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Development of Nickel-Catalyzed Cross-Coupling, Cross-Electrophile Coupling and Domino Reaction

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### Publication Date

2022

Peer reviewed|Thesis/dissertation

UNIVERSITY OF CALIFORNIA,  
IRVINE

Development of Nickel-Catalyzed Cross-Coupling, Cross-Electrophile Coupling, and Domino  
Reactions

DISSERTATION

submitted in partial satisfaction of the requirements for the degree of

DOCTOR OF PHILOSOPHY

in Chemistry

by

Kirsten Anne Hewitt

Dissertation Committee:  
Professor Elizabeth R. Jarvo, Chair  
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2022

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## **DEDICATION**

To Mom, Dad, Kasey, Stephen and Brennan

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## ACKNOWLEDGEMENTS

First, I would like to thank my advisor Liz Jarvo for providing me the opportunity to work in her lab. During my summer rotation before my first year, I realized how supportive and encouraging you were and knew I wanted to pursue my doctoral studies in your group. Your support and encouragement along the way has transformed me into the scientist I am today. Thank you for teaching me how to think critically, write effectively, and be a better scientist.

I would also like to thank my thesis committee, Scott Rychnovsky and Sergey Pronin. Our yearly meetings have helped me tremendously as both a scientist and graduate student. I thank you for your insight into my projects and help tackling tough problems throughout my PhD.

I am also grateful to Alan Heyduk. You have been a wonderful mentor. I have enjoyed teaching both Inorganic and Organometallics with you. Thank you for providing me the opportunity to lecture in the graduate course.

I am also grateful for the wonderful support I had as an undergraduate at University of Pennsylvania. I would like to specifically acknowledge Dr. Sally Mallory who took a chance on an eager freshmen and introduced me to organic chemistry. I am also grateful to Dr. Marisa Kozlowski for allowing me to perform undergraduate research in her lab. Finally, I am grateful to my mentor Dr. Hounk Kang for teaching me laboratory skills and for his patience and kindness.

I acknowledge MDPI for permission to include Chapter 1, which was originally published in *Molecules*. I acknowledge Elsevier for permission to include portions of Chapter 2, which was originally published in *Comprehensive Organometallic Chemistry IV*. I acknowledge the American Chemical Society for permission to include portions of Chapter 3 and Chapter 4, which were originally published in *Journal of Organic Chemistry* and *ACS Catalysis*, respectively. Finally, I am I am thankful to the Allergan graduate research fellowship and UCI department of chemistry dissertation fellowship for providing financial support for my research.

I am thankful to the members of the Jarvo lab both past and present for their support, encouragement, and laughs throughout the last five years. I especially want to thank Erika Lucas for being a great mentor and friend. I learned so much about chemistry, budgeting and everything in between from you and I appreciate everything you have done for me. I also want to acknowledge Amberly Sanford. Thank you for your friendship and advice and I am looking forward to being your coworker again. I am also so grateful for Taylor Thane, my first friend in California. Thank you for being there through all of the ups and downs of graduate school, I couldn't have done it without you. I would also like to thank Patricia Lin for being a great friend, coauthor, and coworker. Thank you for keeping me sane throughout the book chapter, for all the laughs and matching outfits. Finally, I'd like to acknowledge Claire Herbert. It has been an absolute pleasure working with you and watching you grow from an summer rotation student to an accomplished third year graduate student. I cannot wait to see all that you accomplish.

I would like to acknowledge my friends from graduate school, Chuck, Carly, Leah, Tyler, and Angelika. You have all made my graduate school experience so great. I am grateful for all of our

happy hours, dinners, and everything else! I would like to specifically acknowledge Meghan Goulet for being an amazing roommate and friend.

I am also grateful to my friends from Philadelphia, Brianna, Victoria, Nick, Chris, and Joe. Thank you for your constant support and love from 2,000 miles away. I am very excited to be back on the same coast as you all.

To Kasey, Stephen and Brennan, thank you for being my first students when I wanted to play school and for being supportive throughout my life. I am the luckiest girl in the world to have siblings like you.

To my Dad, my first teacher and one of my biggest supporters. Thank you for always answering my questions since I started speaking, for always supporting my dreams and for your endless dad jokes. I am grateful for you every day.

To my Mom, as you say “you only get one” and I am very thankful you are my “one”. I would not be here today without your endless support and love along the way. I appreciate you more than I can put into words.

Finally, to Justin. I cannot even begin to describe the impact you have had on my life. Thank you for your love, your encouragement, your advice and for being you. I am endlessly grateful for you.

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Hewitt, K. A.; Lin, P. C.; Raffman, E.; Jarvo, E. R. C–C Bond Formation Through Cross-Electrophile Coupling. In *Comprehensive Organometallic Chemistry IV*. **2021**. DOI: 10.1016/B978-0-12-820206-7.00092-5



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#### CONFERENCE PRESENTATIONS

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Hewitt, K. A.; Jarvo E. R. Nickel-Catalyzed Domino Cross-Electrophile Coupling Dicarbofunctionalization Reaction of Sulfonamides. Division of Organic Chemistry, Graduate Research Symposium. Albuquerque, NM. November 2021.

Hewitt, K. A.; Jarvo, E. R. Nickel-Catalyzed Cross-Electrophile Coupling and Domino Reactions of Sulfonamides. Empowering Women in Organic Chemistry, Joint Virtual Symposium. October 2021.

Hewitt, K. A.; Lucas, E. L.; Thane, T. A.; Hirbawi, N.; Matus, A. C.; Jarvo, E. R. Nickel-Catalyzed Cross-Electrophile Coupling Reactions of Sulfonamides. Merck SoCal Symposium. Virtual Poster Presentation. July 2021.

Hewitt, K. A.; Jarvo E. R. Nickel-Catalyzed Cross-Electrophile Coupling Reactions of Sulfonamides: Expansion to Domino Reactions. Virtual 262<sup>nd</sup> ACS National Meeting and Exposition. August 2021.

Hewitt, K. A.; Lucas, E. L.; Thane, T. A.; Hirbawi, N.; Matus, A. C.; Jarvo, E. R. Nickel-Catalyzed Cross-Electrophile Coupling Reactions of Sulfonamides: Expansion to Domino Reactions. Empowering Women in Organic Chemistry National Conferences. Virtual Poster Presentation. June 2021.

Hewitt, K. A.; Jarvo E. R. Nickel-Catalyzed Domino Reactions for the Synthesis of Substituted Vinylcyclopropanes. 25<sup>th</sup> Annual Green Chemistry & Engineering Conference. June 2021.

Hewitt, K. A.; Jarvo, E. R. Nickel Catalyzed Domino Reactions of Alkynyl Piperidines. Virtual 261<sup>st</sup> ACS National Meeting and Exposition. April 2021.

Hewitt, K. A.; Lucas, E. L.; Thane, T. A.; Hirbawi, N.; Matus, A. C., Chen, P.-P.; Hong, X.; Jarvo, E. R. Nickel-Catalyzed Cross-Electrophile Coupling Reactions of Sulfonamides. Bristol Meyer Squib Women in Chemistry Day. Virtual Poster Presentation. November 2020.

Hewitt, K. A.; Lucas, E. L.; Castro, A. J.; Jarvo, E. R. Nickel Catalyzed Domino Reactions. 259<sup>th</sup> ACS National Meeting & Exposition, Philadelphia, PA. March 2020.

Hewitt, K. A.; Lucas, E. L.; Jarvo, E. R. Stereospecific Nickel-Catalyzed Cross-Coupling and Cross-Electrophile Couplings of Sulfonamides. 258<sup>th</sup> ACS National Meeting & Exposition, San Diego, CA. August 2019.

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## ABSTRACT OF THE DISSERTATION

Development of Nickel-Catalyzed Cross-Coupling, Cross-Electrophile Coupling, and Domino Reactions

by

Kirsten Anne Hewitt

Doctor of Philosophy in Chemistry

University of California, Irvine, 2022

Professor Elizabeth R. Jarvo, Chair

The world around us, from the medicines we take, to the food we eat, to the plants and animals surrounding us, is composed of carbon-carbon (C–C) bonds. In order to construct these C–C bonds, we rely on previously developed methodologies. In an ever growing world, the need for new technologies to tackle more challenging problems has grown significantly. Catalysis has emerged in the last century as a reliable, efficient and sustainable method to synthesize C-C bonds. In particular, first row transition-metal catalysis has stood out for its unique reactivity and interesting mechanistic pathways. Herein, four nickel-catalyzed transformations that provide highly complex carbon scaffolds will be discussed. In addition, key mechanistic features will be examined.

First, a nickel-catalyzed Kumada cross-coupling reaction of benzylic sulfonamides will be discussed (Chapter 1). A significant by-product that was initially observed was substituted styrenes resulting from  $\beta$ -hydride elimination. In order to address this problem, we synthesized highly branched substrates and this diminished the formation of the undesired by-product. In addition, the scope of the includes heterocycles and alternative Grignard reagents.

Next, cross-electrophile coupling reactions of benzylic, allylic and propargylic sulfonamides will be presented (Chapter 2–4). These strategies allow for the synthesis of highly strained cyclopropanes in a diastereoselective manner. During the mechanistic investigation, we discovered that catalytic turnover was rate determining and we hypothesized that known elementary steps could be inserted to develop a domino reaction. To this end, we established a domino reaction of propargylic sulfonamides that involved discrete cross-electrophile coupling and dicarbofunctionalization to afford tetrasubstituted vinylcyclopropanes. Based on experimental and mechanistic experiments, we proposed a bifurcated mechanism that produces both diastereomers of product.

Finally, a conjunctive cross-electrophile coupling reaction involving mesylate electrophiles and an internal olefin will be discussed (Chapter 5). This methodology builds on our laboratory's previously developed cross-electrophile coupling reaction 1,3-dimesylates. The scope of the reaction contains electron-withdrawing and electron-donating functional groups. Additionally, we performed preliminary mechanistic experiments to demonstrate that the mechanism likely generates diiodides in-situ, initiates at the secondary center and proceeds through radical intermediates.

## Nickel-Catalyzed Kumada Cross-Coupling Reaction of Benzylic Sulfonamides

### 1.1 Introduction

Transition-metal catalyzed cross-coupling (XC) reactions have transformed modern synthetic organic chemistry by creating an arsenal of carbon-carbon bond forming reactions.<sup>1,2</sup> The significance of these transformations was highlighted when Richard Heck, Akira Suzuki, and Ei-ichi Negishi were awarded the 2010 Nobel Prize in chemistry for their work on palladium-catalyzed XC reactions.<sup>3</sup> In the past several decades, nickel complexes have emerged as a powerful and efficient transition metal catalyst with comparable reactivity to palladium.<sup>4</sup> One major advantage is that nickel is more cost effective than palladium and platinum. In its elemental form nickel costs approximately 2,000 times less than palladium and 10,000 less than platinum.<sup>5</sup> Additionally, nickel is more electropositive than its d<sup>10</sup> counterparts which allows for more facile oxidative addition.<sup>6</sup> Consequently, electrophiles that are considered unreactive under palladium catalysis can now be activated via oxidative addition with nickel. Moreover, nickel and palladium complexes display differing reactivity towards  $\beta$ -hydride elimination. This elementary step typically leads to unwanted side products in catalytic reactions and tends to be slower for nickel

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<sup>1</sup> Portions of this chapter have been published in *Molecules*: Hewitt, K. A.; Herbert, C. A.; Matus, A. C.; Jarvo, E. R. *Molecules* **2021**, *26*, 5947–5971.

<sup>2</sup> For reviews on XC reactions see: (a) *Metal-Catalyzed Cross-Coupling Reactions*. 2nd ed. De Meijere, A. Diederich, F., Eds.; Wiley-VCH: Weinheim, Germany **2004**. (b) Hartwig, J. F. *Organotransition Metal Chemistry: From Bonding to Catalysis*; University Science Books. Sausalito, CA, **2010**. (c) Jana, R.; Pathak, T. P.; Sigman, M. S. *Chem. Rev.* **2011**, *111*, 1417–1492. (d) Cherney, A. H.; Kadunce, N. T.; Reisman, S. E. *Chem. Rev.* **2015**, *115*, 9587–9652. (e) Choi, J.; Fu, G. C. *Science*, **2017**, *356*, eaaf7230. (f) Campeau, L.-C.; Hazari, N. *Organometallics*, **2019**, *38*, 3–35.

<sup>3</sup> Seechurn, C. C. C.; Kitching, M. O.; Colacot, T. J.; Snieckus, V. *Angew. Chem. Int. Ed.* **2012**, *51*, 5062–5085.

<sup>4</sup> (a) Tamaru, Y. *Modern Organonickel Chemistry*; Wiley-VCH: Weinheim, Germany, 2005. (b) Ogoshi, S. (Ed.) *Nickel Catalysis in Organic Synthesis: Methods and Reactions*; Wiley: Hoboken, NJ, USA, 2020. (c) Singer, R. A.; Monfette, S.; Bernhardson, D.; Tcyrulnikov, S.; Hubbell, A. K.; Hansen, E. C. *Org. Process. Res. Dev.* **2021**, *25*, 1802–1815.

<sup>5</sup> Tasker, S. Z.; Standley, E. A.; Jamison, T. F. *Nature* **2014**, *509*, 299–309.

<sup>6</sup> Tsou, T. T.; Kochi, J. K. *J. Am. Chem. Soc.* **1979**, *101*, 6319–6332.

than for palladium.<sup>7</sup> Finally, nickel exhibits additional modes of reactivity compared to palladium because it can easily access oxidation states ranging from 0–IV whereas palladium typically accesses the 0 and II oxidation states. Common catalytic cycles for nickel include both polar and radical mechanisms.<sup>5</sup>

Intense research efforts have been employed in the development of nickel-catalyzed XC reactions of sluggish electrophiles, such as aryl and alkyl halides, sulfonates, esters, and ethers.<sup>2,8</sup> However, the XC reaction of alkyl amine derivatives has remained a significant challenge.<sup>9</sup> Historically, in order to facilitate nickel-catalyzed reactions, activation of these carbon-nitrogen bonds has been achieved via incorporation into strained aziridine rings, transformation to ammonium salts or activation of amides.<sup>10</sup>

Ring-strain promoted XC of aziridines has been accomplished (Scheme 1.1).<sup>11</sup> Early stoichiometric work by Hillhouse established that aziridines undergo facile oxidative addition with nickel complexes.<sup>12</sup> Catalytic Negishi reactions of sulfonylaziridines have subsequently been established. The Doyle laboratory reported a regioselective Negishi XC reaction of styrenyl aziridines with alkylzinc reagents with substitution at the benzylic position (Scheme 1.1a).<sup>13</sup> Key to their success was the use of an electron deficient fumarate ligand.<sup>14</sup> Shortly thereafter, the Doyle

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<sup>7</sup> (a) Koga, N.; Obara, S.; Kitaura, K.; Morokuma, K. *J. Am. Chem. Soc.* **1985**, *107*, 7109–7116. (b) Lin, B.-L.; Liu, L.; Fu, Y.; Luo, S.-W.; Chen, Q.; Gui, Q.-X. *Organometallics* **2004**, *23*, 2114–2123.

<sup>8</sup> For selected reviews, see: (a) Rosen, B. M.; Quasdorf, K. W.; Wilson, D. A.; Zhang, N.; Resmerita, A.-M.; Garg, N. K.; Percec, V. *Chem. Rev.* **2011**, *111*, 1346–1416. (b) Tollefson, E. J.; Hanna, L. E.; Jarvo, E. R. *Acc. Chem. Res.* **2015**, *48*, 2344–2353. (c) Yu, D.-G.; Li, B.-J.; Shi, Z.-J. *Acc. Chem. Res.* **2010**, *43*, 1486–1495.

<sup>9</sup> For recent reviews, see (a) Ouyang, K.; Hao, W.; Zhang, W.-X.; Xi, Z. *Chem. Rev.* **2015**, *115*, 12045–12090. (b) Wang, W.; Su, Y.; Li, L.; Huang, H. *Chem. Soc. Rev.* **2016**, *45*, 1257–1272. (c) Pound, S. M.; Watson, M. P. *Chem. Commun.* **2018**, *54*, 12286–12301. (d) Dander, J. E.; Garg, N. K. *ACS Catal.* **2017**, *7*, 1413–1423. (e) Li, G.; Ma, S.; Szostak, M. *Trends Chem.* **2020**, *2*, 914–928.

<sup>10</sup> For an example of a stereoablative copper-catalyzed Kumada XC reaction of sulfonimides, see: Li, M.-B.; Tang, X.-L.; Tian, S.-K. *Adv. Synth. Catal.* **2011**, *353*, 1980–1984.

<sup>11</sup> Huang, C.-Y.; Doyle, A. G. *Chem. Rev.* **2014**, *114*, 8153–8198.

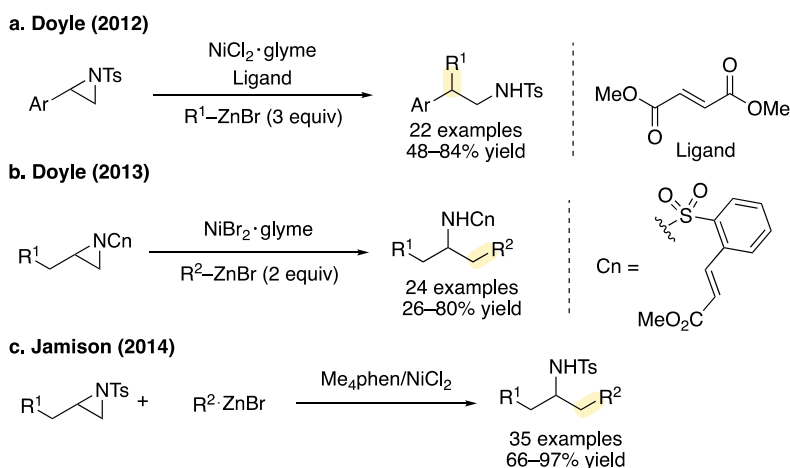
<sup>12</sup> Lin, B. L.; Clough, C. R.; Hillhouse, G. L. *J. Am. Chem. Soc.* **2002**, *124*, 2890–2891.

<sup>13</sup> (a) Huang, C.-Y.; Doyle, A. G. *J. Am. Chem. Soc.* **2012**, *134*, 9541–9544. (b) Huang, C.-Y.; Doyle, A. G. *J. Am. Chem. Soc.* **2015**, *137*, 5638–5641

<sup>14</sup> Estrada, J. G.; Williams, W. L.; Ting, S. I.; Doyle, A. G. *J. Am. Chem. Soc.* **2020**, *142*, 8928–8937.

and Jamison groups independently described a regioselective Negishi XC reaction of alkyl aziridines with alkylzinc reagents to forge the desired carbon-carbon bond (Scheme 1.1b and c).<sup>15</sup> The differing regioselectivity of these reactions can be explained by comparing the oxidative addition events of the C–N bonds. Styrenyl aziridines preferentially undergo oxidative addition at the benzylic center to afford  $\eta^3$ -benzylnickel complexes. In contrast, alkyl aziridines, which do not contain an aromatic ring to direct the nickel complex, preferentially undergo oxidative addition at the less hindered position.<sup>12</sup> These reports demonstrate the ability to activate the C–N bond in strained rings.

### Scheme 1.1 Cross-Coupling (XC) Reactions of Aziridines



Development of XC reactions of acyclic benzylamine derivatives has relied upon formation of highly reactive electrophiles (i.e. charged ammonium salts).<sup>16</sup> The earliest reports of carbon-nitrogen XC coupling reactions utilized aniline derivatives (Scheme 1.2). In a seminal report, the Wenkert laboratory disclosed the Kumada XC reaction of aryl trimethylammonium salts with Grignard reagents (Scheme 1.2a).<sup>17</sup> In 2003, the MacMillan group reported the Suzuki-Miyaura

<sup>15</sup> (a) Nielsen, D. K.; Huang, C.-Y.; Doyle, A. G. *J. Am. Chem. Soc.* **2013**, *135*, 13605–13609. (b) Jensen, K. L.; Standley, E. A.; Jamison, T. F. *J. Am. Chem. Soc.* **2014**, *136*, 11145–11152.

<sup>16</sup> For a recent review see: Wang, Z.-X.; Yang, B. *Org. Biomol. Chem.* **2020**, *18*, 1057.

<sup>17</sup> Wenkert, E.; Han, A.-L.; Jenny, C.-J. *J. Chem. Soc., Chem. Commun.* **1998**, 975–976.

XC reaction of a similar trimethylammonium derivative.<sup>18</sup> The modifications implemented included the utilization of a weakly coordinating counterion (i.e. triflate) and the use of boronic acids as the organometallic reagent, which significantly improved the yield and functional group tolerance of the reaction (Scheme 1.2b). In 2011, the Wang laboratory developed Negishi XC reactions of aryl trimethylammonium iodides with aryl zinc reagents (Scheme 1.2c).<sup>19</sup> This reaction displayed similar functional group tolerance to the Suzuki-Miyaura XC reaction and proceeded in moderate to excellent yields. The Wang group has extensively studied this reaction and have developed an effective pincer ligand for the nickel-catalyzed XC reactions.<sup>20</sup> Additionally, it has been demonstrated that trimethylammonium triflates were tolerated under these reaction conditions.<sup>21</sup> More recently, Uchiyama and coworkers described the Stille XC reaction of aryl ammonium salts with aryl trimethyl tin reagents (Scheme 1.2d).<sup>22</sup> Finally, the Uchiyama and Wang groups independently demonstrated the XC reaction of trimethylammonium salts with organoaluminium reagents (Scheme 1.2e).<sup>23</sup>

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<sup>18</sup> Blakey, S. B.; MacMillan, D. W. C. *J. Am. Chem. Soc.* **2003**, *125*, 6046–6047.

<sup>19</sup> Xie, L.-G.; Wang, Z.-X. *Angew. Chem. Int. Ed.* **2011**, *50*, 4901–4904.

<sup>20</sup> (a) Zhang, X.-Q.; Wang, Z.-X. *J. Org. Chem.* **2012**, *77*, 3658–3663. (b) Yang, X.; Wang, Z.-X. *Organometallics* **2014**, *33*, 5863–5873.

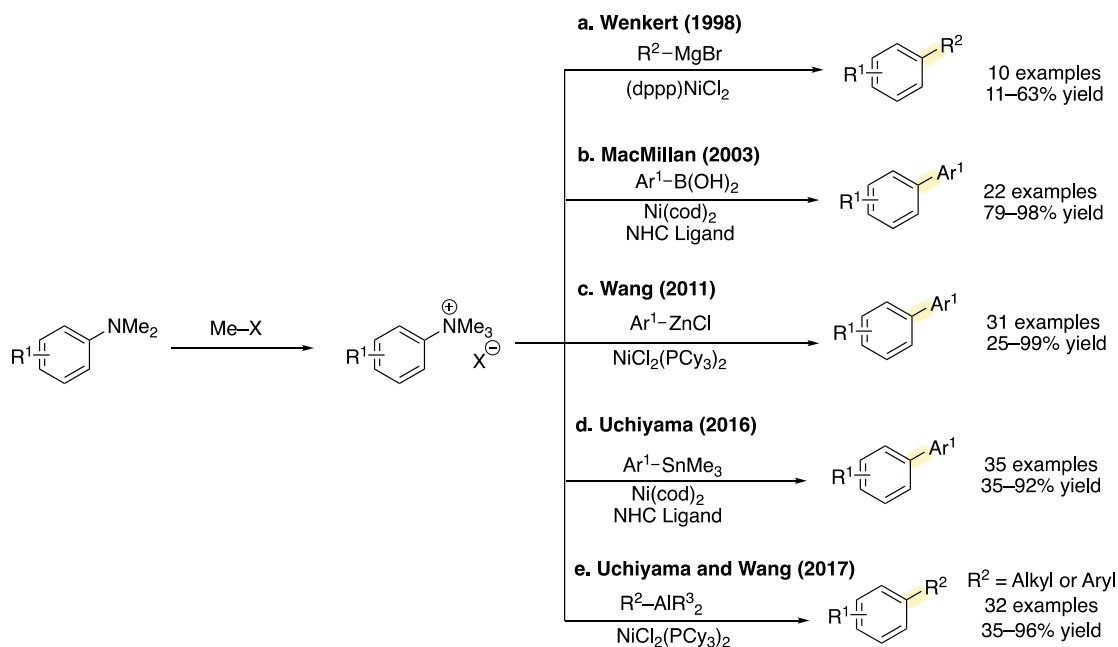
<sup>21</sup> Wu, D.; Tao, J.-L.; Wang, Z.-X. *Org. Chem. Front.* **2015**, *2*, 265–273.

<sup>22</sup> Wang, D.-Y.; Kawahata, M.; Yang, Z.-K.; Miyamoto, K.; Komagawa, S.; Yamaguchi, K.; Wang, C.; Uchiyama, M. *Nat. Commun.* **2016**, *7*, 12937.

<sup>23</sup> (a) Ogawa, H.; Yang, Z.-K.; Minami, H.; Kojima, K.; Saito, T.; Wang, C.; Uchiyama, M. *ACS Catal.* **2017**, *7*, 3988–3994. (b) He, F.; Wang, Z.-X. *Tetrahedron* **2017**, *73*, 4450–4457.



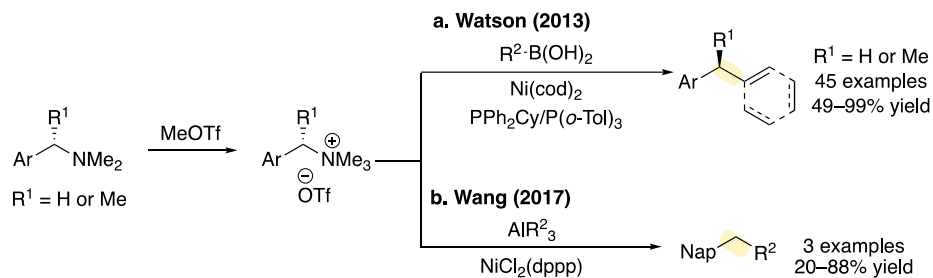
## Scheme 1.2 Cross-Coupling Reactions of Aryl Trimethylammonium Salts



The extension of XC reactions to include alkyl electrophiles has proven difficult due to unwanted  $\beta$ -hydride elimination of the alkylmetal species. One particularly successful strategy has been the implementation of substituted benzylamines. For example, the Watson laboratory demonstrated that benzylic trimethylammonium salts are competent electrophiles in Suzuki-Miyaura XC reactions with aryl and vinylboronic acids (Scheme 1.3a).<sup>24</sup> Similarly, the Wang laboratory disclosed the XC reaction of benzylic trimethylammonium salts with organoaluminum reagents to forge the desired carbon-carbon bond (Scheme 1.3b).<sup>23b</sup>

<sup>24</sup>(a) Maity, P.; Shacklady-McAtee, D. M.; Yap, G. P. A.; Sirianni, E. R.; Watson, M. P. *J. Am. Chem. Soc.* **2013**, *135*, 280–285. (b) Shacklady-McAtee, D. M.; Roberts, K. M.; Basch, C. H.; Song, Y.-G.; Watson, M. P. *Tetrahedron* **2014**, *70*, 4257–4263.

**Scheme 1.3** Cross-Coupling Reactions of Trimethylammonium salts.



The use of Katritzky salts to activate amines has proven to be sufficient for activation of benzylic and alkyl amines for Suzuki-Miyaura and Negishi XC reactions. Previously, it has been observed that Katritzky salts participate in  $S_N2$ , radical, and Minisci-type reactions, and in recent years, many transition-metal catalyzed reactions have been developed.<sup>25,26</sup> The Watson laboratory hypothesized that these air and moisture stable salts would be suitable electrophiles in a XC reaction.<sup>27</sup> To test their hypothesis, primary amines were converted to Katritzky salts via a condensation reaction with 2,4,6-triphenylpyrylium tetrafluoroborate and the corresponding salts were subjected to Suzuki-Miyaura XC reactions with aryl boronic acids. The desired cross-coupled products were obtained in good yields (Scheme 1.4a).<sup>28</sup> This strategy was amenable to the coupling of primary benzylic Katritzky salts as well (Scheme 1.4b).<sup>29</sup> Additionally, vinyl boranes and alkylborane reagents, generated in situ by hydroboration of alkenes, participated in XC with

<sup>25</sup> (a) Bapat, J. B.; Blade, R. J.; Boulton, A. J.; Epszajn, J.; Katritzky, A. R.; Lewis, J.; Molina-Buendia, P.; Nie, P.-L.; Ramsden, C. A. *Tet. Lett.* **1976**, *17*, 2691–2694. (b) Katritzky, A. R.; De Ville, G.; Patel, R. C. *Tetrahedron* **1981**, *37*, 25–30. (c) Katritzky, A. R.; Marson, C. M. *Angew. Chem. Int. Ed.* **1984**, *23*, 420–429. (d) Said, S. A.; Fiksdahl, A. *Tetrahedron: Asymmetry* **2001**, *12*, 1947–1951. (e) Klauck, F. J.; James, M. J.; Glorius, F. *Angew. Chem. Int. Ed.* **2017**, *56*, 12336–12339.

<sup>26</sup> For reviews on transition metal-catalyzed reactions with Katritzky salts see (a) He, F.-S.; Ye, S.; Wu, J. *ACS Catal.* **2019**, *9*, 8943–8960. (b) Pang, Y.; Moser, D.; Cornella, J. *Synthesis* **2020**, *52*, 489–503. (c) Rössler, S. L.; Jelier, B. J.; Magnier, E.; Dagousset, G.; Carreira, E. M.; Togni, A. *Angew. Chem. Int. Ed.* **2020**, *59*, 9264–9280. (d) Li, Y.-N.; Xiao, F.; Guo, Y.; Zeng, Y.-F. *Eur. J. Org. Chem.* **2021**, *2021*, 1215–1228. (e) Kong, D.; Moon, P. J.; Lundgren, R. J. *Nat. Catal.* **2019**, *2*, 473–476

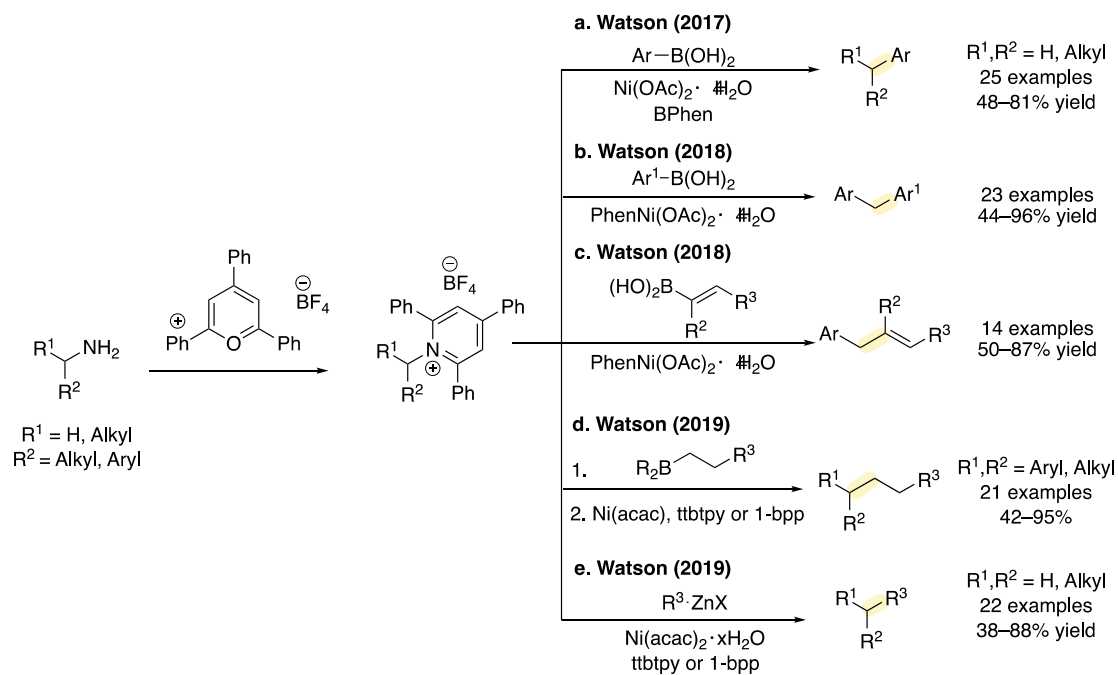
<sup>27</sup> Basch, C. H.; Liao, J.; Xu, J.; Pian, J. J.; Watson, M. P. *J. Am. Chem. Soc.* **2017**, *139*, 5313–5316.

<sup>28</sup> Hoerner, M. E.; Baker, K. M.; Basch, C. H.; Bampo, E. M.; Watson, M. P. *Org. Lett.* **2019**, *21*, 7356–7360.

<sup>29</sup> Liao, J.; Guan, W.; Boscoe, B. P.; Tucker, J. W.; Tomlin, J. W.; Garnsey, M. R.; Watson, M. P. *Org. Lett.* **2018**, *20*, 3030–3033.

Katritzky salts (Scheme 1.4c and d).<sup>30</sup> This strategy has been extended beyond Suzuki-Miyaura reactions to include Negishi XC reactions with alkylzinc reagents (Scheme 1.4e).<sup>31</sup>

### Scheme 1.4. Cross-Coupling Reactions of Katritzky Salts



These methods establish strain- and charge-based strategies to activate amines for use as the electrophilic partner in XC reactions. However the requirement for aziridines or functionalization as highly reactive ammonium salts remains a major limitation in broad application of these methods. In this Chapter, we report the first nickel-catalyzed Kumada XC reaction of simple benzylic sulfonamides with methylmagnesium iodide (Scheme 1.5). Previously, the Jarvo laboratory disclosed the Kumada XC reaction of benzylic ethers which proceeded in excellent yields, and enantio- and diastereoselectivity.<sup>8b,32</sup> Building on this work, we aimed to develop an analogous reaction that employed benzylic sulfonamides. Ethers and sulfonamides

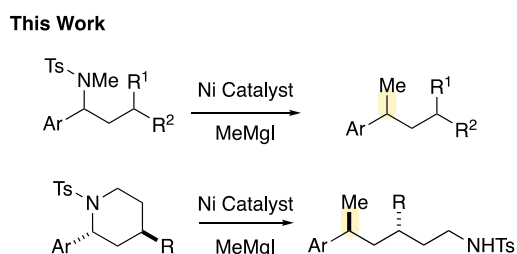
<sup>30</sup> (a) Baker, K. M.; Baca, D. L.; Plunkett, S.; Daneker, M. E.; Watson, M. P. *Org. Lett.* **2019**, *21*, 9738–9741. (b) Guan, W.; Liao, J.; Watson, M. P. *Synthesis* **2018**, *50*, 3231–3237.

<sup>31</sup> Plunkett, S.; Basch, C. H.; Santana, S. O.; Watson, M. P. *J. Am. Chem. Soc.* **2019**, *141*, 2257–2262.

<sup>32</sup> (a) Taylor, B. L. H.; Swift, E. C.; Waetzig, J. D.; Jarvo, E. R. *J. Am. Chem. Soc.* **2011**, *133*, 389–391. (b) Tollefson, E. J.; Dawson, D. D.; Osborne, C. A.; Jarvo, E. R. *J. Am. Chem. Soc.* **2014**, *136*, 14951–14958.

have similar leaving group abilities, as the conjugate bases have similar pK<sub>a</sub>'s, and we hypothesized sulfonamides would behave similarly to ethers in a XC reaction.<sup>33,34</sup> In addition, these moieties are appealing because they are common functional groups in synthesis. Furthermore, we demonstrate that sulfonamides undergo stereospecific XC reactions, in contrast to the stereoablative reactivity typically observed with styrenyl aziridines and Katritzky salts.<sup>35,36,37,38</sup> This stereospecific manifold allows for rapid diastereoselective construction of acyclic fragments bearing 1,3-substitution.<sup>39</sup>

### Scheme 1.5 Kumada Cross-Coupling Reactions of Benzylic Sulfonamides



## 1.2. Results and Discussion

### 1.2.1 Optimization of Kumada XC Reaction of Benzylic Sulfonamides

We began our investigation into the Kumada XC reaction with benzylic sulfonamide **1.1**, which was synthesized in three steps from the commercially available aldehyde.<sup>40</sup> Previously,

<sup>33</sup> Marshall, D. R.; Thomas, P. J.; Stirling, C. J. M. *J. Chem. Soc., Chem. Commun.* **1975**, 940–941.

<sup>34</sup> We have demonstrated that sulfonamides and ethers behave similarly in XEC reactions, see: Lucas, E. L.; Hewitt, K. A.; Chen, P.-P.; Castro, A. J.; Hong, X.; Jarvo, E. R. *J. Org. Chem.* **2020**, *85*, 1775–1793.

<sup>35</sup> Stereochemical outcome with styrenyl aziridines, see: ref. 13.

<sup>36</sup> For representative mechanistic experiments for XEC reactions of styrenyl aziridines which are proposed to proceed through alkyl iodides, see: Steinman, T. J.; Liu, J.; Mengiste, A.; Doyle, A. G. *J. Am. Chem. Soc.* **2020**, *142*, 7598–7605.

<sup>37</sup> Stereochemical outcome with Katritzky ammonium salts, see: Teyrulnikov, S.; Cai, Q.; Twitty, J. C.; Xu, J.; Atifi, A.; Bercher, O. P.; Yap, G. P. A.; Rosenthal, J.; Watson, M. P.; Kozlowski, M. C. *ACS Catal.* **2021**, *11*, 8456–8466. (b) ref 26

<sup>38</sup> Reactions of ammonium salts can be stereospecific or stereoablative: (a) Xu, J.; Bercher, O. P.; Talley, M. R.; Watson, M. P. *ACS Catal.* **2021**, *11*, 1604–1612. (b) Moragas, T.; Gaydou, M.; Martin, R. *Angew. Chem. Int. Ed.* **2016**, *55*, 5053–5057

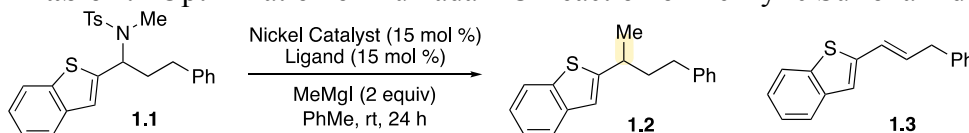
<sup>39</sup> (a) Hanessian, S.; Giroux, S.; Mascitti, V. *Synthesis* **2006**, *7*, 1057–1076. (b) Chen, R.; Shen, Y.; Yang, S.; Zhang, Y. *Angew. Chem. Int. Ed.* **2020**, *59*, 14198–14210.

<sup>40</sup> See Experimental Section for substrate synthesis.

Kumada XC reactions of benzylic ethers employed Ni(cod)<sub>2</sub> and racemic BINAP as the optimal reaction conditions.<sup>8b,32</sup> Under these conditions, we were excited to observe 25% yield of the desired cross-coupled product **1.2** (Table 1.1, entry 1). Increasing the catalyst loading to 15 mol % improved the yield of the reaction (entry 2). However, it also increased the yield of the undesired styrene product **1.3** arising from β-hydride elimination. In an effort to improve the ratio between desired product **1.2** and styrene product **1.3**, we investigated a series of bidentate phosphine, NHC, and pyridine ligands. DPEPhos improved the yield of **1.2** and decreased the amount of styrene **1.3** (entry 3). However, all other ligands evaluated did not improve the yield of **1.2** (entries 4–7).

We next investigated an alternative precatalyst. Previously, the Jarvo laboratory reported the cross-electrophile coupling (XEC) reaction of benzylic and allylic sulfonamides which employed a BINAP-ligated nickel (II) precatalyst.<sup>34,41</sup> Utilizing these conditions, with 15 mol % of catalyst, we were delighted to observe the desired product in 54% yield and 40% yield of styrene **1.3**. We elected to proceed with the nickel (II) precatalyst as it provided the desired product in the highest yield.

**Table 1.1** Optimization of Kumada XC Reaction of Benzylic Sulfonamides



Entry	Nickel Catalyst	Ligand	Yield <b>1.2</b> (%) <sup>a</sup>	Yield <b>1.3</b> (%) <sup>a</sup>	RSM <b>1.1</b> (%) <sup>a</sup>
1 <sup>b</sup>	Ni(cod) <sub>2</sub>	<i>rac</i> -BINAP	25	10	19
2	Ni(cod) <sub>2</sub>	<i>rac</i> -BINAP	34	30	7
3	Ni(cod) <sub>2</sub>	DPEPhos	42	20	0
4	Ni(cod) <sub>2</sub>	XantPhos	0	<5	37
5	Ni(cod) <sub>2</sub>	dppe	0	<5	65
6	Ni(cod) <sub>2</sub>	SiMes-BF <sub>4</sub>	12	0	86
7	Ni(cod) <sub>2</sub>	BPhen	0	0	61
8	( <i>R</i> -BINAP)NiCl <sub>2</sub>	–	54	40	0

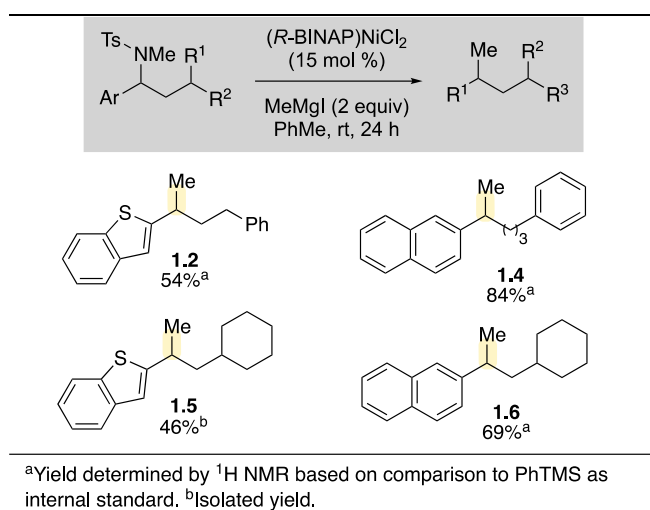
<sup>a</sup>Yield of **1.2** and **1.3** and Recovered Starting Material (RSM) determined by <sup>1</sup>H NMR based on comparison to PhTMS as internal standard. <sup>b</sup>5 mol % Ni(cod)<sub>2</sub>

<sup>41</sup> For the original synthesis and characterization of (*R*-BINAP)NiCl<sub>2</sub> see: (a) Standley, E. A.; Smith, S. J.; Müller, P.; Jamison, T. F. *A Organometallics* **2014**, *33*, 2012–2018. (b) Dawson, D. D.; Oswald, V. F.; Borovik, A. S.; Jarvo, E. R. *Chem. Eur. J.* **2020**, *26*, 3044–3048.

## 1.2.2 Evaluation of the Substrate Scope of the Kumada XC Reaction

With optimized conditions in hand, we evaluated the scope of the Kumada XC reaction (Scheme 1.6).<sup>42</sup> Naphthyl substrates were well tolerated under the standard reaction conditions and product **1.4** was observed in 84% yield. Notably, products such as **1.5** and **1.6** with branching at the  $\beta$ -position provided good yields of cross-coupled products with lesser amounts of styrenes formed from  $\beta$ -hydride elimination (20–30%) when compared to product **1.2**. We hypothesized that this increase in steric bulk destabilized the conformation necessary for  $\beta$ -hydride elimination to proceed.

**Scheme 1.6** Scope of the Kumada XC Reaction of Acyclic Sulfonamides.

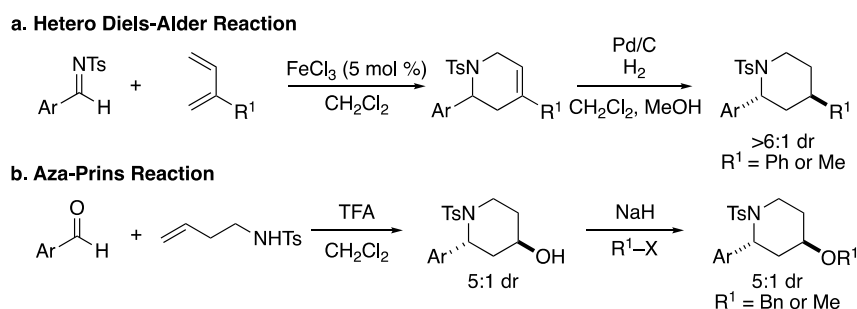


We also sought to evaluate a series of arylpiperidines, with the expectation that a stereospecific XC reaction at the benzylic position would provide synthetic access to highly substituted acyclic fragments. Furthermore, products would bear a pendant sulfonamide moiety, available for subsequent functionalization.<sup>34</sup> Rapid synthesis of the requisite cyclic sulfonamides

<sup>42</sup> Substrates **1.4–1.6** synthesized by Alissa C. Matus.

was achieved by hetero Diels-Alder (HDA) cycloadditions or aza-Prins reactions.<sup>43,44</sup> For substrates with alkyl substituents in the 4-position, [4+2] HDA reactions provided the requisite starting materials (Scheme 1.7a). For substrates bearing ether groups in the 4-position, an aza-Prins reaction provided the requisite 2-aryl-4-hydroxypiperidine that could be subsequently methylated or benzylated. (Scheme 1.7b).

**Scheme 1.7** Arylpiperidine Synthesis via (a) Hetero Diels-Alder (HDA) Reaction and (b) Aza-Prins Reaction.



With rapid and diastereoselective access to the desired piperidines, we examined these cyclic substrates in ring-opening Kumada XC reactions (Scheme 1.8).<sup>45</sup> Phenyl and methyl substituents (products **1.7** and **1.8**) were well tolerated and minimal amounts (<5%) of  $\beta$ -hydride elimination were observed. Methylated and benzylated ethers were well tolerated and provided the desired products in good yields (**1.9**, **1.10**, and **1.11**).<sup>46</sup> It is important to note that the diastereomeric ratio observed in the products is consistent with the diastereomeric ratio of the starting material.<sup>47</sup> Therefore, preliminary data supports a stereospecific Kumada XC reaction.

<sup>43</sup> Tomifuji, R.; Maeda, K.; Takahashi, T.; Kurahashi, T.; Mastubara, S. *Org. Lett.* **2018**, *20*, 7474–7477.

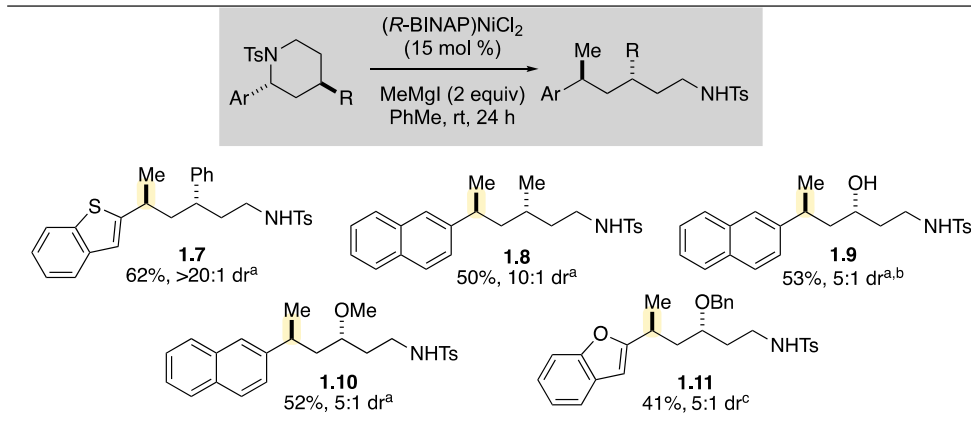
<sup>44</sup> (a) Sabitha, G.; Reddy, N. M.; Prasad, M. N.; Yadav, J. S. *Helv. Chim. Acta* **2009**, *92*, 967–976. (b) Tollefson, E. J.; Erickson, L. W.; Jarvo, E. R. *J. Am. Chem. Soc.* **2015**, *137*, 9760–9763.

<sup>45</sup> Substrates **1.8–1.11** synthesized by Claire A. Herbert.

<sup>46</sup> Deprotection of benzyl group of substrate **9** was accomplished under the standard reaction conditions. For a previous report of benzyl deprotection with a Grignard reagent see: Kawana, M. *Chem. Lett.* **1981**, 1541–1542.

<sup>47</sup> See Experimental Details section for more information.

### Scheme 1.8 Scope of the Kumada XC Reaction of Cyclic Sulfonamides.



<sup>a</sup>Isolated yield.

<sup>b</sup>R = OBn in starting material and provided the free alcohol in product.

<sup>c</sup>Yield determined by <sup>1</sup>H NMR based on comparison to PhTMS as internal standard.

To further develop the potential scope of this reaction, we sought to establish ring-opening of a sulfonyl piperidine with an aryl Grignard reagent (Scheme 1.9). Such transformations would provide synthetic access to diarylalkanes bearing pendant sulfonamides, including rapid assembly of stereochemically-rich analogs of ATPase inhibitor **1.14**.<sup>48,49</sup> We have previously observed that in Kumada XC reactions of benzylic ethers employing aryl Grignard reagents, the optimal nickel catalyst is ligated by dppe.<sup>50</sup> We were pleased to see that this trend applied to benzylic

<sup>48</sup> Pöhler, R.; Krahn, J. H.; van den Boom, J.; Dobrynin, G.; Kaschani, F.; Eggenweiler, H.-M.; Zenke, F. T.; Kaiser, M.; Meyer, H. *Angew. Chem. Int. Ed.* **2018**, *57*, 1576–1580.

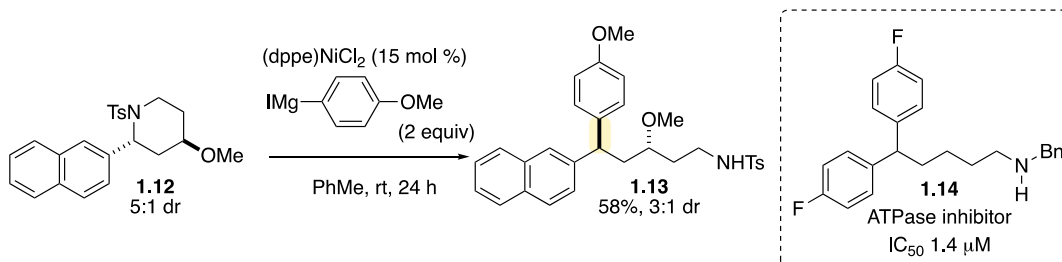
<sup>49</sup> Diarylalkanes are known to exhibit potent biological activity. For selected references see: (a) Wetterau, J. R.; Gregg, R. E.; Harrity, T. W.; Arbeeny, C.; Cap, M.; Connolly, F.; Chu, C.-H.; George, R. J.; Gordon, D. A.; Jamil, H.; Jolibois, K. G.; Kunselman, L. K.; Lan, S.-J.; Maccagnan, T. J.; Ricci, B.; Yan, M.; Young, D.; Chen, Y.; Fryszman, O. M.; Logan, J. V. H.; Musial, C. L.; Poss, M. A.; Robl, J. A.; Simpkins, L. M.; Slusarchyk, W. A.; Sulsky, R.; Taunk, P.; Magnin, D. R.; Tino, J. A.; Lawrence, R. M.; Dickson, J. K.; Biller, S. A. *Science* **1998**, *282*, 751. (b) Kimura, M.; Masuda, T.; Yamada, K.; Mitani, M.; Kubota, N.; Kawakatsu, N.; Kishii, K.; Inazu, M.; Kiuchi, Y.; Oguchi, K.; Namiki, T. *Bioorg. Med. Chem.* **2003**, *11*, 1621–1630. (c) Dei, S.; Coronello, M.; Bartolucci, G.; Manetti, D.; Romanelli, M. N.; Udomtanakunchai, C.; Salerno, M.; Teodori, E. *Eur. J. Med. Chem.* **2018**, *147*, 7–20. (d) Ameen, D.; Snape, T. J. *MedChemComm* **2013**, *4*, 893–907.

<sup>50</sup> Yonova, I. M.; Johnson, A. G.; Osborne, C. A.; Moore, C. E.; Morrisette, N. S.; Jarvo, E. R. *Angew. Chem. Int. Ed.* **2014**, *53*, 2422–2427.



sulfonamides: employing the commercially available precatalyst, (dppe)NiCl<sub>2</sub>, the XC reaction proceeded smoothly to provide the desired product **1.13** in 58% isolated yield.<sup>51,52</sup>

### Scheme 1.9 Kumada XC Reaction with Aryl Grignard Reagent



### 1.3 Proposed Mechanism of the Kumada XC Reaction

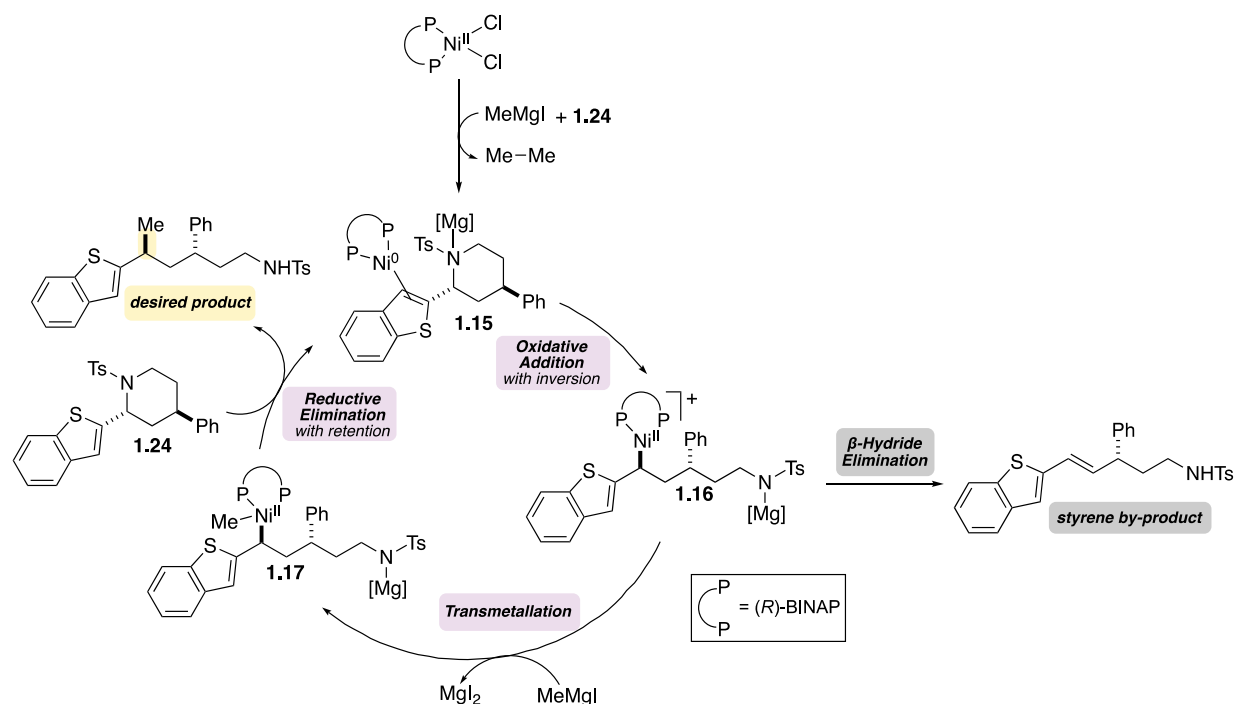
We propose the following catalytic cycle for the Kumada XC reaction based on related mechanisms for the Kumada XC reaction of benzylic ethers and the XEC reaction of benzylic sulfonamides (Scheme 1.10).<sup>34,53</sup> First, reduction of the nickel(II) precatalyst with the Grignard reagent provides the substrate ligated active Ni(0) catalyst **1.15**. Next, oxidative addition of the benzylic sulfonamide affords the Ni(II) intermediate **1.16**. Based on the calculated reaction coordinate diagram and transition state energies for related transformations, we hypothesize that rate-determining oxidative addition occurs with inversion of the benzylic carbon.<sup>8b,32,53</sup> This step is facilitated by Lewis acidic magnesium salts that activate the sulfonamide moiety. Transmetalation with the Grignard reagent provides alkylnickel complex **1.17**. Subsequent reductive elimination, which occurs with retention at the benzylic center, affords the desired product and turns over the catalyst. Alternatively, intermediate **1.16** can undergo β-hydride elimination to afford the observed styrene by-product.

<sup>51</sup> Experiment performed by Claire A. Herbert.

<sup>52</sup> When (*R*-BINAP)NiCl<sub>2</sub> was employed as the precatalyst, the desired product was not observed and 44% of starting material was recovered. For more information, see ref. 1.

<sup>53</sup> Chen, P.-P.; Lucas, E. L.; Greene, M. A.; Zhang, S.; Tollefson, E. J.; Erickson, L. W.; Taylor, B. L.; Jarvo, E. R.; Hong, X. *J. Am. Chem. Soc.* **2019**, *141*, 5835–5855.

**Scheme 1.10** Proposed Mechanism of Kumada XC Reaction.



## 1.4 Conclusions

In conclusion, we have developed a Kumada XC reaction of benzylic sulfonamides with Grignard reagents including methylmagnesium iodide and arylmagnesium iodide. This reaction utilizes readily available starting materials that are not activated prior to the XC reaction. We have demonstrated that increasing the steric bulk adjacent to the reactive center destabilizes the conformation necessary for  $\beta$ -hydride elimination to occur. A stereospecific ring opening Kumada XC reaction has been established to synthesize highly substituted acyclic fragments. This work provides a basis for the XC reaction of simple benzylic sulfonamides.

## 1.5 Experimental Details

### 1.5.1 General Procedures

All reactions were carried out under an atmosphere of  $N_2$ , or Ar when noted. All glassware was oven- or flame-dried prior to use. Tetrahydrofuran (THF), diethyl ether ( $Et_2O$ ),

dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>), and toluene (PhMe) were degassed with Ar and then passed through two 4 x 36 inch columns of anhydrous neutral A-2 alumina (8 x 14 mesh; LaRoche Chemicals; activated under a flow of argon at 350 °C for 12 h) to remove H<sub>2</sub>O.<sup>54</sup> All other solvents utilized were purchased anhydrous commercially, or purified as described. <sup>1</sup>H NMR spectra were recorded on Bruker DRX-400 (400 MHz <sup>1</sup>H, 100 MHz <sup>13</sup>C, 376.5 MHz <sup>19</sup>F), GN-500 (500 MHz <sup>1</sup>H, 125.4 MHz <sup>13</sup>C), or CRYO-500 (500 MHz <sup>1</sup>H, 125.8 MHz <sup>13</sup>C) spectrometers. Proton chemical shifts are reported in ppm (δ) relative to internal tetramethylsilane (TMS, δ 0.00). Data are reported as follows: chemical shift (multiplicity [singlet (s), broad singlet (br s), doublet (d), doublet of doublet (dd), doublet of doublet of doublets (ddd), doublet of doublet of doublet of doublets (dddd), doublet of triplet (dt), doublet of doublet of triplet (ddt), doublet of triplet of doublet (dtd), triplet (t), broad triplet (br t), triplet of doublet (td), triplet of doublet of doublet (tdd), triplet of triplet (tt), quartet (q), quartet of doublet (qd), quartet of doublet of doublets (qdd), quintet (quint), apparent quintet (appar quint), sextet, apparent sextet (appar sextet), multiplet (m)]. coupling constants [Hz], integration). Carbon chemical shifts are reported in ppm (δ) relative to TMS with the respective solvent resonance as the internal standard (CDCl<sub>3</sub>, δ 77.16 ppm). Unless otherwise indicated, NMR data were collected at 25 °C. Infrared (IR) spectra were obtained on a Thermo Scientific Nicolet iS5 spectrometer with an iD5 ATR tip (neat) and are reported in terms of frequency of absorption (cm<sup>-1</sup>). Analytical thin-layer chromatography (TLC) was performed using Silica Gel 60 F<sub>254</sub> precoated plates (0.25 mm thickness). Visualization was accomplished by irradiation with a UV lamp and/or staining with KMnO<sub>4</sub> or CAM. Flash chromatography was performed using SiliaFlash F60 (40-63 μm, 60 Å) from SiliCycle. Automated chromatography was carried out on a Teledyne Isco CombiFlash Rf Plus. Melting points (m.p.) were obtained using a Mel-Temp melting point

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<sup>54</sup> Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518–1520.

apparatus and are uncorrected. High resolution mass spectrometry was performed by the University of California, Irvine Mass Spectrometry Center.

Bis(1,5-cyclooctadiene)nickel was purchased from Strem, stored in a glove box freezer (–20 °C) under an atmosphere of N<sub>2</sub> and used as received. All ligands were purchased from Strem or Sigma Aldrich and were stored in a glovebox and used as received. The methylmagnesium iodide was titrated with iodine prior to use.<sup>55</sup> All other chemicals were purchased commercially and used as received, unless otherwise noted.

## **1.5.2 General Kumada Cross-Coupling Reaction Procedures**

### **1.5.2.1 Method A: Kumada Cross-Coupling Reaction**

In a glovebox, a flame-dried 7 mL vial equipped with a stir bar was charged with sulfonamide substrate (1.0 equiv), nickel precatalyst (15 mol %) and PhMe (0.10–0.20 M in substrate). The Grignard reagent (2.0 equiv) was then added dropwise via a syringe. After 24 h, the reaction was removed from the glovebox, quenched with methanol, filtered through a plug of silica gel eluting with 100% Et<sub>2</sub>O and concentrated in vacuo. Phenyltrimethylsilane (PhTMS; 8.6 μL, 0.050 mmol) was added and the yield was determined by <sup>1</sup>H NMR based on comparison to PhTMS as internal standard before purification by column chromatography.

For reactions in which 1.0 equiv of MgI<sub>2</sub> is added, the vial is wrapped in aluminum foil for the duration of the reaction due to the light sensitivity of MgI<sub>2</sub>.

#### **1.5.2.1.1 Preparation of Grignard Reagent**

Under a N<sub>2</sub> atmosphere, a three-necked flask equipped with a stir bar, reflux condenser, and Schlenk filtration apparatus was charged with magnesium turnings (1.1 g, 45 mmol). The flask and magnesium turnings were then flame-dried under vacuum and the flask was back-filled with

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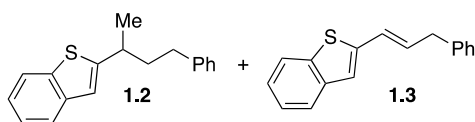
<sup>55</sup> Krasoviskiy, A.; Knochel, P. *Synthesis* **2006**, 5, 890.

N<sub>2</sub>. Anhydrous Et<sub>2</sub>O (7.0 mL) and a crystal of iodine (ca. 2.0 mg) were added to the flask. Freshly distilled iodomethane (1.9 mL, 31 mmol) or 4-iodoanisole as a solution in Et<sub>2</sub>O (4.7 g, 20. mmol, 6.7 M in Et<sub>2</sub>O) was slowly added over 30 min to maintain a gentle reflux. The mixture was stirred for 2 h at room temperature then filtered through the fritted Schlenk filter into a Schlenk flask under N<sub>2</sub> atmosphere. The magnesium turnings were washed with Et<sub>2</sub>O (2 x 1.0 mL) then the Schlenk flask was sealed, removed, and placed under an N<sub>2</sub> atmosphere. The resulting methylmagnesium iodide was typically between 2.4 and 3.0 M as titrated by Knochel's method<sup>55</sup> and could be stored, sealed under N<sub>2</sub> atmosphere or in a glovebox, for up to 4 weeks.

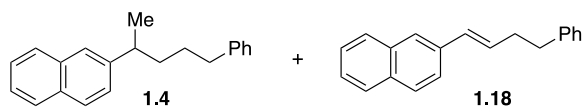
#### 1.5.2.1.2 Preparation of (*R*-BINAP)NiCl<sub>2</sub>

This method was adapted from a procedure reported by Jamison.<sup>41a</sup> To a flame-dried 50 mL round bottom flask equipped with a stir bar was added NiCl<sub>2</sub>·6H<sub>2</sub>O (0.24 g, 1.0 mmol, 1.0 equiv). The flask was placed under vacuum and flame-dried until nearly all of the nickel compound had turned from emerald green to yellow-orange. Some of the green hexahydrate is necessary for the reaction to proceed. The flask was allowed to cool to room temperature then (*R*-BINAP) (0.62 g, 1.0 mmol, 1.0 equiv) was added. The flask was then equipped with a reflux condenser and was evacuated and backfilled with N<sub>2</sub>. The solids were then dissolved in MeCN (20 mL, 0.05 M) and the reaction mixture was allowed to reflux for 24 h. Upon completion, the reaction was cooled to room temperature and the black crystalline precipitate was filtered under vacuum to yield a fine black powder (0.53 g, 0.71 mmol, 71% yield).

#### 1.5.3 Characterization Data for Kumada Cross-Coupled Products



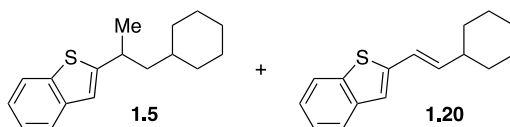
**2-(4-Phenylbutan-2-yl)benzo[*b*]thiophene (1.2)** was prepared according to Method A. The following amounts of reagents were used: sulfonamide **1.1** (87 mg, 0.20 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (23 mg, 30. μmol, 15 mol %), PhMe (1.0 mL, 0.20 M), and methylmagnesium iodide (0.16 mL, 0.40 mmol, 2.0 equiv, 2.5 M in Et<sub>2</sub>O). Before purification, a <sup>1</sup>H NMR yield of 54% was obtained containing 40% styrene **1.3** based on comparison to PhTMS as an internal standard. The residue was purified by flash chromatography (0–5% EtOAc/hexanes) to yield a mixture of the title compound and styrene **1.3**. To separate the major product and the styrene, an Upjohn dihydroxylation was performed.<sup>44</sup> The following amounts of reagents were used: substrate (30 mg, 0.12 mmol, 1.0 equiv), OsO<sub>4</sub> (7.6 μL, 1.2 μmol, 1.0 mol%, 4% solution in H<sub>2</sub>O), *N*-methylmorpholine *N*-oxide (NMO) (16 mg, 0.13 mmol, 1.1 equiv), acetone (0.25 mL) and H<sub>2</sub>O (0.05 mL). The residue was purified by flash column chromatography to afford the title compound as a colorless oil. (13 mg, 48 μmol, 24% yield over two steps) with a small amount of styrene **1.3** (1.1 mg, 4.5 μmol, 2.2% yield). **TLC** R<sub>f</sub> = 0.8 (5% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.78 (d, *J* = 7.9 Hz, 1H), 7.68 (d, *J* = 7.9 Hz, 1H), 7.45–7.22 (m, 4H), 7.22–7.12 (m, 3H), 7.04 (s, 1H), 3.12 (sextet, *J* = 7.0 Hz, 1H), 2.73–2.50 (m, 2H), 2.12–1.92 (m, 2H), 1.41 (d, *J* = 7.0 Hz, 3H); **<sup>13</sup>C NMR** (125.7 MHz, CDCl<sub>3</sub>) δ 152.5, 142.1, 140.0, 139.0, 128.5 (2C), 128.4 (2C), 125.8, 124.1, 123.5, 122.9, 122.3, 119.5, 40.5, 35.8, 33.6, 23.1; **IR** (neat) 2927, 1456, 904, 726 cm<sup>-1</sup>; **HRMS** (TOF MS ES+) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>18</sub>H<sub>18</sub>SNa 289.1027, found 289.1024.



**2-(5-Phenylpentan-2-yl)naphthalene (1.4)**<sup>56</sup> was prepared according to Method A. The following amounts of reagents were used: sulfonamide **1.19** (22 mg, 50. μmol, 1.0 equiv), (*R*-

<sup>56</sup> Compound **1.4** synthesized by Alissa C. Matus.

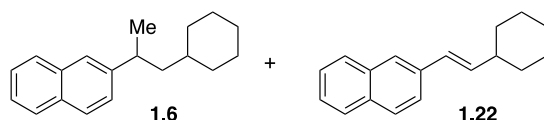
BINAP)NiCl<sub>2</sub> (5.6 mg, 7.5. μmol, 15 mol %), PhMe (0.25 mL, 0.20 M), and methylmagnesium iodide (40. μL, 0.10 mmol, 2.0 equiv, 2.8 M in Et<sub>2</sub>O). Before purification, a <sup>1</sup>H NMR yield of 84% was obtained containing 13% styrene **1.18** based on comparison to PhTMS as an internal standard. The residue was purified by flash chromatography (100% hexanes) to yield the title compound as yellow oil (10. mg, 36 μmol, 74% yield) containing styrene **1.18** (1.3 mg, 5.0 μmol, 10%) and CH<sub>2</sub>Cl<sub>2</sub> (0.8 mg, 9.4 μmol, 19%). **TLC** R<sub>f</sub> = 0.8 (5% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.81–7.74 (m, 3H), 7.60–7.55 (s, 1H), 7.42 (dddd, *J* = 16.3, 8.2, 6.8, 1.4 Hz, 2H), 7.33 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.23 (d, *J* = 7.5 Hz, 2H), 7.17–7.09 (m, 3H), 2.88 (sextet, *J* = 7.0 Hz, 1H), 2.64–2.53 (m, 2H), 1.79–1.55 (m, 4H), 1.31 (d, *J* = 6.9 Hz, 3H); **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 145.2, 142.7, 133.8, 132.3, 128.5 (2C), 128.4 (2C), 128.0, 127.7, 127.7, 125.9, 125.9, 125.8, 125.3, 125.2, 40.2, 38.0, 36.1, 29.7, 22.5; **HRMS** (TOF MS Cl<sup>+</sup>) *m/z*: [M]<sup>+</sup> calcd for C<sub>21</sub>H<sub>22</sub> 274.1721, found 274.1710.



**2-(1-Cyclohexylpropan-2-yl)benzo[*b*]thiophene (1.5)**<sup>57</sup> was prepared according to Method A. The following amounts of reagents were used: sulfonamide **1.21** (43 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (11 mg, 15 μmol, 15 mol %), PhMe (0.50 mL, 0.20 M), and methylmagnesium iodide (70. μL, 0.20 mmol, 2.0 equiv, 2.8 M in Et<sub>2</sub>O). The residue was purified by flash column chromatography (0–15% Et<sub>2</sub>O/pentanes) to afford the title compound as a clear, colorless oil (12 mg, 46 μmol, 46% yield) containing styrene **1.20** (7.3 mg, 30. μmol, 30%) and minimal amounts of solvent. **TLC** R<sub>f</sub> = 0.7 (100% pentanes); **<sup>1</sup>H NMR**: (600 MHz, CDCl<sub>3</sub>) δ 7.83 (d, *J* = 7.2 Hz, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.30 (td, *J* = 7.6, 1.2 Hz, 1H), 7.26–7.22 (m, 1H), 7.00 (s, 1H), 3.27

<sup>57</sup> Compound **1.5** synthesized by Alissa C. Matus.

(sextet,  $J = 12.6$  Hz, 1H), 1.84–1.76 (m, 1H), 1.70–1.57 (m, 5H), 1.53–1.44 (m, 1H), 1.34 (d,  $J = 6.9$  Hz, 3H), 1.32–1.23 (m, 2H), 1.19–1.11 (m, 2H), 0.96–0.85 (m, 2H).  $^{13}\text{C}$  NMR (150.9 MHz,  $\text{CDCl}_3$ )  $\delta$  153.6, 140.2, 138.9, 124.1, 123.4, 122.9, 122.4, 119.0, 66.0, 46.7, 35.2, 33.7, 33.3, 26.8, 26.4, 23.8, 15.4; **HRMS** (TOF MS  $\text{CI}^+$ )  $m/z$  [ $\text{M}$ ] $^+$  calcd for  $\text{C}_{17}\text{H}_{22}\text{S}$  258.1442, found 258.1453.

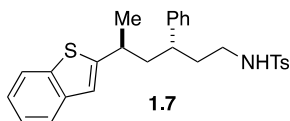


**2-(1-Cyclohexylpropan-2-yl)naphthalene (1.6)**<sup>58</sup> was prepared according to Method A. The following amounts of reagents were used: sulfonamide **1.23** (44 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (11 mg, 15  $\mu\text{mol}$ , 15 mol %), PhMe (0.50 mL, 0.20 M), and methylmagnesium iodide (70.  $\mu\text{L}$ , 0.20 mmol, 2.0 equiv, 2.9 M in Et<sub>2</sub>O). Before purification, a  $^1\text{H}$  NMR yield of 69% was obtained with 22% styrene **1.22** based on comparison to PhTMS as an internal standard. The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a colorless oil (14 mg, 54  $\mu\text{mol}$ , 54% yield) with a small amount of styrene **1.22** (3.7 mg, 15  $\mu\text{mol}$ , 15% yield). **TLC**  $R_f = 0.7$  (100% hexanes);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.85–7.73 (m, 3H), 7.60 (s, 1H), 7.47–7.38 (m, 2H), 7.35 (dd,  $J = 8.4, 1.8$  Hz, 1H), 2.99 (sextet,  $J = 6.8$  Hz, 1H), 1.81 (d,  $J = 13.0$  Hz, 1H), 1.63 (tdd,  $J = 14.2, 8.1, 5.0$  Hz, 5H), 1.50–1.41 (m, 1H), 1.28 (d,  $J = 6.9$  Hz, 3H), 1.19–1.06 (m, 4H), 0.95–0.83 (m, 2H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  145.8, 133.8, 132.3, 128.0, 127.72, 127.68, 126.0, 125.9, 125.2, 125.1, 46.3, 37.0, 35.3, 33.9, 33.5, 26.9, 26.4, 26.4, 23.1; **HRMS** (TOF MS  $\text{CI}^+$ )  $m/z$ : [ $\text{M}$ ] $^+$  calcd for  $\text{C}_{19}\text{H}_{24}$  252.1878, found 252.1868.

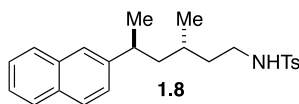
#### 1.5.4 Characterization Data for Ring Opening Kumada XC Products

<sup>58</sup> Compound **1.6** synthesized by Alissa C. Matus.





***N*-(5-(benzo[*b*]thiophen-2-yl)-3-phenylhexyl)-4-methylbenzenesulfonamide (1.7)** was prepared according to Method A. The following amounts of reagents were used: piperidine **1.24** (38 mg, 80  $\mu$ mol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (9.0 mg, 12  $\mu$ mol, 15 mol %), methylmagnesium iodide (60.  $\mu$ L, 0.16 mmol, 2.0 equiv, 2.6 M in Et<sub>2</sub>O), and PhMe (0.5 mL). Before purification, a <sup>1</sup>H NMR yield of 64% was obtained. The residue was purified by column chromatography (0–20% EtOAc/hexanes) to afford the title compound as pale yellow oil (23 mg, 49  $\mu$ mol, 62% yield). The ratio of diastereomers was determined by integration of the resonances attributed to amine hydrogen in the <sup>1</sup>H NMR spectrum. The relative configuration of **1.7** was assigned based on analogy to a compound that has been previously reported.<sup>32</sup> **TLC** *R<sub>f</sub>* = 0.8 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.74 (d, *J* = 7.9 Hz, 1H), 7.67–7.56 (m, 3H), 7.37–7.13 (m, 9H), 7.07–7.00 (m, 2H), 6.93 (s, 1H), 2.85 (q, *J* = 6.7 Hz, 1H), 2.83–2.69 (m, 2H), 2.70–2.59 (m, 1H), 2.40 (s, 3H), 2.07–1.95 (m, 1H), 1.93–1.77 (m, 2H), 1.76–1.64 (m, 1H), 1.30 (d, *J* = 6.8 Hz, 3H); **<sup>13</sup>C NMR** (151 MHz, CDCl<sub>3</sub>)  $\delta$  152.7, 143.5, 143.4, 140.0, 138.9, 136.9, 129.9, 129.8 (2C), 128.9 (2C), 127.7, 127.6 (2C), 127.2 (2C), 126.8, 124.2, 123.6, 123.0, 122.3, 119.1, 45.8, 41.5, 40.9, 36.6, 33.4, 22.0, 21.7; **HRMS** (TOF MS ES<sup>+</sup>) *m/z* [M+Na] calcd for C<sub>27</sub>H<sub>29</sub>NO<sub>2</sub>S<sub>2</sub>Na 486.1537, found 486.1524.



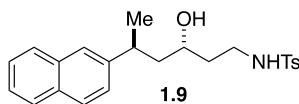
**4-Methyl-*N*-(3-methyl-5-(naphthalen-2-yl)hexyl)benzenesulfonamide (1.8)**<sup>59</sup> was prepared according to Method A. The following amounts of reagents were used: piperidine **1.25** (10. mg,

<sup>59</sup> Compound **1.8** synthesized by Claire A. Herbert.

30.  $\mu\text{mol}$ , 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.0 mg, 40.  $\mu\text{mol}$ , 15 mol %), methylmagnesium iodide (10.  $\mu\text{L}$ , 60.  $\mu\text{mol}$ , 2.0 equiv, 2.9 M in Et<sub>2</sub>O), and PhMe (0.30 mL). Before purification, a <sup>1</sup>H NMR yield of 48% and 10:1 dr was obtained based on comparison to PhTMS as an internal standard. The residue was purified by flash column chromatography (0–15% EtOAc/hexanes) to afford the title compound as a colorless oil (5.4 mg, 14  $\mu\text{mol}$ , 50% yield, 6:1 dr) with a small amount of styrene (0.6 mg, 0.2  $\mu\text{mol}$ , 6%). The ratio of diastereomers was determined by integration of the resonances attributed to amine hydrogen in the <sup>1</sup>H NMR spectrum. The relative configuration of the major **1.8** was assigned based on analogy to ring opened compound **1.7**. For clarity, the <sup>1</sup>H NMR and <sup>13</sup>C NMR data of the major and minor diastereomers have been tabulated individually. **TLC R<sub>f</sub>** = 0.7 (30% EtOAc/hexanes, stained with CAM); **HRMS** (TOF MS E+) *m/z* [M+Na] calcd for C<sub>24</sub>H<sub>29</sub>NO<sub>2</sub>SNa, 418.1817; found, 418.1830.

**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.81–7.76 (m, 3H), 7.69 (d, *J* = 8.3 Hz, 2H), 7.6 (s, 1H), 7.47–7.40 (m, 2H), 7.31–7.26 (m, 3H), 4.29 (t, *J* = 5.9 Hz, 1H), 3.03–2.82 (m, 3H), 2.41 (s, 3H), 1.49 (t, *J* = 6.2 Hz, 1H), 1.43–1.46 (m, 1H), 1.30–1.25 (m, 3H), 1.25 (d, *J* = 6.9 Hz, 3H), 0.79 (d, *J* = 6.5 Hz, 3H); <sup>13</sup>C NMR (125.8, CDCl<sub>3</sub>)  $\delta$  145.1, 148.4, 133.7, 132.2, 129.7 (2C), 128.1, 127.6, 127.6, 127.1 (2C), 126.0, 125.6, 125.2, 125.0, 45.6, 41.1, 37.2, 36.5, 28.1, 29.1, 22.1, 21.5, 19.6.

**Minor Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.81–7.76 (m, 3H), 7.69 (d, *J* = 8.3, 2H), 7.63 (s, 1H), 7.47–7.40 (m, 2H), 7.31–7.26 (m, 3H), 4.39 (t, *J* = 5.5 Hz, 1H), 3.03–2.82 (m, 3H), 2.41 (s, 3H), 1.49 (t, *J* = 6.2 Hz, 1H), 1.43–1.46 (m, 1H), 1.30–1.25 (m, 3H), 1.25 (d, *J* = 6.9 Hz, 3H), 1.08 (d, *J* = 6.7, 3H); <sup>13</sup>C NMR (125.8, CDCl<sub>3</sub>)  $\delta$  145.1, 148.4, 133.7, 132.2, 129.7 (2C), 128.1, 127.6, 127.6, 127.1 (2C), 126.0, 125.7, 125.2, 125.0, 45.6, 41.5, 37.2, 36.5, 29.7, 29.1, 22.1, 21.5, 19.6.



*N*-(3-hydroxy-5-(naphthalen-2-yl)hexyl)-4-methylbenzenesulfonamide (**1.9**)<sup>60</sup> was prepared according to Method A. The following amounts of reagents were used: piperidine **1.26** (93 mg, 0.20 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (23 mg, 30. μmol, 15 mol %), methylmagnesium iodide (0.14 mL, 0.40 mmol, 2.0 equiv, 2.9 M in Et<sub>2</sub>O), and PhMe (2.0 mL). The residue was purified by flash column chromatography (0–15% EtOAc/hexanes) to afford the title compound as a colorless oil (42 mg, 0.11 mmol, 53% yield, 5:1 dr). The ratio of diastereomers was determined by integration of the resonances attributed to amine hydrogen in the <sup>1</sup>H NMR spectrum. The relative configuration of the major **1.9** was assigned based on analogy to ring opened compound **1.7**. For clarity, the <sup>1</sup>H NMR and <sup>13</sup>C NMR data of the major and minor diastereomers have been tabulated individually.

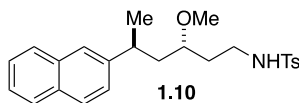
**TLC** R<sub>f</sub> = 0.5 (30% EtOAc/hexanes, stained with CAM); **HRMS** (TOF MS E+) *m/z* [M+Na] calcd for C<sub>23</sub>H<sub>27</sub>NO<sub>3</sub>SNa, 420.1609; found, 420.1604.

**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.79–7.75 (m, 3H), 7.69 (d, *J* = 8.15 Hz, 2H), 7.59 (s, 1H), 7.43 (appar quint, *J* = 7.44 Hz, 2H), 7.31 (d, *J* = 8.82 Hz, 1H), 7.23 (d, *J* = 8.17 Hz, 2H), 5.27 (t, *J* = 5.55 Hz, 1H), 3.74–3.70 (m, 1H), 3.12–3.06 (m, 1H), 3.01–2.93 (m, 2H), 2.37 (s, 3H), 1.89 (br s, 1H), 1.86–1.80 (m, 1H), 1.72–1.62 (m, 2H), 1.53–1.46 (m, 1H), 1.28 (d, *J* = 7.23 Hz, 3H); <sup>13</sup>C NMR (125.8, CDCl<sub>3</sub>) δ 144.4, 143.3, 136.9, 133.7, 132.3, 129.7 (2C), 128.4, 127.7, 127.6, 127.1 (2C), 126.1, 125.5, 124.4, 125.0, 69.2, 45.9, 40.8, 37.0, 36.0, 22.2, 21.5.

**Minor Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.79–7.75 (m, 3H), 7.65 (d, *J* = 8.14 Hz, 2H), 7.59 (s, 1H), 7.43 (appar quint, *J* = 7.44 Hz, 2H), 7.29 (d, *J* = 8.91 Hz, 1H), 7.19 (d, *J* = 8.05

<sup>60</sup> Compound **1.9** synthesized by Claire A. Herbert.

Hz, 2H), 5.19 (t,  $J = 5.57$  Hz, 1H), 3.43–3.39 (m, 1H), 3.12–3.06 (m, 1H), 2.91–2.82 (m, 2H), 2.35 (s, 3H), 1.86–1.80 (m, 1H), 1.76 (br s, 1H), 1.72–1.62 (m, 2H), 1.53–1.46 (m, 1H), 1.29 (d,  $J = 6.40$  Hz, 3H);  $^{13}\text{C}$  NMR (125.8,  $\text{CDCl}_3$ )  $\delta$  143.7, 143.3, 136.8, 133.7, 132.3, 129.7 (2C), 128.4, 127.7, 127.6, 127.1 (2C), 126.1, 125.5, 124.4, 125.0, 68.7, 45.6, 40.8, 36.45, 36.42, 23.2, 21.5.



***N*-(3-methoxy-5-(naphthalen-2-yl)hexyl)-4-methylbenzenesulfonamide (1.10)**<sup>61</sup> was prepared according to Method A. The following amounts of reagents were used: piperidine **1.12** (58 mg, 0.15 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (15 mg, 20.  $\mu\text{mol}$ , 15 mol %), methylmagnesium iodide (0.11 mL, 0.30 mmol, 2.0 equiv, 2.8 M in Et<sub>2</sub>O), and PhMe (1.5 mL). The residue was purified by flash column chromatography (0–25% EtOAc/hexanes) to afford the title compound as a colorless oil (32 mg, 80.  $\mu\text{mol}$ , 52% yield, 5:1 dr). The ratio of diastereomers was determined by integration of the resonances attributed to amine hydrogen in the  $^1\text{H}$  NMR spectrum. The relative configuration of the major **1.10** was assigned based on analogy to ring opened compound **1.7**. For clarity, the  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR data of the major and minor diastereomers have been tabulated individually.

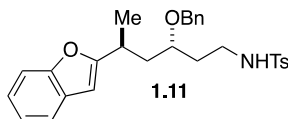
**TLC**  $R_f = 0.4$  (30% EtOAc/hexanes, stained with CAM); **HRMS** (TOF MS E+)  $m/z$  [M+Na] calcd for C<sub>24</sub>H<sub>29</sub>NO<sub>3</sub>SNa, 424.1766; found, 434.1775.

**Major Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.81–7.76 (m, 3H), 7.68 (d,  $J = 8.37$  Hz, 2H), 7.55 (s, 1H), 7.47–7.40 (m, 2H), 7.29 (dd,  $J = 8.48, 1.70$  Hz, 1H), 7.26–7.24 (m, 1H), 7.21 (d,  $J = 7.97$  Hz, 1H), 5.11 (t,  $J = 5.53$  Hz, 1H), 3.15 (s, 3H), 3.08–2.91 (m, 3H), 2.86 (appar sextet,  $J = 7.37$  Hz, 1H), 2.34 (s, 3H), 1.96 (ddd,  $J = 14.3, 8.50, 5.78$  Hz, 1H), 1.82–1.76 (m, 1H), 1.58–

<sup>61</sup> Compound **1.10** synthesized by Claire A. Herbert.

1.50 (m, 2H), 1.28 (d,  $J = 6.98$  Hz, 3H);  $^{13}\text{C}$  NMR (125.8,  $\text{CDCl}_3$ )  $\delta$  144.1, 143.2, 136.8, 133.6, 132.3, 129.7 (2C), 128.3, 127.7, 127.6, 127.1 (2C), 126.1, 125.4, 125.24, 125.17 78.4, 56.3, 40.9, 40.5, 36.3, 32.0, 22.9, 21.5.

**Minor Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.81–7.76 (m, 3H), 7.70 (d,  $J = 8.60$  Hz, 2H), 7.55 (s, 1H), 7.47–7.40 (m, 2H), 7.29 (dd,  $J = 8.48, 1.70$  Hz, 1H), 7.26–7.24 (m, 1H), 7.21 (d,  $J = 7.97$  Hz, 1H), 5.03 (t,  $J = 5.52$  Hz, 1H), 3.20 (s, 3H), 3.08–2.91 (m, 3H), 2.86 (apparent sextet,  $J = 7.37$  Hz, 1H), 2.38 (s, 3H), 1.96 (ddd,  $J = 14.3, 8.50, 5.78$  Hz, 1H), 1.71–1.66 (m, 1H), 1.47–1.40 (m, 2H), 1.27 (d,  $J = 7.32$  Hz, 3H)  $^{13}\text{C}$  NMR (125.8,  $\text{CDCl}_3$ )  $\delta$  144.2, 143.2, 136.9, 133.6, 132.3, 129.7 (2C), 128.3, 127.7, 127.6, 127.1 (2C), 126.1, 125.52, 125.46 125.4, 78.4, 56.3, 40.9, 40.5, 36.3, 32.0, 22.9, 21.5.



***N*-(5-(benzofuran-2-yl)-3-(benzyloxy)hexyl)-4-methylbenzenesulfonamide (1.11)**<sup>62</sup> was prepared according to Method A. The following amounts of reagents were used: piperidine **1.27** (78 mg, 0.17 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (22 mg, 30.  $\mu\text{mol}$ , 15 mol %), methylmagnesium iodide (0.23 mL, 0.68 mmol, 4.0 equiv, 2.9 M in Et<sub>2</sub>O), and PhMe (1.5 mL). The residue was purified by flash column chromatography (0–15% EtOAc/hexanes) to afford a mixture of the title compound and styrene. To separate the major product and the styrene, a dihydroxylation was performed. The following amounts of reagents were used: AD-mix- $\beta$  (52 mg, 1.4 g/mmol), *t*-BuOH (1.0 mL), and H<sub>2</sub>O (1.0 mL). The residue was purified by flash column chromatography to afford the title compound as a colorless oil. (7.0 mg, 15  $\mu\text{mol}$ , 8.6 % yield over two steps, 5:1 dr). The ratio of diastereomers was determined by integration of the resonances attributed to amine

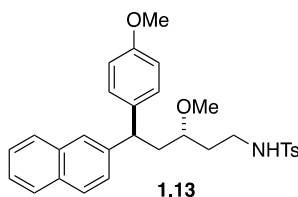
<sup>62</sup> Compound **1.11** synthesized by Claire A. Herbert.

hydrogen in the  $^1\text{H}$  NMR spectrum. The relative configuration of the major **1.11** was assigned based on analogy to ring opened compound **1.7**. When the reaction was performed with 2.0 equivalents of methylmagnesium iodide, a  $^1\text{H}$  NMR yield of 41% was obtained based on comparison to PhTMS as an internal standard before purification. For clarity, the  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR data of the major and minor diastereomers have been tabulated individually.

**TLC**  $R_f$  = 0.5 (30% EtOAc/hexanes, stained with CAM); **HRMS** (TOF MS E+)  $m/z$  [M+Na] calcd for  $\text{C}_{28}\text{H}_{31}\text{NO}_4\text{SNa}$ , 500.1872; found, 500.1861.

**Major Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.64 (d,  $J$  = 7.9 Hz, 2H), 7.48 (d,  $J$  = 7.4 Hz, 1H), 7.42 (d,  $J$  = 7.9 Hz, 1H), 7.27–7.16 (m, 8H), 6.33 (s, 1H), 4.79 (t,  $J$  = 6.0 Hz, 1H), 4.45 (d,  $J$  = 11.5 Hz, 1H), 4.31 (d,  $J$  = 11.4 Hz, 1H), 3.49–3.42 (m, 1H), 3.09–2.95 (m, 3H), 2.38 (s, 3H), 2.12 (appar quint,  $J$  = 6.9 Hz, 1H), 1.87–1.81 (m, 1H), 1.65–1.54 (m, 3H), 1.29 (d,  $J$  = 6.9 Hz, 3H)  $^{13}\text{C}$  NMR (125.8,  $\text{CDCl}_3$ )  $\delta$  162.6, 154.5, 143.3, 137.9, 136.9, 129.7 (2C), 128.5 (2C), 128.1 (2C), 127.9, 127.1 (2C), 123.4, 122.6, 120.5, 110.9, 101.1, 75.0, 70.7, 40.2, 39.1, 32.9, 30.3, 29.7, 21.5, 19.7.

**Minor Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.80 (d,  $J$  = 7.9 Hz, 2H), 7.75 (d,  $J$  = 7.7 Hz, 1H), 7.58 (d,  $J$  = 8.4 Hz, 1H), 7.27–7.16 (m, 8H), 6.25 (s, 1H), 4.79 (t,  $J$  = 6.0 Hz, 1H), 4.40 (s, 2H), 3.53–3.48 (m, 1H), 3.09–2.95 (m, 3H), 2.73 (appar quint,  $J$  = 6.5 Hz, 1H), 2.40 (s, 3H), 1.80–1.77 (m, 1H), 1.65–1.60 (m, 3H), 0.88 (d,  $J$  = 7.0 Hz, 3H);  $^{13}\text{C}$  NMR (125.8,  $\text{CDCl}_3$ )  $\delta$  162.6, 154.5, 143.3, 137.9, 136.9, 129.4 (2C), 128.7 (2C), 128.3 (2C), 127.9, 127.1 (2C), 123.4, 122.6, 120.5, 110.9, 101.4, 75.6, 71.4, 39.9, 39.1, 32.8, 30.5, 29.7, 21.5, 20.2.



**N-3-methoxy-5-(4-methoxyphenyl)-5-(naphthalen-2-yl)pentyl)-4-**

**methylbenzenesulfonamide (1.13)**<sup>63</sup> was prepared according to Method A. The following amounts of reagents were used: piperidine **1.12** (37 mg, 90  $\mu$ mol, 1.0 equiv), Ni(dppe)Cl<sub>2</sub> (7.0 mg, 10.  $\mu$ mol, 15 mol %), (4-methoxyphenyl)magnesium iodide (0.11 mL, 0.18 mmol, 2.0 equiv, 1.7 M in Et<sub>2</sub>O), and PhMe (0.90 mL). Before purification, a <sup>1</sup>H NMR yield of 56% was obtained based on comparison to PhTMS as an internal standard. The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a yellow oil (26 mg, 50.  $\mu$ mol, 58% yield, 3:1 dr). The ratio of diastereomers was determined by integration of the resonances attributed to methyl hydrogens of the tosyl group in the <sup>1</sup>H NMR spectrum. The relative configuration of the major **1.13** was assigned based on analogy to ring opened compound **1.7**. For clarity, the <sup>1</sup>H NMR and <sup>13</sup>C NMR data of the major and minor diastereomers have been tabulated individually.

**TLC R<sub>f</sub>** = 0.3 (30% EtOAc/hexanes, stained with CAM); **HRMS** (TOF MS E+) *m/z* [M+H] calcd for C<sub>30</sub>H<sub>34</sub>NO<sub>4</sub>S, 504.2209; found, 504.2206.

**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.76 (d, *J* = 7.3 Hz, 2H), 7.70–7.69 (m, 3H), 7.61 (s, 1H), 7.43 (dt, *J* = 20.4, 7.3 Hz, 2H), 7.29–7.19 (m, 3H), 7.13 (d, *J* = 8.5 Hz, 2H), 6.81 (d, *J* = 8.6 Hz, 2H), 5.09–5.05 (m, 1H), 4.12 (t, *J* = 7.8 Hz, 1H), 3.76 (s, 3H), 3.20 (s, 3H), 3.13–3.09 (m, 1H), 3.05–2.97 (m, 2H), 2.30 (s, 3H), 2.25 (appar sext, *J* = 7.3 Hz, 1H), 2.02–1.97 (m, 1H), 1.85–1.80 (m, 1H), 1.58–1.52 (m, 1H); <sup>13</sup>C NMR (125.8, CDCl<sub>3</sub>)  $\delta$  158.2, 143.4, 142.4, 136.8, 135.2, 133.6, 129.7 (2C), 129.0 (2C), 128.8, 128.3, 127.8, 127.7, 127.2 (2C), 126.5, 126.2, 125.6, 125.5, 114.1 (2C), 78.0, 56.7, 55.3, 46.5, 40.3, 39.1, 31.8, 21.5.

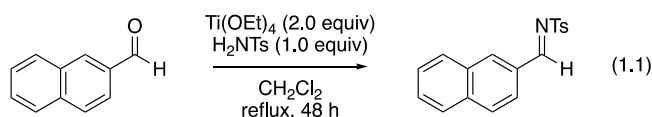
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<sup>63</sup> Compound **1.12** synthesized by Claire A. Herbert.

**Minor Diastereomer:**  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.76 (d,  $J = 7.3$  Hz, 2H), 7.73–7.70 (m, 3H), 7.63 (s, 1H), 7.43 (dt,  $J = 20.4, 7.3$  Hz, 2H), 7.29–7.19 (m, 3H), 7.14 (d,  $J = 7.7$  Hz, 2H), 6.81 (d,  $J = 8.8$  Hz, 2H), 5.09–5.05 (m, 1H), 4.12 (t,  $J = 7.8$  Hz, 1H), 3.76 (s, 3H), 3.19 (s, 3H), 3.13–3.09 (m, 1H), 3.05–2.97 (m, 2H), 2.35 (s, 3H), 2.25 (apparent sextet,  $J = 7.3$  Hz, 1H), 2.06–2.03 (m, 1H), 1.76–1.78 (m, 1H), 1.58–1.52 (m, 1H);  $^{13}\text{C NMR}$  (125.8,  $\text{CDCl}_3$ )  $\delta$  158.2, 143.4, 142.0, 136.9, 136.5, 133.6, 129.7 (2C), 129.0 (2C), 128.8, 128.4, 127.8, 127.7, 127.2 (2C), 126.5, 126.2, 125.9, 125.6, 114.1 (2C), 78.0, 56.7, 55.3, 46.5, 40.3, 39.1, 31.9, 21.6.

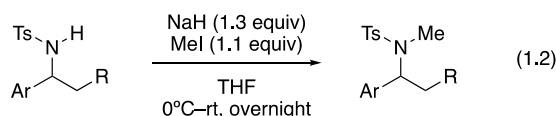
## 1.5.5 General Procedures for the Synthesis of Starting Materials

### 1.5.5.1 Method B: Condensation Reaction



This method was adapted from a procedure reported by Ruano et al.<sup>64</sup> A flame-dried two-neck flask equipped with a stir bar, condenser, septum and  $\text{N}_2$  inlet was charged with aldehyde (1.0 equiv), and *p*-toluenesulfonamide (1.0 equiv) and  $\text{CH}_2\text{Cl}_2$  (330 mL). Then  $\text{Ti(OEt)}_4$  (2.0 equiv) was added dropwise. The deep orange solution was brought to reflux ( $\sim 45$  °C) and allowed to stir for 48 h. The solution was cooled to room temperature and was quenched with  $\text{H}_2\text{O}$ . The mixture was vacuum filtered and the filtrate was concentrated in vacuo.

### 1.5.5.2 Method C: Methylation of Sulfonamide with Methyl Iodide



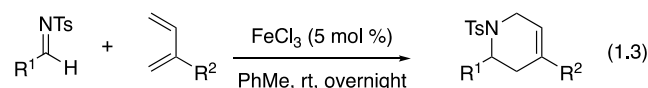
This method was adapted from a procedure reported by Jarvo.<sup>32a</sup> To a suspension of NaH (1.3 equiv) in THF (0.10 M) was added a solution of sulfonamide (1.0 equiv) in THF (0.15 M) at 0 °C.

<sup>64</sup> García Ruano, J. L.; Alemán, J.; Belén Cid, M.; Parra, A. *Org. Lett.* **2005**, *7*, 179–182.



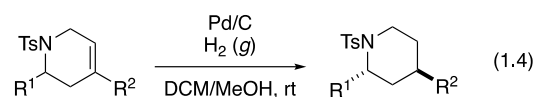
The mixture was warmed to rt and allow to stir for 1 h before the addition of iodomethane (1.1 equiv). The reaction was allowed to stir overnight at rt. The excess NaH was quenched with sat. NH<sub>4</sub>Cl and the solution was extracted with EtOAc (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, concentrated in vacuo, and purified by flash column chromatography.

### 1.5.5.3 Method D: Fe-Catalyzed Formal [4+2] Cycloaddition



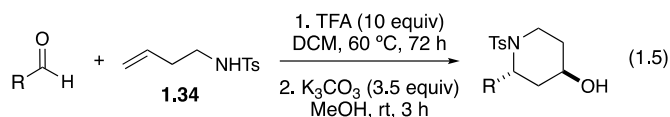
This method was adapted from a procedure reported by Matsubara.<sup>43</sup> To a flame-dried round-bottom flask equipped with a stir bar was added imine (1.0 equiv),  $FeCl_3$  (5.0 mol %), and PhMe (0.1 M). Once the solution was homogenous, diene (2.0 equiv) was added. The reaction mixture was allowed to stir at rt overnight. After completion, the reaction mixture was filtered through a short pad of silica, washed with excess ethyl acetate, and concentrated in vacuo.

### 1.5.5.4 Method E: Pd/C Reduction of Alkenes



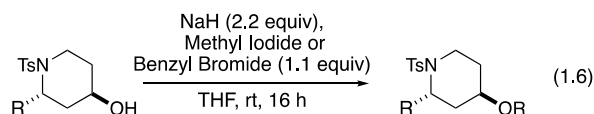
A flame-dried round-bottom flask with stir bar was charged with palladium on carbon (1.0 mg/3.5 mmol of substrate), flushed with N<sub>2</sub>, and capped with septum. Slowly, DCM was added, until Pd/C was fully submerged. Then MeOH (0.2 M in substrate), and alkene (1.0 equiv) were added. Vacuum was pulled on the flask until the solvent began to bubble, at which point the flask was backfilled with N<sub>2</sub> (x 3). An H<sub>2</sub> balloon was added and the reaction mixture was allowed to stir vigorously until complete by <sup>1</sup>H NMR. The balloon was then removed, and the flask was purged with N<sub>2</sub> for 30 min. The septum was removed, and the reaction mixture was filtered through Celite using MeOH (100 mL). The collected solvent was then concentrated in vacuo.

### 1.5.5.5 Method F: TFA Mediated aza-Prins Cyclization



This method was adapted from a procedure reported by Sabitha.<sup>44</sup> To a flame-dried pressure tube equipped with a stir bar was added aldehyde (1.0 equiv), homoallylic sulfonamide **1.34** (1.1 equiv), and CH<sub>2</sub>Cl<sub>2</sub> (0.10 M). Then trifluoroacetic acid (10.0 equiv) was added slowly via syringe. The solution was warmed to 60 °C and allowed to stir for 72 h. The solution was then cooled to rt and quenched with saturated aq. NaHCO<sub>3</sub>. Then the pH was adjusted to >7 by the addition of Et<sub>3</sub>N. The solution was transferred to a separatory funnel, and the aqueous layer was extracted with CH<sub>2</sub>Cl<sub>2</sub> (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated in vacuo. The residue was then redissolved in MeOH, and K<sub>2</sub>CO<sub>3</sub> (3.5 equiv) was added to the flask and the slurry was allowed to stir at rt for 3 h. The solvent was removed under reduced pressure, then H<sub>2</sub>O was added and the residue was transferred to a separatory funnel. The aqueous layer was extracted with EtOAc (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated in vacuo.

### 1.5.5.6 Method G: Alkylation of Secondary Alcohol

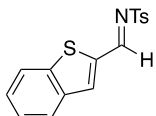


This method was adapted from a procedure reported by Yang.<sup>65</sup> In a glovebox to a flame-dried round bottom flask equipped with a stir bar was added NaH (2.2 equiv). The flask was removed from the glovebox and NaH was dissolved in THF (0.2 M). Alcohol (1.0 equiv) was added dropwise as a solution in THF (0.3 M) and the reaction mixture was allowed to stir at rt for 1 h.

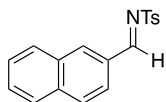
<sup>65</sup> Fu, M.; Chen, L.; Jiang, Y.; Jiang, Z.-X.; Yang, Z. *Org. Lett.* **2016**, *18*, 348–351.

Methyl iodide or benzyl bromide was then added dropwise to the stirring slurry and the reaction mixture was allowed to stir at rt overnight. The reaction was then quenched with saturated aq. NH<sub>4</sub>Cl and extracted with EtOAc (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated in vacuo.

### 1.5.6 Synthesis and Characterization Data of Sulfonamide Starting Materials

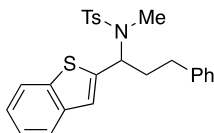


**N-(benzo[*b*]thiophen-2-ylmethylene)-4-methylbenzenesulfonamide (1.28)** was prepared according to Method B. The following amounts of reagents were used: benzo[*b*]thiophene-2-carbaldehyde (3.2 g, 20. mmol, 1.0 equiv), *p*-toluenesulfonamide (3.1 g, 20. mmol, 1.0 equiv), Ti(OEt)<sub>4</sub> (8.4 mL, 40. mmol, 2.0 equiv), and CH<sub>2</sub>Cl<sub>2</sub> (330 mL). The residue was purified by flash column chromatography (5–25% EtOAc/hexanes) to yield the title compound as a pale yellow solid (5.0 g, 16 mmol, 80 %). **m.p.** 148–150°C; **TLC** R<sub>f</sub> = 0.5 (25% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 9.23 (s, 1H), 8.02 (s, 1H), 7.90 (d, *J* = 8.3, 3H), 7.86 (d, *J* = 8.2, 1H), 7.50 (td, *J* = 8.3, 1.2, 1H), 7.42 (td, *J* = 8.4, 1.2, 1H), 7.35 (d, *J* = 8.4, 2H), 2.44 (s, 3H); **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 163.3, 144.8, 143.6, 138.9, 138.3, 137.3, 135.3, 130.0 (2C), 128.7, 128.2 (2C), 126.1, 125.5, 123.2, 21.8; **IR** (neat) 3259, 2921, 1566, 1305, 1292, 1152, 1087, 752 cm<sup>-1</sup>; **HRMS** (TOF MS ES<sup>+</sup>) *m/z* calcd for C<sub>16</sub>H<sub>13</sub>NO<sub>2</sub>S<sub>2</sub> [M + Na]<sup>+</sup> 338.0285, found 338.0283.

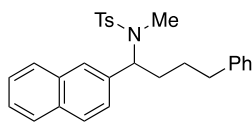


**4-Methyl-N-(naphthalen-2-ylmethylene)benzenesulfonamide (1.29)** was prepared according to Method B. The following amounts of reagents were used: 2-naphthaldehyde (0.31 g, 2.0 mmol, 1.0 equiv), *p*-toluenesulfonamide (0.34 g, 2.0 mmol, 1.0 equiv), Ti(OEt)<sub>4</sub> (0.59 mL, 4.0 mmol, 2.0

equiv), and CH<sub>2</sub>Cl<sub>2</sub> (33 mL). The residue was purified by flash column chromatography (0–25% EtOAc/hexanes) to yield the title compound as a pale yellow solid (0.31 g, 1.0 mmol, 50% yield). Analytical data is consistent with literature values.<sup>66</sup> **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 9.18 (s, 1H), 8.34 (s, 1H), 8.04 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.98–7.86 (m, 5H), 7.67–7.55 (m, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 2.44 (s, 3H).

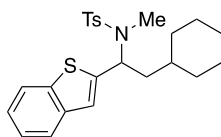


***N*-(1-(benzo[*b*]thiophen-2-yl)-3-phenylpropyl)-*N*,4-dimethylbenzenesulfonamide (1.1)** was prepared according to Method C. The following amounts of reagents were used: *N*-(1-(benzo[*b*]thiophen-2-yl)-3-phenylpropyl)-4-methylbenzenesulfonamide (0.68 g, 1.6 mmol, 1.0 equiv), NaH (50. mg, 2.1 mmol, 1.3 equiv), methyl iodide (0.11 mL, 1.8 mmol, 1.1 equiv) and THF (30 mL). The residue was purified by flash column chromatography (5–25 % EtOAc/hexanes) to yield the title compound as a yellow oil (0.52 g, 1.2 mmol, 68% yield) **TLC** *R<sub>f</sub>* = 0.8 (25% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.73 (d, *J* = 7.9 Hz, 1H), 7.70–7.62 (m, 3H), 7.40–7.27 (m, 4H), 7.27–7.19 (m, 3H), 7.16 (d, *J* = 7.7 Hz, 2H), 7.06 (s, 1H), 5.42 (t, *J* = 7.5 Hz, 1H), 2.77 (s, 3H), 2.69 (qdd, *J* = 14.1, 10.4, 5.9 Hz, 2H), 2.40 (s, 3H), 2.30 (dddd, *J* = 13.7, 10.3, 7.2, 5.4 Hz, 1H), 2.08 (dddd, *J* = 13.9, 10.5, 7.8, 6.3 Hz, 1H); **<sup>13</sup>C NMR** (125.7 MHz, CDCl<sub>3</sub>) δ 143.31, 143.30, 141.0, 139.6, 139.2, 137.0, 129.6 (2C), 128.6 (2C), 128.5 (2C), 127.3 (2C), 126.3, 124.5, 124.4, 123.6, 122.9, 122.3, 56.5, 34.6, 33.0, 29.0, 21.6; **HRMS** (TOF MS ES+) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>25</sub>H<sub>25</sub>NO<sub>2</sub>S<sub>2</sub>Na 458.1224, found 458.1235.



<sup>66</sup> Syu, S.; Lee, Y.-T.; Jang, Y.-J.; Lin, W. *J. Org. Chem.* **2011**, *76*, 2888–2891.

***N*,4-dimethyl-*N*-(1-(naphthalen-2-yl)-4-phenylbutyl)benzenesulfonamide (1.19)**<sup>67</sup> was prepared according to Method C. The following amounts of reagents were used: 4-methyl-*N*-(1-(naphthalen-2-yl)-4-phenylbutyl)benzenesulfonamide (0.50 g, 1.2 mmol, 1.0 equiv), NaH (36 mg, 1.5 mmol, 1.3 equiv), methyl iodide (80.  $\mu$ L, 1.3 mmol, 1.1 equiv) and THF (23 mL). The residue was purified by flash column chromatography (0–25% EtOAc/hexanes) to yield the title compound as a pale yellow solid (0.42 g, 0.95 mmol, 82% yield). **TLC**  $R_f$  = 0.3 (25% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.85 (td,  $J$  = 8.1, 7.1, 4.1 Hz, 2H), 7.80 (d,  $J$  = 8.5 Hz, 1H), 7.75 (dt,  $J$  = 6.1, 3.7 Hz, 1H), 7.72 (d,  $J$  = 8.1 Hz, 2H), 7.58–7.50 (m, 3H), 7.46–7.39 (m, 1H), 7.36–7.31 (m, 2H), 7.26 (t,  $J$  = 8.3 Hz, 2H), 7.20 (d,  $J$  = 7.3 Hz, 2H), 5.34 (t,  $J$  = 7.7 Hz, 1H), 2.72 (td,  $J$  = 7.5, 2.3 Hz, 2H), 2.68 (s, 3H), 2.44 (s, 3H), 2.16–2.06 (m, 1H), 1.88 (ddd,  $J$  = 15.6, 14.0, 7.6 Hz, 1H), 1.70 (quint,  $J$  = 9.0 Hz, 2H); **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  143.1, 141.9, 135.9, 133.1, 132.9, 129.9, 129.6 (2C), 128.53 (2C), 128.46 (2C), 128.3, 128.1, 127.7, 127.6, 127.3 (2C), 126.7, 126.4, 126.3, 126.0, 60.0, 35.5, 30.0, 28.9, 28.3, 21.6; **HRMS** (TOF MS ES+)  $m/z$ : [M + Na] calcd for C<sub>28</sub>H<sub>29</sub>NO<sub>2</sub>S 466.1817, found 466.1816.

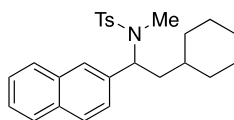


***N*-(1-benzo[*b*]thiophen-2-yl)-2-cyclohexylethyl)-*N*,4-dimethylbenzenesulfonamide (1.21)**<sup>68</sup> was prepared according to Method C. The following amounts of reagents were used: *N*-(1-(benzo[*b*]thiophen-2-yl)-2-cyclohexylethyl)-4-methylbenzenesulfonamide (170 mg, 0.41 mmol, 1.0 equiv), NaH (31 mg, 0.53 mmol, 1.3 equiv), methyl iodide (30.  $\mu$ L, 0.45 mmol, 1.1 equiv), and THF (8.2 mL). The residue was purified by flash column chromatography (20% EtOAc/hexanes)

<sup>67</sup> Compound **1.19** was synthesized by Alissa C. Matus.

<sup>68</sup> Compound **1.21** was synthesized by Alissa C. Matus.

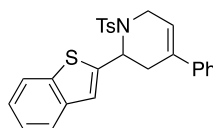
to afford the title compound as a clear, colorless oil (170 mg, 0.40 mmol, 98% yield). **TLC R<sub>f</sub>** = 0.40 (10% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.73 (d, *J* = 7.8 Hz, 1H), 7.70 (d, *J* = 8.3 Hz, 2H), 7.66 (d, *J* = 7.2 Hz, 1H), 7.30 (dtd, *J* = 16.4, 7.2, 1.3 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.05 (s, 1H), 5.50 (t, *J* = 7.6, 1H), 2.71 (s, 3H), 2.39 (s, 3H), 1.92–1.78 (m, 2H), 1.76–1.59 (m, 5H), 1.33–1.23 (m, 1H), 1.20–1.10 (m, 3H), 1.04–0.83 (m, 2H); **<sup>13</sup>C NMR** (125.8 MHz, CDCl<sub>3</sub>) δ 144.1, 143.0, 139.4, 139.1, 137.0, 129.4 (2C), 127.2 (2C), 124.2, 123.3, 122.3, 122.5, 122.1, 53.9, 40.3, 34.0, 33.4, 33.0, 28.7, 26.3, 26.0, 25.9, 21.4; **HRMS** (TOF MS ES<sup>+</sup>) *m/z* [M + Na]<sup>+</sup> calcd for C<sub>24</sub>H<sub>29</sub>NO<sub>2</sub>S<sub>2</sub>Na 450.1537, found 450.1530.



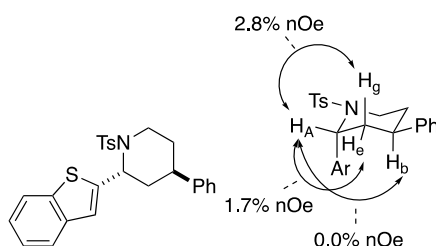
***N*-(2-cyclohexyl-1-(naphthalen-2-yl)ethyl)-*N*,4-dimethylbenzenesulfonamide (1.23)**<sup>69</sup> was prepared according to Method C. The following amounts of reagents were used: *N*-(2-cyclohexyl-1-(naphthalen-2-yl)ethyl)-4-methylbenzenesulfonamide (190 mg, 0.46 mmol, 1.0 equiv), NaH (17 mg, 0.70 mmol, 1.5 equiv), methyl iodide (40. μL, 0.60 mmol, 1.1 equiv), and THF (11 mL). The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a yellow oil (180 mg, 0.43 mmol, 86% yield). **TLC R<sub>f</sub>** = 0.4 (10% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.87 (d, *J* = 9.2 Hz, 1H), 7.82 (dd, *J* = 11.3, 7.5 Hz, 2H), 7.75 (d, *J* = 8.2 Hz, 2H), 7.65 (br s, 1H), 7.56–7.51 (m, 2H), 7.49 (dd, *J* = 8.5, 1.5 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 5.45 (br t, *J* = 7.7 Hz, 1H), 2.72 (s, 3H), 2.46 (s, 3H), 2.02–1.88 (m, 2H), 1.81 (d, *J* = 12.5 Hz, 1H), 1.78–1.72 (m, 2H), 1.72–1.60 (m, 2H), 1.26–1.12 (m, 4H), 1.09–0.87 (m, 2H); **<sup>13</sup>C NMR** (125.8 MHz, CDCl<sub>3</sub>) δ 143.1, 137.6, 136.3, 133.1, 132.9, 129.6 (2C), 128.2, 128.1, 127.6,

<sup>69</sup> Compound **1.23** was synthesized by Alissa C. Matus.

127.3 (2C), 126.8, 126.6, 126.2, 57.5 (2C), 38.2, 34.2, 33.5, 33.4, 28.9, 26.6, 26.22, 26.21, 21.6;  
**HRMS** (TOF MS ES+)  $m/z$  [M + Na]<sup>+</sup> calcd for C<sub>26</sub>H<sub>31</sub>NO<sub>2</sub>SNa 444.1973, found 444.1968.



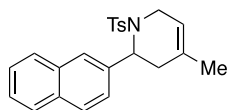
**2-(Benzo[b]thiophen-2-yl)-4-phenyl-1-tosyl-1,2,3,6-tetrahydropyridine (1.30)** was prepared according to Method D. The following amounts of reagents were used: imine **1.28** (240 mg, 0.75 mmol, 1.0 equiv), buta-1,3-dien-2-ylbenzene (190 mg, 1.5 mmol, 2.0 equiv)<sup>70</sup>, FeCl<sub>3</sub> (6.0 mg, 40. μmol, 5.0 mol %), and PhMe (10. mL). The residue was purified by column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a yellow oil (154 mg, 0.34 mmol, 46% yield). **TLC** R<sub>f</sub> = 0.5 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.71 (d, *J* = 8.1 Hz, 2H), 7.68 (d, *J* = 7.8 Hz, 1H), 7.62 (d, *J* = 7.8 Hz, 1H), 7.36–7.22 (m, 8H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.10 (s, 1H), 5.95 (s, 1H), 5.80 (d, *J* = 6.1 Hz, 1H), 4.35 (dt, *J* = 18.6, 3.5 Hz, 1H), 3.84 (dq, *J* = 18.7, 2.8 Hz, 1H), 2.96 (ddt, *J* = 16.3, 6.4, 3.2 Hz, 1H), 2.87 (d, *J* = 17.2 Hz, 1H), 2.33 (s, 3H); **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 144.9, 144.6, 143.6, 140.0, 139.9, 129.9 (2C), 129.2, 128.8 (2C), 128.4, 127.4 (2C), 126.8 (2C), 124.5, 124.3, 123.5, 122.3, 122.3, 53.9, 42.2, 37.1, 36.7, 31.7, 21.7.



**2-(Benzo[b]thiophen-2-yl)-4-phenyl-1-tosylpiperidine (1.24)** was prepared according to Method E. The following amounts of reagents were used: substrate **1.30** (100 mg, 0.22 mmol, 1.0 equiv), Pd/C (20 mg), DCM (2.0 mL) and MeOH (5.0 mL). The residue was purified by column

<sup>70</sup> Buta-1,3-dien-2-ylbenzene was prepared according to the following method: Fiorito, D.; Folliet, S.; Liu, Y.; Mazet, C. *ACS Catal.* **2018**, 8, 1392–1398.

chromatography (0–10% EtOAc/hexanes) to afford the title compound as a pale yellow oil (24 mg, 53  $\mu$ mol, 25% yield, >20:1 dr trans:cis). The dr was determined based on the integration of the resonances attributed to the benzylic hydrogens in the  $^1\text{H}$  NMR spectrum. The relative configuration was assigned based on nOe analysis. **TLC**  $R_f$  = 0.5 (20% EtOAc/hexanes);  **$^1\text{H}$  NMR** (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.81 (d,  $J$  = 8.4 Hz, 2H), 7.77 (d,  $J$  = 7.9 Hz, 1H), 7.68 (d,  $J$  = 7.7 Hz, 1H), 7.40–7.26 (m, 6H), 7.23–7.18 (m, 1H), 7.13 (d,  $J$  = 1.5 Hz, 1H), 7.06 (d,  $J$  = 7.4 Hz, 2H), 5.70 (d,  $J$  = 5.2 Hz, 1H), 4.03 (d,  $J$  = 14.0 Hz, 1H), 3.34 (ddd,  $J$  = 14.1, 12.7, 3.0 Hz, 1H), 2.93 (tt,  $J$  = 12.6, 3.6 Hz, 1H), 2.43 (s, 3H), 2.33 (d,  $J$  = 13.0 Hz, 1H), 2.01 (td,  $J$  = 13.5, 5.5 Hz, 1H), 1.68 (d,  $J$  = 12.5 Hz, 1H), 1.61 (td,  $J$  = 12.7, 4.4 Hz, 1H);  **$^{13}\text{C}$  NMR** (151 MHz,  $\text{CDCl}_3$ )  $\delta$  144.8, 144.5, 139.9, 139.8, 129.8 (2C), 129.1, 128.7 (2C), 128.3, 127.3 (2C), 126.7 (2C), 124.4, 124.2, 123.4, 122.2, 122.2, 76.8, 53.8, 42.1, 36.9, 36.6, 31.6, 21.6; **HRMS** (TOF MS ES+)  $m/z$   $[\text{M}+\text{Na}]$  calcd for  $\text{C}_{26}\text{H}_{25}\text{NO}_2\text{S}_2\text{Na}$  470.1224, found 470.1228.

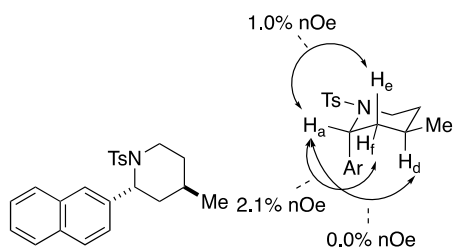


**4-Methyl-2-(naphthalen-2-yl)-1-tosyl-1,2,3,6-tetrahydropyridine (1.31)**<sup>71</sup> was prepared according to Method D. The following amounts of reagents were used: imine **1.29** (0.31 g, 1.0 mmol, 1.0 equiv), isoprene (1.5 mL, 15 mmol, 15 equiv),  $\text{FeCl}_3$  (16 mg, 0.10 mmol, 10. mol %), and PhMe (10. mL, 0.10 M). The residue was purified by flash column chromatography (0–5% EtOAc/hexanes) to afford the title compound as a yellow oil (150 mg, 0.40 mmol, 40% yield). **TLC**  $R_f$  = 0.5 (20% EtOAc/hexanes);  **$^1\text{H}$  NMR** (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.78–7.71 (m, 3H), 7.69 (d,  $J$  = 8.3 Hz, 2H), 7.56 (s, 1H), 7.48–7.40 (m, 3H), 7.20 (d,  $J$  = 8.1 Hz, 2H), 5.43 (d,  $J$  = 3.5 Hz,

<sup>71</sup> Compound **1.31** was synthesized by Claire A. Herbert.



1H), 5.29 (s, 1H), 4.11 (d,  $J = 18.0$  Hz, 1H), 3.35 (d,  $J = 18.1$  Hz, 1H), 2.40–2.30 (m, 2H), 2.35 (s, 3H), 1.68 (s, 3H).



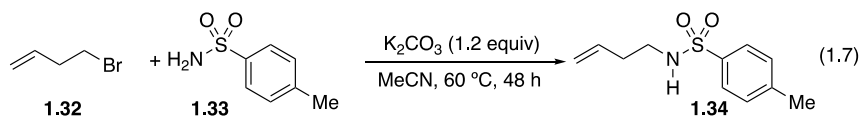
**4-Methyl-2-(naphthalen-2-yl)-1-tosylpiperidine (1.25)**<sup>72</sup> was prepared according to Method E. The following amounts of reagents were used: substrate **1.31** (53 mg, 0.14 mmol, 1.0 equiv), Pd/C (27 mg), DCM (1.0 mL) and MeOH (1.0 mL). The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a pale yellow oil (9.6 mg, 25  $\mu$ mol, 18% yield, 6:1 dr cis:trans). The dr was determined based on the integration of the resonances attributed to the benzylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on nOe analysis. For clarity, the <sup>1</sup>H NMR and <sup>13</sup>C NMR data of the major and minor diastereomers have been tabulated individually.

**TLC**  $R_f = 0.5$  (10% EtOAc/hexanes); **HRMS** (TOF MS ES+)  $m/z$  [M+H] calcd for C<sub>23</sub>H<sub>26</sub>NO<sub>2</sub>S<sub>2</sub> 380.1684, found 380.1689.

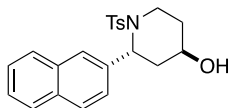
**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.80–7.76 (m, 4H), 7.73–7.71 (m, 1H), 7.63 (s, 1H), 7.46–7.44 (m, 3H), 7.28 (d,  $J = 8.0$  Hz, 2H), 5.48 (d,  $J = 4.5$  Hz, 1H), 3.96 (d,  $J = 14.4$  Hz, 1H), 3.06 (ddd,  $J = 14.0, 13.2, 3.1$  Hz, 1H), 2.69 (d,  $J = 25.9$  Hz, 1H), 2.42 (s, 3H), 2.30 (d,  $J = 13.3$  Hz, 1H), 1.43–1.36 (m, 2H), 0.98 (ddd,  $J = 24.5, 12.4, 4.5$  Hz, 1H), 0.82 (d,  $J = 6.5$  Hz, 3H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  143.1, 138.8, 136.7, 133.3, 132.3, 129.7 (2C), 128.4, 128.0, 127.5, 127.1 (2C), 126.1, 125.9, 125.8, 125.1, 55.6, 42.0, 36.0, 33.0, 25.3, 22.2, 21.5.

<sup>72</sup> Compound **1.25** was synthesized by Claire A. Herbert.

**Minor Diastereomer:**  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.80–7.76 (m, 4H), 7.73–7.71 (m, 1H), 7.63 (s, 1H), 7.46–7.44 (m, 3H), 7.28 (d,  $J = 8.0$  Hz, 2H), 5.25 (d,  $J = 4.9$  Hz, 1H), 3.88 (d,  $J = 10.6$  Hz, 1H), 3.04–2.98 (m, 1H), 2.42 (s, 3H), 2.38 (d,  $J = 3.86$  Hz, 1H), 2.14 (d,  $J = 13.7$  Hz, 1H), 1.43–1.36 (m, 2H), 0.79 (d,  $J = 6.5$  Hz, 3H), 0.75 (d,  $J = 6.4$  Hz, 1H);  $^{13}\text{C NMR}$  (151 MHz,  $\text{CDCl}_3$ )  $\delta$  143.1, 138.8, 136.7, 133.3, 132.3, 129.6 (2C), 129.3, 128.0, 127.6, 127.1 (2C), 126.1, 125.9, 125.8, 124.0, 55.2, 41.8, 36.0, 33.1, 25.2, 23.3, 21.5.



**N-(but-3-en-1-yl)-4-methylbenzenesulfonamide (1.34)** was prepared according to a procedure reported by Jiang.<sup>73</sup> To a flame-dried flask equipped with a stir bar was added 4-bromo-1-butene **1.32** (4.1 mL, 40. mmol, 1.0 equiv), *p*-toluenesulfonamide **1.33** (6.8 g, 40. mmol, 1.0 equiv),  $\text{K}_2\text{CO}_3$  (6.6 g, 48 mmol, 1.2 equiv), and MeCN (160 mL). The mixture was heated to 60 °C and allowed to stir for 3 d. The reaction mixture was quenched with saturated  $\text{NH}_4\text{Cl}$  (100 mL) and extracted with EtOAc (3 x 50 mL). The combined organic layers were washed with  $\text{H}_2\text{O}$  (50 mL) and brine (50 mL), dried over  $\text{Na}_2\text{SO}_4$ , and concentrated in vacuo. The residue was purified by column chromatography (5–25% EtOAc/hexanes) to afford the title compound as a clear, colorless oil (5.4 g, 24 mmol, 60 %). Analytical data is consistent with literature values.<sup>73</sup>  $^1\text{H NMR}$ : (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.76 (d,  $J = 8.2$ , 2H), 7.30 (d,  $J = 8.1$ , 2H), 5.63 (ddt,  $J = 17.1, 10.4, 6.8$ , 1H), 5.11 (br s, 1H), 5.02–4.93 (m, 2H), 2.99 (q,  $J = 6.7$ , 2H), 2.41 (s, 3H), 2.20 (q,  $J = 6.9$ , 2 H).



<sup>73</sup> Huang, J.; Zheng, J.; Wu, W.; Li, J.; Ma, J.; Ren, Y.; Jiang, H. *J. Org. Chem.* **2017**, *82*, 8191–8198.

**2-(Naphthalen-2-yl)-1-tosylpiperidin-4-ol (1.35)**<sup>74</sup> was prepared according to Method F. The following amounts of reagents were used: 2-naphthaldehyde (0.94 g, 6.0 mmol, 1.0 equiv), homoallylic sulfonamide **1.34** (1.1 mL, 6.0 mmol, 1.0 equiv), TFA (4.6 mL, 60. mmol, 10 equiv), and CH<sub>2</sub>Cl<sub>2</sub> (60 mL, 0.10 M). The residue was purified by flash column chromatography (0–30% EtOAc/hexanes) to afford the title compound as an orange solid (0.72 g, 1.8 mmol, 31 % yield, 5:1 dr trans:cis). The dr was determined based on the integration of the resonances attributed to the benzylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration of the major **34** was assigned based on analogy to compound **1.24**. For clarity, the <sup>1</sup>H NMR data of the major and minor diastereomers have been tabulated individually.

**TLC** R<sub>f</sub> = 0.1 (30% EtOAc/hexanes, stained with CAM); **HRMS** (TOF MS ES+) *m/z* [M+H] calcd for C<sub>22</sub>H<sub>24</sub>NO<sub>3</sub>S 382.1477, found 382.1483.

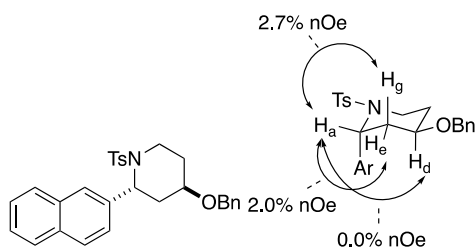
**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.79–7.77 (m, 4H), 7.68 (s, 1H), 7.49–7.41 (m, 4H), 7.29 (d, *J* = 8.1 Hz, 2H), 5.54 (d, *J* = 4.5 Hz, 1H), 3.99 (d, *J* = 15.0 Hz, 1H), 3.74 (tt, *J* = 10.9, 7.9 Hz, 1H), 3.03 (td, *J* = 15.3, 2.7 Hz, 1H), 2.63 (dt, *J* = 13.3, 2.0 Hz 1H), 2.43 (s, 3H), 1.70 (br s, 1H), 1.58 (ddd *J* = 13.6, 11.3, 5.5 Hz, 1H), 1.26–1.18 (m, 2H). <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 143.5, 138.3, 135.9, 133.3, 132.5, 130.0 (2C), 128.7, 128.0, 127.5, 127.0 (2C), 126.3, 126.2, 125.5, 124.7, 64.7, 55.8, 40.7, 36.2, 33.8, 21.6.

**Minor Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.74–7.70 (m, 4H), 7.63 (s, 1H), 7.60 (d, *J* = 8.3 Hz, 2H), 7.49–7.41 (m, 2H), 7.16 (d, *J* = 8.4 Hz, 2H), 5.10 (t, *J* = 5.1 Hz, 1H), 3.99 (d, *J* = 15.0 Hz, 1H), 3.67 (tt, *J* = 13.4, 4.6 Hz, 1H), 3.03 (td, *J* = 15.3, 2.7 Hz, 1H), 2.63 (dt, *J* = 13.3, 2.0 Hz 1H), 2.36 (s, 3H), 1.81–1.73 (m, 1H), 1.67 (br s, 1H), 1.26–1.18 (m, 2H). <sup>13</sup>C NMR (151 MHz,

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<sup>74</sup> Compound **1.35** was synthesized by Claire A. Herbert.

CDCl<sub>3</sub>)  $\delta$  143.3, 137.7, 135.9, 133.2, 132.5, 129.6 (2C), 128.7, 128.2, 127.5, 127.2 (2C), 126.3, 126.0, 125.3, 124.8, 65.1, 55.5, 39.0, 37.0, 31.9, 21.5.



**4-(Benzyloxy)-2-(naphthalen-2-yl)-1-tosylpiperidine (1.26)**<sup>75</sup> was prepared according to Method G. The following amounts of reagents were used: alcohol **1.35** (0.25 g, 0.66 mmol, 1.0 equiv), NaH (63 mg, 2.6 mmol, 4.0 equiv), benzyl bromide (90.  $\mu$ L, 0.73 mmol, 1.1 equiv), and THF (2.3 mL, 0.2 M). The residue was purified by column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a white solid (140 mg, 0.30 mmol, 56 % yield, 5:1 dr trans:cis). The dr was determined based on the integration of the resonances attributed to the benzylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on nOe analysis. For clarity, the <sup>1</sup>H NMR and <sup>13</sup>C NMR data of the major and minor diastereomers have been tabulated individually.

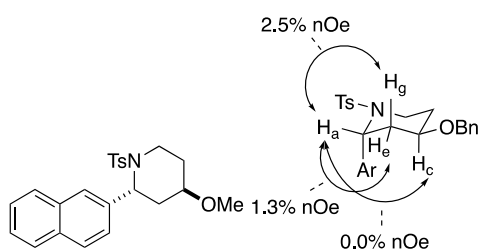
**TLC**  $R_f$  = 0.8 (20% EtOAc/hexanes, stained with CAM); **HRMS** (TOF MS ES+)  $m/z$  [M+Na] calcd for C<sub>29</sub>H<sub>29</sub>NO<sub>3</sub>SNa 494.1766, found 494.1758.

**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.80 (d,  $J$  = 8.1 Hz, 4H), 7.70 (d, 1H), 7.61 (s, 1H), 7.48–7.45 (m, 3H), 7.32–7.26 (m, 7H), 5.55 (d,  $J$  = 3.8 Hz, 1H), 4.50 (d,  $J$  = 11.9 Hz, 1H), 4.43 (d,  $J$  = 11.9 Hz, 1H), 4.02 (d,  $J$  = 14.7 Hz, 1H), 3.52 (tt,  $J$  = 10.8 Hz, 1H), 3.04 (td,  $J$  = 14.5, 2.5 Hz, 1H), 2.68 (d,  $J$  = 13.6 Hz, 1H), 2.43 (s, 3H), 1.80 (d,  $J$  = 11.5 Hz, 1H), 1.62 (ddd,  $J$  = 17.7, 11.9, 6.1 Hz, 1H), 1.31–1.34 (m, 1H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  143.4, 138.3, 136.0, 133.2,

<sup>75</sup> Compound **1.26** was synthesized by Claire A. Herbert.

132.5, 130.0 (2C), 129.5, 128.6, 128.5 (2C), 128.0, 127.8, 127.7 (2C), 127.5, 127.0 (2C), 126.2, 126.1, 125.5, 124.8, 71.3, 70.2, 55.8, 40.8, 33.4, 30.8, 21.6.

**Minor Diastereomer:**  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.80 (d,  $J = 8.1$  Hz, 4H), 7.70 (d, 1H), 7.59 (s, 1H), 7.48–7.45 (m, 3H), 7.32–7.26 (m, 2H), 7.15 (d,  $J = 8.2$  Hz, 1H), 7.07 (t,  $J = 7.6$  Hz, 1H), 6.96 (t,  $J = 7.3$  Hz, 2H), 6.69 (d,  $J = 7.6$  Hz, 1H), 5.17 (t,  $J = 5.1$  Hz, 1H), 4.24, 4.20 (ABq,  $J_{\text{AB}} = 12.3$  Hz, 2H), 3.80–3.68 (m, 3H), 2.53 (dt,  $J = 14.4, 9.4$  Hz, 1H), 2.35 (s, 3H), 2.06 (ddd,  $J = 14.3, 5.3, 2.9$  Hz, 1H), 1.81–1.80 (m, 2H), 1.31–1.34 (m, 1H);  $^{13}\text{C NMR}$  (151 MHz,  $\text{CDCl}_3$ )  $\delta$  143.4, 138.4, 136.0, 133.2, 132.5, 130.0 (2C), 129.5, 128.6, 128.5 (2C), 128.0, 127.8, 127.7 (2C), 127.5, 127.19 (2C), 127.16, 125.9, 125.6, 125.2, 71.2, 69.8, 55.8, 39.3, 34.2, 30.8, 21.6.



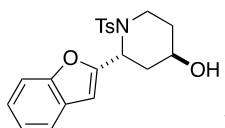
**4-Methoxy-2-(naphthalen-2-yl)-1-tosylpiperidine (1.12)**<sup>76</sup> was prepared according to Method G. The following amounts of reagents were used: alcohol **1.35** (110 mg, 0.30 mmol, 1.0 equiv), NaH (16 mg, 0.67 mmol, 2.2 equiv), methyl iodide (20.  $\mu\text{L}$ , 0.33 mmol, 1.1 equiv), and THF (1.5 mL, 0.20 M). The residue was purified by column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a pale yellow solid (61 mg, 0.15 mmol, 52 % yield, 5:1 dr trans:cis). The dr was determined based on the integration of the resonances attributed to the benzylic hydrogens in the  $^1\text{H NMR}$  spectrum. The relative configuration was assigned based on nOe analysis. For clarity, the  $^1\text{H NMR}$  and  $^{13}\text{C NMR}$  data of the major and minor diastereomers have been tabulated individually.

<sup>76</sup> Compound **1.12** was synthesized by Claire A. Herbert.

**TLC R<sub>f</sub>** = 0.6 (30% EtOAc/hexanes, stained with CAM); **HRMS** (TOF MS ES+) *m/z* [M+Na] calcd for C<sub>23</sub>H<sub>25</sub>NO<sub>3</sub>SNa 396.1633, found 396.1636.

**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.83–7.76 (m, 5H), 7.72 (s, 1H), 7.54–7.51 (m, 1H), 7.48–7.46 (m, 2H), 7.29 (d, *J* = 7.9 Hz, 2H), 5.56 (s, 1H), 4.04 (d, *J* = 14.1 Hz, 1H), 3.29 (tt, *J* = 7.4, 3.0 Hz, 1H), 3.25 (s, 3H), 3.09 (t, *J* = 13.1 Hz, 1H), 2.68 (d, *J* = 13.2 Hz, 1H), 2.42 (s, 3H), 1.80 (d, *J* = 11.5 Hz, 1H), 1.53 (td, *J* = 12.2 Hz, 1H), 1.16 (qd, *J* = 11.6, 5.8 Hz, 1H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 143.4, 138.4, 136.1, 133.4, 132.5, 130.0 (2C), 126.7, 128.1, 127.6, 127.0 (2C), 126.3, 126.1, 125.5, 124.7, 73.2, 55.8, 55.5, 40.8, 33.2, 30.2, 21.6.

**Minor Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.83–7.76 (m, 5H), 7.72 (s, 1H), 7.54–7.51 (m, 1H), 7.48–7.46 (m, 2H), 7.29 (d, *J* = 7.9 Hz, 2H), 4.99 (t, *J* = 5.38 Hz, 1H), 3.77–3.71 (m, 1H), 3.60 (dt, *J* = 13.6, 4.6 Hz, 1H), 3.42 (br s, 1H), 3.03 (s, 3H), 2.37–2.29 (m, 1H), 2.33 (s, 3H), 2.02 (d, *J* = 15.2 Hz, 1H), 1.86–1.82 (m, 1H), 1.72–1.68 (m, 1H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 143.0, 138.3, 137.4, 133.1, 132.5, 129.4 (2C), 126.7, 128.0, 127.5, 127.2 (2C), 125.8, 125.7, 125.5, 125.2, 73.5, 56.4, 55.5, 40.0, 34.1, 29.6, 21.5.



**2-(Naphthalen-2-yl)-1-tosylpiperidin-4-ol (1.36)**<sup>77</sup> was prepared according to Method F. The following amounts of reagents were used: 2-benzofurancarboxaldehyde (0.60 mL, 5.0 mmol, 1.0 equiv), homoallylic sulfonamide **1.34** (0.91 mL, 5.0 mmol, 1.0 equiv), TFA (3.8 mL, 50. mmol, 10 equiv), CH<sub>2</sub>Cl<sub>2</sub> (50 mL, 0.10 M). The residue was purified by flash column chromatography (0–50% EtOAc/hexanes) to afford the title compound as an orange solid (0.42 g, 1.1 mmol, 22% yield, 5:1 dr trans:cis). The dr was determined based on the integration of the resonances attributed to

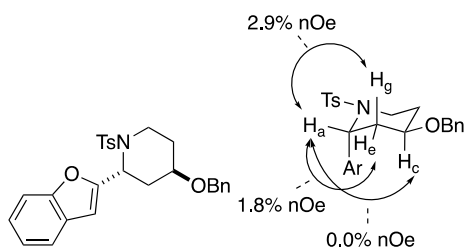
<sup>77</sup> Compound **1.36** was synthesized by Claire A. Herbert.

the benzylic hydrogens in the  $^1\text{H}$  NMR spectrum. The relative configuration was assigned based on analogy to compound **1.26**. For clarity, the  $^1\text{H}$  NMR data of the major and minor diastereomers have been tabulated individually.

**TLC R<sub>f</sub>** = 0.1 (30% EtOAc/hexanes, stained with CAM).

**Major Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.65 (d,  $J$  = 8.29 Hz, 2H), 7.46 (dd,  $J$  = 6.75, 2.12 Hz, 1H), 7.21–7.18 (m, 3H), 7.15 (d,  $J$  = 8.61 Hz, 2H), 6.49 (t,  $J$  = 2.0 Hz, 1H), 5.51 (d,  $J$  = 5.48 Hz, 1H), 3.97–3.90 (m, 2H), 3.23 (td,  $J$  = 13.5, 2.7 Hz, 1H), 2.51–2.45 (m, 1H), 2.33 (s, 3H), 1.94–1.88 (m, 1H), 1.75 (ddd,  $J$  = 13.0, 11.6, 5.9 Hz, 1H), 1.53 (d,  $J$  = 5.0 Hz, 1H), 1.44 (ddd,  $J$  = 24.1, 12.8, 4.5 Hz, 1H).

**Minor Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.65 (d,  $J$  = 8.29 Hz, 2H), 7.46 (dd,  $J$  = 6.75, 2.12 Hz, 1H), 7.21–7.18 (m, 3H), 7.15 (d,  $J$  = 8.61 Hz, 2H), 6.51 (t,  $J$  = 1.1 Hz, 1H), 5.33 (d,  $J$  = 5.6 Hz, 1H), 4.14–4.10 (m, 2H), 3.64 (td,  $J$  = 10.9, 4.0 Hz, 1H), 2.51–2.45 (m, 1H), 2.32 (s, 3H), ), 2.12 (ddd,  $J$  = 14.4, 6.7, 3.3 Hz, 1H), 1.94–1.88 (m, 1H), 1.53 (d,  $J$  = 5.0 Hz, 1H), 1.44 (ddd,  $J$  = 24.1, 12.8, 4.5 Hz, 1H).



**2-(benzofuran-2-yl)-4-(benzyloxy)-1-tosylpiperidine (1.27)**<sup>78</sup> was prepared according to method G. The following amounts of reagents were used: alcohol **1.36** (0.15 g, 0.40 mmol, 1.0 equiv), NaH (46 mg, 1.9 mmol, 4.7 equiv), benzyl bromide (52  $\mu\text{L}$ , 0.44 mmol, 1.1 equiv), and THF (3.0 mL, 0.2 M). The residue was purified by column chromatography (0–10% EtOAc/hexanes) to

<sup>78</sup> Compound **1.27** was synthesized by Claire A. Herbert.

afford the title compound as a yellow solid (87 mg, 0.19 mmol, 47% yield, 5:1 dr trans:cis). The dr was determined based on the integration of the resonances attributed to the benzylic hydrogens in the  $^1\text{H}$  NMR spectrum. The relative configuration was assigned based on nOe analysis. For clarity, the  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR data of the major and minor diastereomers have been tabulated individually.

**TLC**  $R_f$  = 0.8 (30% EtOAc/hexanes, stained with CAM); **HRMS** (TOF MS ES+)  $m/z$  [M+Na] calcd for  $\text{C}_{27}\text{H}_{27}\text{NO}_4\text{SNa}$  484.1559, found 484.1542.

**Major Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.65 (d,  $J$  = 8.3 Hz, 2H), 7.45, (d,  $J$  = 8.3 Hz, 1H), 7.30–7.17 (m, 8H), 7.15 (d,  $J$  = 8.2 Hz, 2H), 6.44 (s, 1H), 5.52 (d,  $J$  = 5.3 Hz, 1H), 4.49 (s, 2H), 3.94 (d,  $J$  = 13.7 Hz, 1H), 3.66 (tt,  $J$  = 11.2, 4.0 Hz, 1H), 3.20 (td,  $J$  = 13.4, 2.6 Hz, 1H), 2.57 (dt,  $J$  = 13.2, 1.8 Hz, 1H), 2.32 (s, 3H), 1.97 (d,  $J$  = 12.3 Hz, 1H), 1.78 (ddd,  $J$  = 13.0, 11.7, 5.8 Hz, 1H), 1.45 (qd,  $J$  = 12.8, 4.8 Hz, 1H);  $^{13}\text{C}$  NMR (151 MHz,  $\text{CDCl}_3$ )  $\delta$  155.3, 154.7, 143.3, 138.2, 137.2, 129.5 (2C), 128.5 (2C), 128.1, 128.0, 127.8, 127.1, 126.9, 124.1, 122.9, 120.9, 111.1, 104.8, 71.8, 70.2, 51.7, 41.4, 34.2, 31.3, 21.5.

**Minor Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.68 (d,  $J$  = 8.3 Hz, 2H), 7.35, (d,  $J$  = 4.4 Hz, 1H), 7.30–7.17 (m, 5H), 7.07 (t,  $J$  = 7.5 Hz, 1H), 6.99 (t,  $J$  = 7.6, 2H), 6.79 (d,  $J$  = 7.6 Hz, 2H), 6.42 (s, 1H), 5.36 (d,  $J$  = 6.4 Hz, 1H), 4.28 (s, 2H), 3.76–3.73 (m, 2H), 3.66 (tt,  $J$  = 11.2, 4.0 Hz, 1H), 2.70 (d,  $J$  = 14.1 Hz, 1H), 2.32 (s, 3H), 1.97 (d,  $J$  = 12.3 Hz, 1H), 1.83 (d,  $J$  = 13.8 Hz, 1H), 1.72–1.68 (m, 1H);  $^{13}\text{C}$  NMR (151 MHz,  $\text{CDCl}_3$ )  $\delta$  155.3, 154.7, 143.3, 138.2, 137.2, 129.5 (2C), 128.5 (2C), 128.1, 128.0, 127.8, 127.1, 126.9, 123.6, 122.6, 120.7, 110.9, 103.2, 71.8, 70.0, 49.6, 37.6, 31.0, 29.3, 15.3.



## C–C Bond Formation Through Cross-Electrophile Coupling Reactions

## 2.1 Introduction

Cross-electrophile coupling (XEC) reactions are excellent methods to construct carbon-carbon bonds from widely commercially available electrophiles.<sup>1,2</sup> An advantage of XEC reactions is that they do not require preformed organometallic reagents. Typically, the organometallic reagents used in traditional cross-coupling (XC) reactions are prepared from the corresponding alkyl or aryl halides, whereas XEC reactions allow for the direct conversion of these electrophiles. Unlike traditional XC reactions where the electrophile favors oxidative addition and the organometallic reagent favors transmetalation, XEC reactions contain two electrophiles that favor oxidative addition. Therefore, the transition metal catalyst must be able to differentiate the two electrophiles in order to obtain cross-selectivity. This inherent challenge resulted in delayed efforts to explore and optimize these reactions, when compared to traditional XC reactions. Some of the strategies for optimal cross-selectivity that have been developed include electronic differentiation of starting materials, steric matching between catalyst and substrate, and tethering of the electrophiles.<sup>2a,2h</sup>

In this Chapter, inter- and intramolecular XEC reactions will be discussed. It will focus on the dimerization and cross-selective XEC reactions of C(sp<sup>2</sup>) electrophiles, including aryl and vinyl halides (Scheme 2.1, Type A). The next two classes of XEC reactions between C(sp<sup>2</sup>)

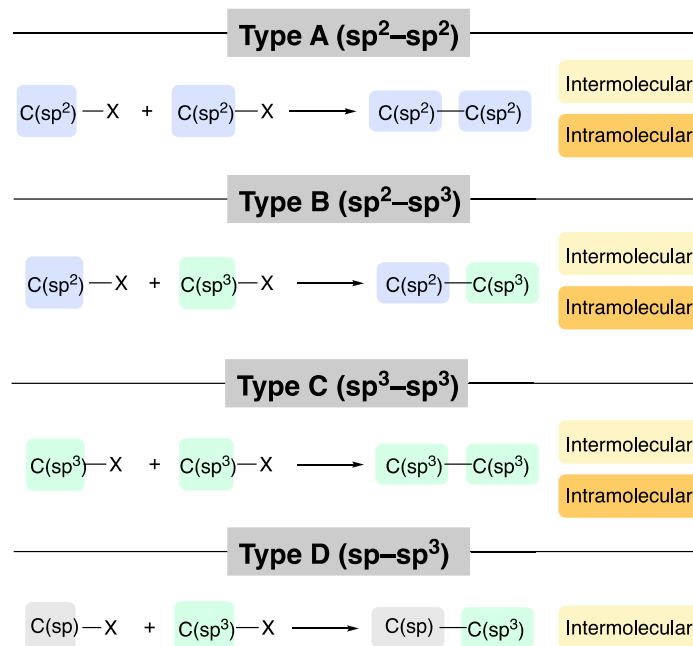
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<sup>1</sup> Portions of this Chapter have been published in *Comprehensive Organometallic Chemistry*, see: Hewitt, K. A.; Lin, P. C.; Raffman, E. T. A.; Jarvo, E. R. C–C Bond Formation Through Cross-Electrophile Coupling Reactions. In *Comprehensive Organometallic Chemistry IV*; Elsevier, **2021**.

<sup>2</sup> (a) Everson, D. A.; Weix, D. J. *J. Org. Chem.* **2014**, *79*, 4793–4798. (b) Knappke, C. E. I.; Grupe, S.; Gärtner, D.; Corpet, M.; Gosmini, C.; Jacobi Von Wangelin, A. *Chem. Eur. J.* **2014**, *20*, 6828–6842. (c) Poremba, K. E.; Dibrell, S. E.; Reisman, S. E. *ACS Catal.* **2020**, *10*, 8237–8246. (d) Gu, J.; Wang, X.; Xue, W.; Gong, H. *Org. Chem. Front.* **2015**, *2*, 1411–1421. (e) Weix, D. J. *Acc. Chem. Res.* **2015**, *48*, 1767–1775. (f) Iwasaki, T.; Kambe, N. *Top. Curr. Chem.* **2016**, *374*, 1–36. (g) Wang, X.; Dai, Y.; Gong, H. *Top. Curr. Chem.* **2016**, *374*, 1–29. (h) Lucas, E. L.; Jarvo, E. R. *Nat. Rev. Chem.* **2017**, *1*, 0065. (i) Campeau, L. C.; Hazari, N. *Organometallics* **2019**, *38*, 3–35. (j) Jin, Y.; Wang, C. *Synlett* **2020**, *31*, 1843–1850. (k) Liu, J.; Ye, Y.; Sessler, J. L.; Gong, H. *Acc. Chem. Res.* **2020**, *53*, 1833–1845. (l) Sanford, A. B.; Jarvo, E. R. *Synlett* **2021**, *32*, 1151–1156. (m) Xue, W.; Jia, X.; Wang, X.; Tao, X.; Yin, Z.; Gong, H. *Chem. Soc. Rev.* **2021**, *50*, 4162–4184. (n) Li, Y.; Fan, Y.; Jia, Q. *Chinese J. Org. Chem.* **2019**, *39*, 350.

and C(sp<sup>3</sup>) electrophiles (Type B) and C(sp<sup>3</sup>) electrophiles (Type C) are covered elsewhere.<sup>1,3</sup> Finally, the least common XEC reaction, coupling of C(sp) and C(sp<sup>3</sup>) electrophiles, will be outlined (Type D). This Chapter will be broken into two sections to highlight the use of stoichiometric reducing metals (Section 2.3) and electrochemical reductions (Section 2.4).

**Scheme 2.1** Patterns in Cross-Electrophile Coupling Reactions



The focus of this Chapter will be transition metal-catalyzed XEC reactions with stoichiometric reducing agents and electrochemical reductions to forge C–C bonds. Commonly in the literature, these transformations are referred to as “reductive coupling”, “reductive cross-coupling” or “cross-electrophile coupling” reactions. Furthermore, XEC Reactions of type A are often referred to as “Ullman-type coupling” reactions.<sup>4</sup> I will employ the term XEC reaction for all of these carbon-carbon bond forming reactions conducted under reducing conditions. I will not include examples of dual photoredox-transition metal catalysis for the formation of C–

<sup>3</sup> For a full discussion on Type B and Type C written by Patricia C. Lin and Ethan T. A. Raffman, see ref. 1

<sup>4</sup> (a) Fanta, P. E. *Chem. Rev.* **1946**, *38*, 139–196. (b) Sainsbury, M. *Tetrahedron* **1980**, *36*, 3327–3359. (c) Bringmann, G.; Walter, R.; Weirich, R. *Angew. Chem. Int. Ed.* **1990**, *29*, 977–991. (d) Hassan, J.; Sévignon, M.; Gozzi, C.; Schulz, E.; Lemaire, M. *Chem. Rev.* **2002**, *102*, 1359–1470. (e) Fanta, P. E. *Synthesis* **1974**, *1974*, 9–21.

C bonds.<sup>5</sup> In addition, reductive coupling reactions utilizing CO<sub>2</sub><sup>6</sup> or carbonyls<sup>7</sup> have been extensively covered elsewhere. Conjunctive or interrupted XEC reactions, where two organohalides are combined with an olefin or alkyne, will also not be included in this Chapter.<sup>8</sup>

## 2.2 Proposed Mechanisms of Cross-Electrophile Coupling (XEC) Reactions

Several mechanisms have been proposed for XEC reactions to account for formation of the observed products.<sup>2c,2e,2h</sup> The mechanisms are outlined below, using as an example a nickel catalyst and reducing metal, designated as M, to illustrate general features of each catalytic cycle. The common mechanisms include: (I) in-situ formation of the organometallic reagent (II) sequential reduction (III) radical chain and (IV) oxidative addition, intramolecular S<sub>N</sub>2 (Figure 2.1). All mechanisms share certain key features with traditional cross-coupling (XC) reactions such as oxidative addition (OA) and reductive elimination (RE) steps.<sup>9</sup> As in XC reactions, oxidative events can occur via polar or radical pathways. However in XEC reactions, excluding mechanism (I), there are no stoichiometric organometallic reagents, and reduction events are crucial for product formation and catalytic turnover. In addition, the reduction steps can be achieved with stoichiometric reducing metals, such as Zn or Mn, or electrochemically.

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<sup>5</sup> (a) Prier, C. K.; MacMillan, D. W. C. Dual Photoredox Catalysis: The Merger of Photoredox Catalysis with Other Catalytic Activation Modes. In *Visible Light Photocatalysis in Organic Chemistry*; Wiley-VCH: Weinheim, Germany, 2018; pp 299–333. (b) Milligan, J. A.; Phelan, J. P.; Badir, S. O.; Molander, G. A. *Angew. Chem. Int. Ed.* **2019**, *58*, 6152–6163. (c) Tellis, J. C.; Kelly, C. B.; Primer, D. N.; Jouffroy, M.; Patel, N. R.; Molander, G. A. *Acc. Chem. Res.* **2016**, *49*, 1429–1439. (d) Tóth, B. L.; Tischler, O.; Novák, Z. *Tetrahedron Lett.* **2016**, *57*, 4505–4513. (e) Skubi, K. L.; Blum, T. R.; Yoon, T. P. *Chem. Rev.* **2016**, *116*, 10035–10074. (f) Gui, Y.-Y.; Sun, L.; Lu, Z.-P.; Yu, D.-G. *Org. Chem. Front.* **2016**, *3*, 522–526.

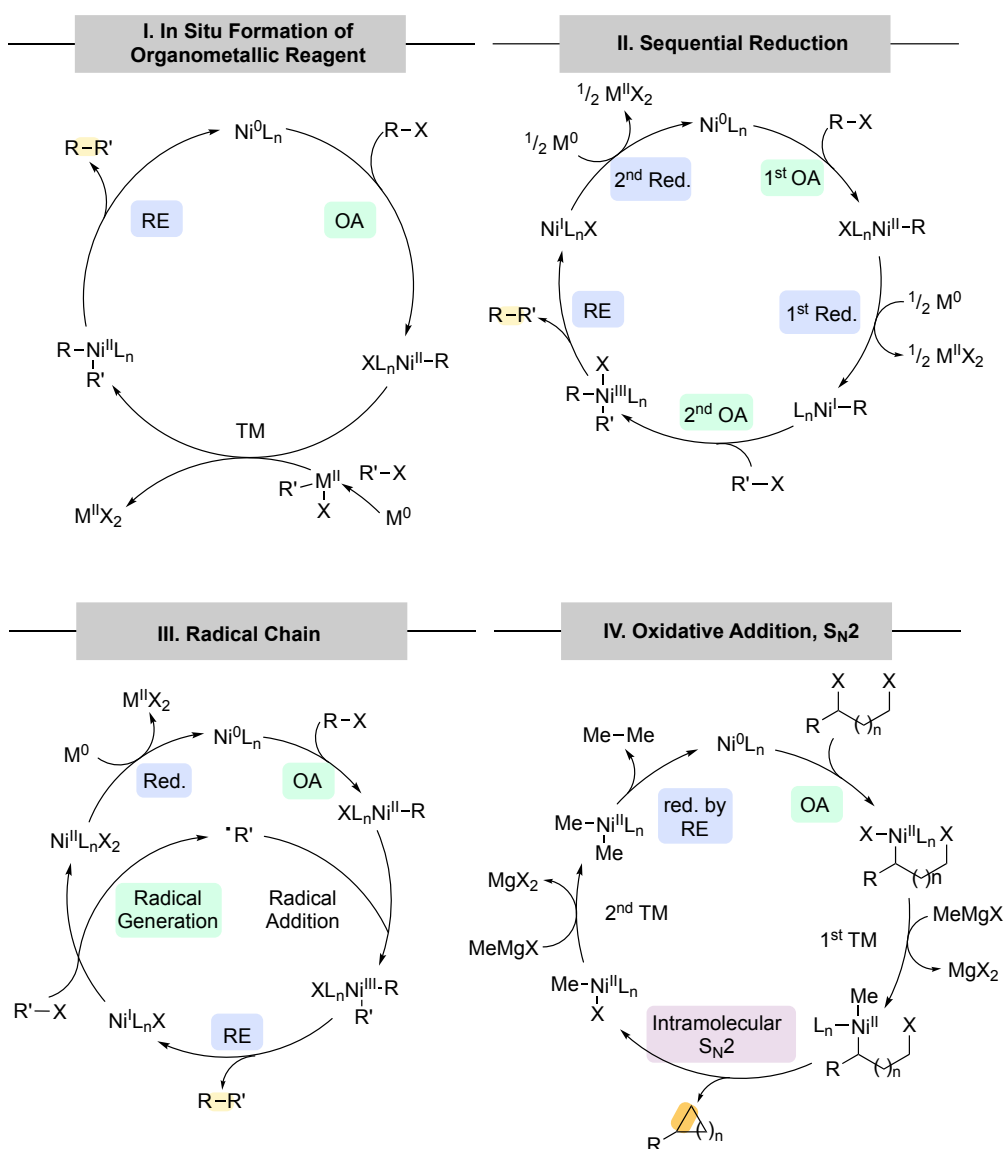
<sup>6</sup> (a) Börjesson, M.; Moragas, T.; Gallego, D.; Martin, R. *ACS Catal.* **2016**, *6*, 6739–6749. (b) Moragas, T.; Correa, A.; Martin, R. *Chem. Eur. J.* **2014**, *20*, 8242–8258. (c) Correa, A.; León, T.; Martin, R. *J. Am. Chem. Soc.* **2014**, *136*, 1062–1069. (d) Tortajada, A.; Börjesson, M.; Martin, R. *Acc. Chem. Res.* **2021**, *54*, 3941–3952. (e) Davies, J.; Lyonnet, J. R.; Zimin, D. P.; Martin, R. *Chem.* **2021**, *7*, 2927–2942.

<sup>7</sup> (a) Fürstner, A. *Chem. Rev.* **1999**, *99*, 991–1046. (b) Hargaden, G. C.; Guiry, P. J. *Adv. Synth. Catal.* **2007**, *349*, 2407–2424. (c) Santana, C. G.; Krische, M. J. *ACS Catal.* **2021**, *11*, 5572–5585.

<sup>8</sup> (a) Durandetti, M.; Hardou, L.; Lhermet, R.; Rouen, M.; Maddaluno, J. *Chem. Eur. J.* **2011**, *17*, 12773–12783. (b) Derosa, J.; Tran, V. T.; van der Puyl, V. A.; Engle, K. M. *Aldrichimica Acta* **2018**, *51*, 21–32. (c) Dhungana, R. K.; KC, S.; Basnet, P.; Giri, R. *Chem. Rec.* **2018**, *18*, 1314–1340. (d) Qi, X.; Diao, T. *ACS Catal.* **2020**, *10*, 8542–8556. (e) Derosa, J.; Apolinar, O.; Kang, T.; Tran, V. T.; Engle, K. M. *Chem. Sci.* **2020**, *11*, 4287–4296. (f) Diccianni, J.; Lin, Q.; Diao, T. *Acc. Chem. Res.* **2020**, *53*, 906–919.

<sup>9</sup> Hartwig, J. F. *Organotransition Metal Chemistry: From Bonding to Catalysis*; University Science Books, **2010**.

**Figure 2.1:** Proposed Mechanisms for Cross-Electrophile Coupling (XEC) Reactions.



When stoichiometric reducing metals, such as Zn, are employed it is feasible to propose in-situ formation of organometallic reagents (Figure 2.1, I). The catalytic cycle is then that of a traditional cross-coupling reaction, wherein oxidative addition occurs with one electrophile. Transmetalation (TM) occurs with the organometallic reagent that was formed in situ. Finally, reductive elimination (RE) produces the desired C–C bond. Identifying this mechanistic manifold is often reasonably straightforward by substitution of one electrophile and the reducing metal with the corresponding organometallic reagent. An alternative test to exclude this mechanism is to demonstrate that the XEC reaction can be conducted with organic

reductants, such as tetrakis(dimethylamino)ethylene (TDAE), which is inconsistent with the formation of an organometallic reagent in situ.<sup>2b,10</sup>

The majority of XEC reactions typically undergo either sequential reduction or radical chain mechanisms (Figure 2.1, II and III). The Weix and Durandetti groups have performed extensive studies to elucidate the key steps of these mechanisms.<sup>2a,2e,11,12</sup> The two catalytic cycles are similar, in that both involve two oxidative events and two reductive events, and one oxidative event often involves formation of an alkyl radical. In a sequential reduction mechanism (Figure 2.1, II), one electrophile selectively engages the catalyst for oxidative addition, frequently by a concerted oxidative addition. This step is followed by reduction of the catalyst to form the Ni(I) intermediate. A second OA then occurs, and when C(sp<sup>3</sup>) electrophiles are employed, this step frequently involves halogen atom abstraction and a short-lived organic radical.<sup>13,14</sup> Subsequent reductive elimination produces the C–C bond. A second reduction step turns over the catalyst.

In contrast, the radical chain mechanism involves a radical propagation step (Figure 2.1, III). The Ni(II) intermediate generated from oxidative addition captures the radical intermediate and leads to a Ni(III) complex. This species promptly undergoes reductive elimination, generating the desired product and a Ni(I) intermediate.<sup>15,16</sup> Next the Ni(I) species generates the alkyl radical via halogen atom abstraction. The resulting Ni(II) halide complex can be reduced to Ni(0) to re-enter the catalytic cycle.

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<sup>10</sup> Kuroboshi, M.; Waki, Y.; Tanaka, H. *J. Org. Chem.* **2003**, *68*, 3938–3942.

<sup>11</sup> Biswas, S.; Weix, D. J. *J. Am. Chem. Soc.* **2013**, *135*, 16192–16197.

<sup>12</sup> Durandetti, M.; Nédélec, J. Y.; Périchon, J. *J. Org. Chem.* **1996**, *61*, 1748–1755.

<sup>13</sup> Kehoe, R.; Mahadevan, M.; Manzoor, A.; McMurray, G.; Wienefeld, P.; Baird, M. C.; Budzelaar, P. H. M. *Organometallics* **2018**, *37*, 2450–2467.

<sup>14</sup> Diccianni, J. B.; Katigbak, J.; Hu, C.; Diao, T. *J. Am. Chem. Soc.* **2019**, *141*, 1788–1796.

<sup>15</sup> Tsou, T. T.; Kochi, J. K. *J. Am. Chem. Soc.* **1979**, *101*, 6319–6332.

<sup>16</sup> Tsou, T. T.; Kochi, J. K. *J. Am. Chem. Soc.* **1979**, *101*, 7547–7560.

Recently, the Jarvo and Hong groups reported a distinct mechanism for the intramolecular XEC reaction (Figure 2.1, IV).<sup>17,18</sup> In contrast to the sequential reduction and radical chain mechanisms, this mechanism involves a single oxidative addition and reductive elimination event. These reactions have employed methylmagnesium iodide, not as a coupling partner, but as the terminal reducing agent. In this mechanism, oxidative addition selectively occurs at the benzylic or secondary electrophile. Transmetalation with the Grignard reagent produces the key alkylnickel intermediate which undergoes the proposed intramolecular S<sub>N</sub>2-type reaction to afford the cyclized product. Subsequent transmetalation and reductive elimination of ethane regenerates the active nickel catalyst.

The mechanism for a given XEC reaction can be difficult to predict by simple inspection of the starting materials, catalyst, and reagents. In particular, deciphering whether the sequential reduction or radical chain mechanism is operative is quite difficult and requires extensive mechanistic studies. However, it is crucial to identify and elucidate the key mechanistic steps as it allows for development of new methods that build upon these elementary steps.

## 2.3 XEC Reactions Employing Stoichiometric Reductants

### 2.3.1 Reactions of C(sp<sup>2</sup>) Electrophiles (Type A)

#### 2.3.1.1 Dimerization Reactions of C(sp<sup>2</sup>) electrophiles

The reductive dimerization reaction of aryl halides has been known since the early twentieth century when Ullman and co-workers reported the first reductive dimerization reaction of an aryl bromide in the presence of copper powder in 1901.<sup>4,19</sup> This original report employed stoichiometric copper powder and high temperatures (>200 °C). Since this

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<sup>17</sup> Chen, P.-P.; Lucas, E. L.; Greene, M. A.; Zhang, S.-Q.; Tollefson, E. J.; Erickson, L. W.; Taylor, B. L. H.; Jarvo, E. R.; Hong, X. *J. Am. Chem. Soc.* **2019**, *141*, 5835–5855.

<sup>18</sup> Sanford, A. B.; Thane, T. A.; McGinnis, T. M.; Chen, P. P.; Hong, X.; Jarvo, E. R. *J. Am. Chem. Soc.* **2020**, *142*, 5017–5023.

<sup>19</sup> Ullmann, F.; Bielecki, J. *Berichte der Dtsch. Chem. Gesellschaft* **1901**, *34*, 2174–2185.

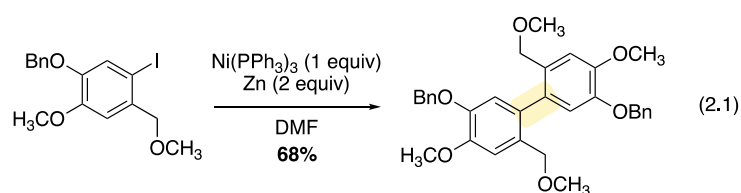
publication, stoichiometric and catalytic variations have been developed to accomplish C–C bond formation. A wide range of aryl, heteroaryl, and vinyl halides and pseudohalides have been employed (Table 2.1).

**Table 2.1:** Dimerization Reactions of C(sp<sup>2</sup>) Electrophiles

Entry	Starting Material <sup>a</sup>	Product	Metal Catalyst	Reducing Agent
1			Ni, Pd, Co	Cu, Zn, Mn, TDAE, Mg, LiH
2			Ni, Pd	Cu, Zn, Polyethylene Glycol
3			Cu, Ni	Zn
4			Cu, Ni	Zn

<sup>a</sup>X = I, Br, Cl, OTs, OTf, or OMs

Early examples of reductive dimerization reactions were mediated by reducing metals such as Cu, Ni and Pd to accomplish the C–C bond formation.<sup>10,19,20,21,22,23,24,25,26</sup> In 1975, Kende and co-workers reported the synthesis of highly substituted biaryls with one equivalent of a nickel triphenylphosphine complex (eq 2.1).<sup>21</sup>



<sup>20</sup> Semmelhack, M. F.; Helquist, P. M.; Jones, L. D. *J. Am. Chem. Soc.* **1971**, *93*, 5908–5910.

<sup>21</sup> Kende, A. S.; Liebeskind, L. S.; Braitsch, D. M. *Tetrahedron Lett.* **1975**, *16*, 3375–3378.

<sup>22</sup> Semmelhack, M. F.; Helquist, P.; Jones, L. D.; Keller, L.; Mendelson, L.; Ryono, L. S.; Gorzynski Smith, J.; Stauffer, R. D. *J. Am. Chem. Soc.* **1981**, *103*, 6460–6471.

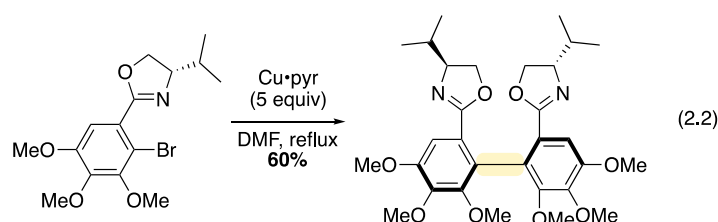
<sup>23</sup> Inaba, S.; Matsumoto, H.; Rieke, R. D. *Tetrahedron Lett.* **1982**, *23*, 4215–4216.

<sup>24</sup> Matsumoto, H.; Inaba, S.; Rieke, R. D. *J. Org. Chem.* **1983**, *48*, 840–843.

<sup>25</sup> Reisch, H. A.; Enkelmann, V.; Scherf, U. *J. Org. Chem.* **1999**, *64*, 655–658.

<sup>26</sup> Zhang, S.; Zhang, D.; Liebeskind, L. S. *J. Org. Chem.* **1997**, *62*, 2312–2313.

In addition, stoichiometric metals have been utilized to synthesize dimeric heterocycles.<sup>27</sup> Asymmetric variations have been developed for the synthesis of biphenyl compounds by incorporating a chiral auxiliary on the aryl halide moiety (eq 2.2).<sup>28,29,30,31</sup> This strategy has been applied to the total synthesis of axially chiral natural products, such as *o*-permethyl tellimagrandin I.<sup>32</sup>



It was realized in the early 1990s that catalytic quantities of metal complexes could also accomplish biaryl bond formation, if a stoichiometric amount of a sacrificial reducing agent was included.<sup>21,22,33,34,35,36,37,38,39,40,41,42,43,44,45,46</sup> Such bimetallic methods are advantageous because an abundant and inexpensive reducing agent can be employed to affect turnover of a transition-metal catalyst that is more precious and/or more toxic.<sup>47</sup> Typically, the sacrificial metal reductants utilized in XEC reactions are zinc and manganese. As an example, in 1995 Percec and co-workers reported the dimerization reaction of aryl sulfonates in the presence of

<sup>27</sup> Tiecco, M.; Testaferri, L.; Tingoli, M.; Chianelli, D.; Montanucci, M. *Synthesis* **1984**, 1984, 736–738.

<sup>28</sup> Meyers, A. I.; McKennon, M. J. *Tetrahedron Lett.* **1995**, 36, 5869–5872.

<sup>29</sup> Nelson, T. D.; Meyers, A. I. *Tetrahedron Lett.* **1993**, 34, 3061–3062.

<sup>30</sup> Nelson, T. D.; Meyers, A. I. *Tetrahedron Lett.* **1994**, 35, 3259–3262.

<sup>31</sup> Nelson, T. D.; Meyers, A. I. *J. Org. Chem.* **1994**, 59, 2655–2658.

<sup>32</sup> Nelson, T. D.; Meyers, A. I. *J. Org. Chem.* **1994**, 59, 2577–2580.

<sup>33</sup> Zembayashi, M.; Tamao, K.; Yoshida, J.; Kumada, M. *Tetrahedron Lett.* **1977**, 18, 4089–4091.

<sup>34</sup> Colon, I.; Kelsey, D. R. *J. Org. Chem.* **1986**, 51, 2627–2637.

<sup>35</sup> Iyoda, M.; Otsuka, H.; Sato, K.; Nisato, N.; Oda, M. *Bull. Chem. Soc. Jpn.* **1990**, 63, 80–87.

<sup>36</sup> Percec, V.; Bae, J. Y.; Zhao, M.; Hill, D. H. *J. Org. Chem.* **1995**, 60, 176–185.

<sup>37</sup> Massicot, F.; Schneider, R.; Fort, Y. *J. Chem. Res.* **1999**, 2, 664–665.

<sup>38</sup> Leadbeater, N. E.; Resouly, S. M. *Tetrahedron Lett.* **1999**, 40, 4243–4246.

<sup>39</sup> Howarth, J.; James, P.; Dai, J. *Tetrahedron Lett.* **2000**, 41, 10319–10321.

<sup>40</sup> Zuo, Z.; Kim, R. S.; Watson, D. A. *J. Am. Chem. Soc.* **2021**, 143, 1328–1333.

<sup>41</sup> Takagi, K.; Hayama, N.; Sasaki, K. *Bull. Chem. Soc. Jpn.* **1984**, 57, 1887–1890.

<sup>42</sup> Jutand, A.; Mosleh, A. *Synlett* **1993**, 568–570.

<sup>43</sup> Jutand, A.; Mosleh, A. *J. Org. Chem.* **1997**, 62, 261–274.

<sup>44</sup> Wang, L.; Zhang, Y.; Liu, L.; Wang, Y. *J. Org. Chem.* **2006**, 71, 1284–1287.

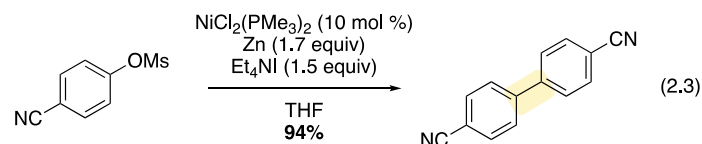
<sup>45</sup> Bhattacharjya, A.; Klumphu, P.; Lipshutz, B. H. *Nat. Commun.* **2015**, 6, 1–6.

<sup>46</sup> Moncomble, A.; Le Floch, P.; Gosmini, C. *Chem. Eur. J.* **2009**, 15, 4770–4774.

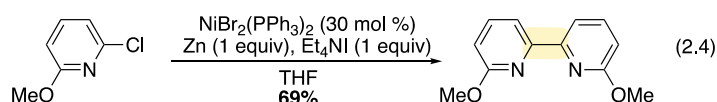
<sup>47</sup> Kasprzak, K. S.; Diwan, B. A.; Rice, J. M.; Misra, M.; Riggs, C. W.; Olinski, R.; Dizdaroglu, M. *Chem. Res. Toxicol.* **1992**, 5, 809–815.



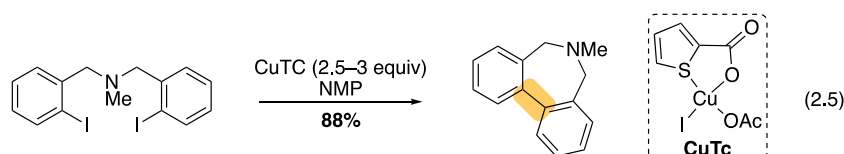
a nickel catalyst and zinc powder (eq 2.3).<sup>36</sup> In addition to aryl electrophiles, the reductive dimerization reactions of vinyl halides have been employed to afford symmetrical dienes.<sup>21,22,26,40</sup>



The dimerization reaction of halopyridines has also been established (eq. 2.4).<sup>35,38,44,48</sup> This method has been employed for the synthesis of bipyridine ligands, which are a common ligand scaffold used in XEC reactions themselves.<sup>49,50,51,52</sup> Additionally, heterocycles such as thiophene, thiazole, pyrrole, and furan undergo efficient dimerization reactions.<sup>22,26,33,34,41,44,45,53,54</sup>



Finally, intramolecular variations have been reported, including formation of medium-sized rings.<sup>22,26</sup> For example, Liebeskind and co-workers demonstrated that seven membered rings could be constructed via an intramolecular XEC reaction (eq 2.5).<sup>26</sup>



### 2.3.1.2 Cross-Selective XEC Reactions of C(sp<sup>2</sup>) Electrophiles

The XEC reaction between two different C(sp<sup>2</sup>) hybridized electrophiles was not realized until the early 2000's.<sup>55</sup> Since then, a wide range of cross-selective reactions that

<sup>48</sup> Chan, K. S.; Tse, A. K. S. *Synth. Commun.* **1993**, *23*, 1929–1934.

<sup>49</sup> Constable, E. C.; Morris, D.; Carr, S. *New J. Chem.* **1998**, *22*, 287–294.

<sup>50</sup> Zhang, B.; Breslow, R. *J. Am. Chem. Soc.* **1997**, *119*, 1676–1681.

<sup>51</sup> Janiak, C. *Synthesis* **1999**, 959–964.

<sup>52</sup> Wong, H. L.; Tian, Y.; Chan, K. S. *Tetrahedron Lett.* **2000**, *41*, 7723–7726.

<sup>53</sup> Venkatraman, S.; Li, C.-J. *Org. Lett.* **1999**, *1*, 1687–1687.

<sup>54</sup> Sessler, J. L.; Hoehner, M. C. *Synlett* **1994**, 211–212.

<sup>55</sup> Hassan, J.; Hathroubi, C.; Gozzi, C.; Lemaire, M. *Tetrahedron* **2001**, *57*, 7845–7855.

combine aryl halides and pseudohalides, as well as vinyl halides and pseudohalides, acid chlorides, and enones have been reported (Table 2.2).

**Table 2.2:** Cross-Selective XEC Reactions of C(sp<sup>2</sup>) Electrophiles

Entry	Starting Material 1 <sup>a</sup>	Starting Material 2 <sup>a</sup>	Product	Metal Catalyst	Reducing Agent
1				Ni, Pd, Co, Fe	Zn, Mn, Mg, B <sub>2</sub> (pin) <sub>2</sub> Polyethylene glycol
2				Ni, Co, Fe	Zn, Mn, Mg
3				Ni, Pd	Zn
4				Ni, Pd	Zn
5				Ni	Mn
6				Co	Mn

<sup>a</sup>X = I, Br, Cl, CN, OTs, OTf, OMs, OAc, SO<sub>2</sub>CF<sub>2</sub>H, NR<sub>2</sub>, OCOR, or O*t*-Bu

A major goal in the development of these reactions is achieving cross-selectivity between two electrophiles under practical conditions with synthetically useful yields. There have been several successful strategies employed including using excess of one C(sp<sup>2</sup>) electrophile,<sup>41,55,56</sup> electronic differentiation of the two electrophiles<sup>57,58,59</sup> or the use of co-catalysts.<sup>60,61,62,63,64</sup>

<sup>56</sup> Amatore, M.; Gosmini, C. *Angew. Chem. Int. Ed.* **2008**, *47*, 2089–2092.

<sup>57</sup> Qian, Q.; Zang, Z.; Wang, S.; Chen, Y.; Lin, K.; Gong, H. *Synlett* **2013**, *24*, 619–624.

<sup>58</sup> Chen, Z.; Wang, X. *A Org. Biomol. Chem.* **2017**, *15*, 5790–5796.

<sup>59</sup> Dorval, C.; Tricoire, M.; Begouin, J. M.; Gandon, V.; Gosmini, C. *ACS Catal.* **2020**, *10*, 12819–12827.

<sup>60</sup> Ackerman, L. K. G.; Lovell, M. M.; Weix, D. J. *Nature* **2015**, *524*, 454.

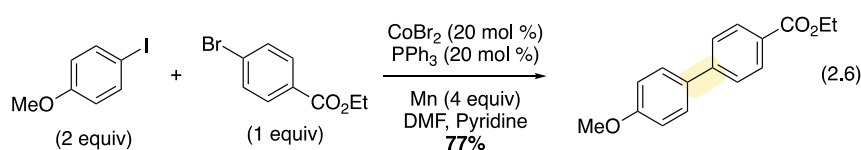
<sup>61</sup> Hanna, L. E.; Jarvo, E. R. *Angew. Chem. Int. Ed.* **2015**, *54*, 15618–15620.

<sup>62</sup> Huang, L.; Ackerman, L. K. G.; Kang, K.; Parsons, A. M.; Weix, D. J. *J. Am. Chem. Soc.* **2019**, *141*, 10978–10983.

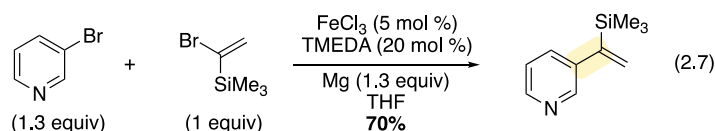
<sup>63</sup> Xiong, B.; Li, Y.; Wei, Y.; Kramer, S.; Lian, Z. *Org. Lett.* **2020**, *22*, 6334–6338.

<sup>64</sup> Kang, K.; Huang, L.; Weix, D. J. *J. Am. Chem. Soc.* **2020**, *142*, 10634–10640.

For example, in a reaction of aryl halides, Gosmini and co-workers utilized an excess of the more reactive aryl iodide to ensure conversion of the less reactive aryl bromide to the cross product (eq 2.6). In addition, the authors noted that the aryl halides must be electronically differentiated in order to achieve the desired unsymmetrical biaryl.<sup>56</sup> This requirement can lead to limitations in substrate scope. Similar methods, including nickel- and palladium-catalyzed reactions, involve the XEC reaction of heterocyclic electrophiles.<sup>10,20,21,44,55,56,57,60,62,65,66,67,68,69</sup>



Another successful strategy to promote cross-reactivity is to employ two C(sp<sup>2</sup>) electrophiles with different electronic or steric features. For example, Wangelin and co-workers exhibited cross-selectivity between aryl bromides and vinyl bromides (eq 2.7).<sup>69</sup> Interestingly, they utilized an earth abundant iron catalyst. The XEC reaction of vinyl halides have also been realized with nickel- and cobalt-catalysts.<sup>69,70,71,72,73,74,75</sup> In addition, functionalized ketones have been synthesized via XEC reactions of acyl electrophiles.<sup>76,77,78</sup>



<sup>65</sup> Gosmini, C.; Bassene-Ernst, C.; Durandetti, M. *Tetrahedron* **2009**, *65*, 6141–6146.

<sup>66</sup> Liao, L. Y.; Kong, X. R.; Duan, X. F. *J. Org. Chem.* **2014**, *79*, 777–782.

<sup>67</sup> Gong, C.; Huo, C.; Wang, X.; Quan, Z. *Chinese J. Chem.* **2017**, *35*, 1137–1366.

<sup>68</sup> Miao, W.; Ni, C.; Xiao, P.; Jia, R.; Zhang, W.; Hu, J. *Org. Lett.* **2021**, *23*, 711–715.

<sup>69</sup> Czaplik, W. M.; Mayer, M.; Jacobi von Wangelin, A. *ChemCatChem* **2011**, *3*, 135–138.

<sup>70</sup> Liu, J.; Ren, Q.; Zhang, X.; Gong, H. *Angew. Chem. Int. Ed.* **2016**, *55*, 15544–15548.

<sup>71</sup> Xiong, B.; Wang, T.; Sun, H.; Li, Y.; Kramer, S.; Cheng, G. J.; Lian, Z. *ACS Catal.* **2020**, *10*, 13616–13623.

<sup>72</sup> Amatore, M.; Gosmini, C.; Périchon, J. *Eur. J. Org. Chem.* **2005**, *2005*, 989–992.

<sup>73</sup> Moncomble, A.; Floch, P. Le; Lledos, A.; Gosmini, C. *J. Org. Chem.* **2012**, *77*, 5056–5062.

<sup>74</sup> Olivares, A. M.; Weix, D. J. *J. Am. Chem. Soc.* **2018**, *140*, 2446–2449.

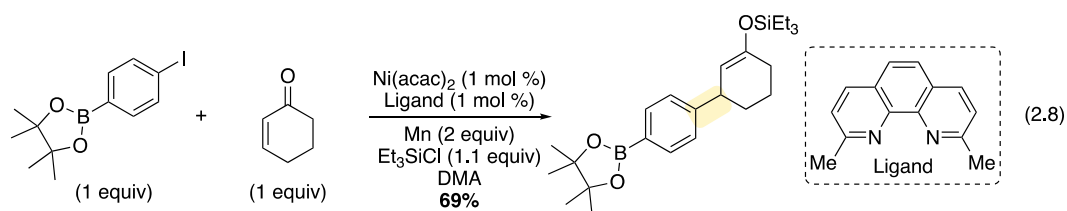
<sup>75</sup> Sha, Y.; Liu, J.; Wang, L.; Liang, D.; Wu, D.; Gong, H. *Org. Biomol. Chem.* **2021**, *19*, 4887–4890.

<sup>76</sup> Ni, S.; Zhang, W.; Mei, H.; Han, J.; Pan, Y. *Org. Lett.* **2017**, *19*, 2536–2539.

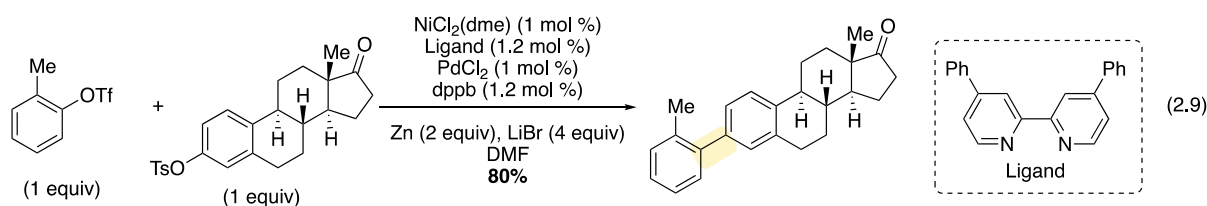
<sup>77</sup> Lin, T.; Mi, J.; Song, L.; Gan, J.; Luo, P.; Mao, J.; Walsh, P. *J. Org. Lett.* **2018**, *20*, 1191–1194.

<sup>78</sup> Zhu, Z.; Gong, Y.; Tong, W.; Xue, W.; Gong, H. *Org. Lett.* **2021**, *23*, 2158–2163.

Reductive coupling reactions have been expanded to include  $\alpha,\beta$ -unsaturated ketones<sup>79</sup> and  $\alpha$ -bromo enones.<sup>80</sup> An interesting example of a reductive conjugate addition involves an aryl iodide with  $\alpha,\beta$ -unsaturated enone (eq 2.8).<sup>79</sup> The authors demonstrated that aryl boronic acids and esters are well tolerated which can potentially lead to subsequent Suzuki-Miyaura coupling reactions.<sup>81</sup>



Finally, the Weix and Lian research groups independently demonstrated that a co-catalysis strategy could selectively engage two different sulfonates to achieve cross-selectivity (eq 2.9).<sup>63,64</sup> In order for this strategy to be successful, each catalyst must preferentially engage one electrophile. It was previously demonstrated that electron-rich palladium complexes could selectively engage aryl triflates over aryl halides.<sup>82</sup> The authors propose that the nickel complex catalyzes conversion of the aryl tosylate to the arylzinc reagent, which then undergoes palladium-catalyzed XC with the aryl triflate.<sup>60,83</sup>



### 2.3.2 XEC Reactions of C(sp) Electrophiles (Type D)

While the XEC reactions of C(sp<sup>2</sup>) and C(sp<sup>3</sup>) have been well established, the incorporation of C(sp) electrophiles has lagged. Traditionally, the Sonogashira coupling

<sup>79</sup> Shrestha, R.; Dorn, S. C. M.; Weix, D. J. *J. Am. Chem. Soc.* **2013**, *135*, 751–762.

<sup>80</sup> Beng, T. K.; Sincavage, K.; Silaire, A. W. V; Alwali, A.; Bassler, D. P.; Spence, L. E.; Beale, O. *Org. Biomol. Chem.* **2015**, *13*, 5349–5353.

<sup>81</sup> Miyaura, N.; Suzuki, A. *Chem. Rev.* **1995**, *95*, 2457–2483.

<sup>82</sup> Kamikawa, T.; Hayashi, T. *Tetrahedron Lett.* **1997**, *38*, 7087–7090.

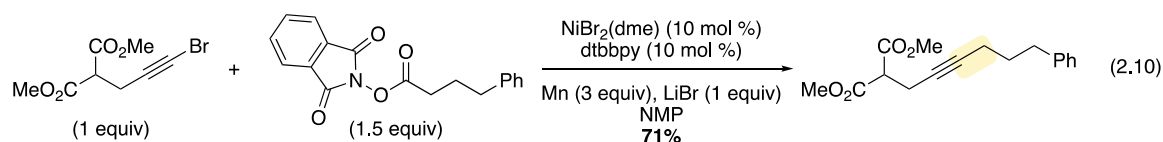
<sup>83</sup> Roy, A. H.; Hartwig, J. F. *Organometallics* **2004**, *23*, 194–202.

reaction is used for the formation of C(sp)–C(sp<sup>2</sup>) bonds and is typically a straightforward approach to synthesis of these compounds.<sup>84,85</sup> XEC reactions provide a mechanistically interesting alternative that allows for facile coupling of bromoalkynes with alkyl electrophiles. (Table 2.3)

**Table 2.3:** XEC Reactions of C(sp) and C(sp<sup>3</sup>) Electrophiles

Entry	Starting Material 1	Starting Material 2	Product	Metal Catalyst	Reducing Agent
1				Ni	Mn
2				Ni	Zn

Both reported examples employed alkynyl bromides and radical precursors to achieve the C(sp)–C(sp<sup>3</sup>) coupling reaction.<sup>86,87</sup> For example, in 2016, the Weix group reported the XEC reaction of bromoalkynes and NHP esters (eq 2.10).<sup>87</sup>



## 2.4 XEC Reactions Employing Electrochemical Reductions

Electrochemical reductions have been utilized in XEC reactions.<sup>88,89,90</sup> These methods avoid the need for stoichiometric reducing metals. The difficulties associated with employing metal powders such as Zn and Mn are the formation of heterogenous reaction mixtures and the necessity to use high boiling point amide solvents, such as DMA or DMF. Electrochemistry,

<sup>84</sup> Chinchilla, R.; Nájera, C. *Chem. Rev.* **2007**, *107*, 874–922.

<sup>85</sup> Chinchilla, R.; Nájera, C. *Chem. Soc. Rev.* **2011**, *40*, 5084.

<sup>86</sup> Ni, S.; Li, C. X.; Mao, Y.; Han, J.; Wang, Y.; Yan, H.; Pan, Y. *Sci. Adv.* **2019**, *5*, 9516–9544.

<sup>87</sup> Huang, L.; Olivares, A. M.; Weix, D. J. *Angew. Chem. Int. Ed.* **2017**, *56*, 11901–11905.

<sup>88</sup> Jutand, A. *Chem. Rev.* **2008**, *108*, 2300–2347.

<sup>89</sup> Nédélec, J.-Y.; Périchon, J.; Troupel, M. *Top. Curr. Chem.* **1997**, 141–173.

<sup>90</sup> Gale-Day, Z. J. *Synthesis* **2021**, *53*, 879–888.

on the other hand, is a simple, homogenous and inexpensive method that provides the electrons necessary for catalyst turnover. Additionally, electroreductive methods are ideal for industrial scale processes because large amounts of metallic waste are not produced. Furthermore, there is less of a need for metal scavengers to remove trace metals in reaction products. This can facilitate rapid and efficient syntheses of potential drug candidates. Despite these advantages, electroreductive coupling reactions are less common than the methods employing stoichiometric reductants. Nonetheless, it has been demonstrated that electrochemical conditions can be used for the majority of substrate classes typically employed in XEC reactions.

#### **2.4.1 XEC Reactions of C(sp<sup>2</sup>) Electrophiles (Type A)**

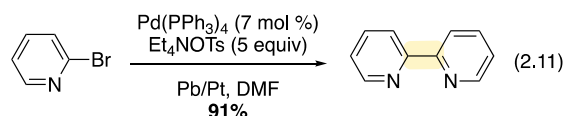
Some of the earliest examples of XEC reactions of aryl electrophiles employed electroreductive methods to forge the desired C–C bond. The methods have allowed for the synthesis of unsymmetrical biaryls including heterocyclic compounds. In addition, styrenes, dienes and unsymmetrical ketones have also been efficiently constructed with electroreductive methods. With judicious choice of cathode, anode, electrolyte, and solvent, successful XEC reactions are possible (Table 2.4).

**Table 2.4:** Electrochemical XEC Reactions of C(sp<sup>2</sup>) Electrophiles

Entry	Starting Material 1	Starting Material 2	Product	Metal Catalyst	Electrode <sup>a</sup>
1				Ni, Pd, Co	Ni/Cu; Pb/Pt; Pt/Pt; Au/Mg; Ni/Fe; Ni/Zn; Pt/Ni; Fe/Fe; Glassy Carbon/Pt; Carbon Cloth/Mg
2				Ni, Co, Fe	Fe/Fe; Ni/Zn; Ni/Al
3				Ni	Au/Zn
4				Ni, Pd	Au/Mg; Ni/Al
5				Ni	Ni/Ni

X = I, Br, Cl, OTf, OAc  
<sup>a</sup>cathode/anode

Dimerization reactions have been a useful method to forge biaryls.<sup>43, 91,92,93,94,95,96,97,98,99,100,101,102</sup> This concept has been applied to the synthesis of bipyridines (eq 2.11).<sup>95,103,104</sup>



Cross-selective examples of electroreductive XEC reactions lagged behind the dimerization of aryl electrophiles because of the difficulties associated with cross-selectivity

<sup>91</sup> Jennings, P. W.; Pillsbury, D. G.; Hall, J. L.; Brice, V. T. *J. Org. Chem.* **1976**, *41*, 719–722.

<sup>92</sup> Schiavon, G.; Bontempelli, G.; Corain, B. *J. Chem. Soc., Dalt. Trans.* **1981**, *202*, 1074–1081.

<sup>93</sup> Mori, M.; Hashimoto, Y.; Ban, Y. *Tetrahedron Lett.* **1980**, *21*, 631–634.

<sup>94</sup> Troupel, M.; Rollin, Y.; Sibille, S.; Perichon, J.; Fauvarque, J.-F. *J. Organomet. Chem.* **1980**, *202*, 435–446.

<sup>95</sup> Torii, S.; Tanaka, H.; Morisaki, K. *Tetrahedron Lett.* **1985**, *26*, 1655–1658.

<sup>96</sup> Rollin, Y.; Troupel, M.; Tuck, D. G.; Perichon, J. *J. Organomet. Chem.* **1986**, *303*, 131–137.

<sup>97</sup> Meyer, G.; Rollin, Y.; Perichon, J. *J. Organomet. Chem.* **1987**, *333*, 263–267.

<sup>98</sup> Fox, M. A.; Chandler, D. A.; Lee, C. *J. Org. Chem.* **1991**, *56*, 3246–3255.

<sup>99</sup> Jutand, A.; Négri, S.; Mosleh, A. *J. Chem. Soc., Chem. Commun.* **1992**, 1729–1730.

<sup>100</sup> Courtois, V.; Barhdadi, R.; Troupel, M.; Perichon, J. *Tetrahedron* **1997**, *53*, 11569–11576.

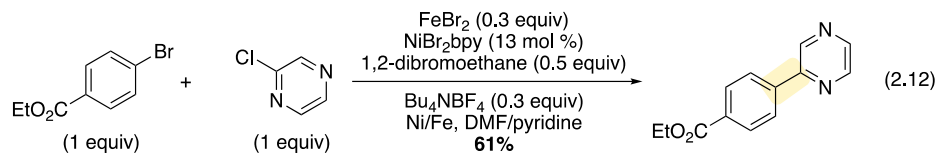
<sup>101</sup> Yasuhara, A.; Kasano, A.; Sakamoto, T. *J. Synth. Org. Chem. Japan* **2000**, *58*, 192–198.

<sup>102</sup> Courtois, V.; Barhdadi, R.; Condon, S.; Troupel, M. *Tetrahedron Lett.* **1999**, *40*, 5993–5996.

<sup>103</sup> Cassol, T. M.; Demnitz, F. W. J.; Navarro, M.; Neves, E. A. *Tetrahedron Lett.* **2000**, *41*, 8203–8206.

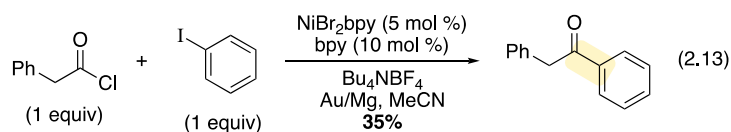
<sup>104</sup> de França, K. W. R.; Navarro, M.; Léonel, É.; Durandetti, M.; Nédélec, J.-Y. *J. Org. Chem.* **2002**, *67*, 1838–1842.

(vide supra).<sup>97,98,105,106,107</sup> The Gosmini group reported a cross-selective reaction between aryl bromides and 2-chloropyrimidine (eq 2.12).<sup>108</sup> Key to their success was the use of substoichiometric FeBr<sub>2</sub> which prevented strong ligation of pyrimidine to the nickel catalyst.



Similar strategies have been employed to achieve the cross-selective XEC reaction of other aryl electrophiles and heterocyclic substrates.<sup>108,109,110,111,112,113,114</sup> Additionally, vinyl electrophiles are well suited to undergo reductive dimerization reactions<sup>96,115</sup> and cross selective reactions.<sup>12,115,116</sup>

Finally, XEC reactions have been achieved with acid chlorides.<sup>117,118</sup> For example, Marzouk et. al reported the cross-selective XEC reaction between aryl iodides and acid chlorides (eq 2.13).<sup>117</sup>



## 2.4.2 XEC Reactions of C(sp<sup>2</sup>) and C(sp<sup>3</sup>) Electrophiles (Type B)

Similar to XEC reactions with reducing agents, the most common combination reported for XEC is C(sp<sup>2</sup>)–C(sp<sup>3</sup>) coupling reactions (Table 2.5). This is because the partners often

<sup>105</sup> Amatore, C.; Carré, E.; Jutand, A.; Tanaka, H.; Ren, Q.; Torii, S. *Chem. Eur. J.* **1996**, *2*, 957–966.

<sup>106</sup> Meyer, G.; Troupel, M.; Périchon, J. *J. Organomet. Chem.* **1990**, *393*, 137–142.

<sup>107</sup> Gomes, P.; Fillon, H.; Gosmini, C.; Labbé, E.; Périchon, J. *Tetrahedron* **2002**, *58*, 8417–8424.

<sup>108</sup> Gosmini, C.; Nédélec, J. Y.; Périchon, J. *Tetrahedron Lett.* **2000**, *41*, 201–203.

<sup>109</sup> Gosmini, C.; Lasry, S.; Nédélec, J. Y.; Périchon, J. *Tetrahedron* **1998**, *54*, 1289–1298.

<sup>110</sup> Gosmini, C.; Nédélec, J. Y.; Périchon, J. *Tetrahedron Lett.* **2000**, *41*, 5039–5042.

<sup>111</sup> Sengmany, S.; Léonel, E.; Polissaint, F.; Nédélec, J.-Y.; Pipelier, M.; Thobie-Gautier, C.; Dubreuil, D. *J. Org. Chem.* **2007**, *72*, 5631–5636.

<sup>112</sup> Sengmany, S.; Le Gall, E.; Léonel, E. *Molecules* **2011**, *16*, 5550–5560.

<sup>113</sup> Sengmany, S.; Vitu-Thiebaud, A.; Le Gall, E.; Condon, S.; Léonel, E.; Thobie-Gautier, C.; Pipelier, M.; Lebreton, J.; Dubreuil, D. *J. Org. Chem.* **2013**, *78*, 370–379.

<sup>114</sup> Sengmany, S.; Vasseur, S.; Lajnef, A.; Le Gall, E.; Léonel, E. *Eur. J. Org. Chem.* **2016**, *2016*, 4865–4871.

<sup>115</sup> Cannes, C.; Condon, S.; Durandetti, M.; Périchon, J.; Nédélec, J.-Y. *J. Org. Chem.* **2000**, *65*, 4575–4583.

<sup>116</sup> Gomes, P.; Gosmini, C.; Périchon, J. *Tetrahedron* **2003**, *59*, 2999–3002.

<sup>117</sup> Marzouk, H.; Rollin, Y.; Folest, J. C.; Nédélec, J. Y.; Périchon, J. *J. Organomet. Chem.* **1989**, *369*, C47–C50.

<sup>118</sup> Folest, J. C.; Pereira-Martins, E.; Troupel, M.; Périchon, J. *Tetrahedron Lett.* **1993**, *34*, 7571–7574.



engage the catalyst in mechanistically distinct pathways (vide supra). The partners are differentiated therefore promoting cross-selectivity.

**Table 2.5:** Electrochemical XEC Reactions of C(sp<sup>2</sup>) and C(sp<sup>3</sup>) Electrophiles

Entry	Starting Material 1	Starting Material 2	Product	Metal Catalyst	Electrode <sup>a</sup>
1		X-R <sup>2</sup>		Ni	RVC/Zn; Ni/Zn; RVC/HN( <i>i</i> -Pr) <sub>2</sub> Graphite/Ni
2				Ni	Ni/Zn; Ni/Al Graphite/Ni
3		X-CH <sub>2</sub> -CH=CH <sub>2</sub>		Ni	Ni/Zn; Ni/Al
4				Ni	Au/Hg; Ni/Zn; Ni/Al Carbon Fiber/Al Carbon Fiber/Zn
5				Ni	RVC/RVC; RVC/Zn
6				Ni	RVC/Zn; Ni/Zn
7				Ni	Ni/Al
8		X-R <sup>2</sup>		Ni	Au/Zn; Au/Mg
9		X-CH <sub>2</sub> -C <sub>6</sub> H <sub>4</sub> -R <sup>2</sup>		Ni	Au/Zn; Au/Mg

X = I, Br, Cl, OH, OAc  
<sup>a</sup>cathode/anode

A common functional group utilized in the 1990s and early 2000s was the  $\alpha$ -chlorocarbonyl.<sup>12,115,119,120,121,122</sup> This electrophile cleanly undergoes an XEC reaction with a variety of electrophiles including aryl, heterocyclic and vinylic halides. For example,

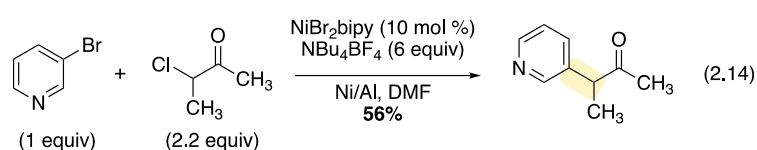
<sup>119</sup> Folest, J. C.; Périchon, J.; Fauvarque, J. F.; Jutand, A. *J. Organomet. Chem.* **1988**, 342, 259–261.

<sup>120</sup> Conan, A.; Sibille, S.; D'Incan, E.; Périchon, J. *J. Chem. Soc., Chem. Commun.* **1990**, 48–49.

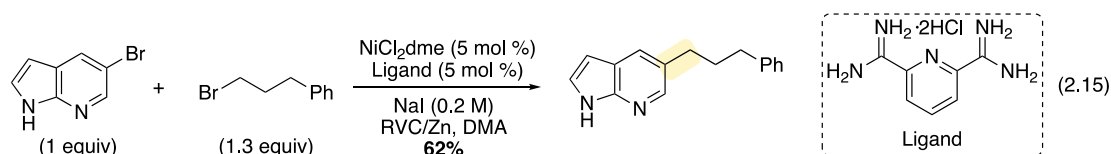
<sup>121</sup> Durandetti, M.; Sibille, S.; Nédélec, J.-Y.; Périchon, J. *Synth. Commun.* **1994**, 24, 145–151.

<sup>122</sup> Durandetti, M.; Périchon, J.; Nédélec, J.-Y. *J. Org. Chem.* **1997**, 62, 7914–7915.

Durandetti and co-workers reported the coupling of aryl bromides with  $\alpha$ -chloroketones (eq 2.14).<sup>121</sup>



Additionally, electroreductive methods have been applied to coupling of aryl and alkyl electrophiles.<sup>12,123,124,125,126,127,128,129</sup> Recently, a group at Pfizer developed an electrochemical XEC reaction with a range of heterocyclic bromides, including an aza-indole (eq 2.15).<sup>123</sup> The authors noted that XEC reactions are ideal targets for drug design and electrochemistry can improve their scale up processes significantly compared to stoichiometric metals.



Building on previous enantioselective XEC reactions of vinyl bromides from their laboratory,<sup>130</sup> the Reisman group demonstrated that an enantioselective reaction under electroreductive conditions was feasible (eq 2.16).<sup>131</sup> The high enantioselectivity achieved with a bisoxazoline ligand is consistent with the proposed mechanisms involving halogen atom abstraction, since the enantiodetermining step should remain the same whether catalyst turnover is electrochemical or mediated by a reducing metal.

<sup>123</sup> Perkins, R. J.; Pedro, D. J.; Hansen, E. C. *Org. Lett.* **2017**, *19*, 3755–3758.

<sup>124</sup> Perkins, R. J.; Hughes, A. J.; Weix, D. J.; Hansen, E. C. *Org. Process Res. Dev.* **2019**, *23*, 1746–1751.

<sup>125</sup> Truesdell, B. L.; Hamby, T. B.; Sevov, C. S. *J. Am. Chem. Soc.* **2020**, *142*, 5884–5893.

<sup>126</sup> Li, Z.; Sun, W.; Wang, X.; Li, L.; Zhang, Y.; Li, C. *J. Am. Chem. Soc.* **2021**, *143*, 3536–3543.

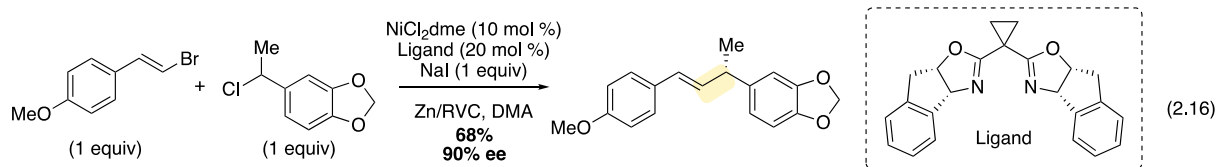
<sup>127</sup> Li, H.; Breen, C. P.; Seo, H.; Jamison, T. F.; Fang, Y. Q.; Bio, M. M. *Org. Lett.* **2018**, *20*, 1338–1341.

<sup>128</sup> Koyanagi, T.; Herath, A.; Chong, A.; Ratnikov, M.; Valiere, A.; Chang, J.; Molteni, V.; Loren, J. *Org. Lett.* **2019**, *21*, 816–820.

<sup>129</sup> Gomes, P.; Gosmini, C.; Périchon, J. *J. Org. Chem.* **2003**, *68*, 1142–1145.

<sup>130</sup> Cherney, A. H.; Reisman, S. E. *J. Am. Chem. Soc.* **2014**, *136*, 14365–14368.

<sup>131</sup> Delano, T. J.; Reisman, S. E. *ACS Catal.* **2019**, *9*, 6751–6754.



### 2.4.3 XEC Reactions of C(sp<sup>3</sup>) Electrophiles (Type C)

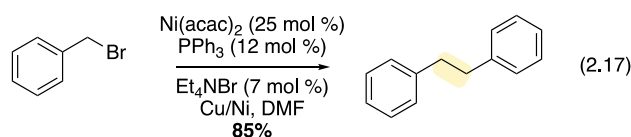
Although less common than the cross-selective XEC reactions of C(sp<sup>2</sup>) and C(sp<sup>3</sup>) electrophiles, reductive dimerization reactions of benzylic and aliphatic electrophiles have been established (Table 2.6).<sup>91,92,96,132</sup>

**Table 2.6:** Electrochemical Dimerization Reactions of C(sp<sup>3</sup>) Electrophiles

Entry	Starting Material	Product	Metal Catalyst	Electrode <sup>a</sup>
1			Ni	Cu/Ni; Pt/Pt; Au/Mg
2			Ni	Au/Mg
3			Ni, Fe	Ni/Ni; Al/Al; Pt/Pt Au/Mg; Hg/Pt
4			Fe	Ni/Ni

X = I, Br, Cl, OTf  
<sup>a</sup>cathode/anode

To illustrate, the Jennings group reported the dimerization reaction of benzyl bromide in good yield (eq 2.17).<sup>91</sup> In this report they were also able to achieve a good yield for a simple aliphatic electrophile, 1-octylbromide. Another report has demonstrated that secondary alkyl halides can efficiently undergo an iron-catalyzed dimerization reaction utilizing a Ni/Ni undivided cell.<sup>132</sup>



<sup>132</sup> Mabrouk, S.; Pellegrini, S.; Folest, J.-C.; Rollin, Y.; Perichon, J. *J. Organomet. Chem.* **1986**, *301*, 391–400.

## 2.5 Conclusions

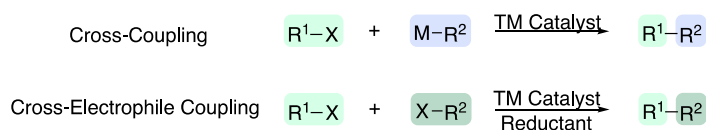
Transition metal-catalyzed XEC reactions have gained popularity over the past two decades as an efficient method to construct carbon-carbon bonds. Many of the methods discussed employ sustainable first-row transition metal catalysts, earth abundant metal reductants or electrochemistry and mild reaction conditions. We anticipate that as mechanistic insights grow and more general methods emerge, XEC reactions will become an essential tool in the organic chemist synthetic toolbox.

## Intramolecular Nickel-Catalyzed Cross-Electrophile Coupling Reactions of Sulfonamides

## 3.1 Introduction

Cross-electrophile coupling (XEC) reactions have emerged as a powerful tool in synthetic organic chemistry.<sup>1,2</sup> In contrast to traditional cross-coupling (XC) reactions where an electrophile (R–X) and an organometallic reagent (R–M) are coupled together in the presence of a transition metal (TM) catalyst, XEC reactions utilize two electrophilic reagents, a TM catalyst, and a terminal reductant to forge the desired carbon-carbon bond. (Scheme 3.1).

## Scheme 3.1 Cross-Coupling v. Cross-Electrophile Coupling Reactions



There are several advantages of XEC reactions. The first is that alkyl and aryl halides typically have greater commercial availability than the commonly used organometallic reagents. Additionally, the organometallic reagents are often prepared from the corresponding alkyl or aryl halides, whereas, XEC reactions would allow for the direct coupling of these electrophiles. Finally, due to the reactivity of organometallic reagents, they frequently require the extrusion of oxygen and moisture and are typically less functional group tolerant than the corresponding electrophiles which can limit the scope of the reaction.

<sup>1</sup> Portions of this chapter have been published in the Journal of Organic Chemistry, see: Lucas, E. L.; Hewitt, K. A.; Chen, P.-P.; Castro, A. J.; Hong, X.; Jarvo, E. R. *J. Org. Chem.* **2020**, *85*, 1775–1793.

<sup>2</sup> For selected reviews on XEC reactions, see: (a) Hewitt, K. A.; Lin, P. C.; Raffman, E. T. A.; Jarvo, E. R. C–C Bond Formation via Cross-Electrophile Coupling Reactions. *Comprehensive Organometallic Chemistry IV*. **2021**. 10.1016/B978-0-12-820206-7.00092-5. (b) Everson, D. A.; Weix, D. *J. Org. Chem.* **2014**, *79*, 4793–4798. (c) Knappke, C. E. I.; Grupe, S.; Gärtner, D.; Corpet, M.; Gosmini, C.; Jacobi von Wangelin, A. *Chem. Eur. J.* **2014**, *20*, 6828–6842. (d) Gu, J.; Wang, X.; Xue, W.; Gong, H. *Org. Chem. Front.* **2015**, *2*, 1411–1421. (e) Wang, X.; Dai, Y.; Gong, H. *Top. Curr. Chem.* **2016**, *374*, 43. (f) Lucas, E. L.; Jarvo, E. R. *Nat. Rev. Chem.* **2017**, *1*, 0065. (g) Campeau, L.-C.; Hazari, N. *Organometallics* **2019**, *38*, 3–35. (h) Goldfogel, M. J.; Huang, L.; Weix, D. J. Cross-Electrophile Coupling: Principles and New Reactions. In *Nickel Catalysis in Organic Synthesis*; Ogoshi, S. Ed.; Wiley, 2020; pp 183–222.

The major challenge associated with this strategy is the homocoupling of electrophiles. The first reported example of the transition-metal mediated reductive homocoupling of electrophiles was the Ullman dimerization of aryl halides in 1901.<sup>3,4</sup> The selective XEC between two different electrophiles was not realized until later in the 20<sup>th</sup> century.<sup>5</sup> Unlike traditional XC reactions where the electrophile favors oxidative addition and the nucleophilic reagent favors transmetalation, in XEC reactions both reagents favor oxidative addition and the catalyst must be able to distinguish them in order to obtain cross-selectivity. This inherent challenge resulted in delayed efforts to explore and optimize these reactions.

Several strategies have been developed for optimal cross-selectivity (Scheme 3.2).<sup>2a,2b,2f</sup> One strategy includes employing an excess of one of the electrophiles. This allows for the isolation of statistical mixtures of homocoupled products and the desired cross-coupled product. As an example, the Gong group demonstrated that secondary and primary bromides could be coupled together by utilizing three equivalents of the more reactive primary bromide (Scheme 3.2a).<sup>6</sup> Another strategy is to electronically differentiate the starting materials. As an example, the Reisman group reported the XEC reaction between benzylic chlorides with acid chlorides and observed exclusive formation of the desired cross-coupled product (Scheme 3.2b).<sup>7</sup> Co-catalytic strategies have also been successful for the cross-selective XEC reactions. The Weix group reported a nickel and titanium co-catalytic strategy to forge the desired benzylic alcohols in high yield, diastereoselectivity and enantioselectivity (Scheme 3.2c).<sup>8</sup> Finally, the electrophiles can be

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<sup>3</sup> Ullmann, F.; Bielecki, J. *Chem. Ber.* **1901**, *34*, 2174–2185.

<sup>4</sup> For reviews on the Ullman coupling, see: (a) Fanta, P. E. *Chem. Rev.* **1946**, *38*, 139–196. (b) Sainsbury, M. *Tetrahedron* **1980**, *36*, 3327–3359. (c) Bringmann, G.; Walter, R.; Weirich, R. *Angew. Chem. Int. Ed.* **1990**, *29*, 977–991. (d) Hassan, J.; Sévignon, M.; Gozzi, C.; Schulz, E.; Lemaire, M. *Chem. Rev.* **2002**, *102*, 1359–1470. (e) Fanta, P. E. *Synthesis* **1974**, *1974*, 9–21.

<sup>5</sup> For progress on cross-selective XEC reactions between two electrophiles, see ref 2.

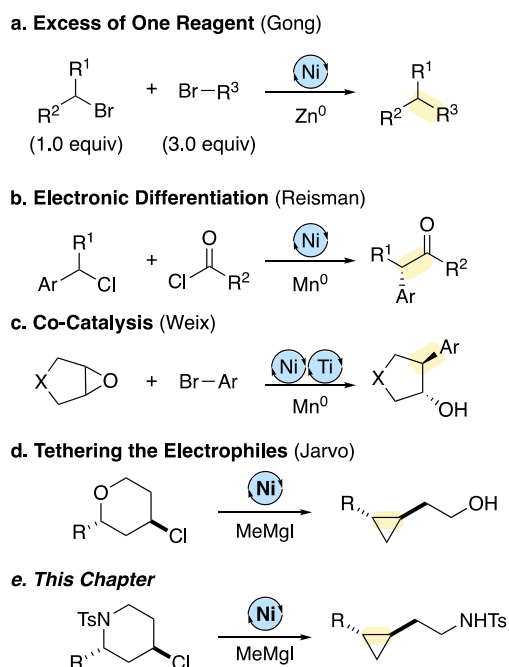
<sup>6</sup> Yu, X.; Yang, T.; Wang, S.; Xu, H.; Gong, H. *Org. Lett.* **2011**, *13*, 2138–2141.

<sup>7</sup> Cherney, A. H.; Kadunce, N. T.; Reisman, S. E. *J. Am. Chem. Soc.* **2013**, *135*, 7442–7445.

<sup>8</sup> Zhao, Y.; Weix, D. J. *J. Am. Chem. Soc.* **2015**, *137*, 3237–3240.

tethered together and the cyclized product can be isolated in high yield. For example, the Jarvo group reported the ring contraction of tetrahydrofurans to afford disubstituted cyclopropanes in high yield and diastereoselectivity (Scheme 3.2d).<sup>9</sup> We aimed to exploit this strategy of tethering the electrophiles together to develop an analogous XEC reaction of benzylic and allylic sulfonamides (Scheme 3.2e).<sup>1</sup>

**Scheme 3.2** Representative Examples of Strategies for Cross-Selective XEC Reactions



In addition, the goal of this method was to activate the carbon-nitrogen bond in the sulfonamide. Amine derivatives are elusive electrophiles in XEC reactions and have been occasionally activated via transition-metal catalysis.<sup>10</sup> This functional group is also prevalent in natural products, biomolecules, and pharmaceutical agents which makes it an ideal candidate for

<sup>9</sup> (a) Tollefson, E. J.; Erickson, L. W.; Jarvo, E. R. *J. Am. Chem. Soc.* **2015**, *137*, 9760–9763. (b) Erickson, L. W.; Lucas, E. L.; Tollefson, E. J.; Jarvo, E. R. *J. Am. Chem. Soc.* **2016**, *138*, 14006–14011.

<sup>10</sup> For recent reviews on cross-coupling reaction of C–N bonds, see: (a) Ouyang, K.; Hao, W.; Zhang, W.-X.; Xi, Z. *Chem. Rev.* **2015**, *115*, 12045–12090. (b) Wang, Q.; Su, Y.; Li, L.; Huang, H. *Chem. Soc. Rev.* **2016**, *45*, 1257–1272. (c) Pound, S. M.; Watson, M. P. *Chem. Commun.* **2018**, *54*, 12286–12301. (d) Huang, C.-Y.; Doyle, A. G. *Chem. Rev.* **2014**, *114*, 8153–8198.

further manipulation.<sup>11</sup> Finally, the carbon-nitrogen bond exhibits orthogonal reactivity compared with traditional electrophiles, such as halides and pseudohalides, which allows for selective functionalization.

Similar to the traditional XC reaction of amine derivatives, the current examples of XEC reactions require the activation of the carbon-nitrogen bond by the formation of ammonium salts or incorporation into strained rings.<sup>10c,10d,12</sup> For example, the Shu laboratory reported the XEC reaction of aryl ammonium triflates with vinylic acetates to afford the desired carbon-carbon bond (Scheme 3.3a).<sup>13</sup> Similarly, the Martin group reported the XEC reaction between benzylic trimethylammonium salts and CO<sub>2</sub> to afford carboxylic acids (Scheme 3.3b).<sup>14</sup> More recently, the Watson group developed the XEC reaction of benzylic Katritzky salts with aryl bromides to forge the desired carbon-carbon bond (Scheme 3.3c).<sup>15</sup> Finally, the Doyle and Sigman laboratories disclosed the XEC reaction of styrenyl aziridines with aryl iodides to yield enantioenriched 2-arylphenethylsulfonamides (Scheme 3.3d).<sup>16</sup> These examples demonstrate the feasibility of the development of XEC reactions of alkyl amine derivatives but also exemplify the current limitations.

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<sup>11</sup> (a) Nicolaou, K. C.; Montagnon, T. *Molecules that Changed the World*; Wiley-VCH: Germany, 2008. (b) Transition-Metal Catalyzed C–N Bond Activation; Fattorusso, E.; Tagliatela-Scafati, O., Eds. *Modern Alkaloids: Structure, Isolation, Synthesis and Biology*; Wiley-VCH, 2008. (c) Lawrence, S. A. *Amines: Synthesis, Properties, and Applications*. Cambridge University Press: New York, 2004.

<sup>12</sup> For examples of traditional XC reactions of amine derivatives, see (a) ref 10. (b) Chapter 1 of this thesis.

<sup>13</sup> He, R.-D.; Li, C.-L.; Pan, Q.-Q.; Guo, O.; Liu, X.-Y.; Shu, X.-Z. *J. Am. Chem. Soc.* **2019**, *141*, 12481–12486.

<sup>14</sup> Moragas, T.; Gaydou, M.; Martin, R. *Angew. Chem. Int. Ed.* **2016**, *55*, 5053–5057.

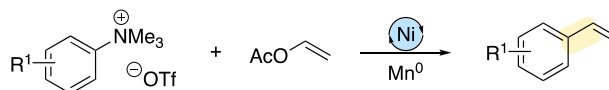
<sup>15</sup> Liao, J.; Basch, C. H.; Hoerner, M. E.; Talley, M. R.; Boscoe, B. P.; Tucker, J. W.; Garnsey, M. R.; Watson, M. P. *Org. Lett.* **2019**, *21*, 2941–2946.

<sup>16</sup> Woods, B. P.; Orlandi, M.; Huang, C.-Y.; Sigman, M. S.; Doyle, A. G. *J. Am. Chem. Soc.* **2017**, *139*, 5688–5691.

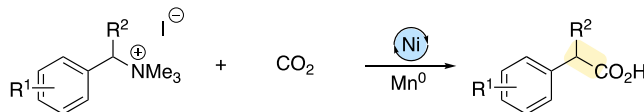


### Scheme 3.3 Representative Example of XEC Reactions of Amine Derivatives

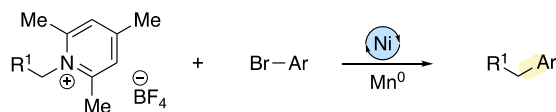
#### a. Aryl Ammonium Salts (Shu)



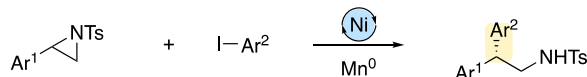
#### b. Benzylic Ammonium salts (Martin)



#### c. Katritzky salts (Watson)



#### d. Strained sulfonamides (Doyle and Sigman)



In this Chapter, we report a nickel-catalyzed XEC reaction of benzylic and allylic sulfonamides (Scheme 3.2e). Optimization and implementation of the aza-Prins reaction is presented for the synthesis of the requisite starting materials. In addition, we demonstrate that *N*-tosyl-4-chloropiperidines undergo a ring contraction reaction to afford *trans*-substituted cyclopropanes in high yield and excellent diastereoselectivity. This work is a significant development because activation of the carbon-nitrogen bond is achieved without the utilization of a charged ammonium salt or a strained ring. Moreover, it demonstrates the ability to manipulate the structure of sulfonamides. New methods for the manipulation of these functional groups would be valuable because sulfonamides comprise nearly 30% of the newly FDA approved drugs bearing sulfur-containing functional groups.<sup>17</sup> This work sets the foundation for further development of nickel-catalyzed XEC reactions of amine derivatives and provides a structural rearrangement of sulfonamides by transforming *N*-tosylpiperidines to *N*-tosyl(aminoethyl)cyclopropanes.

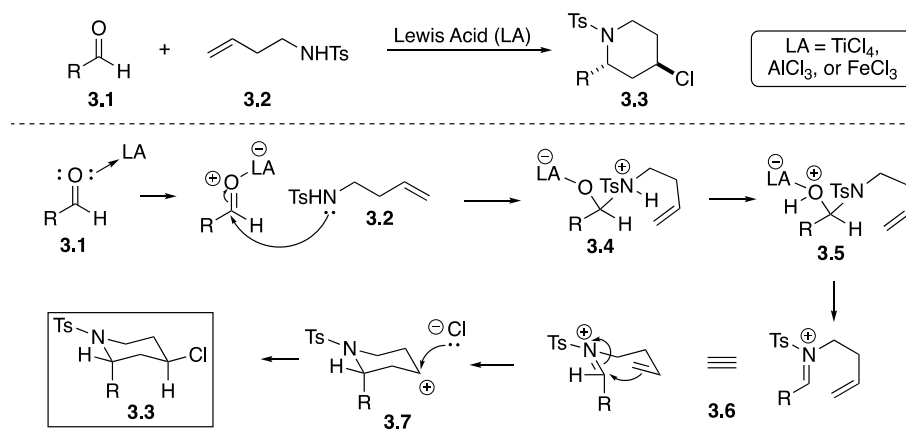
## 3.2 Results and Discussion

<sup>17</sup> Ilardi, E. A.; Vitaku, E.; Njardarson, J. T. *J. Med. Chem.* **2014**, *57*, 2832–2842.

### 3.2.1 Optimization of the Aza-Prins Cyclization

*N*-Tosyl-4-chloropiperidines were selected as the starting materials for the XEC reaction because they can be synthesized in one step via highly diastereoselective aza-Prins cyclizations from commercially available aldehydes.<sup>18</sup> The mechanism of the aza-Prins reaction is outlined in Figure 3.1. Coordination of the Lewis acid to the aldehyde (**3.1**) increases the electrophilicity of the carbonyl moiety and allows for the nucleophilic addition of the sulfonamide (**3.2**). A condensation reaction cleanly affords the imine with the tethered olefin (**3.6**). Olefin attack of the imine leads to the formation of the cyclized intermediate **3.7**. The resulting carbocation can be quenched with a nucleophile ( $\text{Cl}^-$ ) in solution in a diastereoselective fashion to afford the desired piperidine **3.3**.

**Figure 3.1** Mechanism of the Prins Cyclization



The aza-Prins cyclization selectively forms the *trans* diastereomer of the piperidine. The allylic strain imparted by the sulfonyl group drives the aryl or vinyl group to the axial position.<sup>19</sup> Addition of the chloride is highly stereoselective to avoid 1,3-diaxial interactions (see Figure 3.1,

<sup>18</sup> For reviews on the Prins cyclization see: (a) Arundale, E.; Mikeska, L. A. *Chem. Rev.* **1952**, *51*, 505–555. (b) Adams, D. R.; Bhatnagar, S. P. *Synthesis*, **1977**, 661–672. (c) Olier, C.; Kaafarani, M.; Gastaldi, S.; Bertrand, M. P. *Tetrahedron*, **2010**, *66*, 413–445. (d) Pastor, I. M.; Yus, M. *Curr. Org. Chem.* **2012**, *16*, 1277–1312. (e) Reddy, B. V. S.; Nair, P. N.; Antony, A.; Lalli, C.; Grée, R. *Eur. J. Org. Chem.* **2017**, 1805–1819.

<sup>19</sup> (a) Zhang, H.; Muñoz, K. *ACS Catal.* **2017**, *7*, 4122–4125. (b) Seel, S.; Thaler, T.; Takatsu, K.; Zhang, C.; Zipse, H.; Straub, B. F.; Mayer, P.; Knochel, P. *J. Am. Chem. Soc.* **2011**, *133*, 4774–4777.

intermediate **3.7**). Given the high diastereoselectivity and range of commercially available aldehydes, it is clear that the aza-Prins reaction would be an ideal synthesis for *N*-tosyl-4-chloropiperidines. In this section, we describe the modification and optimization of the aza-Prins cyclization for the synthesis of a range of *N*-tosyl-4-chloropiperidines.

The initial condensation reaction to form imine intermediate **3.6** often requires the coordination of a Lewis acid (LA) to the aldehyde. This increases the electrophilicity of the aldehyde and allows for facile formation of the imine. Some of the common Lewis acids utilized in these reactions include FeCl<sub>3</sub>, BiCl<sub>3</sub>, BF<sub>3</sub>·Et<sub>2</sub>O, InCl<sub>3</sub>, AlCl<sub>3</sub>, and TiCl<sub>4</sub>.<sup>18</sup> We found that the choice of Lewis acid for the reaction conditions needs to match the reactivity of aldehyde starting material. For example, when simple aromatic substrates or an extended aromatic heterocycles were employed, titanium-promoted aza-Prins reactions were utilized.<sup>20</sup> However, for aromatic rings that are sensitive to strong Lewis acids, a milder AlCl<sub>3</sub>-catalyzed aza-Prins cyclization can be employed.<sup>21</sup> Finally, for substituted cinnamaldehydes, FeCl<sub>3</sub>-mediated aza-Prins reactions were found to afford the desired products in synthetically useful yields.<sup>22</sup>

Our investigation into the aza-Prins cyclization began with 2-naphthaldehyde. Utilizing previously reported conditions by the van de Weghe laboratory, piperidine **3.8** was synthesized in 41% yield via TiCl<sub>4</sub> and *p*-TsOH·H<sub>2</sub>O promoted aza-Prins cyclization (Table 3.1, entry 1).<sup>20,23</sup> With this result in hand, we aimed to synthesize substrates bearing heterocycles. However, when the same reaction conditions were applied to benzofuran-2-carbaldehyde for 48 h there was no product formation observed (entry 2). Increasing the reaction time to seven days provided a modest

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<sup>20</sup> Durel, V.; Lalli, C.; Roisnel, T.; van de Weghe, P. *J. Org. Chem.* **2016**, *81*, 849–859.

<sup>21</sup> Liu, G. Q.; Cui, B.; Xu, R.; Li, Y. M. *J. Org. Chem.* **2016**, *81*, 5144–5161.

<sup>22</sup> Osawa, C.; Tateyama, M.; Miura, K.; Tayama, E.; Iwamoto, H.; Hasegawa, E. *Heterocycles* **2012**, *86*, 1211–1226.

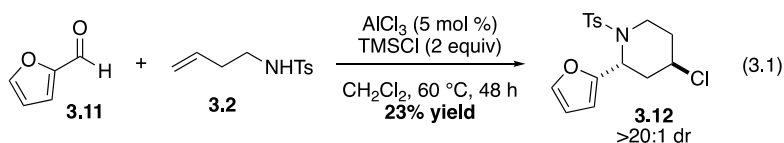
<sup>23</sup> This substrate was synthesized by Dr. Erika Lucas. For more information, see: Lucas, E. L. Development of Stereospecific Nickel-Catalyzed Cross-Electrophile Coupling Reactions for Cyclopropane Synthesis. Ph.D. Dissertation, University of California, Irvine, Irvine, CA, **2019**.

yield of the desired product, which was encouraging for further optimization (Table 3.1, entry 3). The conditions developed by van de Weghe contained one source of chlorine anion ( $\text{TiCl}_4$ ) in solution and this anion is necessary for product formation (See Figure 3.1, intermediate **3.7** to product **3.3**). We hypothesized that if the Brønsted acid was modified to a chlorine containing acid the amount of chlorine ions in solution would increase and allow for more facile preparation of the desired product. To test this hypothesis, we employed HCl as the Brønsted acid rather than *p*-TsOH·H<sub>2</sub>O and piperidine **3.9** was isolated in a 48% yield (entry 4). Applying the modified conditions to another heterocyclic aldehyde benzo[*b*]thiophene-2-carbaldehyde, piperidine **3.10** was isolated in a 26% yield (entry 5). Although the aza-Prins cyclization proceeds in moderate yields, it allows for the diastereoselective formation of the desired product in one step from commercially available aldehydes.

**Table 3.1** Optimization of Titanium-Promoted Aza-Prins Reaction

Entry	R	Compound	Brønsted Acid	Time	Yield (%)
1		<b>3.8</b>	<i>p</i> -TsOH·H <sub>2</sub> O	48 h	41
2		<b>3.9</b>	<i>p</i> -TsOH·H <sub>2</sub> O	48 h	0
3		<b>3.9</b>	<i>p</i> -TsOH·H <sub>2</sub> O	7 d	16
4		<b>3.9</b>	HCl	48 h	48
5		<b>3.10</b>	HCl	48 h	26

Encouraged by the success of benzofuran and benzothiophene, we aimed to expand the scope of the aza-Prins cyclization to include non-extended aromatic heterocycles, such as furan. 2-furaldehyde (**3.11**) was subjected to a TiCl<sub>4</sub>-promoted aza-Prins reaction; unfortunately, with either *p*-TSA·H<sub>2</sub>O or HCl, the reaction failed to provide the desired product and no starting material was recovered. Substituted furan rings are known to be sensitive to hydrolysis under strongly acidic conditions,<sup>24</sup> so we investigated milder Lewis acids. Inspired by work published by the Li laboratory,<sup>21</sup> we employed catalytic AlCl<sub>3</sub> and one equivalent of trimethylsilyl chloride (TMSCl) and only starting material was recovered after 24 h. We were encouraged, however, that the furaldehyde was not decomposing or hydrolyzing under these reactions conditions. Further optimization, such as increasing catalyst loading, the equivalents of TMSCl, or heating the reaction to reflux, failed to afford any desired product. Finally, I found that 5 mol% of AlCl<sub>3</sub>, two equiv of TMSCl, and heating the reaction to 60 °C in a sealed pressure tube for 48 hours afforded piperidine **3.12** in a 23% yield and as single diastereomer (eq 3.1).

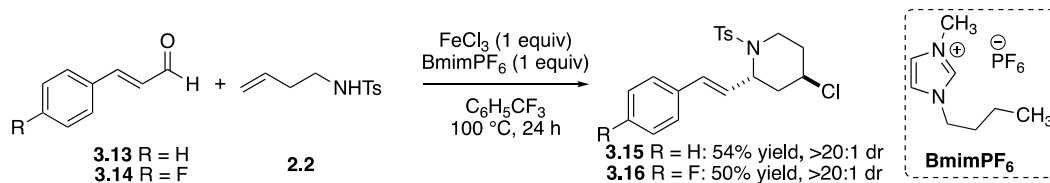


The next targeted substrates were allylic sulfonamides. The aza-Prins cyclizations presented thus far were not compatible with the cinnamaldehyde starting material and only lead to decomposition by-products. Recently, Iwamoto and co-workers disclosed a FeCl<sub>3</sub>- and BmimPF<sub>6</sub>-mediated aza-Prins cyclization which included substituted cinnamaldehydes in their scope.<sup>22</sup> Therefore, aldehyde **3.13** was subjected to these reported conditions and piperidine **3.15** was isolated in an 11% yield, with 80% of the recovered aldehyde. While this result indicated that this

<sup>24</sup> For selected examples, see (a) Joule, J. A.; Keith, M. *Heterocyclic Chemistry*; Blackwell Publishing Ltd.: Chichester, U. K., 2010. (b) Liang, X.; Haynes, B. S.; Montoya, A. *Energy Fuels*, **2018**, *32*, 4139–4148. (c) Nikbin, N.; Caratzoulas, S.; Vlachos, D. G. *ChemSusChem* **2013**, *6*, 2066–2068.

reaction was suitable for vinyl aldehydes, further optimization was needed. Gratifyingly, heating the reaction mixture to reflux afforded the piperidine **3.15** in 54% yield and as a single diastereomer (Scheme 3.4). The scope was expanded to include a cinnamaldehyde derivative bearing a fluorine atom (**3.16**) in 50% yield (Scheme 3.4).

**Scheme 3.4** FeCl<sub>3</sub>- and BmimPF<sub>6</sub>-Mediated Aza-Prins Reaction



In summary, the aza-Prins cyclization provided an efficient and successful synthetic route to *N*-tosyl-4-chloropiperidines. In one step, the desired *trans*-substituted piperidines were produced in synthetically useful yields and excellent diastereoselectivity. The reported conditions were modified and optimized to improve reaction efficiency.

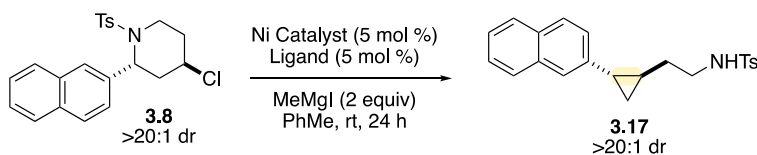
### 3.2.2 Development of XEC Reaction of *N*-Tosyl-4-chloropiperidines

Naphthyl-substituted piperidine **3.8** was selected to evaluate the XEC reaction. The optimal conditions for the previously developed ring contraction of aryl substituted tetrahydropyrans utilized Ni(cod)<sub>2</sub> as the precatalyst and *rac*-BINAP as the ligand (see Scheme 3.2d).<sup>9a</sup> Applying these conditions in a reaction with piperidine **3.8**, a 30% <sup>1</sup>H NMR yield of the cyclopropane **3.17** was observed (Table 3.2, entry 1). Five additional ligands were evaluated and surprisingly all failed to provide the cyclopropane product (entries 2–6). We next turned our attention to Ni(II) precatalysts. When the reaction was performed with (*R*-BINAP)NiCl<sub>2</sub>, the desired cyclopropane was observed in a 77% yield (entry 7).<sup>25</sup> Lewis acidic magnesium iodide (MgI<sub>2</sub>) is hypothesized

<sup>25</sup> For synthesis and characterization of (*R*-BINAP)NiCl<sub>2</sub>, see: (a) Standley, E. A.; Smith, S. J.; Müller, P.; Jamison, T. F. *Organometallics* **2014**, *33*, 2012–2018. (b) Dawson, D. D.; Oswald, V. F.; Borovik, A. S.; Jarvo, E. R. *Chem. Eur. J.* **2020**, *26*, 3044–3048. (c) Vogler, A. *Inorg. Chem. Commun.* **2016**, *65*, 39–40.

to accelerate the rate of oxidative addition of sluggish electrophiles.<sup>26,27</sup> The addition of one equivalent of MgI<sub>2</sub> increased the yield to 96% (entry 8).

**Table 3.2** Optimization of XEC reaction<sup>28</sup>



Entry	Nickel Catalyst	Ligand	Yield (%) <sup>a</sup>
1	Ni(cod) <sub>2</sub>	<i>rac</i> -BINAP	30
2	Ni(cod) <sub>2</sub>	XantPhos	0
3	Ni(cod) <sub>2</sub>	dppe	0
4	Ni(cod) <sub>2</sub>	BPhen	0
5	Ni(cod) <sub>2</sub>	bipy	0
6	Ni(cod) <sub>2</sub>	SiMes·BF <sub>4</sub>	0
7	( <i>R</i> -BINAP)NiCl <sub>2</sub>	–	77
8 <sup>b</sup>	( <i>R</i> -BINAP)NiCl <sub>2</sub>	–	96

<sup>a</sup>Yield determined by <sup>1</sup>H NMR based on the comparison to PhTMS as internal standard.

<sup>b</sup>Reaction performed with 1 equiv of MgI<sub>2</sub> added

In order to confirm that either enantiomer of the catalyst would deliver identical results and that stereochemical information is not conferred from the chiral catalyst, a control experiment was designed. When piperidine **3.9** was subjected to the XEC reaction conditions with each enantiomer of the catalyst, the reactions afforded similar yields of cyclopropane **3.18** and the same diastereoselectivity (Table 3.3, entries 1 and 2). When a mixture of both diastereomers of the catalyst was employed in the reaction conditions, it provided cyclopropane **3.18** in a similar yield

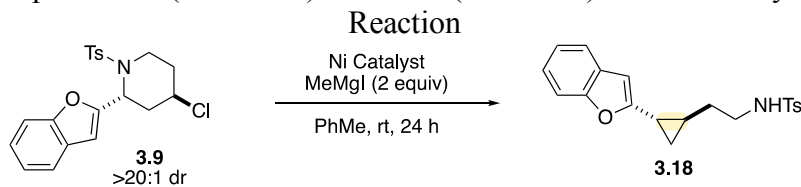
<sup>26</sup> (a) Greene, M. A.; Yonova, I. M.; Williams, F. J.; Jarvo, E. R. *Org. Lett.* **2012**, *14*, 4293–4296. (b) Felkin, H.; Swierczewski, G. *Tetrahedron Lett.* **1972**, *13*, 1433–1436

<sup>27</sup> Chen, P.-P.; Lucas, E. L.; Greene, M. A.; Zhang, S.-Q.; Tollefson, E. J.; Erickson, L. W.; Taylor, B. L. H.; Jarvo, E. R.; Hong, X. *J. Am. Chem. Soc.* **2019**, *141*, 5835–5855.

<sup>28</sup> Optimization of the XEC reaction with piperidine **3.8** was performed by Dr. Erika Lucas.

and diastereoselectivity (entry 3).<sup>29</sup> This experiment demonstrates that either enantiomer of catalyst can be employed in this XEC reaction.

**Table 3.3** Comparison of (*R*-BINAP)NiCl<sub>2</sub> and (*S*-BINAP)NiCl<sub>2</sub> as catalysts in the XEC



Entry	Ni Catalyst	Catalyst Loading	Yield (%) <sup>a</sup>	dr (trans:cis)
1	( <i>R</i> -BINAP)NiCl <sub>2</sub>	5 mol %	86	>20:1
2	( <i>S</i> -BINAP)NiCl <sub>2</sub>	5 mol %	85	>20:1
3	( <i>R</i> -BINAP)NiCl <sub>2</sub> + ( <i>S</i> -BINAP)NiCl <sub>2</sub>	2.5 mol % + 2.5 mol %	88	>20:1

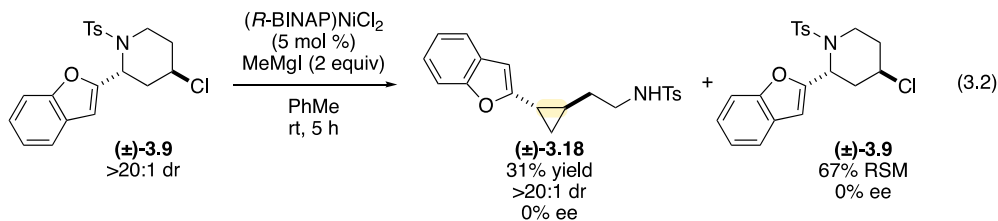
<sup>a</sup>Yield determined by <sup>1</sup>H NMR based on comparison to PhTMS as internal standard

Next, we hypothesized that the chiral catalyst could selectively react with one enantiomer over the other enantiomer and we wanted to investigate if a kinetic resolution was occurring with the chiral catalyst.<sup>30</sup> We designed a control reaction to test this hypothesis where the reaction would be quenched at low conversion of starting material and the enantiomeric excess (ee) of the starting material and product would be assessed (eq. 3.2). Racemic starting material **3.9** was subjected to the standard reaction conditions and the reaction was quenched after 5 h with 33% conversion of starting material. The starting material was recovered, the product was isolated and the enantiomeric excess (ee) was determined using chiral SFC. The starting material and product were both racemic (0% ee). This experiment confirms the absence of a kinetic resolution.

<sup>29</sup> The racemic catalyst, (*rac*-BINAP)NiCl<sub>2</sub>, was synthesized and resulted in a viscous oil which led to difficulties in reaction set-up. We elected to utilize the chiral catalyst because it afforded fine black powder which could easily be added to the reaction flask

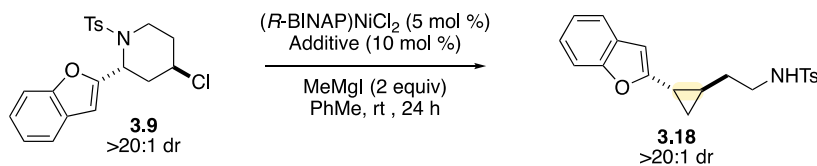
<sup>30</sup> For reviews on kinetic resolutions, see: (a) Huerta, F. F.; Minidis, A. B. E.; Bäckvall, J.-E. *Chem. Soc. Rev.* **2001**, *30*, 321–331. (b) Cook, G. R. *Curr. Org. Chem.* **2000**, *4*, 869.





In order to understand the importance of the nickel (II) precatalyst and why diminished yields were observed when Ni(cod)<sub>2</sub> was employed as the precatalyst, a control experiment was designed. We hypothesized that 1,5-cyclooctadiene (cod) was inhibiting the XEC reaction by strong ligation to the catalyst and producing a stable, off-cycle nickel complex. This would prevent the catalyst from engaging the substrate and entering the catalytic cycle. In order to confirm this hypothesis, the XEC reaction of piperidine **3.9** was performed with (*R*-BINAP)NiCl<sub>2</sub> as the catalyst and exogenous cod as an additive. It was confirmed that cod significantly inhibits the XEC reaction of piperidine **3.9** and the desired product **3.18** was observed in only 6% yield (Table 3.4, entry 2).

**Table 3.4** Cod Inhibition Experiment with Piperidine **3.9**



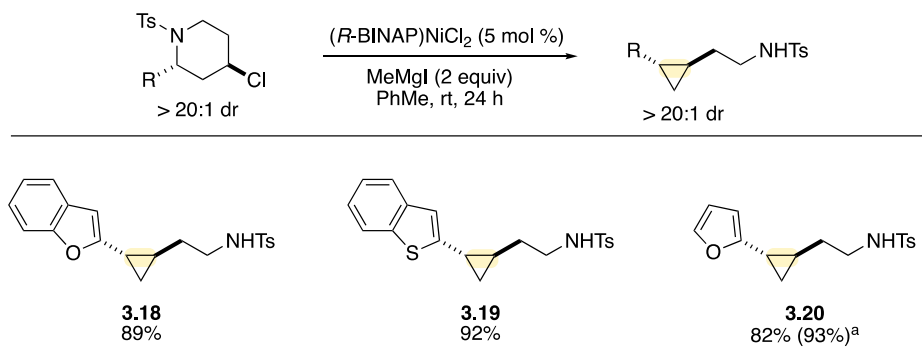
Entry	Additive	Yield (%) <sup>a</sup>	RSM (%) <sup>a</sup>
1	–	93	0
2	cod	6	90

<sup>a</sup>Yield determined by <sup>1</sup>H NMR based on the comparison to PhTMS as internal standard

Once the optimal conditions for the XEC reaction were identified, the scope was expanded to include substrates with pendant heterocyclic functionalities (Scheme 3.5). When benzofuran and benzothiophene substituted piperidines **3.9** and **3.10** were subjected to the standard XEC reaction, disubstituted cyclopropanes **3.18** and **3.19** were isolated in 89% and 92% yields,

respectively. Additionally, non-extended aromatic systems were well-tolerated under these reaction conditions and furanyl-substituted cyclopropane (**3.20**) was isolated in an 82% yield. Importantly, all cyclopropane products were synthesized with excellent diastereoselectivity.

**Scheme 3.5** Substrate Scope for Ring Contraction of Heteroaryl-Piperidines

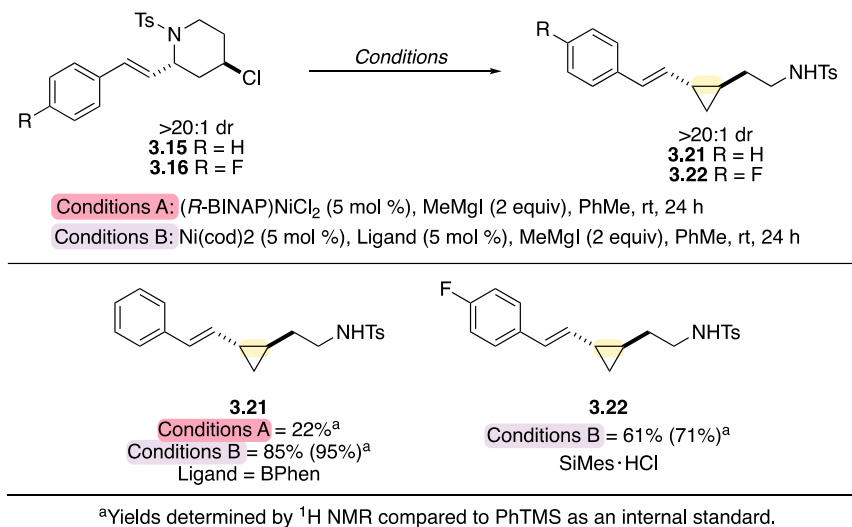


<sup>a</sup>Yields in parenthesis determined by <sup>1</sup>H NMR compared to PhTMS as an internal standard.

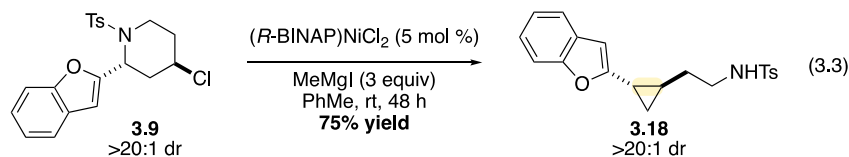
Motivated by the previously developed method by the Jarvo laboratory to prepare vinylcyclopropanes from vinyltetrahydropyrans, we aimed to develop a complementary approach to synthesize vinylcyclopropanes from vinylpiperidines.<sup>9b</sup> It was hypothesized that these substrates would perform similarly to the aryl substrates in this ring contraction because the allylic carbon-nitrogen bond is a comparable electrophile to the benzylic sulfonamides. Surprisingly, when piperidine **3.15** was subjected the optimal reaction conditions, cyclopropane **3.21** was observed in a 22% yield (Scheme 3.6, Conditions A). We hypothesized that the vinylpiperidine is capable of competing with cod for the ligation to the nickel catalyst. To test this hypothesis, the XEC reaction of piperidine **3.15** was performed with Ni(cod)<sub>2</sub> and bathophenanthroline (BPhen) and cyclopropane **3.21** was observed in 95% <sup>1</sup>H NMR yield (Scheme 3.6, Conditions B). We applied these conditions to the remaining vinylpiperidine that was synthesized and the scope was expanded

to include an electron withdrawing group (**3.22**) utilizing Conditions B. This substrate cleanly underwent the XEC reaction and furnished the disubstituted cyclopropane **3.22** in 61% yield.<sup>31</sup>

**Scheme 3.6** Substrate Scope for Ring Contraction of Vinyl Piperidines



Interestingly, the XEC reaction employed the air stable (*R*-BINAP)NiCl<sub>2</sub>, however, all of the reactions were performed in the glovebox. To demonstrate that a glovebox was not necessary, the XEC reaction of piperidine **3.9** was performed on the benchtop with three equivalents of methylmagnesium iodide for 48 h, and the desired cyclopropane **3.18** was isolated in a 75% yield (eq. 3.3).<sup>32</sup>

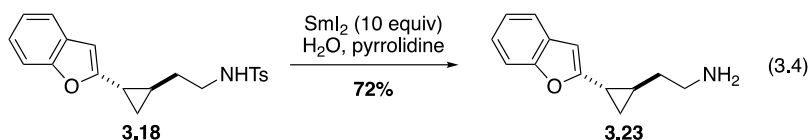


Finally, we demonstrated that the pendant sulfonamide was a synthetically useful intermediate. The tosyl group was efficiently deprotected in the presence of samarium (II) diiodide

<sup>31</sup> It is important to note that various ligands were evaluated for the synthesis of the vinylcyclopropanes and all of these ligands provided the product in similar yields. The yields and ligands reported in Scheme 3.6 are the conditions that produced the highest yields of the vinylcyclopropane. See Experimental Section for more information.

<sup>32</sup> The standard reaction conditions were modified to increase the yield of the XEC reaction on the benchtop. See Experimental Section for more information.

to afford the primary amine **3.23** in a 72% yield (eq. 3.4). Primary amines themselves are important synthetic intermediates that can be further functionalized and are prevalent in biologically active compounds, pharmaceuticals, and natural products.<sup>33</sup>



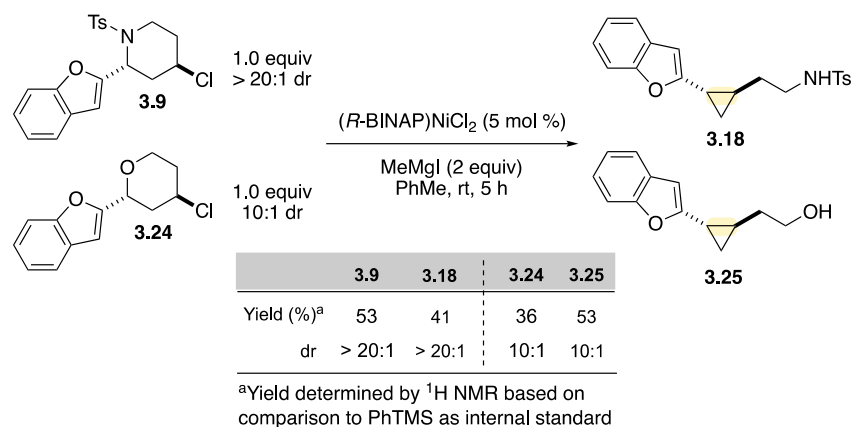
### 3.3 Proposed Mechanism

In 2019, the Jarvo and Hong laboratories published an experimentally and computationally supported mechanism for the nickel-catalyzed XC and XEC reactions of benzylic ethers.<sup>9,27</sup> These reactions share similar features with the ring contraction of piperidines including a BINAP-ligated nickel complex and methylmagnesium iodide as the reductant. Because of these similar features, we hypothesized that these reactions may also share some fundamental mechanistic details. A competition experiment between piperidine **3.9** and ether **3.24** was designed to gain further insight into the mechanistic information (Scheme 3.7). Piperidine **3.9** and ether **3.24** were subjected to the reaction conditions and after 5 h the corresponding cyclopropanes were produced in a 1.2:1 ratio slightly favoring the alcohol **3.25**. The Hong group also calculated the transition state energies for the oxidative addition events and confirmed that the barrier heights are similar for the ether and the sulfonamide and in agreement with the results of the competition experiment.<sup>34</sup> This allowed us to conclude that the two reactions shared the same rate-determining step for cyclopropane formation which we hypothesized is the oxidative addition of the carbon-heteroatom bond.

<sup>33</sup> (a) Ruiz-Castillo, P.; Buchwald, S. L. *Chem. Rev.* **2016**, *116*, 12564–12649.; (b) McGrath, N. A.; Brichacek, M.; Njardarson, J. T. *J. Chem. Educ.* **2010**, *87*, 1348; (c) Liu, Y.; Ge, H. *Nat. Chem.* **2017**, *9*, 26.

<sup>34</sup> For more information of the computational results, see ref 1.

**Scheme 3.7** Competition Experiment of Piperidine **3.9** and Ether **3.24**<sup>35</sup>



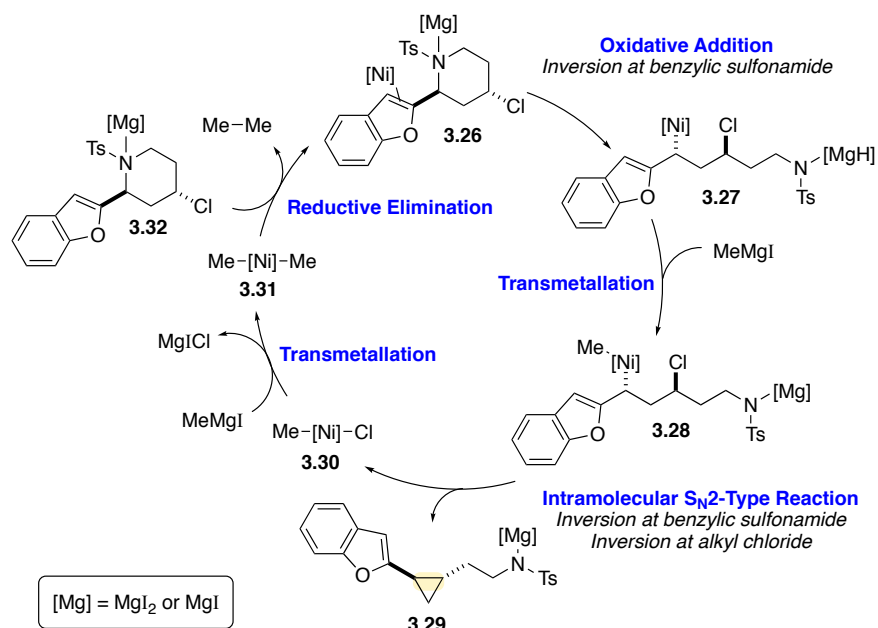
Based on this insight, we propose the following plausible catalytic cycle (Figure 3.2). The nickel catalyst engages the arene or alkene (**3.32**), followed by oxidative addition of the carbon-nitrogen bond. This step can be accelerated with the addition of Lewis acidic magnesium diiodide (see Table 3.1). Next, transmetalation with methylmagnesium iodide affords nickel(II) species **3.28**. This key intermediate undergoes the proposed intramolecular S<sub>N</sub>2-type reaction to afford the magnesium ligated cyclopropane product **3.29**. A second transmetalation and subsequent reductive elimination of ethane turns over the catalytic cycle.

The XEC reaction is proposed to be a stereospecific reaction. To understand the overall stereochemical outcome of the reaction, we need to understand the individual elementary steps. We hypothesized that the oxidative addition of the C–N bond proceeds via an S<sub>N</sub>2 mechanism with inversion of stereochemistry at the benzylic carbon. Next, the S<sub>N</sub>2-type reaction for the formation of the cyclopropane (**3.28** to **3.29**), can be envisaged as an S<sub>E</sub>2-type reaction at the benzylic nickel center and an S<sub>N</sub>2-type reaction at the alkyl chloride.<sup>36</sup> This leads to double inversion (i.e., net retention) at the benzylic center and inversion at the alkyl chloride.

<sup>35</sup> Competition experiment performed by Dr. Erika Lucas.

<sup>36</sup> Fukuto, J. M.; Jensen, F. R. *Acc. Chem. Res.* **1983**, *16*, 177–184.

**Figure 3.2** Proposed Catalytic Cycle for the XEC Reaction of *N*-Tosyl-Piperidines



### 3.4 Conclusion

In conclusion, we have optimized the aza-Prins cyclization for the synthesis of heterocyclic and vinylic piperidines. This reaction proceeds in moderate to good yield and excellent diastereoselectivity and furnishes the desired piperidine in a single step from commercially available aldehydes. Additionally, we have developed the first intramolecular nickel-catalyzed XEC reaction of *N*-tosyl-4-chloropiperidines. The XEC reaction utilizes an air-stable nickel (II) precatalyst and can be performed without the use of a glovebox. The reaction proceeds in great yield and with excellent diastereoselectivity. It is tolerant of heterocyclic functionalities and has been expanded to include vinylcyclopropanes. In this Chapter, we have established precedent for the activation of unstrained, neutral carbon-nitrogen bonds and have provided a framework for the development of new catalysts for the activation of other carbon-nitrogen bonds.<sup>37</sup>

<sup>37</sup> For the nickel-catalyzed XC reaction of unstrained, neutral carbon-nitrogen bond, see: (a) Chapter 1 of this thesis. (b) Hewitt, K. A.; Herbert, C. A.; Matus, A. C.; Jarvo, E. R. *Molecules* **2021**, *26*, 5947–5971.

## 3.5 Experimental Details

### 3.5.1 General Procedures

All reactions were carried out under an atmosphere of N<sub>2</sub> when noted. All glassware was oven- or flame-dried prior to use. Tetrahydrofuran (THF), diethyl ether (Et<sub>2</sub>O), dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>), methanol (MeOH), and toluene (PhMe) were degassed with Ar and then passed through two 4 x 36 inch columns of anhydrous neutral A-2 alumina (8 x 14 mesh; LaRoche Chemicals; activated under a flow of argon at 350 °C for 12 h) to remove H<sub>2</sub>O.<sup>38</sup> All other solvents utilized were purchased “anhydrous” commercially, or purified as described. <sup>1</sup>H NMR spectra were recorded on Bruker DRX-400 (400 MHz <sup>1</sup>H, 100 MHz <sup>13</sup>C), GN-500 (500 MHz <sup>1</sup>H, 125.7 MHz <sup>13</sup>C), CRYO-500 (500 MHz <sup>1</sup>H, 125.7 MHz <sup>13</sup>C), or AVANCE-600 (600 MHz <sup>1</sup>H, 150 MHz <sup>13</sup>C) spectrometers. Proton chemical shifts are reported in ppm (δ) relative to internal tetramethylsilane (TMS, δ 0.00). Data are reported as follows: chemical shift (multiplicity [singlet (s), broad singlet (br s), doublet (d), doublet of doublets (dd), doublet of doublet of doublets (ddd), doublet of doublet of triplets (ddt), doublet of triplets (dt), triplet (t), triplet of doublets (td), triplet of triplets (tt), quartet (q), quartet of doublets (qd), quintet (quin), apparent singlet (as), apparent doublet (ad), apparent doublets of doublets (add), apparent doublet of triplets (adt), apparent doublet of quintets (adquin), apparent doublet of septets (adsept), apparent triplet (at), apparent triplet of doublets (atd), apparent triplet of triplets (att), apparent quartet (aq), apparent quartet of doublets (aqd), apparent quartet of triplets (aqt), apparent quintet of doublets (aquin d), apparent sextet (asext), apparent septet (asept), multiplet (m)], coupling constants [Hz], integration). Carbon chemical shifts are reported in ppm (δ) relative to TMS with the respective solvent resonance as the internal standard (CDCl<sub>3</sub>, δ 77.16 ppm). Unless otherwise indicated, NMR data were collected at 25 °C.

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<sup>38</sup> Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518–1520.

Infrared (IR) spectra were obtained on a Thermo Scientific Nicolet iS5 spectrometer with an iD5 ATR tip (neat) and are reported in terms of frequency of absorption ( $\text{cm}^{-1}$ ). Analytical thin-layer chromatography (TLC) was performed using Silica Gel 60 F254 precoated plates (0.25 mm thickness). Visualization was accomplished by irradiation with a UV lamp. Flash chromatography was performed using SiliaFlash F60 (40-63  $\mu\text{m}$ , 60 Å) from SiliCycle. Automated chromatography was carried out on a Teledyne Isco CombiFlash Rf Plus. Melting points (M.p.) were obtained using a MelTemp melting point apparatus and are uncorrected. SFC Determinations of enantiopurity were determined by chiral SFC analysis and performed on an Agilent Technologies HPLC (1260 series) system. Analytical Instruments using OD-H Chiralpak column (100 bar, 50°C, 254 nm). High resolution mass spectrometry was performed by the University of California, Irvine Mass Spectrometry Center.

Bis(1,5-cyclooctadiene)nickel was purchased from Strem, stored in a glove box freezer (–20 °C) under an atmosphere of  $\text{N}_2$  and used as received. All ligands were purchased from Strem or Sigma Aldrich and were stored under  $\text{N}_2$  atmosphere and used as received. The methylmagnesium iodide was titrated with iodine prior to use.<sup>39</sup> All other chemicals were purchased commercially and used as received, unless otherwise noted.

### **3.5.2 General Cross-Electrophile Coupling Reaction Procedures**

#### **3.5.2.1 Method A: Cross-Electrophile Coupling Reaction with (*R*-BINAP)NiCl<sub>2</sub>**

In a glovebox, an oven-dried 7 mL vial equipped with a stir bar was charged with substrate (1 equiv), (*R*-BINAP)NiCl<sub>2</sub> (5 mol %), and PhMe (0.2 M in substrate). A solution of MeMgI in Et<sub>2</sub>O (2 equiv) was then added dropwise via syringe. After 24 h, the reaction vial was removed from the glovebox, quenched with MeOH, filtered through a plug of silica gel eluting with Et<sub>2</sub>O,

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<sup>39</sup> Krasoviskiy, A.; Knochel, P. *Synthesis*, **2006**, 5, 890–891.



and concentrated in vacuo. Phenyltrimethylsilane (PhTMS; 8.6  $\mu\text{L}$ , 50.  $\mu\text{mol}$ ) was added and the yield was determined by  $^1\text{H}$  NMR based on comparison to PhTMS as internal standard before purification by column chromatography.

For challenging substrates, 1.0–1.5 equiv  $\text{MgI}_2$  was added to the reaction mixture and the vial was wrapped in foil for the duration of the reaction due to the light sensitivity of  $\text{MgI}_2$ .

### 3.5.2.2 Preparation of Methylmagnesium Iodide

Under a  $\text{N}_2$  atmosphere, a three-necked flask equipped with a stir bar, reflux condenser, and Schlenk filtration apparatus was charged with magnesium turnings (2.80 g, 115 mmol). The flask and magnesium turnings were then flame-dried under vacuum and the flask was backfilled with  $\text{N}_2$ . Anhydrous  $\text{Et}_2\text{O}$  (25 mL) and a crystal of iodine (ca. 2 mg) were added to the flask. Freshly distilled iodomethane (5.0 mL, 80 mmol) was slowly added over 30 min to maintain a gentle reflux. The mixture was stirred for 2 h at room temperature then filtered through the fritted Schlenk filter into a pear-shaped flask under a  $\text{N}_2$  atmosphere. The pear-shaped flask was capped with a septum, sealed with parafilm, and stored in the glovebox under a  $\text{N}_2$  atmosphere for up to eight weeks. The resulting methyl Grignard reagent was typically between 2.4 and 3.0 M as titrated by Knochel's method.<sup>39</sup>

### 3.5.2.3 Preparation of (*R*-BINAP) $\text{NiCl}_2$

(*R*-BINAP) $\text{NiCl}_2$  was synthesized according to a procedure reported by Jamison.<sup>25a</sup> To a 50 mL round-bottom flask equipped with a stir bar was added  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  (240 mg, 1.0 mmol, 1.0 equiv). The flask was placed under vacuum and flame-dried until nearly all of the nickel compound had turned from green to yellow-orange (a small amount of remaining green of the hexahydrate is necessary for the reaction to proceed). After cooling to room temperature, (*R*-BINAP) (620 mg, 1.0 mmol, 1.0 equiv) was added to the flask and a reflux condenser was attached.

The flask was evacuated, backfilled with N<sub>2</sub>, and then anhydrous MeCN (20 mL) was added. The reaction mixture was allowed to reflux for 24 h, at which point the solution was cooled to room temperature and filtered under vacuum to yield a fine, black powder (530 mg, 0.71 mmol, 71% yield)

#### **3.5.2.4 Method B: Cross-Electrophile Coupling Reaction with Ni(cod)<sub>2</sub>**

In a glovebox, an oven-dried 7 mL vial equipped with a stir bar was charged with substrate (1 equiv), Ni(cod)<sub>2</sub> (5 mol %), ligand (5 mol %) and PhMe (0.2 M in substrate). A solution of MeMgI in Et<sub>2</sub>O (2 equiv) was then added dropwise via syringe. After 24 h, the reaction vial was removed from the glovebox, quenched with MeOH, filtered through a plug of silica gel eluting with Et<sub>2</sub>O, and concentrated in vacuo. Phenyltrimethylsilane (PhTMS; 8.6 μL, 50. μmol) was added and the yield was determined by <sup>1</sup>H NMR based on comparison to PhTMS as internal standard before purification by column chromatography.

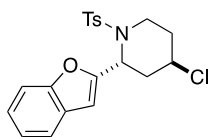
#### **3.5.3 Control Experiment: Comparison of (*R*-BINAP)NiCl<sub>2</sub> to (*S*-BINAP)NiCl<sub>2</sub> in the XEC Reaction**

In order to confirm that either enantiomer of the nickel catalyst would deliver identical results when employed in the XEC reaction, a control experiment was designed. The control experiment was performed according to Method A. Vial 1 was charged with substrate **3.9** (39 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.7 mg, 5.0 μmol, 5.0 mol %), methylmagnesium iodide (80. μL, 0.20 mmol, 2.0 equiv, 2.5 M in Et<sub>2</sub>O) and PhMe (0.50 mL). Vial 2 was charged with substrate **3.9** (39 mg, 0.10 mmol, 1.0 equiv), (*S*-BINAP)NiCl<sub>2</sub> (3.7 mg, 5.0 μmol, 5.0 mol %), methylmagnesium iodide (80. μL, 0.20 mmol, 2.0 equiv, 2.5 M in Et<sub>2</sub>O) and PhMe (0.50 mL). Vial 3 was charged with substrate **3.9** (39 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (1.9 mg, 2.5 μmol, 2.5 mol %), (*S*-BINAP)NiCl<sub>2</sub> (1.9 mg, 2.5 μmol, 2.5 mol %), methylmagnesium iodide

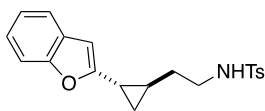
(80.  $\mu\text{L}$ , 0.20 mmol, 2.0 equiv, 2.5 M in  $\text{Et}_2\text{O}$ ) and PhMe (0.50 mL). After 24 h, the reaction vials were removed from the glovebox, quenched with MeOH, filtered through a plug of silica gel eluting with  $\text{Et}_2\text{O}$ , and concentrated in vacuo. Phenyltrimethylsilane (PhTMS; 8.6  $\mu\text{L}$ , 50.  $\mu\text{mol}$ ) was added and the yields were determined by  $^1\text{H}$  NMR compared to PhTMS as an internal standard. The results of this control experiment are tabulated in **Table 3.3**.

### 3.5.4 Control Experiment: Kinetic Resolution with Chiral Catalyst

In order to identify the presence or absence of a kinetic resolution, a control experiment was designed. The control experiment was performed according to Method A. The following amounts of reagents were used: substrate **3.9** (39 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.8 mg, 5.0  $\mu\text{mol}$ , 5.0 mol %), methylmagnesium iodide (70.  $\mu\text{L}$ , 0.20 mmol, 2.0 equiv, 2.9 M in  $\text{Et}_2\text{O}$ ), and PhMe (0.5 mL). After 5 h, the reaction vial was removed from the glovebox, quenched with MeOH, filtered through a plug of silica gel eluting with  $\text{Et}_2\text{O}$ , and concentrated in vacuo. The results of this control experiment are outlined in eq. 3.2. The analytical data for both starting material and product are tabulated below.



Before purification, it was determined that 67% of the starting material was recovered by  $^1\text{H}$  NMR based on comparison to PhTMS. The residue was purified by column chromatography to recover piperidine **3.9** (24 mg, 63  $\mu\text{mol}$ , 63% recovery of starting material). Refer to **3.9** below for analytical data. **SFC Analysis** (Chiralcel OD-H, 20% IPA, 2.0 mL/min, 250 nm) indicated 0% ee.  $t_{\text{R}}$  (only diastereomer, enantiomer 1) = 27.7 minutes,  $t_{\text{R}}$  (only diastereomer, enantiomer 2) = 29.9 minutes.

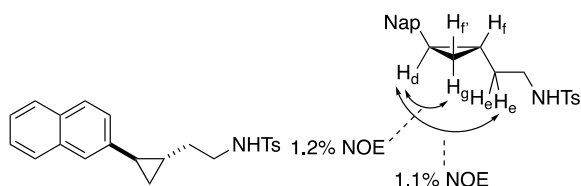


Before purification, a  $^1\text{H}$  NMR yield of 31% yield was determined based on comparison to PhTMS. The residue was purified by column chromatography to yield the desired product **3.18** (11 mg, 31  $\mu\text{mol}$ , 31% yield). Refer to **3.18** below for analytical data. **SFC Analysis** (Chiralcel OD-H, 5% IPA, 2.0 mL/min, 250 nm) indicated 0% ee.  $t_{\text{R}}$  (only diastereomer, enantiomer 1) = 10.4 minutes,  $t_{\text{R}}$  (only diastereomer, enantiomer 2) = 12.9 minutes.

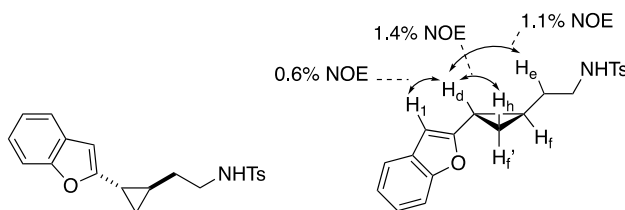
### 3.5.5 Cod Inhibition Experiment

In order to determine whether 1,5-cyclooctadiene (cod) was inhibiting the XEC reaction when  $\text{Ni}(\text{cod})_2$  was employed as a precatalyst, a control experiment was designed. In a glovebox, an oven-dried 7 mL vial equipped with a stir bar was charged with substrate (39 mg, 0.10 mmol, 1.0 equiv) and (*R*-BINAP) $\text{NiCl}_2$  (3.8 mg, 5.0  $\mu\text{mol}$ , 5 mol %). Then 1,5-cyclooctadiene (PhMe (0.5 mL). Then methylmagnesium iodide (70.  $\mu\text{L}$ , 0.20 mmol, 2.0 equiv, 2.9 M in  $\text{Et}_2\text{O}$ ) was added dropwise via syringe. After 24 h, the reaction vial was removed from the glovebox, quenched with MeOH, filtered through a plug of silica gel eluting with  $\text{Et}_2\text{O}$ , and concentrated in vacuo. Cyclopropane **3.18** was observed in 6%  $^1\text{H}$  NMR yield and piperidine **3.9** was recovered in 90%  $^1\text{H}$  NMR yield compared to PhTMS. The results from this control experiment are tabulated in **Table 3.4**.

### 3.5.6 Characterization Data for Cyclopropane Products



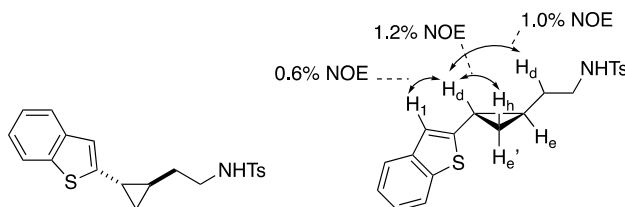
**4-Methyl-*N*-(2-(*trans*-2-(naphthalen-2-yl)cyclopropyl)ethyl)benzenesulfonamide (3.17)**<sup>40</sup> was prepared according to Method A. The following amounts of reagents were used: piperidine **3.8** (40. mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.8 mg, 5.0 μmol, 5.0 mol %), MgI<sub>2</sub> (28 mg, 0.10 mmol, 1.0 equiv), PhMe (0.5 mL, 0.2 M in substrate) and methylmagnesium iodide (0.10 mL, 0.20 mmol, 2.0 equiv, 2.1 M in Et<sub>2</sub>O). Before purification, a <sup>1</sup>H NMR yield of 96% was obtained. The residue was purified by column chromatography (0–30% EtOAc/hexanes) to afford the product as a white solid (30. mg, 82. μmol, 82% yield, > 20:1 dr *trans*:*cis*). The dr was determined based on the integration of the resonances attributed to the benzylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **mp** 93–95 °C; **TLC R<sub>f</sub>** = 0.2 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.76 (d, *J* = 8.0 Hz, 1H), 7.71 (ad, *J* = 8.2 Hz, 4H), 7.45–7.36 (m, 3H), 7.22 (d, *J* = 8.1 Hz, 2H), 7.09 (d, *J* = 8.6 Hz, 1H), 4.68 (t, *J* = 6.2 Hz, 1H), 3.08 (q, *J* = 6.7 Hz, 2H), 2.37 (s, 3H), 1.79–1.73 (m, 1H), 1.63–1.55 (m, 2H), 1.06–0.96 (m, 2H), 0.83–0.78 (m, 1H); **<sup>13</sup>C NMR** (125.8 MHz, CDCl<sub>3</sub>) δ 143.5, 140.4, 137.0, 133.7, 132.1, 129.8 (2C), 128.1, 127.7, 127.4, 127.2 (2C), 126.3, 125.2, 124.6, 123.8, 43.4, 34.4, 23.3, 21.6, 20.9, 15.8; **HRMS** (TOF MS ES+) *m/z* [M + Na]<sup>+</sup> calcd for C<sub>22</sub>H<sub>23</sub>NO<sub>2</sub>SNa, 388.1347, found 388.1348.



***N*-(2-(*trans*-2-(Benzofuran-2-yl)cyclopropyl)ethyl)-4-methylbenzenesulfonamide (3.18)** was prepared according to Method A. The following amounts of reagents were used: (*R*-BINAP)NiCl<sub>2</sub> (7.5 mg, 10. μmol, 5.0 mol %), piperidine **3.9** (77 mg, 0.20 mmol, 1.0 equiv), PhMe (1.0 mL, 0.20

<sup>40</sup> Substrate synthesized by Dr. Erika Lucas.

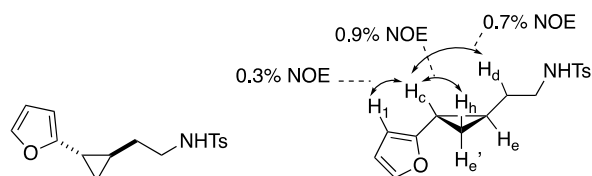
M in substrate), and methylmagnesium iodide (0.16 mL, 0.40 mmol, 2.0 equiv, 2.5 M solution in Et<sub>2</sub>O). The residue was purified by column chromatography (5–25% EtOAc/hexanes) to afford the product as a white solid (44 mg, 0.12 mmol, 89% yield, > 20:1 dr trans:cis). The dr was based on the integration of the resonances attributed to the benzylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **mp** 85–87°C; **TLC** *R<sub>f</sub>* = 0.5 (5–25% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.74 (d, *J* = 8.0 Hz, 2H), 7.43 (dd, *J* = 6.6 Hz, 1.9 Hz, 1H), 7.34 (d, *J* = 7.8 Hz, 1H), 7.26 (t, *J* = 2.8 Hz, 2H), 7.17 (att, *J* = 7.3 Hz, 5.8 Hz, 2H), 6.31 (s, 1H), 4.65 (t, *J* = 7.5 Hz, 1H), 3.12–3.05 (m, 2H), 2.39 (s, 3H), 1.76 (dt, *J* = 9.2 Hz, 4.9 Hz, 1H), 1.66–1.49 (m, 2H), 1.23–1.17 (m, 1H), 1.17–1.10 (m, 1H), 0.79 (dt, *J* = 8.6 Hz, 5.3 Hz, 1H); **<sup>13</sup>C NMR** (125.8 MHz, CDCl<sub>3</sub>) δ 159.3, 154.3, 143.6, 137.0, 129.9 (2C), 129.1, 127.2 (2C), 123.2, 122.7, 120.1, 110.7, 100.6, 43.2, 33.8, 21.6, 19.0, 16.6, 13.7; **IR** (neat) 3295, 2923, 1602, 1455, 1417, 1362, 1256, 1154, 1090, 1074, 891, 820, 754, 661 cm<sup>-1</sup>; **HRMS** (TOF MS ES+) *m/z* [M + H]<sup>+</sup> calcd for C<sub>20</sub>H<sub>22</sub>NO<sub>3</sub>S, 356.1320, found 356.1317.



### ***N*-(2-(*trans*-2-(Benzo[*b*]thiophene-2-yl)cyclopropyl)ethyl)-4-methylbenzenesulfonamide**

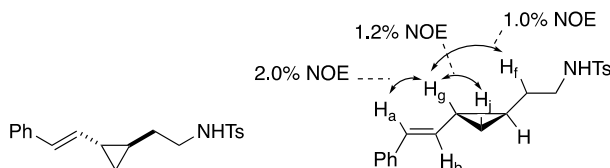
**(3.19)** was prepared according to Method A. The following amounts of reagents were used: (*R*-BINAP)NiCl<sub>2</sub> (7.5 mg, 10. μmol, 5.0 mol %), piperidine **3.10** (81 mg, 0.20 mmol, 1.0 equiv), PhMe (1.0 mL, 0.20 M in substrate), and methylmagnesium iodide (0.14 mL, 0.40 mmol, 2.0 equiv, 2.9 M solution in Et<sub>2</sub>O). The residue was purified by chromatography (5–25% EtOAc/hexanes) to afford the product as a white solid (68 mg, 0.18 mmol, 92% yield, > 20:1 dr trans:cis). The dr was based on the integration of the resonances attributed to the benzylic

hydrogens in the  $^1\text{H}$  NMR spectrum. The relative configuration was assigned based on NOE analysis. **mp** 93–94 °C; **TLC**  $R_f$  = 0.5 (25% EtOAc/hexanes);  **$^1\text{H}$  NMR** (500 MHz,  $\text{CDCl}_3$ ) 7.74 (d,  $J$  = 8.0 Hz, 2H), 7.70 (d,  $J$  = 8.0 Hz, 1H), 7.60 (d,  $J$  = 7.8 Hz, 1H), 7.37–7.16 (m, 4H), 6.89 (s, 1H), 4.66 (t,  $J$  = 6.2 Hz, 1H), 3.09 (q,  $J$  = 6.7 Hz, 2H), 2.39 (s, 3H), 1.88 (dt,  $J$  = 9.1 Hz, 4.8 Hz, 1H), 1.58 (q,  $J$  = 6.8 Hz, 2H), 1.11–1.03 (m, 1H), 1.01 (dt,  $J$  = 8.9 Hz, 4.9 Hz, 1H), 0.85 (dt,  $J$  = 8.6 Hz, 4.7 Hz, 1H);  **$^{13}\text{C}$  NMR** (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  148.0, 143.5, 140.2, 138.4, 136.9, 129.8 (2C), 127.1 (2C), 124.3, 123.5, 122.6, 122.1, 118.8, 43.1, 34.0, 21.8, 21.6, 19.1, 16.6; **IR** (neat) 3249, 2919, 1596, 141.01 37, 1293, 1144, 1092, 1782 1067, 1043, 899, 745, 645  $\text{cm}^{-1}$ ; **HRMS** (TOF MS ES+)  $m/z$   $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{20}\text{H}_{21}\text{NO}_2\text{S}_2\text{Na}$  394.0912, found 394.0911.



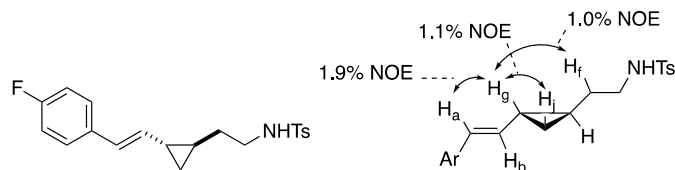
***N*-(2-(*trans*-2-(Furan-2-yl)cyclopropyl)ethyl)-4-methylbenzenesulfonamide (3.20)** was prepared according to Method A. The following amounts of reagents were used: (*R*-BINAP)NiCl<sub>2</sub> (7.5 mg, 10.  $\mu\text{mol}$ , 5.0 mol %), piperidine **3.12** (69 mg, 0.20 mmol, 1.0 equiv), PhMe (1.0 mL, 0.20 M in substrate), and methylmagnesium iodide (0.14 mL, 0.40 mmol, 2.0 equiv, 2.8 M solution in Et<sub>2</sub>O). Before purification, a  $^1\text{H}$  NMR yield of 93% was obtained. The residue was purified by column chromatography (5–20% EtOAc/hexanes) to afford the product as a yellow oil (50 mg, 0.16 mmol, 82% yield, > 20:1 dr *trans*:*cis*). The dr was based on the integration of the resonances attributed to the benzylic hydrogens in the  $^1\text{H}$  NMR spectrum. The relative configuration was assigned based on NOE analysis. **TLC**  $R_f$  = 0.5 (25% EtOAc/hexanes);  **$^1\text{H}$  NMR** (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75 (d,  $J$  = 8.3 Hz, 2H), 7.30 (d,  $J$  = 8.4 Hz, 2H), 7.22 (dd,  $J$  = 1.8 Hz, 0.9 Hz, 1H), 6.24 (dd,  $J$  = 3.3 Hz, 1.9 Hz, 1H), 5.89 (d,  $J$  = 3.2 Hz, 1H), 4.55 (t,  $J$  = 6.3 Hz, 1H), 3.07 (septet,  $J$  = 6.4 Hz, 2H), 2.42 (s, 3H), 1.61 (dt,  $J$  = 9.3 Hz, 4.8 Hz, 1H), 1.52 (q,  $J$  = 6.9 Hz, 2H), 1.04– 0.96

(m, 1H), 0.94 (dt,  $J = 8.5$  Hz, 4.8 Hz, 1H), 0.65 (dt,  $J = 8.8$  Hz, 4.8 Hz, 1H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  156.3, 143.5, 140.6, 137.1, 129.8 (2C), 127.2 (2C), 110.4, 103.7, 43.2, 33.7, 21.7, 18.2, 16.0, 13.1; HRMS (TOF MS ES+)  $m/z$   $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{16}\text{H}_{19}\text{NO}_3\text{SNa}$  328.0983, found 328.0981.



**(E)-4-Methyl-N-(2-(trans-2-styrylcyclopropyl)ethyl)benzenesulfonamide (3.21)** was prepared according to Method B. The following amounts of reagents were used: piperidine **3.15** (38 mg, 0.10 mmol, 1.0 equiv),  $\text{Ni}(\text{cod})_2$  (1.4 mg, 5.0  $\mu\text{mol}$ , 5.0 mol %), BPhen (1.7 mg, 5.0  $\mu\text{mol}$ , 5.0 mol %), PhMe (0.50 mL, 0.20 M in substrate), and methylmagnesium iodide (0.080 mL, 0.40 mmol, 2.0 equiv, 2.5 M solution in  $\text{Et}_2\text{O}$ ). Before purification, a  $^1\text{H}$  NMR yield of 95% was obtained. The residue was purified by column chromatography (5–20%  $\text{EtOAc}$ / hexanes) to afford the product as a clear, colorless oil (29 mg, 85  $\mu\text{mol}$ , 85% yield, > 20:1 dr trans/cis). The dr was determined based on the integration of the resonances attributed to the allylic hydrogens in the  $^1\text{H}$  NMR spectrum. The relative configuration was assigned based on NOE analysis. TLC  $R_f = 0.3$  (25%  $\text{EtOAc}$ / hexanes);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.74 (d,  $J = 8.2$  Hz, 2H), 7.32–7.23 (m, 6H), 7.17 (quin,  $J = 8.5$  Hz, 1H), 6.40 (d,  $J = 15.8$  Hz, 1H), 5.69 (dd,  $J = 15.8$  Hz, 8.8 Hz, 1H), 4.42 (t,  $J = 6.4$  Hz, 1H), 3.23–2.93 (m, 2H), 2.41 (s, 3H), 1.51 (q,  $J = 6.9$  Hz, 2H), 1.39–1.18 (m, 1H), 0.82–0.73 (m, 1H), 0.69 (dt,  $J = 8.7$  Hz, 4.9 Hz, 1H), 0.61 (dt,  $J = 8.2$  Hz, 4.1 Hz, 1H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  143.4, 137.5, 137.0, 133.2, 129.8 (2C), 128.6 (2C), 127.8, 127.2 (2C), 126.8, 125.6 (2C), 43.3, 33.8, 21.9, 21.6, 18.7, 14.0; IR (neat) 3284, 3026, 2926, 2254, 1651, 1598, 1448, 1324, 1160, 1094, 1042, 959, 908, 731, 694  $\text{cm}^{-1}$ ; HRMS (TOF MS ES+)  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{20}\text{H}_{24}\text{NO}_2\text{S}$  342.1528, found 342.1545.





***N*-(2-(*trans*-2-((*E*)-4-fluorostyryl)cyclopropyl)ethyl)-4-methylbenzenesulfonamide (3.22)**

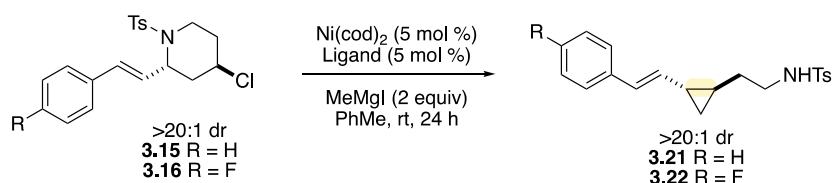
was prepared according to Method B. The following amounts of reagents were used: piperidine **3.16** (61 mg, 0.15 mmol, 1.0 equiv), Ni(cod)<sub>2</sub> (2.1 mg, 7.5 μmol, 5.0 mol %), SiMes·HCl (2.6 mg, 7.5 μmol, 5.0 mol %), PhMe (0.75 mL, 0.2 M in substrate), and methylmagnesium iodide (0.10 mL, 0.30 mmol, 2.0 equiv, 2.9 M solution in Et<sub>2</sub>O). Before purification a <sup>1</sup>H NMR yield of 71% was obtained. The residue was purified by column chromatography (5–25% EtOAc/ hexanes) to afford the product a clear, colorless oil (32 mg, 89 μmol, 61% yield, > 20:1 dr *trans*/*cis*). The dr was determined based on the integration attributed to the allylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **TLC** *R<sub>f</sub>* = 0.2 (25% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.75 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.25–7.20 (m, 2H), 6.96 (t, *J* = 8.7 Hz, 2H), 6.36 (d, *J* = 15.8 Hz, 1H), 5.61 (dd, *J* = 15.8 Hz, 8.8 Hz, 1H), 4.50 (t, *J* = 6.3 Hz, 1H), 3.05 (qd, *J* = 6.7 Hz, 2.0 Hz, 2H), 2.41 (s, 3H), 1.49 (qd, *J* = 6.9 Hz, 2.6 Hz, 2H), 1.28 (ddd, *J* = 12.9 Hz, 8.6 Hz, 4.2 Hz, 1H), 0.81–0.73 (m, 1H), 0.68 (dt, *J* = 8.3 Hz, 4.8 Hz, 1H), 0.61 (dt, *J* = 8.2 Hz, 5.1 Hz, 1H); **<sup>13</sup>C NMR** (125.8 MHz, CDCl<sub>3</sub>) δ 161.9, 143.6, 137.1, 133.8 (d, *J* = 245.5 Hz), 133.1 (d, *J* = 3.3 Hz), 129.9 (2C), 127.3 (2C), 127.1 (d, *J* = 7.9 Hz, 2C), 126.8, 115.5 (d, *J* = 21.7, 2C), 43.3, 33.9, 22.0, 21.7, 18.7, 14.1; **HRMS** (TOF MS ES+) *m/z* [M + H]<sup>+</sup> calcd for C<sub>20</sub>H<sub>23</sub>FNO<sub>2</sub>S 360.1433, found 360.1440.

**3.5.7 Evaluation of Various Ligands in the XEC Reaction of Vinylpiperidines**

Ligand evaluation was performed according to Method C. The following amounts of reagents were used: substrate **3.15** or **3.16** (39 mg, 0.10 mmol, 1.0 equiv), Ni(cod)<sub>2</sub> (1.4 mg, 5.0 μmol, 5.0 % mol%), methylmagnesium iodide (70. μL, 0.20 mmol, 2.0 equiv, 2.9 M in Et<sub>2</sub>O), and PhMe (0.5

mL). Ligands evaluated: *rac*-BINAP (3.1 mg, 5.0  $\mu$ mol, 5.0 mol %), BPhen (1.7 mg, 5.0  $\mu$ mol, 5.0 mol %), XantPhos (2.9 mg, 5.0  $\mu$ mol, 5.0 mol %), SiMes $\cdot$ BF<sub>4</sub> (2.0 mg, 5.0  $\mu$ mol, 5.0 mol %), PCy<sub>3</sub> (1.4 mg, 5.0  $\mu$ mol, 5.0 mol %), and SiMes $\cdot$ HCl (1.7 mg, 5.0  $\mu$ mol, 5.0 mol %). After 24 h, the reaction vials were removed from the glovebox, quenched with MeOH, filtered through a plug of silica gel eluting with Et<sub>2</sub>O, and concentrated in vacuo. Phenyltrimethylsilane (PhTMS; 8.6  $\mu$ L, 50.  $\mu$ mol) was added and the yield was determined by <sup>1</sup>H NMR based on comparison to PhTMS as internal standard. The results from this screen are tabulated in Table 3.5.

**Table 3.5 Ligand Screen for XEC Reaction of Vinylpiperidines**



Entry	R	Ligand	Yield (%) <sup>a</sup>
1	H	<i>rac</i> -BINAP	19
2	H	BPhen	95
3	H	XantPhos	90
4	H	SiMes $\cdot$ BF <sub>4</sub>	88
5	H	PCy <sub>3</sub>	73
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6	F	<i>rac</i> -BINAP	25
7	F	BPhen	53
8	F	SiMes $\cdot$ HCl	62
9	F	PCy <sub>3</sub>	68

<sup>a</sup>Yield determined by <sup>1</sup>H NMR based on the comparison to PhTMS as internal standard

### 3.5.8 Benchtop Reaction to Demonstrate Stability of (*R*-BINAP)NiCl<sub>2</sub>

This experiment was performed according to a modified version of Method A. On the benchtop, a flame dried 7-mL vial was charged with substrate **3.9** (78 mg, 0.20 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (7.5 mg, 1.0  $\mu$ mol, 5.0 mol %). The vial was evacuated and backfilled with N<sub>2</sub> (x 3). Then anhydrous PhMe (1.0 mL) was added followed by methylmagnesium iodide (0.22 mL, 0.60 mmol, 3.0 equiv, 2.8 M in Et<sub>2</sub>O) dropwise via a syringe. The reaction was allowed to stir for

48 h at rt. After 48 h, the reaction vials were removed from the glovebox, quenched with MeOH, filtered through a plug of silica gel eluting with Et<sub>2</sub>O, and concentrated in vacuo. Before purification, a <sup>1</sup>H NMR yield of 79% was determined based on comparison to PhTMS as internal standard. The residue was purified by column chromatography to yield the cyclopropane **3.19** as a white solid (53 mg, 0.15 mmol, 75% yield, >20:1 dr). Refer above for analytical data of the product.

### 3.5.9 General Procedures for the Synthesis of Piperidine Substrates

#### 3.5.9.1 Method C: Aza-Prins Reaction with *p*-TsOH·H<sub>2</sub>O and TiCl<sub>4</sub> for Piperidine Synthesis

This method is based on a procedure reported by van de Weghe.<sup>20</sup> To a flame-dried pressure tube equipped with a stir bar was added aryl aldehyde (1 equiv), homoallylic sulfonamide **3.2** (1 equiv), and CH<sub>2</sub>Cl<sub>2</sub> (0.1 M). To the stirring reaction mixture was added *p*TsOH·H<sub>2</sub>O (0.10 equiv) or HCl (0.10 equiv, 1.0 M) The reaction mixture was stirred open to air for an additional 15 min, and then TiCl<sub>4</sub> (1 equiv) was added. The pressure tube was capped, and the reaction mixture was allowed to stir at 60 °C for 7 days. The solution was cooled to room temperature, quenched with sat aq NaHCO<sub>3</sub>, and extracted with CH<sub>2</sub>Cl<sub>2</sub> (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, concentrated in vacuo, and purified by column chromatography. The allylic strain imparted by the sulfonyl group drives the aryl group to the axial position; therefore, only the trans diastereomer was isolated for most of the aza-Prins reactions.

Frequently, the desired Prins product was isolated as a mixture with unreacted aldehyde. To remove unreacted aldehyde from the desired product, the crude mixture was subjected to NaBH<sub>4</sub> reduction according to a modified procedure reported by Wang and Franzén.<sup>41</sup> The unpurified aza-Prins reaction mixture was concentrated and dissolved in MeOH. NaBH<sub>4</sub> (1.6 equiv

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<sup>41</sup> Wang, Y.; Franzén, R. *Synlett* **2012**, 23, 925–929.

relative to the remaining aldehyde as determined by  $^1\text{H}$  NMR integration) was added in one portion and the reaction stirred for 20 min at rt. The reaction mixture was then concentrated in vacuo to remove the MeOH. The resulting solid was dissolved in EtOAc and transferred to a separatory funnel. The organic layer was washed with water (x3) and brine, dried over  $\text{Na}_2\text{SO}_4$ , filtered, and concentrated in vacuo. Note: A CAPFE O-ring was used for the pressure tube cap.  $\text{CH}_2\text{Cl}_2$  will disintegrate Viton O-rings.

### 3.5.9.2 Method D: $\text{AlCl}_3$ -Catalyzed Aza-Prins Reaction for Piperidine Synthesis

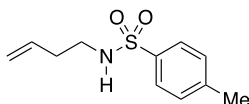
This method is a modified version of a procedure reported by Li.<sup>21</sup> In a glovebox,  $\text{AlCl}_3$  (33 mg, 0.25 mmol, 0.050 equiv) was added to a flame-dried pressure tube equipped with a stir bar. The tube was sealed with a septum, brought out of the glovebox and placed under an  $\text{N}_2$  atmosphere. Using a syringe, the sealed tube was then charged with the 2-furaldehyde **3.11** (0.42 mL, 5.0 mmol, 1.0 equiv), homoallylic sulfonamide **3.2** (0.91 mL, 5.0 mmol, 1.0 equiv), and  $\text{CH}_2\text{Cl}_2$  (25 mL, 0.20 M in aldehyde). Then freshly distilled  $\text{TMSCl}$  (1.3 mL, 10. mmol, 2.0 equiv) was added dropwise. The septum was then removed and the pressure tube was sealed with a Teflon screw cap, heated to 60 °C, and was allowed to stir for 48 h. The reaction mixture was cooled to room temperature and allowed to stir for an additional 1 h. Then excess  $\text{CH}_2\text{Cl}_2$  was added, and the reaction mixture was quenched with  $\text{H}_2\text{O}$ . The two phases were separated, and the aqueous layer was extracted with  $\text{CH}_2\text{Cl}_2$  (x3). The combined organic layers were dried over  $\text{Na}_2\text{SO}_4$ , filtered, concentrated in vacuo, and purified by column chromatography.

### 3.5.9.3 Method E: : $\text{FeCl}_3/\text{BmimPF}_6$ -Mediated Aza-Prins Reaction

This method was adapted from a procedure reported by Iwamoto.<sup>22</sup> In a glovebox,  $\text{FeCl}_3$  (1.5 equiv) was added to a flame-dried round-bottom flask equipped with a stir bar. The flask was sealed with a septum and removed from the glovebox. Benzotrifluoride (0.10 M in homoallylic

sulfonamide) was added to the flask, followed by dropwise addition of BmimPF<sub>6</sub> (1.5 equiv). In a separate flask, a solution of aldehyde (1.5 equiv) and homoallylic sulfonamide **3.2** (1.0 equiv) in benzotrifluoride (0.10 M in aldehyde) was prepared. Using a syringe, the solution of aldehyde and sulfonamide was added to the flask containing FeCl<sub>3</sub>. The flask was fitted with a reflux condenser and N<sub>2</sub> inlet. The solution was heated to reflux in an oil bath and allowed to stir for 24 h. The reaction mixture was cooled to room temperature and was then quenched with H<sub>2</sub>O. The layers were separated, and the aqueous layer was extracted with Et<sub>2</sub>O (x 2). The combined organic layers were washed sequentially with sat aq Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, sat aq NaHCO<sub>3</sub>, and brine. The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, concentrated in vacuo, and purified by column chromatography.

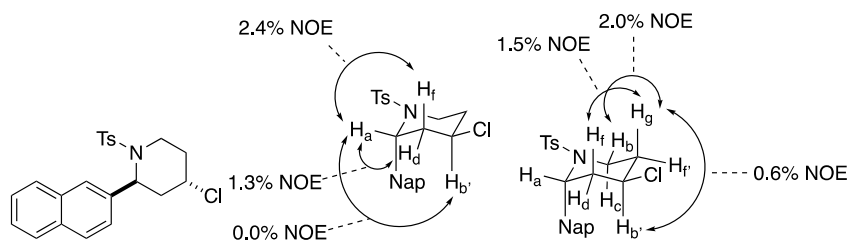
### 3.5.10 Characterization Data for Piperidine Substrates



**N-(but-3-en-1-yl)-4-methylbenzenesulfonamide (3.2)** was synthesized according to a procedure reported by Jiang.<sup>42</sup> To a flame-dried flask equipped with a stir bar was added 4-bromo-1-butene (4.1 mL, 40. mmol, 1.0 equiv), *p*-toluenesulfonamide (6.8 g, 40. mmol, 1.0 equiv), K<sub>2</sub>CO<sub>3</sub> (6.6 g, 48 mmol, 1.2 equiv), and MeCN (160 mL). The mixture was heated to 60 °C in an oil bath and allowed to stir for 3 d. The reaction mixture was quenched with saturated NH<sub>4</sub>Cl (100 mL) and extracted with EtOAc (3 x 50 mL). The combined organic layers were washed with H<sub>2</sub>O (50 mL) and brine (50 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, and was concentrated in vacuo. The residue was purified by column chromatography (5–25% EtOAc/hexanes) to afford the product as a clear, colorless oil (5.4 g, 24 mmol, 60 % yield). Analytical data is consistent with literature values.<sup>42</sup> **<sup>1</sup>H NMR** (400

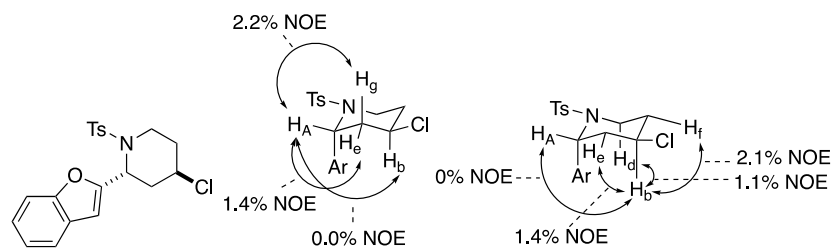
<sup>42</sup> Huang, J.; Zheng, J.; Wu, W.; Li, J.; Ma, J.; Ren, Y.; Jiang, H. *J. Org. Chem.* **2017**, *82*, 8191–8198.

MHz, CDCl<sub>3</sub>)  $\delta$  7.76 (d,  $J = 8.2$ , 2H), 7.30 (d,  $J = 8.1$ , 2H), 5.63 (ddt,  $J = 17.1$ , 10.4, 6.8, 1H), 5.11 (br s, 1H), 5.02–4.93 (m, 2H), 2.99 (q,  $J = 6.7$ , 2H), 2.41 (s, 3H), 2.20 (q,  $J = 6.9$ , 2H).

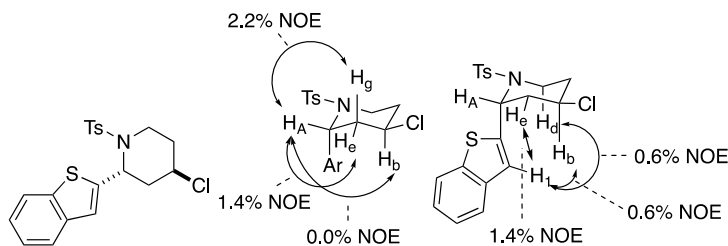


***trans*-4-Chloro-2-(naphthalen-2-yl)-1-tosylpiperidine (3.8)**<sup>43</sup> was synthesized according to Method C. The following amounts of reagents were used: 2-naphthaldehyde **3.33** (1.61 g, 10.3 mmol, 1.00 equiv), homoallylic sulfonamide **3.2** (2.63 g, 10.3 mmol, 1.00 equiv), *p*-TsOH·H<sub>2</sub>O (196 mg, 1.03 mmol, 10.0 mol %), TiCl<sub>4</sub> (1.13 mL, 10.3 mmol, 1.00 equiv), and CH<sub>2</sub>Cl<sub>2</sub> (103 mL, 0.100 M). The reaction mixture was allowed to stir at 60 °C for 48 h. The residue was purified by column chromatography (0–10% EtOAc/hexanes) to afford the product as a white solid (1.68 g, 4.20 mmol, 41% yield, > 20:1 dr *trans*:*cis*). The dr was determined based on the integration of the resonances attributed to the benzylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **mp** 136–138 °C; **TLC**  $R_f = 0.3$  (10% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.84–7.71 (m, 5H), 7.65 (s, 1H), 7.50–7.43 (m, 3H), 7.33 (d,  $J = 8.0$  Hz, 2H), 5.54 (as, 1H), 4.05–3.94 (m, 2H), 3.10 (at,  $J = 13.8$  Hz, 1H), 2.86 (ad,  $J = 14.1$  Hz, 1H), 2.45 (s, 3H), 2.01–1.90 (m, 2H), 1.62 (qd,  $J = 12.7$  Hz, 4.8 Hz, 1H); **<sup>13</sup>C NMR** (125.5 MHz, CDCl<sub>3</sub>)  $\delta$  143.8, 138.2, 134.9, 133.4, 132.7, 130.1 (2C), 129.0, 128.1, 127.7, 127.2 (2C), 126.6, 126.4, 125.7, 124.6, 56.4, 53.0, 41.7, 37.9, 35.4, 21.7; **HRMS** (TOF MS ES+)  $m/z$  [M + Na]<sup>+</sup> calcd for C<sub>22</sub>H<sub>22</sub>ClNO<sub>2</sub>SNa 422.0957, found 422.0938.

<sup>43</sup> This substrate was synthesized by Dr. Erika Lucas.

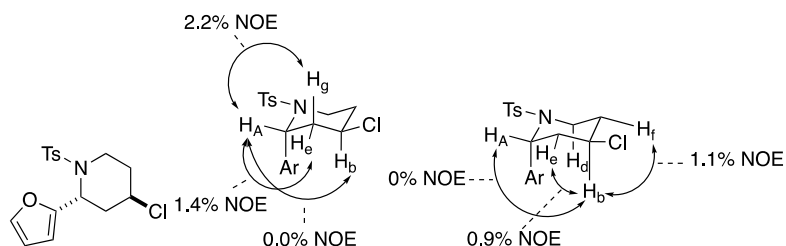


***trans*-2-(Benzofuran-2-yl)-4-chloro-1-tosylpiperidine (3.9)** was prepared according to a modified version of Method C where HCl was used instead of *p*-TsOH·H<sub>2</sub>O. The following amounts of reagents were used: benzo[*b*]furan-2-carboxaldehyde **3.34** (0.12 mL, 1.0 mmol, 1.0 equiv), homoallylic sulfonamide **3.2** (0.18 mL, 1.0 mmol, 1.0 equiv), HCl (0.10 mL, 0.10 mmol, 10. mol %, 1.0 M aqueous solution), TiCl<sub>4</sub> (0.11 mL, 1.0 mmol, 1.0 equiv), CH<sub>2</sub>Cl<sub>2</sub> (10. mL, 0.10 M in aldehyde). The residue was purified by column chromatography (5–25% EtOAc/hexanes) to afford the product as a tan solid (190 mg, 0.48 mmol, 48% yield, > 20:1 dr *trans*/*cis*). The dr was based on the integration of the resonances attributed to the benzylic protons in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **mp** 89–91 °C; **TLC** **R<sub>f</sub>** = 0.8 (25% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.62 (d, *J* = 8.2 Hz, 2H), 7.47 (d, *J* = 8.2 Hz, 1H), 7.36–7.17 (m, 3H), 7.14 (d, *J* = 8.2 Hz, 2H), 6.50 (s, 1H), 5.49 (ad, *J* = 5.6 Hz, 1H), 4.16 (att, *J* = 12.0 Hz, 4.2 Hz, 1H), 3.93 (ad, *J* = 13.5 Hz, 1H), 3.26 (atd, *J* = 13.4 Hz, 2.7 Hz, 1H), 2.66 (ad, *J* = 13.5 Hz, 1H), 2.32 (s, 3H), 2.13 (atd, *J* = 12.6 Hz, 5.6 Hz, 2H), 1.81 (aqd, *J* = 12.6 Hz, 4.8 Hz, 1H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (125.8 MHz, CDCl<sub>3</sub>) δ 154.8, 154.5, 143.6, 136.8, 129.6 (2C), 127.9, 127.2 (2C), 124.5, 123.2, 121.1, 111.2, 105.4, 53.0, 52.0, 42.4, 38.9, 35.7, 21.6.; **IR** (neat) 2952, 2868, 1596, 1454, 1375, 1256, 1155, 1090, 741 cm<sup>-1</sup>; **HRMS** (TOF MS ES<sup>+</sup>) *m/z* [M + Na]<sup>+</sup> calcd for C<sub>20</sub>H<sub>20</sub>ClNO<sub>3</sub>SNa 412.0750, found 412.0756.



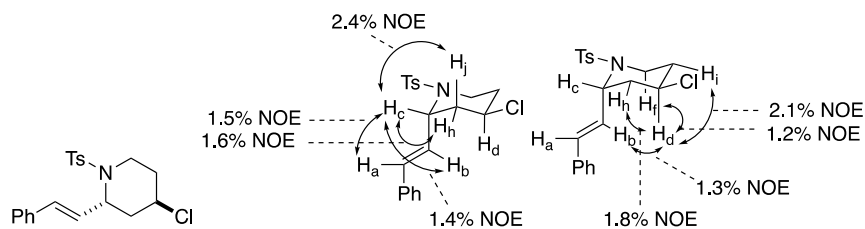
***trans*-2-(Benzo[b]thiophene-2-yl)-4-chloro-1-tosylpiperidine (3.10)** was prepared according to a modified version of method C where HCl was used instead of *p*-TsOH·H<sub>2</sub>O. The following amounts of reagents were used: benzo[b]thiophene-2-carbaldehyde **3.35** (810 mg, 5.0 mmol, 1.0 equiv), homoallylic sulfonamide **3.2** (0.91 mL, 5.0 mmol, 1.0 equiv), HCl (0.10 mL, 0.10 mmol, 10. mol %, 5.0 M aqueous solution), TiCl<sub>4</sub> (0.50 mL, 5.0 mmol, 1.0 equiv), CH<sub>2</sub>Cl<sub>2</sub> (50. mL, 0.10 M). The residue was purified by column chromatography (5–25% EtOAc/hexanes) to afford the product as a pale yellow solid (0.52 g, 1.3 mmol, 26% yield, > 20:1 dr *trans*/*cis*). The dr was based on the integration of the resonances attributed to the benzylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **mp** 120–123 °C; **TLC** *R<sub>f</sub>* = 0.7 (25% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.77 (d, *J* = 8.1 Hz, 2H), 7.74 (d, *J* = 8.0 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.37–7.27 (m, 4H), 7.11 (s, 1H), 5.64 (d, *J* = 5.4 Hz, 1H), 4.13 (tt, *J* = 12.1 Hz, 4.2 Hz, 1H), 3.96 (dt, *J* = 14.4 Hz, 2.4 Hz, 1H), 3.22 (td, *J* = 14.0 Hz, 2.6 Hz, 1H), 2.67 (dt, *J* = 13.6 Hz, 1.9 Hz, 1H), 2.43 (s, 3H), 2.09 (td, *J* = 12.9 Hz, 5.8 Hz, 1H), 2.00 (add, *J* = 12.9 Hz, 2.9 Hz, 1H), 1.68 (qd, *J* = 12.8 Hz, 4.7 Hz, 1H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (125.8 MHz, CDCl<sub>3</sub>) δ 144.0, 143.4, 139.8, 139.8, 137.5, 130.0 (2C), 127.3 (2C), 124.7, 124.6, 123.6, 122.4, 122.3, 54.3, 52.8, 41.7, 39.3, 35.3, 21.7; **IR** (neat) 3104, 2928, 1238, 1154, 1056, 751 cm<sup>-1</sup>; **HRMS** (TOF MS ES +) *m/z* [M + Na]<sup>+</sup> calcd for C<sub>20</sub>H<sub>20</sub>ClNO<sub>2</sub>S<sub>2</sub>Na 428.0522, found 428.0541.



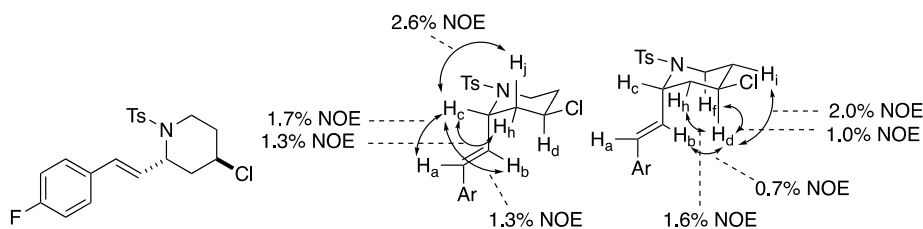


**trans-4-Chloro-2-(furan-2-yl)-1-tosylpiperidine (3.12)** was prepared according to Method D.

The following amount of reagents were used: the 2-furaldehyde **3.11** (0.42 mL, 5.0 mmol, 1.0 equiv), homoallylic sulfonamide **3.2** (0.91 mL, 5.0 mmol, 1.0 equiv),  $\text{AlCl}_3$  (33 mg, 0.25 mmol, 0.050 equiv),  $\text{TMSCl}$  (1.3 mL, 10. mmol, 2.0 equiv), and  $\text{CH}_2\text{Cl}_2$  (25 mL, 0.20 M). The residue was purified by column chromatography (5–25% EtOAc/hexanes) to yield product as a white solid (388 mg, 1.14 mmol, 23% yield, > 20:1 dr trans/cis). The dr was based on the integration of the resonances attributed to the benzylic hydrogens in the  $^1\text{H}$  NMR spectrum. The relative configuration was assigned based on NOE analysis. **mp** 65–67 °C; **TLC**  $R_f$  = 0.6 (25% EtOAc/hexanes);  **$^1\text{H}$  NMR** (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.61 (d,  $J$  = 8.3 Hz, 2H), 7.23 (d,  $J$  = 8.3 Hz, 3H), 6.26 (dd,  $J$  = 3.3 Hz, 1.9 Hz, 1H), 6.12 (dd,  $J$  = 3.5 Hz, 1.0 Hz, 1H), 5.36 (d,  $J$  = 5.8 Hz, 1H), 4.15 (tt,  $J$  = 12.1 Hz, 4.2 Hz, 1H), 3.84 (dt,  $J$  = 13.8 Hz, 2.4 Hz, 1H), 3.12 (td,  $J$  = 13.3 Hz, 2.7 Hz, 1H), 2.54 (ddt,  $J$  = 13.2 Hz, 4.2 Hz, 2.2 Hz, 1H), 2.41 (s, 3H), 2.15–2.08 (m, 1H), 2.04 (dt,  $J$  = 12.7 Hz, 6.3 Hz, 1H), 1.76 (qd,  $J$  = 12.7 Hz, 4.8 Hz, 1H);  **$^{13}\text{C}$  NMR** (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  152.1, 143.5, 142.3, 137.1, 129.7 (2C), 127.3 (2C), 110.5, 108.4, 53.2, 51.6, 42.1, 39.0, 35.7, 21.7. **IR** (neat) 2959, 2928, 1597, 1495, 1339, 1304, 1157, 1092, 854, 728, 655  $\text{cm}^{-1}$ ; **HRMS** (TOF MS ES+)  $m/z$   $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{16}\text{H}_{18}\text{ClINO}_3\text{SNa}$  362.0594, found 362.0581.



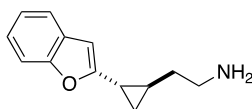
***trans*-4-Chloro-2-((*E*)-styryl)-1-tosylpiperidine (3.15)** was prepared according to Method E. The following amounts of reagents were used: *trans*-cinnamaldehyde **3.13** (0.18 mL, 1.5 mmol, 1.5 equiv), homoallylic sulfonamide **3.2** (0.18 mL, 1.0 mmol, 1.0 equiv), FeCl<sub>3</sub> (0.24 g, 1.5 mmol, 1.5 equiv), BmimPF<sub>6</sub> (0.30 mL, 1.5 mmol, 1.5 equiv), and benzotrifluoride (10. mL, 0.10 M). The residue was purified by column chromatography (5–25% EtOAc/hexanes) to afford the product as a white solid (150 mg, 0.41 mmol, 54% yield, > 20:1 dr *trans*/*cis*). The dr was based on the integration of the resonances attributed to allylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **mp** 127–129 °C; **TLC** R<sub>f</sub> = 0.7 (25% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.68 (d, *J* = 8.1 Hz, 2H), 7.28 (t, *J* = 7.2 Hz, 2H), 7.26–7.21 (m, 3H), 7.18 (d, *J* = 7.3 Hz, 2H), 6.41 (d, *J* = 16.1 Hz, 1H), 5.89 (dd, *J* = 16.1 Hz, 5.7 Hz, 1H), 4.86 (s, 1H), 4.06 (tt, *J* = 11.7 Hz, 4.1 Hz, 1H), 3.87 (dt, *J* = 14.2 Hz, 3.9 Hz, 1H), 3.11 (td, *J* = 13.1 Hz, 2.8 Hz, 1H), 2.37 (s, 3H), 2.28 (dt, *J* = 13.2 Hz, 4.3 Hz, 1H), 2.10 (d, *J* = 12.5 Hz, 1H), 1.99 (td, *J* = 12.5 Hz, 5.3 Hz, 1H), 1.76 (qd, *J* = 12.3 Hz, 4.7 Hz, 1H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (125.7 MHz, CDCl<sub>3</sub>) δ 143.6, 137.3, 136.1, 133.0, 129.9 (2C), 128.7 (2C), 128.2, 127.5 (2C), 126.5 (2C) 125.3, 55.7, 53.2, 41.5, 40.7, 35.8, 21.6; **IR** (neat) 2923, 2862, 1595, 1494, 1446, 1330, 1153, 1089, 935, 723 cm<sup>-1</sup>; **HRMS** (TOF MS ES<sup>+</sup>) *m/z* [M + Na]<sup>+</sup> calcd for C<sub>20</sub>H<sub>22</sub>ClNO<sub>2</sub>SNa 398.0957, found 398.0951.



***trans*-4-Chloro-2-((*E*)-4-fluorostyryl)-1-tosylpiperidine (3.16)** was prepared accord to Method E. The following amounts of reagents were used: (*E*)-4-fluorocinnamaldehyde **3.14** (0.40 mL, 3.0

mmol, 1.5 equiv), homoallylic sulfonamide **3.2** (0.37 mL, 2.0 mmol, 1.0 equiv), FeCl<sub>3</sub> (0.48 g, 3.0 mmol, 1.5 equiv), BmimPF<sub>6</sub> (0.62 mL, 3.0 mmol, 1.5 equiv) and benzotrifluoride (20. mL, 0.10 M). The residue was purified by column chromatography (5–25% EtOAc/ hexanes) to afford the product as a yellow oil (390 mg, 1.0 mmol, 50% yield, > 20:1 dr trans:cis). The dr was based on the integration of the resonances attributed to allylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **TLC R<sub>f</sub>** = 0.6 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.67 (d, *J* = 8.3 Hz, 2H), 7.23 (d, *J* = 8.3 Hz, 2H), 7.20–7.10 (m, 2H), 6.96 (t, *J* = 8.6 Hz, 2H), 6.38 (dd, *J* = 16.0 Hz, 1.7 Hz, 1H), 5.83 (dd, *J* = 16.0 Hz, 5.8 Hz, 1H), 4.82 (s, 1H), 4.05 (tt, *J* = 11.6 Hz, 4.1 Hz, 1H), 3.85 (dt, *J* = 13.6 Hz, 3.2 Hz, 1H), 3.11 (td, *J* = 12.9 Hz, 2.8 Hz, 1H), 2.36 (s, 3H), 2.25 (d, *J* = 13.1 Hz, 1H), 2.09 (dd, *J* = 12.8 Hz, 2.2 Hz, 1H), 1.96 (td, *J* = 12.4 Hz, 5.5 Hz, 1H), 1.73 (qd, *J* = 12.2 Hz, 4.6 Hz, 1H); **<sup>13</sup>C NMR** (125.7 MHz, CDCl<sub>3</sub>) δ 162.5 (d, *J* = 247.8 Hz), 143.6, 137.1, 132.2 (d, *J* = 3.7 Hz), 131.7, 129.8 (2C), 128.1 (d, *J* = 7.9 Hz, 2C), 127.3 (2C), 125.0 (d, *J* = 2.3 Hz), 115.5 (d, *J* = 21.7 Hz, 2C), 55.6, 53.1, 41.4, 40.6, 35.6, 21.5; **HRMS** (TOF MS ES+) *m/z* [M + Na]<sup>+</sup> calcd for C<sub>20</sub>H<sub>21</sub>ClFNO<sub>2</sub>SNa 416.0863, found 416.0872.

### 3.5.11 Characterization Data for Derivatized Products

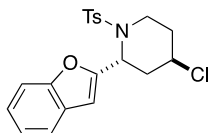


**2-(trans-2-(Benzofuran-2-yl)cyclopropyl)ethan-1-amine (3.33)**. was prepared according to a procedure reported by Hilmersson.<sup>44</sup> To a flame-dried Schlenk flask equipped with a stir bar was added cyclopropane **3.18** (67 mg, 0.20 mmol, 1.0 equiv). The flask was sealed with a septum and placed under an atmosphere of N<sub>2</sub>. Using a syringe, the flask was then charged with SmI<sub>2</sub> (54 mL,

<sup>44</sup> Ankner, T.; Hilmersson, G. *Org. Lett.* **2009**, *11*, 503–506

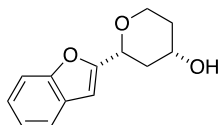
2.0 mmol, 10. equiv, 40. mM in THF),<sup>45</sup> followed by H<sub>2</sub>O (0.88 mL, 49 mmol, 240 equiv) and pyrrolidine (2.64 mL, 32 mmol, 160 equiv). The solution became white upon addition of the amine. The reaction mixture was diluted with CH<sub>2</sub>Cl<sub>2</sub> (50 mL) and treated with a solution of potassium sodium tartrate (50 mL, 10% w/v) and K<sub>2</sub>CO<sub>3</sub> (50 mL, 10% w/v). The aqueous layer was extracted with CH<sub>2</sub>Cl<sub>2</sub> (2 x 50 mL). The combined organic layers were washed with brine (50 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated in vacuo. The residue was purified by column chromatography (0–30% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) to afford the product as a light yellow solid (29 mg, 0.14 mmol, 72% yield, >20:1 dr trans/cis). The dr was based on the integration of the resonances attributed to allylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **mp** 141–143 °C; **TLC** R<sub>f</sub> = 0.3 (10% MeOH/CH<sub>2</sub>Cl<sub>2</sub>, CAM stain); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.40 (dd, *J* = 6.4 Hz, 2.3 Hz, 1H), 7.33 (d, *J* = 7.2 Hz, 1H), 7.13 (tt, *J* = 7.3 Hz, 5.6 Hz, 2H), 6.31 (s, 1H), 3.13 (t, *J* = 7.6 Hz, 2H), 1.78 (dq, *J* = 14.8 Hz, 7.6 Hz, 1H), 1.85 (dt, *J* = 9.2 Hz, 4.9 Hz, 1H), 1.78 (dq, *J* = 14.8 Hz, 7.6 Hz, 1H), 1.39–1.29 (m, 1H), 1.19 (dt, *J* = 9.1 Hz, 5.0 Hz, 1H), 0.88 (dt, *J* = 8.8 Hz, 5.2 Hz, 1H); **<sup>13</sup>C NMR** (125.4 MHz, CDCl<sub>3</sub>) δ 158.9, 154.4, 129.1, 123.3, 122.7, 120.2, 110.8, 100.9, 40.0, 31.8, 18.3, 16.6, 14.0; **HRMS** (TOF MS ES+) *m/z* [M + H]<sup>+</sup> calcd for C<sub>13</sub>H<sub>16</sub>NO 202.1232, found 202.1231.

### 3.5.12 Synthesis and Characterization for the Competition Experiment



**trans-2-(benzofuran-2-yl)-4-chloro-1-tosylpiperidine (3.9)** refer above for analytical data.

<sup>45</sup> SmI<sub>2</sub> was freshly prepared from Sm metal and diiodoethane. For the preparation, see: Szostak, M.; Spain, M.; Procter, D. J. *J. Org. Chem.* **2012**, *77*, 3049–3059.

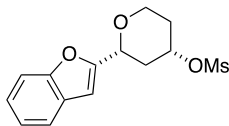


***trans*-2-(Benzofuran-2-yl)tetrahydro-2H-pyran-4-ol (3.36)**<sup>46</sup> was prepared according to a procedure reported by Yadav.<sup>47</sup> To a flame-dried round-bottom flask under N<sub>2</sub> equipped with a stir bar and septum were added benzo[*b*]furan-2-carboxaldehyde **3.34** (1.8 mL, 15 mmol, 1.0 equiv) and CH<sub>2</sub>Cl<sub>2</sub> (63 mL, 0.24 M in aldehyde). Trifluoroacetic acid (11.6 mL, 150. mmol, 10.0 equiv) was slowly added to the flask. The reaction mixture was allowed to stir at room temperature for 3 h, then saturated aq. NaHCO<sub>3</sub> (60 mL) was slowly added. Et<sub>3</sub>N was then added to the flask until the pH reached >7. The aqueous layer was extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 x 60 mL), and then the combined organic layers were washed with brine (60 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo. The residue was then redissolved in MeOH (50. mL, 0.30 M in aldehyde), K<sub>2</sub>CO<sub>3</sub> (7.1 g, 51 mmol, 3.4 equiv) was added, and the reaction mixture was allowed to stir for 30 m at room temperature. The solution was concentrated in vacuo, and then H<sub>2</sub>O (60 mL) was added to the residue. The solution was extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 x 60 mL), then the combined organic layers were washed with brine (60 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo. The residue was purified by column chromatography to afford the title compound as an off-white solid (1.1 g, 5.0 mmol, 33% yield, >20:1 dr *cis/trans*). The dr was determined based on the integration of the resonances attributed to the benzylic hydrogens in the <sup>1</sup>H NMR spectrum. Analytical data is consistent with literature values.<sup>9a</sup> **TLC R<sub>f</sub>** = 0.7 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.55 (d, *J* = 7.7 Hz, 1H), 7.47 (d, *J* = 8.2 Hz, 1H), 7.29–7.25 (m, 1H), 7.21 (at, *J* = 7.3 Hz, 1H), 6.65 (s, 1H), 4.53 (dd, *J* = 11.7 Hz, 1.3 Hz, 1H), 4.19 (add, *J* = 12.0 Hz,

<sup>46</sup> This substrate was synthesized by Dr. Erika Lucas.

<sup>47</sup> Sabitha, G.; Reddy, N. M.; Prasad, M. N.; Yadav, J. S. *Helv. Chim. Acta* **2009**, *92*, 967.

4.9 Hz, 1H), 3.99–3.91 (m, 1H), 3.63 (atd,  $J = 12.5$  Hz, 2.0 Hz, 1H), 2.33 (adquin,  $J = 12.4$  Hz, 2.2 Hz, 1H), 1.99 (adquin,  $J = 12.8$  Hz, 2.5 Hz, 1H), 1.85 (aq,  $J = 11.9$  Hz, 1H), 1.76 (d,  $J = 3.8$  Hz, 1H), 1.69 (aqd,  $J = 12.6$  Hz, 5.1 Hz, 1H).

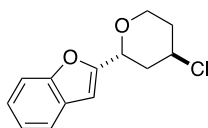


***trans*-2-(Benzofuran-2-yl)tetrahydro-2H-pyran-4-yl methanesulfonate (3.37)**<sup>48</sup> was prepared according to a procedure reported by Wallace.<sup>49</sup> To a flame-dried flask equipped with a stir bar was added **3.36** (1.1 g, 5.0 mmol, 1.0 equiv). The flask was evacuated, backfilled with N<sub>2</sub>, and capped with a septum. CH<sub>2</sub>Cl<sub>2</sub> (83 mL, 0.060 M in substrate) was added to the flask, and the reaction mixture was cooled to 0 °C. Et<sub>3</sub>N (1.04 mL, 7.44 mmol, 1.50 equiv) was added, and the reaction mixture was allowed to stir at 0 °C for 10 min. Methanesulfonyl chloride (0.46 mL, 6.0 mmol, 1.2 equiv) was slowly added to the flask, and the reaction mixture was allowed to stir at room temperature for 3 h. The reaction mixture was quenched with saturated aq. NaHCO<sub>3</sub> (80 mL) and then extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 80 mL). The combined organic layers were washed with brine (80 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo. The residue was purified by column chromatography (0–40% EtOAc/hexanes) to afford the title compound as a yellow oil (1.08 g, 3.66 mmol, 74% yield, >20:1 dr cis/trans). The dr was determined based on the integration of the resonances attributed to the benzylic hydrogens in the <sup>1</sup>H NMR spectrum. Analytical data is consistent with literature values.<sup>9a</sup> **TLC R<sub>f</sub>** = 0.3 (30% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.55 (d,  $J = 7.7$  Hz, 1H), 7.47 (d,  $J = 8.3$  Hz, 1H), 7.27 (at,  $J = 7.7$  Hz, 1H), 7.21 (t,  $J = 7.7$  Hz, 1H), 6.66 (s, 1H), 5.00–4.91 (m, 1H), 4.58 (add,  $J = 11.7$  Hz, 1.6 Hz, 1H), 4.20 (add,  $J =$

<sup>48</sup> This substrate was synthesized by Dr. Erika Lucas.

<sup>49</sup> Crombie, B. S.; Smith, C.; Varnavas, C. Z.; Wallace, T. W. *J. Chem. Soc., Perkin Trans.* **2001**, 1, 206–215.

12.1 Hz, 1.2 Hz, 1H), 3.66 (atd,  $J = 12.5$  Hz, 1.8 Hz, 1H), 3.05 (s, 3H), 2.55–2.48 (m, 1H), 2.19–2.11 (m, 2H), 1.97 (aqd,  $J = 12.5$  Hz, 5.0 Hz, 1H).



**2-(trans-4-Chlorotetrahydro-2H-pyran-2-yl)benzofuran (3.24)**<sup>50</sup> was prepared according to a modified procedure reported by Moyeux.<sup>51</sup> To a round-bottom flask equipped with a stir bar was added mesylate **3.37** (1.1 g, 3.7 mmol, 1.0 equiv) and tetrabutylammonium chloride (2.0 g, 7.3 mmol, 2.0 equiv). The flask was equipped with a reflux condenser and then evacuated and backfilled with N<sub>2</sub>. The condenser was capped with a septum, THF (50. mM in tosylate) was added, and the reaction mixture was refluxed in an oil bath for 18 h. The reaction mixture was quenched with H<sub>2</sub>O and extracted with EtOAc (x 2). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo. The residue was purified by column chromatography to afford the title compound as a white solid (0.78 g, 3.3 mmol, 90% yield, 10:1 dr trans/cis). The dr was determined based on the integration of the resonances attributed to the benzylic hydrogens in the <sup>1</sup>H NMR spectrum. Analytical data is consistent with literature values.<sup>9a</sup> **TLC Rf** = 0.5 (5% EtOAc/ hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.54 (d,  $J = 7.7$  Hz, 1H), 7.47 (d,  $J = 8.2$  Hz, 1H), 7.27 (at,  $J = 7.6$  Hz, 1H), 7.21 (at,  $J = 7.6$  Hz, 1H), 6.65 (s, 1H), 5.10 (dd,  $J = 10.8$  Hz, 2.3 Hz, 1H), 4.69 (aquin,  $J = 3.6$  Hz, 1H), 4.16 (atd,  $J = 11.7$  Hz, 2.2 Hz, 1H), 4.02–3.96 (m, 1H), 2.48–2.39 (m, 1H), 2.28–2.18 (m, 2H), 1.94–1.88 (m, 1H).

<sup>50</sup> Substrate **3.24** was synthesized by Dr. Erika Lucas.

<sup>51</sup> Cahiez, G.; Lefevre, N.; Poizat, M.; Moyeux, A. *Synthesis* **2013**, 45, 231–236.

## Nickel-Catalyzed Domino Cross-Electrophile Coupling Dicarbofunctionalization Reaction to Afford Tetrasubstituted Vinylcyclopropanes

### 4.1 Introduction

Domino reactions provide powerful strategies to build molecular complexity by forging multiple C–C bonds and stereogenic centers in a single reaction flask.<sup>1,2</sup> Development of these reactions typically builds on mechanistic understanding of discrete elementary steps, established in simpler transformations. Incorporation of cross-electrophile coupling (XEC) reactions into domino sequences is in its early stages.<sup>3</sup> Recent efforts have established nickel-catalyzed conjunctive XEC reactions where two organohalides are utilized to functionalize an alkene in the presence of a reducing agent (Scheme 4.1a).<sup>4,5,6</sup> These reactions intersect traditional XEC reaction mechanisms because they involve two oxidative addition events and, frequently, alkyl radical

<sup>1</sup> Portions of this chapter have been published in *ACS Catalysis*, see: Hewitt, K. A.; Xie, P.-P.; Thane, T. A.; Hirbawi, N.; Zhang, S.-Q.; Matus, A. C.; Lucas, E. L.; Hong, X.; Jarvo, E. R. *ACS Catal.* **2021**, *11*, 14369–14380.

<sup>2</sup> For reviews of transition-metal catalyzed domino reactions see: (a) Tietze, L. F. *Chem. Rev.* **1996**, *96*, 115–136. (b) Ikeda, S.-I. *Acc. Chem. Res.* **2000**, *33*, 511–519. (c) Montgomery, J. *Angew. Chem. Int. Ed.* **2004**, *43*, 3890–3908. (d) Nicolaou, K. C.; Edmonds, D. J.; Bulger, P. G. *Angew. Chem. Int. Ed.* **2006**, *45*, 7134–7186. (e) Pellissier, H. *Chem. Rev.* **2013**, *113*, 442–524. (f) Tietze, L. F. *Domino Reactions: Concepts for Efficient Organic Synthesis*. Wiley-VCH: Weinheim, **2014**.

<sup>3</sup> For reviews of XEC reactions, see: (a) Knappke, C. E. I.; Grupe, S.; Gärtner, D.; Corpet, M.; Gosmini, C.; Jacobi von Wangelin, A. *Chem. Eur. J.* **2014**, *20*, 6828–6842. (b) Goldfogel, M. J.; Huang, L.; Weix, D. J. *Cross-Electrophile Coupling: Principles and New Reactions*. In *Nickel Catalysis in Organic Synthesis*; Ogoshi, S., Ed.; Wiley, **2020**; pp 183–222. (c) Wang, X.; Dai, Y.; Gong, H. *Top. Curr. Chem. (Z)* **2016**, *374*, 61–89. (d) Lucas, E. L.; Jarvo, E. R. *Nat. Rev. Chem.* **2017**, *1*, 0065. (e) Poremba, K. E.; Dibrell, S. E.; Reisman, S. E. *ACS Catal.* **2020**, *10*, 8237–8246. (f) Campeau, L.-C.; Hazari, N. *Organometallics* **2019**, *38*, 3–35. (g) Hewitt, K. A.; Lin, P. C.; Raffiman, E. T. A.; Jarvo, E. R. C–C Bond Formation via Cross-Electrophile Coupling Reactions. *Comprehensive Organometallic Chemistry IV*. **2021**. 10.1016/B978-0-12-820206-7.00092-5

<sup>4</sup> For recent reviews on conjunctive XEC reactions see: (a) Qi, X.; Diao, T. *ACS Catal.* **2020**, *10*, 8542–8556. (b) Derosa, J.; Apolinar, O.; Kang, T.; Tran, V. T.; Engle, K. M. *Chem. Sci.* **2020**, *11*, 4287–4296.

<sup>5</sup> For recent reviews on nickel-catalyzed conjunctive XC reactions see: (a) Derosa, J.; Tran, V. T.; van der Puyl, V. A.; Engle, K. M. *Aldrichimica Acta*. **2018**, *51*, 21–32. (b) Dhungana, R. K.; KC, S.; Basnet, P.; Giri, R. *Chem. Rec.* **2018**, *18*, 1314–1340. (c) Luo, Y.-C. Xu, C.; Zhang, X. *Chin. J. Chem.* **2020**, *38*, 1371–1394. For lead examples with alkynes: (d) Terao, H.; Bando, F.; Kambe, N. *Chem. Commun.* **2009**, 7336–7338. (e) Xue, F.; Zho, J.; Hor, T. S. A.; Hayashi, T. *J. Am. Chem. Soc.* **2015**, *137*, 3189–3192. (f) Wickham, L. M.; Giri, R. *Acc. Chem. Res.* **2021**, *54*, 3415–3437.

<sup>6</sup> For a recent review of the mechanisms of inter- and intramolecular conjunctive XEC reactions see: (a) Diccianni, J.; Lin, Q.; Diao, T. *Acc. Chem. Res.* **2020**, *53*, 906–919. (b) Lin, Q.; Diao, T. *J. Am. Chem. Soc.* **2019**, *141*, 17937–17948.



intermediates.<sup>7,8</sup> *Domino methods that combine XEC reactions with additional C–C bond-forming reactions have not been reported.*

We sought to develop a domino reaction building on our nickel-catalyzed XEC reactions of ethers and sulfonamides (Scheme 4.1b).<sup>9,10</sup> In contrast with other XEC mechanisms, these reactions avoid radical intermediates, initiate with a polar oxidative addition of an activated C–O or C–N bond,<sup>11</sup> and cascade forward via an intramolecular S<sub>N</sub>2-type reaction. Based on the calculated reaction coordinate, catalyst turnover by reduction of Ni(II) to Ni(0) with formation of ethane is rate-determining. Therefore, this catalytic cycle presents an opportunity to develop new domino transformations by inserting elementary steps known for Ni(II) intermediates.<sup>12,13,14</sup> In this Chapter, I describe a nickel-catalyzed domino reaction that combines intramolecular XEC and

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<sup>7</sup> For lead examples of three-component conjunctive XEC reactions of alkenes, see: (a) García-Domínguez, A.; Li, Z.; Nevado, C. *J. Am. Chem. Soc.* **2017**, *139*, 6835–6838. (b) Shu, W.; García-Domínguez, A.; Quirós, M. T.; Mondal, R.; Cárdenas, D. J.; Nevado, C. *J. Am. Chem. Soc.* **2019**, *141*, 13812–13821. (c) Anthony, D.; Lin, Q.; Baudet, J.; Diao, T. *Angew. Chem. Int. Ed.* **2019**, *58*, 3198–3202. (d) Wei, X.; Shu, W.; García-Domínguez, A.; Merino, E.; Nevado C. *J. Am. Chem. Soc.* **2020**, *142*, 13515–13522. (e) Anthony, D.; Diao, T. *Synlett* **2020**, *31*, 1443–1447. (f) García-Domínguez, A.; Li, Z.; Nevado, C. *Chimia*. **2018**, *72*, 212–215. (g) Zhao, X.; Tu, H.-Y.; Guo, L.; Zhu, S.; Qing, F.-L.; Chu, L. *Nat. Commun.* **2018**, *9*, 3488. (h) Yang, T.; Chen, X.; Rao, W.; Koh, M. *J. Chem.* **2020**, *6*, 738–751. (i) Tu, H.-T.; Wang, F.; Huo, L.; Li, Y.; Zhu, S.; Zhao, X.; Li, H.; Qing, F.-L.; Chu, L. *J. Am. Chem. Soc.* **2020**, *142*, 9604–9611.

<sup>8</sup> For lead examples of two component conjunctive XEC reactions of alkenes, see: (a) Yan, C.-S.; Peng, Y.; Xu, X.-B.; Wang, Y.-W. *Chem. Eur. J.* **2012**, *18*, 6039–6048. (b) Xiao, J.; Wang, Y.-W.; Peng, Y. *Synthesis* **2017**, *49*, 3576–3581. (c) Kuang, Y.; Wang, X.; Anthony, D.; Diao, T. *Chem. Commun.* **2018**, *54*, 2558. (d) Wang, K.; Ding, Z.; Zhou, Z.; Kong, W. *J. Am. Chem. Soc.* **2018**, *140*, 12364–12368. (e) Jin, Y.; Wang, C. *Angew. Chem. Int. Ed.* **2019**, *58*, 6722–6726. (f) Xu, S.; Wang, K.; Kong, W. *Org. Lett.* **2019**, *21*, 7498–7503. (g) Tian, Z.-X.; Qiao, J.-B.; Xu, G.-L.; Pang, X.; Qi, L.; Ma, W.-Y.; Zhao, Z.-Z.; Duan, J.; Du, Y.-F.; Su, P.; Liu, X.-Y.; Shu, X.-Z. *J. Am. Chem. Soc.* **2019**, *141*, 7637–764

<sup>9</sup> (a) Tollefson, E. J.; Erickson, L. W.; Jarvo, E. R. *J. Am. Chem. Soc.* **2015**, *137*, 9760–9763. (b) Erickson, L. W.; Lucas, E. L.; Tollefson, E. J.; Jarvo, E. R. *J. Am. Chem. Soc.* **2016**, *138*, 14006–14011. (c) Lucas, E. L.; Hewitt, K. A.; Chen, P.-P.; Castro, A. J.; Hong, X.; Jarvo, E. R. *J. Org. Chem.* **2020**, *85*, 1775–1793.

<sup>10</sup> Chen, P.-P.; Lucas, E. L.; Greene, M. A.; Zhang, S.-Q.; Tollefson, E. J.; Erickson, L. W.; Taylor, B. L. H.; Jarvo, E. R.; Hong, X. *J. Am. Chem. Soc.* **2019**, *141*, 5835–5855.

<sup>11</sup> (a) Tollefson, E. J.; Hanna, L. E.; Jarvo, E. R. *Acc. Chem. Res.* **2015**, *48*, 2344–2353. (b) Zhang, S.-Q.; Hong, X. *Acc. Chem. Res.* **2021**, *54*, 2158–2171.

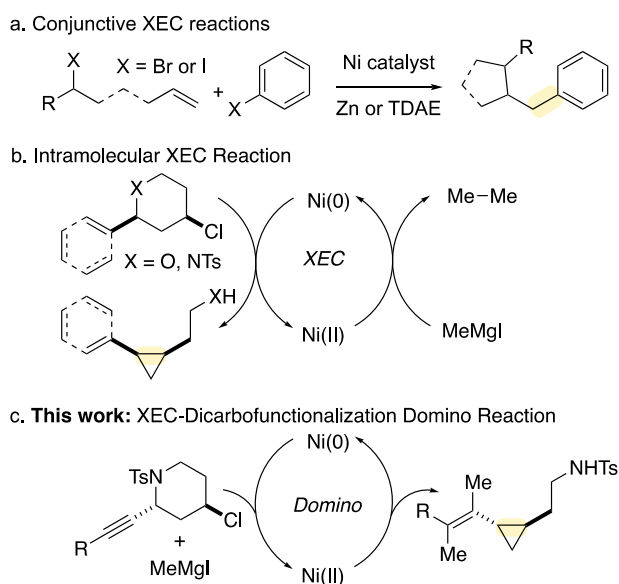
<sup>12</sup> For reviews of nickel catalysis, see: (a) *Modern Organonickel Chemistry*; Tamaru, Y., Ed.; Wiley-VCH: Weinheim; 2005. (b) Tasker, S. Z.; Standley, E. A.; Jamison, T. F. *Nature* **2014**, *509*, 299–309. (c) *Nickel Catalysis in Organic Synthesis: Methods and Reactions*; Ogoshi, S., Ed.; Wiley, **2020**.

<sup>13</sup> Dicarbofunctionalization of alkynes with two equivalents of Grignard reagents have been reported, see: Dong, C.-G.; Yeung, P.; Hu, Q.-S. *Org. Lett.* **2007**, *9*, 363–366.

<sup>14</sup> For an example of a nickel-catalyzed domino reaction that builds on XEC reactivity, see: Ping, Y.; Wang, K.; Pan, Q.; Ding, Z.; Zhou, Z.; Guo, Y.; Jong, W. *ACS Catal.* **2019**, *9*, 7335–7342.

dicarbofunctionalization reactions to produce substituted vinylcyclopropanes (Scheme 4.1c). This multicomponent transformation produces three new carbon–carbon bonds and a strained ring. The cyclopropane moiety is produced with high stereochemical fidelity. In addition, we provide experimental and computational evidence that the mechanism involves a single oxidative addition, an intramolecular  $S_N2$ -type reaction and a single reductive elimination. All elementary steps are stereospecific and product isomers result from divergent pathways from a key allenylnickel intermediate.

### Scheme 4.1 Nickel-Catalyzed Domino XEC Reactions

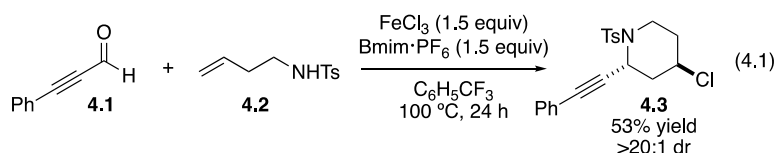


## 4.2 Results and Discussion

### 4.2.1 Starting Material Synthesis via the Aza-Prins Reaction

At the outset, we recognized that a critical component of our approach would be the rapid and efficient synthesis of the requisite starting materials. We envisioned that an aza-Prins reaction could generate 4-halo-2-alkynyl piperidines directly from ynals. Unfortunately, there were no Prins

or aza-Prins reactions of simple ynals in the literature.<sup>15</sup> The closest precedence was a cobalt-mediated reaction, where the cobalt complex serves to mask the alkyne to produce protected 2-alkynyl tetrahydropyrans.<sup>16</sup> We were encouraged, however, by iron-promoted aza-Prins reactions of enals for synthesis of 2-vinyl piperidines.<sup>17</sup> We applied similar conditions for the aza-Prins reaction of ynals (eq 4.1). Gratifyingly, this reaction afforded the desired 2-alkynyl-4-chloropiperidine **4.3** in 53% yield and as a single diastereomer. As expected, the *trans*-diastereomer was preferred due to minimization of A(1,3)-strain.<sup>18</sup> This transformation proved to be robust across a range of aryl- and alkyl-substituted ynals (*vide infra*).<sup>19</sup>



#### 4.2.2 Development of Nickel-Catalyzed Domino Reactions

We investigated the feasibility of the proposed domino reaction employing *N*-tosyl piperidine **4.3**.<sup>20</sup> We began with the previously developed conditions for the XEC reaction of aryl and vinyl sulfonamides with (*R*-BINAP)NiCl<sub>2</sub> as the precatalyst (Table 4.1, entry 1).<sup>9c,21</sup> We

<sup>15</sup> For reviews of the Prins/aza-Prins reaction see (a) Arundale, E.; Mikeska, L. A. *Chem. Rev.* **1952**, *51*, 505–555. (b) Adams, D. R.; Bhatnagar, S. P. *Synthesis*, **1977**, 661–672. (c) Olier, C.; Kaafarani, M.; Gastaldi, S.; Bertrand, M. P. *Tetrahedron*, **2010**, *66*, 413–445. (d) Pastor, I. M.; Yus, M. *Curr. Org. Chem.* **2012**, *16*, 1277–1312. (e) Reddy, B. V. S.; Nair, P. N.; Antony, A.; Lalli, C.; Grée, R. *Eur. J. Org. Chem.* **2017**, 1805–1819.

<sup>16</sup> Olier, C.; Gastaldi, S.; Gil, G.; Bertrand, M. P. *Tetrahedron Lett.* **2007**, *48*, 7801–7804.

<sup>17</sup> (a) Osawa, C.; Tateyama, M.; Miura, K.; Tayama, E.; Iwamoto, H.; Hasegawa, E. *Heterocycles* **2012**, *86*, 1211–1226. (b) ref 9c

<sup>18</sup> (a) Zhang, H.; Muñiz, K. *ACS Catal.* **2017**, *7*, 4122–4125. (b) Seel, S.; Thaler, T.; Takatsu, K.; Zhang, C.; Zipse, H.; Straub, B. F.; Mayer, P.; Knochel, P. *J. Am. Chem. Soc.* **2011**, *133*, 4774–4777.

<sup>19</sup> See Experimental Section for more information.

<sup>20</sup> For lead references of XEC of ammonium salts, aziridines, and pyridinium salts, see: (a) Moragas, T.; Gaydou, M.; Martin, R. *Angew. Chem., Int. Ed.* **2016**, *55*, 5053–5057. (b) Woods, B. P.; Orlandi, M.; Huang, C.-Y.; Sigman, M. H.; Doyle, A. G. *J. Am. Chem. Soc.* **2017**, *139*, 5688–5691. (c) Liao, J.; Basch, C. H.; Hoermer, M. E.; Talley, M. R.; Boscoe, B. P.; Tucker, J. W.; Garnsey, M. R.; Watson, M. P. *Org. Lett.* **2019**, *21*, 2941–2946. (d) Wang, J.; Hoerner, M. E.; Watson, M. P.; Weix, D. J. *Angew. Chem. Int. Ed.* **2020**, *59*, 13484–13489. For reviews on the activation of C–N bonds see: (e) Ouyang, K.; Hao, W.; Zhang, W.-X.; Xi, Z. *Chem. Rev.* **2015**, *115*, 12045–12090. (f) Wang, Q.; Su, Y.; Li, L.; Huang, H. *Chem. Soc. Rev.* **2016**, *45*, 1257–1272.

<sup>21</sup> For synthesis of (*R*-BINAP)NiCl<sub>2</sub>, see: (a) Standley, E. A.; Smith, S. J.; Muller, P.; Jamison, T. F. *Organometallics* **2014**, *33*, 2012–2018. (b) Dawson, D. D.; Oswald, V. F.; Borovik, A. S.; Jarvo, E. R. *Chem. Eur. J.* **2020**, *26*, 3044–3048.

evaluated two halide leaving groups, and the chloride produced the functionalized cyclopropane **4.4** in greater yield as compared to the fluoride (entries 1 and 2).<sup>22</sup> We determined by NMR analysis that the cyclopropane moiety is exclusively produced as the *trans* configuration while the olefin is generated as a mixture of (*E*)- and (*Z*)-alkene isomers.<sup>23</sup> In an attempt to improve diastereoselectivity, we cooled the reaction to 0 °C, however, no product was formed and 98% starting material was recovered (entry 3). Previously, our laboratory has found that Lewis acidic MgI<sub>2</sub> can accelerate the rate of oxidative addition of sluggish electrophiles,<sup>10,24</sup> however, the yield of the domino reaction did not improve with the addition of one equivalent of MgI<sub>2</sub> (entry 4). When the number of equivalents of the Grignard reagent was lowered, the yield improved (entries 5 and 6). Next, we evaluated alternative precatalysts. The commercially available Jamison catalyst, *cis*-(*R*-BINAP)Ni(*o*-tolyl)Cl, afforded similar yield and *E*:*Z* ratios of cyclopropane **4.4** (entry 7).<sup>21a</sup> Ni(cod)<sub>2</sub> and racemic BINAP provided decreased yield, consistent with our prior observation that cod inhibits XEC reactions of sulfonamides.<sup>9c</sup> Other precursors also afforded modest yields (entries 9 and 10).<sup>25</sup> Finally, we observed that the nickel catalyst was essential for the domino reaction to take place. In the absence of catalyst, simple propargylic displacement by the Grignard reagent occurred in 15% yield (entry 11).<sup>26,27</sup> We hypothesized that allenylcopper intermediates

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<sup>22</sup> Fluoride leaving groups were evaluated due to the success of the XEC Reactions of vinyl tetrahydropyrans. See ref 9b for further information.

<sup>23</sup> See Experimental Section for more information.

<sup>24</sup> (a) Greene, M. A.; Yonova, I. M.; Williams, F. J.; Jarvo, E. R. *Org. Lett.* **2012**, *14*, 4293–4296. (b) Felkin, H.; Swierczewski, G. *Tetrahedron Lett.* **1972**, *13*, 1433–1436.

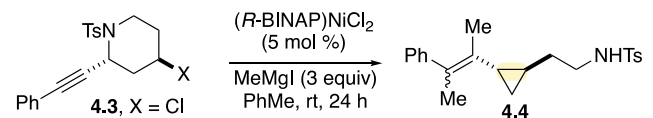
<sup>25</sup> Tran, V. T.; Li, Z.-Q.; Apolinar, O.; Derosa, J.; Joannou, M. V.; Wisniewski, S. R.; Eastgate, M. D.; Engle, K. M. *Angew. Chem. Int. Ed.* **2020**, *59*, 7409–7413.

<sup>26</sup> Allene **4.61** was the observed product. See Experimental Details Section **4.4.6** for full characterization of product.

<sup>27</sup> For examples of propargylic displacement by Grignard reagents, see (a) Gore, J.; Dulcere, J. P. *J. Chem. Soc., Chem. Commun.* **1972**, 866–867. (b) Krause, N.; Hoffmann-Röder, A. *Tetrahedron* **2004**, *60*, 11671–11694.

could potentially engage in an XEC reaction.<sup>28,29,30</sup> However, in preliminary investigations, when the nickel precatalyst was replaced with a copper salt no reaction was observed (entry 12). Therefore, we concluded that the optimal domino conditions employ a nickel(II) precatalyst, BINAP as the ligand, and two equivalents of methylmagnesium iodide.

**Table 4.1.** Optimization of Nickel-Catalyzed Domino Reaction



Entry	Deviation from Standard Conditions	Yield (%) <sup>a</sup>	E:Z
1	none	61	1.5 : 1
2	X = F	51	1.1 : 1
3	0 °C instead of rt	<5	–
4	1.0 equiv of MgI <sub>2</sub> added	61	1.7 : 1
5	2.5 equiv MeMgI	68	1.5 : 1
6	<b>2 equiv MeMgI</b>	<b>72 (71)<sup>b</sup></b>	<b>1.7 : 1</b>
<i>(R-BINAP)NiCl<sub>2</sub> replaced with 5 mol% of:</i>			
7	<i>cis</i> -( <i>R</i> -BINAP)Ni( <i>o</i> -tolyl)Cl	74	1.2 : 1
8	Ni(cod) <sub>2</sub> , <i>rac</i> -BINAP	30	1 : 2
9 <sup>c</sup>	Ni(cod)(DQ), <i>rac</i> -BINAP	42	1 : 1.2
10 <sup>c</sup>	Ni(acac) <sub>2</sub> , <i>rac</i> -BINAP	52	1 : 1.6
11	No Nickel Catalyst	<5	–
12 <sup>d</sup>	Cu(CH <sub>3</sub> CN) <sub>4</sub> OTf	<5	–

<sup>a</sup>Determined by <sup>1</sup>H NMR based on comparison to PhTMS as an internal standard.

<sup>b</sup>Isolated yield. <sup>c</sup>2 equiv of methylmagnesium iodide. <sup>d</sup> PhMe replaced with DCM.

To determine the functional group compatibility of this reaction, a series of experiments to probe the reaction robustness were performed (Figure 4.1).<sup>31</sup> Since this transformation employs a nucleophilic Grignard reagent, we first targeted functional groups that are sensitive to methylmagnesium iodide. We began by evaluating acidic moieties. Phenol, thiophenol, and aniline

<sup>28</sup> Copper catalysts are also known to promote propargyl substitution reactions. For lead references, see (a) Zhang, D.-E.; Hu, X.-P. *Tetrahedron Lett.* **2015**, *56*, 283–295. (b) Roy, R.; Saha, S. *RSC Adv.* **2018**, *8*, 31129–31193. (c) Miyake, Y.; Uemura, S.; Nishibayashi, Y. *ChemCatChem* **2009**, *1*, 342–356.

<sup>29</sup> For copper-catalyzed reactions with propargylic ammonium salts see: (a) Guisán-Ceinos, M.; Martín-Heras, V.; Soler-Yanes, R.; Cárdenas, D. J.; Tortosa, M. *Chem. Commun.* **2018**, *54*, 8343–8346. (b) Guisán-Ceinos, M.; Martín-Heras, V.; Tortosa, M. *J. Am. Chem. Soc.* **2017**, *139*, 8448–8451.

<sup>30</sup> Copper catalysts are known to promote dicarbofunctionalization reactions. For representative examples, see: (a) You, W.; Brown, M. K. *J. Am. Chem. Soc.* **2014**, *136*, 14730–14733. (b) You, W.; Brown, M. K. *J. Am. Chem. Soc.* **2015**, *137*, 14578–14581. (c) Thapa, S.; Basnet, P.; Giri, R. *J. Am. Chem. Soc.* **2017**, *139*, 5700–5703.

<sup>31</sup> Collins, K.; Glorius, F. A. *Nature Chem.* **2013**, *5*, 597–601.

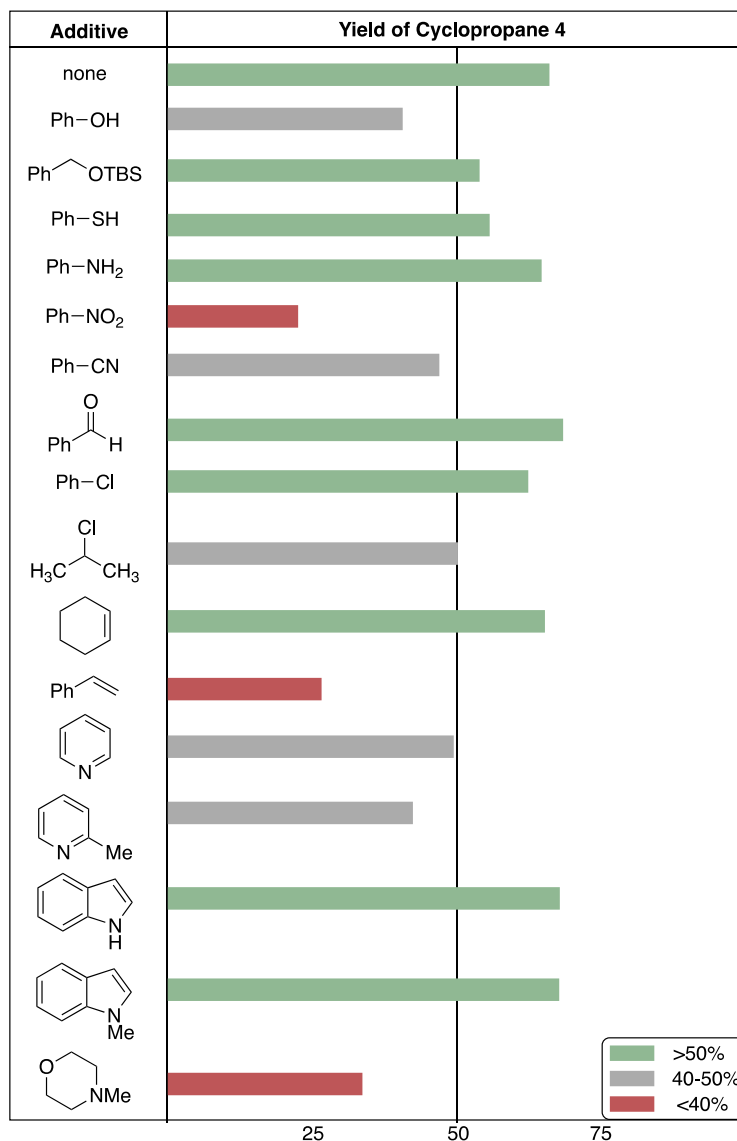
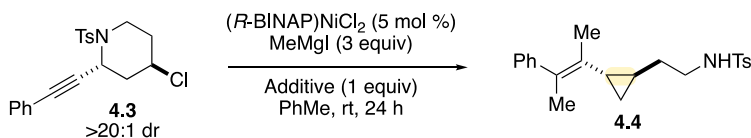
were all tolerated under the reaction conditions, given excess Grignard reagent to account for proton transfer, allowing for moderate to good yields of cyclopropane **4.4**. We were also pleased to note that a silyl ether was well tolerated. Nitrobenzene inhibited the nickel catalyst and poor yields of cyclopropane **4.4** were observed. With excess Grignard reagent, a nitrile and an aldehyde were cleanly converted to the amine and alcohol, respectively, and these moieties did not interfere with nickel catalyst: cyclopropane **4.4** was generated in good yields.

Next, we targeted additives with functional groups that can react with or serve as ligands for nickel catalysts (Figure 4.1). Aryl and alkyl chlorides did not outcompete the propargyl sulfonamide; the substituted vinylcyclopropane was observed in good yield. Alkenes strongly ligate nickel catalysts, so we evaluated two alkenes: cyclohexene and styrene. Cyclohexene was well-tolerated and did not inhibit the reaction, however, styrene inhibited the domino reaction likely due to strong ligation to the active catalyst. Finally, we evaluated nitrogen-containing heterocycles, common ligands for transition metals. In the presence of pyridine and a substituted pyridine the yield of cyclopropane **4.4** was lowered. Interestingly, when pyridine was the additive, (*Z*)-alkene **4.4** was the major product.<sup>32</sup> Indole and *N*-methyl indole did not affect the yields of the domino transformation. Finally, *N*-methylmorpholine inhibited the domino reaction resulting in diminished yield of cyclopropane **4.4**. Taken together, numerous functional groups are tolerated under our reaction conditions providing a robust domino reaction.

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<sup>32</sup> See Experimental Section **4.4.3** for more information.

**Figure 4.1** Robustness Screen to Establish Functional Group Compatibility<sup>a,b</sup>



(a) Yield of cyclopropane **4** determined by <sup>1</sup>H NMR based on comparison to PhTMS as an internal standard. (b) Unless otherwise noted, products were observed as ~1.5:1 mixture of (*E*)- and (*Z*)-alkene isomers.

Next, we evaluated the scope of the domino reaction (Scheme 4.2). Historically, cyclopropanes have been synthesized via Simmons–Smith reactions, transition metal-catalyzed

carbene insertions, cycloisomerizations, and nucleophilic displacement reactions.<sup>33</sup> This method establishes an alternative to these strategies, while also providing a rapid synthesis of highly substituted vinylcyclopropanes.<sup>34,35,36</sup> First, we evaluated substituted aromatic rings (**4.4–4.12**).<sup>37</sup> We found that electron donating groups were well tolerated in the domino reaction, producing the desired product in excellent yields and moderate *E:Z* ratios. Interestingly, this reaction could be scaled up tenfold (to 1.0 mmol) and still retain good yield. The electron withdrawing CF<sub>3</sub> group provided the desired product albeit in moderate yield and diastereoselectivity. Interestingly, the sterically bulky *t*-Bu substrate provided (*Z*)-alkene **4.11** as the major product. We hypothesize that the *t*-Bu groups hinder the carbometallation step and favor the intramolecular transmetalation leading to (*Z*)-vinylcyclopropane as the major product (*vide infra*). We were delighted to observe that aromatic groups were not necessary and alkyl substrates were able to undergo the transformation in excellent yields (**4.13–4.18**).<sup>38</sup> It is important to note that cyclopropane **4.13** derived from propyne is obtained in excellent diastereoselectivity (>20:1) further confirming that cyclopropane formation is highly stereoselective.

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<sup>33</sup> For reviews on the synthesis of vinylcyclopropanes see: (a) Lebel, H.; Marcoux, J.-F.; Molinaro, C.; Charett, A. B. *Chem. Rev.* **2003**, *103*, 977–1050. (b) Bartoli, G.; Bencivenni, G.; Dalpozzo, R. *Synthesis*, **2014**, *46*, 979–1029. (c) Ebner, C.; Carreira, E. M. *Chem. Rev.* **2017**, *117*, 11651–11679; (d) Wu, W.; Lin, Z.; Jiang, H. *Org. Biomol. Chem.* **2018**, *16*, 7315–7329. (e) Mato, M.; Franchino, A.; García-Morales, C.; Echavarren, A. M. *Chem. Rev.* **2021**, *121*, 8613–8684.

<sup>34</sup> For selected cross-coupling approaches for the synthesis of vinylcyclopropanes, see (a) Charette, A. B.; Giroux, A. *J. Org. Chem.* **1996**, *61*, 8718–8719. (b) Yao, M.-L.; Deng, M.-Z. *Tetrahedron Lett.* **2000**, *41*, 9083–9087. (c) Hu, L.; Shen, P.-X.; Shao, Q.; Hong, K.; Qiao, J.; Yu, J.-Q. *Angew. Chem. Int. Ed.* **2018**, *58*, 2134–2138.

<sup>35</sup> For transformations of vinylcyclopropanes, see: (a) Jiao, L.; Yu, Z.-X. *J. Org. Chem.* **2013**, *78*, 6842–6848. (b) Rubin, M.; Rubina, M.; Gevorgyan, V. *Chem. Rev.* **2007**, *107*, 3117–3179 (d) Souillart, L.; Cramer, N. *Chem. Rev.* **2015**, *115*, 9410–9464. (e) Wang, J.; Blaszczyk, S. A.; Li, X.; Tang, W. *Chem. Rev.* **2020**, *121*, 110–139.

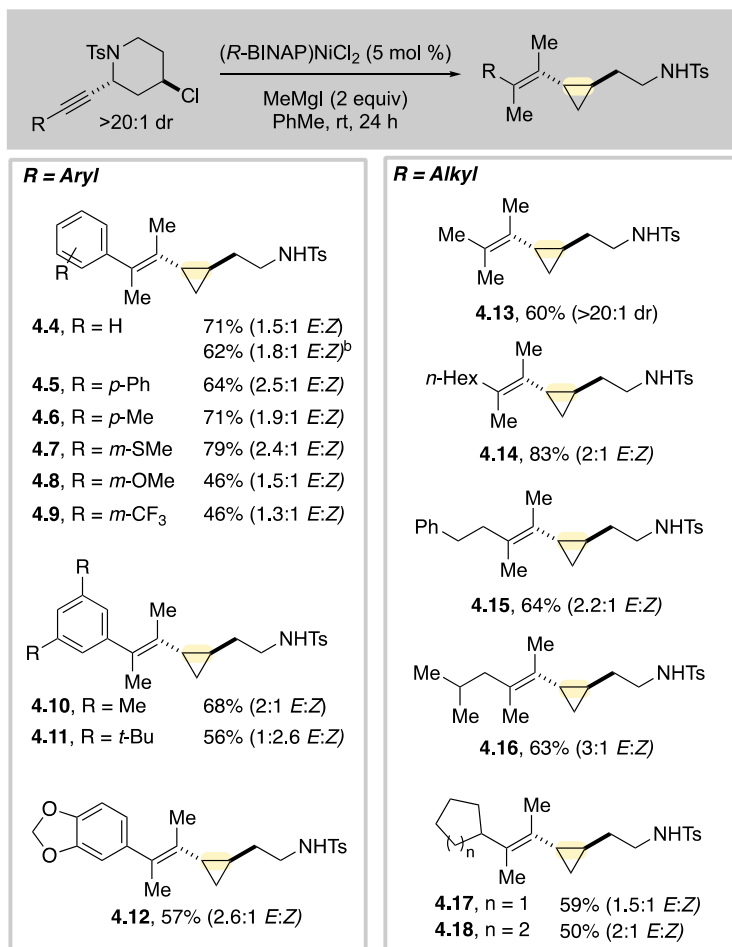
<sup>36</sup> For Fe-catalyzed conjunctive XC reactions of vinylcyclopropanes that proceeds with ring-opening, see: (f) Liu, L.; Lee, W.; Yuan, M.; Acha, C.; Geherty, M. B.; Williams, B.; Guitierrez, O. *Chem. Sci.* **2020**, *11*, 3146–3151

<sup>37</sup> Substrates **4.8** and **4.9** synthesized by Nadia Hirbawi.

<sup>38</sup> Substrates **4.15**, **4.16**, and **4.17** synthesized by Taylor A. Thane



### Scheme 4.2 Substrate Scope<sup>a</sup>



<sup>a</sup>Reactions performed on 0.1–0.2 mmol scale. Isolated yields.

<sup>b</sup>Reaction performed on 1.0 mmol scale.

Vinylcyclopropane and amine-substituted cyclopropane moieties are present in numerous biologically active compounds, natural products and pharmaceutical agents.<sup>39,40</sup> For example, amino-substituted vinylcyclopropanes have been established as lysine specific demethylase (LSD1) inhibitors with potent anti-leukemia activity.<sup>41</sup> To demonstrate the synthetic potential of

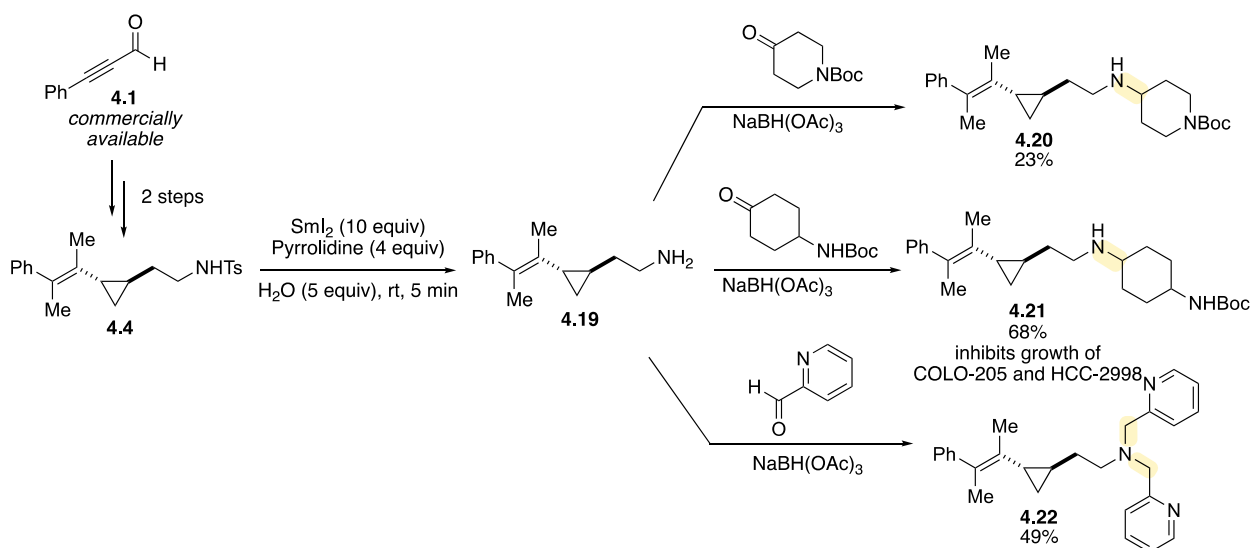
<sup>39</sup> For reviews on biological activity of substituted vinyl cyclopropanes, see: (a) Talele, T. T. *J. Med. Chem.* **2016**, *59*, 8712–8756. (b) Salaün, J. Cyclopropane Derivatives and their Diverse Biological Profile. In *Small Ring Compounds in Organic Synthesis VI*. de Meijere, A. Ed. **2000**. pp. 1–67.

<sup>40</sup> For discussions on the biological activity of substituted alkenes, see: (a) Avendano, C.; Menendez, J. C. *Medicinal Chemistry of Anticancer Drugs*, Elsevier, Oxford, **2015**, pp. 87–95. (b) Flynn, A. B.; Ogilvie, W. W. *Chem. Rev.* **2007**, *107*, 4698–4745.

<sup>41</sup> Albrecht, B. K.; Audia, J. E.; Cote, A.; Duplessis, M.; Gehling, V. S.; Harmange, J.-C.; Vaswani, R. G. LSD1 Inhibitors and Uses Thereof. WO 2016/172496 A1. 20

our domino reaction, we synthesized a series of analogs of LSD1 inhibitors (Scheme 4.3). First, we performed a  $\text{SmI}_2$  deprotection of the pendant sulfonamide.<sup>42</sup> The resulting primary amine **4.19** was subjected to three different reductive amination reactions to produce **4.20**, **4.21**, and **4.22**. Interestingly, amine **4.21** demonstrated significant cell death against colon cancer cell lines (COLO-205 and HCC-2998).<sup>43,44</sup> Therefore, this domino reaction provides rapid access to bioactive structural motifs in four steps from commercially available materials.

**Scheme 4.3** Derivatization of Cyclopropane Products



### 4.3 Proposed Mechanism of the Domino XEC Reaction

#### 4.3.1 Mechanistic Experiments to Probe the Domino Reaction.

Next, we aimed to elucidate the mechanism of this multicomponent reaction. A series of control experiments were consistent with initiation of the catalytic cycle by oxidative addition of the nickel catalyst with the propargylic sulfonamide moiety. First, employing an alternative Grignard reagent,  $\text{EtMgI}$ , led to formation of alkynylcyclopropane **4.23** in 15% yield (Scheme

<sup>42</sup> (a) Szostak, M.; Spain, M.; Procter, D. J. *J. Org. Chem.* **2012**, *77*, 3049–3059. (b) Ankner, T.; Hilmersson, G. *Org. Lett.* **2009**, *11*, 503–506.

<sup>43</sup> (a) Shoemaker, R. H. *Nat. Rev. Cancer* **2006**, *6*, 813–823. (b) National Institute of Health, “National Cancer Institute Developmental Therapeutics Program,” can be found under <https://dtp.cancer.gov/>, 2021.

<sup>44</sup> Product **4.21** synthesized by Nadia Hirbawi and product **4.22** synthesized by Taylor A. Thane.

4a).<sup>45</sup> This observation is consistent with oxidative addition of the propargylic sulfonamide to produce an allenylnickel complex that proceeds through an XEC pathway. A simple propargylic sulfonamide, **4.24**, lacking a pendant alkyl halide, underwent substitution to form allene **4.25** (Scheme 4.4b). This further supports that the mechanism initiates by oxidative addition of the propargylic sulfonamide. Additionally, simple alkynes did not undergo dicarbofunctionalization under standard reaction conditions (Scheme 4.4c and 4.4d). Alkynyl cyclopropane **4.26** did not undergo a 1,2-dicarbofunctionalization under standard reaction conditions, employing catalytic or stoichiometric quantities of nickel complex.<sup>46</sup> Similarly, in a competition experiment, propargylic sulfonamide **4.3** reacted while alkyne **4.27** was not functionalized.<sup>47</sup> Therefore, even in the presence of relevant catalytically active organonickel intermediates, a simple alkyne was not engaged. Taken together, these experiments support initiation of the domino reaction by oxidative addition of the propargylic sulfonamide moiety.

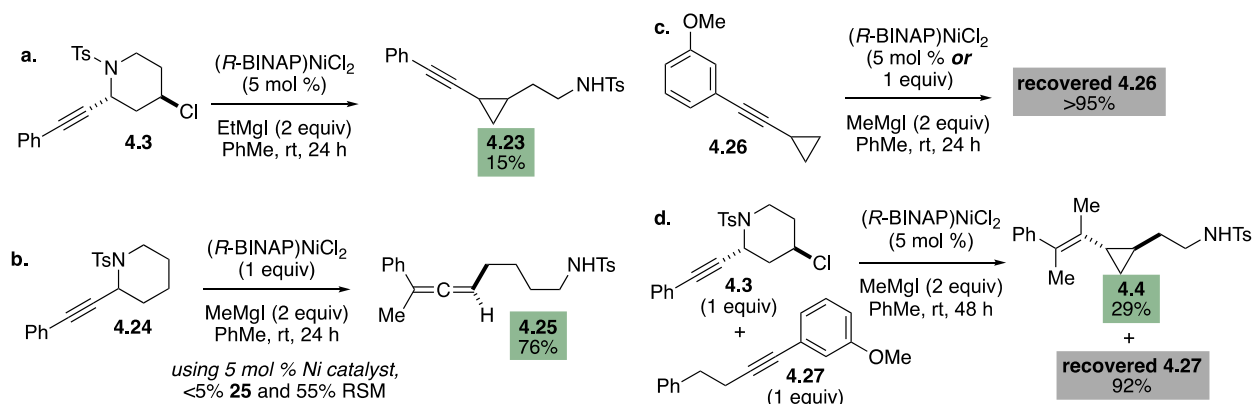
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<sup>45</sup> Control experiment in Scheme 4.4a performed by Taylor A. Thane

<sup>46</sup> Control experiment in Scheme 4.4c performed by Taylor A. Thane

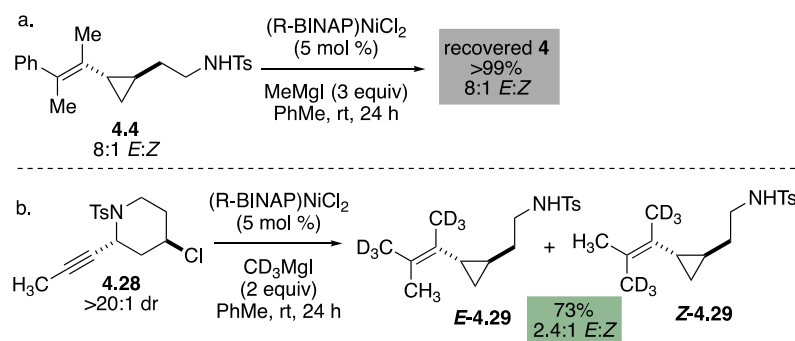
<sup>47</sup> Control experiment in Scheme 4.4d performed by Nadia Hirbawi

### Scheme 4.4 Mechanistic Control Experiments



Next, we examined subsequent steps in the catalytic cycle, with a particular focus on the steps that would bifurcate to afford (*E*)- and (*Z*)-isomers of the alkene. To ensure that the product distribution was a kinetic distribution, we performed a control experiment (Scheme 4.5a). Resubjecting vinylcyclopropane **4.4** (8:1 *E:Z*) to the reaction conditions resulted in no isomerization. To determine whether the relative stability of product diastereomers is reflected in key transition states, we examined a substrate devoid of a steric preference for product formation (Scheme 4.5b).<sup>48</sup> Employing a deuterated Grignard reagent provided vinylcyclopropane **4.29** as a 1:2.4 mixture (*E:Z*). Therefore, we concluded that the relative stability of product isomers does not impact the selectivity of the reaction.

### Scheme 4.5 Kinetic and Thermodynamic Stability of Products



<sup>48</sup> Control reaction in Scheme 4.5b performed by Taylor A. Thane.

Next, we aimed to understand the stereoselective formation of the cyclopropane moiety. A stereochemical proof was performed to experimentally confirm that the cyclopropane ring formation is enantiospecific and occurs with retention (double inversion) at the sulfonamide-bearing carbon, and inversion at the alkyl chloride. To synthesize the desired enantioenriched *trans*-piperidine (+)-**4.34**, the following synthesis was performed (Scheme 4.6). First, a diastereoselective TFA-promoted aza-Prins reaction afforded racemic alkynylpiperidine **4.31**. Enantioenriched alkynylpiperidine (+)-**4.32** was prepared by lipase-catalyzed kinetic resolution.<sup>49</sup> Deprotection of the acetate group followed by an oxidation with DMP and reduction with L-selectride afforded the *cis*-alkynylpiperidine (+)-**4.33**. Absolute configuration of alcohol (+)-**4.33** was unambiguously assigned by X-ray crystallography. Finally, a mesylation reaction followed by a chlorination reaction produced alkynylpiperidine (+)-**4.34**. The enantioenriched piperidine was subjected to the standard conditions and the major diastereomer was isolated in a 31% yield. I attempted to crystallize this material and confirm the absolute configuration by X-ray crystallography, however, all attempts to grow X-ray quality crystals were unsuccessful. The product was derivatized to sulfonimide (+)-**4.35** and the absolute configuration was determined by comparing the experimental circular dichroism (CD) spectrum to the simulated spectrum.<sup>50,51</sup>

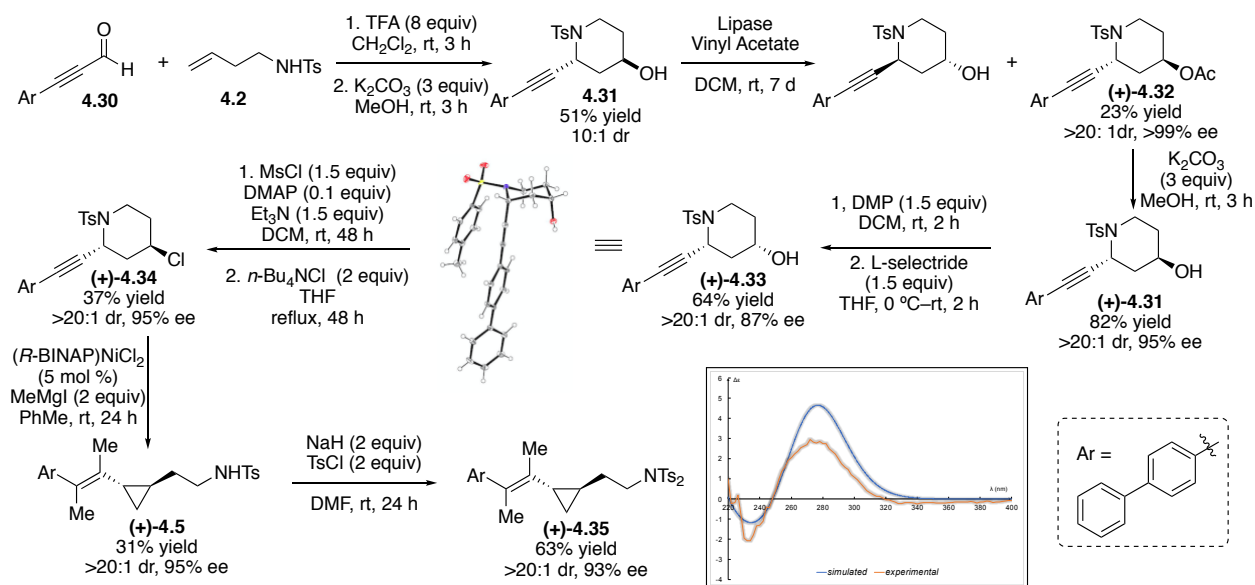
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<sup>49</sup> Yadav, J. S.; Reddy, B. V. S.; Padmavani, B.; Venugopal, C.; Rao, A. B. *Tetrahedron Lett.* **2007**, *48*, 4631–4633.

<sup>50</sup> Bannwarth, C.; Grimme, S. *Comput. Theor. Chem.* **2014**, *1040–1041*, 45–53.

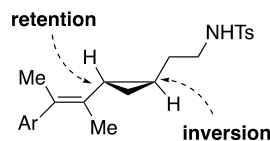
<sup>51</sup> Experimental CD spectra collected by Dr. Dima Fishman and the UCI laser spectroscopy lab

### Scheme 4.6 Stereochemical Outcome of the Domino Reaction



In our previous investigation of the XEC reaction of benzylic and allylic sulfonamides, we hypothesized that the ring contraction was stereospecific.<sup>9c,52</sup> The proposed oxidative addition proceeded with inversion and the intramolecular  $\text{S}_{\text{N}}2$ -type reaction proceeded with inversion at both the benzylic position and the alkyl chloride. We aimed to prove this experimentally but we were unable to obtain X-ray crystallographic data for the cyclopropane product. With the stereochemical proof presented in Scheme 4.6, we are able to definitely prove that the ring contraction of sulfonamides proceeds with double inversion (net retention) at the propargylic carbon and inversion at the alkyl chloride (Figure 4.2).

**Figure 4.2** Stereochemical Outcome of the Domino XEC Reaction



<sup>52</sup> For more information, see Chapter 3, section 3.3.

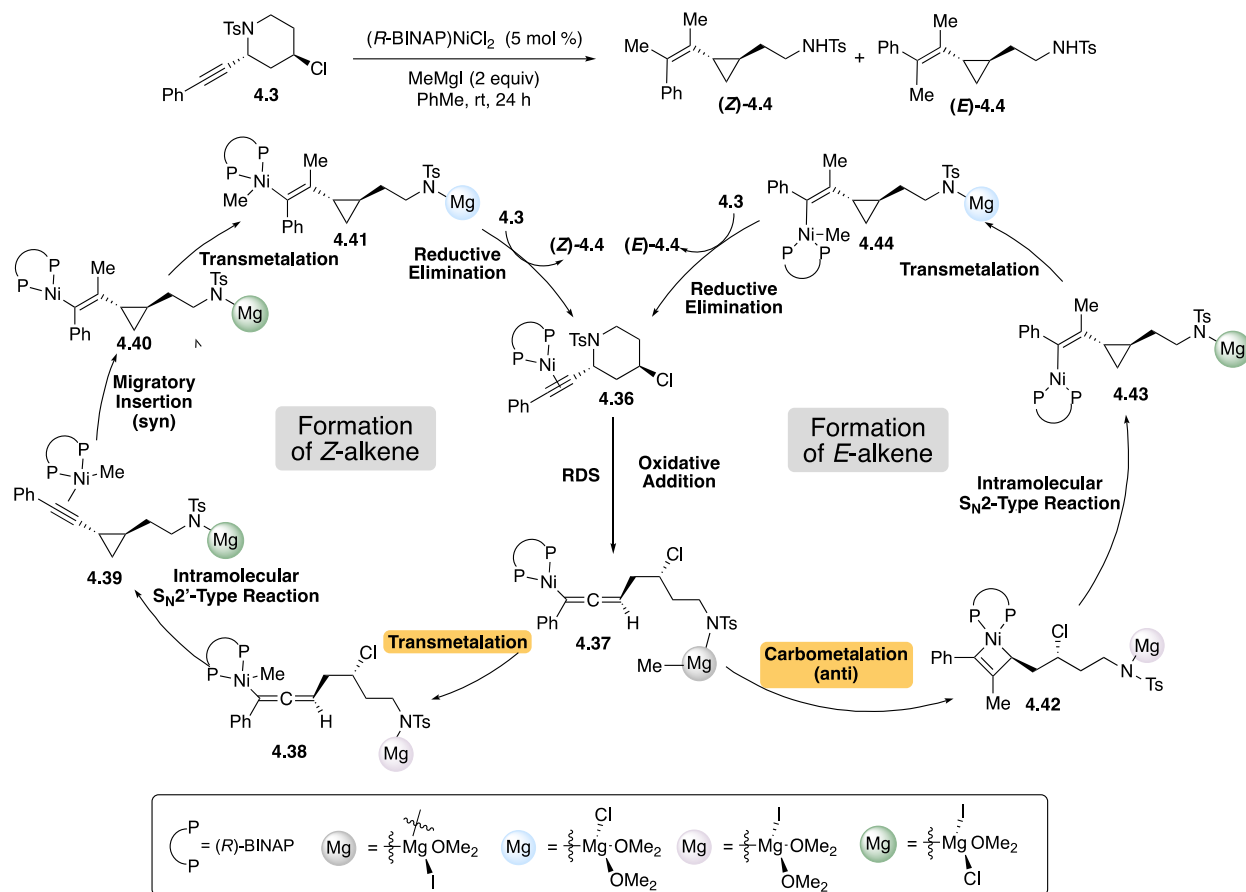
### 4.3.2 Proposed Mechanism of the Domino Reaction

Based on the experimental and computational mechanistic experiments, we propose the following domino mechanism (Scheme 4.7).<sup>1,10,53</sup> Initiation of the catalytic cycle occurs with oxidative addition of the propargylic sulfonamide. The key allenylnickel intermediate **4.37** can react via two pathways. Carbometallation by nucleophilic attack of the pendant organomagnesium complex affords metallacyclobutane **4.42**, and eventually leads to formation of the major diastereomer, (*E*)-**4.4**. Alternatively, allenylnickel complex **4.37** can undergo transmetallation and subsequently attack the pendant electrophilic alkyl chloride, leading to formation of the minor diastereomer, (*Z*)-**4.4**. The pathways that diverge from allenylnickel intermediate **4.37** have transition states that are similar in energy, consistent with the observed modest levels of diastereoselectivity.<sup>1</sup> Each elementary step is stereospecific, with *anti*-carbometallation and *syn*-reductive elimination providing (*E*)-**4.4** and *syn*-migratory insertion and *syn*-reductive elimination generating (*Z*)-**4.4**

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<sup>53</sup> For details on the computational data and calculated reaction coordinate diagrams, see ref 1

### Scheme 4.7 Proposed Mechanism of the Domino Reaction



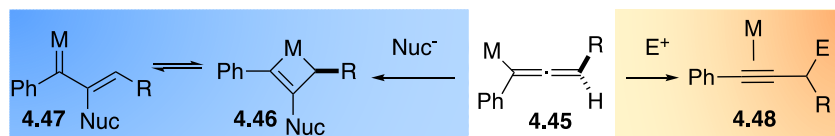
Interestingly, this proposed bifurcated mechanism is consistent with the known ambiphilic reactivity of allenylmetal intermediates (Scheme 4.8).<sup>54</sup> Nucleophiles tend to add to the central carbon, leading to formation of metallocyclobutene intermediates **4.46**. These metallocyclobutenes can equilibrate with vinylcarbene complexes **4.47**. In contrast, electrophiles tend to add to the terminal carbon. In our system, allenylmetal complex **4.37** reacts with coordinated nucleophilic Grignard reagent to form metallocyclobutene **4.42**. However, if transmetalation proceeds, the

<sup>54</sup> (a) Minami, I.; Yuhara, M.; Watanabe, H.; Tsuji, J. *J. Organomet. Chem.* **1987**, *334*, 225–242. (b) Casey, C. P.; Nash, J. R.; Yi, C. S.; Selmeczy, A. D.; Chung, S.; Powell, D. R.; Hayashi, R. K. *J. Am. Chem. Soc.* **1998**, *120*, 722–733. (c) Chen, J.-T. *Coord. Chem. Rev.* **1999**, *190–192*, 1143–1168. (d) Tsuji, J.; Mandai, T. *Angew. Chem. Int. Ed. Engl.* **1995**, *34*, 2589–2612. (e) Ma, S. *Eur. J. Org. Chem.* **2004**, *6*, 1175–1183. (f) Cheng, Y.-C.; Chen, Y.-K.; Huang, T.-M.; Yu, C.-I.; Lee, G.-H.; Wang, Y.; Chen, J.-T. *Organometallics* **1998**, *17*, 2953–2957. (g) Holland, R. L.; Bunker, K. D.; Chen, C. H.; DiPasquale, A. G.; Rheingold, A. L.; Baldrige, K. K.; O'Connor, J. M. *J. Am. Chem. Soc.* **2008**, *130*, 10093–10095.



resultant electron-rich allenylnickel moiety of **4.36** quickly attacks the pendant electrophilic alkyl chloride.

**Scheme 4.8** Ambiphilic Pathways for Allenylmetal Species



## 4.4 Conclusions

In conclusion, we report a nickel-catalyzed domino reaction, to the best of our knowledge, the first example that includes separate XEC and dicarbomethylation reactions. The net transformation consists of ring-contraction of the piperidine and dicarbomethylation of the alkyne, generating highly substituted cyclopropanes bearing tetrasubstituted olefins and aminoethyl substituents. The reaction products are structurally related to histone demethylase inhibitors and we report the synthesis of derivatives that demonstrate selective inhibition of colon and non-small cell lung cancer cell lines. In addition, to accomplish rapid synthesis of the requisite starting materials, we report the first aza-Prins reaction of simple ynals to afford alkynyl piperidines, the starting materials for this domino reaction. Experimental and computational analysis of the reaction mechanism support a bifurcated reaction mechanism that accounts for product distributions. After oxidative addition, a key allenylnickel intermediate partitions into two different pathways, each with similar barrier heights, that lead to the observed reaction products. Future studies will include the development of related domino reactions and transformations that employ ambiphilic allenylnickel complexes.

## 4.5 Experimental Details

### 4.5.1 General Procedures

All reactions were carried out under an atmosphere of N<sub>2</sub>, or Ar when noted. All glassware was oven- or flame-dried prior to use. Tetrahydrofuran (THF), diethyl ether (Et<sub>2</sub>O), dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>), dichloroethane (DCE), dimethylformamide (DMF), triethylamine (Et<sub>3</sub>N), and toluene (PhMe) were degassed with Ar and then passed through two 4 x 36 inch columns of anhydrous neutral A-2 alumina (8 x 14 mesh; LaRoche Chemicals; activated under a flow of argon at 350 °C for 12 h) to remove H<sub>2</sub>O.<sup>55</sup> All other solvents utilized were purchased anhydrous commercially, or purified as described. <sup>1</sup>H NMR spectra were recorded on Bruker DRX-400 (400 MHz <sup>1</sup>H, 100 MHz <sup>13</sup>C, 376.5 MHz <sup>19</sup>F), GN-500 (500 MHz <sup>1</sup>H, 125.4 MHz <sup>13</sup>C), CRYO-500 (500 MHz <sup>1</sup>H, 125.8 MHz <sup>13</sup>C) or AVANCE-600 (600 MHz <sup>1</sup>H, 150 MHz <sup>13</sup>C, 564.6 MHz <sup>19</sup>F) spectrometers. Proton chemical shifts are reported in ppm (δ) relative to internal tetramethylsilane (TMS, δ 0.00). Data are reported as follows: chemical shift (multiplicity [singlet (s), doublet (d), triplet (t), quartet (q), sextet (sext), septet (sept), multiplet (m), broad singlet (bs), broad triplet (bt), doublet of doublet (dd), doublet of triplet (dt), doublet of quartet (dq), doublet of doublet of doublet (ddd), doublet of doublet of triplet (ddt), doublet of triplet of doublet (dtd), triplet of doublet (td), triplet of triplet (tt), triplet of doublet of doublet (tdd), quartet of doublet (qd), quartet of triplet (qt), quartet of doublet of doublet (qdd), apparent singlet (as), apparent triplet (at), apparent quintet (aquin), apparent doublet of quintet (aquin), apparent triplet of doublet (aqd), apparent quartet of doublet (aqd)], coupling constants [Hz], integration). Carbon chemical shifts are reported in ppm (δ) relative to TMS with the respective solvent resonance as the internal standard (CDCl<sub>3</sub>, δ 77.16 ppm). Fluorine chemical shifts are reported in ppm (δ) relative to the absolute frequency of 0.00 ppm in the proton spectrum. Unless otherwise indicated, NMR data were collected at 25 °C. Infrared (IR) spectra were obtained on a Thermo Scientific Nicolet iS5

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<sup>55</sup> Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518–1520.

spectrometer with an iD5 ATR tip (neat) and are reported in terms of frequency of absorption ( $\text{cm}^{-1}$ ). Analytical thin-layer chromatography (TLC) was performed using Silica Gel 60 F<sub>254</sub> precoated plates (0.25 mm thickness). Visualization was accomplished by irradiation with a UV lamp and/or staining with  $\text{KMnO}_4$  or CAM. Flash chromatography was performed using SiliaFlash F60 (40-63  $\mu\text{m}$ , 60 Å) from SiliCycle. Automated chromatography was carried out on a Teledyne Isco CombiFlash Rf Plus. Melting points (m.p.) were obtained using a Mel-Temp melting point apparatus and are uncorrected. Optical rotations were measured on a Rudolph Research Analytical Autopol III Automatic Polarimeter. SFC Determinations of enantiopurity were determined by chiral SFC analysis and performed on an Agilent Technologies HPLC (1260 series) system or Agilent Technologies HPLC (1200 series) Analytical Instruments using OJ-H, OD-H, and AD-H Chiralpak columns (100 bar, 50°C, 254 nm). High resolution mass spectrometry was performed by the University of California, Irvine Mass Spectrometry Center. Circular dichroism spectra were performed by the University of California, Irvine Laser Spectroscopy Labs and were collected on a Jasco J-810 spectropolarimeter (163–900 nm).

Bis(1,5-cyclooctadiene)nickel was purchased from Strem, stored in a glove box freezer (–20 °C) under an atmosphere of  $\text{N}_2$  and used as received. All ligands were purchased from Strem or Sigma Aldrich and were stored in a glovebox and used as received. The methylmagnesium iodide was titrated with iodine prior to use.<sup>56</sup> All other chemicals were purchased commercially and used as received, unless otherwise noted.

## **4.5.2 General Domino XEC 1,2-Dicarbonylfunctionalization Reaction Procedures**

### **4.5.2.1 Method A: Domino XEC 1,2-Dicarbonylfunctionalization with (*R*-BINAP)NiCl<sub>2</sub>**

In a glovebox, an oven-dried 7 mL vial equipped with a stir bar was charged with substrate

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<sup>56</sup> Krasoviskiy, A.; Knochel, P. *Synthesis* **2006**, 5, 890.

(1 equiv), (*R*-BINAP)NiCl<sub>2</sub> (5 mol %), and PhMe (0.2 M in substrate). A solution of MeMgI in Et<sub>2</sub>O (2 equiv) was then added dropwise via syringe. After 24 h, the reaction vial was removed from the glovebox, quenched with MeOH, filtered through a plug of silica gel eluting with Et<sub>2</sub>O, and concentrated in vacuo. Phenyltrimethylsilane (PhTMS; 8.6 μL, 50. μmol) was added and the yield was determined by <sup>1</sup>H NMR based on comparison to PhTMS as internal standard before purification by column chromatography.

#### 4.4.2.2. Preparation of Methylmagnesium Iodide

Under a N<sub>2</sub> atmosphere, a three-necked flask equipped with a stir bar, reflux condenser, and Schlenk filtration apparatus was charged with magnesium turnings (2.80 g, 115 mmol). The flask and magnesium turnings were then flame-dried under vacuum and the flask was backfilled with N<sub>2</sub>. Anhydrous Et<sub>2</sub>O (25 mL) and a crystal of iodine (ca. 2 mg) were added to the flask. Freshly distilled iodomethane (5.0 mL, 80 mmol) was slowly added over 30 min to maintain a gentle reflux. The mixture was stirred for 2 h at room temperature then filtered through the fritted Schlenk filter into a pear-shaped flask under a N<sub>2</sub> atmosphere. The pear-shaped flask was capped with a septum, sealed with parafilm, and stored in the glovebox under a N<sub>2</sub> atmosphere for up to eight weeks. The resulting methyl Grignard reagent was typically between 2.4 and 3.0 M as titrated by Knochel's method.<sup>56</sup>

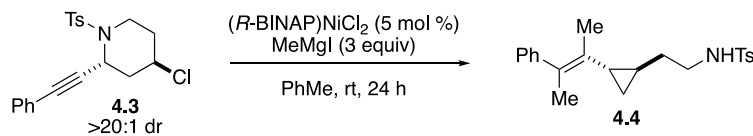
#### 4.5.2.3 Preparation of (*R*-BINAP)NiCl<sub>2</sub>

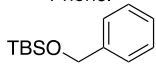
(*R*-BINAP)NiCl<sub>2</sub> was synthesized according to a procedure reported by Jamison.<sup>21a</sup> To a 50 mL round-bottom flask equipped with a stir bar was added NiCl<sub>2</sub>·6H<sub>2</sub>O (152 mg, 0.64 mmol, 1.0 equiv). The flask was placed under vacuum and flame-dried until nearly all of the nickel compound had turned from green to yellow-orange (a small amount of remaining green of the hexahydrate is necessary for the reaction to proceed). After cooling to room temperature, (*R*-

BINAP (0.40 g, 0.64 mmol, 1.0 equiv) was added to the flask and a reflux condenser was attached. The flask was evacuated, backfilled with N<sub>2</sub>, and then anhydrous MeCN (20 mL) was added. The reaction mixture was heated to reflux in an oil bath for 24 h, at which point the solution was cooled to room temperature and filtered under vacuum to yield a fine, black powder (0.33 mg, 0.44 mmol, 68% yield).

#### 4.5.3 Robustness Screen to Establish Functional Group Compatibility

In a glovebox, an oven-dried 7 mL vial equipped with a stir bar was charged with piperidine 3 (1 equiv), (*R*-BINAP)NiCl<sub>2</sub> (5 mol %), and PhMe (0.4 M in substrate). The reaction was sealed with a septum cap, brought out of the glovebox, and placed under an N<sub>2</sub> atmosphere. Then the additive (0.1 mmol, 1.0 equiv) was added via microsyringe followed by dropwise addition of a solution of MeMgI in Et<sub>2</sub>O (3 equiv). After 24 h, the reaction mixture was quenched with MeOH, filtered through a plug of silica gel eluting with Et<sub>2</sub>O, and concentrated in vacuo. Phenyltrimethylsilane (PhTMS; 8.6 μL, 50. μmol) was added and the yield was determined by <sup>1</sup>H NMR based on comparison to PhTMS as internal standard before purification by column chromatography. The results from this experiment are pictorially presented via a bar graph in Figure 4.1 and tabulated below in Table 4.2.

**Table 4.2** Robustness Screen Results.

Entry	Additive	Conversion (Additive) <sup>a</sup>	Yield ( <i>E:Z</i> ) <sup>a</sup>
1	none	n/a	61% (1.6 : 1)
2	Phenol	55%	43% (1.2 : 1)
3		<5%	53% (1.8 : 1)
4	Thiophenol	37%	56% (1.8 : 1)
5	Aniline	30%	59% (1 : 1)
6	Nitrobenzene	>95%	23% (2.2 : 1)
7	Benzonitrile	>95%	48% (2.4 : 1)
8	Benzaldehyde	>95%	65% (2.2 : 1)
9	Chlorobenzene	64%	57% (2.1 : 1)
10	2-Chloropropane	53%	50% (2.3 : 1)
11	Cyclohexene	82%	60% (1.3 : 1)
12	Styrene	89%	28% (2.1 : 1)
13	Pyridine	>95%	49% (1 : 1.6)
14	2-Methylpyridine	50%	41% (1 : 1.2)
15	Indole	<5%	65% (1.6 : 1)
16	1-Methyl-1 <i>H</i> -indole	30%	66% (1.5 : 1)
17	4-Methylmorpholine	>95%	38% (1.2 : 1)

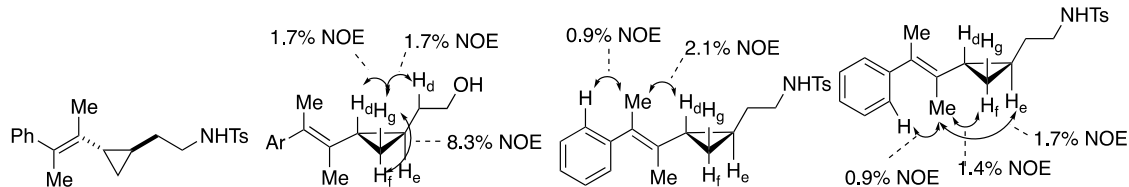
<sup>a</sup>Yield determined by <sup>1</sup>H NMR based on comparison to PhTMS as an internal standard.

#### 4.5.4 Characterization Data for Cyclopropane Products

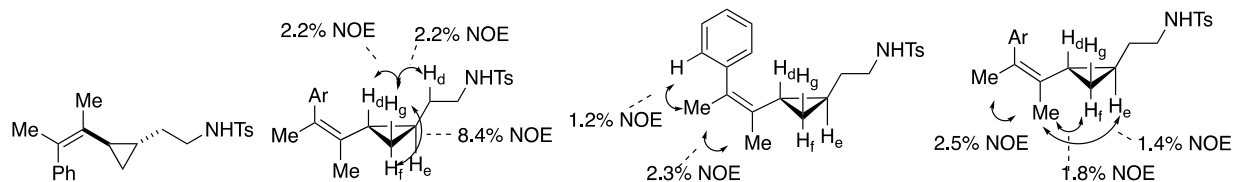
##### *N*-(2-*trans*-2-(3-phenylbut-2-en-2-yl)cyclopropyl)ethyl)4-methylbenzenesulfonamide (**4.4**):

was prepared according to Method A. The following amounts of reagents were used: piperidine **4.3** (37 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.8 mg, 5.0 μmol, 5.0 mol %), MeMgI (90. μL, 0.20 mmol, 2.3 M in Et<sub>2</sub>O, 2.0 equiv), PhMe (0.50 mL, 0.20 M in substrate). Before purification a <sup>1</sup>H NMR yield of 72% was obtained based on comparison to PhTMS as an internal standard. The residue was purified by column chromatography (0–10% EtOAc/hexanes) to afford the title compound a pale yellow oil (26 mg, 71. μmol, 71% yield, 1.5:1 *E:Z*). The ratio of alkene isomers was determined by integration of the resonances attributed to H<sub>g</sub> in the <sup>1</sup>H NMR spectrum. The relative configuration of the major (*E*)- and minor (*Z*)-**4.4** were assigned based on NOE analysis. A second column was performed and the diastereomers were separated. The alkene

isomers were characterized separately to demonstrate the stereochemical outcome of the reaction.

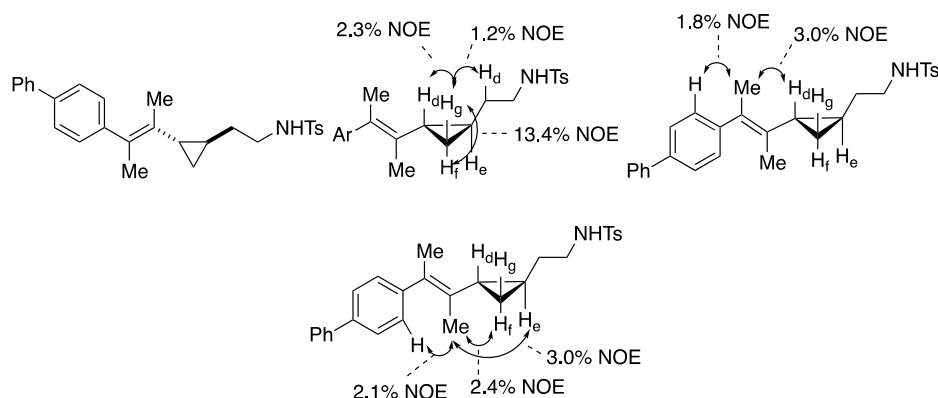


**Major Diastereomer:** TLC  $R_f = 0.6$  (25% EtOAc/hexanes);  $^1\text{H NMR}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.74 (d,  $J = 8.4$  Hz, 2H), 7.34–7.27 (m, 4H), 7.23–7.14 (m, 1H), 7.11–7.05 (m, 2H), 4.41 (s, 1H), 3.08 (qd,  $J = 6.7, 2.3$  Hz, 2H), 2.42 (s, 3H), 2.04 (d,  $J = 1.5$  Hz, 3H), 1.71–1.55 (m, 1H), 1.52–1.39 (m, 2H), 1.25 (d,  $J = 1.6$  Hz, 3H), 0.87–0.77 (m, 1H), 0.77–0.70 (m, 1H), 0.49 (dt,  $J = 9.3, 4.8$  Hz, 1H);  $^{13}\text{C NMR}$  ( $\text{CDCl}_3$ , 125.7 MHz)  $\delta$  145.5, 143.5, 137.0, 132.0, 129.8 (2C), 129.5, 128.4 (2C), 128.0 (2C), 127.2 (2C), 125.9, 76.8, 43.4, 34.3, 22.2, 21.6, 20.8, 16.0, 15.6, 11.7; **IR** (neat) 3284, 2925, 2860, 1599, 1324, 1160, 905, 728, 650  $\text{cm}^{-1}$ ; **HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M} + \text{Na}]^+$  calculated for  $\text{C}_{22}\text{H}_{27}\text{NO}_2\text{SNa}$ , 392.1660; found, 392.1667.



**Minor Diastereomer:** TLC  $R_f = 0.6$  (25% EtOAc/hexanes);  $^1\text{H NMR}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.70 (d,  $J = 8.1$  Hz, 2H), 7.28 (t,  $J = 7.8$  Hz, 5H), 7.15 (d,  $J = 7.9$  Hz, 2H), 4.30 (t,  $J = 6.2$  Hz, 1H), 2.98–2.83 (m, 2H), 2.42 (s, 3H), 1.96 (s, 3H), 1.43 (d,  $J = 1.5$  Hz, 3H), 1.30–1.15 (m, 3H), 0.69–0.63 (m, 1H), 0.59 (dt,  $J = 8.8, 5.0$  Hz, 1H), 0.16 (dt,  $J = 9.4, 4.9$  Hz, 1H);  $^{13}\text{C NMR}$  ( $\text{CDCl}_3$ , 125.7 MHz)  $\delta$  143.3, 131.3, 129.7 (2C), 129.5, 128.7 (2C), 128.5, 128.1 (2C), 127.8, 127.1 (2C), 125.9, 43.2, 33.7, 22.9, 21.6, 21.6, 15.6, 13.9, 11.6; **IR** (neat) 3284, 2925, 2860, 1599, 1324, 1160, 905, 728, 650  $\text{cm}^{-1}$ ; **HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M} + \text{Na}]^+$  calculated for  $\text{C}_{22}\text{H}_{27}\text{NO}_2\text{SNa}$ , 392.1660; found, 392.1667.

**Scale Up Reaction:** In a glovebox, an oven-dried 7 mL vial equipped with a stir bar was charged with piperidine **4.3** (0.37 g, 1.0 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (38 mg, 50. μmol, 5.0 mol %), and PhMe (5.0 mL, 0.2 M in substrate). Then MeMgI (0.80 mL, 2.0 mmol, 2.0 equiv, 2.5 M in Et<sub>2</sub>O) was then added dropwise via syringe. After 24 h, the reaction vial was removed from the glovebox, quenched with MeOH, filtered through a plug of silica gel eluting with Et<sub>2</sub>O, and concentrated in vacuo. The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a mixture of alkene diastereomers as a clear and colorless oil (230 mg, 0.62 mmol, 62% yield, 1.8:1 *E:Z*). Refer above to cyclopropane **4.4** for analytical data.

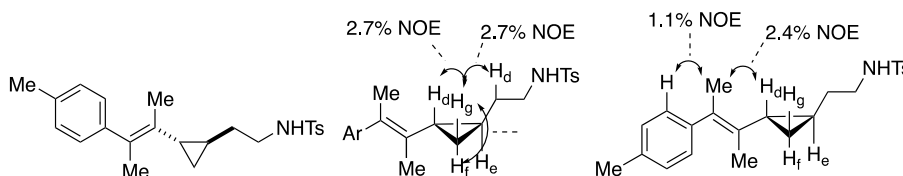


***N*-(2-(*trans*-2-(3-([1,1'-Biphenyl]-4-yl)but-2-en-2-yl)cyclopropyl)ethyl)-4-**

**methylbenzenesulfonamide (4.5)** was prepared according to Method A. The following amounts of reagents were used: piperidine **4.34** (45 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.8 mg, 50. μmol, 5.0 mol %), MeMgI (90. μL, 0.20 mmol, 2.3 M in Et<sub>2</sub>O, 2.0 equiv), PhMe (0.50 mL, 0.20 M in substrate). Before purification, a <sup>1</sup>H NMR yield of 70% and 2.8:1 *E:Z* was obtained based on comparison to PhTMS as an internal standard. The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a mixture of alkene diastereomers as a pale yellow solid (29 mg, 65 μmol, 64% yield, 2.5:1 *E:Z*). A second column



was performed and the diastereomers were separated. The major diastereomer is fully characterized below. The ratio of alkene isomers was determined by integration of the resonances attributed to H<sub>g</sub> in the <sup>1</sup>H NMR spectrum. The relative configuration of the major (*E*)-**4.5** was assigned based on NOE analysis. **m.p.** 134–137 °C; **TLC R<sub>f</sub>** = 0.2 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.78 (d, *J* = 8.4 Hz, 2H), 7.59 (d, *J* = 8.3 Hz, 2H), 7.53 (d, *J* = 8.2 Hz, 2H), 7.42 (t, *J* = 7.4 Hz, 2H), 7.34–7.28 (m, 3H), 7.16 (d, *J* = 8.2 Hz, 2H), 4.75 (t, *J* = 6.2 Hz, 1H), 3.07 (qd, *J* = 6.9 Hz, 1.3 Hz, 2H), 2.41 (s, 3H), 2.06 (q, *J* = 1.4 Hz, 3H), 1.69–1.57 (m, 1H), 1.51–1.39 (m, 2H), 1.31 (q, *J* = 1.4 Hz, 3H), 0.88–0.79 (m, 1H), 0.77–0.71 (m, 1H), 0.52–0.46 (m, 1H); **<sup>13</sup>C NMR** (125.8 MHz, CDCl<sub>3</sub>) δ 144.6, 143.5, 141.1, 138.7, 137.1, 131.5, 130.1, 129.8 (2C), 128.9 (2C), 128.8 (2C), 127.3 (2C), 127.2, 127.1 (2C), 126.8 (2C), 43.4, 34.4, 22.4, 21.6, 20.8, 16.2, 15.8, 11.9. **IR** (neat) 3304, 2922, 2861, 1597, 1318, 1151, 812, 727, 657 cm<sup>-1</sup>; **HRMS** (TOF MS ES+) *m/z*: [M + Na]<sup>+</sup> calculated for C<sub>28</sub>H<sub>31</sub>NO<sub>2</sub>SNa, 468.1973; found, 468.1978.



**4-methyl-*N*-(2-(*trans*-2-(3-(*p*-tolyl)but-2-en-2-yl)cyclopropyl)ethyl)benzenesulfonamide (4.6)**

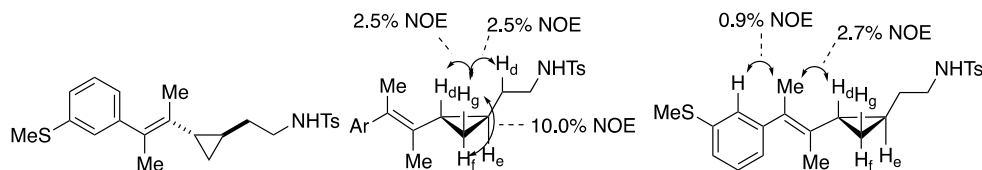
was prepared according to Method A. The following amounts of reagents were used: piperidine **4.49** (39 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.8 mg, 5.0 μmol, 5.0 mol %), MeMgI (90 μL, 0.20 mmol, 2.3 M in Et<sub>2</sub>O, 2.0 equiv), PhMe (0.50 mL, 0.20 M in substrate). Before purification, a <sup>1</sup>H NMR yield of 65% and 1.4:1 *E:Z* was obtained based on comparison to PhTMS as an internal standard. The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a mixture of alkene diastereomers as a clear yellow oil (27 mg, 0.070 mmol, 71% yield, 1.9:1 *E:Z*). The ratio of alkene isomers was determined by

integration of the resonances attributed to H<sub>g</sub> in the <sup>1</sup>H NMR spectrum. The relative configuration of the major (*E*)-**4.6** was assigned based on NOE analysis. For clarity, the <sup>1</sup>H NMR and <sup>13</sup>C NMR data of the major and minor diastereomers have been tabulated individually.

**TLC** R<sub>f</sub> = 0.5 (20% EtOAc/hexanes); **IR** (neat) 3280, 2923, 1599, 1485, 1325, 1238, 1160, 1039, 905, 729 cm<sup>-1</sup>; **HRMS** (TOF MS ES+) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>23</sub>H<sub>29</sub>NO<sub>2</sub>SNa, 406.1817; found, 406.1805.

**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.76 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 3H), 7.11 (d, *J* = 7.9 Hz, 2H), 6.98 (d, *J* = 7.9 Hz, 2H), 4.55 (t, *J* = 6.2 Hz, 1H), 3.07 (qd, *J* = 6.3, 2.2 Hz, 2H), 2.42 (s, 7H), 2.33 (s, 7H), 2.02 (s, 3H), 1.68–1.62 (m, 1H), 1.50–1.42 (m, 2H), 1.26 (s, 5H), 0.86–0.75 (m, 1H), 0.72 (dt, *J* = 9.2, 5.1 Hz, 1H), 0.47 (dt, *J* = 9.6, 5.0 Hz, 1H); <sup>13</sup>C NMR (125.7 MHz, CDCl<sub>3</sub>) δ 143.4, 142.5, 137.0, 135.4, 129.8 (2C), 128.8, 128.7 (2C), 128.6, 128.2 (2C), 127.2 (2C), 43.4, 34.3, 22.2, 21.6, 21.1, 20.8, 16.1, 15.6, 11.7.

**Minor Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.70 (d, *J* = 7.8 Hz, 2H), 7.28 (s, 2H), 7.08 (s, 2H), 7.04 (d, *J* = 7.6 Hz, 2H), 4.36 (t, *J* = 6.2 Hz, 1H), 2.91 (q, *J* = 6.7 Hz, 2H), 2.42 (s, 3H), 2.33 (s, 3H), 1.94 (s, 3H), 1.41 (s, 3H), 1.24–1.18 (m, 2H), 0.92–0.83 (m, 1H), 0.71–0.63 (m, 1H), 0.59 (dt, *J* = 9.0, 5.1 Hz, 1H), 0.16 (dt, *J* = 9.4, 4.9 Hz, 1H); <sup>13</sup>C NMR (125.7 MHz, CDCl<sub>3</sub>) δ <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 143.4, 142.2, 135.5, 129.9 (2C), 129.8 (2C), 129.3, 129.1, 128.9 (2C), 128.7 (2C), 127.3, 43.3, 33.8, 23.0, 21.8, 21.3, 20.1, 15.6, 13.8, 11.6.



#### 4-methyl-*N*-(2-(*trans*-2-(3-(3-(methylthio)phenyl)but-2-en-2-

*yl*)cyclopropyl)ethyl)benzenesulfonamide (**4.7**) was prepared according to Method A. The following amounts of reagents were used: piperidine **4.50** (42 mg, 0.10 mmol, 1.0 equiv), (*R*-

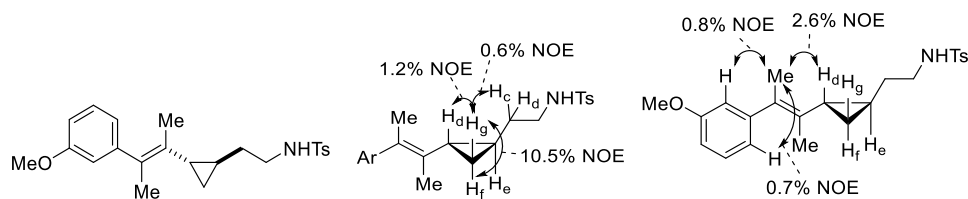
BINAP)NiCl<sub>2</sub> (3.8 mg, 5.0 μmol, 5.0 mol %), MeMgI (90. μL, 0.20 mmol, 2.3 M in Et<sub>2</sub>O, 2.0 equiv), PhMe (0.50 mL, 0.20 M in substrate). Before purification, a <sup>1</sup>H NMR yield of 65% and 2.8:1 *E:Z* was obtained based on comparison to PhTMS as an internal standard. The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a mixture of alkene diastereomers as a clear yellow oil (33 mg, 79 μmol, 79% yield, 2.4:1 *E:Z*). The ratio of alkene isomers was determined by integration of the resonances attributed to H<sub>g</sub> in the <sup>1</sup>H NMR spectrum. The relative configuration of the major product (*E*)-**4.7** was assigned based on NOE analysis. For clarity, the <sup>1</sup>H NMR and <sup>13</sup>C NMR data of the major and minor diastereomers have been tabulated individually.

**TLC** R<sub>f</sub> = 0.5 (20% EtOAc/hexanes); **IR** (neat) 3279, 2921, 1585, 1323, 1158, 1094, 905, 728 cm<sup>-1</sup>; **HRMS** (TOF MS ES+) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>23</sub>H<sub>29</sub>NO<sub>2</sub>S<sub>2</sub>Na, 438.1537; found, 438.1533.

**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.77 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 7.22 (t, *J* = 6.6 Hz, 1H), 7.09 (d, *J* = 7.7 Hz, 1H), 6.99 (s, 1H), 6.86 (d, *J* = 7.6 Hz, 1H), 4.57 (t, *J* = 6.2 Hz, 1H), 3.14–3.01 (m, 2H), 2.47 (s, 3H), 2.42 (s, 3H), 2.02 (s, 3H), 1.71–1.61 (m, 1H), 1.51–1.46 (m, 2H), 1.25 (s, 3H), 0.87–0.77 (m, 1H), 0.73 (dt, *J* = 8.4, 5.1 Hz, 1H), 0.49 (dt, *J* = 9.5, 5.0 Hz, 1H); <sup>13</sup>C NMR (125.7 MHz, CDCl<sub>3</sub>) δ 146.2, 143.5, 137.9, 137.0, 131.5, 130.1, 129.8 (2C), 128.5, 127.2 (2C), 126.5, 125.2, 124.1, 43.4, 34.3, 22.1, 21.6, 20.7, 16.0, 15.9, 15.7, 11.7.

**Minor Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.71 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.3 Hz, 2H), 7.20 (d, *J* = 5.2 Hz, 1H), 7.06 (s, 1H), 6.99 (s, 1H), 6.92 (d, *J* = 7.6 Hz, 1H), 4.40 (t, *J* = 6.1 Hz, 1H), 2.91 (q, *J* = 6.7 Hz, 2H), 2.47 (s, 3H), 2.42 (s, 3H), 1.94 (s, 3H), 1.42 (s, 3H), 1.31 (dt, *J* = 13.8, 6.8 Hz, 1H), 1.20–1.13 (m, 2H), 0.70–0.64 (m, 1H), 0.59 (dt, *J* = 8.4, 5.1 Hz, 1H), 0.18 (dt, *J* = 9.5, 5.0 Hz, 1H); <sup>13</sup>C NMR (125.7 MHz, CDCl<sub>3</sub>) δ 145.7, 143.3, 137.1, 130.8, 130.0,

129.7 (2C), 128.6, 127.1 (2C), 127.1, 126.9, 125.6, 124.0, 43.1, 33.6, 23.0, 21.5, 20.6, 15.8, 15.7, 13.9, 11.7.



***N*-(2-(*trans*-2-(3-(3-Methoxyphenyl)but-2-en-2-yl)cyclopropyl)ethyl)-4-**

**methylbenzenesulfonamide (4.8)**<sup>57</sup> was prepared according to Method A. The following amounts of reagents were used: piperidine **4.51** (40.4 mg, 0.100 mmol, 1.00 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.8 mg, 5.0 μmol, 5.0 mol %), MeMgI (71 μL, 0.20 mmol, 2.8 M in Et<sub>2</sub>O, 2.0 equiv), PhMe (0.50 mL, 0.20 M in substrate). Before purification, a <sup>1</sup>H NMR yield of 47% and 2.2:1 *E:Z* was obtained based on comparison to PhTMS as an internal standard. The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a mixture of alkene diastereomers as a clear colorless oil (18 mg, 46 μmol, 46% yield, 1.5:1 *E:Z*). The ratio of alkene isomers was determined by integration of the resonances attributed to H<sub>g</sub> in the <sup>1</sup>H NMR spectrum. The relative configuration of the major (*E*)-**4.8** was assigned based on NOE analysis. For clarity, the <sup>1</sup>H NMR and <sup>13</sup>C NMR data of the major and minor diastereomers have been tabulated individually.

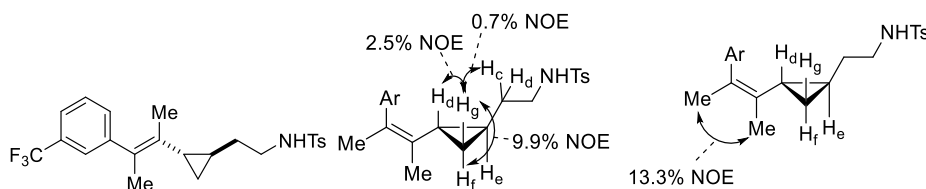
**TLC** R<sub>f</sub> = 0.4 (20% EtOAc/hexanes); **IR** (neat) 3279, 2921, 1597, 1575, 1320, 1157, 1093, 1043 cm<sup>-1</sup>; **HRMS** (TOF MS ES+) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>23</sub>H<sub>29</sub>NO<sub>3</sub>SNa, 422.1766; found, 422.1760.

**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.77 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 7.25–7.17 (m, 1H), 6.78–6.70 (m, 1H), 6.68 (d, *J* = 7.6 Hz, 1H), 6.64 (as, 1H), 4.63 (t, *J*

<sup>57</sup> Cyclopropane **4.8** synthesized by Nadia Hirbawi.

= 5.9 Hz, 1H), 3.79 (s, 3H), 3.07 (qd,  $J = 6.6, 3.0$  Hz, 2H), 2.42 (s, 3H), 2.02 (as, 3H), 1.69–1.55 (m, 1H), 1.50–1.48 (m, 2H), 1.26 (as, 3H), 0.87–0.77 (m, 1H), 0.72 (dt,  $J = 8.5, 5.0$  Hz, 1H), 0.48 (dt,  $J = 8.7, 5.0$  Hz, 1H);  $^{13}\text{C}$  NMR (125.9 MHz,  $\text{CDCl}_3$ )  $\delta$  159.38, 147.1, 143.5, 137.0, 131.9, 129.8 (2C), 129.66, 129.03, 127.19 (2C), 120.9, 114.1, 111.3, 55.2, 43.4, 34.3, 22.1, 21.6 (2C), 16.0, 15.64, 11.7.

**Minor Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.71 (d,  $J = 8.3$  Hz, 2H), 7.29 (d,  $J = 8.3$  Hz, 2H), 7.25–7.17 (m, 1H), 6.78–6.70, (m, 1H), 6.68 (d,  $J = 7.6$  Hz, 1H), 6.64 (as, 1H), 4.45 (t,  $J = 5.8$  Hz, 1H), 3.79 (s, 3H), 2.91 (q,  $J = 6.6$  Hz, 2H), 2.42 (s, 3H), 1.95 (as, 3H), 1.41 (as, 3H), 1.33–1.23 (m, 1H), 1.23–1.12 (m, 2H), 0.69–0.61 (m, 1H), 0.59 (dt,  $J = 8.5, 5.0$  Hz, 1H), 0.18 (dt,  $J = 8.8, 5.0$  Hz, 1H);  $^{13}\text{C}$  NMR (125.9 MHz,  $\text{CDCl}_3$ )  $\delta$  159.42, 146.6, 143.3, 137.1, 131.1, 129.71 (2C), 129.6, 129.07, 127.15 (2C), 121.3, 114.4, 111.4, 55.2, 43.2, 33.7, 22.9, 20.7 (2C), 15.56, 13.8, 11.5.



***N*-(2-(*trans*-2-(3-(3-Trifluoromethylphenyl)but-2-en-2-yl)cyclopropyl)ethyl)-4-**

**methylbenzenesulfonamide (4.9)**<sup>58</sup> was prepared according to Method A. The following amounts of reagents were used: piperidine **4.52** (44 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.8 mg, 5.0  $\mu\text{mol}$ , 5.0 mol %), MeMgI (80.  $\mu\text{L}$ , 0.20 mmol, 2.5 M in Et<sub>2</sub>O, 2.0 equiv), PhMe (0.50 mL, 0.20 M in substrate). Before purification, a  $^1\text{H}$  NMR yield of 47% and 1.4:1 *E:Z* was obtained based on comparison to PhTMS as an internal standard. The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a mixture of alkene

<sup>58</sup> Cyclopropane **4.9** synthesized by Nadia Hirbawi.

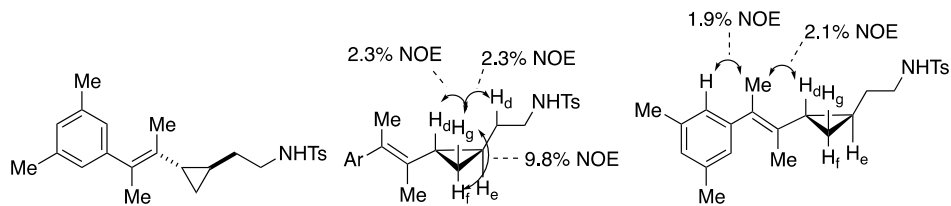
diastereomers as a clear yellow oil (20. mg, 46  $\mu$ mol, 46% yield, 1.3:1 *E*:*Z*). The ratio of alkene isomers was determined by integration of the resonances attributed to H<sub>g</sub> in the <sup>1</sup>H NMR spectrum. Due to overlapping peaks of the major (*E*)-**4.9**, the relative configuration of the minor (*Z*)-**4.9** was assigned based on NOE analysis. For clarity, the <sup>1</sup>H NMR and <sup>13</sup>C NMR data of the major and minor diastereomers have been tabulated individually.

**TLC** R<sub>f</sub> = 0.5 (25% EtOAc/hexanes); **IR** (neat) 3278, 2923, 1331, 1159, 1122, 1094, 1072 cm<sup>-1</sup>;

**HRMS** (TOF MS ES+) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>23</sub>H<sub>26</sub>F<sub>3</sub>NO<sub>2</sub>SNa, 460.1534; found, 460.1545.

**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.77 (d, *J* = 8.1 Hz, 2H), 7.46–7.38 (m, 3H), 7.36–7.33 (m, 1H), 7.29 (d, *J* = 8.1 Hz, 2H), 4.61 (t, *J* = 5.8 Hz, 1H), 3.07 (q, *J* = 6.7 Hz, 2H), 2.42 (s, 3H), 2.09–2.02 (m, 3H), 1.63 (quin, *J* = 6.9 Hz, 1H), 1.50–1.40 (m, 2H), 1.23 (s, 3H), 0.89–0.80 (m, 1H), 0.77–0.71 (m, 1H), 0.51 (dt, *J* = 8.7, 4.7 Hz, 1H); <sup>13</sup>C NMR (150.9 MHz, CDCl<sub>3</sub>)  $\delta$  146.2, 143.5, 136.94, 131.8, 131.1, 130.6, 130.4 (q, *J* = 31.7 Hz), 129.73 (2C), 128.50, 127.14 (2C), 125.1 (q, *J* = 3.7 Hz), 124.3 (q, *J* = 272.4 Hz), 122.67 (q, *J* = 3.6 Hz), 43.3, 34.3, 22.1, 21.50, 20.6, 16.0, 15.7, 11.78; <sup>19</sup>F NMR (564.7 MHz, CDCl<sub>3</sub>)  $\delta$  –62.53 (3F).

**Minor Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.71 (d, *J* = 8.2 Hz, 2H), 7.46–7.38 (m, 3H), 7.36–7.33 (m, 1H), 7.31 (d, *J* = 8.1 Hz, 2H), 4.43 (t, *J* = 6.0 Hz, 1H), 2.91 (q, *J* = 6.7, 2H), 2.42 (s, 3H), 1.97 (s, 3H), 1.45 (s, 3H), 1.35 (quin, *J* = 6.8 Hz, 1H), 1.16–1.05 (m, 2H), 0.73–0.67 (m, 1H), 0.59 (dt, *J* = 8.4, 5.2 Hz, 1H), 0.20 (dt, *J* = 8.8, 4.9 Hz, 1H); <sup>13</sup>C NMR (150.9 MHz, CDCl<sub>3</sub>)  $\delta$  145.7, 143.4, 136.96, 132.1 (s), 131.2, 130.3 (q, *J* = 32.1 Hz), 129.8, 129.69 (2C), 128.53, 127.08 (2C), 125.6 (q, *J* = 3.7 Hz), 124.3 (q, *J* = 272.4 Hz), 122.65 (q, *J* = 3.6 Hz), 43.1, 33.5, 22.9, 21.51, 21.3, 15.8, 14.0, 11.82; <sup>19</sup>F NMR (564.7 MHz, CDCl<sub>3</sub>)  $\delta$  –62.49 (3F).



***N*-(2-(*trans*-2-(3-(3,5-dimethylphenyl)but-2-en-2-yl)cyclopropyl)ethyl)-4-**

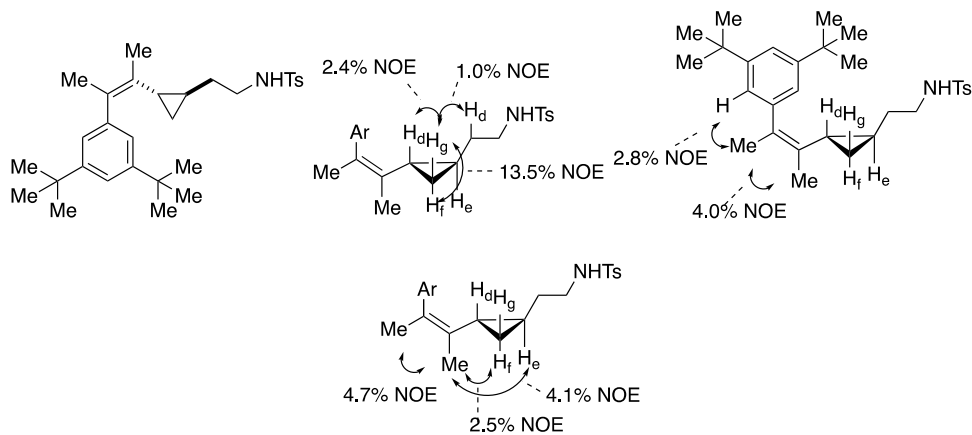
**methylbenzenesulfonamide (4.10)** was prepared according to Method A. The following amounts of reagents were used: piperidine **4.53** (50 mg, 0.12 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (4.6 mg, 6.0 μmol, 5.0 mol %), MeMgI (0.10 mL, 0.24 mmol, 2.5 M in Et<sub>2</sub>O, 2.0 equiv), PhMe (0.60 mL, 0.20 M in substrate). The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a mixture of alkene diastereomers as a clear yellow oil (32 mg, 80. μmol, 68% yield, 2.0:1 *E:Z*). The ratio of alkene isomers was determined by integration of the resonances attributed to H<sub>g</sub> in the <sup>1</sup>H NMR spectrum. The relative configuration of the major (*E*)-**4.10** was assigned based on NOE analysis. For clarity, the <sup>1</sup>H NMR and <sup>13</sup>C NMR data of the major and minor diastereomers have been tabulated individually.

**TLC** R<sub>f</sub> = 0.4 (20% EtOAc/hexanes); **IR** (neat) 3280, 2922, 2860, 1598, 1323, 1184, 907, 724 cm<sup>-1</sup>; **HRMS** (TOF MS ES<sup>+</sup>) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>24</sub>H<sub>31</sub>NO<sub>2</sub>SNa, 398.2154; found, 398.2155.

**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.77 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 6.83 (s, 1H), 6.70 (s, 2H), 4.50 (t, *J* = 6.3 Hz, 1H), 3.07 (qd, *J* = 6.5, 2.9 Hz, 2H), 2.42 (s, 3H), 2.29 (s, 6H), 2.00 (s, 3H), 1.67–1.59 (m, 1H), 1.49–1.41 (m, 2H), 1.25 (d, *J* = 1.9 Hz, 3H), 0.84–0.75 (m, 1H), 0.71 (dt, *J* = 8.9, 5.0 Hz, 1H), 0.47 (dt, *J* = 9.5, 4.9 Hz, 1H); <sup>13</sup>C NMR (125.7 MHz, CDCl<sub>3</sub>) δ 145.6, 143.5, 137.5, 132.3, 129.8 (2C), 129.2, 127.6 (2C), 127.3 (2C), 126.6, 126.2 (2C), 43.5, 34.4, 22.2, 21.7, 21.5 (2C), 20.9, 16.2, 15.7, 11.8.

**Minor Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.70 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 7.9 Hz, 2H), 6.83 (s, 1H), 6.76 (s, 2H), 4.32 (t, *J* = 6.1 Hz, 1H), 2.92 (q, *J* = 6.8 Hz, 2H), 2.42 (s, 3H),

2.28 (s, 6H), 1.94 (s, 3H), 1.40 (s, 3H), 1.33–1.26 (m, 1H), 1.23–1.15 (m, 2H), 0.69–0.63 (m, 1H), 0.59 (dt,  $J = 8.9, 5.1$  Hz, 1H), 0.16 (dt,  $J = 9.5, 4.9$  Hz, 1H);  $^{13}\text{C}$  NMR (125.7 MHz,  $\text{CDCl}_3$ )  $\delta$  145.1, 143.4, 137.6, 137.1, 131.5, 129.8 (2C), 129.1, 127.6 (2C), 127.2 (2C), 126.2 (2C), 43.3, 33.7, 23.1, 21.7, 21.6, 20.9, 15.6, 13.8, 11.6.



***N*-(2-(*trans*-2-(3-(3,5-di-*tert*-butylphenyl)but-2-en-2-yl)cyclopropyl)ethyl)-4-**

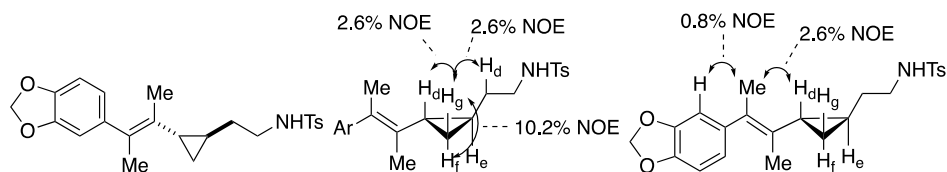
**methylbenzenesulfonamide (4.11)** was prepared according to Method A. The following amounts of reagents were used: piperidine **4.54** (44 mg, 90.  $\mu\text{mol}$ , 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.4 mg, 4.5  $\mu\text{mol}$ , 5.0 mol %), MeMgI (80.  $\mu\text{L}$ , 0.18 mmol, 2.3 M in Et<sub>2</sub>O, 2.0 equiv), PhMe (0.45 mL, 0.20 M in substrate). Before purification, a  $^1\text{H}$  NMR yield of 56% and 2.3:1 *E:Z* was obtained based on comparison to PhTMS as an internal standard. The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a mixture of alkene diastereomers as a clear yellow oil (24 mg, 50.  $\mu\text{mol}$ , 56% yield, 1:2.6 *E:Z*). The ratio of alkene isomers was determined by integration of the resonances attributed to H<sub>g</sub> in the  $^1\text{H}$  NMR spectrum. The relative configuration of the major (*Z*)-**4.11** was assigned based on NOE analysis. For clarity, the  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR data of the major and minor diastereomers have been tabulated individually.



**TLC R<sub>f</sub>** = 0.6 (20% EtOAc/hexanes); **IR** (neat) 3277, 2962, 2865, 1591, 1324, 1159, 1094, 907, 730 cm<sup>-1</sup>; **HRMS** (TOF MS ES<sup>+</sup>) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>30</sub>H<sub>43</sub>NO<sub>2</sub>SNa, 504.2919; found, 504.2912.

**Major (Z) Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.77 (d, *J* = 8.0 Hz, 2H), 7.33 – 7.30 (m, 2H), 7.07 (d, *J* = 1.8 Hz, 2H), 7.00 (d, *J* = 1.8 Hz, 1H), 4.43 (s, 1H), 2.99 (q, *J* = 6.6 Hz, 2H), 2.48 (d, *J* = 2.9 Hz, 3H), 2.05 (s, 3H), 1.59–1.51 (m, 1H), 1.49 (s, 3H), 1.37 (s, 18H), 1.33–1.32 (m, 1H), 1.08 (dq, *J* = 14.5, 7.2 Hz, 1H), 0.76 (tt, *J* = 8.3, 5.2 Hz, 1H), 0.64 (dt, *J* = 8.2, 5.1 Hz, 1H), 0.22 (dt, *J* = 9.5, 5.0 Hz, 1H); <sup>13</sup>C NMR (125.7 MHz, CDCl<sub>3</sub>) δ 150.2, 144.1, 143.4, 137.2, 132.5, 129.8 (2C), 129.0, 127.2 (2C), 123.1 (2C), 119.8 (2C), 43.4, 34.9, 33.8, 31.7 (6C), 23.0, 21.9, 21.7, 15.7 (2C), 13.9, 11.7.

**Minor (E) Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.84 (d, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.07 (d, *J* = 1.8 Hz, 2H), 7.00 (d, *J* = 1.8 Hz, 1H), 4.60 (d, *J* = 7.0 Hz, 1H), 3.15 (hept, *J* = 6.4 Hz, 2H), 2.48 (s, 3H), 2.12 (d, *J* = 1.9 Hz, 3H), 1.76–1.67 (m, 1H), 1.59–1.50 (m, 1H), 1.39 (s, 18H), 1.33 (s, 3H), 1.05–0.85 (m, 2H), 0.85–0.78 (m, 1H), 0.55 (dt, *J* = 9.4, 5.0 Hz, 1H); <sup>13</sup>C NMR (125.7 MHz, CDCl<sub>3</sub>) δ 150.1, 144.4, 143.4, 137.1, 132.4, 129.7 (2C), 129.0, 127.2 (2C), 127.1, 122.6 (2C), 119.6 (2C), 43.4, 34.9, 34.3, 31.6, 31.5 (6C), 22.2, 20.9, 16.1, 15.6, 11.7.



***N*-(2-(*trans*-2-(3-(benzo[*d*][1,3]dioxol-5-yl)but-2-en-2-yl)cyclopropyl)ethyl)-4-**

**methylbenzenesulfonamide (4.12)** was prepared according to Method A. The following amounts of reagents were used: piperidine **4.55** (32 mg, 80. μmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (2.9 mg, 3.8 μmol, 5.0 mol %), MeMgI (70. μL, 0.16 mmol, 2.3 M in Et<sub>2</sub>O, 2.0 equiv), PhMe (0.40 mL,

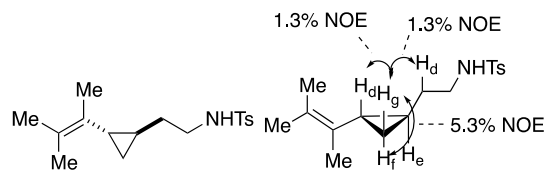
0.20 M in substrate). Before purification, a  $^1\text{H}$  NMR yield of 55% and 2.6:1 *E:Z* was obtained based on comparison to PhTMS as an internal standard. The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a mixture of alkene diastereomers as a clear pale yellow oil (19 mg, 45  $\mu\text{mol}$ , 57% yield, 2.8:1 *E:Z*). The ratio of alkene isomers was determined by integration of the resonances attributed to  $\text{H}_g$  in the  $^1\text{H}$  NMR spectrum. The relative configuration of the major (*E*)-**4.12** was assigned based on NOE analysis. For clarity, the  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR data of the major and minor diastereomers have been tabulated individually.

**TLC**  $R_f$  = 0.7 (20% EtOAc/hexanes); **IR** (neat) 3276, 2922, 1598, 1323, 1094, 903, 726  $\text{cm}^{-1}$ ;

**HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{23}\text{H}_{27}\text{NO}_4\text{SNa}$ , 436. 1559; found, 436.1570.

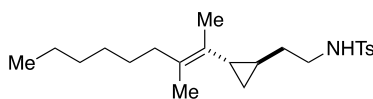
**Major Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.76 (d,  $J$  = 8.0 Hz, 2H), 7.31 (d,  $J$  = 7.8 Hz, 2H), 6.75 (d,  $J$  = 8.0, 1H), 6.58 (s, 1H), 6.53 (d,  $J$  = 7.9 Hz, 1H), 5.93 (s, 2H), 4.58 (t,  $J$  = 6.1 Hz, 1H), 3.14–3.00 (m, 2H), 2.42 (s, 3H), 1.99 (d,  $J$  = 1.7 Hz, 3H), 1.72–1.54 (m, 1H), 1.48–1.40 (m, 2H), 1.27–1.25 (m, 3H), 0.85–0.75 (m, 1H), 0.74–0.66 (m, 1H), 0.47 (dt,  $J$  = 9.5, 4.9 Hz, 1H);  $^{13}\text{C}$  NMR (125.7 MHz,  $\text{CDCl}_3$ )  $\delta$  147.2, 145.5, 143.4, 139.5, 137.0, 131.5, 129.8 (2C), 127.2 (2C), 121.4, 109.0, 108.0, 100.8, 43.4, 34.3, 22.9, 22.2, 21.6, 20.9, 16.1, 15.7, 11.7.

**Minor Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.71 (d,  $J$  = 8.2 Hz, 2H), 7.34–7.30 (m, 2H), 6.71 (s, 1H), 6.65 (d,  $J$  = 1.7 Hz, 1H), 6.60 (d,  $J$  = 1.6 Hz, 1H), 5.94 (s, 2H), 4.37 (t,  $J$  = 6.3 Hz, 1H), 2.92 (q,  $J$  = 6.7 Hz, 2H), 2.42 (s, 3H), 1.92–1.90 (m, 3H), 1.39 (s, 3H), 1.35–1.27 (m, 2H), 1.25–1.17 (m, 1H), 0.85–0.75 (m, 1H), 0.62–0.56 (m, 1H), 0.19 (dt,  $J$  = 9.4, 4.9 Hz, 1H);  $^{13}\text{C}$  NMR (125.7 MHz,  $\text{CDCl}_3$ )  $\delta$  147.3, 145.6, 143.4, 139.6, 139.0, 130.7, 129.7 (2C), 127.1 (2C), 121.7, 109.3, 108.9, 108.0, 100.8, 43.2, 33.7, 22.9, 21.7, 20.6, 15.5, 13.8, 11.6.



**4-methyl-N-(2-(*trans*-2-(3-methylbut-2-en-2-yl)cyclopropyl)ethyl)benzenesulfonamide (4.13)**

was prepared according to Method A. The following amounts of reagents were used: piperidine **4.28** (24 mg, 80.  $\mu\text{mol}$ , 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (2.9 mg, 4.0  $\mu\text{mol}$ , 5.0 mol %), MeMgI (70.  $\mu\text{L}$ , 0.16 mmol, 2.3 M in Et<sub>2</sub>O, 2.0 equiv), PhMe (0.40 mL, 0.20 M in substrate). The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a clear yellow oil (15 mg, 49  $\mu\text{mol}$ , 60% yield, >20:1 dr). The dr was based on the integration of the resonances attributed to H<sub>g</sub> in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **TLC** R<sub>f</sub> = 0.3 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.74 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 8.2 Hz, 2H), 4.51 (s, 1H), 3.03 (ddt, *J* = 12.8, 10.3, 6.1 Hz, 2H), 2.43 (s, 3H), 1.73 (d, *J* = 1.5 Hz, 3H), 1.65 (s, 3H), 1.60 (d, *J* = 4.0 Hz, 1H), 1.53 (td, *J* = 14.0, 7.1 Hz, 1H), 1.39 (dq, *J* = 13.9, 6.9 Hz, 1H), 1.32 (s, 3H), 0.69–0.62 (m, 1H), 0.59 (dt, *J* = 8.3, 5.0 Hz, 1H), 0.36 (dt, *J* = 9.3, 4.7 Hz, 1H); **<sup>13</sup>C NMR** (125.7 MHz, CDCl<sub>3</sub>)  $\delta$  143.5, 137.1, 129.81 (2C), 129.78, 127.3 (2C), 126.0, 43.5, 34.3, 22.0, 21.7, 21.3, 20.5, 15.4, 14.6, 11.6; **IR** (neat) 3279, 2921, 2859, 1598, 1324, 1159, 1094, 905, 729 cm<sup>-1</sup>; **HRMS** (TOF MS ES<sup>+</sup>) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>17</sub>H<sub>25</sub>NO<sub>2</sub>SNa, 330.1504; found, 330.1500.



**4-methyl-N-(2-(*trans*-2-(3-methylnon-2-en-2-yl)cyclopropyl)ethyl)benzenesulfonamide**

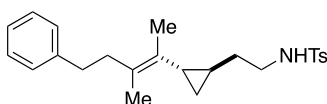
**(4.14)** was prepared according to Method A. The following amounts of reagents were used: piperidine **4.56** (42 mg, 0.11 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (4.1 mg, 5.5  $\mu\text{mol}$ , 5.0 mol %), MeMgI (0.10 mL, 0.22 mmol, 2.3 M in Et<sub>2</sub>O, 2.0 equiv), PhMe (0.55 mL, 0.20 M in substrate).

Before purification, a  $^1\text{H}$  NMR yield of 60% was obtained based on comparison to PhTMS as an internal standard. The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a mixture of alkene diastereomers as a clear yellow oil (35 mg, 0.093 mmol, 83% yield, 2:1 *E:Z*). The ratio of alkene isomers was determined by integration of the resonances in the  $^{13}\text{C}$  NMR spectrum. The relative configuration of the major (*E*)-**4.14** was assigned based on analogy to cyclopropane **4.4**.

**TLC**  $R_f$  = 0.2 (20% EtOAc/hexanes); **IR** (neat) 3278, 2924, 2856, 1598, 1455, 1324, 1158, 1094, 841, 660  $\text{cm}^{-1}$ ; **HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{22}\text{H}_{35}\text{NO}_2\text{SNa}$ , 400.2286; found, 400.2289.

**Major Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75 (d,  $J$  = 8.1 Hz, 2H), 7.30 (d,  $J$  = 8.1 Hz, 2H), 4.54 (t,  $J$  = 6.5 Hz, 1H), 3.09–2.96 (m, 2H), 2.43 (s, 3H), 2.10 (td,  $J$  = 7.1, 3.3 Hz, 1H), 1.98 (td,  $J$  = 7.0, 2.2 Hz, 1H), 1.62 (d,  $J$  = 1.0 Hz, 3H), 1.6–1.47 (m, 1H), 1.42–1.19 (m, 13H), 0.88 (t,  $J$  = 6.6 Hz, 3H), 0.73–0.62 (m, 1H), 0.62–0.55 (m, 1H), 0.40–0.30 (m, 1H);  $^{13}\text{C}$  NMR (125.7 MHz,  $\text{CDCl}_3$ )  $\delta$  143.3, 137.1, 130.4, 129.7 (2C), 127.2 (2C), 126.1, 43.3, 35.3, 34.3, 34.3, 31.9, 29.5, 28.4, 22.7, 21.6, 19.3, 15.1, 14.3, 14.2, 11.4.

**Minor Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75 (d,  $J$  = 8.1 Hz, 2H), 7.30 (d,  $J$  = 8.1 Hz, 2H), 4.54 (t,  $J$  = 6.5 Hz, 1H), 3.09–2.96 (m, 2H), 2.43 (s, 3H), 2.10 (td,  $J$  = 7.1, 3.3 Hz, 1H), 1.98 (td,  $J$  = 7.0, 2.2 Hz, 1H), 1.70 (d,  $J$  = 1.3 Hz, 3H), 1.61–1.47 (m, 1H), 1.42–1.19 (m, 13H), 0.88 (t,  $J$  = 6.6 Hz, 3H), 0.73–0.62 (m, 1H), 0.62–0.55 (m, 1H), 0.40–0.30 (m, 1H);  $^{13}\text{C}$  NMR (125.7 MHz,  $\text{CDCl}_3$ )  $\delta$  143.3, 137.0, 130.7, 129.7 (2C), 127.2 (2C), 126.1, 43.4, 35.3, 34.2, 31.9, 29.4, 28.2, 22.7, 22.1, 21.4, 18.5, 15.5, 14.2, 14.2, 11.6.



**4-Methyl-*N*-(2-(*trans*-2-(3-methyl-5-phenylpent-2-en-2-**

**yl)cyclopropyl)ethyl)benzenesulfonamide (4.15)**<sup>59</sup> was prepared according to Method A. The following amounts of reagents were used: piperidine **4.57** (40. mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.8 mg, 5.0 μmol, 5.0 mol %), MeMgI (90. μL, 0.20 mmol, 2.0 equiv), and PhMe (0.5 mL, 0.2 M in substrate). The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a clear oil (26 mg, 0.64 mmol, 64% yield, 2.2:1 *E:Z*). The ratio of alkene isomers was determined by integration of the resonances in the <sup>13</sup>C NMR spectrum. The relative configuration of the major (*E*)-**4.15** was assigned based on analogy to cyclopropane **4.4**. For clarity, the <sup>1</sup>H NMR and <sup>13</sup>C NMR data of the major and minor diastereomers have been tabulated individually.

**TLC R<sub>f</sub>** = 0.5 (25% EtOAc/hexanes); **IR** (neat) 3276, 3062, 3026, 2998, 2924, 2860, 1599 cm<sup>-1</sup>;

**HRMS** (TOF MS ES<sup>+</sup>) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>24</sub>H<sub>31</sub>NO<sub>2</sub>SNa, 420.1973; found, 420.1975.

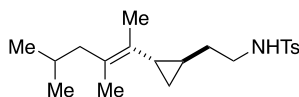
**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.76–7.72 (m, 2H), 7.31–7.24 (m, 4H), 7.17–7.14 (m, 3H), 4.58–4.52 (m, 1H), 3.04–2.96 (m, 2H), 2.68–2.60 (m, 2H), 2.43–2.41 (m, 2H), 2.39 (s, 3H), 1.68 (s, 3H), 1.55–1.48 (m, 1H), 1.30 (s, 3H), 1.28–1.20 (m, 2H), 0.67–0.62 (m, 1H), 0.58–0.53 (m, 1H), 0.32–0.28 (m, 1H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 143.4, 142.6, 137.0, 129.7 (2C), 129.3, 128.4 (2C), 128.3 (2C), 127.3, 127.2 (2C), 125.8, 43.3, 36.6, 34.8, 34.3, 21.6, 21.4, 19.3, 15.2, 14.5, 11.5.

**Minor Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.76–7.72 (m, 2H), 7.31–7.24 (m, 4H), 7.17–7.14 (m, 3H), 4.58–4.52 (m, 1H), 3.04–2.96 (m, 2H), 2.68–2.60 (m, 2H), 2.39 (s, 3H), 2.31–2.27 (m, 2H), 1.75 (s, 3H), 1.40–1.36 (m, 1H), 1.28–1.20 (m, 2H), 1.23 (s, 3H), 0.67–0.62 (m, 1H), 0.58–0.53 (m, 1H), 0.38–0.34 (m, 1H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 143.4, 142.5, 137.0,

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<sup>59</sup> Cyclopropane **4.15** was synthesized by Taylor A. Thane

129.8 (2C), 129.3, 128.5 (2C), 128.3 (2C), 127.4, 127.2 (2C), 125.7, 43.4, 37.5, 34.5, 34.3, 22.1, 21.6, 18.6, 15.5, 14.1, 11.7.



***N*-(2-(*trans*-2-(3,5-dimethylhex-2-en-2-yl)cyclopropyl)ethyl)-4-methylbenzenesulfonamide**

**(4.16)**<sup>60</sup> was prepared according to Method A. The following amounts of reagents were used: piperidine **4.58** (35 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.8 mg, 5.0 μmol, 5.0 mol %), MeMgI (90. μL, 0.20 mmol, 2.0 equiv), and PhMe (0.5 mL, 0.2 M in substrate). The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a clear oil (23 mg, 0.66 mmol, 66% yield, 3:1 *E*:*Z*). The ratio of alkene isomers was determined by integration of the resonances in the <sup>13</sup>C NMR spectrum. The relative configuration of the major (*E*)-**4.16** was assigned based on analogy to cyclopropane **4.4**.

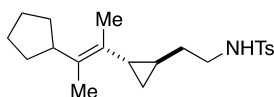
**TLC** R<sub>f</sub> = 0.5 (25% EtOAc/hexanes); **IR** (neat) 3279, 3056, 2951, 2924, 2866 cm<sup>-1</sup>; **HRMS** (TOF MS ES<sup>+</sup>) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>20</sub>H<sub>31</sub>NO<sub>2</sub>SNa, 372.1973; found, 372.1969.

**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.74 (d, *J* = 7.9 Hz, 2H), 8.30 (d, *J* = 7.9 Hz, 2H), 5.50 (t, *J* = 6.2 Hz, 1H), 4.01 (q, *J* = 6.7 Hz, 2H), 3.01 (d, *J* = 7.6 Hz, 2H), 2.77–2.71 (m, 1H), 2.61 (s, 3H), 2.58–2.53 (m, 1H), 2.38–2.30 (m, 2H), 2.27 (s, 3H), 1.89–1.79 (m, 6H), 1.69–1.67 (m, 1H), 1.61–1.58 (m, 1H), 1.34–1.31 (m, 1H), 0.73–0.64 (m, 1H), 0.61–0.58 (m, 1H), 0.34–0.31 (m, 1H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 143.4, 137.2, 129.8 (2C), 129.4, 127.3 (2C), 127.2, 43.43, 43.41, 34.3 (2C), 27.5 (2C), 22.81, 22.76, 21.75, 21.65, 15.3, 11.5.

**Minor Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.74 (d, *J* = 7.9 Hz, 2H), 8.30 (d, *J* = 7.9 Hz, 2H), 5.50 (t, *J* = 6.2 Hz, 1H), 4.01 (q, *J* = 6.7 Hz, 2H), 2.90 (d, *J* = 7.5 Hz, 2H), 2.77–2.71 (m,

<sup>60</sup> Cyclopropane **4.16** was synthesized by Taylor A. Thane

1H), 2.69 (s, 3H), 2.58–2.53 (m, 1H), 2.38–2.30 (m, 2H), 2.33 (s, 3H), 1.89–1.79 (m, 6H), 1.69–1.67 (m, 1H), 1.61–1.58 (m, 1H), 1.37–1.34 (m, 1H), 0.73–0.64 (m, 1H), 0.61–0.58 (m, 1H), 0.39–0.35 (m, 1H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 143.4, 137.2, 129.8 (2C), 129.4, 127.3 (2C), 127.2, 44.4, 43.5, 34.4 (2C), 22.7, 22.6, 22.3, 21.8, 19.1, 15.7, 14.9, 11.8.



***N*-(2-(*trans*-2-(3-cyclopentylbut-2-en-2-yl)cyclopropyl)ethyl)-4-methylbenzenesulfonamide (4.17)**<sup>61</sup> was prepared according to Method A. The following amounts of reagents were used: piperidine **4.59** (36 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.8 mg, 5.0 μmol, 5.0 mol %), MeMgI (80. μL, 0.20 mmol, 2.0 equiv), and PhMe (0.5 mL, 0.2 M in substrate). The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a mixture of alkene diastereomers as a clear oil (22 mg, 0.59 mmol, 59% yield, 1.5:1 *E*:*Z*). The ratio of alkene isomers was determined by the integration of the resonances attributed to cyclopentyl methine in the <sup>1</sup>H NMR spectrum. The relative configuration of the major (*E*)-**4.17** was assigned based on analogy to cyclopropane **4.4**. For clarity, the <sup>1</sup>H NMR and <sup>13</sup>C NMR data of the major and minor diastereomers have been tabulated individually.

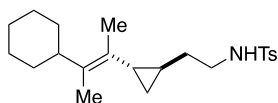
**TLC** R<sub>f</sub> = 0.5 (25% EtOAc/hexanes); **IR** (neat) 3278, 2949, 2865, 1599 cm<sup>-1</sup>; **HRMS** (TOF MS ES+) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>21</sub>H<sub>31</sub>NO<sub>2</sub>SNa, 384.1973; found, 384.1976.

**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.75 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 8.2 Hz, 2H), 4.56 (bs, 1H), 3.17–3.11 (m, 1H), 3.02 (q, *J* = 6.7 Hz, 2H), 2.42 (s, 3H), 1.65–1.47 (m, 11H), 1.43–1.25 (m, 6H), 0.69–0.63 (m, 1H), 0.61–0.56 (m, 1H), 0.38–0.33 (m, 1H); <sup>13</sup>C NMR

<sup>61</sup> Cyclopropane **4.17** was synthesized by Taylor A. Thane

(125.8 MHz, CDCl<sub>3</sub>)  $\delta$  143.5, 137.1, 132.9, 129.8 (2C), 127.3 (2C), 126.6, 43.4, 42.2, 34.5, 30.6, 30.4, 26.2 (2C), 21.64, 21.59, 15.58, 15.55, 14.2, 11.9;

**Minor Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.75 (d,  $J$  = 8.2 Hz, 2H), 7.30 (d,  $J$  = 8.2 Hz, 2H), 4.56 (bs, 1H), 3.02 (q,  $J$  = 6.7 Hz, 2H), 2.90–2.83 (m, 1H), 2.42 (s, 3H), 1.65–1.47 (m, 11H), 1.43–1.25 (m, 6H), 0.69–0.63 (m, 1H), 0.61–0.56 (m, 1H), 0.38–0.33 (m, 1H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>)  $\delta$  143.5, 137.1, 132.6, 129.8 (2C), 127.3 (2C), 126.2, 43.5, 43.1, 34.3, 30.4, 30.3, 26.1, 26.0, 22.6, 21.6, 15.7, 14.3, 13.6, 11.8.



***N*-(2-(*trans*-2-(3-cyclohexylbut-2-en-2-yl)cyclopropyl)ethyl)-4-methylbenzenesulfonamide**

**(4.18)** was prepared according to Method A. The following amounts of reagents were used: piperidine **4.60** (38 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.8 mg, 5.0  $\mu$ mol, 5.0 mol %), MeMgI (90.  $\mu$ L, 0.20 mmol, 2.3 M in Et<sub>2</sub>O, 2.0 equiv), PhMe (0.50 mL, 0.20 M in substrate). Before purification, a <sup>1</sup>H NMR yield of 52% was obtained based on comparison to PhTMS as an internal standard. The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a mixture of alkene diastereomers as a clear yellow oil (19 mg, 51  $\mu$ mol, 50% yield, 2:1 *E*:*Z*). The ratio of alkene isomers was determined by integration of the resonances in the <sup>13</sup>C NMR spectrum. The relative configuration of the major (*E*)-**4.18** was assigned based on analogy to cyclopropane **4.4**.

**TLC** *R*<sub>f</sub> = 0.2 (20% EtOAc/hexanes); **IR** (neat) 3278, 2924, 2851, 1599, 1446, 1325, 1158, 1094, 838, 660 cm<sup>-1</sup>; **HRMS** (TOF MS ES<sup>+</sup>) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>22</sub>H<sub>33</sub>NO<sub>2</sub>SNa, 398.2130; found, 398.2119.

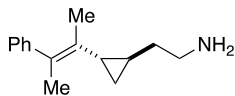
**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.75 (dd,  $J$  = 8.3, 2.8 Hz, 2H), 7.31 (d,  $J$  = 8.0 Hz, 2H), 4.46 (t,  $J$  = 6.2 Hz, 1H), 3.03 (qt,  $J$  = 8.1, 4.1 Hz, 2H), 2.71–2.63 (m, 1H), 2.43 (s,



3H), 1.77–1.71 (m, 2H), 1.68–1.64 (m, 1H), 1.61 (d,  $J = 1.7$  Hz, 1H), 1.53–1.51 (m, 3H), 1.46–1.34 (m, 3H), 1.34 (s, 3H), 1.32–1.20 (m, 5H), 1.19–1.09 (m, 1H), 0.69–0.60 (m, 1H), 0.58 (dt,  $J = 8.5, 5.1$  Hz, 1H), 0.36 (dt,  $J = 9.4, 4.9$  Hz, 1H);  $^{13}\text{C}$  NMR (125.7 MHz,  $\text{CDCl}_3$ )  $\delta$  143.4, 137.0, 135.5, 129.7 (2C), 127.1 (2C), 125.2, 43.4, 41.2, 34.4, 31.0, 30.9, 26.9, 26.8, 26.4, 21.6, 21.3, 15.6, 15.5, 14.3, 11.9.

**Minor Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75 (dd,  $J = 8.3, 2.8$  Hz, 2H), 7.31 (d,  $J = 8.0$  Hz, 2H), 4.46 (t,  $J = 6.2$  Hz, 1H), 3.03 (qt,  $J = 8.1, 4.1$  Hz, 2H), 2.71–2.63 (m, 1H), 2.43 (s, 3H), 1.77–1.71 (m, 2H), 1.68–1.64 (m, 1H), 1.53–1.51 (m, 2H), 1.61 (d,  $J = 1.7$  Hz, 3H), 1.47–1.36 (m, 2H), 1.35 (d,  $J = 1.6$  Hz, 3H), 1.32–1.20 (m, 5H), 1.19–1.09 (m, 1H), 0.69–0.60 (m, 1H), 0.58 (dt,  $J = 8.5, 5.1$  Hz, 1H), 0.36 (dt,  $J = 9.4, 4.9$  Hz, 1H);  $^{13}\text{C}$  NMR (151 MHz,  $\text{CDCl}_3$ )  $\delta$  143.4, 137.0, 135.3, 129.7 (2C), 127.2 (2C), 125.1, 43.4, 41.9, 34.2, 30.8, 30.7, 26.8, 26.8, 26.4, 22.4, 21.3, 15.7, 15.5, 14.1, 13.7.

#### 4.5.5 Synthesis and Characterization of Derivatization of Cyclopropane Products

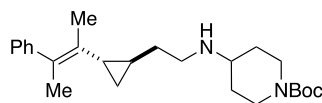


***trans*-2-(3-phenylbut-2-en-2-yl)cyclopropyl)ethan-1-amine (4.19)** was prepared according to modified procedure reported by Hilmerson.<sup>42b</sup> To a flame-dried round bottom flask equipped with a stir bar was added freshly prepared  $\text{SmI}_2$  (40. mL, 3.2 mmol, 10. equiv, 80. mM in THF).<sup>42a</sup> Then cyclopropane **4.4** (120 mg, 0.32 mmol, 1.0 equiv) was added a solution in minimal amount of THF. This was immediately followed by  $\text{H}_2\text{O}$  (0.17 mL, 9.6 mmol, 30. equiv), and pyrrolidine (0.53 mL, 6.4 mmol, 20. equiv). The solution became white upon the addition of the amine. The reaction mixture was diluted with  $\text{CH}_2\text{Cl}_2$  and treated with a solution of potassium sodium tartrate (50 mL, 10% w/w) and  $\text{K}_2\text{CO}_3$  (50 mL, 10% w/w). The aqueous layer was extracted with  $\text{CH}_2\text{Cl}_2$ . The

combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated in vacuo. The unpurified reaction mixture was carried onto the next step without further purification.

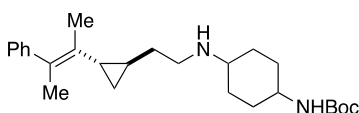
#### 4.5.5.1 Method B: Reductive Amination of Primary Amines

This method was adapted from a procedure reported by Albrecht.<sup>41</sup> To a suspension of amine **4.19** (1.0–1.3 equiv) in dichloroethane (0.10–0.20 M in amine) was added the corresponding ketone or aldehyde (1.0 equiv). Then NaBH(OAc)<sub>3</sub> (2.0–3.2 equiv) was added at rt and was allowed to stir for 30 mins. Then additional ketone or aldehyde (0.3 equiv) was added and the reaction mixture was allowed to stir for 30 mins. The mixture was quenched with saturated NaHCO<sub>3</sub> and extracted with methyl *t*-Butyl ether (x 3). The combined organic layers were dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo. The residue was purified by column chromatography.



**tert-butyl-4-trans-2-(3-phenylbut-2-en-2-yl)cyclopropyl)ethyl)amino)piperidine-1-carboxylate (4.20)** was prepared according Method B. The following amounts of reagents were used: amine **4.19** (26 mg, 0.12 mmol, 1.3 equiv), 1-boc-piperidone (19 mg, 90.  $\mu$ mol, 1.0 equiv), NaBH(OAc)<sub>3</sub> (61 mg, 0.29 mmol, 3.2 equiv), and DCE (1.2 mL, 0.10 M in amine). The residue was purified by column chromatography (0–30% EtOAc/hexanes) to afford the title compound as a yellow oil (27 mg, 70.  $\mu$ mol, 23% yield over two steps). **TLC R<sub>f</sub>** = 0.3 (25% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.41–7.33 (m, 4H, both diastereomers), 7.26 (d,  $J$  = 7.5 Hz, 4H, both diastereomers), 7.20–7.13 (m, 2H, both diastereomers), 4.10 (bs, 4H, both diastereomers), 2.96–2.77 (m, 6H, both diastereomers), 2.77–2.56 (m, 3H, one diastereomer), 2.18–2.10 (m, 3H, one diastereomer), 2.03 (s, 3H, one diastereomer), 1.99–1.79 (m, 3H, one diastereomer), 1.76–1.57 (m, 2H, one diastereomer), 1.54 (s, 3H, one diastereomer), 1.52 (s, 16H, both diastereomers),

1.46–1.26 (m, 9H, both diastereomers), 1.09–0.88 (m, 1H, one diastereomer), 0.88–0.79 (m, 1H, one diastereomer), 0.69 (dt,  $J = 9.0, 5.1$  Hz, 1H, one diastereomer), 0.60 (dt,  $J = 9.5, 5.0$  Hz, 1H, one diastereomer), 0.30 (dt,  $J = 9.4, 5.0$  Hz, 1H, one diastereomer);  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  155.0 (one diastereomer), 154.9 (one diastereomer), 145.8 (one diastereomer), 145.3 (one diastereomer), 140.1 (one diastereomer), 131.7 (one diastereomer), 130.9 (one diastereomer), 130.3 (one diastereomer), 130.2 (one diastereomer), 128.9 (2C, one diastereomer), 128.53 (one diastereomer), 128.51 (2C, one diastereomer), 128.45 (one diastereomer), 128.4 (one diastereomer), 128.11 (4C, both diastereomers), 128.09 (one diastereomer), 125.92 (one diastereomer), 125.90 (one diastereomer), 125.8 (one diastereomer), 79.6 (one diastereomer), 55.4 (one diastereomer), 55.1 (one diastereomer), 51.6 (one diastereomer), 46.7 (one diastereomer), 46.3 (one diastereomer), 34.9 (one diastereomer), 34.2 (one diastereomer), 28.6 (6C, both diastereomers), 23.1 (2C, one diastereomer), 22.4 (2C, one diastereomer), 21.7 (2C, one diastereomer), 20.9 (2C, one diastereomer), 16.5 (one diastereomer), 16.4 (one diastereomer), 16.2 (one diastereomer), 14.1 (one diastereomer), 12.0 (one diastereomer), 11.9 (one diastereomer). **IR** (neat) 2926, 2854, 1682, 1427, 1169, 906, 716  $\text{cm}^{-1}$ ; **HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{25}\text{H}_{39}\text{N}_2\text{O}_2$ , 399.3011; found, 399.3011.

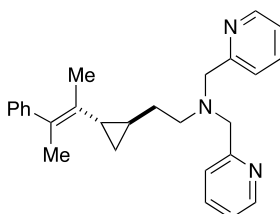


**tert-butyl-(4-trans-2-(3-phenylbut-2-en-2-yl)cyclopropyl)ethyl)amino)cyclohexyl) carbamate (4.21)**<sup>62</sup> was prepared according to Method B. The following amounts of reagents were used: amine **4.19** (50 mg, 0.23 mmol, 1.0 equiv), *N*-4-Boc-aminocyclohexanone (49 mg, 0.23 mmol, 1.0 equiv),  $\text{NaBH}(\text{OAc})_3$  (98 mg, 0.46 mmol, 2.0 equiv), and DCE (1.0 mL, 0.20 M in amine). The

<sup>62</sup> Substrate **4.21** synthesized by Nadia Hirbawi.

residue was purified by column chromatography (0–10% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) to afford the title compound as a yellow oil (64 mg, 0.16 mmol, 68% yield over two steps). The product was characterized as a 1.7:1.7:1.0:1 mixture of diastereomers. **TLC R<sub>f</sub>** = 0.6 (10% MeOH/CH<sub>2</sub>Cl<sub>2</sub>); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.34–7.24 (m, 8H, four diastereomers), 7.22–7.14 (m, 8H, four diastereomers), 7.09 (d, *J* = 7.9 Hz, 4H, four diastereomers), 4.72 (bs, 2H, two diastereomers), 4.39 (bs, 2H, two diastereomers), 3.68 (bs, 2H, two diastereomers), 3.42 (bs, 2H, two diastereomers), 2.83 (t, *J* = 7.3 Hz, 8H, four diastereomers), 2.69–2.43 (m, 8H, four diastereomers), 2.11–1.94 (m, 12H, two diastereomers), 2.07 (s, 6H, two diastereomers), 1.97 (s, 6H, two diastereomers), 1.84–1.50 (m, 16H, two diastereomers), 1.50–1.22 (m, 12H, four diastereomers), 1.48 (s, 6H, two diastereomers), 1.44 (s, 36H, four diastereomers), 1.30 (s, 6H, two diastereomers), 1.30–1.26 (m, 2H, two diastereomers), 1.21–1.03 (m, 4H, two diastereomers), 0.98–0.84 (m, 2H, two diastereomers), 0.82–0.71 (m, 2H, two diastereomers), 0.68–0.59 (m, 2H, two diastereomers), 0.59–0.50 (m, 2H, two diastereomers), 0.24 (dt, *J* = 13.2, 4.4 Hz, 2H, two diastereomers); **<sup>13</sup>C NMR** (125.8 MHz, CDCl<sub>3</sub>) δ 155.4 (2C, two diastereomers), 155.3 (2C, two diastereomers), 145.7 (2C, two diastereomers), 145.2 (2C, two diastereomers), 131.8 (2C, two diastereomers), 131.03 (one diastereomer), 130.99 (one diastereomer), 130.0 (2C, two diastereomers), 129.97 (one diastereomer), 129.95 (one diastereomer), 128.8 (4C, two diastereomers), 128.4 (4C, two diastereomers), 128.1 (2C, two diastereomers), 128.07 (2C, two diastereomers), 128.06 (2C, two diastereomers), 125.9 (2C, two diastereomers), 125.86 (2C, two diastereomers), 125.85 (2C, two diastereomers), 79.2 (4C, four diastereomers), 56.4 (2C, two diastereomers), 56.1 (2C, two diastereomers), 54.6 (2C, two diastereomers), 54.3 (2C, two diastereomers), 49.3 (4C, four diastereomers), 46.5 (2C, two diastereomers), 46.07 (one diastereomer), 46.02 (one diastereomer), 31.94 (4C, two diastereomers), 31.91 (4C, two diastereomers), 30.4 (2C, two diastereomers), 29.8

(2C, two diastereomers), 28.5 (12C, four diastereomers), 23.02 (one diastereomer), 22.99 (one diastereomer), 22.39 (one diastereomer), 22.35 (one diastereomer), 21.7 (4C, two diastereomers), 20.9 (4C, two diastereomers), 16.28 (2C, two diastereomers), 16.25 (2C, two diastereomers), 16.20 (2C, two diastereomers), 14.1 (2C, two diastereomers), 11.9 (2C, two diastereomers), 11.8 (2C, two diastereomers). **IR** (neat) 3367, 2928, 2855, 1686, 1519, 1455, 1364, 1247, 1169, 764, 702  $\text{cm}^{-1}$ ; **HRMS** (TOF MS ES+)  $m/z$ :  $[M + H]^+$  calcd for  $\text{C}_{26}\text{H}_{41}\text{N}_2\text{O}_2$ , 413.3168; found, 413.3159.



***trans*-2-(3-Phenylbut-2-en-2-yl)cyclopropyl-*N,N*-bis(pyridin-2-ylmethyl)ethan-1-amine**

**(4.22)**<sup>63</sup> was prepared according to Method B. The following amounts of reagents were used: amine **4.19** (43 mg, 0.20 mmol, 1.1 equiv), 2-pyridinecarbozaldehyde (17  $\mu\text{L}$ , 0.18 mmol, 1.0 equiv),  $\text{NaBH}(\text{OAc})_3$  (0.11 g, 0.50 mmol, 2.8 equiv), and DCE (2 mL). The residue was purified by flash column chromatography (0–10% MeOH/DCM) to afford the title compound as a clear oil (34 mg, 86  $\mu\text{mol}$ , 49% yield over two steps). For clarity, the  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR data of the major and minor diastereomers have been tabulated individually.

**TLC**  $R_f$  = 0.3 (10% MeOH/DCM); **HRMS** (TOF MS  $\text{Cl}^+$ )  $m/z$ :  $[M + H]^+$  calcd for  $\text{C}_{27}\text{H}_{32}\text{N}_3$ , 398.2596; found, 398.2598.

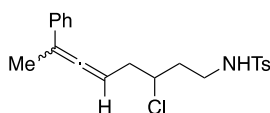
**Major Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.53–8.49 (m, 2H), 7.66–7.55 (m, 4H), 7.30–7.23 (m, 2H), 7.22–7.07 (m, 5H), 3.85 (s, 4H), 2.70 (t,  $J$  = 7.5 Hz, 2H), 1.98 (s, 3H), 1.70–1.64 (m, 1H), 1.38–1.28 (m, 2H), 1.26 (s, 3H), 0.92–0.84 (m, 1H), 0.71–0.67 (m, 1H), 0.46–0.41 (m, 1H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  160.2 (2C), 149.11 (2C), 145.9, 136.5 (2C), 130.51,

<sup>63</sup> Substrate **4.22** synthesized by Taylor A. Thane.

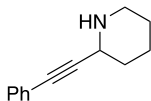
128.5 (2C), 128.1 (2C), 125.8 (2C), 122.9 (2C), 122.0 (2C), 60.7 (2C), 54.4, 32.3, 23.3, 20.8, 16.6, 16.0, 12.0.

**Minor Diastereomer:**  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.53–8.49 (m, 2H), 7.66–7.55 (m, 4H), 7.30–7.23 (m, 2H), 7.22–7.07 (m, 5H), 3.85 (s, 4H), 2.54 (t,  $J = 6.6$  Hz, 2H), 1.95 (s, 3H), 1.59–1.52 (m, 1H), 1.44 (s, 3H), 1.37–1.17 (m, 2H), 0.83–0.76 (m, 1H), 0.59–0.54 (m, 1H), 0.17–0.12 (m, 1H);  $^{13}\text{C NMR}$  (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  160.3 (2C), 149.1 (2C), 145.3, 136.4 (2C), 130.48, 128.9 (2C), 128.0 (2C), 125.8 (2C), 122.9 (2C), 121.9 (2C), 60.6 (2C), 54.3, 31.8, 22.6, 21.7, 16.59, 13.9, 11.9.

#### 4.5.6 Experimental Data for Mechanistic Experiments



***N*-(3-chloro-7-phenylocta-5,6-dien-1-yl)-4-methylbenzenesulfonamide (4.61)** was prepared according to a modified version of Method A. The following amounts of reagents were used: piperidine **4.3** (37 mg, 0.10 mmol, 1.0 equiv), MeMgI (0.10 mL, 0.30 mmol, 3.0 equiv, 3.0 M in  $\text{Et}_2\text{O}$ ), PhMe (0.5 mL, 0.2 M). The residue was purified by column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a clear and colorless oil (6.0 mg, 15  $\mu\text{mol}$ , 15% yield, 4:1 dr). **TLC**  $R_f = 0.3$  (20% EtOAc/hexanes);  $^1\text{H NMR}$  (600 MHz,  $\text{CDCl}_3$ )  $\delta$  7.82–7.74 (m, 2H), 7.48–7.43 (m, 2H), 7.42–7.33 (m, 4H), 7.28 (t,  $J = 7.3$  Hz, 1H), 5.48 (tt,  $J = 6.6, 3.1$  Hz, 1H), 4.55 (t,  $J = 6.4$  Hz, 1H), 4.08 (td,  $J = 8.5, 7.1, 4.8$  Hz, 1H), 3.30–3.13 (m, 2H), 2.56 (qd,  $J = 6.9, 6.3, 3.5$  Hz, 2H), 2.48 (s, 3H), 2.15 (dd,  $J = 5.3, 2.9$  Hz, 3H), 1.96–1.82 (m, 1H);  $^{13}\text{C NMR}$  (151 MHz,  $\text{CDCl}_3$ )  $\delta$  205.6, 143.7, 136.9, 131.8, 129.9 (2C), 128.5 (2C), 127.2 (2C), 126.9, 125.9 (2C), 101.4, 88.5, 59.6, 40.8, 38.3, 37.6, 32.1, 29.9, 21.7, 17.2, 14.3; **HRMS** (TOF MS  $\text{Cl}^+$ )  $m/z$ :  $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{21}\text{H}_{25}\text{ClNO}_2\text{S}$ , 390.1295; found, 390.1306.



**2-(phenylethynyl)piperidine (4.62)** was prepared according to a procedure reported by Seidel.<sup>64</sup>

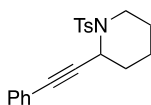
To a solution of piperidine (0.49 mL, 5.0 mmol, 1.0 equiv) in Et<sub>2</sub>O (10 mL, 0.5 M), was added *n*-BuLi (2.0 mL, 5.0 mmol, 1.0 equiv, 2.5 M in hexanes) at  $-78\text{ }^{\circ}\text{C}$ . The reaction mixture was allowed to stir for 10 mins. Then 2,2,2-trifluoroacetophenone (0.84 mL, 6.0 mmol, 1.2 equiv) was added as a solution in Et<sub>2</sub>O (6.0 mL, 1.0 M) and allowed to stir for an additional 10 mins. This was followed by the addition of freshly prepared lithium acetylide (7.5 mmol, 1.5 equiv). The reaction mixture was warmed to rt and allowed to stir for 2 h.

The lithium acetylide was prepared according the following method. In a flame dried round bottom flask equipped with a stir bar was added phenylacetylene (0.82 mL, 7.5 mmol, 1.5 equiv), PhMe (7.5 mL, 1.0 M), and THF (2.0 mL, 3.8 M). The flask was cooled to  $-78\text{ }^{\circ}\text{C}$  and *n*-BuLi (3.0 mL, 7.5 mmol, 1.5 equiv) was added dropwise via syringe. The reaction mixture was allowed to stir for 30 mins at  $-78\text{ }^{\circ}\text{C}$ .

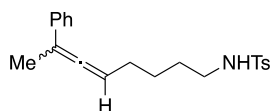
To quench, the flask was cooled to  $0\text{ }^{\circ}\text{C}$  and MeOH was added. The mixture was transferred to a separatory funnel, was diluted with Et<sub>2</sub>O and washed with 1.0 M NaOH. The aqueous layer was extracted with Et<sub>2</sub>O (x 3). The combined organic layers were washed sequentially with H<sub>2</sub>O and brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo. The residue was purified by column chromatography (0–5% MeOH/DCM) to afford the title compound as a pale yellow oil (0.31 g, 1.7 mmol, 33% yield). **TLC R<sub>f</sub>** = 0.1 (5% MeOH/DCM). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.46 – 7.39 (m, 2H), 7.34–7.23 (m, 3H), 3.88 (dd,  $J$  = 7.4, 3.6 Hz, 1H), 3.21–3.10 (m, 1H), 2.91 (bs, 1H),

<sup>64</sup> Paul, A.; Seidel, D. *J. Am. Chem. Soc.* **2019**, *141*, 8778–8782.

2.77 (ddd,  $J = 11.7, 6.8, 4.3$  Hz, 1H), 1.94 (ddt,  $J = 12.4, 6.9, 3.4$  Hz, 1H), 1.88–1.78 (m, 1H), 1.72 (dtd,  $J = 11.6, 7.6, 3.6$  Hz, 1H), 1.6–1.47 (m, 3H).



**2-(phenylethynyl)-1-tosylpiperidine (4.24)** was prepared according to a procedure reported by Smith.<sup>65</sup> To a stirring solution of piperidine **4.62** (0.31 g, 1.7 mmol, 1.0 equiv) in DCM (3.4 mL, 0.5 M) was added Et<sub>3</sub>N (0.48 mL, 3.4 mmol, 2.0 equiv) and TsCl (0.30 g, 1.9 mmol, 1.1 equiv) at rt. The reaction mixture was allowed to stir overnight. To quench, 1.0 M HCl was added slowly. The biphasic mixture was transferred to a separatory funnel and the aqueous layer was extracted with DCM (x 3). The combined organic layers were washed with NaHCO<sub>3</sub>, then brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated in vacuo. The residue was purified by column chromatography (0–25% EtOAc/hexanes) to afford the title compound as a white solid (350 mg, 1.0 mmol, 67% yield). Analytical data is consistent with literature values.<sup>66</sup> **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) 7.71 (d,  $J = 8.3$  Hz, 2H), 7.31–7.14 (m, 5H), 6.98 (dt,  $J = 7.0, 1.5$  Hz, 2H), 5.05 (s, 1H), 3.76 (dd,  $J = 12.1, 3.6$  Hz, 1H), 2.88 (td,  $J = 11.8, 2.8$  Hz, 1H), 2.27 (s, 3H), 1.97–1.82 (m, 2H), 1.77–1.61 (m, 4H); **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  143.3, 135.1, 131.5 (2C), 129.3 (2C), 128.2, 128.1 (2C), 128.0 (2C), 122.4, 86.9, 84.4, 46.9, 42.4, 31.7, 25.4, 21.4, 19.5; **HRMS** (TOF MS Cl<sup>+</sup>)  $m/z$ : [M + Na]<sup>+</sup> calcd for C<sub>20</sub>H<sub>21</sub>NO<sub>2</sub>SNa, 362.1191; found, 362.1187.

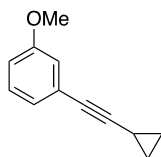


<sup>65</sup> Spoehrle, S. S. M.; West, T. H.; Taylor J. E.; Slawin, A. M.; Smith, A. D. *J. Am. Chem. Soc.* **2017**, *139*, 11895–11902.

<sup>66</sup> Daniels, D. S. B.; Jones, A. S.; Thompson, A. L.; Paton, R. S.; Anderson, E. A. *Angew. Chem. Int. Ed.* **2014**, *53*, 1915–1920.



**4-Methyl-*N*-(7-phenylocta-5,6-dien-1-yl)benzenesulfonamide (4.25)** was prepared according to Method A. The following amounts of reagents were used: piperidine **4.24** (28 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (75 mg, 0.10 mmol, 1.0 equiv), MeMgI (80. μL, 0.20 mmol, 2.0 equiv), and PhMe (0.5 mL, 0.2 M in substrate). The residue was purified by flash column chromatography (0–25% EtOAc/hexanes) to afford the title compound as a clear oil (21 mg, 76 μmol, 76% yield). **TLC** R<sub>f</sub> = 0.4 (25% hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.71 (d, *J* = 7.8 Hz, 2H), 7.37 (d, *J* = 7.8 Hz, 2H), 7.32–7.25 (m, 4H), 7.19 (t, *J* = 7.3 Hz, 1H), 5.37 (bs, 1H), 4.40 (bt, 1H), 2.91 (q, *J* = 6.5 Hz, 2H), 2.41 (s, 3H), 2.06 (s, 3H), 1.54–1.48 (m, 2H), 1.46–1.40 (m, 2H); **<sup>13</sup>C NMR** (125.8 MHz, CDCl<sub>3</sub>) δ 204.3, 143.5, 137.6, 137.1, 129.8 (2C), 128.4 (2C), 127.2 (2C), 126.6, 125.7 (2C), 100.9, 92.5, 43.2, 29.2, 28.4, 26.0, 21.6, 17.3; **IR** (neat) 3282, 3082, 3060, 3027, 2931, 2858, 1948 cm<sup>-1</sup>.

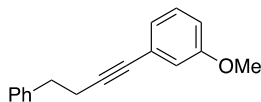


**1-(Cyclopropylethynyl)-3-methoxybenzene (4.26)**<sup>67</sup> was prepared according to Method D. The following amounts of reagents were used: cyclopropylacetylene (0.13 mL, 1.5 mmol, 1.1 equiv), 3-iodoanisole (0.17 mL, 1.4 mmol, 1.0 equiv), Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (13 mg, 0.18 mmol, 1.0 mol %), CuI (6.9 mg, 40 μmol, 2.0 mol %), and Et<sub>3</sub>N (6 mL). The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a clear oil (94 mg, 0.55 mmol, 39% yield). Analytical data are consistent with literature values.<sup>68</sup> **TLC** R<sub>f</sub> = 0.8 (10% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.16 (t, *J* = 7.9, 1H), 6.97–6.95 (m, 1H), 6.91–

<sup>67</sup> Cyclopropane **4.26** synthesized by Taylor A. Thane.

<sup>68</sup> Siebeneicher, H.; Doye, S. *Eur. J. Org. Chem.* **2002**, 7, 1213–1220.

6.90 (m, 1H), 6.82–6.79 (m, 1H), 3.77 (s, 3H), 1.47–1.40 (m, 1H), 0.88–0.82 (m, 2H), 0.81–0.72 (m, 2H).



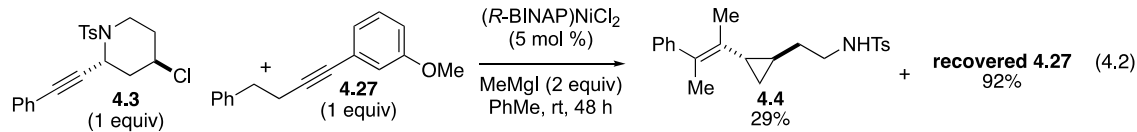
**1-methoxy-3-(4-phenylbut-1-yn-1-yl)benzene (4.27)**<sup>69</sup> was prepared according to a procedure reported by Guant.<sup>70</sup> In a glovebox, CuI (15 mg, 80.  $\mu\text{mol}$ , 4.0 mol %) was added to a flame-dried round bottom flask equipped with a stir bar. The flask was sealed with a septum, removed from the glovebox, and placed under a  $\text{N}_2$  atmosphere. Then  $\text{Pd}(\text{PPh}_3)\text{Cl}_2$  (28 mg, 40.  $\mu\text{mol}$ , 2.0 mol %) was added, followed by THF (10. mL, 0.20 M in 3-iodoanisole). The suspension was allowed to stir at room temperature for 5 min. Then, 3-iodoanisole (0.24 mL, 2.0 mmol, 1.0 equiv),  $\text{Et}_3\text{N}$  (2.4 mL, 14 mmol, 7.0 equiv), and but-3-yn-1-ylbenzene (0.31 mL, 2.2 mmol, 1.1 equiv) were added via syringe. The reaction was allowed to stir at rt and monitored by TLC until the aryl iodide was completely consumed. To quench, the reaction mixture was filtered through a pad of Celite and washed with excess  $\text{Et}_2\text{O}$ . The filtrate concentrated in vacuo and purified by flash column chromatography (0–2%  $\text{EtOAc}$ /hexanes) to afford the title compound as a colorless oil (180 mg, 0.74 mmol, 37% yield). **TLC**  $R_f$  = 0.6 (5%  $\text{EtOAc}$ /hexanes);  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.34–7.14 (m, 6H), 6.97 (d,  $J$  = 7.6 Hz, 1H), 6.90 (as, 1H), 6.83 (dd,  $J$  = 8.3, 2.4 Hz, 1H), 3.79 (s, 3H), 2.93 (t,  $J$  = 7.5 Hz, 2H), 2.69 (t,  $J$  = 7.5 Hz, 2H). Analytical data are consistent with literature values.<sup>71</sup>

<sup>69</sup> alkyne **4.27** synthesized by Nadia Hirbawi

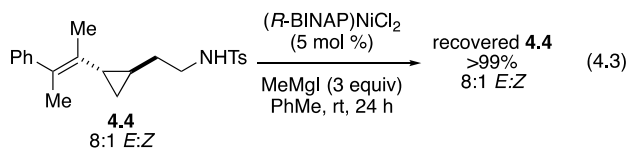
<sup>70</sup> Walkinshaw, A. J.; Xu, W.; Suero, M. G.; Guant, M. J. *J. Am. Chem. Soc.* **2013**, *135*, 12532–12535.

<sup>71</sup> Basu, B.; Das, S.; Das, P.; Mandal, B.; Banerjee, D.; Almqvist, F. *Synthesis* **2009**, *7*, 1137–1146.

### 4.5.6.1 Crossover Experiment<sup>72</sup>



In a glovebox, an oven-dried 7 mL vial equipped with a stir bar was charged with piperidine **4.3** (37 mg, 0.10 mmol, 1.0 equiv), alkyne **4.27** (24 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (3.8 mg, 5.0 μmol, 5.0 mol %), and PhMe (0.50 mL, 0.20 M in substrate). Then MeMgI (81 μL, 0.20 mmol, 2.5 M in Et<sub>2</sub>O, 2.0 equiv) was added dropwise via syringe. After 48 h, the reaction vial was removed from the glovebox, quenched with MeOH, filtered through a plug of silica gel eluting with Et<sub>2</sub>O, and concentrated in vacuo. The reaction was judged to be 35% recovered piperidine >20:1 dr, 29% yield of cyclopropane **4.4** 1.4:1 *E:Z*, 92% recovered alkyne **4.27** based on comparison to PhTMS as an internal standard by <sup>1</sup>H NMR spectroscopy. For piperidine **4.3**, the dr was determined based on the integration of the resonances attributed to the propargylic protons in the <sup>1</sup>H NMR spectrum. For cyclopropane **4.4**, the dr was determined based on the integration of the resonances attributed to H<sub>g</sub> in the <sup>1</sup>H NMR spectrum. See above (cyclopropane **4.4**) for full characterization of the product. See below (piperidine **4.3**) for full characterization of the starting material.



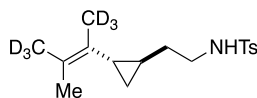
The control reaction was carried out according to a modified version of Method A. The following amounts of reagents were used: cyclopropane **4.4** (16 mg, 40. μmol, 1.0 equiv, 8:1 *E:Z*), (*R*-BINAP)NiCl<sub>2</sub> (1.5 mg, 2.0 μmol, 5 mol %), MeMgI (40. μL, 0.12 mmol, 2.8 M in Et<sub>2</sub>O, 3.0 equiv),

<sup>72</sup> Crossover experiment performed by Nadia Hirbawi.

PhMe (0.2 mL, 0.2 M in substrate). A  $^1\text{H}$  NMR yield of >99% and 8:1 *E:Z* was obtained based on comparison to PhTMS as internal standard.

#### 4.5.6.2 Preparation of $\text{CD}_3\text{MgI}$ :

Under a  $\text{N}_2$  atmosphere, a three-necked flask equipped with a stir bar, reflux condenser, and Schlenk filtration apparatus was charged with magnesium turnings (2.80 g, 115 mmol). The flask and magnesium turnings were then flame-dried under vacuum and the flask was backfilled with  $\text{N}_2$ . Anhydrous  $\text{Et}_2\text{O}$  (25 mL) and a crystal of iodine (ca. 2 mg) were added to the flask. Freshly distilled  $\text{D}_3$ -iodomethane (5.0 mL, 80 mmol) was slowly added over 30 min to maintain a gentle reflux. The mixture was stirred for 2 h at room temperature then filtered through the fritted Schlenk filter into a pear-shaped flask under a  $\text{N}_2$  atmosphere. The pear-shaped flask was capped with a septum, sealed with parafilm, and stored in the glovebox under a  $\text{N}_2$  atmosphere for up to eight weeks. The resulting methyl Grignard reagent titrated to 2.9 M as titrated by Knochel's method.<sup>56</sup>



**4-Methyl-*N*-(2-(*trans*-2-(3-methylbut-2-en-2-yl-1,1,4,4,4-*d*<sub>6</sub>)cyclopropyl)ethyl)benzene sulfonamide (4.29)**<sup>73</sup> was prepared according to Method A. The following amounts of reagents were used: piperidine **4.28** (31 mg, 0.10 mmol, 1.0 equiv), (*R*-BINAP) $\text{NiCl}_2$  (3.8 mg, 5.0  $\mu\text{mol}$ , 5.0 mol %),  $\text{MeMgI}$  (80.  $\mu\text{L}$ , 0.20 mmol, 2.0 equiv), and PhMe (0.50 mL, 0.20 M in substrate). A  $^1\text{H}$  NMR yield of 70% was obtained based on comparison to PhTMS as internal standard. The residue was purified by flash column chromatography (0–25% EtOAc/hexanes) to afford the title

<sup>73</sup> cyclopropane **4.29** synthesized by Taylor A. Thane.

compound as a clear oil (23 mg, 73  $\mu\text{mol}$ , 73% yield, 1:2.4 *E:Z*). The *E:Z* ratio was determined based on the integration of the resonances attributed to  $\text{CD}_3$  in the  $^2\text{H}$  NMR spectrum.

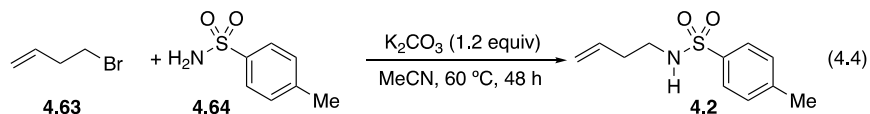
**TLC**  $R_f = 0.5$  (25% hexanes); **IR** (neat) 3279, 3065, 2996, 2923, 2859, 2236, 2189, 2058, 1598  $\text{cm}^{-1}$ ; **HRMS** (TOF MS  $\text{Cl}^+$ )  $m/z$ :  $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{17}\text{H}_{20}\text{D}_6\text{NO}_2\text{S}$ , 314.2061; found, 314.2164. >99%  $^2\text{H}$  Incorporation.

**Major Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75 (d,  $J = 8.3$  Hz, 2H), 7.30 (d,  $J = 8.3$  Hz, 2H), 4.60 (bt,  $J = 6.0$  Hz, 1H), 3.05–2.98 (m, 2H), 2.43 (s, 3H), 1.71 (s, 3H), 1.57–1.48 (m, 1H), 1.43 (m, 1H), 1.32–1.25 (m, 1H), 0.68–0.62 (m, 1H), 0.60–0.55 (m, 1H), 0.37–0.33 (m, 1H);  $^2\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  1.62 (bs, 3H), 1.29 (bs, 3H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  143.5, 137.1, 129.8 (2C), 127.3 (2C), 126.0, 125.9, 43.6, 43.5, 34.3, 29.8, 21.9, 21.6, 20.41, 15.4, 11.5;

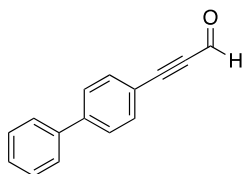
**Minor Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75 (d,  $J = 8.3$  Hz, 2H), 7.30 (d,  $J = 8.3$  Hz, 2H), 4.60 (bt,  $J = 6.0$  Hz, 1H), 3.05–2.98 (m, 2H), 2.43 (s, 3H), 1.64 (s, 3H), 1.57–1.48 (m, 1H), 1.43 (m, 1H), 1.32–1.25 (m, 1H), 0.68–0.62 (m, 1H), 0.60–0.55 (m, 1H), 0.37–0.33 (m, 1H);  $^2\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  1.70 (bs, 3H), 1.29 (bs, 3H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  143.5, 137.1, 129.8 (2C), 127.3 (2C), 126.0, 125.9, 43.6, 43.5, 34.3, 29.8, 21.9, 21.6, 20.41, 15.4, 11.5.

## 4.5.7 Proof of Stereochemical Outcome

### 4.5.7.1 Characterization Data for Stereochemical Proof

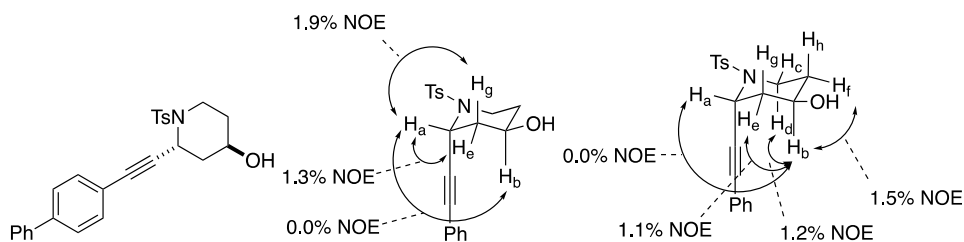


***N*-(but-3-en-1-yl)-4-methylbenzenesulfonamide (4.2)** was prepared according to a procedure reported by Jiang.<sup>74</sup> To a flame-dried flask equipped with a stir bar was added 4-bromo-1-butene **4.63** (4.1 mL, 40. mmol, 1.0 equiv), *p*-toluenesulfonamide **4.64** (6.8 g, 40. mmol, 1.0 equiv), K<sub>2</sub>CO<sub>3</sub> (6.6 g, 48 mmol, 1.2 equiv), and MeCN (160 mL). The mixture was heated to 60 °C and allowed to stir for 3 d. The reaction mixture was quenched with saturated NH<sub>4</sub>Cl (100 mL) and extracted with EtOAc (3 x 50 mL). The combined organic layers were washed with H<sub>2</sub>O (50 mL) and brine (50 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, and was concentrated in vacuo. The residue was purified by column chromatography (5–25% EtOAc/hexanes) to afford the title compound as a clear, colorless oil (5.4 g, 24 mmol, 60 %). Analytical data is consistent with literature values.<sup>74</sup> **<sup>1</sup>H NMR:** (400 MHz, CDCl<sub>3</sub>) δ 7.76 (d, *J* = 8.2, 2H), 7.30 (d, *J* = 8.1, 2H), 5.63 (ddt, *J* = 17.1, 10.4, 6.8, 1H), 5.11 (br s, 1H), 5.02–4.93 (m, 2H), 2.99 (q, *J* = 6.7, 2H), 2.41 (s, 3H), 2.20 (q, *J* = 6.9, 2 H).



**3-([1,1'-Biphenyl]-4-yl)propionaldehyde (4.30)** was prepared according to Method C. The following amounts of reagents were used: 4-ethynylbiphenyl (0.71 g, 4.0 mmol, 1.0 equiv), *n*-BuLi (1.7 mL, 4.0 mmol, 1.0 equiv, 2.4 M solution in hexanes), DMF (0.62 mL, 8.0 mmol, 2.0 equiv), THF (10. mL, 0.40 M). The residue was purified by column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a yellow solid (0.21 g, 1.0 mmol, 25% yield). **TLC R<sub>f</sub>** = 0.6 (10% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 9.45 (s, 1H), 7.70–7.58 (m, 6H), 7.47 (at, *J* = 7.1 Hz, 2H), 7.40 (tt, *J* = 7.3 Hz, 1.3 Hz, 1H).

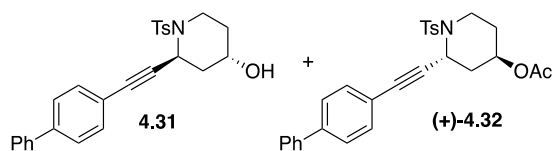
<sup>74</sup> Huang, J.; Zheng, J.; Wu, W.; Li, J.; Ma, J.; Ren, Y.; Jiang, H. *J. Org. Chem.* **2017**, *82*, 8191–8198.



***trans*-2-((1,1'-biphenyl)-4-ylethynyl)-1-tosylpiperidin-4-ol (4.31)** was prepared according to a procedure reported by Sabitha.<sup>75</sup> To a flame dried round bottom flask equipped with a stir bar was added aldehyde **4.30** (1.5 g, 5.0 mmol, 1.0 equiv), homoallylic sulfonamide **4.2** (1.3 g, 5.5 mmol, 1.1 equiv), and CH<sub>2</sub>Cl<sub>2</sub> (50. mL, 0.10 M). Then trifluoroacetic acid (3.1 mL, 40. mmol, 8.0 equiv) was added slowly via syringe. The mixture was allowed to stir for 3 h at rt and was quenched with saturated aq. NaHCO<sub>3</sub>. Then the pH was adjusted to >7 by the addition of Et<sub>3</sub>N. The solution was transferred to a separatory funnel, and the aqueous layer was extracted with CH<sub>2</sub>Cl<sub>2</sub> (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated in vacuo. The residue was then redissolved in MeOH and K<sub>2</sub>CO<sub>3</sub> (2.4 g, 18 mmol, 3.5 equiv) was added to the flask and was allowed to stir at rt for 1 h. The solvent was removed under reduced pressure, then H<sub>2</sub>O was added the residue was transferred to separatory funnel. The aqueous layer was extracted with EtOAc (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated in vacuo. The residue was purified by column chromatography (0–50% EtOAc/hexanes) to afford the title compound as an orange solid (1.1 g, 2.5 mmol, 51% yield, 10:1 dr, *trans*:*cis*). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration of the major (*trans*) diastereomer was determined by NOE analysis. **m.p.** = 68–72 °C; **TLC R<sub>f</sub>** = 0.4 (50% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.75 (d, *J* = 8.4 Hz, 2H), 7.58–7.53 (m,

<sup>75</sup> (a) Sabitha, G.; Reddy, N. M.; Prasad, M. N.; Yadav, J. S. *Helv. Chim. Acta* **2009**, *92*, 967–976. (b) see ref 9b.

2H), 7.48–7.42 (m, 4H), 7.40–7.34 (m, 1H), 7.23 (d,  $J = 8.0$  Hz, 2H), 7.05 (d,  $J = 8.0$  Hz, 2H), 5.17 (s, 1H), 4.14–3.99 (m, 1H), 3.87 (dt,  $J = 12.0, 2.1$  Hz, 1H), 2.99 (td,  $J = 12.5, 2.7$  Hz, 1H), 2.30 (s, 3H), 2.22 (ddt,  $J = 12.2, 4.6, 2.4$  Hz, 1H), 2.09–2.02 (m, 1H), 1.84 (td,  $J = 11.9, 4.9$  Hz, 1H), 1.66 (qd,  $J = 12.0, 4.8$  Hz, 1H), 1.55 (bs, 1H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  143.6, 141.4, 140.3, 135.1, 132.0 (2C), 129.6 (2C), 129.1 (2C), 128.2 (2C), 127.9, 127.1 (2C), 126.9 (2C), 120.9, 87.1, 84.5, 65.7, 46.8, 41.2, 40.2, 34.7, 21.6; IR (neat) 3536, 2941, 2219, 1515, 1161, 986, 766  $\text{cm}^{-1}$ ; HRMS (TOF MS ES+)  $m/z$ :  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{26}\text{H}_{25}\text{NO}_3\text{SNa}$ , 454.1453; found, 454.1445.



**(2*S*,4*S*)-2-([1,1'-biphenyl]-4-ylethynyl)-1-tosylpiperidin-4-ol (4.31)** and **(2*R*,4*R*)-2-([1,1'-biphenyl]-4-ylethynyl)-1-tosylpiperidin-4-yl acetate ((+)-4.32)** were prepared according to a lipase resolution procedure reported by Yadav.<sup>76</sup> To a round bottom flask equipped with a stir bar was added piperidine **4.31** (0.91 g, 2.1 equiv, 1.0 equiv), lipase from *Candida artica* (0.82 g, 20% w/w), vinyl acetate (6.3 mL, 68 mmol, 16 equiv), and  $\text{CH}_2\text{Cl}_2$  (42 mL, 0.050 M in piperidine). The reaction was allowed to stir open to air for 120 h. The reaction mixture was filtered through a pad of Celite and the solvent was removed under reduced pressure. The residue was purified by column chromatography (0–50% EtOAc/hexanes) to separate the alcohol and the acetate products.

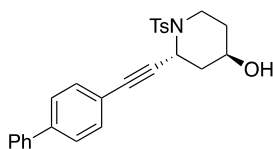
**Analytical Data for 4.31 (Alcohol):** The title compound was isolated as an orange solid (0.62 g, 1.4 mmol, 67% recovered). TLC  $R_f = 0.4$  (50% EtOAc/hexanes);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75 (d,  $J = 8.2$  Hz, 2H), 7.59–7.54 (m, 2H), 7.50–7.42 (m, 4H), 7.41–7.34 (m, 1H), 7.24 (d,  $J = 8.0$  Hz, 2H), 7.05 (d,  $J = 8.2$  Hz, 2H), 5.18 (s, 1H), 4.09 (ddt,  $J = 15.8, 11.1, 5.9$  Hz, 1H), 3.88 (dt,

<sup>76</sup> Yadav, J. S.; Reddy, B. V. S.; Padmavani, B.; Venugopal, C.; Rao, A. B. *Tetrahedron Lett.* **2007**, *48*, 4631–4633.



$J = 13.2, 2.8$  Hz, 1H), 3.01 (td,  $J = 12.5, 2.7$  Hz, 1H), 2.31 (s, 3H), 2.23 (ddt,  $J = 12.3, 4.6, 2.4$  Hz, 1H), 2.11–2.02 (m, 1H), 1.85 (td,  $J = 11.9, 4.9$  Hz, 1H), 1.67 (qd,  $J = 12.5, 4.8$  Hz, 1H), 1.55 (bs, 1H).

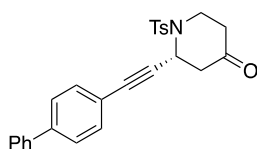
**Analytical Data for (+)-4.32 (Acetate):** The title compound was isolated as an off white solid (230 mg, 0.49 mmol, 23% yield, >20:1 dr, >99% ee) **m.p.** = 147–149 °C; **TLC**  $R_f = 0.6$  (50% EtOAc/hexanes);  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.76 (d,  $J = 8.4$  Hz, 2H), 7.59–7.53 (m, 2H), 7.50–7.42 (m, 4H), 7.41–7.35 (m, 1H), 7.24 (d,  $J = 8.1$  Hz, 2H), 7.07 (d,  $J = 8.3$  Hz, 2H), 5.21 (s, 1H), 5.13 (tt,  $J = 11.5, 4.5$  Hz, 1H), 3.89 (dt,  $J = 12.2, 2.1$  Hz, 1H), 3.06 (td,  $J = 12.5, 2.7$  Hz, 1H), 2.31 (s, 3H), 2.28–2.21 (m, 1H), 2.19–2.11 (m, 1H), 2.06 (s, 3H), 1.98 (td,  $J = 12.0, 4.9$  Hz, 1H), 1.73 (qd,  $J = 12.3, 4.9$  Hz, 1H);  **$^{13}\text{C NMR}$**  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  170.4, 143.7, 141.4, 140.3, 135.0, 132.1 (2C), 129.6 (2C), 129.0 (2C), 128.2 (2C), 127.9, 127.1 (2C), 126.8 (2C), 120.8, 87.5, 83.8, 68.1, 46.7, 41.0, 36.8, 31.1, 21.6, 21.3; **IR** (neat) 2927, 2867, 2224, 1730, 1595, 1349, 1159, 1031, 767, 588  $\text{cm}^{-1}$ ; **HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{28}\text{H}_{27}\text{NO}_4\text{SNa}$ , 496.1559; found, 496.1564;  $[\alpha]_D^{23} + 10.52$  ( $c$  8.6 mg/1.5 mL  $\text{CHCl}_3$ ); **SFC Analysis** (Chiralcel OJ-H, 5% IPA, 2.0 mL/min, 254 nm) indicated >99% ee  $t_R$  (only diastereomer, major enantiomer) = 12.8 minutes,  $t_R$  (only diastereomer, minor enantiomer) = 15.7 minutes.



**(2R,4R)-2-([1,1'-biphenyl]-4-ylethynyl)-1-tosylpiperidin-4-ol ((+)-4.31)** was prepared according to a procedure reported by Sabitha.<sup>77</sup> To a round bottom flask equipped with a stir bar was added acetate (+)-4.32 (0.34 g, 0.71 mmol, 1.0 equiv) and dissolved in excess MeOH and a minimal amount of THF. Then  $\text{K}_2\text{CO}_3$  (0.34 g, 2.5 mmol, 3.5 equiv) was added and the mixture

<sup>77</sup> Sabitha, G.; Reddy, N. M.; Prasad, M. N.; Yadav, J. S. *Helv. Chim. Acta* **2009**, *92*, 967–976.

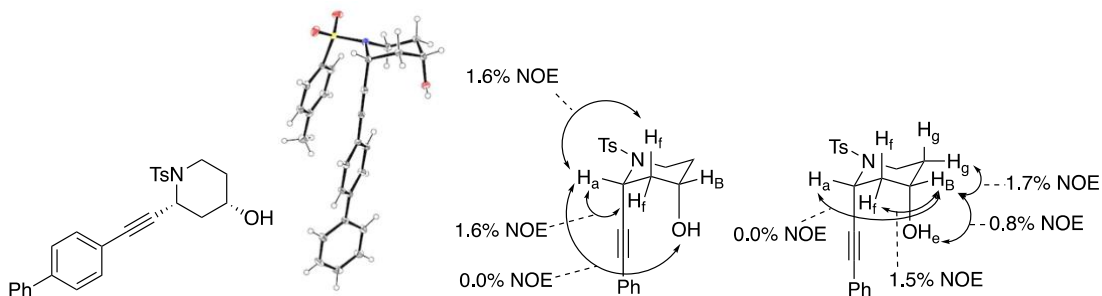
was allowed to stir at rt for 3 h. Then solvent was removed under reduced pressure, then H<sub>2</sub>O was added the residue was transferred to separatory funnel. The aqueous layer was extracted with EtOAc (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated in vacuo. The residue was purified by column chromatography (0–50% EtOAc/hexanes) to afford the title compound as a white solid (0.25 g, 0.58 mmol, 82% yield, >20:1 dr, 95% ee). Refer to **4.31** above for analytical data.  $[\alpha]^{23}_{\text{D}} + 52.19$  (*c* 10 mg/mL CHCl<sub>3</sub>); **SFC Analysis** (Chiralcel AD-H, 15% IPA, 2.0 mL/min, 254 nm) indicated 95% ee *t<sub>R</sub>* (only diastereomer, minor enantiomer) = 17.1 minutes, *t<sub>R</sub>* (only diastereomer, major enantiomer) = 19.3 minutes.



**(R)-2-([1,1'-biphenyl]-4-ylethynyl)-1-tosylpiperidin-4-one (4.65)** was prepared according to a modified procedure reported by Huang.<sup>78</sup> To a stirring solution of piperidine (+)-**4.31** (240 mg, 0.55 mmol, 1.0 equiv) in CH<sub>2</sub>Cl<sub>2</sub> (2.8 mL, 0.20 M), was added DMP (0.36 g, 0.84 mmol, 1.5 equiv). The mixture was allowed to stir at rt for 2 h. To quench, saturated NaHCO<sub>3</sub> was added and the reaction was extracted with CH<sub>2</sub>Cl<sub>2</sub> (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated in vacuo. The residue was purified by column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a white solid (220 mg, 0.55 mmol, 98% yield). **m.p.** = 163–166 °C; **TLC R<sub>f</sub>** = 0.6 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.78 (d, *J* = 8.3 Hz, 2H), 7.57–7.50 (m, 2H), 7.44 (dtd, *J* = 8.1, 3.8, 1.6 Hz, 4H), 7.39–7.33 (m, 1H), 7.29 (d, *J* = 7.9 Hz, 2H), 7.03 (d, *J* = 8.5 Hz, 2H), 5.43 (dt, *J* = 6.3, 2.1 Hz, 1H), 4.12 (ddt, *J* = 11.6, 7.5, 1.9 Hz, 1H), 3.31 (td, *J* = 12.2, 3.6 Hz, 1H), 2.98 (dd, *J* = 14.4, 6.3

<sup>78</sup> Huang, R.; Chen, X.; Mou, C.; Luo, G.; Li, Y.; Li, X.; Xue, W.; Jin, Z.; Chi, Y. R. *Org. Lett.* **2019**, *21*, 4340–4344.

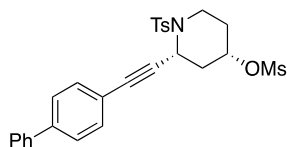
Hz, 1H), 2.73 (ddd,  $J = 15.1, 12.2, 7.5$  Hz, 1H), 2.62 (dt,  $J = 14.4, 2.1$  Hz, 1H), 2.47 (dt,  $J = 15.2, 1.6$  Hz, 1H), 2.34 (s, 3H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  204.0, 144.2, 141.8, 140.2, 134.6, 132.2 (2C), 129.8 (2C), 129.0 (2C), 128.2 (2C), 128.0, 127.1 (2C), 126.9 (2C), 120.3, 88.7, 82.9, 48.0, 47.5, 41.7, 40.7, 21.6; IR (neat) 2873, 2224, 1722, 1598, 1486, 1092, 765  $\text{cm}^{-1}$ ; HRMS (TOF MS ES+)  $m/z$ :  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{26}\text{H}_{23}\text{NO}_3\text{SNa}$ , 452.1296; found, 452.1300.



**(2R,4S)-2-((1,1'-biphenyl)-4-ylethynyl)-1-tosylpiperidin-4-ol ((+)-4.33)** was prepared according to a procedure reported by Andersen.<sup>79</sup> To a solution of ketone **4.65** (240 mg, 0.55 mmol, 1.0 equiv) in THF (2.8 mL, 0.20 M) at 0 °C, was added L-selectride (0.61 mL, 0.61 mmol, 1.1 equiv, 1.0 M in THF) slowly via syringe. The mixture was allowed to stir at 0 °C for 30 mins and then was allowed to warm to rt and stir for an additional 2 h. To quench, 1 M HCl was added and extracted with EtOAc (x 3). The combined organic layer was washed with  $\text{NaHCO}_3$ , dried over  $\text{Na}_2\text{SO}_4$ , filtered, and concentrated in vacuo. The residue was purified by column chromatography (0–50% EtOAc/hexanes) to afford the title compound as a white solid (160 mg, 0.34 mmol, 65% yield, >20:1 dr, 87% ee). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the  $^1\text{H}$  NMR spectrum. The relative configuration was determined by NOE analysis. Absolute configuration was determined by X-ray crystallography (CCDC 2104034). **m.p.** = 145–147 °C; **TLC**  $R_f$  = 0.4 (50% EtOAc/hexanes);  $^1\text{H}$  NMR (500 MHz,

<sup>79</sup> Andersen, C.; Ferey, V.; Daumas, M.; Bernardelli, P.; Guérinot, A.; Cossy, J. *Org. Lett.* **2019**, *21*, 2285–2289.

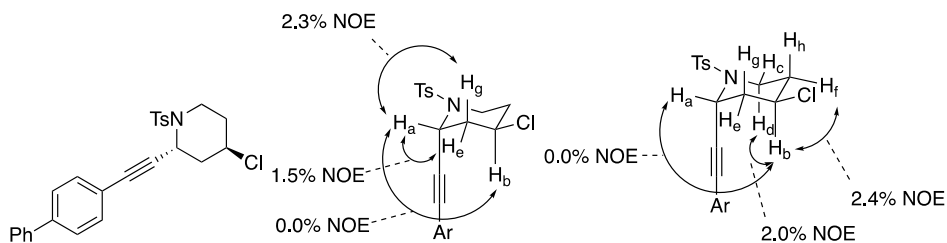
CDCl<sub>3</sub>) δ 7.76 (d, *J* = 8.4 Hz, 2H), 7.57–7.53 (m, 2H), 7.45 (t, *J* = 7.9 Hz, 4H), 7.40–7.34 (m, 1H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.05 (d, *J* = 8.1 Hz, 2H), 5.08 (t, *J* = 3.4 Hz, 1H), 4.16 (as, 1H), 3.75 (dt, *J* = 12.0, 3.0 Hz, 1H), 3.38–3.21 (m, 1H), 2.65 (d, *J* = 7.4 Hz, 1H), 2.27 (s, 3H), 2.21–2.17 (m, 2H), 1.98–1.91 (m, 2H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 143.6, 141.6, 140.2, 135.1, 132.0 (2C), 129.6 (2C), 129.1 (2C), 128.2 (2C), 128.0, 127.1 (2C), 126.9 (2C), 120.4, 87.5, 86.1, 64.4, 43.4, 37.6, 37.1, 32.7, 21.6; **IR** (neat) 3503, 2921, 2870, 2224, 1651, 1594, 1487, 1181, 901, 757 cm<sup>-1</sup>; **HRMS** (TOF MS ES+) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>25</sub>H<sub>25</sub>NO<sub>3</sub>SNa, 454.1453; found, 454.1445; [α]<sup>23</sup><sub>D</sub> + 27.58 (*c* 9.3 mg/1.5 mL CHCl<sub>3</sub>); **SFC Analysis** (Chiralcel AD-H, 15% IPA, 2.0 mL/min, 254 nm) indicated 87% ee *t<sub>R</sub>* (only diastereomer, minor enantiomer) = 15.2 minutes, *t<sub>R</sub>* (only diastereomer, major enantiomer) = 18.2 minutes.



**(2R,4S)-2-([1,1'-biphenyl]-4-ylethynyl)-1-tosylpiperidin-4-yl methanesulfonate (4.66)** was prepared according to a procedure reported by Sanford.<sup>80</sup> To a flame dried round bottom flask equipped with a stir bar was added alcohol (+)-**4.33** (150 mg, 0.34 mmol, 1.0 equiv) and CH<sub>2</sub>Cl<sub>2</sub> (1.7 mL, 0.20 M). Then Et<sub>3</sub>N (70. μL, 0.51 mmol, 1.5 equiv), DMAP (4.1 mg, 3.4 μmol, 0.10 equiv), and MsCl (40. μL, 0.51 mmol, 1.5 equiv) were added in that order. The reaction mixture was allowed to stir at rt for 48 h. Once complete by TLC, sat. NaHCO<sub>3</sub> was added and the mixture was extracted with CH<sub>2</sub>Cl<sub>2</sub> (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated in vacuo. The residue was carried onto the next step without further purification. **TLC** *R<sub>f</sub>* = 0.5 (50% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.76 (d,

<sup>80</sup> Sanford, A. B.; Thane, T. A.; McGinnis, T. M.; Chen, P.-P.; Hong, X.; Jarvo, E. R. *J. Am. Chem. Soc.* **2020**, *142*, 5017–5023.

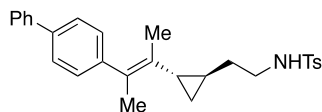
$J = 8.3$  Hz, 2H), 7.59–7.54 (m, 2H), 7.49–7.43 (m, 4H), 7.41–7.35 (m, 1H), 7.22 (d,  $J = 8.1$  Hz, 2H), 7.11 (d,  $J = 8.4$  Hz, 2H), 5.18–5.12 (m, 2H), 3.81 (d,  $J = 12.6$  Hz, 1H), 3.34 (td,  $J = 12.5, 2.7$  Hz, 1H), 2.89 (s, 3H), 2.44 (dd,  $J = 14.8, 2.6$  Hz, 1H), 2.29–2.26 (m, 4H), 2.26–2.17 (m, 1H), 2.16–2.04 (m, 1H).



**(2*R*,4*R*)-2-([1,1'-biphenyl]-4-ylethynyl)-4-chloro-1-tosylpiperidine ((+)-4.34)** was prepared according to a procedure reported by Cahiez.<sup>81</sup> In a glovebox, *n*-Bu<sub>4</sub>NCl (100 mg, 0.44 mmol, 2.0 equiv) was added to a flame dried round bottom flask. The flask was sealed with a septum and removed from the glovebox. In a separate flask, a solution mesylate **4.66** (110 mg, 0.22 mmol, 1.0 equiv) in THF (1.1 mL, 0.10 M) was prepared. The solution of mesylate was then transferred to the reaction flask containing *n*-Bu<sub>4</sub>NCl via syringe. The mixture was heated to reflux and allowed to stir for 48 h. The reaction mixture was then cooled to rt and quenched with H<sub>2</sub>O. The mixture was transferred to a separatory funnel and extracted with EtOAc (x 2), then hexanes (x 1). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated in vacuo. The residue was purified by column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a white solid (51 mg, 0.11 mmol, 52% yield, >20:1 dr trans:cis, 91% ee). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration of the major diastereomer was determined by NOE analysis. Absolute configuration was determined by analogy to piperidine (+)-**4.33**. **m.p.** =

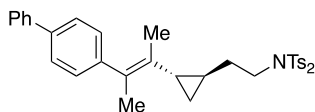
<sup>81</sup> (a) Cahiez, G.; Lefèvre, N.; Poizat, M.; Moyeux, A. *Synthesis*, **2013**, 45, 213–216. (b) see ref 9b.

111–114 °C; **TLC**  $R_f$  = 0.4 (15% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.73 (d,  $J$  = 8.3 Hz, 2H), 7.57–7.53 (m, 2H), 7.48–7.42 (m, 4H), 7.37 (tt,  $J$  = 7.3 Hz, 1.3 Hz, 1H), 7.23 (d,  $J$  = 7.9 Hz, 2H), 7.05 (d,  $J$  = 8.4 Hz, 2H), 5.17–5.13 (m, 1H), 4.22 (att,  $J$  = 11.9 Hz, 4.3 Hz, 1H), 3.86 (adquin,  $J$  = 12.2 Hz, 2.3 Hz, 1H), 2.99 (atd,  $J$  = 12.4 Hz, 2.5 Hz, 1H), 2.40 (adquin,  $J$  = 12.8 Hz, 2.0 Hz, 1H), 2.31–2.13 (m, 5H), 1.98 (aqd,  $J$  = 12.6 Hz, 4.9 Hz, 1H); **<sup>13</sup>C NMR** (125.8 MHz, CDCl<sub>3</sub>) δ 143.8, 141.6, 140.2, 134.9, 132.0 (2C), 129.6 (2C), 129.1 (2C), 128.2 (2C), 128.0, 127.1 (2C), 126.9 (2C), 120.6, 87.7, 83.5, 53.0, 47.5, 42.1, 41.6, 36.0, 21.6; **IR** (neat) 2063, 3032, 2921, 2864, 2223, 1596, 1344, 1161, 766, 725 cm<sup>-1</sup>; **HRMS** (TOF MS ES<sup>+</sup>)  $m/z$ : [M + Na]<sup>+</sup> calcd for C<sub>26</sub>H<sub>24</sub>ClNO<sub>2</sub>SNa, 472.1114; found, 472.1122; **[α]<sup>D</sup><sub>23</sub>** + 7.17 (*c* 6.9 mg/1.5 mL CHCl<sub>3</sub>); **SFC Analysis** (Chiralcel AD-H, 15% IPA, 2.0 mL/min, 254 nm) indicated 91% ee  $t_R$  (only diastereomer, major enantiomer) = 13.3 minutes,  $t_R$  (only diastereomer, minor enantiomer) = 20.6 minutes.



***N*-(2-((1*R*,2*S*)-2-((*E*)-3-([1,1'-biphenyl]-4-yl)but-2-en-2-yl)cyclopropyl)ethyl)-4-methylbenzenesulfonamide ((+)-4.5)** was prepared according to Method A. The following amounts of reagents were used: piperidine (+)-**4.34** (70. mg, 0.16 mmol, 1.0 equiv), (*R*-BINAP)NiCl<sub>2</sub> (6.0 mg, 8.0 μmol, 5.0 mol %), MeMgI (0.12 mL, 0.32 mmol, 2.8 M in Et<sub>2</sub>O, 2.0 equiv), and PhMe (0.8 mL). The residue was purified by column chromatography (0–10% EtOAc/hexanes) and the diastereomers were separated to afford the major (*E*)-diastereomer as a pale yellow solid (22 mg, 50. μmol, 31% yield, >20:1 *E*:*Z*, 95% ee). Refer to cyclopropane **4.5** above for analytical data. **[α]<sup>D</sup><sub>23</sub>** + 9.34 (*c* 22 mg/1.5 mL CHCl<sub>3</sub>); **SFC Analysis** (Chiralcel OD-H,

20% IPA, 2.0 mL/min, 250 nm) indicated 95% ee  $t_R$  (only diastereomer, minor enantiomer) = 27.9 minutes,  $t_R$  (only diastereomer, major enantiomer) = 29.7 minutes.



***N*-(2-((1*R*,2*S*)-2-((*E*)-3-([1,1'-biphenyl]-4-yl)but-2-en-2-yl)cyclopropyl)ethyl)-4-methyl-*N*-tosylbenzenesulfonamide ((+)-4.36)** was prepared according to a literature procedure.<sup>9c</sup> In a glovebox, NaH (1.4 mg, 60.  $\mu$ mol, 2.0 equiv) was added to a flame dried round bottom flask equipped with a stir bar. The flask was sealed with a septum, brought out of the glovebox, and placed under N<sub>2</sub> atmosphere. Then DMF (1.0 mL) was added followed by a solution of cyclopropane (+)-4.5 (14 mg, 30.  $\mu$ mol, 1.0 equiv) in DMF (0.5 mL). The solution was allowed to stir at rt for 1 h and then TsCl (11 mg, 60.  $\mu$ mol, 2.0 equiv) was added as a solution in DMF (0.5 mL). The resulting solution was allowed to stir at rt for 24 h. The reaction mixture was quenched with saturated NH<sub>4</sub>Cl and the aqueous layer was extracted with EtOAc ( $\times$  3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated in vacuo. The residue was purified by column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a white solid (11 mg, 18  $\mu$ mol, 63% yield, >20:1 *E*:*Z*, 93% ee). The *E*:*Z* ratio was determined based on the integration of the resonances attributed to protons on the cyclopropane in the <sup>1</sup>H NMR spectrum. The relative configuration was determined by NOE analysis. Absolute configuration was determined by comparing the experimental CD spectrum with the calculated CD spectrum (see below). **TLC**  $R_f$  = 0.6 (30% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.92 (d,  $J$  = 7.8 Hz, 4H), 7.63–7.58 (m, 2H), 7.54 (d,  $J$  = 8.1 Hz, 2H), 7.43 (t,  $J$  = 7.7 Hz, 2H), 7.38–7.30 (m, 5H), 7.17 (d,  $J$  = 8.0 Hz, 2H), 3.78 (t,  $J$  = 8.1 Hz, 2H), 2.46 (s, 6H), 2.10 (d,  $J$  = 1.0 Hz, 3H), 1.84 (dq,  $J$  = 14.9, 7.5 Hz, 1H), 1.67 (dq,  $J$  = 14.7, 7.7 Hz, 1H), 1.33 (d,  $J$  = 1.7 Hz, 3H), 0.92–0.79 (m, 2H),

0.76 (dt,  $J = 8.2, 4.8$  Hz, 1H), 0.54 (dt,  $J = 9.4, 4.8$  Hz, 1H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  145.0, 144.7, 141.2, 138.8, 137.3, 130.3, 129.8 (4C), 129.0 (2C), 128.9 (2C), 128.4 (4C), 127.20, 127.18, 127.1 (2C), 126.8 (2C), 49.1, 35.0, 29.9, 22.4, 21.8 (2C), 20.9, 16.3, 15.7, 11.9, 1.2; IR 2969, 2926, 1597, 1376, 1162, 950, 767  $\text{cm}^{-1}$ ; HRMS (TOF MS ES+)  $m/z$ :  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{35}\text{H}_{37}\text{NO}_4\text{S}_2\text{Na}$ , 622.2062; found, 622.2072;  $[\alpha]^{23}_{\text{D}} + 66$  ( $c$  5.7 mg/1.5 mL  $\text{CHCl}_3$ ); SFC Analysis (Chiralcel OD-H, 15% IPA, 2.0 mL/min, 250 nm) indicated 93% ee  $t_{\text{R}}$  (only diastereomer, minor enantiomer) = 27.7 minutes,  $t_{\text{R}}$  (only diastereomer, major enantiomer) = 29.5 minutes.

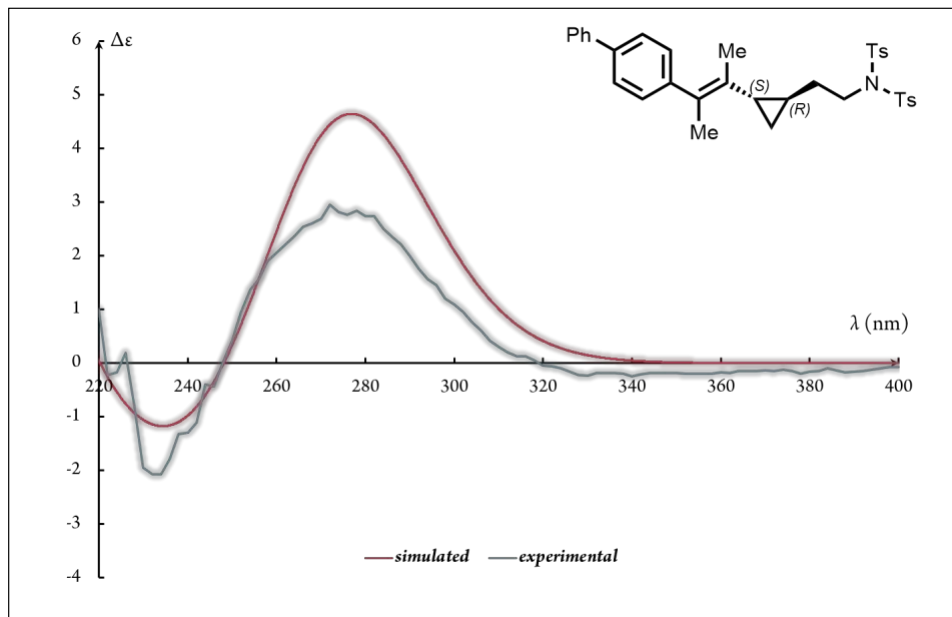
#### 4.5.7.2 Experimental (blue) and Simulated (red) CD Spectra.

Experimental data collected was collected in  $\text{CH}_2\text{Cl}_2$  (concentration =  $7.48 \times 10^{-4}$  M). Calculated conformational averaged ECD spectrum and experimental ECD spectrum of (+)-**4.36** at TD- $\omega$ B97XD/def2-TZVPP-SMD( $\text{CH}_2\text{Cl}_2$ )/B3LYPD3(BJ)/def2-SV(P)-SMD( $\text{CH}_2\text{Cl}_2$ ). Conformers population were obtained with electronic energies (EE) at  $\omega$ B97XD/def2-TZVPP-SMD( $\text{CH}_2\text{Cl}_2$ )/B3LYP-D3(BJ)/def2-SV(P)-SMD( $\text{CH}_2\text{Cl}_2$ ). Number of states solved during TD-DFT calculation were set to 50. Full width at half maximum (FWHM) was set to 0.5 eV with Gaussian broadening function.<sup>1,82</sup>

**Figure 4.3:** Experiment and Computation Circular Dichroism Spectra to Confirm Absolute Configurations

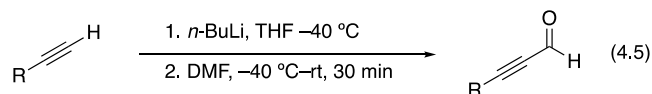
<sup>82</sup> Bannwarth, C.; Grimme, S. *Comput. Theor. Chem.* **2014**, 1040–1041, 45–53.





#### 4.5.8 General Procedures for the Synthesis of Starting Materials

##### 4.5.8.1 Method C: Synthesis of Propargyl Aldehydes with *n*-BuLi and DMF

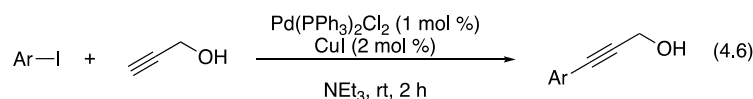


This method was adapted from a procedure reported by Larsen.<sup>83</sup> To a flame-dried round-bottom flask equipped with a stir bar was added alkyne (1 equiv). The flask was evacuated, backfilled with N<sub>2</sub>, and capped with a septum. THF (0.4 M in alkyne) was added and the solution was cooled to -40 °C. Then *n*-BuLi (1 equiv) was added dropwise, followed by DMF (2 equiv). The mixture was allowed to warm to room temperature and stir for an additional 30 mins. The solution was poured into a vigorously stirred biphasic solution of 10% aqueous KH<sub>2</sub>PO<sub>4</sub> and Et<sub>2</sub>O cooled over ice. The layers were separated, and the organic layer was washed with H<sub>2</sub>O (x 2). The combined aqueous layers were extracted with Et<sub>2</sub>O. Then the combined organic layers were dried over Na<sub>2</sub>SO<sub>4</sub>, concentrated in vacuo, and purified by column chromatography.

<sup>83</sup> Journet, M.; Cai, D.; DiMichele, L. M.; Larsen, R. D. *Tetrahedron Lett.* **1998**, 39, 6427–6428.

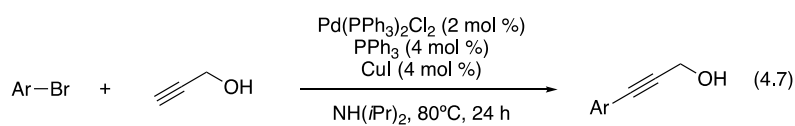
## 4.5.8.2 Method D: Sonogashira Cross-Coupling Reaction of Aryl Halides and Propargyl Alcohol

### 4.5.8.2.1 Sonogashira Cross-Coupling Reactions with Aryl Iodides



This method was adapted from a procedure reported by Tambar.<sup>84</sup> To a flame-dried round bottom flask equipped with a stir bar was added Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (1 mol %) and CuI (2 mol %). Et<sub>3</sub>N (0.01 M in CuI) was added to the flask, and the suspension was allowed to stir for five minutes. A solution of aryl iodide (1.0 equiv) and propargyl alcohol (1.1 equiv) in Et<sub>3</sub>N (1.0 M in aryl iodide) was prepared, and the solution was added dropwise to the reaction mixture. The reaction was allowed to stir at rt until the aryl iodide was consumed. The reaction mixture was filtered through a pad of Celite and washed with excess EtOAc. The combined solution was concentrated in vacuo and purified by column chromatography.

### 4.5.8.2.1 Sonogashira Cross-Coupling Reactions with Aryl Bromides



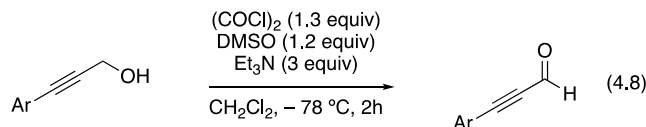
The method was adapted from a procedure reported by Nagumo.<sup>85</sup> In a glovebox, CuI (4.0 mol %) was added to a flame-dried round bottom flask. The flask was seal with a septum, removed from the glovebox and placed under a N<sub>2</sub> atmosphere. Then Pd(PPh<sub>3</sub>)Cl<sub>2</sub> (2.0 mol %) and Ph<sub>3</sub>P (4.0 mol %) was added followed by freshly distilled diisopropylamine (0.5 M in aryl bromide). The suspension was allowed to stir at room temperature for 5 min. In a separate flask, a solution of aryl bromide (1.0 equiv) and propargyl alcohol (1.3 equiv) in diisopropylamine (1.0 M in aryl bromide)

<sup>84</sup> Xu, B.; Gartman, J. A.; Tambar, U. K. *Tetrahedron* **2017**, *73*, 4150–4159.

<sup>85</sup> Sakata, Y.; Yasui, E.; Mizukami, M.; Nagumo, S. *Tetrahedron Lett.* **2019**, *60*, 755–759.

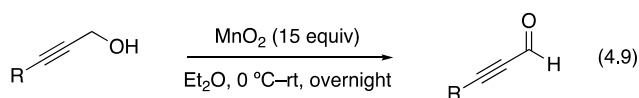
was prepared and added dropwise to the reaction flask. The flask was then fitted with a reflux condenser, heated to reflux, and was allowed to stir for 24 h. The reaction mixture was cooled to rt and filtered through a pad of Celite and washed with EtOAc (x 3). Then the solution was concentrated in vacuo and purified by column chromatography.

#### 4.5.8.3 Method E: Swern Oxidation of Propargyl Alcohols



This method was adapted from a procedure reported by Kobayashi.<sup>86</sup> A flame-dried round bottom flask equipped with stir bar was charged with oxalyl chloride (1.3 equiv) and CH<sub>2</sub>Cl<sub>2</sub> (0.4 M). The flask was cooled to -78 °C and DMSO (1.2 equiv) was added dropwise under N<sub>2</sub> with a vent. This was allowed to stir at -78 °C for 15 min. Then propargyl alcohol (1.0 equiv) was added as a solution in CH<sub>2</sub>Cl<sub>2</sub> (0.2 M). The reaction mixture was allowed to stir at -78 °C for 2 h. Then, triethylamine (3.0 equiv) was added and the reaction was allowed to warm to rt. The reaction mixture was quenched with saturated NH<sub>4</sub>Cl, extracted with CH<sub>2</sub>Cl<sub>2</sub> (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, concentrated in vacuo, and purified by column chromatography.

#### 4.5.8.4 Method F: MnO<sub>2</sub> Oxidation of Propargyl Alcohols



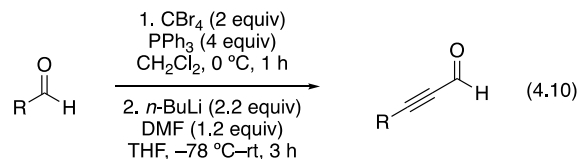
This method was adapted from a procedure reported by Fürstner.<sup>87</sup> Propargyl alcohol (1.0 equiv) was added to a vigorously stirring suspension of MnO<sub>2</sub> (10. equiv) in Et<sub>2</sub>O at 0 °C. This suspension

<sup>86</sup> Shinohara, R.; Morita, M.; Ogawa, N.; Kobayashi, Y. *Org. Lett.* **2019**, *21*, 3247–3251.

<sup>87</sup> Persich, P.; Llaveria, J.; Lhermet, R.; de Haro, T.; Stade, R.; Kondoh, A.; Fürstner, A. *Chem. Eur. J.* **2013**, *19*, 13047–13058.

was warmed to rt and allowed to stir overnight. Additional MnO<sub>2</sub> (5.0 equiv) was added and was allowed to stir for an additional 4 h. The reaction mixture was filtered through a pad of Celite and the filtrate was concentrated in vacuo. For lower boiling point substrates, a stream of air could be used to evaporate the Et<sub>2</sub>O.

#### 4.5.8.5 Method G: Corey-Fuchs Reaction

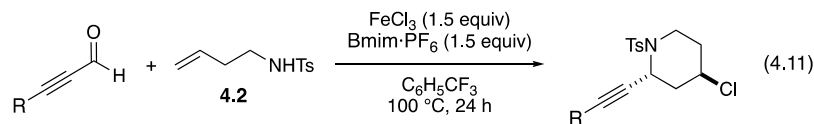


This method was adapted from a procedure reported by Ghosh.<sup>88</sup> To flame-dried round bottom flask equipped with a stir bar was added PPh<sub>3</sub> (4 equiv) and DCM (0.3 M in substrate). The reaction mixture was cooled to 0 °C and CBr<sub>4</sub> (2 equiv) was added. Then the desired aldehyde (1 equiv) was added dropwise to the reaction flask and was allowed to stir at 0 °C for 1 hour. The reaction mixture was concentrated. The residue was diluted with 1:1 Et<sub>2</sub>O:hexanes and filtered through a pad of silica gel. The filter cake was washed with excess 1:1 mixture of Et<sub>2</sub>O:hexanes.

To a flame-dried round bottom flask, was added the residue (1.0 equiv) and THF (0.4 M in substrate). The reaction mixture was cooled to -78 °C and *n*-BuLi (2.2 equiv) was added dropwise. After stirring at -78 °C for 1 h, the reaction flask was allowed to stir at rt for 1 h. The reaction mixture was cooled to -78 °C and the DMF (1.2 equiv) was added in one portion. After stirring at -78 °C for 1 h, the reaction mixture was quenched with NH<sub>4</sub>Cl, extracted with Et<sub>2</sub>O (x3), dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated. The residue was carried forward without purification.

<sup>88</sup> Ghosh, A. K.; Wang, Y. *J. Am. Chem. Soc.* **2000**, *122*, 11027–11028.

#### 4.5.8.6 Method H: FeCl<sub>3</sub>/Bmim·PF<sub>6</sub>-Promoted aza-Prins Reaction



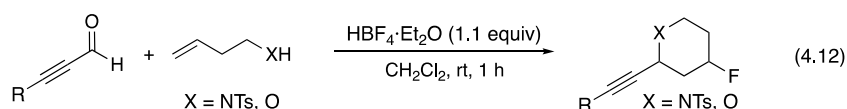
This method was adapted from a procedure reported by Iwamoto.<sup>17a</sup> In a glovebox, FeCl<sub>3</sub> (1.5 equiv) was added to a flame-dried round-bottom flask equipped with a stir bar. The flask was sealed with a septum and removed from the glovebox. Benzotrifluoride (0.3 M in FeCl<sub>3</sub>) was added to the flask, followed by dropwise addition of Bmim·PF<sub>6</sub> (1.5 equiv). In a separate flask, a solution of aldehyde (1.5 equiv) and homoallylic sulfonamide **2** (1.0 equiv) in benzotrifluoride (0.3 M in homoallylic sulfonamide) was prepared. Using a syringe, the solution of aldehyde and sulfonamide was added to the flask containing FeCl<sub>3</sub>. The reaction flask was fitted with a reflux condenser and N<sub>2</sub> inlet. The solution was heated to reflux and allowed to stir for 24 h. The reaction mixture was cooled to room temperature and was then quenched with H<sub>2</sub>O. The layers were separated, and the aqueous layer was extracted with Et<sub>2</sub>O (x 2). The combined organic layers were washed sequentially with sat. aq. Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, sat. aq. NaHCO<sub>3</sub>, and brine. The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, concentrated in vacuo, and purified by column chromatography. After purification by column chromatography, the bright orange product was passed through an activated charcoal plug to remove the colored impurities.

Frequently, the desired product was isolated as a mixture with unreacted aldehyde. To remove unreacted aldehyde from the desired product, the mixture was subjected to NaBH<sub>4</sub> reduction by a modified procedure reported by Wang and Franzén.<sup>89</sup> The unpurified reaction mixture was concentrated and dissolved in MeOH. NaBH<sub>4</sub> (1.6 equiv relative to 1.0 equiv of remaining aldehyde as determined by <sup>1</sup>H NMR integration) was added in one portion and the reaction stirred

<sup>89</sup> Wang, Y.; Franzén, R. *Synlett* **2012**, 23, 925–929.

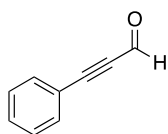
20 min at rt. The reaction mixture was then concentrated in vacuo to remove the MeOH. The resulting solid was dissolved in CH<sub>2</sub>Cl<sub>2</sub> and transferred to a separatory funnel. The organic layer was washed with water (x 3), brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo.

#### 4.4.8.7 Method I: HBF<sub>4</sub>·Et<sub>2</sub>O Mediated Prins/aza-Prins Reaction



This method was adapted from a procedure reported by Reddy.<sup>90</sup> A flame dried round bottom flask was charged with aldehyde (1 equiv) and homoallylic sulfonamide **4.2** or 3-buten-1-ol (1.1 equiv) in CH<sub>2</sub>Cl<sub>2</sub> (0.2 M in aldehyde). Then HBF<sub>4</sub>·Et<sub>2</sub>O (1 equiv) was added dropwise via syringe over 10 mins. The resulting deep red solution was allowed to stir for 1 h. Then the reaction was quenched with saturated NH<sub>4</sub>Cl and extracted with CH<sub>2</sub>Cl<sub>2</sub> (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, concentrated in vacuo, and purified by column chromatography.

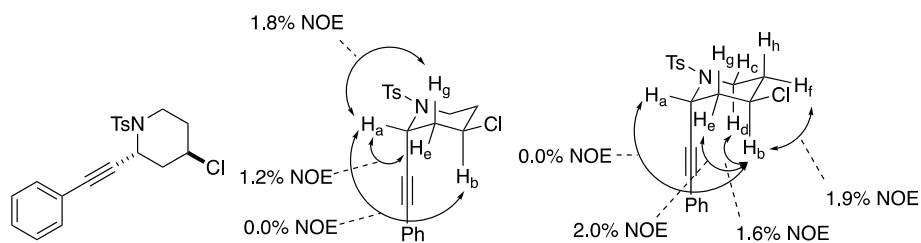
#### 4.5.9 Characterization Data for Starting Materials



**3-phenylpropionaldehyde (4.1)** was prepared according to Method C. The following amounts of reagents were used: phenylacetylene (0.55 mL, 5.0 mmol, 1.0 equiv), *n*-BuLi (4.2 mL, 5.0 mmol, 1.2 equiv, 1.2 M in THF), DMF (0.78 mL, 10. mmol, 2.0 equiv), THF (13 mL, 0.40 M). The residue was purified by column chromatography (0–5% EtOAc/hexanes) to afford the title compound as an orange oil (0.56 g, 4.3 mmol, 86% yield). **TLC R<sub>f</sub>** = 0.6 (10% EtOAc/hexanes); **<sup>1</sup>H NMR** (400

<sup>90</sup> Yadav, J. S.; Reddy, B. V. S.; Anusha, B.; Reddy, U. V. S.; Reddy, V. V. B. *Tetrahedron Lett.* **2010**, *51*, 2872–2874.

MHz, CDCl<sub>3</sub>)  $\delta$  9.42 (s, 1H), 7.63–7.57 (m, 2H), 7.48 (tt,  $J$  = 7.4, 1.4 Hz, 1H), 7.40 (ddd,  $J$  = 8.7, 6.7, 1.6 Hz, 2H). Analytical data are consistent with literature values.<sup>91</sup>

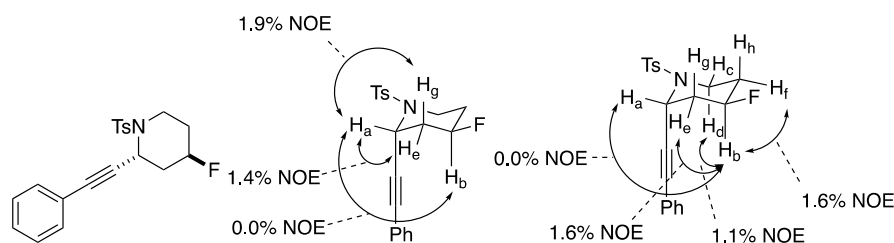


***trans*-4-Chloro-2-(phenylethynyl)-1-tosylpiperidine (4.3)** was prepared according to Method H.

The following amounts of reagents were used: 3-phenylpropionaldehyde **4.1** (0.45 g, 3.5 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (0.43 mL, 2.3 mmol, 1.0 equiv), FeCl<sub>3</sub> (0.56 g, 3.5 mmol, 1.5 equiv), Bmim·PF<sub>6</sub> (0.72 mL, 3.5 mmol, 1.5 equiv), benzotrifluoride (23 mL, 0.10 M in homoallylic sulfonamide). The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a pale yellow solid (0.46 g, 1.2 mmol, 53% yield, >20:1 dr *trans*:*cis*). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **m.p.** 107–109 °C; **TLC**  $R_f$  = 0.5 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (CDCl<sub>3</sub>, 500 MHz)  $\delta$  7.78 (d,  $J$  = 8.3 Hz, 2H), 7.38–7.33 (m, 1H), 7.32–7.25 (m, 4H), 7.04 (d,  $J$  = 8.5 Hz, 2H), 5.19 (as, 1H), 4.26 (tt,  $J$  = 12.0, 4.3 Hz, 1H), 3.91 (dt,  $J$  = 12.4, 2.1 Hz, 1H), 3.04 (td,  $J$  = 12.4, 2.6 Hz, 1H), 2.44 (ddt,  $J$  = 12.5, 4.1, 2.3 Hz, 1H), 2.34 (s, 3H), 2.33–2.27 (m, 1H), 2.23 (td,  $J$  = 12.7, 4.7 Hz, 1H), 2.04 (qd,  $J$  = 12.6, 4.8 Hz, 1H); **<sup>13</sup>C NMR** (CDCl<sub>3</sub>, 126 MHz)  $\delta$  143.7, 134.7, 131.5 (2C), 129.5 (2C), 128.7, 128.1 (2C), 128.1 (2C), 121.7, 87.7, 82.8, 52.9, 47.3, 42.0, 41.5, 35.9, 21.4; **IR** (neat) 2971, 2934, 2235, 1596, 1488, 1397, 1340, 1160, 1084, 856, 762, 723, 691 cm<sup>-1</sup>;

<sup>91</sup> Noro, M.; Masuda, T.; Ichimura, A. S.; Koga, N.; Iwamura, H. *J. Am. Chem. Soc.* **1994**, *116*, 6179–6190.

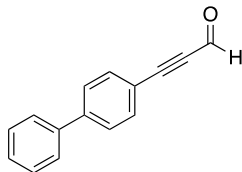
**HRMS** (TOF MS ES+)  $m/z$ : [M + Na] calculated for C<sub>20</sub>H<sub>20</sub>ClNO<sub>2</sub>SNa, 396.0801; found, 396.0807.



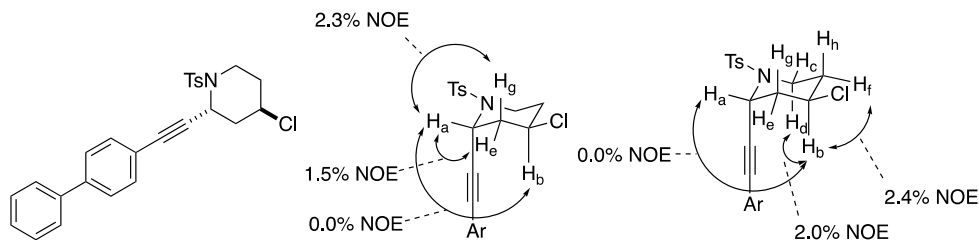
***trans*-4-Fluoro-2-(phenylethynyl)-1-tosylpiperidine (4.67)** was prepared according to Method I.

The following amounts of reagents were used: 3-phenylpropionaldehyde **4.1** (93 mg, 0.71 mmol, 1.0 equiv), homoallylic sulfonamide **4.2** (0.14 mL, 0.78 mmol, 1.1 equiv), HBF<sub>4</sub>·Et<sub>2</sub>O (0.10 mL, 0.71 mmol, 1.0 equiv), CH<sub>2</sub>Cl<sub>2</sub> (3.6 mL, 0.20 M). The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a pale yellow solid (118 mg, 0.33 mmol, 47% yield, 10:1 dr *trans*:*cis*). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. The major (*trans*) diastereomer is characterized below. **m.p.** 122–125 °C; **TLC** R<sub>f</sub> = 0.7 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (CDCl<sub>3</sub>, 500 MHz) δ 7.78 (d, *J* = 8.0 Hz, 2H), 7.38–7.21 (m, 5H), 7.04 (d, *J* = 7.1 Hz, 2H), 5.25 (s, 1H), 4.95 (dt, *J* = 48.2, 10.9, 4.8 Hz, 1H), 3.96 (d, *J* = 10.4 Hz, 1H), 3.04 (t, *J* = 12.5 Hz, 1H), 2.49–2.37 (m, 1H), 2.35 (s, 3H), 2.28–2.21 (m, 1H), 2.08 (tdd, *J* = 12.1, 8.2, 4.9 Hz, 1H), 1.99–1.84 (m, 1H); **<sup>13</sup>C NMR** (CDCl<sub>3</sub>, 126 MHz) δ 140.8, 134.7, 131.5 (2C), 129.5 (2C), 128.7, 128.2 (2C), 128.03 (2C), 121.6, 88.2, 86.8, 46.4 (d, *J* = 15.3 Hz), 40.5 (d, *J* = 14.0 Hz), 37.5 (d, *J* = 20.8 Hz), 32.5, 31.9 (d, *J* = 20.0 Hz), 21.5; **<sup>19</sup>F NMR** (565 MHz, CDCl<sub>3</sub>) δ –180.41 (d, *J* = 48.0 Hz); **IR** (neat) 2958, 2864, 2228, 1598, 1489, 1147, 1065, 814, 754 cm<sup>-1</sup>; **HRMS** (TOF MS ES+)  $m/z$ : [M + Na] calculated for C<sub>20</sub>H<sub>20</sub>FNO<sub>2</sub>SNa, 380.1096; found, 380.1096.

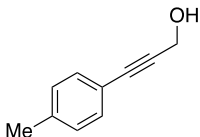




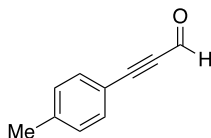
**3-([1,1'-Biphenyl]-4-yl)propionaldehyde (4.30)** see above for full characterization.



***trans*-2-([1,1'-Biphenyl]-4-ylethynyl)-4-chloro-1-tosylpiperidine (4.34)** was prepared according to Method H. The following amounts of reagents were used: aldehyde **4.30** (160 mg, 0.75 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (110 mg, 0.50 mmol, 1.0 equiv), FeCl<sub>3</sub> (120 mg, 0.75 mmol, 1.5 equiv), Bmim·PF<sub>6</sub> (0.15 mL, 0.75 mmol, 1.5 equiv), and benzotrifluoride (5.0 mL, 0.10 M in sulfonamide). The residue was purified by column chromatography (0–15% EtOAc/hexanes) to yield the title compound as a yellow solid (118 mg, 0.261 mmol, 52% yield, > 20:1 dr *trans*:*cis*). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **TLC R<sub>f</sub>** = 0.4 (15% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.73 (d, *J* = 8.3 Hz, 2H), 7.57–7.53 (m, 2H), 7.48–7.42 (m, 4H), 7.37 (tt, *J* = 7.3 Hz, 1.3 Hz, 1H), 7.23 (d, *J* = 7.9 Hz, 2H), 7.05 (d, *J* = 8.4 Hz, 2H), 5.17–5.13 (m, 1H), 4.22 (att, *J* = 11.9 Hz, 4.3 Hz, 1H), 3.86 (adquin, *J* = 12.2 Hz, 2.3 Hz, 1H), 2.99 (atd, *J* = 12.4 Hz, 2.5 Hz, 1H), 2.40 (adquin, *J* = 12.8 Hz, 2.0 Hz, 1H), 2.31–2.13 (m, 5H), 1.98 (aqd, *J* = 12.6 Hz, 4.9 Hz, 1H). See above ((+)-**35**) for full characterization.

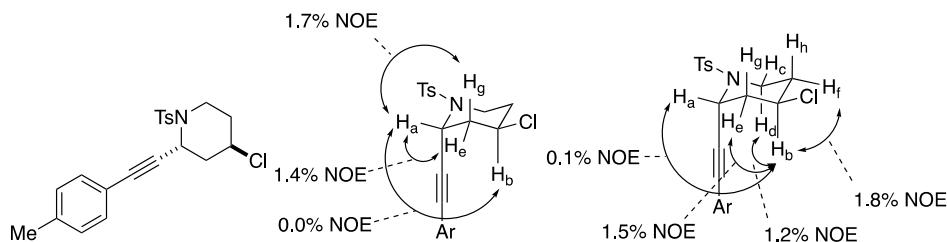


**3-(*p*-tolyl)prop-2-yn-1-ol (4.68)**<sup>92</sup> was prepared according to Method D. The following amounts of reagents were used: 1-bromo-4-methylbenzene (1.7 g, 10. mmol, 1.0 equiv), propargyl alcohol (0.76 mL, 13 mmol, 1.3 equiv), Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (140 mg, 0.20 mmol, 2.0 mol %), CuI (76 mg, 0.40 mmol, 4.0 mol %), triphenylphosphine (0.11 g, 0.40 mmol, 4.0 mol %), diisopropylamine (15 mL). The residue was purified by column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a yellow oil (0.65 g, 4.4 mmol, 44 % yield). **TLC** *R<sub>f</sub>* = 0.4 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.34 (d, *J* = 8.1 Hz, 2H), 7.13 (d, *J* = 3.2 Hz, 2H), 4.56–4.45 (m, 2H), 2.36 (s, 3H), 1.70 (s, 1H).

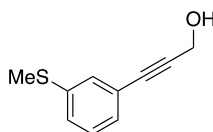


**3-(*p*-tolyl)propionaldehyde (4.69)** was prepared according to Method E. This reaction was performed in triplicate. The following amounts of reagents were used in each reaction flask: propargyl alcohol **4.68** (0.31 g, 2.2 mmol, 1.0 equiv), oxalyl chloride (0.24 mL, 2.8 mmol, 1.3 equiv), dimethyl sulfoxide (0.18 mL, 2.6 mmol, 1.2 equiv), triethylamine (0.91 mL, 6.5 mmol, 3.0 equiv), CH<sub>2</sub>Cl<sub>2</sub> (11 mL). The three reaction mixtures were combined, and the residue was purified by column chromatography (0–40% CH<sub>2</sub>Cl<sub>2</sub>/hexanes) to afford the title compound as a yellow oil (0.52 g, 3.6 mmol, 56% yield). **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 9.43 (s, 1H), 7.52 (d, *J* = 8.1 Hz, 2H), 7.23 (d, *J* = 8.1 Hz, 2H), 2.42 (s, 3H).

<sup>92</sup> Substrates **4.68** and **4.69** synthesized by Alissa C. Matus

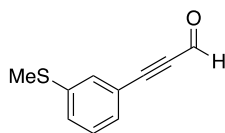


***trans*-4-chloro-2-(*p*-tolylethynyl)-1-tosylpiperidine (4.49)**<sup>93</sup> was prepared according to Method H. The following amounts of reagents were used: aldehyde **4.69** (0.52 g, 3.6 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (0.54 g, 2.4 mmol, 1.0 equiv), FeCl<sub>3</sub> (0.58 g, 3.6 mmol, 1.5 equiv), Bmim·PF<sub>6</sub> (0.74 mL, 3.6 mmol, 1.5 equiv), benzotrifluoride (60 mL). The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a pale yellow solid (0.60 g, 1.6 mmol, 66% yield, >20:1 dr *trans*:*cis*). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **m.p.** 107–109 °C; **TLC R<sub>f</sub>** = 0.6 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (CDCl<sub>3</sub>, 500 MHz) δ 7.71 (d, *J* = 8.3 Hz, 2H), 7.23–7.20 (m, 2H), 7.06–7.01 (m, 2H), 6.87 (d, *J* = 8.1 Hz, 2H), 5.12 (s, 1H), 4.20 (tt, *J* = 12.0, 4.3 Hz, 1H), 3.83 (ddt, *J* = 12.1, 4.5, 2.1 Hz, 1H), 2.97 (td, *J* = 12.5, 2.6 Hz, 1H), 2.37 (ddt, *J* = 12.6, 4.6, 2.4 Hz, 1H), 2.32 (s, 3H), 2.30 (s, 3H), 2.27–2.19 (m, 1H), 2.19–2.11 (m, 1H), 1.97 (qd, *J* = 12.6, 4.8 Hz, 1H); **<sup>13</sup>C NMR** (CDCl<sub>3</sub>, 126 MHz) δ 143.8, 138.9, 134.9, 131.5 (2C), 129.6 (2C), 129.0 (2C), 128.1 (2C), 118.7, 87.9, 82.1, 53.1, 47.5, 42.1, 41.6, 36.0, 21.6, 21.5; **IR** (neat) 2926, 2860, 2227, 1509, 1346, 1159, 915, 725 cm<sup>-1</sup>; **HRMS** (TOF MS ES<sup>+</sup>) *m/z*: [M + Na] calculated for C<sub>21</sub>H<sub>22</sub>ClNO<sub>2</sub>SNa, 410.0957; found, 410.0962.



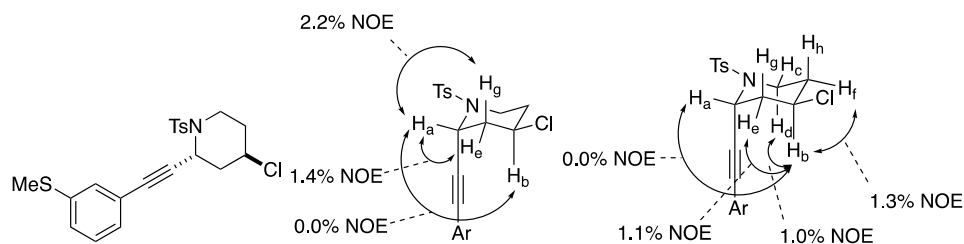
<sup>93</sup> Piperidine **4.49** synthesized by Alissa C. Matus. Characterization data collected by Kirsten A. Hewitt

**3-(3-(methylthio)phenyl)prop-2-yn-1-ol (4.70)**<sup>94</sup> was prepared according to Method D. The following amounts of reagents were used: (3-bromophenyl)(methyl)sulfane (1.4 mL, 10. mmol, 1.0 equiv), propargyl alcohol (0.76 mL, 13 mmol, 1.3 equiv), Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (140 mg, 0.20 mmol, 2.0 mol %), CuI (76 mg, 0.40 mmol, 4.0 mol %), triphenylphosphine (0.11 mg, 0.40 mmol, 4.0 mol %), diisopropylamine (30 mL). The residue was purified by column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a yellow oil (1.21g, 6.76 mmol, 68% yield). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.33–7.29 (m, 1H), 7.24–7.17 (m, 3H), 4.49 (d, *J* = 6.1 Hz, 2H), 2.48 (s, 3H), 1.67 (t, *J* = 6.2 Hz, 1H).



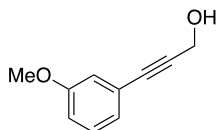
**3-(3-(methylthio)phenyl)propionaldehyde (4.71)** was prepared according to Method E. This reaction was run in quadruplicate. The following amounts of reagents were used in each reaction flask: propargyl alcohol **4.70** (0.30 g, 1.7 mmol, 1.0 equiv), oxalyl chloride (0.19 mL, 2.2 mmol, 1.3 equiv), dimethyl sulfoxide (0.14 mL, 2.1 mmol, 1.2 equiv), triethylamine (0.71 mL, 5.1 mmol, 3.0 equiv), CH<sub>2</sub>Cl<sub>2</sub> (9.7 mL). The four reaction mixtures were combined, and the residue was purified by column chromatography (0–40% CH<sub>2</sub>Cl<sub>2</sub>/hexanes) to afford the title compound as a yellow oil (0.65 g, 3.7 mmol, 55% yield). **TLC** *R<sub>f</sub>* = 0.4 (65% CH<sub>2</sub>Cl<sub>2</sub>/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 9.43 (s, 1H), 7.45 (t, *J* = 1.8 Hz, 1H), 7.38 (dd, *J* = 2.8, 1.7 Hz, 1H), 7.36 (dd, *J* = 1.7, 1.1 Hz, 1H), 7.34–7.29 (m, 1H), 2.51 (s, 3H).

<sup>94</sup> Substrates **4.70** and **4.71** synthesized by Alissa C. Matus.

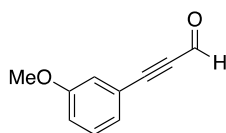


***trans*-4-chloro-2-((3-(methylthio)phenyl)ethynyl)-1-tosylpiperidine (4.50)**<sup>95</sup> was prepared according to Method H. The following amounts of reagents were used: aldehyde **4.71** (0.65 g, 3.6 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (0.53 g, 2.4 mmol, 1.0 equiv), FeCl<sub>3</sub> (0.59 g, 3.6 mmol, 1.5 equiv), Bmim·PF<sub>6</sub> (0.73 mL, 3.6 mmol, 1.5 equiv), benzotrifluoride (59 mL). The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a yellow solid (0.31 mg, 0.75 mmol, 31% yield, >20:1 dr *trans*:*cis*). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **m.p.** 99–101 °C; **TLC** *R<sub>f</sub>* = 0.4 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (CDCl<sub>3</sub>, 500 MHz) δ 7.71 (d, *J* = 8.3 Hz, 2H), 7.23 (d, *J* = 8.1 Hz, 2H), 7.18–7.12 (m, 2H), 6.84 (s, 1H), 6.78–6.73 (m, 1H), 5.13 (t, *J* = 2.1 Hz, 1H), 4.18 (tt, *J* = 12.0, 4.3 Hz, 1H), 3.85 (ddt, *J* = 12.2, 4.6, 2.2 Hz, 1H), 2.96 (td, *J* = 12.5, 2.7 Hz, 1H), 2.46 (s, 3H), 2.37 (ddt, *J* = 12.7, 4.5, 2.3 Hz, 1H), 2.32 (s, 3H), 2.27–2.20 (m, 1H), 2.16 (td, *J* = 12.4, 4.8 Hz, 1H), 1.97 (qd, *J* = 12.6, 4.8 Hz, 1H); **<sup>13</sup>C NMR** (CDCl<sub>3</sub>, 126 MHz) δ 143.9, 138.9, 134.7, 129.5 (2C), 129.4, 128.5, 128.06 (2C), 128.05, 126.6, 122.2, 87.2, 83.1, 52.9, 47.3, 42.0, 41.4, 35.9, 21.5, 15.7; **IR** (neat) 2923, 2859, 2250, 1584, 1345, 1158m 936, 725 cm<sup>-1</sup>; **HRMS** (TOF MS ES+) *m/z*: [M + Na] calculated for C<sub>21</sub>H<sub>22</sub>ClNO<sub>2</sub>S<sub>2</sub>Na, 442.0678; found, 442.0670.

<sup>95</sup> Piperidine **4.50** synthesized by Alissa C. Matus. Characterization data collected by Kirsten A. Hewitt

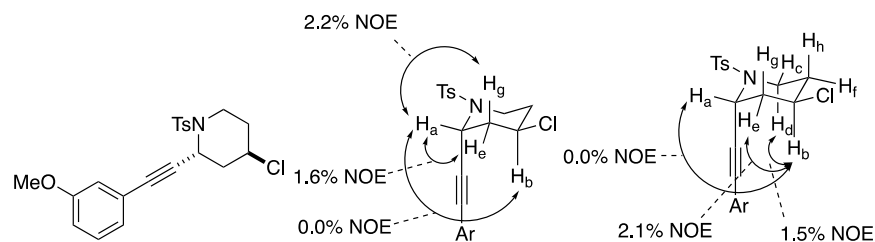


**3-(3-methoxyphenyl)prop-2-yn-1-ol (4.72)**<sup>96</sup> was prepared according to Method D. The following amounts of reagents were used: 1-iodo-3-methoxybenzene (0.60 mL, 5.0 mmol, 1.0 equiv), propargyl alcohol (0.32 mL, 0.32 mmol, 5.5 equiv), Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (35 mg, 50. μmol, 1.0 mol %), CuI (19 mg, 0.10 mmol, 2.0 mol %), triethylamine (15 mL). The residue was purified by column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a yellow oil (0.75 g, 4.7 mmol, 93% yield). **TLC** R<sub>f</sub> = 0.1 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.22 (t, *J* = 8.0 Hz, 1H), 7.03 (d, *J* = 7.6 Hz, 1H), 6.97 (s, 1H), 6.88 (dd, *J* = 8.3, 1.8 Hz, 1H), 4.50 (s, 2H), 3.80 (s, 3H), 1.72 (s, 1H).

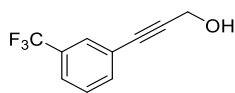


**3-(3-methoxyphenyl)propionaldehyde (4.73)** was prepared according to Method E. The reaction was run in triplicate. The following amounts of reagents were used in each reaction flask: propargyl alcohol **4.72** (0.25 g, 1.5 mmol, 1.0 equiv), oxalyl chloride (0.17 mL, 2.0 mmol, 1.3 equiv), dimethyl sulfoxide (0.13 mL, 1.8 mmol, 1.2 equiv), triethylamine (0.64 mL, 4.6 mmol, 3.0 equiv), CH<sub>2</sub>Cl<sub>2</sub> (9.2 mL). The three reaction mixtures were combined, and the residue was purified by column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a yellow oil (0.29 mg, 1.8 mmol, 59% yield). **TLC** R<sub>f</sub> = 0.5 (20% EtOAc/hexanes). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 9.42 (s, 1H), 7.31 (t, *J* = 7.8 Hz, 1H), 7.20 (dt, *J* = 7.6, 1.3 Hz, 1H), 7.13–7.10 (m, 1H), 7.04 (ddd, *J* = 8.3, 2.7, 1.0 Hz, 1H), 3.82 (s, 3H).

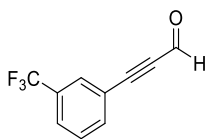
<sup>96</sup> Substrates **4.72**, **4.73** and **4.51** synthesized by Nadia Hirbawi.



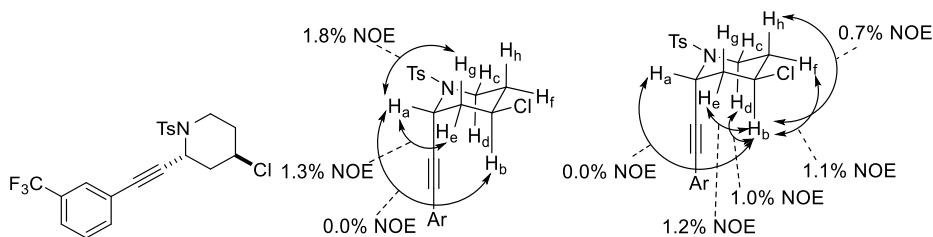
**4-chloro-2-((3-methoxyphenyl)ethynyl)-1-tosylpiperidine (4.51)** was prepared according to Method H. The following amounts of reagents were used: aldehyde **4.73** (0.29 g, 1.8 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (0.60 mL, 1.2 mmol, 1.0 equiv), FeCl<sub>3</sub> (0.29 mg, 1.8 mmol, 1.5 equiv), Bmim·PF<sub>6</sub> (0.37 mL, 1.8 mmol, 1.5 equiv), C<sub>6</sub>H<sub>5</sub>CF<sub>3</sub> (18 mL, 0.10 M in sulfonamide). The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a yellow solid (0.16 mg, 0.39 mmol, 32%, >20:1 dr trans:cis). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **m.p.** 115–117 °C; **TLC** R<sub>f</sub> = 0.5 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.71 (d, *J* = 8.1 Hz, 2H), 7.23 (d, *J* = 8.1 Hz, 2H), 7.14 (t, *J* = 8.0 Hz, 1H), 6.84 (dd, *J* = 8.4, 2.6 Hz, 1H), 6.55 (d, *J* = 7.6 Hz, 1H), 6.49 (s, 1H), 5.12 (s, 1H), 4.20 (tt, *J* = 12.0, 4.3 Hz, 1H), 3.85 (dt, *J* = 12.3, 2.2 Hz, 1H), 3.78 (s, 3H), 2.96 (td, *J* = 12.5, 2.6 Hz, 1H), 2.38 (ddt, *J* = 12.7, 4.5, 2.4 Hz, 1H), 2.30 (s, 3H), 2.23 (ddt, *J* = 12.1, 4.6, 2.3 Hz, 1H), 2.16 (td, *J* = 12.4, 4.8 Hz, 1H), 1.97 (qd, *J* = 12.6, 4.9 Hz, 1H); **<sup>13</sup>C NMR** (151 MHz, CDCl<sub>3</sub>) δ 159.3, 143.9, 134.8, 129.6 (2C), 129.3, 128.1 (2C), 124.1, 122.7, 117.1, 114.6, 87.7, 82.6, 55.4, 53.0, 47.4, 42.1, 41.5, 36.0, 21.5; **IR** (neat) 2925, 2856, 2232, 1597, 1345, 1158, 926, 725 cm<sup>-1</sup>; **HRMS** (TOF MS ES<sup>+</sup>) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>21</sub>H<sub>22</sub>ClNO<sub>3</sub>SNa, 426.0907; found, 426.0903.



**3-(3-Trifluoromethylphenyl)prop-2-yn-1-ol (4.74)**<sup>97</sup> was prepared according to Method D. The following amounts of reagents were used: 3-iodobenzotrifluoride (0.55 mL, 3.8 mmol, 1.0 equiv), propargyl alcohol (0.24 mL, 4.2 mmol, 1.1 equiv), CuI (14 mg, 76  $\mu$ mol, 2.0 mol %), Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (26 mg, 38  $\mu$ mol, 1.0 mol %), Et<sub>3</sub>N (11 mL). The compound was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as an orange oil (0.72 g, 3.6 mmol, 95% yield). **TLC**  $R_f$  = 0.4 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.70 (s, 1H), 7.63–7.54 (m, 2H), 7.44 (t,  $J$  = 7.8 Hz, 1H), 4.51 (as, 2H), 1.75 (br s, 1H). Analytical data are consistent with literature values.<sup>98</sup>



**3-(3-Trifluoromethylphenyl)propionaldehyde (4.75)** was prepared according to Method F. The following amounts of reagents were used: propargylic alcohol **4.74** (0.72 mg, 3.6 mmol, 1.0 equiv), MnO<sub>2</sub> (4.7 g, 54 mmol, 15 equiv), CH<sub>2</sub>Cl<sub>2</sub> (36 mL, 0.10 M). The compound was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a yellow oil (0.45 g, 2.4 mmol, 66% yield). **TLC**  $R_f$  = 0.6 (10% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  9.45 (s, 1H), 7.88 (s, 1H), 7.79 (d,  $J$  = 8.1 Hz, 1H), 7.75 (d,  $J$  = 7.8 Hz, 1H), 7.57 (t,  $J$  = 7.8 Hz, 1H).

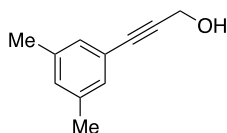


<sup>97</sup> Substrates **4.74**, **4.75**, and **4.52** synthesized by Nadia Hirbawi.

<sup>98</sup> Donnelly, K.; Zhang, H.; Baumann, M. *Molecules* **2019**, *24*, 3658–3671.

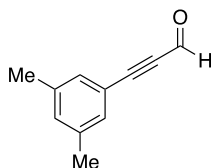


**trans-4-Chloro-2-((3-trifluoromethylphenyl)ethynyl)-1-tosylpiperidine (4.52)** was prepared according to Method H. The following amounts of reagents were used: aldehyde **4.75** (0.47 g, 2.4 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (0.39 mg, 1.6 mmol, 1.0 equiv), FeCl<sub>3</sub> (0.39 mg, 2.4 mmol, 1.5 equiv), Bmim·PF<sub>6</sub> (0.49 mL, 2.4 mmol, 1.5 equiv), and benzo-trifluoride (16 mL, 0.10 M). The residue was purified by column chromatography (0–10% EtOAc/hexanes) to yield the title compound as a white solid (160 mg, 0.35 mmol, 22% yield, >20:1 dr trans:cis). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **m.p.** 124–127°C; **TLC** R<sub>f</sub> = 0.7 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.72 (d, *J* = 8.1 Hz, 2H), 7.54 (d, *J* = 7.9 Hz, 1H), 7.38 (t, *J* = 7.8 Hz, 1H), 7.26–7.18 (m, 4H), 5.15 (as, 1H), 4.17 (tt, *J* = 12.0, 4.3 Hz, 1H), 3.88 (ddt, *J* = 12.4, 4.5, 2.2 Hz, 1H), 2.96 (td, *J* = 12.6, 2.7 Hz, 1H), 2.39 (ddt, *J* = 12.7, 4.2, 2.4 Hz, 1H), 2.30–2.14 (m, 2H), 2.27 (s, 3H), 1.99 (qd, *J* = 12.6, 4.8 Hz, 1H); **<sup>13</sup>C NMR** (150.9 MHz, CDCl<sub>3</sub>) δ 144.0, 134.7, 134.6, 130.9 (q, *J* = 32.6 Hz), 129.5 (2C), 128.9, 128.2 (q, *J* = 3.9 Hz), 128.1 (2C), 125.3 (q, *J* = 3.9 Hz), 123.6 (q, *J* = 272.5 Hz), 122.6, 86.1, 84.5, 52.7, 47.2, 42.1, 41.2, 35.8, 21.3; **<sup>19</sup>F NMR** (564.7 MHz, CDCl<sub>3</sub>) δ –63.0; **IR** (neat) 2926, 1596, 1434, 1329, 1248, 1158, 1122, 1072 cm<sup>-1</sup>; **HRMS** (TOF MS ES+) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>21</sub>H<sub>19</sub>ClF<sub>3</sub>NO<sub>2</sub>SNa, 464.0675; found, 464.0669.

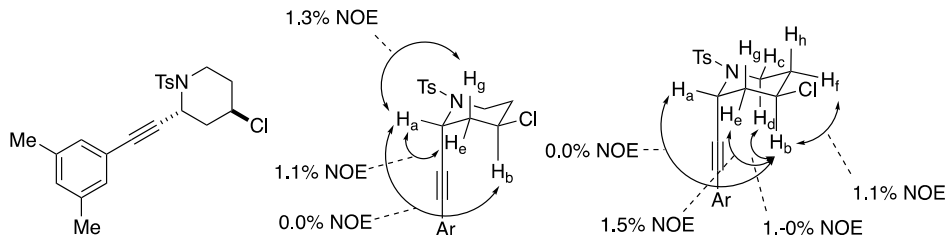


**3-(3,5-dimethylphenyl)prop-2-yn-1-ol (4.76)** was prepared according to Method D. The following amounts of reagents were used: 1-iodo-3,5-dimethylbenzene (0.85 mL, 3.0 mmol, 1.0 equiv), propargyl alcohol (0.19 mL, 3.3 mmol, 1.1 equiv), Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (21 mg, 30. μmol, 1.0 mol %), CuI (11 mg, 60 μmol, 2.0 mol %), triethylamine (9 mL). The residue was purified by column

chromatography (0–20% EtOAc/hexanes) to afford the title compound as a yellow oil (294 mg, 1.83 mmol, 61% yield).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.07 (s, 2H), 6.96 (s, 1H), 4.48 (d,  $J = 5.8$  Hz, 2H), 2.28 (d,  $J = 0.7$  Hz, 6H), 2.02–1.90 (m, 1H).

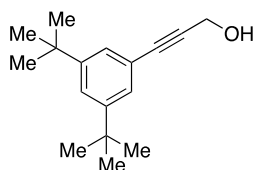


**3-(3,5-dimethylphenyl)propiolaldehyde (4.77)** was prepared according to Method F. The following amounts of reagents were used: propargyl alcohol **4.76** (0.29 mg, 1.8 equiv, 1.0 mmol),  $\text{MnO}_2$  (2.4 g, 27 mmol, 15 equiv),  $\text{Et}_2\text{O}$  (4.0 mL). The residue was purified by column chromatography (0–25% EtOAc/hexanes) to yield the title compound a yellow oil (130 mg, 0.82 mmol, 45% yield).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.38 (s, 1H), 7.20 (s, 2H), 7.09 (s, 1H), 2.30 (s, 6H).

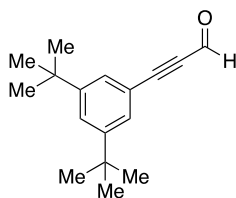


***trans*-4-chloro-2-((3,5-dimethylphenyl)ethynyl)-1-tosylpiperidine (4.53)** was prepared according to Method H. The following amounts of reagents were used: aldehyde **4.77** (130 mg, 0.82 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (0.10 mL, 0.55 mmol, 1.0 equiv),  $\text{FeCl}_3$  (0.11 mg, 0.82 mmol, 1.5 equiv),  $\text{Bmim}\cdot\text{PF}_6$  (0.17 mL, 0.82 mmol, 1.5 equiv), benzotrifluoride (5.5 mL). The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as tan solid (72 mg, 0.18 mmol, 22% yield, >20:1 dr *trans*:*cis*). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the  $^1\text{H NMR}$  spectrum. The relative configuration was assigned based on NOE analysis. **m.p.** 123–

125 °C TLC R<sub>f</sub> = 0.6 (20% EtOAc/hexanes); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 7.72 (d, *J* = 8.3 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 6.92 (s, 1H), 6.61 (s, 2H), 5.12 (t, *J* = 2.0 Hz, 1H), 4.20 (tt, *J* = 12.0, 4.3 Hz, 1H), 3.83 (ddt, *J* = 12.1, 4.4, 2.1 Hz, 1H), 2.97 (td, *J* = 12.5, 2.7 Hz, 1H), 2.37 (ddt, *J* = 12.4, 4.5, 2.4 Hz, 1H), 2.32 (s, 3H), 2.25 (s, 6H), 2.21 (dt, *J* = 4.5, 2.5 Hz, 1H), 2.16 (td, *J* = 12.4, 4.8 Hz, 1H), 1.97 (qd, *J* = 12.6, 5.0 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 126 MHz) δ 143.7, 137.8, 134.9, 130.6 (2C), 129.6 (2C), 129.3 (2C), 128.2 (2C), 121.4, 88.2, 82.0, 53.1, 47.4, 42.0, 41.7, 36.0, 21.6, 21.2 (2C); IR (neat) 2923, 2860, 2224, 1598, 1346, 1184, 932, 853, 726 cm<sup>-1</sup>; HRMS (TOF MS ES<sup>+</sup>) *m/z*: [M + Na] calculated for C<sub>22</sub>H<sub>24</sub>ClNO<sub>2</sub>SNa, 424.1114; found, 424.1113.

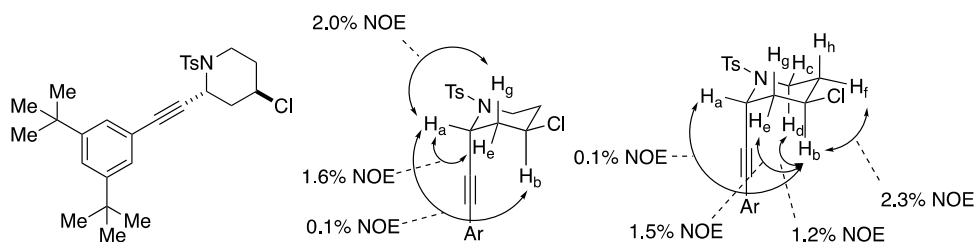


**3-(3,5-di-*tert*-butylphenyl)prop-2-yn-1-ol (4.78)**<sup>99</sup> was prepared according to Method D. The following amounts of reagents were used: 1-bromo-3,5-di-*tert*-butylbenzene (2.7 g, 10. mmol, 1.0 equiv), propargyl alcohol (0.76 mL, 13 mmol, 1.3 equiv), Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (140 mg, 0.20 mmol, 2.0 mol %), CuI (76 mg, 0.40 mmol, 4.0 mol %), triphenylphosphine (110 mg, 0.40 mmol, 4.0 mol %), diisoproylamine (15 mL). The residue was purified by column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a yellow solid (2.27 g, 9.20 mmol, 92 % yield). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) 7.41–7.36 (m, 1H), 7.31–7.28 (m, 2H), 7.27–7.23 (m, 1H), 4.50 (d, *J* = 6.0 Hz, 2H), 1.31 (s, 18H).



<sup>99</sup> Substrate **4.78** and **4.79** synthesized by Alissa Matus.

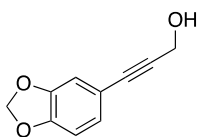
**3-(3,5-di-*tert*-butylphenyl)propionaldehyde (4.79)** was prepared according to Method E. Due to issue with scale, the reaction was run in four reaction flasks. The following amounts of reagents were used in each reaction flask: propargyl alcohol **4.78** (0.49 mg, 2.0 mmol, 1.0 equiv), oxalyl chloride (0.22 mL, 2.6 mmol, 1.3 equiv), dimethyl sulfoxide (0.17 mL, 2.4 mmol, 1.2 equiv), triethylamine (0.84 mL, 6.0 mmol, 3.0 equiv), CH<sub>2</sub>Cl<sub>2</sub> (12 mL). The four reaction mixtures were combined, and residue was purified by column chromatography (0–30% CH<sub>2</sub>Cl<sub>2</sub>/hexanes) to afford the title compound as a pale yellow oil (0.69 mg, 3.0 mmol, 33% yield). **TLC** *R<sub>f</sub>* = 0.3 (40% CH<sub>2</sub>Cl<sub>2</sub>/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 9.44 (s, 1H), 7.57 (t, *J* = 1.9 Hz, 1H), 7.48 (d, *J* = 1.9 Hz, 2H), 1.34 (s, 18H).



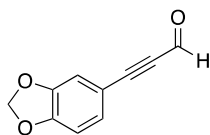
***trans*-4-Chloro-2-((3,5-di-*tert*-butylphenyl)ethynyl)-1-tosylpiperidine (4.54)**<sup>100</sup> was prepared according to Method H. The following amounts of reagents were used: Aldehyde **4.79** (0.69 g, 3.0 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (0.46 g, 2.0 mmol, 1.0 equiv), FeCl<sub>3</sub> (0.49 g, 3.0 mmol, 1.5 equiv), Bmim·PF<sub>6</sub> (0.62 mL, 3.0 mmol, 1.5 equiv), benzotrifluoride (50 mL). The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a pale yellow solid (0.35 g, 0.73 mmol, 36% yield, >20:1 dr *trans*:*cis*). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **m.p.** 60–62 °C; **TLC** *R<sub>f</sub>* = 0.7 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (CDCl<sub>3</sub>, 500 MHz) δ 7.74 (d, *J* = 8.4 Hz,

<sup>100</sup> Piperidine **4.54** synthesized by Alissa C. Matus. Characterization data collected by Kirsten A. Hewitt

2H), 7.39 (t,  $J = 1.9$  Hz, 1 H), 7.21 (d,  $J = 8.1$  Hz, 2H), 6.93 (d,  $J = 1.9$  Hz, 2H), 5.19 (t,  $J = 2.1$  Hz, 1H), 4.25 (tt,  $J = 12.0, 4.3$  Hz, 1H), 3.83 (dt,  $J = 12.3, 2.2$  Hz, 1H), 3.01 (td,  $J = 12.5, 2.6, 1$  H), 2.41 (ddt,  $J = 12.7, 4.4, 2.3$  Hz, 1H), 2.27–2.24 (m, 1H), 2.23 (s, 3H), 2.16 (td,  $J = 12.4, 4.8$  Hz, 1H), 1.97 (qd,  $J = 12.6, 4.9$  Hz, 1H), 1.30 (s, 18H);  $^{13}\text{C}$  NMR (CDCl<sub>3</sub>, 126 MHz)  $\delta$  150.9 (2C), 143.7, 135.1, 129.5 (2C), 128.2 (2C), 125.7 (2C), 123.2, 121.0, 88.8, 81.6, 53.2, 47.5, 42.0, 41.8, 36.1, 34.9 (2C), 31.4 (6C), 21.6; IR (neat) 2964, 2905, 2867, 2254, 1590, 1346, 1186, 904, 724 cm<sup>-1</sup>; HRMS (TOF MS ES+)  $m/z$ : [M + Na] calculated for C<sub>28</sub>H<sub>36</sub>ClNO<sub>2</sub>SNa, 508.2053; found, 508.2067.

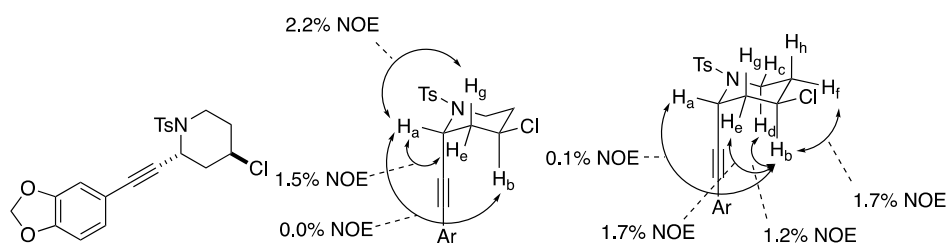


**3-(benzo[*d*][1,3]dioxol-5-yl)prop-2-yn-1-ol (4.80)**<sup>101</sup> was prepared according to Method D. The following amounts of reagents were used: 5-iodobenzo[*d*][1,3]dioxole (1.0 mL, 8.0 mmol, 1.0 equiv), propargyl alcohol (0.51 mL, 8.8 mmol, 1.1 equiv), Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (56 mg, 80.  $\mu$ mol, 1.0 mol %), CuI (30 mg, 0.16 mmol, 2.0 mol %), triethylamine (24 mL). The residue was purified by column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a white solid (1.29 g, 7.31 mmol, 91 % yield).  $^1\text{H}$  NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  6.97 (dd,  $J = 8.0, 1.6$  Hz, 1H), 6.88 (d,  $J = 1.6$  Hz, 1H), 6.75 (d,  $J = 8.0$  Hz, 1H), 5.97 (s, 2H), 4.47 (d,  $J = 6.2$  Hz, 2H), 1.57 (t,  $J = 6.2$  Hz, 1H).



<sup>101</sup> Substrates **4.80** and **4.81** synthesized by Alissa C. Matus.

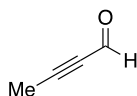
**3-(benzo[*d*][1,3]dioxol-5-yl)propionaldehyde (4.81)** was prepared according to Method E. Due to issue with scale, the reaction was run in four reaction flasks. The following amounts of reagents were used in each reaction flask: propargyl alcohol **4.80** (0.31 g, 1.6 mmol, 1.0 equiv), oxalyl chloride (0.20 mL, 2.3 mmol, 1.3 equiv), dimethyl sulfoxide (0.15 mL, 2.1 mmol, 1.2 equiv), triethylamine (0.74 mL, 5.3 mmol, 3.0 equiv), CH<sub>2</sub>Cl<sub>2</sub> (11 mL). The four reaction mixtures were combined, and the residue was purified by column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a yellow solid (108 mg, 1.20 mmol, 37 % yield). **TLC** *R<sub>f</sub>* = 0.6 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 9.38 (s, 1H), 7.19 (dd, *J* = 8.1, 1.6 Hz, 1H), 7.02 (d, *J* = 1.6 Hz, 1H), 6.83 (d, *J* = 8.0 Hz, 1H), 6.04 (s, 2H).



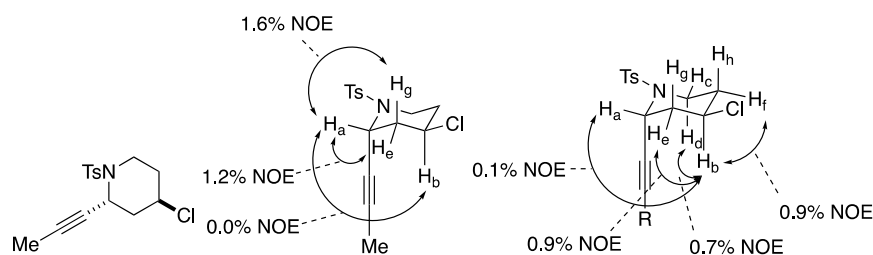
***trans*-2-(Benzo[*d*][1,3]dioxol-5-ylethynyl)-4-chloro-1-tosylpiperidine (4.55)**<sup>102</sup> was prepared according to Method H. The following amounts of reagents were used: aldehyde **4.81** (0.45 g, 2.6 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (0.39 mg, 1.7 mmol, 1.0 equiv), FeCl<sub>3</sub> (0.42 g, 2.6 mmol, 1.5 equiv), Bmim·PF<sub>6</sub> (0.54 mL, 2.6 mmol, 1.5 equiv), benzotrifluoride (43 mL). The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a white solid (58 mg, 0.14 mmol, 17% yield, >20:1 dr *trans*:*cis*). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **m.p.** 132–134 °C; **TLC** *R<sub>f</sub>* = 0.4 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (CDCl<sub>3</sub>, 500 MHz) δ 7.71 (d, *J* = 8.3 Hz,

<sup>102</sup> Piperidine **4.55** synthesized by Alissa C. Matus. Characterization data collected by Kirsten A. Hewitt

2H), 7.24 (d,  $J = 8.1$  Hz, 2H), 6.66 (d,  $J = 8.0$  Hz, 1H), 6.53 (dd,  $J = 8.0, 1.6$  Hz, 1H), 6.33 (d,  $J = 1.6$  Hz, 1H), 5.95 (d,  $J = 1.8$  Hz, 2H), 5.10 (t,  $J = 2.0$  Hz, 1H), 4.18 (tt,  $J = 12.0, 4.3$  Hz, 1H), 3.84 (dt,  $J = 12.1, 2.4$  Hz, 1H), 2.95 (td,  $J = 12.5, 2.6$  Hz, 1H), 2.38–2.35 (m, 1H), 2.34 (s, 3H), 2.26–2.19 (m, 1H), 2.15 (td,  $J = 12.4, 4.7$  Hz, 1H), 1.96 (qd,  $J = 12.6, 4.8$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 126 MHz)  $\delta$  148.2, 147.3, 143.8, 134.9, 129.6 (2C), 128.1 (2C), 126.2, 114.9, 111.6, 108.3, 101.51, 87.6, 81.2, 53.0, 47.4, 42.1, 41.6, 36.0, 21.5; IR (neat) 2927, 2254, 11598, 1346, 1160, 904, 723  $\text{cm}^{-1}$ ; HRMS (TOF MS ES+)  $m/z$ :  $[\text{M} + \text{Na}]$  calculated for  $\text{C}_{21}\text{H}_{20}\text{ClNO}_4\text{SNa}$ , 440.0699; found, 440.0692.

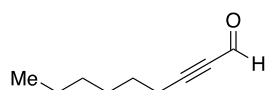


**but-2-ynal (4.82)** was prepared according to Method C. The following amounts of reagents were used: propyne (0.23 mL, 3.0 mmol, 1.0 equiv, 3% solution in heptane), *n*-BuLi (1.2 mL, 3.0 mmol, 1.0 equiv, 2.5 M in hexanes), DMF (0.46 mL, 6.0 mmol, 2 equiv), THF (7.5 mL, 0.40 M). Due to the volatility of the compound, it was carried onto the next step without further purification.

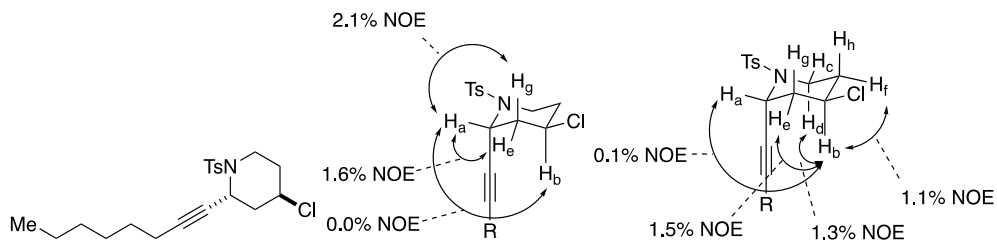


**Trans-4-chloro-2-(prop-1-yn-1-yl)-1-tosylpiperidine (4.28)** was prepared according to Method H. The following amounts of reagents were used: aldehyde **4.82** (3.0 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (0.37 mL, 2.0 mmol, 1.0 equiv),  $\text{FeCl}_3$  (0.48 g, 3.0 mmol, 1.5 equiv),  $\text{Bmim} \cdot \text{PF}_6$  (0.62 mL, 3.0 mmol, 1.5 equiv), benzotrifluoride (30 mL). The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound white solid (24 mg, 76  $\mu\text{mol}$ , 3% yield, >20:1 dr trans:cis). The dr was determined based on the integration of

the resonances attributed to the propargylic hydrogens in the  $^1\text{H}$  NMR spectrum. The relative configuration was assigned based on NOE analysis. **m.p.** 109–111 °C; **TLC**  $R_f$  = 0.3 (20% EtOAc/hexanes);  **$^1\text{H}$  NMR** ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  7.69 (d,  $J$  = 8.3 Hz, 2H), 7.29 (d,  $J$  = 8.0 Hz, 2H), 4.88 (dq,  $J$  = 4.5, 2.3 Hz, 1H), 4.18–4.07 (m, 1H), 3.73 (ddt,  $J$  = 12.3, 4.6, 2.2 Hz, 1H), 2.87 (td,  $J$  = 12.5, 2.6 Hz, 1H), 2.42 (s, 3H), 2.27–2.13 (m, 2H), 2.03 (td,  $J$  = 12.3, 4.7 Hz, 1H), 1.89 (qd,  $J$  = 12.6, 4.8 Hz, 1H), 1.46 (d,  $J$  = 2.3 Hz, 3H);  **$^{13}\text{C}$  NMR** ( $\text{CDCl}_3$ , 126 MHz)  $\delta$  143.6, 135.0, 129.2 (2C), 128.3 (2C), 83.7, 73.1, 53.1, 47.1, 41.8, 41.7, 36.0, 21.7, 3.3; **IR** (neat) 2918, 2860, 2229, 1596, 1340, 1159, 927, 727  $\text{cm}^{-1}$ ; **HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M} + \text{Na}]$  calculated for  $\text{C}_{15}\text{H}_{18}\text{ClNO}_2\text{SNa}$ , 334.0645; found, 334.0642



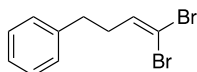
**non-2-ynal (4.83)** was prepared according to Method C. The following amounts of reagents were used: octyne (0.44 mL, 3.0 mmol, 1.0 equiv), *n*-BuLi (1.2 mL, 3.0 mmol, 1.0 equiv, 2.5 M in hexanes), dimethylformamide (0.46 mL, 6.0 mmol, 2.0 equiv), THF (7.5 mL). The residue was purified by column chromatography (0–10% EtOAc/hexanes) to afford the title compound as an orange oil (0.38 g, 2.7 mmol, 91% yield). The residue was carried into the next step without further purification.



**trans-4-Chloro-2-(oct-1-yn-1-yl)-1-tosylpiperidine (4.56)** was prepared according to Method H. The following amounts of reagents were used: aldehyde **4.83** (0.38 g, 2.7 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (0.33 mL, 1.8 mmol, 1.0 equiv),  $\text{FeCl}_3$  (0.44 mg, 2.7 mmol, 1.5

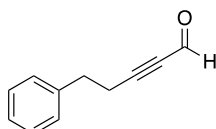


equiv), Bmim·PF<sub>6</sub> (0.56 mL, 2.7 mmol, 1.5 equiv), benzotrifluoride (18 mL). The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound as bright red oil (0.33 g, 0.86 mmol, 47% yield, >20:1 dr trans:cis). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **TLC** *R<sub>f</sub>* = 0.8 (10% EtOAc/hexanes); **<sup>1</sup>H NMR** (CDCl<sub>3</sub>, 500 MHz) δ 7.68 (d, *J* = 8.3 Hz, 2H), 7.27 (d, *J* = 8.2 Hz, 2H), 4.90 (t, *J* = 2.0 Hz, 1H), 4.13 (tt, *J* = 12.0, 4.3 Hz, 1H), 3.75 (ddt, *J* = 12.2, 4.6, 2.2 Hz, 1H), 2.87 (td, *J* = 12.5, 2.7 Hz, 1H), 2.41 (s, 3H), 2.28–2.13 (m, 2H), 2.04 (td, *J* = 12.3, 4.7 Hz, 1H), 1.98–1.73 (m, 3H), 1.33–1.22 (m, 2H), 1.21–1.13 (m, 6H), 0.88 (t, *J* = 7.1 Hz, 3H); **<sup>13</sup>C NMR** (CDCl<sub>3</sub>, 126 MHz) δ 143.6, 135.1, 129.3 (2C), 128.2 (2C), 88.5, 73.7, 53.1, 47.0, 41.8, 41.7, 36.0, 31.3, 28.6, 28.3, 22.6, 21.6, 18.5, 14.1; **IR** (neat) 2931, 2859, 2255, 1598, 1346, 1161, 904, 724 cm<sup>-1</sup>; **HRMS** (TOF MS ES+) *m/z*: [M + Na] calculated for C<sub>20</sub>H<sub>28</sub>ClNO<sub>2</sub>SNa, 404.1427; found, 404.1426.

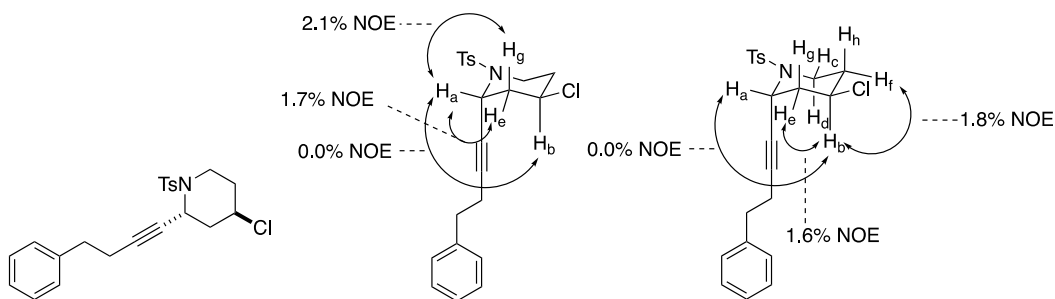


**(4,4-Dibromobut-3-en-1-yl)benzene (4.84)**<sup>103</sup> was prepared according to Method G. The following amounts of reagents were used: phenyl propionaldehyde (0.66 mL, 5.0 mmol, 1.0 equiv), CBr<sub>4</sub> (3.3 g, 10. mmol, 2.0 equiv), PPh<sub>3</sub> (5.2 g, 20. mmol, 4.0 equiv), and CH<sub>2</sub>Cl<sub>2</sub> (16 mL, 0.30 M in substrate). The residue was carried forward without further purification. **TLC** *R<sub>f</sub>* = 0.9 (100% hexanes) **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.33–7.27 (m, 2H), 7.24–7.16 (m, 3H), 6.41 (t, *J* = 7.1 Hz, 1H), 2.73 (t, *J* = 7.6 Hz, 2H), 2.41 (q, *J* = 7.53 Hz, 2H).

<sup>103</sup> substrates **4.84**, **4.85**, and **4.57** synthesized by Taylor A. Thane.

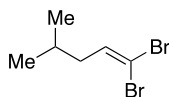


**5-Phenylpent-2-ynal (4.85)** was prepared according to Method G. The following amounts of reagents were used: **4.84** (0.92 g, 3.1 mmol, 1.0 equiv), *n*-BuLi (3.1 mL, 6.8 mmol, 2.2 equiv), DMF (0.29 mL, 3.7 mmol, 1.2 equiv), and THF (8 mL, 0.4 M in substrate). The residue was carried forward without further purification. **TLC**  $R_f$  = 0.5 (10% EtOAc/hexanes) **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  9.15 (s, 1H), 7.34–7.27 (m, 2H), 7.25–7.16 (m, 3H), 2.91 (t,  $J$  = 7.5 Hz, 2H), 2.71 (t,  $J$  = 7.4 Hz, 2H).

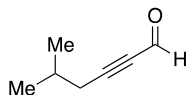


**trans-4-Chloro-2-(4-phenylbut-1-yn-1-yl)-1-tosylpiperidine (4.57)** was prepared according to Method H. The following amounts of reagents were used: **4.84** (0.28 g, 1.8 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (0.27 g, 1.2 mmol, 1.0 equiv), FeCl<sub>3</sub> (0.29 g, 1.8 mmol, 1.5 equiv), Bmim·PF<sub>6</sub> (0.37 mL, 1.8 mmol, 1.5 equiv), and benzotrifluoride (18 mL). The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a yellow solid (113 mg, 0.28 mmol, 23% yield, >20:1 dr trans:cis). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **m.p.** 111–113°C; **TLC**  $R_f$  = 0.7 (25% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.64 (d,  $J$  = 8.3 Hz, 2H), 7.29–7.19 (m, 5H), 7.02 (d,  $J$  = 7.4 Hz, 2H), 4.84–4.82 (m, 1H), 3.85 (tt,  $J$  = 11.9, 4.3 Hz, 1H), 3.65–3.60 (m, 1H), 2.60 (td,  $J$  = 12.4, 2.5 Hz, 1H), 2.56–2.43 (m, 2H), 2.41 (s, 3H), 2.24–2.07 (m, 3H), 2.04–

1.94 (m, 2H), 1.81 (qd,  $J = 12.6, 4.9$  Hz, 1H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  143.5, 140.2, 135.1, 129.3 (2C), 128.5 (2C), 128.4 (2C), 128.2 (2C), 126.6, 87.5, 74.9, 52.9, 46.9, 41.7 (2C), 35.9, 34.4, 21.7, 20.3; IR (neat) 3062, 3027, 2928, 2860 2232  $\text{cm}^{-1}$ ; HRMS (TOF MS ES+)  $m/z$ :  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{22}\text{H}_{24}\text{ClNO}_2\text{SNa}$ , 424.1114; found, 424.1102.

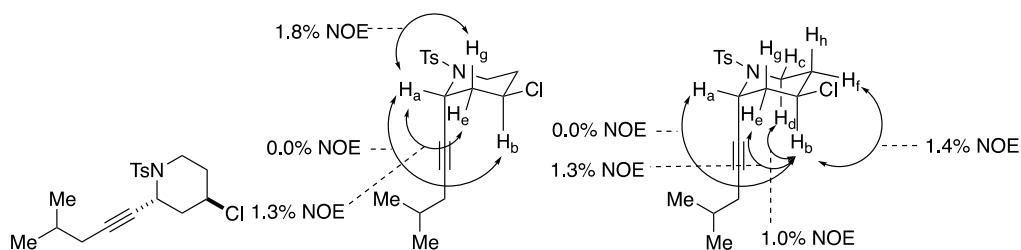


**1,1-Dibromo-4-methylpent-1-ene (4.86)**<sup>104</sup> was prepared according to Method G. The following amounts of reagents were used: isovaleraldehyde (0.54 mL, 5.0 mmol, 1.0 equiv),  $\text{CBr}_4$  (3.3 g, 10. mmol, 2.0 equiv),  $\text{PPh}_3$  (5.2 g, 20. mmol, 4.0 equiv), and DCM (15 mL). The residue was passed through a silica plug eluting with 100% hexanes. The resulting oil was carried forward without further purification. TLC  $R_f = 0.9$  (100% hexanes);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.40 (t,  $J = 7.5$  Hz, 1H), 1.99 (t,  $J = 7.1$  Hz, 2H), 1.75 (sept,  $J = 6.7$  Hz, 1H), 0.93 (d,  $J = 6.6$  Hz, 6H).

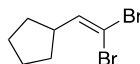


**5-Methylhex-2-ynal (4.87)** was prepared according to Method G. The following amounts of reagents were used: **4.86** (0.88 g, 3.7 mmol, 1.0 equiv),  $n\text{-BuLi}$  (3.2 mL, 8.0 mmol, 2.2 equiv), DMF (0.34 mL, 4.4 mmol, 1.2 equiv), and THF (9 mL). The residue was carried forward without further purification. TLC  $R_f = 0.5$  (10% hexanes);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  9.19 (s, 1H), 2.31 (d,  $J = 6.6$  Hz, 2H), 1.94 (sept,  $J = 6.6$  Hz, 1H), 1.02 (d,  $J = 6.8$  Hz, 6H).

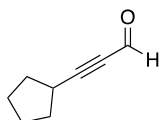
<sup>104</sup> Substrates **4.86**, **4.87**, and **4.58** synthesized by Taylor A. Thane



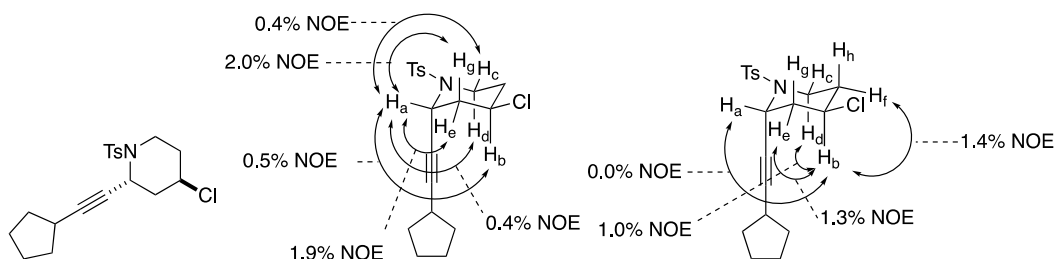
***trans*-4-Chloro-2-(4-methylpent-1-yn-1-yl)-1-tosylpiperidine (4.58)** was prepared according to Method H. The following amounts of reagents were used: aldehyde **4.87** (0.17 g, 1.6 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (0.23 g, 1.0 mmol, 1.0 equiv), FeCl<sub>3</sub> (0.25 g, 1.6 mmol, 1.5 equiv), Bmim·PF<sub>6</sub> (0.32 mL, 1.6 mmol, 1.5 equiv), and benzotrifluoride (15 mL). The residue was purified by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as pale yellow solid (59 mg, 0.16 mmol, 10% yield, >20:1 dr *trans*:*cis*). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **m.p.** 63–65 °C; **TLC** **R<sub>f</sub>** = 0.7 (25% EtOAc/hexanes); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.69 (d, *J* = 8.3 Hz, 2H), 7.27 (d, *J* = 8.3 Hz, 2H), 4.94–4.91 (m, 1H), 4.14 (tt, *J* = 12.1, 4.2 Hz, 1H), 3.78–3.74 (m, 1H), 2.90 (td, *J* = 12.4, 2.7 Hz, 1H), 2.41 (s, 3H), 2.27–2.23 (m, 1H), 2.21–2.16 (m, 1H), 2.0 (td, *J* = 12.5, 4.8, 1H), 1.90 (qd, *J* = 12.5, 4.4, 1H), 1.73 (qdd, *J* = 13.8, 6.9, 1.9, 2H), 1.48 (sept, *J* = 6.7, 1H), 0.79 (d, *J* = 6.7, 3H), 0.77 (d, *J* = 6.7, 3H); **<sup>13</sup>C NMR** (125.8 MHz, CDCl<sub>3</sub>) δ 143.5, 135.3, 129.4 (2C), 128.2 (2C), 87.3, 74.8, 53.2, 47.0, 42.0, 41.8, 36.1, 27.82, 27.76, 22.07, 22.05, 21.6; **IR** (neat) 2957, 2927, 2868, 2230 cm<sup>-1</sup>; **HRMS** (TOF MS ES<sup>+</sup>) *m/z*: [M + Na]<sup>+</sup> calcd for C<sub>18</sub>H<sub>24</sub>ClNO<sub>2</sub>SNa, 376.1114; found, 376.1108.



**(2,2-dibromovinyl)cyclopentane (4.88)**<sup>105</sup> was prepared according to Method G. The following amounts of reagents were used: cyclopentanecarboxaldehyde (0.53 mL, 5.0 mmol, 1.0 equiv), CBr<sub>4</sub> (3.3 g, 10. mmol, 2.0 equiv), PPh<sub>3</sub> (5.2 g, 20. mmol, 4.0 equiv), and CH<sub>2</sub>Cl<sub>2</sub> (16 mL, 0.30 M in substrate). The residue was carried forward without further purification. **TLC R<sub>f</sub>** = 0.9 (100% hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 6.31 (d, *J* = 9.0 Hz, 1H), 2.67 (sext, *J* = 8.7 Hz, 1H), 1.91–1.87 (m, 2H), 1.69–1.56 (m, 4H), 1.32–1.26 (m, 2H).



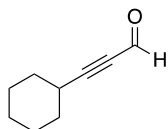
**3-cyclopentylpropionaldehyde (4.89)** was prepared according to Method G. The following amounts of reagents were used: **4.88** (0.75 g, 2.9 mmol, 1.0 equiv), *n*-BuLi (2.6 mL, 6.5 mmol, 2.2 equiv), DMF (0.28 mL, 3.6 mmol, 1.2 equiv), and THF (8.0 mL, 0.40 M in substrate). The residue was carried forward without further purification. **TLC R<sub>f</sub>** = 0.6 (10% EtOAc/hexanes) **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 9.18 (s, 1H), 2.86–2.78 (m, 1H), 2.03–1.96 (m, 2H), 1.79–1.67 (m, 4H), 1.66–1.58 (m, 2H).



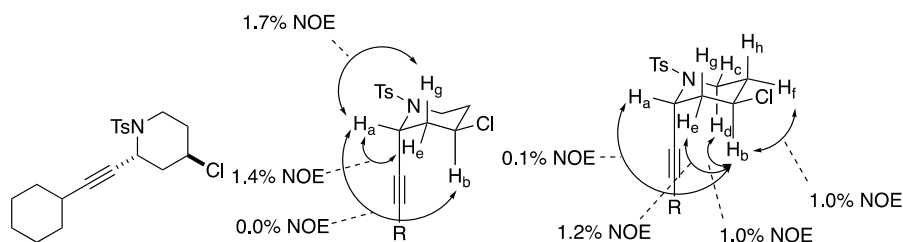
**trans-4-chloro-2-(cyclopentylethynyl)-1-tosylpiperidine (4.59)** was prepared according to Method H. The following amounts of reagents were used: **4.89** (180 mg, 1.5 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (230 mg, 1.0 mmol, 1.0 equiv), FeCl<sub>3</sub> (240 mg, 1.5 mmol, 1.5 equiv), Bmim·PF<sub>6</sub> (0.31 mL, 1.5 mmol, 1.5 equiv), and benzotrifluoride (15 mL). The residue was purified

<sup>105</sup> Substrates **4.88**, **4.89**, and **4.59** synthesized by Taylor A. Thane.

by flash column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a pale-yellow solid (220 mg, 0.59 mmol, 5.9% yield over 3 steps, >20:1 dr trans:cis). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the  $^1\text{H}$  NMR spectrum. The relative configuration was assigned based on NOE analysis. **m.p.** 84–86 °C; **TLC**  $R_f$  = 0.7 (25% EtOAc/hexanes);  **$^1\text{H}$  NMR** (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.67 (d,  $J$  = 8.2 Hz, 2H), 7.27 (d,  $J$  = 8.2 Hz, 2H), 4.91–4.88 (m, 1H), 4.13 (tt,  $J$  = 11.8, 4.4 Hz, 1H), 3.78–3.73 (m, 1H), 2.89 (td,  $J$  = 12.5, 2.5 Hz, 1H), 2.41 (s, 3H), 2.31–2.15 (m, 3H), 2.05 (td,  $J$  = 12.3, 4.9 Hz, 1H), 1.90 (qd,  $J$  = 12.3, 4.9 Hz, 1H), 1.72–1.59 (m, 2H), 1.69–1.62 (m, 2H), 1.51–1.42 (m, 2H), 1.23–1.13 (m, 2H);  **$^{13}\text{C}$  NMR** (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  143.5, 135.4, 129.5 (2C), 128.1 (2C), 92.8, 73.3, 53.2, 47.0, 42.0, 41.7, 36.1, 33.6, 33.5, 29.8, 25.04, 25.01, 21.6; **IR** (neat) 2958, 2868, 2232, 1598; **HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{19}\text{H}_{24}\text{ClNO}_2\text{SNa}$ , 388.1114; found, 388.1107.

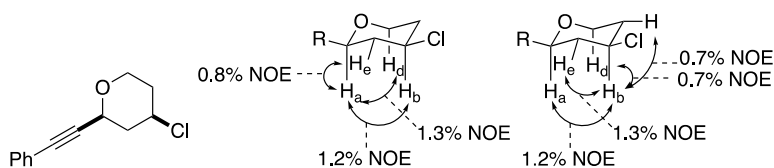


**3-cyclohexylpropionaldehyde (4.90)** was prepared according to Method C. The following amounts of reagents were used: ethynylcyclohexane (0.65 mL, 5.0 mmol, 1.0 equiv), *n*-BuLi (4.5 mL, 5.0 mmol, 1.0 equiv, 2.5 M in hexanes), DMF (0.77 mL, 10. mmol, 2.0 equiv), THF (13 mL). The residue was purified by column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a yellow solid (0.39 g, 2.9 mmol, 58% yield). **TLC**  $R_f$  = 0.8 (5% EtOAc/hexanes);  **$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.18 (s, 1H), 2.58 (tt,  $J$  = 8.8, 3.9 Hz, 1H), 1.84 (ddt,  $J$  = 12.8, 6.7, 3.5 Hz, 2H), 1.75 – 1.65 (m, 2H), 1.51 (ddd,  $J$  = 13.3, 9.6, 6.9 Hz, 4H), 1.34 (tdd,  $J$  = 10.1, 5.9, 2.6 Hz, 3H).



***trans*-4-chloro-2-(cyclohexylethynyl)-1-tosylpiperidine (4.60)** was prepared according to Method H. The following amounts of reagents were used: aldehyde **4.90** (0.63 g, 4.6 mmol, 1.5 equiv), homoallylic sulfonamide **4.2** (0.56 mL, 3.1 mmol, 1.0 equiv), FeCl<sub>3</sub> (0.74 g, 4.63 mmol, 1.5 equiv), Bmim·PF<sub>6</sub> (0.94 mL, 4.6 mmol, 1.5 equiv), benzotrifluoride (31 mL). The residue was purified by flash column chromatography (0–20% EtOAc/hexanes) to afford the title compound white solid (0.67 mg, 1.8 mmol, 57% yield, >20:1 dr *trans*:*cis*). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **m.p.** 110–113 °C; **TLC** R<sub>f</sub> = 0.7 (20% EtOAc/hexanes); **<sup>1</sup>H NMR** (CDCl<sub>3</sub>, 500 MHz) δ 7.74 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.1 Hz, 2H), 4.96 (s, 1H), 4.20 (tt, *J* = 12.0, 4.3 Hz, 1H), 3.82 (ddt, *J* = 12.2, 4.6, 2.2 Hz, 1H), 2.96 (td, *J* = 12.5, 2.7 Hz, 1H), 2.47 (s, 3H), 2.35 – 2.20 (m, 2H), 2.16 – 2.04 (m, 2H), 1.97 (qd, *J* = 12.6, 4.9 Hz, 1H), 1.62 – 1.46 (m, 6H), 1.32 – 1.17 (m, 3H), 1.16 – 1.01 (m, 2H); **<sup>13</sup>C NMR** (CDCl<sub>3</sub>, 126 MHz) δ 143.4, 135.3, 129.4, 128.0, 92.6, 73.6, 53.1, 46.9, 41.9, 41.6, 36.0, 32.3, 32.2, 28.8, 25.7, 24.8, 21.5; **IR** (neat) 2927, 2853, 2230, 1598, 1346, 1185, 933, 726 cm<sup>-1</sup>; **HRMS** (TOF MS ES+) *m/z*: [M + Na]<sup>+</sup> calculated for C<sub>20</sub>H<sub>28</sub>ClNO<sub>2</sub>SNa 402.1270, found 402.1272.

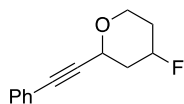
#### 4.5.10. Synthesis and Characterization of Tetrahydropyran Substrates



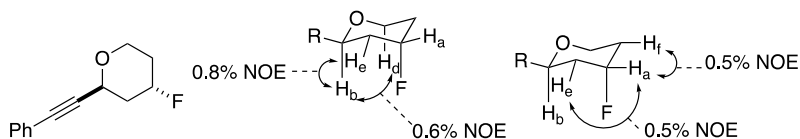
***trans*-4-Chloro-2-(phenylethynyl)tetrahydro-2H-pyran (4.91)** was prepared according to a modified procedure reported by Grée.<sup>106</sup> In a glovebox, ZnCl<sub>2</sub> (0.39 g, 2.9 mmol, 1.1 equiv) was added to a flame dried flask equipped with a stir bar. The flask was sealed with a septum, removed from the glovebox, and placed under an inert atmosphere of N<sub>2</sub>. Then *p*-TsOH (0.54 g, 2.9 mmol, 1.1 equiv) and CH<sub>2</sub>Cl<sub>2</sub> (13 mL) were added via syringe. In a separate flame dried round bottom flask, aldehyde **4.1** (0.33 g, 2.6 mmol, 1.0 equiv), 3-butenol (0.25 mL, 2.9 mmol, 1.1 equiv) were dissolved in CH<sub>2</sub>Cl<sub>2</sub> (13 mL). This mixture was allowed to stir for 5 mins at rt. Then the solution containing aldehyde and 3-butenol was added to the solution of ZnCl<sub>2</sub> and *p*-TsOH. The reaction mixture was allowed to stir at rt overnight. Sat. NaHCO<sub>3</sub> was added to quench the reaction mixture. The biphasic solution was extracted with CH<sub>2</sub>Cl<sub>2</sub> (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated in vacuo. The residue was purified by column chromatography (0–20% EtOAc/hexanes) to afford the title compound as a white solid (0.21 g, 0.97 mmol, 37% yield, >20:1 dr *cis*:*trans*). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the <sup>1</sup>H NMR spectrum. The relative configuration was assigned based on NOE analysis. **m.p.** 51–53 °C; **TLC R<sub>f</sub>** = 0.8 (20% EtOAc/hexanes). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.48–7.42 (m, 2H), 7.35–7.27 (m, 3H), 4.30 (dd, *J* = 11.2, 2.3 Hz, 1H), 4.11 (ddd, *J* = 12.2, 4.7, 2.1 Hz, 1H), 4.02 (tt, *J* = 11.5, 4.5 Hz, 1H), 3.49 (td, *J* = 12.1, 2.1 Hz, 1H), 2.47 (ddt, *J* = 13.1, 4.4, 2.2 Hz, 1H), 2.16–2.06 (m, 2H), 1.95 (qd, *J* = 12.5, 12.0, 4.8 Hz, 1H); **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 132.0 (2C), 128.8, 128.4 (2C), 122.3, 86.6, 85.4, 67.9, 67.0, 54.3, 43.0, 36.5; **IR** (neat) 2861, 2835, 2234, 1595, 1488, 1364, 1212, 1022, 757, 724 cm<sup>-1</sup>; **HRMS** (MS TOF Cl<sup>+</sup>) *m/z*: [M–Cl]<sup>+</sup> calculated for C<sub>13</sub>H<sub>13</sub>O, 185.0966; found, 185.0961.

<sup>106</sup> Borkar, P.; van de Weghe, O.; Reddy, B. V. S.; Yadav, J. S.; Grée, R. *Chem. Commun.* **2012**, 48, 9316–9318.

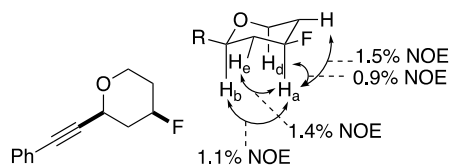




**4-fluoro-2-(phenylethynyl)tetrahydro-2H-pyran (4.92)** was prepared according to Method I. The following amounts of reagents were used: aldehyde **4.1** (0.50 g, 3.8 mmol, 1.0 equiv), 3-butenol (0.36 mL, 4.2 mmol, 1.1 equiv),  $\text{HBF}_4 \cdot \text{Et}_2\text{O}$  (0.52 mL, 3.8 mmol, 1.0 equiv).  $\text{CH}_2\text{Cl}_2$  (19 mL, 0.20 M). The residue was purified by column chromatography and the diastereomers were separated (0–10% EtOAc/hexanes) to afford the *trans*-diastereomer as a pale yellow solid (0.22 g, 1.1 mmol, 29% yield, >20:1 dr *trans:cis*) and the *cis*-diastereomer as a white solid (0.20 g, 0.99 mmol, 26% yield, >20:1 dr *cis:trans*). The dr was determined based on the integration of the resonances attributed to the propargylic hydrogens in the  $^1\text{H}$  NMR spectrum. The relative configuration was assigned based on NOE analysis. For clarity, the data of the major and minor diastereomers have been tabulated individually.

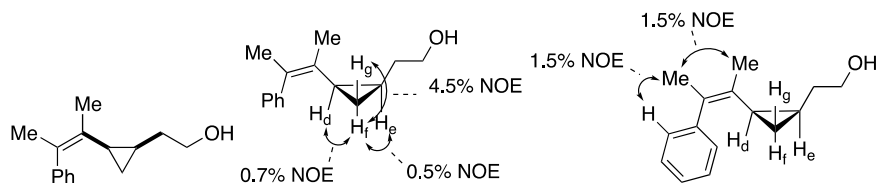
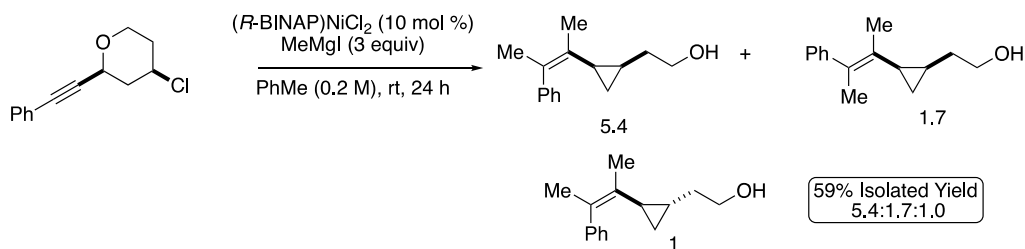


***trans*-diastereomer:** TLC  $R_f = 0.5$  (5% EtOAc/hexanes);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.54–7.47 (m, 2H), 7.41–7.34 (m, 3H), 5.11 (dtt,  $J = 48.1, 5.9, 3.2$  Hz, 1H), 4.88 (dd,  $J = 8.7, 3.6$  Hz, 1H), 4.07–4.00 (m, 1H), 3.96 (tdd,  $J = 11.8, 3.4, 1.9$  Hz, 1H), 2.26–2.11 (m, 2H), 2.10–1.85 (m, 2H);  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  131.9 (2C), 128.7, 128.4 (2C), 122.4, 87.12, 87.09, 85.79 (d,  $J = 23.4$  Hz), 63.92 (d,  $J = 4.7$  Hz), 62.34 (d,  $J = 4.1$  Hz), 37.58 (d,  $J = 19.6$  Hz), 31.35 (d,  $J = 19.7$  Hz);  $^{19}\text{F}$  NMR (565 MHz,  $\text{CDCl}_3$ )  $\delta$  –183.2 (s); IR (neat) 2958, 2864, 2228, 1598, 1498, 1147, 1086, 754, 690  $\text{cm}^{-1}$ ; HRMS (TOF MS  $\text{Cl}^+$ )  $m/z$ :  $[\text{M} + \text{NH}_4]^+$  calculated for  $\text{C}_{13}\text{H}_{21}\text{FN}_2\text{O}$ , 222.1294; found, 222.1285.



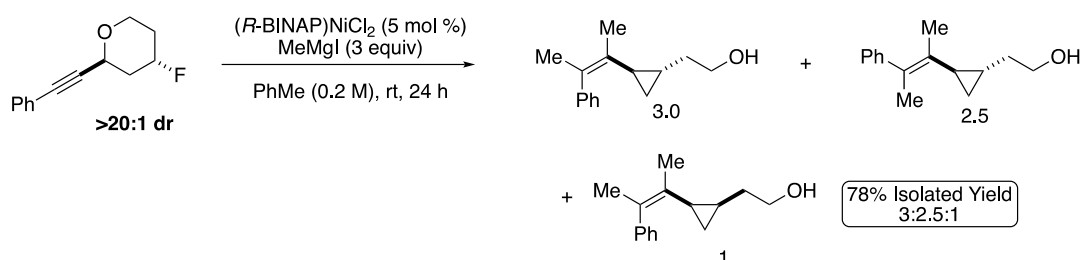
**cis-diastereomer:** TLC  $R_f$  = 0.4 (5% EtOAc/hexanes);  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.48–7.42 (m, 2H), 7.34–7.27 (m, 3H), 4.72 (dt,  $J$  = 48.6, 9.8, 4.7 Hz, 1H), 4.35 (ddd,  $J$  = 10.3, 2.8, 1.9 Hz, 1H), 4.16 (dtd,  $J$  = 12.0, 5.0, 3.4 Hz, 1H), 3.52–3.44 (m, 1H), 2.42–2.34 (m, 1H), 2.08–1.92 (m, 2H), 1.89–1.77 (m, 1H);  $^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  131.9 (2C), 128.6, 128.3 (2C), 122.3, 88.4, 87.0, 86.8 (d,  $J$  = 2.3 Hz), 66.0 (d,  $J$  = 12.4 Hz), 64.2 (d,  $J$  = 11.0 Hz), 38.7 (d,  $J$  = 18.5 Hz), 32.4 (d,  $J$  = 18.2 Hz);  $^{19}\text{F NMR}$  (565 MHz,  $\text{CDCl}_3$ )  $\delta$  –172.41 (d,  $J$  = 48.3 Hz); **IR** (neat) 2958, 2864, 2228, 1598, 1498, 1147, 1086, 754, 690  $\text{cm}^{-1}$ ; **HRMS** (TOF MS  $\text{Cl}^+$ )  $m/z$ :  $[\text{M} + \text{NH}_4]^+$  calculated for  $\text{C}_{13}\text{H}_{21}\text{FN}_2\text{O}$ , 222.1294; found, 222.1283.

#### 4.5.10.1 Results from Tetrahydropyran Ring Contraction

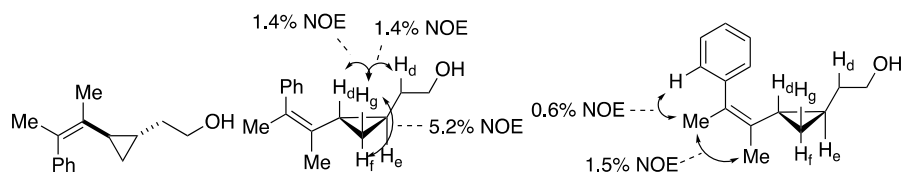


**(3-phenylbut-2-en-2-yl)cyclopropyl)ethan-1-ol (4.93)** was prepared according to a modified version of Method A. The reaction was performed in triplicate. The following amounts of reagents were used in each flask: **4.92** (44 mg, 0.20 mmol, 1.0 equiv), (*R*-BINAP) $\text{NiCl}_2$  (15 mg, 20.  $\mu\text{mol}$ , 10. mol %),  $\text{MeMgI}$  (0.24 mL, 0.60 mmol, 2.5 M in  $\text{Et}_2\text{O}$ , 3.0 equiv), and  $\text{PhMe}$  (1.0 mL, 0.20 M).

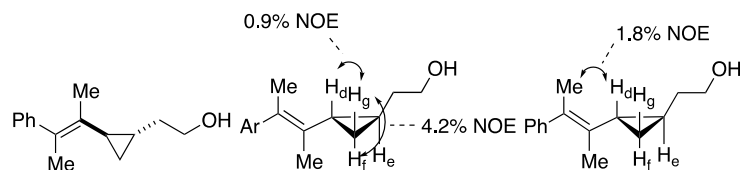
The reaction mixtures were combined, and residue was purified by column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a yellow oil (76 mg, 0.35 mmol, 59% yield, 5.4:1.7:1.0 mixture of diastereomers). The dr was based on the integration of resonances associated with H<sub>g</sub> in the <sup>1</sup>H NMR spectrum. The relative configuration of the major diastereomer was assigned based on NOE analysis. The <sup>1</sup>H NMR of the major diastereomer is given below. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.23–7.15 (m, 4H), 7.13–7.08 (m, 1H), 3.64 (t, *J* = 6.7 Hz, 2H), 2.09–2.06 (m, 2H), 2.01 (s 3H), 1.77 (s, 3H), 1.15–1.03 (m, 2H), 0.82–0.70 (m, 1H), 0.56 (tdd, *J* = 8.4, 5.0, 0.9 Hz, 1H), –0.03 (q, *J* = 5.6 Hz, 1H).



#### 4.5.10.2 NOE correlations for major diastereomer:

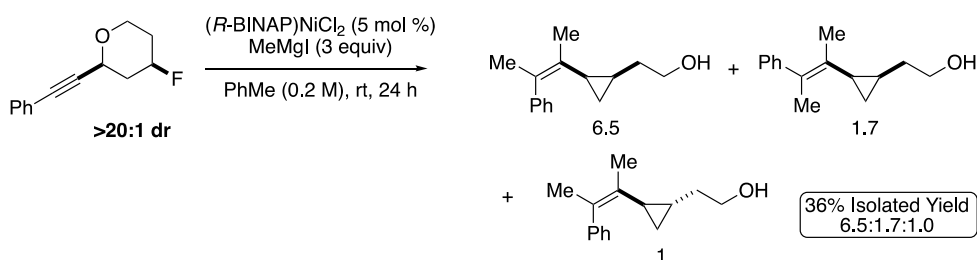


#### 4.5.10.3 NOE correlations for minor diastereomer:



**(3-phenylbut-2-en-2-yl)cyclopropyl)ethan-1-ol (4.93)** was prepared according to a modified version of Method A. The following amounts of reagents were used **4.92** (45 mg, 0.22 mmol, 1.0 equiv), *(R)*-BINAP)NiCl<sub>2</sub> (8.3 mg, 11 μmol, 5.0 mol %), MeMgI (0.26 mL, 0.66 mmol, 2.5 M in Et<sub>2</sub>O, 3.0 equiv), and PhMe (1.1 mL, 0.20 M). The residue was purified by column

chromatography (0–10% EtOAc/hexanes) to afford the title compound as a yellow oil (37 mg, 0.17 mmol, 78% yield, 3:2.5:1 mixture of diastereomers). The dr was based on the integration of resonances associated with Hg in the  $^1\text{H}$  NMR spectrum. The relative configuration of the major and minor diastereomer was assigned based on NOE analysis. The  $^1\text{H}$  NMR of the major diastereomer is given below.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.33–7.27 (m, 2H), 7.20 (d,  $J = 7.5$  Hz, 3H), 3.59 (t,  $J = 6.5$  Hz, 2H), 1.98 (s, 3H), 1.49 (s, 3H), 1.42 (dq,  $J = 13.2, 6.5$  Hz, 1H), 1.31–1.23 (m, 2H), 0.85 (dq,  $J = 13.2, 6.5, 5.9$  Hz, 1H), 0.66 (dt,  $J = 9.3, 5.1$  Hz, 1H), 0.26 (dt,  $J = 9.5, 5.0$  Hz, 1H).

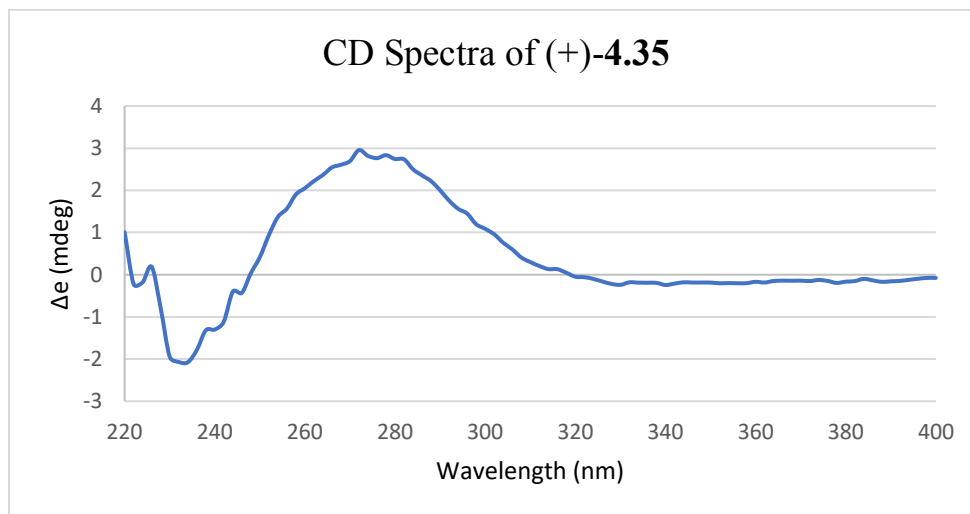


**(3-phenylbut-2-en-2-yl)cyclopropyl)ethan-1-ol (4.93)** was prepared according to a modified version of Method A. The following amounts of reagents were used **4.92** (46 mg, 0.23 mmol, 1.0 equiv), (*R*-BINAP) $\text{NiCl}_2$  (8.6 mg, 12  $\mu\text{mol}$ , 5.0 mol %),  $\text{MeMgI}$  (0.28 mL, 0.69 mmol, 2.5 M in  $\text{Et}_2\text{O}$ , 3.0 equiv), and  $\text{PhMe}$  (1.2 mL, 0.20 M). The residue was purified by column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a yellow oil (17 mg, 79  $\mu\text{mol}$ , 36% yield, 6.5:1.7:1 mixture of diastereomers). The dr was based on the integration of resonances associated with Hg in the  $^1\text{H}$  NMR spectrum. The relative configuration was assigned based on NOE analysis. See above for NOE correlations of the major diastereomer. The  $^1\text{H}$  NMR of the major diastereomer is given below.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.28 (t,  $J = 7.6$  Hz, 2H), 7.20 (d,  $J = 7.9$  Hz, 3H), 3.64 (t,  $J = 6.7$  Hz, 2H), 2.01 (s, 3H), 1.77 (s, 3H), 1.72 (ddt,  $J = 16.3, 11.0, 5.2$  Hz, 1H), 1.55–1.45 (m, 2H), 0.74 (dtd,  $J = 14.3, 9.2, 5.2$  Hz, 1H), 0.56 (td,  $J = 8.4, 5.0$  Hz, 1H),  $-0.03$  (q,  $J = 5.7$  Hz, 1H).

## 4.6 Circular Dichroism Experimental Data

Experimental data collected was collected in CH<sub>2</sub>Cl<sub>2</sub> (concentration = 7.48 x 10<sup>-4</sup> M).

**Figure 4.4** Experimental Circular Dichroism Spectrum of (+)-4.35



**Table 4.3** Experimental Values for CD Spectra of (+)-4.35

Wavelength (nm)	Δε (mdeg)
400	-0.0759497
398	-0.0751904
396	-0.0969728
394	-0.121212
392	-0.147048
390	-0.156384
388	-0.168732
386	-0.133429
384	-0.0984882
382	-0.151537
380	-0.164564
378	-0.194577
376	-0.148106
374	-0.124022
372	-0.151128
370	-0.140436
368	-0.14309
366	-0.141069
364	-0.149368

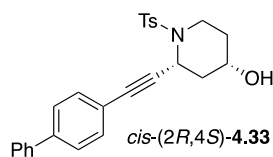
362	-0.184781
360	-0.168379
358	-0.200113
356	-0.202217
354	-0.198336
352	-0.202217
350	-0.186079
348	-0.184813
346	-0.185545
344	-0.179994
342	-0.211313
340	-0.243334
338	-0.192655
336	-0.190551
334	-0.187009
332	-0.179927
330	-0.239985
328	-0.217697
326	-0.160824
324	-0.0992894
322	-0.0588276
320	-0.0472567
318	0.047425
316	0.132352
314	0.133412
312	0.208498
310	0.30016
308	0.410641
306	0.602357
304	0.7589
302	0.958575
300	1.08812
298	1.19775
296	1.44968
294	1.56305
292	1.75573
290	2.00229
288	2.21936
286	2.35494

284	2.49864
282	2.73679
280	2.74398
278	2.83456
276	2.76474
274	2.81699
272	2.95532
270	2.69466
268	2.60762
266	2.54433
264	2.36611
262	2.22114
260	2.05217
258	1.90347
256	1.56613
254	1.36982
252	0.938061
250	0.41649
248	0.0341021
246	-0.430506
244	-0.401459
242	-1.11161
240	-1.30017
238	-1.31937
236	-1.78829
234	-2.076
232	-2.06933
230	-1.94643
228	-0.795606
226	0.186215
224	-0.178137
222	-0.224473
220	1.00859

## 4.7 Crystallographic Data

### 1) X-ray Data Collection, Structure Solution and Refinement for *cis*-(2*R*,4*S*)-4.33

CCDC: 2104034



A colorless crystal of approximate dimensions 0.122 x 0.367 x 0.392 mm was mounted on a glass fiber and transferred to a Bruker SMART APEX II diffractometer system. The APEX2<sup>107</sup> program package was used to determine the unit-cell parameters and for data collection (60 sec/frame scan time). The raw frame data was processed using SAINT<sup>108</sup> and SADABS<sup>109</sup> to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL<sup>110</sup> program package. The diffraction symmetry was *mmm* and the systematic absences were consistent with the orthorhombic space group *P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>* that was later determined to be correct.

The structure was solved by direct methods and refined on  $F^2$  by full-matrix least-squares techniques. The analytical scattering factors<sup>111</sup> for neutral atoms were used throughout the analysis.

Hydrogen atoms associated with C(26) were included using a riding model. The remaining hydrogen atoms were located from a difference-Fourier map and refined (*x*,*y*,*z* and  $U_{iso}$ ).

Least squares analysis yielded  $wR2 = 0.0933$  and  $Goof = 1.093$  for 369 variables refined against 5859 data (0.73 Å),  $R1 = 0.0381$  for those 5519 data with  $I > 2.0\sigma(I)$ . The absolute structure was assigned by refinement of the Flack parameter<sup>112</sup>.

Definitions:

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$$

<sup>107</sup> APEX2 Version 2014.11-0, Bruker AXS, Inc.; Madison, WI 2014.

<sup>108</sup> SAINT Version 8.34a, Bruker AXS, Inc.; Madison, WI 2013.

<sup>109</sup> Sheldrick, G. M. SADABS, Version 2014/5, Bruker AXS, Inc.; Madison, WI 2014.

<sup>110</sup> Sheldrick, G. M. SHELXTL, Version 2014/7, Bruker AXS, Inc.; Madison, WI 2014.

<sup>111</sup> *International Tables for Crystallography* **1992**, Vol. C., Dordrecht: Kluwer Academic Publishers.

<sup>112</sup> Parsons, S., Flack, H. D., Wagner, T. *Acta Cryst.* **2013**, *B69*, 249–259.

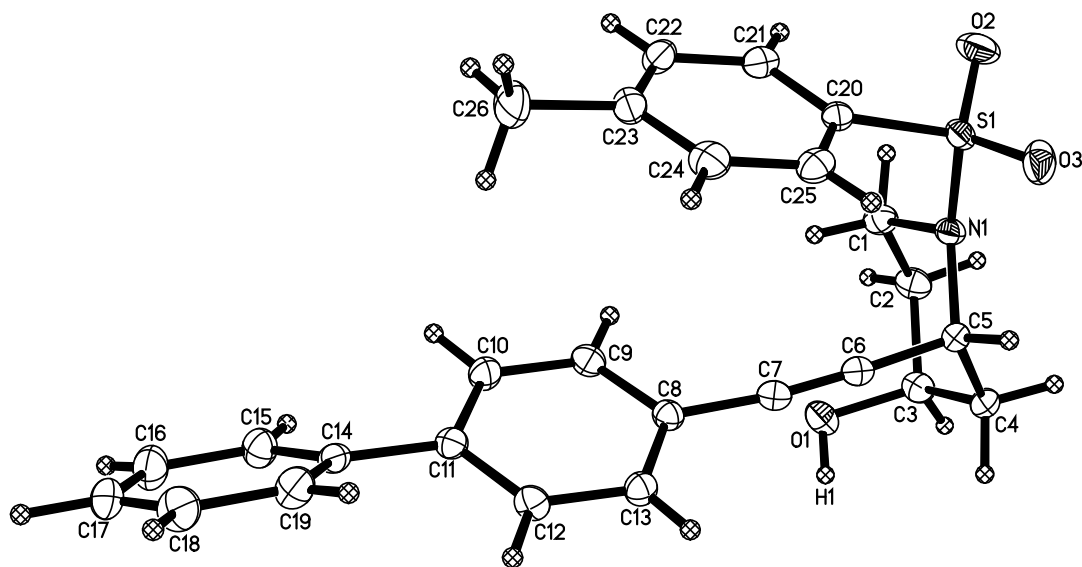


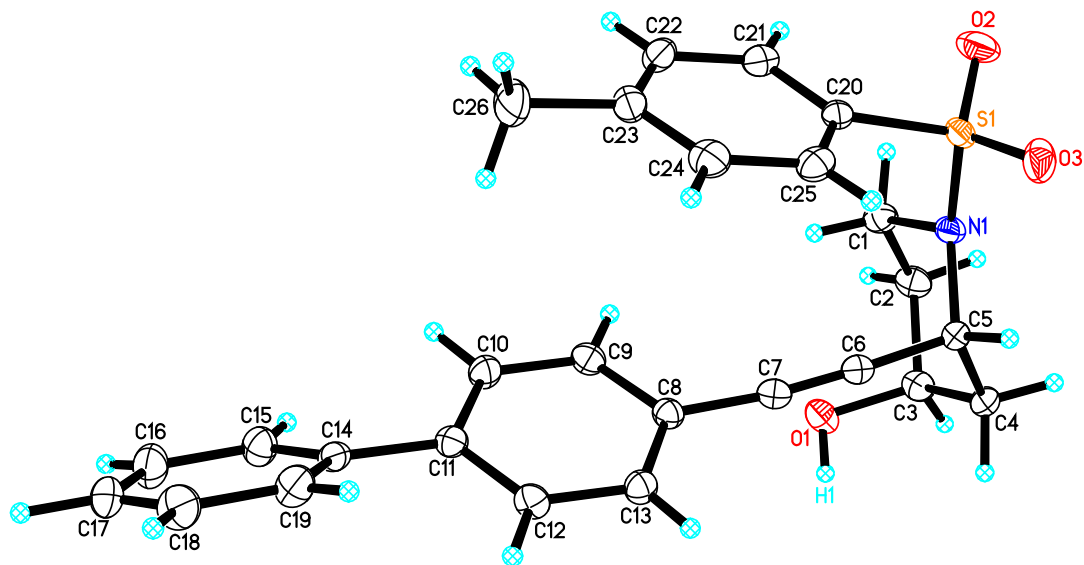
$$R1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}$$

Goof = S =  $[\sum [w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$  where n is the number of reflections and p is the total number of parameters refined.

The thermal ellipsoid plot is shown at the 50% probability level.

**Figure 4.5** X-Ray Crystallographic Structure of (+)-4.33





**Table 4.4** Crystal data and structure refinement for erj39.

Identification code	erj39 (Kirsten Hewitt)	
Empirical formula	$C_{26} H_{25} N O_3 S$	
Formula weight	431.53	
Temperature	133(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	$a = 7.9745(4)$ Å	$\alpha = 90^\circ$ .
	$b = 10.8981(5)$ Å	$\beta = 90^\circ$ .
	$c = 25.0685(13)$ Å	$\gamma = 90^\circ$ .
Volume	$2178.63(19)$ Å <sup>3</sup>	
Z	4	
Density (calculated)	1.316 Mg/m <sup>3</sup>	
Absorption coefficient	0.177 mm <sup>-1</sup>	
F(000)	912	
Crystal color	colorless	
Crystal size	0.392 x 0.367 x 0.122 mm <sup>3</sup>	
Theta range for data collection	1.625 to 29.129°	
Index ranges	$-10 \leq h \leq 10, -14 \leq k \leq 14, -34 \leq l \leq 34$	
Reflections collected	51864	

Independent reflections	5859 [R(int) = 0.0425]
Completeness to theta = 25.242°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.8622 and 0.7838
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	5859 / 0 / 369
Goodness-of-fit on F <sup>2</sup>	1.093
Final R indices [I>2sigma(I) = 5519 data]	R1 = 0.0381, wR2 = 0.0915
R indices (all data, 0.73 Å)	R1 = 0.0416, wR2 = 0.0933
Absolute structure parameter	0.020(16)
Largest diff. peak and hole	0.349 and -0.213 e.Å <sup>-3</sup>

**Table 4.5** Atomic coordinates (x 10<sup>4</sup>) and equivalent isotropic displacement parameters (Å<sup>2</sup>x 10<sup>3</sup>) for erj39.<sup>113</sup>

	x	y	z	U(eq)
S(1)	3362(1)	1493(1)	8849(1)	20(1)
O(1)	8990(2)	3531(2)	9010(1)	23(1)
O(2)	2026(2)	2237(2)	9057(1)	33(1)
O(3)	3392(2)	202(2)	8960(1)	29(1)
N(1)	5082(2)	2069(2)	9106(1)	17(1)
C(1)	5338(3)	3403(2)	9048(1)	21(1)
C(2)	6488(3)	3854(2)	9493(1)	23(1)
C(3)	8143(3)	3173(2)	9485(1)	20(1)
C(4)	7848(3)	1788(2)	9506(1)	19(1)
C(5)	6646(3)	1325(2)	9071(1)	16(1)
C(6)	7410(2)	1368(2)	8535(1)	17(1)
C(7)	7987(2)	1456(2)	8098(1)	17(1)
C(8)	8555(2)	1652(2)	7562(1)	16(1)
C(9)	8168(3)	2765(2)	7308(1)	20(1)
C(10)	8630(3)	2962(2)	6784(1)	19(1)
C(11)	9494(2)	2070(2)	6493(1)	17(1)

<sup>113</sup> U(eq) is defined as one third of the trace of the orthogonalized U<sup>ij</sup> tensor.

C(12)	9902(3)	969(2)	6752(1)	18(1)
C(13)	9451(3)	766(2)	7278(1)	18(1)
C(14)	9980(2)	2277(2)	5929(1)	18(1)
C(15)	10377(3)	3442(2)	5737(1)	23(1)
C(16)	10909(3)	3603(2)	5214(1)	28(1)
C(17)	11044(3)	2605(2)	4873(1)	28(1)
C(18)	10622(3)	1454(2)	5056(1)	29(1)
C(19)	10090(3)	1284(2)	5574(1)	24(1)
C(20)	3422(3)	1696(2)	8154(1)	18(1)
C(21)	2930(3)	2810(2)	7929(1)	22(1)
C(22)	3140(3)	2977(2)	7382(1)	25(1)
C(23)	3839(3)	2071(2)	7065(1)	25(1)
C(24)	4280(3)	960(2)	7298(1)	24(1)
C(25)	4080(3)	762(2)	7838(1)	21(1)
C(26)	4137(3)	2286(3)	6478(1)	38(1)

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**Table 4.6** Bond lengths [ $\text{\AA}$ ] and angles [ $^\circ$ ] for erj39.

S(1)-O(3)	1.4339(18)
S(1)-O(2)	1.4371(18)
S(1)-N(1)	1.6396(18)
S(1)-C(20)	1.758(2)
O(1)-C(3)	1.425(3)
O(1)-H(1)	0.84(3)
N(1)-C(1)	1.476(3)
N(1)-C(5)	1.491(3)
C(1)-C(2)	1.526(3)
C(1)-H(1A)	0.96(3)
C(1)-H(1B)	0.96(3)
C(2)-C(3)	1.515(3)
C(2)-H(2A)	0.97(3)
C(2)-H(2B)	1.01(3)
C(3)-C(4)	1.528(3)
C(3)-H(3A)	0.97(3)
C(4)-C(5)	1.537(3)

C(4)-H(4A)	0.99(3)
C(4)-H(4B)	0.98(3)
C(5)-C(6)	1.477(3)
C(5)-H(5A)	0.93(2)
C(6)-C(7)	1.192(3)
C(7)-C(8)	1.434(3)
C(8)-C(13)	1.397(3)
C(8)-C(9)	1.404(3)
C(9)-C(10)	1.382(3)
C(9)-H(9A)	0.96(3)
C(10)-C(11)	1.397(3)
C(10)-H(10A)	0.98(3)
C(11)-C(12)	1.404(3)
C(11)-C(14)	1.483(3)
C(12)-C(13)	1.383(3)
C(12)-H(12A)	0.92(3)
C(13)-H(13A)	0.88(3)
C(14)-C(15)	1.394(3)
C(14)-C(19)	1.403(3)
C(15)-C(16)	1.390(3)
C(15)-H(15A)	0.93(3)
C(16)-C(17)	1.387(4)
C(16)-H(16A)	0.92(3)
C(17)-C(18)	1.377(4)
C(17)-H(17A)	1.03(3)
C(18)-C(19)	1.380(3)
C(18)-H(18A)	0.83(3)
C(19)-H(19A)	0.92(3)
C(20)-C(25)	1.391(3)
C(20)-C(21)	1.395(3)
C(21)-C(22)	1.394(3)
C(21)-H(21A)	0.96(3)
C(22)-C(23)	1.385(4)
C(22)-H(22A)	0.93(3)
C(23)-C(24)	1.390(3)
C(23)-C(26)	1.508(3)

C(24)-C(25)	1.381(3)
C(24)-H(24A)	0.95(3)
C(25)-H(25A)	0.93(3)
C(26)-H(26A)	0.9800
C(26)-H(26B)	0.9800
C(26)-H(26C)	0.9800
O(3)-S(1)-O(2)	119.70(12)
O(3)-S(1)-N(1)	106.61(10)
O(2)-S(1)-N(1)	105.18(10)
O(3)-S(1)-C(20)	108.34(10)
O(2)-S(1)-C(20)	107.99(10)
N(1)-S(1)-C(20)	108.58(9)
C(3)-O(1)-H(1)	105.9(18)
C(1)-N(1)-C(5)	114.47(16)
C(1)-N(1)-S(1)	117.02(14)
C(5)-N(1)-S(1)	117.95(14)
N(1)-C(1)-C(2)	109.15(17)
N(1)-C(1)-H(1A)	106.6(15)
C(2)-C(1)-H(1A)	108.1(16)
N(1)-C(1)-H(1B)	108.8(18)
C(2)-C(1)-H(1B)	112.4(18)
H(1A)-C(1)-H(1B)	112(2)
C(3)-C(2)-C(1)	110.84(17)
C(3)-C(2)-H(2A)	108.9(19)
C(1)-C(2)-H(2A)	108.4(17)
C(3)-C(2)-H(2B)	111.0(14)
C(1)-C(2)-H(2B)	108.5(14)
H(2A)-C(2)-H(2B)	109(2)
O(1)-C(3)-C(2)	106.84(17)
O(1)-C(3)-C(4)	111.90(17)
C(2)-C(3)-C(4)	110.47(18)
O(1)-C(3)-H(3A)	108.4(15)
C(2)-C(3)-H(3A)	112.5(15)
C(4)-C(3)-H(3A)	106.8(15)
C(3)-C(4)-C(5)	113.30(17)

C(3)-C(4)-H(4A)	110.8(17)
C(5)-C(4)-H(4A)	104.9(17)
C(3)-C(4)-H(4B)	109.6(16)
C(5)-C(4)-H(4B)	109.6(16)
H(4A)-C(4)-H(4B)	108(2)
C(6)-C(5)-N(1)	112.39(16)
C(6)-C(5)-C(4)	112.22(17)
N(1)-C(5)-C(4)	107.57(16)
C(6)-C(5)-H(5A)	107.9(14)
N(1)-C(5)-H(5A)	107.6(15)
C(4)-C(5)-H(5A)	109.1(15)
C(7)-C(6)-C(5)	176.8(2)
C(6)-C(7)-C(8)	174.3(2)
C(13)-C(8)-C(9)	118.54(18)
C(13)-C(8)-C(7)	122.48(18)
C(9)-C(8)-C(7)	118.97(18)
C(10)-C(9)-C(8)	120.50(18)
C(10)-C(9)-H(9A)	122.9(17)
C(8)-C(9)-H(9A)	116.6(17)
C(9)-C(10)-C(11)	121.30(19)
C(9)-C(10)-H(10A)	118.5(16)
C(11)-C(10)-H(10A)	120.2(16)
C(10)-C(11)-C(12)	117.89(19)
C(10)-C(11)-C(14)	121.39(19)
C(12)-C(11)-C(14)	120.72(18)
C(13)-C(12)-C(11)	121.16(19)
C(13)-C(12)-H(12A)	118.8(17)
C(11)-C(12)-H(12A)	120.0(17)
C(12)-C(13)-C(8)	120.59(19)
C(12)-C(13)-H(13A)	116.9(17)
C(8)-C(13)-H(13A)	122.5(18)
C(15)-C(14)-C(19)	117.95(19)
C(15)-C(14)-C(11)	121.79(18)
C(19)-C(14)-C(11)	120.24(19)
C(16)-C(15)-C(14)	120.7(2)
C(16)-C(15)-H(15A)	123.4(18)

C(14)-C(15)-H(15A)	115.7(17)
C(17)-C(16)-C(15)	120.3(2)
C(17)-C(16)-H(16A)	119.2(17)
C(15)-C(16)-H(16A)	120.4(17)
C(18)-C(17)-C(16)	119.4(2)
C(18)-C(17)-H(17A)	124.4(17)
C(16)-C(17)-H(17A)	116.1(17)
C(17)-C(18)-C(19)	120.7(2)
C(17)-C(18)-H(18A)	120(2)
C(19)-C(18)-H(18A)	119(2)
C(18)-C(19)-C(14)	120.9(2)
C(18)-C(19)-H(19A)	122.5(18)
C(14)-C(19)-H(19A)	116.6(18)
C(25)-C(20)-C(21)	120.87(19)
C(25)-C(20)-S(1)	118.79(16)
C(21)-C(20)-S(1)	120.18(17)
C(22)-C(21)-C(20)	118.6(2)
C(22)-C(21)-H(21A)	123.4(16)
C(20)-C(21)-H(21A)	118.1(16)
C(23)-C(22)-C(21)	121.3(2)
C(23)-C(22)-H(22A)	120.9(18)
C(21)-C(22)-H(22A)	117.7(18)
C(22)-C(23)-C(24)	118.8(2)
C(22)-C(23)-C(26)	120.8(2)
C(24)-C(23)-C(26)	120.4(2)
C(25)-C(24)-C(23)	121.3(2)
C(25)-C(24)-H(24A)	118.7(18)
C(23)-C(24)-H(24A)	120.0(18)
C(24)-C(25)-C(20)	119.1(2)
C(24)-C(25)-H(25A)	119.8(16)
C(20)-C(25)-H(25A)	121.0(17)
C(23)-C(26)-H(26A)	109.5
C(23)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	109.5
C(23)-C(26)-H(26C)	109.5
H(26A)-C(26)-H(26C)	109.5



**Table 4.7** Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for erj39.<sup>114</sup>

	U <sup>11</sup>	U <sup>22</sup>	U <sup>33</sup>	U <sup>23</sup>	U <sup>13</sup>	U <sup>12</sup>
S(1)	14(1)	31(1)	16(1)	4(1)	0(1)	-4(1)
O(1)	22(1)	21(1)	27(1)	-1(1)	7(1)	-2(1)
O(2)	15(1)	59(1)	24(1)	-2(1)	3(1)	2(1)
O(3)	28(1)	36(1)	24(1)	10(1)	-7(1)	-14(1)
N(1)	13(1)	24(1)	16(1)	0(1)	0(1)	0(1)
C(1)	19(1)	21(1)	22(1)	-1(1)	0(1)	4(1)
C(2)	21(1)	23(1)	24(1)	-6(1)	2(1)	1(1)
C(3)	18(1)	25(1)	18(1)	-4(1)	0(1)	-1(1)
C(4)	17(1)	24(1)	17(1)	3(1)	-3(1)	0(1)
C(5)	15(1)	17(1)	16(1)	2(1)	0(1)	1(1)
C(6)	15(1)	17(1)	19(1)	0(1)	-1(1)	1(1)
C(7)	15(1)	17(1)	19(1)	-2(1)	-1(1)	0(1)
C(8)	15(1)	19(1)	16(1)	-1(1)	0(1)	-1(1)
C(9)	19(1)	20(1)	19(1)	-3(1)	3(1)	4(1)
C(10)	21(1)	17(1)	20(1)	1(1)	1(1)	4(1)
C(11)	16(1)	19(1)	15(1)	-2(1)	-1(1)	0(1)
C(12)	20(1)	16(1)	18(1)	-4(1)	2(1)	2(1)
C(13)	20(1)	16(1)	19(1)	1(1)	1(1)	1(1)
C(14)	16(1)	21(1)	15(1)	-1(1)	-1(1)	5(1)
C(15)	28(1)	22(1)	19(1)	-2(1)	1(1)	2(1)
C(16)	35(1)	28(1)	23(1)	5(1)	3(1)	2(1)
C(17)	29(1)	42(1)	15(1)	2(1)	0(1)	7(1)
C(18)	34(1)	33(1)	19(1)	-10(1)	-2(1)	9(1)
C(19)	30(1)	21(1)	20(1)	-3(1)	-2(1)	5(1)
C(20)	13(1)	26(1)	17(1)	3(1)	-3(1)	-4(1)

<sup>114</sup> The anisotropic displacement factor exponent takes the form:  $-2\pi^2 [h^2 a^{*2}U^{11} + \dots + 2 h k a^* b^* U^{12}]$

C(21)	18(1)	26(1)	23(1)	1(1)	-2(1)	2(1)
C(22)	21(1)	29(1)	26(1)	10(1)	-5(1)	-2(1)
C(23)	18(1)	41(1)	16(1)	3(1)	-5(1)	-11(1)
C(24)	21(1)	32(1)	21(1)	-5(1)	-3(1)	-2(1)
C(25)	20(1)	21(1)	22(1)	-1(1)	-5(1)	-2(1)
C(26)	32(1)	63(2)	18(1)	6(1)	-4(1)	-11(1)

**Table 4.8** Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for erj39.

	x	y	z	U(eq)
H(26A)	3820	3129	6387	57
H(26B)	3459	1710	6270	57
H(26C)	5327	2159	6397	57
H(1)	9840(40)	3080(20)	8989(11)	22(7)
H(1A)	4260(30)	3780(20)	9094(10)	20(6)
H(1B)	5790(40)	3560(30)	8698(12)	31(7)
H(2A)	6700(40)	4720(30)	9439(11)	29(7)
H(2B)	5890(30)	3740(20)	9845(10)	16(6)
H(3A)	8850(30)	3380(20)	9787(10)	17(6)
H(4A)	7310(40)	1550(30)	9847(12)	31(7)
H(4B)	8930(40)	1360(30)	9477(11)	27(7)
H(5A)	6360(30)	510(20)	9144(9)	11(5)
H(9A)	7560(40)	3350(20)	7513(11)	27(7)
H(10A)	8310(40)	3740(20)	6613(11)	25(7)
H(12A)	10530(40)	380(20)	6577(11)	24(7)
H(13A)	9750(30)	60(20)	7419(10)	20(6)
H(15A)	10180(40)	4090(30)	5970(11)	28(7)
H(16A)	11240(40)	4360(30)	5096(11)	27(7)
H(17A)	11470(40)	2790(30)	4493(12)	39(8)
H(18A)	10700(40)	850(30)	4855(14)	45(9)
H(19A)	9790(40)	530(30)	5705(11)	28(7)
H(21A)	2470(30)	3430(20)	8161(11)	20(6)

H(22A)	2840(40)	3740(30)	7238(12)	32(8)
H(24A)	4770(40)	330(30)	7089(12)	39(8)
H(25A)	4360(30)	10(20)	7985(10)	22(6)

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**Table 4.9** Torsion angles [°] for erj39.

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O(3)-S(1)-N(1)-C(1)	-179.52(15)
O(2)-S(1)-N(1)-C(1)	-51.45(17)
C(20)-S(1)-N(1)-C(1)	63.95(17)
O(3)-S(1)-N(1)-C(5)	37.64(17)
O(2)-S(1)-N(1)-C(5)	165.71(15)
C(20)-S(1)-N(1)-C(5)	-78.89(16)
C(5)-N(1)-C(1)-C(2)	-60.7(2)
S(1)-N(1)-C(1)-C(2)	155.18(14)
N(1)-C(1)-C(2)-C(3)	57.2(2)
C(1)-C(2)-C(3)-O(1)	67.5(2)
C(1)-C(2)-C(3)-C(4)	-54.4(2)
O(1)-C(3)-C(4)-C(5)	-65.4(2)
C(2)-C(3)-C(4)-C(5)	53.5(2)
C(1)-N(1)-C(5)-C(6)	-66.4(2)
S(1)-N(1)-C(5)-C(6)	77.35(19)
C(1)-N(1)-C(5)-C(4)	57.6(2)
S(1)-N(1)-C(5)-C(4)	-158.64(14)
C(3)-C(4)-C(5)-C(6)	71.0(2)
C(3)-C(4)-C(5)-N(1)	-53.1(2)
C(13)-C(8)-C(9)-C(10)	1.6(3)
C(7)-C(8)-C(9)-C(10)	-177.08(19)
C(8)-C(9)-C(10)-C(11)	-0.2(3)
C(9)-C(10)-C(11)-C(12)	-0.9(3)
C(9)-C(10)-C(11)-C(14)	179.6(2)
C(10)-C(11)-C(12)-C(13)	0.6(3)
C(14)-C(11)-C(12)-C(13)	-179.85(19)
C(11)-C(12)-C(13)-C(8)	0.8(3)
C(9)-C(8)-C(13)-C(12)	-1.9(3)
C(7)-C(8)-C(13)-C(12)	176.74(19)
C(10)-C(11)-C(14)-C(15)	31.9(3)
C(12)-C(11)-C(14)-C(15)	-147.6(2)
C(10)-C(11)-C(14)-C(19)	-149.6(2)
C(12)-C(11)-C(14)-C(19)	30.9(3)
C(19)-C(14)-C(15)-C(16)	-1.8(3)

C(11)-C(14)-C(15)-C(16)	176.8(2)
C(14)-C(15)-C(16)-C(17)	0.4(4)
C(15)-C(16)-C(17)-C(18)	0.9(4)
C(16)-C(17)-C(18)-C(19)	-0.8(4)
C(17)-C(18)-C(19)-C(14)	-0.6(4)
C(15)-C(14)-C(19)-C(18)	1.8(3)
C(11)-C(14)-C(19)-C(18)	-176.7(2)
O(3)-S(1)-C(20)-C(25)	-23.0(2)
O(2)-S(1)-C(20)-C(25)	-154.06(17)
N(1)-S(1)-C(20)-C(25)	92.38(18)
O(3)-S(1)-C(20)-C(21)	161.57(17)
O(2)-S(1)-C(20)-C(21)	30.5(2)
N(1)-S(1)-C(20)-C(21)	-83.02(19)
C(25)-C(20)-C(21)-C(22)	-1.1(3)
S(1)-C(20)-C(21)-C(22)	174.15(17)
C(20)-C(21)-C(22)-C(23)	-0.8(3)
C(21)-C(22)-C(23)-C(24)	2.3(3)
C(21)-C(22)-C(23)-C(26)	-177.0(2)
C(22)-C(23)-C(24)-C(25)	-1.9(3)
C(26)-C(23)-C(24)-C(25)	177.4(2)
C(23)-C(24)-C(25)-C(20)	0.0(3)
C(21)-C(20)-C(25)-C(24)	1.5(3)
S(1)-C(20)-C(25)-C(24)	-173.84(16)

**Table 4.10** Hydrogen bonds for erj39 [ $\text{\AA}$  and  $^\circ$ ].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1)...O(2)#1	0.84(3)	1.98(3)	2.804(2)	169(3)

Symmetry transformations used to generate equivalent atoms:

#1  $x+1, y, z$

## Synthesis of Vicinal Carbocycles by Intramolecular Nickel-Catalyzed Conjunctive Cross-Electrophile Coupling Reaction

### 5.1 Introduction

Nickel-catalyzed conjunctive cross-electrophile coupling (XEC) reactions allow for the rapid and efficient synthesis of highly complex scaffolds, beginning with two electrophilic partners and an olefin (Scheme 5.1)<sup>1,2,3</sup> These reactions pose major challenges in achieving high levels of stereo-, regio-, and chemoselectivity, particularly when performing three-component reactions (Scheme 5.1a). Building upon the mechanistic insights from XEC reactions,<sup>4,5</sup> several strategies have been established to achieve selectivity, including mechanistic differentiation of the

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<sup>1</sup> Portions of this chapter have been submitted for publication.

<sup>2</sup>For reviews of nickel catalysis, see: (a) Modern Organonickel Chemistry; Tamaru, Y., Ed.; Wiley-VCH: Weinheim; **2005**. (b) Tasker, S. Z.; Standley, E. A.; Jamison, T. F. *Nature* **2014**, *509*, 299–309. (c) Nickel Catalysis in Organic Synthesis: Methods and Reactions; Ogoshi, S., Ed.; Wiley, **2020**.

<sup>3</sup> For reviews of conjunctive XC and XEC reactions, see: (a) Derosa, J.; Tran, V. T.; van der Puyl, V. A.; Engle, K. M. *Aldrichim. Acta* **2018**, *51*, 21–32. (b) Zhang, J.-S.; Liu, L.; Chen, T.; Han, L.-B. *Chem. Asian J.* **2018**, *13*, 2277–2291. (c) Qi, X.; Diao, T. *ACS Catal.* **2020**, *10*, 8542–8556. (d) Derosa, J.; Apolinar, O.; Kang, T.; Tran, V. T.; Engle, K. M. *Chem. Sci.* **2020**, *11*, 4287–4296. (e) Badir, S. O.; Molander, G. A. *Chem.* **2020**, *6*, 1327–1339. (f) Tu, H.-Y.; Zhu, S.; Qing, F.-L.; Chu, L. *Synthesis* **2020**, *52*, 1346–1356. (g) Ping, Y.; Kong, W. *Synthesis* **2020**, *52*, 979–992. (h) Wickham, L. M.; Giri, R. *Acc. Chem. Res.* **2021**, *54*, 3415–3437. (i) Gao, P.; Niu, Y.-J.; Yang, F.; Guo, L.-N.; Duan, X.-H. *Chem. Commun.* **2022**, *58*, 730–746.

<sup>4</sup> For reviews of XEC reactions, see: (a) Knappke, C. E. I.; Grupe, S.; Gärtner, D.; Corpet, M.; Gosmini, C.; Jacobi von Wangelin, A. *Chem. Eur. J.* **2014**, *20*, 6828–6842. (b) Everson, D. A.; Weix D. J. *J. Org. Chem.* **2014**, *79*, 4793–4798. (c) Gu, J.; Wang, X.; Xue, W.; Gong, H. *Org. Chem. Front.* **2015**, *2*, 1411–1421. (c) Goldfogel, M; Huang, L. Weix, D. J. Cross-Electrophile Coupling. In *Nickel Catalysis in Organic Synthesis*; Ogoshi, S., Ed.; Wiley, **2020**; 183–222. (f) Richmond, E.; Moran, J. *Synthesis* **2019**, *50*, 499–513. (h) Campeau, L.-C.; Hazari, N. *Organometallics* **2019**, *38*, 3–35. (i) Hewitt, K. A.; Lin, P. C.; Raffman, E. T. A.; Jarvo, E. R. *Comprehensive Organometallic Chemistry IV*. 2021. DOI: 10.1016/B978-0-12-820206-7.00092-5.

<sup>5</sup> For a discussion of the mechanism of the conjunctive XEC reaction, see: (a) Lin, Q.; Diao, T. *J. Am. Chem. Soc.* **2019**, *141*, 17937–17948. (b) Diccianni, J.; Lin, Q.; Diao, T. *Acc. Chem. Res.* **2020**, *53*, 906–919.

electrophiles or employing an excess of one reagent.<sup>6,7,8,9</sup> In addition, the use of directing groups can also favor regio- and chemoselective reactions and allow for the use of unactivated conjunctive reagents.<sup>10</sup> An additional strategy, tethering one electrophile and alkene together to afford a two-component reaction, also significantly addresses the selectivity challenges and at the same time constructs cyclic fragments (Scheme 5.1b).<sup>11,12,13</sup> We envisioned a fully intramolecular nickel-catalyzed conjunctive XEC reaction to provide vicinal ring systems (Scheme 5.1c).<sup>14</sup> This

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<sup>6</sup> For representative examples of conjunctive XEC reactions in which an activated alkene is used, see: (a) García-Domínguez, A.; Li, Z.; Nevado, C. *J. Am. Chem. Soc.* **2017**, *139*, 6835–6838. (b) Wang, L.; Wang, C. *Org. Lett.* **2020**, *22*, 8829–8835. (c) Zhao, Q.-W.; Yang, Z.-F.; Fu, X.-P.; Zhang, X. *Synlett* **2021**, *32*, 1565–1569. (d) Wang, X.-X.; Lu, X.; He, S.-J.; Fu, Y. *Chem. Sci.* **2020**, *11*, 7950–7956. (e) Feng, X.; Guo, L.; Zhu, S.; Chu, L. *Synlett* **2021**, *32*, 1519–1524.

<sup>7</sup> For a representative example of conjunctive XEC reactions with terminal alkenes see: Shu, W.; García-Domínguez, A.; Quirós, M. T.; Mondal, R.; Cárdenas, D. J.; Nevado, C. *J. Am. Chem. Soc.* **2019**, *141*, 13812–13821.

<sup>8</sup> For lead examples of enantioselective conjunctive XEC reactions, see: (a) Anthony, D.; Lin, Q.; Baudet, J.; Diao, T. *Angew. Chem., Int. Ed.* **2019**, *58*, 3198–3202. (b) Wei, X.; Shu, W.; García-Domínguez, A.; Merino, E.; Nevado, C. *J. Am. Chem. Soc.* **2020**, *142*, 13515–13522. (c) Tu, H.-Y.; Wang, F.; Huo, L.; Li, Y.; Zhu, S.; Zhao, X.; Li, H.; Qing, F.-L.; Chu, L. *J. Am. Chem. Soc.* **2020**, *142*, 9604–9611.

<sup>9</sup> For lead examples of conjunctive XEC reactions that employ dual photoredox and nickel-catalysis, see: (a) Guo, L.; Tu, H.-Y.; Zhu, S.; Chu, L.; *Org. Lett.* **2019**, *21*, 4771–4776. (b) García-Domínguez, A.; Mondal, R.; Nevado, C.; *Angew. Chem. Int. Ed.* **2019**, *58*, 12286–12290. (c) Qian, P.; Guan, H.; Wang, Y.-E.; Lu, Q.; Zhang, F.; Xiong, D.; Walsh, P. J.; Mao, J. *Nat. Comm.* **2021**, *12*, 6613.

<sup>10</sup> For lead examples of conjunctive XEC reactions with directing groups, see: (a) Yang, T.; Chen, X.; Rao, W.; Koh, M. J. *Chem* **2020**, *6*, 738–751. (b) Yang, T.; Jiang, Y.; Luo, Y.; Lim, J. J. H.; Lan, Y.; Koh, M. J. *J. Am. Chem. Soc.* **2020**, *142*, 21410–21419. (c) Pan, R.; Shi, C.; Zhang, D.; Tian, Y.; Guo, S.; Yao, H.; Lin, A. *Org. Lett.* **2019**, *21*, 8915–8920. (d) Zhao, X.; Tu, H.-Y.; Guo, L.; Zhu, S.; Qing, F.-L.; Chu, L. *Nat. Commun.* **2018**, *9*, 3488.

<sup>11</sup> For lead examples of two component conjunctive XEC reactions where the C(sp<sup>2</sup>) electrophile is tethered to the alkene, see: (a) Yan, C.-S.; Peng, Y.; Xu, X.-B.; Wang, Y.-W. *Chem. Eur. J.* **2012**, *18*, 6039–6048. (b) Wang, K.; Ding, Z.; Zhou, Z.; Kong, W. *J. Am. Chem. Soc.* **2018**, *140*, 12364–12368. (c) Jin, Y.; Wang, C. *Angew. Chem., Int. Ed.* **2019**, *58*, 6722–6726. (d) Tian, Z.-X.; Qiao, J.-B.; Xu, G.-L.; Pang, X.; Qi, L.; Ma, W.-Y.; Zhao, Z.-Z.; Duan, J.; Du, Y.-F.; Su, P.; Liu, X.-Y.; Shu, X.-Z. *J. Am. Chem. Soc.* **2019**, *141*, 7637–7643. (e) Ma, T.; Chen, Y.; Li, Y.; Ping, Y.; Kong, W. *ACS Catal.* **2019**, *9*, 9127–9133. (f) Fan, P.; Lan, Y.; Zhang, C.; Wang, C. *J. Am. Chem. Soc.* **2020**, *142*, 2180–2186. (g) Yang, J.; Yang, L.; Gu, J.; Shuai, L.; Wang, H.; Ouyang, Q.; Li, Y.-L.; Liu, H.; Gong, L. *Org. Lett.* **2022**, *24*, 2376–2380.

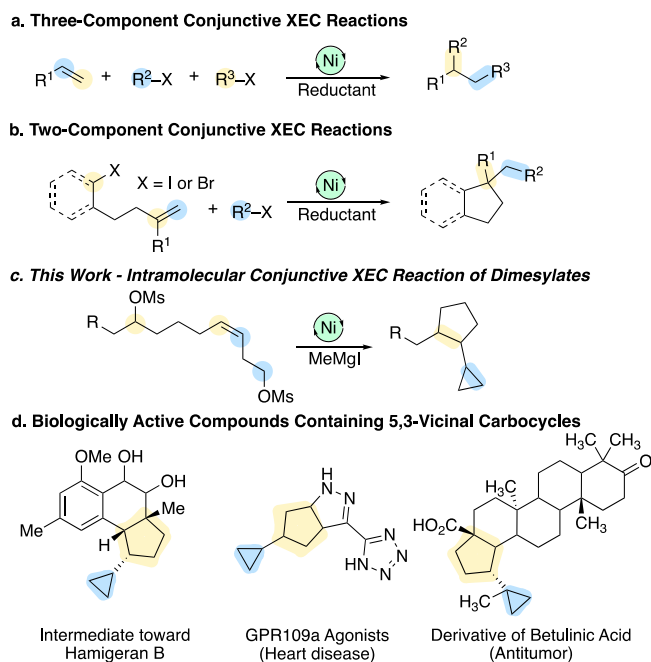
<sup>12</sup> For lead examples of two component conjunctive XEC reactions where the C(sp<sup>3</sup>) electrophile is tethered to the alkene, see: (a) ref. 11 a. (b) Yin, H.; Zhao, C.; You, H.; Lin, K.; Gong, H. *Chem. Commun.* **2012**, *48*, 7034–7036. (c) Peng, Y.; Xu, X.-B.; Xiao, J.; Wang, Y.-W. *Chem. Commun.* **2014**, *50*, 472–474. (d) Kuang, Y.; Wang, X.; Anthony, D.; Diao, T. *Chem. Commun.* **2018**, *54*, 2558–2561. (f) Weires, N. A.; Slutskyy, Y.; Overman, L. E. *Angew. Chem. Int. Ed.* **2019**, *58*, 8561–8565.

<sup>13</sup> For lead examples of two component conjunctive XEC reactions where the two electrophiles are tethered together, see: Wang, H.; Huang, H.; Gong, C.; Diao, Y.; Chen, J.; Wu, S.-H.; Wang, L. *Org. Lett.* **2022**, *24*, 328–333.

<sup>14</sup> For examples of fully intramolecular conjunctive XEC reactions, see: (a) ref. 11 a. (b) Xiao, J.; Wang, Y.-W.; Peng, Y. *Synthesis* **2017**, *49*, 3576–3581. (c) Peng, Y.; Xiao, J.; Xu, X.-B.; Duan, S.-M.; Ren, L.; Shao, Y.-L.; Wang, Y.-W. *Org. Lett.* **2016**, *18*, 5170–5173. (d) Xiao, J.; Cong, X.-W.; Yang, G.-Z.; Wang, Y.-W.; Peng, Y. *Org. Lett.* **2018**, *20*, 1651–1654. (e) Xiao, J.; Wang, Y.-W.; Qiu, Z.-P.; Peng, Y. *Synlett* **2021**, *32*, 1662–1664.

manifold engages two unactivated electrophiles and an internal olefin, and generates two carbocycles in a single step.

### Scheme 5.1 Previous Work in Conjunctive XEC Reactions and Medicinally Relevant Vicinal Carbocycles



Vicinal 3,5-carbocyclic motifs are present in a number of biologically active compounds and natural products.<sup>15,16,17,18</sup> In addition, cyclopropanes themselves a common moieties in

<sup>15</sup> For lead reviews on cascade reactions in total synthesis, see: (a) Nicolaou, K. C.; Edmonds, D. J.; Bulger, P. G. *Angew. Chem. Int. Ed.* **2006**, *45*, 7134–7186. (b) Nicolaou, K. C.; Chen, J. S. *Chem. Soc. Rev.* **2009**, *38*, 2993–3009. (c) Sebren, L. J.; Devery, J. J.; Stephenson, C. R. J. *ACS Catal.* **2014**, *4*, 703–716. (d) Yu, C.; Huang, H.; Sheng, C.; Wang, W. *Designing Efficient Cascade Reactions in Drug Discovery*. In *Green Chemistry in Drug Discovery*; Richardson, P. F., Ed.; **2022**; 213–235.

<sup>16</sup> (a) For the synthesis of the intermediate to Hamigeran B, see: Taber, D. F.; Tan, W. *J. Org. Chem.* **2008**, *73*, 7560–7564. (b) For activity of Hamigeran B, see: Wellington, K. D.; Cambie, R. C.; Rutledge, P. S.; Bergquist, P. C. *J. Nat. Prod.* **2000**, *63*, 79–85.

<sup>17</sup> For the synthesis and biological activity of the GPR109a agonist, see: Imbriglio, J. E.; Chang, S.; Liang, R.; Raghavan, S.; Schmidt, D.; Smenton, A.; Tria, S.; Schrader, T. O.; Jung, J.-K.; Esser, C.; Taggart, A. K. P.; Cheng, K.; Carballo-Jane, E.; Waters, M. G.; Tata, J. R.; Colletti, S. L. *Bioorg. Med. Chem. Lett.* **2009**, *19*, 2121–2124.

<sup>18</sup> For the synthesis and biological activity of the derivative of betulinic Acid, see: Symon, A. V.; Veselova, N. N.; Kaplun, A. P.; Vlasenkova, N. K.; Fedorova, G. A.; Lyutik, A. I.; Gerasimova, G. K.; Shvets, V. I. *Rus. J. Bioorg. Chem.* **2005**, *31*, 286–291.



medicinal chemistry (Scheme 5.1d).<sup>19</sup> We sought to prepare vicinal 3,5-carbocycles by a nickel-catalyzed conjunctive XEC reaction, where a single cascade reaction would construct both carbocyclic moieties. In this manuscript, we report an intramolecular nickel-catalyzed conjunctive XEC reaction (Scheme 5.1c). The mechanistic framework of this reaction builds on our laboratory's development of intramolecular XEC reactions of 1,3-dimesylates.<sup>20</sup> In addition, it compliments traditional radical reactions that initiate at alkyl halides and cascade forward to forge multiple ring systems.<sup>21,22</sup> We provide preliminary mechanistic experiments to demonstrate that this reaction likely involves radical intermediates.

## 5.2 Results and Discussion

### 5.2.1 Optimization and Scope of the Conjunctive XEC Reaction

To begin, we designed a model substrate, dimesylate **5.1**, that contained two alkyl mesylates and an internal olefin. Based on previously developed cross-coupling and XEC reactions in our laboratory, we hypothesized that the secondary mesylate would be activated by the nickel catalyst and cascade forward through a 5-exo-trig cyclization.<sup>20,23,24</sup> The proposed reaction would terminate by 3-exo-tet cyclization to afford the desired vicinal carbocycle **5.2**. First, we examined

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<sup>19</sup> For the biological activity of cyclopropanes, see: (a) Talele, T. *J. Med. Chem.* **2016**, *59*, 8712–8756. (b) Salaün, J. Cyclopropane Derivatives and their Diverse Biological Profile. In *Small Ring Compounds in Organic Synthesis VI*. A. de Meijere, Ed. **2000**. 1–67.

<sup>20</sup> Sanford, A. B.; Thane, T. A.; McGinnis, T. M.; Chen, P.-P.; Hong, X.; Jarvo, E. R. *J. Am. Chem. Soc.* **2020**, *142*, 5017–5023.

<sup>21</sup> For lead reviews on domino or cascade reactions, see: (a) Ikeda, S.-I. *Acc. Chem. Res.* **2000**, *33*, 511–519. (c) Tietze, L. F. *Domino Reactions: Concepts for Efficient Organic Synthesis*. Wiley-VCH: Weinheim, **2014**. (d) Malacria, M. *Chem. Rev.* **1996**, *96*, 289–306. (e) Liu, Z.-Q. Radical Cascade Reactions. In *Free Radicals: Fundamentals and Applications in Organic Synthesis 2*; Georg Thieme Verlag KG: Stuttgart, **2021**. (f) Jahn, U. *Top. Curr. Chem.* **2011**, *323–451*. (g) Zhang, G.; Liu, Y.; Zhao, J.; Li, Y.; Zhang, Q. *Sci. China Chem.* **2019**, *62*, 1476–1491. (h) Romero, K. J.; Galliher, M. S.; Pratt, D. A.; Stephenson, C. R. J. *Chem. Soc. Rev.* **2018**, *47*, 7851–7866. (i) For a review on polyene cyclizations in the synthesis of natural products, see: Yoder, R. A.; Johnston, J. N. *Chem. Rev.* **2005**, *105*, 4730–4756. (j) For a review on “zipper”-type cascade reactions, see: Negishi, E.-I. *Pure & Appl. Chem.* **1992**, *64*, 323–334.

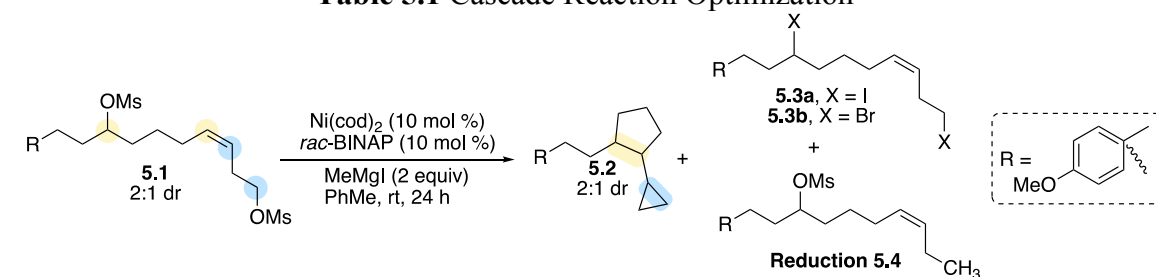
<sup>22</sup> For reviews, see: (a) Plesniak, M. P.; Huang, H.-M.; Procter, D. J. *Nat. Rev.* **2017**, *1*, 0077. (b) Huang, H.-M.; Garduño-Castro, M. H.; Morrill, C.; Procter, D. J. *Chem. Soc. Rev.* **2019**, *48*, 4626–4638.

<sup>23</sup> Harris, M. R.; Konev, M. O.; Jarvo, E. R. *J. Am. Chem. Soc.* **2014**, *136*, 7825–7828.

<sup>24</sup> Dimesylate **5.1** was prepared in five steps from commercially available aryl iodides. See Experimental Section **5.4** for more information.

the previously developed conditions for the synthesis of cyclopropanes from 1,3-dimesylates, employing Ni(cod)<sub>2</sub>, racemic BINAP and methylmagnesium iodide. We were delighted to observe the desired product in 85% yield (Table 5.1, entry 1). Next, to confirm that a *rac*-BINAP ligated nickel-catalyst was responsible for the conjunctive XEC reaction, we performed the reaction without ligand and a decreased yield of 29% was observed (entry 2).

**Table 5.1** Cascade Reaction Optimization



Entry	Deviation From Standard Conditions	Recovered 5.1 (%) <sup>a</sup>	Yield 5.2 (%) <sup>a</sup>	Dihalide 5.3 (%) <sup>a</sup>	Reduction 5.4 (%) <sup>a</sup>
1	None	0	85	0	0
2	No Ligand	0	29	18	12
3	dppm instead of <i>rac</i> -BINAP	0	45	0	0
4	BPhen instead of <i>rac</i> -BINAP	0	82	22	0
5	bipy instead of <i>rac</i> -BINAP	0	72	0	0
6	No Nickel or ligand	0	0	86	0
7 <sup>b</sup>	Zn and NaI instead of MeMgI	15	<5	17	23
8 <sup>c</sup>	Zn and MgBr <sub>2</sub> instead of MeMgI	0	<5	54	19
9 <sup>d</sup>	Mn and NaI/TMSCl instead of MeMgI	38	0	0	11
10 <sup>e</sup>	TDAE and NaI instead of MeMgI	22	<5	0	12

<sup>a</sup>Yields determined by <sup>1</sup>H NMR compared to PhTMS as an internal standard. <sup>b</sup>2 equiv of Zn and 8 equiv of NaI

<sup>c</sup>2 equiv of Zn and MgBr<sub>2</sub>. <sup>d</sup>2 equiv of Mn and NaI and 1 equiv of TMSCl. <sup>e</sup>2 equiv of TDAE and NaI

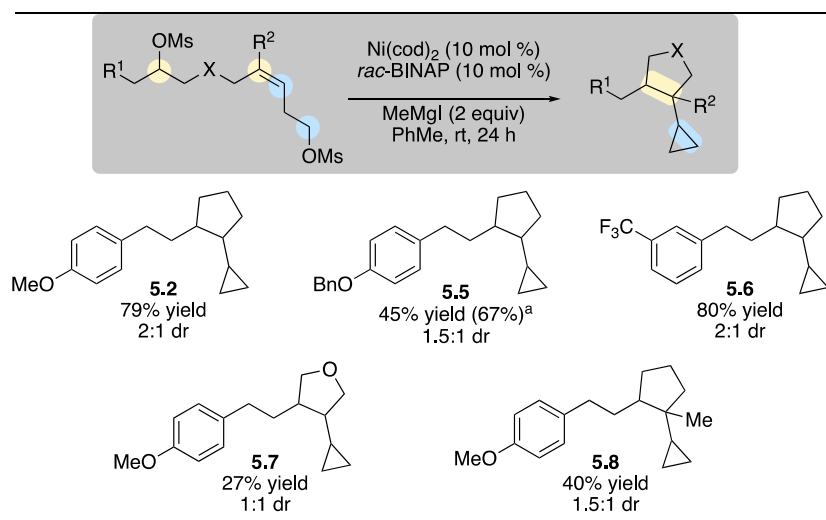
With these results, we then evaluated a series of ligands. While all ligands provided the desired product, *rac*-BINAP proved to be the optimal ligand (compare entry 1 v. entries 3–5). In the absence of the nickel catalyst and ligand, only diiodide (**5.3a**) was observed in an 86% yield (entry 6). This result is consistent with formation of diiodides in situ as reactive intermediates.<sup>20,25</sup> It also confirms that the nickel catalyst is necessary for the conjunctive XEC reaction to occur.

<sup>25</sup> For examples of nickel-catalyzed reactions that produce alkyl halides, see: (a) Prinsell, M. R.; Everson, D. A.; Weix, D. J. *Chem. Commun.* **2010**, 46, 5743–5745. (b) Do, H. Q.; Chandrashekar, E. R. R.; Fu, G. C. *J. Am. Chem. Soc.* **2013**, 135, 16288–16291. (c) Liang, Z.; Xue, W.; Lin, K.; Gong, H. *Org. Lett.* **2014**, 16, 5620–5623. (e) Duan, J.; Du, Y.-F.; Pang, X.; Shu, X.-Z. *Chem. Sci.* **2019**, 10, 8706–8712.

Finally, we evaluated alternative reductants and halide sources, including zinc, manganese, and TDAE, and found that the Grignard reagent provided the highest yield (entries 7–10).

With the optimal reaction conditions in hand, we investigated the scope of the cascade reaction (Scheme 5.2). We were delighted to observe that both electron-donating and electron-withdrawing substituents were well tolerated under our standard reaction conditions (**5.2**, **5.5**, **5.6**). The cascade reaction also allowed for synthesis of a substituted tetrahydrofuran (**5.7**). Finally, a trisubstituted alkene was subjected to the reaction conditions and afforded adjacent quaternary and tertiary centers (**5.8**), albeit in moderate yield.

**Scheme 5.2** Conjunctive XEC Reaction Scope



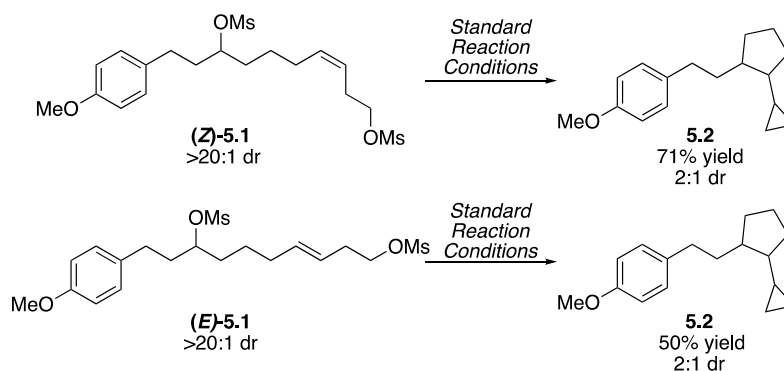
<sup>a</sup>Yield in Parenthesis is <sup>1</sup>H NMR yield compared to PhTMS as an internal standard

### 5.2.2 Investigation of the Mechanism of the Conjunctive XEC Reaction

Next, we turned our attention towards determining key features of the mechanism of this conjunctive XEC reaction. We hypothesized that the mechanism could proceed via two different pathways, involving either migratory insertion of an organonickel intermediate or a radical cyclization. Performing the cascade reaction with single olefin isomer of dimesylate **5.1** provides

a probe for radical versus organometallic cyclization (Scheme 5.3).<sup>26</sup> Migratory insertion is a stereospecific process,<sup>27</sup> and would be expected to provide a single diastereomer of cyclopentane **5.2**. In contrast, radical cyclization would be stereoablative and lead to formation of both diastereomers. We separated the alkene diastereomers, employing silver impregnated silica gel,<sup>28</sup> and subjected them separately to the cascade reaction. We observed that both (*E*)- and (*Z*)-**5.1** produced the same major diastereomer in 2:1 dr. This result is consistent with a radical exo-trig cyclization and not migratory insertion of an organonickel species.

**Scheme 5.3** Control Reaction with Single Alkene Diastereomer



We aimed to further corroborate the proposed radical exo-trig cyclization by examining reactions of diiodide **3a**. First, to confirm that diiodide was a competent intermediate in the catalytic cycle, we subjected **5.3a** to the standard reaction conditions and observed product **5.2** in 73% yield (Table 5.2, entry 1). Therefore, we propose that dimesylate **5.1** is converted to diiodide **5.3a** in situ, and this intermediate engages the nickel catalyst by halogen atom transfer (XAT).<sup>29</sup> If radical intermediates are operative, we should observe a decrease in yield with known radical

<sup>26</sup> For a discussion of hybrid organometallic-radical mechanisms, see: Kwiatkowski, M. R.; Alexanian, E. J. *Acc. Chem. Res.* **2019**, *52*, 1134–1144. (b) Wang, K.; Kong, W. *Chin. J. Chem.* **2018**, *36*, 247–256.

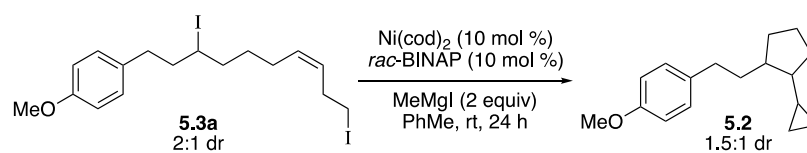
<sup>27</sup> (a) Heck, R. F. *J. Am. Chem. Soc.* **1969**, *91*, 6707–6714. (b) Hartwig, J. F. *Organotransition Metal Chemistry: From Bonding to Catalysis*; University Science Books, 2010.

<sup>28</sup> B. M. Lawrence *J. Chromatog.* **1968**, *38*, 535–537.

<sup>29</sup> For discussion of the mechanism of halogen atom transfer, see: (a) Kehoe, R.; Mahadevan, M.; Manzoor, A.; McMurray, G.; Wienfield, P.; Baird, M. C.; Budzelaar, P. H. M. *Organometallics* **2018**, *37*, 2450–2467. (b) Diccianni, J. B.; Katigbak, J.; Hu, C.; Diao, T. T. *J. Am. Chem. Soc.* **2019**, *141*, 1788–1796. (c) See reference 20.

inhibitors. Indeed, when one equivalent of TEMPO was added to the standard reaction conditions, we observed a decrease in yield (entry 2). In addition, we hypothesized that radical initiators, such as  $\text{SmI}_2$ , should produce the desired carbocyclic system.<sup>22</sup> Upon subjecting diiodide to a reaction with freshly prepared  $\text{SmI}_2$ ,<sup>30</sup> we were excited to observe the desired cascade product in 56% yield and as a 1.5:1 mixture of diastereomers (entry 3). These results are consistent with radical formation at one of the electrophilic centers and indicate that one or both cyclizations are radical mediated.

**Table 5.2** Control Reactions with Diiodide **5.3a**



Entry	Deviation From Standard Conditions	Yield <b>5.2</b> (%) <sup>a</sup>
1	None	73
2	1 equiv of TEMPO added	48
3	$\text{SmI}_2$ and THF instead of Ni, ligand and MeMgI	56 <sup>b</sup>

<sup>a</sup>Yield determined by <sup>1</sup>H NMR compared to PhTMS as an internal standard

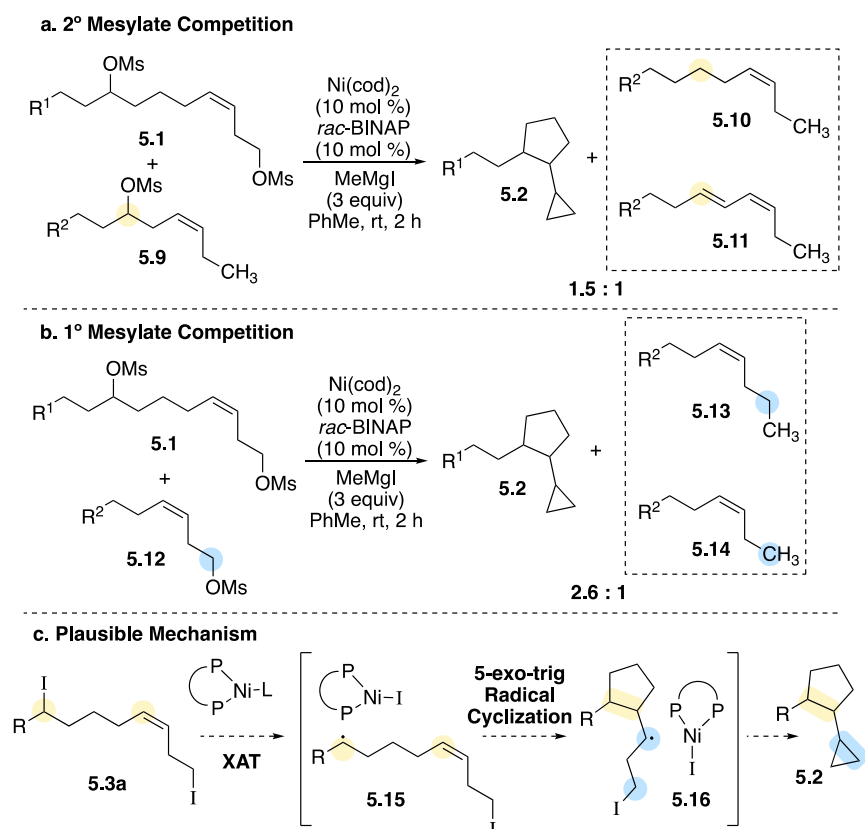
<sup>b</sup>Isolated Yield

Finally, we aimed to understand which electrophile was activated first. Based on the selectivity of XAT reactions, we hypothesized that the reaction initiated at the secondary center.<sup>20,22</sup> However, we had observed the formation of reduction product **5.4** in the optimization studies (vide supra) and considered that the primary iodide may engage the nickel complex first. We designed the following competition experiment to investigate the order of events. We synthesized two mesylates: one with a 2° mesylate (**5.9**) and one with a 1° mesylate (**5.12**). In a competition experiment between dimesylate **5.1** and 2° mesylate (**5.9**), we observed a 1.5:1 ratio of products (Scheme 5.4a). However, in a similar competition experiment, now employing 1° mesylate (**5.12**), the product ratio observed was 2.6:1 (Scheme 5.4b). The 2° mesylate reacted at a

<sup>30</sup> For the preparation of  $\text{SmI}_2$ , see: Szostak, M.; Spain, M.; Procter, D. J. *J. Org. Chem.* **2012**, *77*, 3049–3059.

competitive rate compared to dimesylate **5.1** and demonstrated that the 2° mesylate reacted faster than the 1° mesylate. These results indicate that the productive pathway for the conjunctive XEC reaction initiates at the secondary center. This selectivity is consistent with previous observations that secondary alkyl halides react at a faster rate than primary halides with nickel catalysts.<sup>20,29</sup>

**Scheme 5.4** Competition Experiment between Dimesylate **5.1** and 2° and 1° Mesylates **5.9** and **5.12**, and Proposed Reaction Mechanism.<sup>31</sup>



Based on the mechanistic experiments, we proposed the following plausible reaction mechanism (Scheme 5.4c). Beginning from diiodide **5.3a**, generated in situ, halogen atom transfer occurs at the secondary alkyl iodide to generate alkyl radical **5.15**. This secondary alkyl radical

<sup>31</sup> R<sup>1</sup> = 4-MeO-C<sub>6</sub>H<sub>4</sub>, R<sup>2</sup> = 4-BnO-C<sub>6</sub>H<sub>4</sub>.

cyclizes to afford the cyclopentane ring **5.16**.<sup>32</sup> This radical cyclization is consistent with formation of a mixture of diastereomers from either cis- or trans-alkenes (vide supra). Following this step, radical or nickel-mediated 3-exo-tet cyclization would afford cyclopropane **5.2**. Both pathways lead to a nickel(II) complex, which is reduced by the Grignard reagent to regenerate the nickel(0) catalyst.<sup>33</sup>

### 5.3 Conclusions

In conclusion, we have developed a nickel-catalyzed cascade reaction for synthesis of 3,5-vicinal carbocyclic motifs. We have demonstrated the scope of the reaction to include electron donating and withdrawing groups. In addition, we have provided preliminary mechanistic experiments to demonstrate that alkyl iodides are likely generated in situ and that the cascade reaction likely proceeds through radical intermediates. Future work includes delineating the remaining steps of the reaction mechanism and development of related cascade reactions.

### 5.4 Experimental Details

#### 5.4.1 General Procedures

All reactions were carried out under an atmosphere of N<sub>2</sub> when noted. All glassware was oven- or flame-dried prior to use. Tetrahydrofuran (THF), diethyl ether (Et<sub>2</sub>O), dichloromethane (DCM), methanol (MeOH), triethylamine (Et<sub>3</sub>N), dimethylformamide (DMF), and toluene (PhMe) were degassed with Ar and then passed through two 4 x 36 inch columns of anhydrous neutral A-2 alumina (8 x 14 mesh; LaRoche Chemicals; activated under a flow of argon at 350 °C for 12 h)

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<sup>32</sup> For a nickel-catalyzed Heck reactions that proceed through radical cyclization and/or organonickel intermediates, see: (a) Kwiatkowski, M. R.; Alexanian, E. J. *Angew. Chem. Int. Ed.* **2018**, *57*, 16857–16860. (b) Tang, S.; Liu, C.; Lei, A. *Chem. Commun.* **2013**, *49*, 2442–2444. (c) Liu, C.; Tang, S.; Liu, D.; Yuan, J.; Zheng, L.; Meng, L.; Lei, A. *Angew. Chem. Int. Ed.* **2012**, *51*, 3638–3641. (d) Matsubara, R.; Gutierrez, A. C.; Jamison, T. F. *J. Am. Chem. Soc.* **2011**, *133*, 19020–19023. (e) Standley, E. A.; Jamison, T. F. *J. Am. Chem. Soc.* **2013**, *135*, 1585–1592.

<sup>33</sup> Chen, P.-P.; Lucas, E. L.; Greene, M. A.; Zhang, Z.-Q.; Tollefson, E. J.; Erickson, L. W.; Taylor, B. L. H.; Jarvo, E. R.; Hong, X. *J. Am. Chem. Soc.* **2019**, *141*, 5835–5855.

to remove H<sub>2</sub>O.<sup>34</sup> All other solvents utilized were purchased “anhydrous” commercially, or purified as described. <sup>1</sup>H NMR spectra were recorded on Bruker DRX-400 (400 MHz <sup>1</sup>H, 100 MHz <sup>13</sup>C), GN-500 (500 MHz <sup>1</sup>H, 125.7 MHz <sup>13</sup>C), CRYO-500 (500 MHz <sup>1</sup>H, 125.7 MHz <sup>13</sup>C), or AVANCE-600 (600 MHz <sup>1</sup>H, 150 MHz <sup>13</sup>C) spectrometers. Proton chemical shifts are reported in ppm (δ) relative to internal tetramethylsilane (TMS, δ 0.00). Data are reported as follows: chemical shift (multiplicity [singlet (s), broad singlet (br s), doublet (d), doublet of doublets (dd), doublet of doublet of doublets (ddd), doublet of doublet of triplets (ddt), doublet of triplets (dt), triplet (t), triplet of doublets (td), triplet of triplets (tt), quartet (q), quartet of doublets (qd), quintet (quin), apparent singlet (as), apparent doublet (ad), apparent doublets of doublets (add), apparent doublet of triplets (adt), apparent doublet of quintets (adquin), apparent doublet of septets (adsept), apparent triplet (at), apparent triplet of doublets (atd), apparent triplet of triplets (att), apparent quartet (aq), apparent quartet of doublets (aqd), apparent quartet of triplets (aqt), apparent quintet of doublets (aquin d), apparent sextet (asext), apparent septet (asept), multiplet (m)], coupling constants [Hz], integration). Carbon chemical shifts are reported in ppm (δ) relative to TMS with the respective solvent resonance as the internal standard (CDCl<sub>3</sub>, δ 77.16 ppm). Unless otherwise indicated, NMR data were collected at 25 °C. Infrared (IR) spectra were obtained on a Thermo Scientific Nicolet iS5 spectrometer with an iD5 ATR tip (neat) and are reported in terms of frequency of absorption (cm<sup>-1</sup>). Analytical thin-layer chromatography (TLC) was performed using Silica Gel 60 F254 precoated plates (0.25 mm thickness). Visualization was accomplished by irradiation with a UV lamp. Flash chromatography was performed using SiliaFlash F60 (40-63 μm, 60 Å) from SiliCycle. Automated chromatography was carried out on a Teledyne Isco CombiFlash Rf Plus. Melting points (M.p.) were obtained using a MelTemp melting point

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<sup>34</sup> Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518–1520.



apparatus and are uncorrected. High resolution mass spectrometry was performed by the University of California, Irvine Mass Spectrometry Center.

Bis(1,5-cyclooctadiene)nickel was purchased from Strem, stored in a glove box freezer (–20 °C) under an atmosphere of N<sub>2</sub> and used as received. All ligands were purchased from Strem or Sigma Aldrich and were stored under N<sub>2</sub> atmosphere and used as received. The methylmagnesium iodide was titrated with iodine prior to use.<sup>35</sup> All other chemicals were purchased commercially and used as received, unless otherwise noted.

## 5.4.2 Conjunctive XEC Reaction Procedures

### 5.4.2.1 Method A: Nickel-Catalyzed Conjunctive XEC Reaction

In a glovebox, an oven-dried 7 mL dram vial equipped with a stir bar was charged with dimesylate (1.0 equiv), Ni(cod)<sub>2</sub> (10. mol %), *rac*-BINAP (10. mol %) and PhMe (0.20 M in substrate). A solution of MeMgI in Et<sub>2</sub>O (2.0 equiv) was then added dropwise via syringe. After 24 h, the reaction vial was removed from the glovebox, quenched with MeOH, filtered through a plug of silica gel eluting with Et<sub>2</sub>O, and concentrated in vacuo. Phenyltrimethylsilane (PhTMS; 8.6 μL, 50. μmol) was added and the yield was determined by <sup>1</sup>H NMR based on comparison to PhTMS as internal standard before purification by column chromatography.

To remove olefin by-products resulting from β-hydride elimination, a Sharpless asymmetric dihydroxylation was performed.<sup>36</sup> To a flame-dried 7-mL dram vial was added AD-mix-β (1.4 g/mmol of substrate). The flask was sealed with a septum cap and placed under an atmosphere of N<sub>2</sub>. Then *t*-BuOH (0.50 mL) and H<sub>2</sub>O (0.50 mL) were added via syringe. The vial was cooled to 0 °C and then unpurified reaction mixture was added dropwise as a solution in *t*-

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<sup>35</sup> Krasoviskiy, A.; Knochel, P. *Synthesis*, **2006**, 5, 890–891.

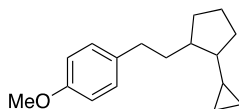
<sup>36</sup> (a) For the original report of the asymmetric dihydroxylation, see: Jacobsen, E. N.; Marko, I.; Mungall, W. S.; Schroeder, G.; Sharpless, K. B. *J. Am. Chem. Soc.* **1988**, *110*, 1968–1970. (b) For the procedure that was employed, see: Holl, K.; Schepmann, D.; Daniliuc, C. G.; Wunsch, B. *Tetrahedron Asymmetry*, **2014**, *25*, 268–277.

BuOH (0.50 mL) and H<sub>2</sub>O (0.5 mL) via syringe. The mixture was allowed to stir at 0 °C for 24 h. To quench, saturated aq. Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (1.0 mL) was added and the mixture was allowed to warm to rt and stir for 1 h. Then the mixture was transferred to a separatory funnel, and the organic layer was extracted with EtOAc (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated in vacuo.

#### 5.4.2.2 Preparation of Grignard Reagent

Under a N<sub>2</sub> atmosphere, a three-necked flask equipped with a stir bar, reflux condenser, and Schlenk filtration apparatus was charged with magnesium turnings (1.1 g, 45 mmol, 1.5 equiv). The flask and magnesium turnings were then flame-dried under vacuum and the flask was back-filled with N<sub>2</sub>. Anhydrous Et<sub>2</sub>O (7.0 mL) and a crystal of iodine (ca. 2.0 mg) were added to the flask. Freshly distilled iodomethane (1.9 mL, 31 mmol, 1.0 equiv) was slowly added over 30 min to maintain a gentle reflux. The mixture was stirred for 2 h at room temperature then filtered through the fritted Schlenk filter into a Schlenk flask under N<sub>2</sub> atmosphere. The magnesium turnings were washed with Et<sub>2</sub>O (2 x 1.0 mL) then the Schlenk flask was sealed, removed, and placed under an N<sub>2</sub> atmosphere. The resulting methylmagnesium iodide was typically between 2.4 and 3.0 M as titrated by Knochel's method<sup>35</sup> and could be stored, sealed under N<sub>2</sub> atmosphere or in a glovebox, for up to 4 weeks.

#### 5.4.3 Characterization Data for Conjunctive XEC Reaction Products



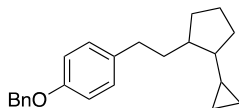
**1-(2-(2-cyclopropylcyclopentyl)ethyl)-4-methoxybenzene (5.2)** The following amounts of reagents were used: dimesylate **5.1** (61 mg, 0.14 mmol, 1.0 equiv), Ni(cod)<sub>2</sub> (3.9 mg, 14 μmol, 10 mol %), *rac*-BINAP (8.7 mg, 14 μmol, 10 mol %), MeMgI (0.11 mL, 0.28 mmol, 2.0 equiv, 2.5

M in Et<sub>2</sub>O), PhMe (0.70 mL), AD-mix-β (0.20 g, 1.4 g/mmol of dimesylate), *t*-BuOH (0.50 mL), and H<sub>2</sub>O (0.50 mL). Purification by column chromatography (100% hexanes) afforded the title compound as a clear and colorless oil (24 mg, 0.10 mmol, 71% yield, 2:1 dr). The dr was determined by the integration of resonances attributed to the aromatic carbons in <sup>13</sup>C NMR. For clarity, the <sup>1</sup>H and <sup>13</sup>C data are tabulated individually.

**TLC** R<sub>f</sub> = 0.7 (5% EtOAc/hexanes, CAM Stain); **HRMS** (TOF MS CI+) *m/z*: [M]<sup>+</sup> calcd for C<sub>17</sub>H<sub>24</sub>O, 244.1827; found 244.1831.

**Major Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.08–6.98 (m, 2H), 6.75 (d, *J* = 8.1 Hz, 2H), 3.70 (d, *J* = 1.6 Hz, 3H), 2.60–2.38 (m, 2H), 1.92–1.80 (m, 1H), 1.79–1.56 (m, 2H), 1.55–1.37 (m, 3H), 1.38–1.00 (m, 3H), 0.94–0.68 (m, 1H), 0.53–0.43 (m, 1H), 0.37 (ddq, *J* = 21.2, 8.5, 4.3 Hz, 1H), 0.26 (dt, *J* = 22.5, 8.9, 4.7 Hz, 1H), 0.05 (tq, *J* = 9.4, 4.6 Hz, 1H), -0.10 (tq, *J* = 9.5, 4.6 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 157.7, 135.6, 129.3 (2C), 113.8 (2C), 55.4, 48.5, 43.1, 34.6, 33.5, 31.8, 30.7, 23.0, 11.8, 5.5, 3.0.

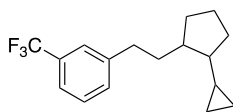
**Minor Diastereomer:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.08–6.98 (m, 2H), 6.75 (d, *J* = 8.1 Hz, 2H), 3.70 (d, *J* = 1.6 Hz, 3H), 2.60–2.38 (m, 2H), 1.92–1.80 (m, 1H), 1.79–1.56 (m, 2H), 1.55–1.37 (m, 3H), 1.38–1.00 (m, 3H), 0.94–0.68 (m, 1H), 0.53–0.43 (m, 1H), 0.37 (ddq, *J* = 21.2, 8.5, 4.3 Hz, 1H), 0.26 (dt, *J* = 22.5, 8.9, 4.7 Hz, 1H), 0.05 (tq, *J* = 9.4, 4.6 Hz, 1H), -0.10 (tq, *J* = 9.5, 4.6 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 157.7, 135.4, 129.3 (2C), 113.8 (2C), 55.4, 51.0, 46.2, 37.6, 34.2, 32.6, 32.1, 23.6, 15.4, 4.4, 2.3.



**1-(benzyloxy)-4-(2-(2-cyclopropylcyclopentyl)ethyl)benzene (5.5)** was prepared according to Method A. The following amounts of reagents were used: dimesylate **5.17** (32 mg, 60. μmol, 1.0

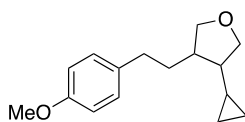
equiv), Ni(cod)<sub>2</sub> (1.7 mg, 6.0 μmol, 10 mol %), *rac*-BINAP (3.7 mg, 6.0 μmol, 10 mol %), MeMgI (40. μL, 0.12 mmol, 2.0 equiv, 3.0 M in Et<sub>2</sub>O), PhMe (0.30 mL), AD-mix-β (84 mg, 1.4 g/mmol), *t*-BuOH (1.0 mL) and H<sub>2</sub>O (1.0 mL). Purification by column chromatography (0–10% EtOAc/hexanes) afforded the title compound as a pale yellow oil (8.6 mg, 28 μmol, 45% yield, 1.5:1 dr). The dr was determined by the integration of resonances attributed to the aromatic carbons in <sup>13</sup>C NMR. **TLC** R<sub>f</sub> = 0.8 (10% EtOAc/hexanes, CAM Stain); **<sup>1</sup>H NMR** (600 MHz, CDCl<sub>3</sub>) δ 7.43 (d, *J* = 7.3 Hz, 4H, both diastereomers), 7.38 (t, *J* = 7.4 Hz, 4H, both diastereomers), 7.34–7.29 (m, 2H, both diastereomers), 7.14–7.09 (m, 4H, both diastereomers), 6.92–6.87 (m, 4H, both diastereomers), 5.04 (s, 4H, both diastereomers), 2.62 (dddd, *J* = 13.6, 10.4, 5.3, 2.8 Hz, 2H, both diastereomers), 2.52 (dtd, *J* = 13.9, 10.0, 6.1, 2H, both diastereomers), 1.99–1.86 (m, 2H, both diastereomers), 1.86–1.61 (m, 6H, both diastereomers), 1.62–1.55 (m, 2H, both diastereomers), 1.54–1.46 (m, 2H, both diastereomers), 1.45–1.27 (m, 2H, both diastereomers), 1.21 (dq, *J* = 12.5, 8.5 Hz, 1H, minor diastereomer), 1.13 (dq, *J* = 11.3, 6.0, 1H, major diastereomer), 0.99–0.76 (m, 4H, both diastereomers), 0.55 (ddd, *J* = 13.1, 7.9, 5.3, 2.9 Hz, 2H, both diastereomers), 0.47 (dddd, *J* = 9.4, 8.1, 5.5, 4.2 Hz, 1H, major diastereomer), 0.44–0.39 (m, 1H, minor diastereomer), 0.40–0.34 (m, 1H, major diastereomer), 0.34–0.29 (m, 1H, minor diastereomer), 0.12 (tdd, *J* = 9.3, 5.2, 4.1 Hz, 2H, both diastereomers), -0.01–0.06 (m, 2H, both diastereomers); **<sup>13</sup>C NMR** (151 MHz, CDCl<sub>3</sub>) δ 156.96 (major diastereomer) 156.95 (minor diastereomer), 137.4 (2C, both diastereomers), 135.9 (minor diastereomer), 135.8 (major diastereomer), 129.4 (2C, major diastereomer), 129.3 (2C, minor diastereomer), 128.7 (4C, both diastereomers), 128.0 (2C, both diastereomers), 127.6 (4C, both diastereomers), 114.78 (2C, minor diastereomer), 114.76 (2C, major diastereomer), 70.20 (2C, both diastereomers), 51.0 (minor diastereomer), 48.5 (major diastereomer), 46.2 (minor diastereomer), 43.1 (major diastereomer), 37.6 (minor diastereomer),

34.6 (major diastereomer), 34.2 (minor diastereomer), 33.5 (major diastereomer), 32.6 (minor diastereomer) 32.1 (minor diastereomer), 31.8 (major diastereomer), 30.7 (major diastereomer), 23.6 (minor diastereomer), 23.0 (major diastereomer), 15.4 (minor diastereomer), 11.8 (major diastereomer), 5.5 (major diastereomer), 4.4 (minor diastereomer), 3.0 (major diastereomer), 2.3 (minor diastereomer); **HRMS** (TOF MS CI+)  $m/z$ :  $[M]^+$  calcd for  $C_{23}H_{28}O$ , 320.2140, found 320.2154.



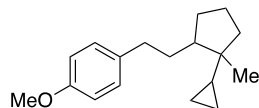
**1-(2-(2-cyclopropylcyclopentyl)ethyl)-3-(trifluoromethyl)benzene (5.6)** was prepared according to Method A. The following amounts of reagents were used: dimesylate **5.18** (43 mg, 92  $\mu$ mol, 1.0 equiv), Ni(cod)<sub>2</sub> (2.5 mg, 9.2  $\mu$ mol, 10 mol %), *rac*-BINAP (5.7 mg, 9.2  $\mu$ mol, 10 mol %), MeMgI (70.  $\mu$ L, 0.18 mmol, 2.0 equiv, 2.5 M in Et<sub>2</sub>O), and PhMe (0.50 mL). Purification by column chromatography (100% hexanes) afforded the title compound as a clear and colorless oil (21 mg, 74  $\mu$ mol, 80% yield, 2:1 dr). The dr was determined by the integration of resonances attributed to the aromatic carbons in <sup>13</sup>C NMR. **TLC**  $R_f$  = 0.8 (10% EtOAc/hexanes, CAM Stain); **<sup>1</sup>H NMR** (600 MHz, CDCl<sub>3</sub>)  $\delta$  7.47–7.41 (m, 4H, both diastereomers), 7.40–7.36 (m, 4H, both diastereomers), 2.74 (ddt,  $J$  = 13.7, 10.3, 5.1 Hz, 2H, both diastereomers), 2.65 (ddt,  $J$  = 13.8, 10.5, 6.8 Hz, 2H, both diastereomers), 2.07–1.88 (m, 4H, both diastereomers), 1.87–1.67 (m, 4H, diastereomers), 1.66–1.55 (m, 2H, both diastereomers), 1.54–1.48 (m, 2H, both diastereomers), 1.47–1.30 (m, 4H, both diastereomers), 1.28–1.19 (m, 1H, major diastereomer), 1.15 (dq,  $J$  = 11.5, 6.2 Hz, 1H, minor diastereomer), 1.00–0.79 (m, 2H, both diastereomers), 0.56 (dddd,  $J$  = 14.9, 8.1, 5.7, 4.0 Hz, 2H, both diastereomers), 0.52–0.46 (m, 1H, major diastereomers), 0.44 (ddd,  $J$  = 8.2, 5.4, 4.2 Hz, 1H, minor diastereomer), 0.39 (dddd,  $J$  = 9.3, 8.2, 5.4, 4.1 Hz, 1H, major

diastereomer), 0.34 (dddd,  $J = 9.4, 8.3, 5.4, 4.2$  Hz, 1H, minor diastereomer), 0.13 (dt,  $J = 9.8, 4.9$  Hz, 2H, both diastereomers), 0.04 to -0.04 (m, 2H, both diastereomers);  $^{13}\text{C}$  NMR (151 MHz,  $\text{CDCl}_3$ )  $\delta$  144.3 (major diastereomer), 144.2 (minor diastereomer), 131.91 (q,  $J = 1.7$  Hz, major diastereomer), 131.89 (q,  $J = 1.7$  Hz, minor diastereomer), 130.7 (q,  $J = 31.5$  Hz, 2C, both diastereomers), 128.8 (2C, both diastereomers), 125.2 (q,  $J = 3.3$  Hz, major diastereomer), 125.1 (q,  $J = 3.9$  Hz, minor diastereomer), 124.5 (q,  $J = 272.6$  Hz, 2C, both diastereomers), 122.6 (q,  $J = 3.9$  Hz, 2C, both diastereomers), 50.9 (minor diastereomer), 48.4 (major diastereomer), 46.2 (minor diastereomer), 43.1 (major diastereomer), 37.1 (minor diastereomer), 35.3 (major diastereomer), 35.0 (minor diastereomer), 33.1 (major diastereomer), 32.6 (minor diastereomer), 32.1 (minor diastereomer), 31.7 (major diastereomer), 30.7 (major diastereomer), 23.6 (minor diastereomer), 23.0 (major diastereomer), 15.4 (minor diastereomer), 11.8 (major diastereomer), 5.5 (major diastereomer), 4.4 (minor diastereomer), 3.1 (major diastereomer), 2.3 (minor diastereomer);  $^{19}\text{F}$  NMR (565 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.5 (3F, major diastereomer), -62.6 (3F, minor diastereomer); HRMS (TOF MS  $\text{CI}^+$ )  $m/z$ :  $[\text{M}]^+$  calcd for  $\text{C}_{17}\text{H}_{21}\text{F}_3$ , 282.1595, found 282.1595.



**3-cyclopropyl-4-(4-methoxyphenethyl)tetrahydrofuran (5.7)** was prepared according to Method A. The following amounts of reagents were used: dimesylate **5.19** (58 mg, 0.13 mmol, 1.0 equiv),  $\text{Ni}(\text{cod})_2$  (3.6 mg, 13  $\mu\text{mol}$ , 10. mol %), *rac*-BINAP (8.1 mg, 13  $\mu\text{mol}$ , 10 mol %),  $\text{MeMgI}$  (90.  $\mu\text{L}$ , 0.26 mmol, 2.0 equiv, 2.9 M in  $\text{Et}_2\text{O}$ ), and PhMe (0.70 mL). Before purification a  $^1\text{H}$  NMR yield of 28% yield was obtained based on comparison to PhTMS as an internal standard. The residue was purified by column chromatography (0–10% EtOAc/hexanes) to afford the title compound as a clear and colorless oil (8.2 mg, 33  $\mu\text{mol}$ , 27% yield, 1:1 dr). The dr was determined

by the integration of resonances attributed to the aromatic carbons in  $^{13}\text{C}$  NMR. **TLC**  $R_f = 0.2$  (10% EtOAc/Hexanes, CAM Stain);  **$^1\text{H}$  NMR** (600 MHz,  $\text{CDCl}_3$ )  $\delta$  7.10 (at,  $J = 8.9$  Hz, 4H, both diastereomers), 6.84 (dd,  $J = 8.9, 3.4$  Hz, 4H, both diastereomers), 4.03 (dd,  $J = 8.4, 7.4$  Hz, 1H, one diastereomer), 3.93 (dt,  $J = 20.4, 7.6$  Hz, 2H, both diastereomers), 3.88 (dd,  $J = 8.3, 6.4$  Hz, 1H, one diastereomer), 3.79 (s, 6H, both diastereomers), 3.69 (dd,  $J = 8.3, 4.6$  Hz, 1H, one diastereomer), 3.59–3.53 (m, 2H, both diastereomers), 3.44 (dd,  $J = 8.4, 7.7$  Hz, 1H, one diastereomer), 2.62–2.52 (m, 4H, both diastereomers), 2.23 (dq,  $J = 12.9, 7.6$  Hz, 1H, one diastereomer), 2.07–1.87 (m, 3H, both diastereomer), 1.70 (dtd,  $J = 13.4, 9.1, 6.5$  Hz, 1H, one diastereomer), 1.56–1.50 (m, 1H, one diastereomer), 1.49–1.42 (m, 1H, one diastereomer), 1.20 (dq,  $J = 9.0, 7.4$  Hz, 1H, one diastereomer), 0.74–0.67 (m, 1H, one diastereomer), 0.67–0.61 (m, 1H, one diastereomer), 0.57 (dddd,  $J = 9.7, 8.2, 5.5, 4.4$  Hz, 1H, one diastereomer), 0.52–0.44 (m, 2H, both diastereomers), 0.41 (dddd,  $J = 9.3, 8.0, 5.4, 4.4$  Hz, 1H, one diastereomer), 0.18 (dq,  $J = 10.1, 5.2$  Hz, 2H, both diastereomers), 0.05 (td,  $J = 9.6, 4.8$  Hz, 2H, both diastereomers);  **$^{13}\text{C}$  NMR** (151 MHz,  $\text{CDCl}_3$ )  $\delta$  157.93 (one diastereomer), 157.92 (one diastereomer), 134.5 (one diastereomer), 134.4 (one diastereomer), 129.33 (2C, one diastereomer), 129.29 (2C, one diastereomer), 113.9 (4C, both diastereomers), 74.2 (one diastereomer), 73.7 (one diastereomer), 73.4 (one diastereomer), 72.7 (one diastereomer), 55.4 (2C, both diastereomers), 50.8 (one diastereomer), 47.9 (one diastereomer), 45.7 (one diastereomer), 42.4 (one diastereomer), 35.4 (one diastereomer), 34.4 (one diastereomer), 34.1 (one diastereomer), 30.6 (one diastereomer), 13.3 (one diastereomer), 10.0 (one diastereomer), 5.0 (one diastereomer), 3.7 (one diastereomer), 3.1 (one diastereomer), 2.7 (one diastereomer); **HRMS** (TOF MS  $\text{CI}^+$ )  $m/z$ :  $[\text{M}]^+$  calcd for  $\text{C}_{16}\text{H}_{22}\text{O}_2$ , 246.1620, found 246.1614.

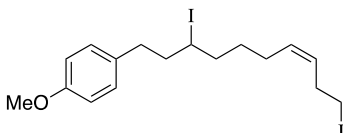


**1-(2-(2-cyclopropyl-2-methylcyclopentyl)ethyl)-4-methoxybenzene (5.8)** was prepared according to Method A. The following amount of reagents were used: dimesylate **5.20** (27 mg, 60.  $\mu\text{mol}$ , 1.0 equiv),  $\text{Ni}(\text{cod})_2$  (1.7 mg, 6.0  $\mu\text{mol}$ , 10 mol %), *rac*-BINAP (3.7 mg, 6.0  $\mu\text{mol}$ , 10 mol %),  $\text{MeMgI}$  (40.  $\mu\text{L}$ , 0.12 mmol, 2.0 equiv, 3.0 M in  $\text{Et}_2\text{O}$ ),  $\text{PhMe}$  (0.30 mL), AD-mix- $\beta$  (84 mg, 1.4 g/mmol), *t*-BuOH (1.0 mL) and  $\text{H}_2\text{O}$  (1.0 mL). Purification by column chromatography (0–10% EtOAc/hexanes) afforded the title compound as a pale yellow oil (6.0 mg, 23  $\mu\text{mol}$ , 40% yield, 1.5:1 dr). The dr was determined by the integration of resonances attributed to the aromatic carbons in  $^{13}\text{C}$  NMR. **TLC**  $R_f$  = 0.7 (10% EtOAc/hexanes, CAM Stain);  **$^1\text{H}$  NMR** (600 MHz,  $\text{CDCl}_3$ )  $\delta$  7.12 (d,  $J$  = 8.4 Hz, 4H, both diastereomers), 6.83 (dd,  $J$  = 9.1, 3.0 Hz, 4H, both diastereomers), 3.79 (s, 6H, both diastereomers), 2.66 (ddd,  $J$  = 15.0, 10.6, 4.9 Hz, 1H, minor diastereomer), 2.45 (ddd,  $J$  = 13.8, 10.2, 6.4 Hz, 1H, major diastereomers), 1.94 (qd,  $J$  = 10.1, 9.0, 1H, major diastereomer), 1.83 (dddd,  $J$  = 13.3, 9.9, 6.4, 3.0 Hz, 1H, minor diastereomer), 1.70–1.55 (m, 2H, both diastereomers), 1.52–1.43 (m, 2H, both diastereomers), 1.44–1.36 (m, 2H, both diastereomers), 1.35–1.24 (m, 6H, both diastereomers), 1.21 (ddd,  $J$  = 12.8, 6.4, 3.3 Hz, 2H, both diastereomers), 0.91–0.79 (m, 4H, both diastereomers), 0.73 (s, 3H, major diastereomer), 0.69 (s, 3H, minor diastereomer), 0.67–0.62 (m, 2H, both diastereomers), 0.24 (dq,  $J$  = 13.0, 8.9, 4.2 Hz, 4H, both diastereomers), 0.18–0.08 (m, 4H, both diastereomers);  **$^{13}\text{C}$  NMR** (151 MHz,  $\text{CDCl}_3$ )  $\delta$  157.72 (minor diastereomer), 157.70 (major diastereomer), 135.59 (major diastereomer), 135.58 (minor diastereomer), 129.4 (2C, minor diastereomer), 129.3 (2C, major diastereomer), 113.80 (2C, major diastereomer), 113.78 (2C, minor diastereomer), 55.4 (2C, both diastereomers) 51.1 (2C, both diastereomers), 43.1 (2C, both diastereomers), 38.1 (2C, both diastereomers), 34.8 (2C,



both diastereomers), 32.7 (2C, both diastereomers), 31.1 (2C, both diastereomers), 23.4 (2C, both diastereomers), 22.0 (2C, both diastereomers), 15.6 (2C, both diastereomers) 1.2 (minor diastereomer), 1.1 (major diastereomer), 0.3 (major diastereomer), 0.2 (minor diastereomer); **HRMS** (TOF MS CI+)  $m/z$ :  $[M]^+$  calcd for  $C_{18}H_{26}O$ , 258.1984, found 258.1980.

#### 5.4.4 Characterization Data for By-Products Formed from Optimization Studies

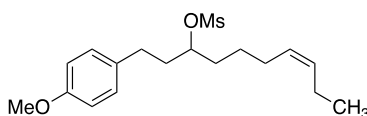


**(3,10-diiododec-7-en-1-yl)-4-methoxybenzene (5.3a)** was observed as a by-product from the optimization studies as a 2:1 mixture of alkene diastereomers (see Table 5.1). Below is the characterization of the title compound. For clarity the  $^1H$  and  $^{13}C$  data are tabulated separately. **TLC**  $R_f$  = 0.7 (10% EtOAc/hexanes, CAM Stain); **HRMS** (TOF MS CI+)  $m/z$ :  $[M]^+$  calcd for  $C_{17}H_{24}I_2O$ , 497.9917; found 497.9906.

**Major Diastereomer:**  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.12 (d,  $J$  = 8.7 Hz, 2H), 6.85 (d,  $J$  = 8.6 Hz, 2H), 5.59–5.42 (m, 1H), 5.40–5.28 (m, 1H), 4.04 (tt,  $J$  = 8.8, 4.3 Hz, 1H), 3.80 (s, 3H), 3.15 (td,  $J$  = 7.2, 4.0 Hz, 2H), 2.84 (ddd,  $J$  = 13.9, 8.9, 5.0 Hz, 1H), 2.74–2.65 (m, 1H), 2.62 (aq,  $J$  = 7.2, 6.1 Hz, 2H), 2.15 (dtd,  $J$  = 14.3, 9.1, 5.0 Hz, 1H), 2.09–1.84 (m, 4H), 1.81–1.69 (m, 1H), 1.68–1.57 (m, 1H), 1.54–1.42 (m, 1H);  $^{13}C$  NMR (101 MHz,  $CDCl_3$ )  $\delta$  158.1, 133.0, 131.8, 129.6 (2C), 128.7, 114.1 (2C), 55.4, 42.6, 40.3, 39.0, 34.8, 31.6, 29.4, 26.7, 5.5.

**Minor Diastereomer:**  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.12 (d,  $J$  = 8.7 Hz, 2H), 6.85 (d,  $J$  = 8.6 Hz, 2H), 5.59–5.42 (m, 1H), 5.40–5.28 (m, 1H), 4.04 (tt,  $J$  = 8.8, 4.3 Hz, 1H), 3.80 (s, 3H), 3.15 (td,  $J$  = 7.2, 4.0 Hz, 2H), 2.84 (ddd,  $J$  = 13.9, 8.9, 5.0 Hz, 1H), 2.74–2.65 (m, 1H), 2.55 (aq,  $J$  = 7.2, 6.7 Hz, 2H), 2.15 (dtd,  $J$  = 14.3, 9.1, 5.0 Hz, 1H), 2.09–1.84 (m, 4H), 1.81–1.69 (m, 1H),

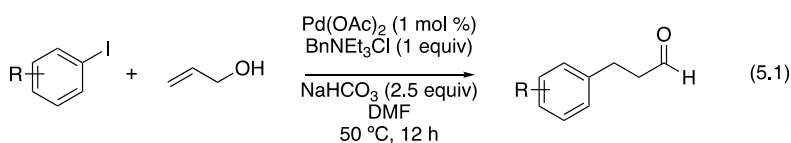
1.68–1.57 (m, 1H), 1.54–1.42 (m, 1H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  158.1, 132.8, 131.8, 129.6 (2C), 129.2, 114.0 (2C), 55.4, 42.6, 40.1, 39.2, 36.7, 31.7, 29.1, 26.7, 6.3.



**(4-Methoxyphenyl)dec-7-en-3-yl methanesulfonate (5.4)** was observed as a by-product from the optimization studies (see Table 1). Below is the characterization for the major diastereomer.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.11 (d,  $J = 8.2$  Hz, 2H), 6.84 (d,  $J = 8.4$  Hz, 2H), 5.57–5.22 (m, 2H), 4.75 (sextet,  $J = 6.1$  Hz, 1H), 3.79 (s, 3H), 2.99 (s, 3H), 2.67 (qdd,  $J = 14.1, 9.5, 6.1$  Hz, 2H), 2.15–1.87 (m, 4H), 1.74 (tt,  $J = 9.1, 6.0$  Hz, 2H), 1.68–1.52 (m, 1H), 1.52–1.14 (m, 3H), 0.96 (tdd,  $J = 7.5, 3.5, 1.1$  Hz, 3H);  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  158.1, 133.1, 132.7, 129.4 (2C), 128.2, 114.1 (2C), 83.4, 55.4, 38.9, 36.5, 32.2, 30.5, 26.8, 24.9, 20.7, 14.1; HRMS (TOF MS ES+)  $m/z$ :  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{18}\text{H}_{28}\text{O}_4\text{SNa}$ , 363.1606; found 363.1615.

## 5.4.5 General Procedures for the Synthesis of Dimesylate Starting Materials

### 5.4.5.1 Method B: Pd-Catalyzed Heck Reaction



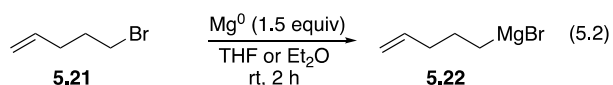
This method was adapted from a procedure reported by Su.<sup>37</sup> A flame dried pressure tube equipped with a stir bar was charged with aryl iodide<sup>38</sup> (1.0 equiv),  $\text{NaHCO}_3$  (2.5 equiv), and  $\text{BnNEt}_3\text{Cl}$  (1.0 equiv). The flask was sealed with a septum and pumped into a glovebox. Then the pressure tube was charged with  $\text{Pd}(\text{OAc})_2$  (1.0 mol %), sealed with septum, removed from the glovebox and placed under an atmosphere of  $\text{N}_2$ . Then DMF (0.20 M) was added via syringe followed by allyl

<sup>37</sup> Shang, Y., Jie, X., Jonnada, K. Zafar, S. N.; Su, W. *Nat Commun.* **2017**, *8*, 2273.

<sup>38</sup> Note: if the aryl iodide starting material is a liquid, it was added via syringe after the flask was removed from the glovebox.

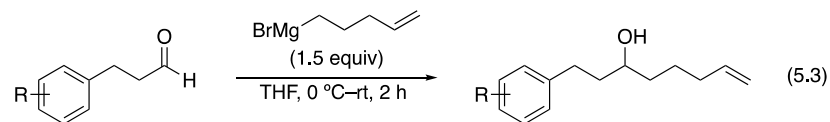
alcohol (1.5 equiv). The pressure tube was sealed with a teflon cap, heated to 50 °C and allowed to stir at that temperature for at least 5 h. After the reaction was complete, the flask was cooled to rt and allowed to stir at that temperature for 1 h. The mixture was then filtered through a pad of silica gel eluting with EtOAc. The filtrate was concentrated to remove the excess EtOAc and then transferred to a separatory funnel. The organic layer was washed with excess H<sub>2</sub>O (x 5) and then brine. The combined organic layers were dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo. The product was purified by column chromatography.

#### 5.4.5.2 Method C: Preparation of Grignard Reagent



To a flame dried two-neck round bottom flask equipped with a stir bar, was added magnesium turnings (1.5 equiv). The flask and magnesium turnings were then flame-dried under vacuum and the flask was back-filled with N<sub>2</sub>. Anhydrous Et<sub>2</sub>O or THF (2.0 M) and a crystal of iodine (ca. 2.0 mg) were added to the flask. Then alkyl bromide **5.21** (1.0 equiv) was slowly added neat over 30 min to maintain a gentle reflux. The mixture was stirred for 2 h at room temperature. The resulting Grignard reagent was titrated according to Knochel's method and used immediately.<sup>35</sup>

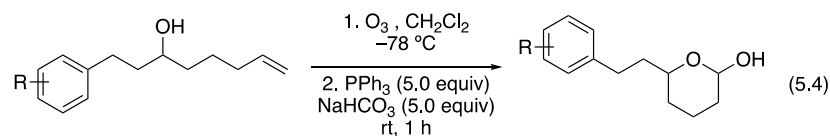
#### 5.4.5.3 Method D: Nucleophilic Grignard Addition into Aldehyde



To a flame dried round bottom flask equipped with a stir bar, was added aldehyde (1.0 equiv) as a solution in THF or Et<sub>2</sub>O (0.20 M). The flask was cooled to 0 °C and the freshly prepared Grignard reagent (1.5 equiv) was added dropwise via syringe. The flask is warmed to rt and allowed to stir for 2 h. To quench, saturated aq. NH<sub>4</sub>Cl was added to the reaction mixture. The biphasic solution was transferred to a separatory funnel and the organic layer was extracted with EtOAc. The

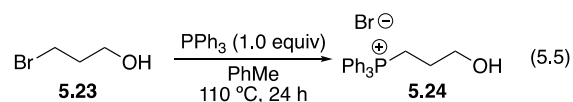
combined organic layers were washed with H<sub>2</sub>O, brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo.

#### 5.4.5.4 Method E: Ozonolysis of Terminal Alkenes



This method was adapted from a procedure reported by was prepared by Taber.<sup>39</sup> A round-bottom flask equipped with stir bar was charged with alkene (1.0 equiv), and CH<sub>2</sub>Cl<sub>2</sub> (0.10 M). The reaction was then cooled to  $-78\text{ }^{\circ}\text{C}$ , and ozone was bubbled through mixture until the solution turned blue. Ozone flow was then discontinued, and O<sub>2</sub> was passed through the solution for 5 min. PPh<sub>3</sub> (5.0 equiv) and NaHCO<sub>3</sub> (5.0 equiv) were then added to flask and the mixture was allowed stirred for at least 1 h. Then H<sub>2</sub>O was added and the biphasic mixture was transferred to a separatory funnel. The organic layer was extracted with CH<sub>2</sub>Cl<sub>2</sub> (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo.

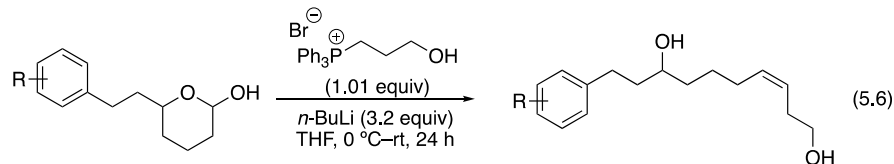
#### 5.4.5.5 Method F: Preparation of Wittig Salt 5.24



To a flame dried round bottom flask equipped with a stir bar and reflux condenser was added 3-bromopropanol **5.23** (1.0 equiv) and PhMe (1.1 M). Then PPh<sub>3</sub> (1.0 equiv) was added, the reaction mixture was heated to reflux and allowed to stir until white precipitate formed (~15–24 h). The flask was cooled to rt, the precipitate was filtered and washed with hexanes. The white solids were collected and dried under vacuum overnight.

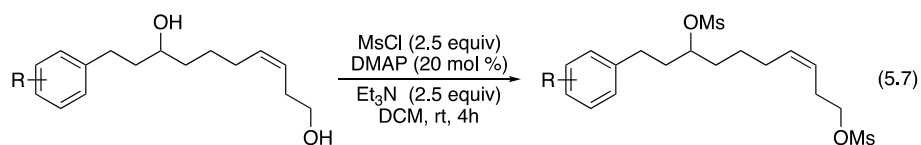
<sup>39</sup> Taber, D. F.; Guo, P. *J. Org. Chem.* **2008**, 73, 9479–9481.

#### 5.4.5.6 Method G: Wittig Reaction



This method was adapted from a procedure reported by Taber.<sup>39</sup> To a flame dried round bottom flask equipped with stir bar was added the Wittig salt **5.24** (1.01 equiv) and THF (0.2 M). The flask is cooled to 0 °C and *n*-BuLi (3.2 equiv, 2.5 M in hexanes) was added dropwise. The dark red solution was allowed to stir at 0 °C for 1 h. Then, a solution of lactol (1.0 equiv) in THF (1.0 M) was added and the reaction mixture was warmed to rt. The resulting orange solution was allowed to stir at rt for 24 h. To quench, saturated aq. NH<sub>4</sub>Cl was added dropwise. The biphasic mixture was transferred to a separatory funnel. The organic layer was extracted with EtOAc (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo.

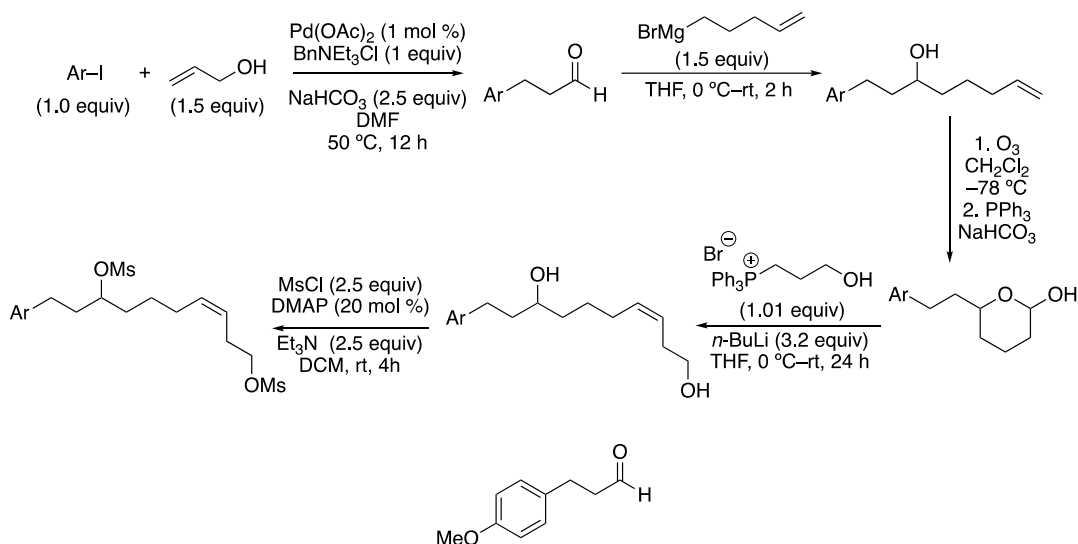
#### 5.4.5.7 Method H: Mesylation Reaction



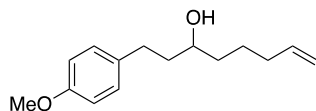
A flame dried round bottom flask equipped with a stir bar was charged with diol (1.0 equiv) and CH<sub>2</sub>Cl<sub>2</sub> (0.20 M) under N<sub>2</sub>. Then, Et<sub>3</sub>N (1.5 equiv), DMAP (20 mol %), and MsCl (2.5 equiv) were added in that order. The reaction mixture was then stirred at rt for at least 4 h. Once complete, sat. NaHCO<sub>3</sub> was added and the reaction mixture was extracted with CH<sub>2</sub>Cl<sub>2</sub> (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated in vacuo.

#### 5.4.6 Synthesis and Characterization Data for Dimesylate Starting Materials

**Scheme 5.5** General Synthesis of Dimesylate Starting Material



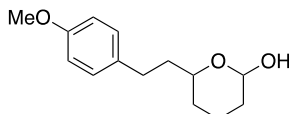
**3-(4-Methoxyphenyl)propanal (5.25)** was prepared according to Method B. The following amounts of reagents were used: 4-iodoanisole (4.9 g, 20. mmol, 1.0 equiv), allyl alcohol (2.0 mL, 30. mmol, 1.5 equiv), Pd(OAc)<sub>2</sub> (45 mg, 0.20 mmol, 1.0 mol %), BnEt<sub>3</sub>NCl (4.6 g, 20. mmol, 1.0 equiv), NaHCO<sub>3</sub> (4.3 g, 50. mmol, 2.5 equiv), and DMF (80 mL). Purification by column chromatography (0–10% EtOAc/hexanes) afforded the title compound as a clear and colorless oil (2.6 g, 16 mmol, 81% yield). Analytical data is consistent with literature values.<sup>40</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.75 (t, *J* = 1.5 Hz, 1H), 7.07 (d, *J* = 8.9 Hz, 2H), 6.81 (d, *J* = 8.7 Hz, 2H), 3.74 (s, 3H), 2.86 (t, *J* = 7.6 Hz, 2H), 2.73 – 2.65 (m, 2H).



**1-(4-Methoxyphenyl)oct-7-en-3-ol (5.26)** was prepared according to Method D. The following amount of reagents were used: aldehyde **5.25** (1.4 g, 8.6 mmol, 1.0 equiv), Grignard reagent **5.22** (10. mL, 13 mmol, 1.5 equiv, 1.3 M in Et<sub>2</sub>O) and Et<sub>2</sub>O (43 mL, 0.20 M). Purification by column chromatography (0–30% EtOAc/hexanes) afforded the title compound as a pale yellow oil (1.9 g,

<sup>40</sup> C. C. Frost, B. C. Hartley, *Org. Lett.* **2007**, *9*, 4259–4261.

8.1 mmol, 94% yield). Analytical data consistent with literature values.<sup>39</sup> **TLC**  $R_f$  = 0.3 (20% EtOAc/hexanes, KMnO<sub>4</sub> stain); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.15–7.10 (m, 2H), 6.87–6.81 (m, 2H), 5.81 (ddt,  $J$  = 16.9, 10.1, 6.6 Hz, 1H), 5.02 (dq,  $J$  = 17.1, 1.7 Hz, 1H), 4.97 (dq,  $J$  = 10.2, 1.7 Hz, 1H), 3.79 (s, 3H), 3.68–3.57 (m, 1H), 2.68 (dddd,  $J$  = 46.8, 13.8, 9.5, 6.3 Hz, 2H), 2.12–2.04 (m, 2H), 1.82–1.66 (m, 2H), 1.60–1.39 (m, 4H).



**6-(4-Methoxyphenethyl)tetrahydro-2H-pyran-2-ol (5.27)** was prepared according to Method E.

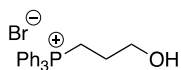
The following amounts of reagents were used: alcohol **5.26** (0.42 g, 1.8 mmol, 1.0 equiv), ozone (excess), CH<sub>2</sub>Cl<sub>2</sub> (18 mL), PPh<sub>3</sub> (0.85 g, 2.0 g/1g of alcohol), and NaHCO<sub>3</sub> (0.85 g, 2.0 g/1g of alcohol). Purification by column chromatography (0–50% EtOAc/hexanes) afforded the title compound as a white solid (0.19 g, 0.83 mmol, 46% yield, 1.5:1 mixtures of diastereomers). For clarity, the <sup>1</sup>H and <sup>13</sup>C of the major and minor diastereomers have been tabulated individually. The analytical data is consistent with literature values.<sup>39</sup>

**TLC**  $R_f$  = 0.3 (20% EtOAc/hexanes, CAM stain); **HRMS** (TOF MS ES+)  $m/z$ : [M+Na]<sup>+</sup> calcd for C<sub>14</sub>H<sub>20</sub>O<sub>3</sub>Na, 259.1310; found 259.1309.

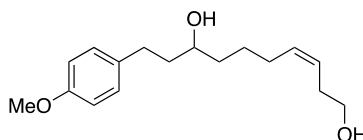
**Major diastereomer:** **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.09 (d,  $J$  = 8.5 Hz, 2H), 6.82 (d,  $J$  = 8.4 Hz, 2H), 4.69 (t,  $J$  = 7.7 Hz, 1H), 3.78 (s, 3H), 3.46–3.34 (m, 1H), 3.19 (s, 1H), 2.65–2.51 (m, 2H), 1.92–1.79 (m, 2H), 1.79–1.55 (m, 3H), 1.54–1.43 (m, 1H), 1.38–1.15 (m, 2H); **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  157.8, 134.3, 129.4 (2C), 113.9 (2C), 96.6, 75.6, 55.4, 38.0, 33.1, 30.9, 30.6, 22.2.

**Minor Diastereomer:** **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.09 (d,  $J$  = 8.5 Hz, 2H), 6.82 (d,  $J$  = 8.4 Hz, 2H), 5.33 (s, 1H), 3.98–3.90 (m, 1H), 3.78 (s, 3H), 3.19 (s, 1H), 3.14 (d,  $J$  = 6.2 Hz, 1H), 2.79–2.66 (m, 1H), 1.92–1.79 (m, 2H), 1.79–1.55 (m, 3H), 1.56–1.42 (m, 1H), 1.38–1.15 (m, 2H); **<sup>13</sup>C**

**NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  157.8, 134.5, 129.4 (2C), 113.9 (2C), 92.1, 68.1, 55.4, 38.2, 31.3, 30.9, 30.0, 17.6.



**(3-Hydroxypropyl)triphenylphosphonium bromide (5.24)** was prepared according to Method F. The following amounts of reagents were used: 4-bromoproanol **5.23** (0.89 mL, 10. mmol, 1.0 equiv), PPh<sub>3</sub> (2.6 g, 10. mmol, 1.0 equiv), and PhMe (9.1 mL). Analytical data is consistent with literature data.<sup>41</sup> **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.84–7.75 (m, 9H), 7.74–7.67 (m, 6H), 4.93 (s, 1H) 3.94–3.62 (m, 4H), 1.95–1.74 (m, 2H).



**(4-Methoxyphenyl)dec-3-ene-1,8-diol (5.28)** was prepared according to Method G. The following amounts of reagents were used: lactol **5.27** (0.19, 0.83 mmol, 1.0 equiv), Wittig salt **5.24** (0.34 g, 0.84 mmol, 1.0 equiv), *n*-BuLi (1.1 mL, 2.7 mmol, 3.2 equiv, 2.5 M in hexane) and THF (4.2 mL). Purification by column chromatography (0–70% EtOAc/hexanes) afforded the title compound as a yellow oil (0.16 g, 0.57 mmol, 69% yield, 2:1 mixture of diastereomers). For clarity, the <sup>1</sup>H and <sup>13</sup>C of the major and minor diastereomers have been tabulated individually.

**TLC** R<sub>f</sub> = 0.2 (20% EtOAc/hexanes, KMnO<sub>4</sub> stain); **HRMS** (TOF MS ES+) *m/z*: [M+Na]<sup>+</sup> calcd for C<sub>17</sub>H<sub>26</sub>O<sub>3</sub>Na, 301.1780; found 301.1788.

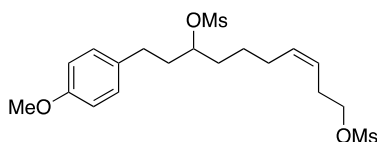
**Major diastereomer:** **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.18 (d, *J* = 8.4 Hz, 2H), 6.89 (d, *J* = 8.5 Hz, 2H), 5.62 (dt, *J* = 10.7, 7.5 Hz, 1H), 5.45 (dtt, *J* = 10.5, 7.3, 1.5 Hz, 1H), 3.85 (s, 3H), 3.70 (t, *J* = 6.5 Hz, 2H), 2.79 (ddd, *J* = 14.8, 9.5, 5.8 Hz, 1H), 2.68 (ddd, *J* = 13.9, 9.4, 6.9 Hz, 1H), 2.37

<sup>41</sup> Kandula, S. R. V.; Kumar, P. *Tetrahedron Asymmetry* **2005**, *16*, 3268–3274.



(q,  $J = 7.0$  Hz, 2H), 2.14 (tquint,  $J = 13.9, 7.5$  Hz, 2H), 1.89–1.71 (m, 2H), 1.66 (br s, 5H), 1.61–1.41 (m, 2H);  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  157.9, 134.3, 133.0, 129.4 (2C), 125.6, 114.0 (2C), 71.3, 62.4, 55.4, 39.5, 37.1, 31.3, 30.9, 27.4, 25.8.

**Minor Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.18 (d,  $J = 8.5$  Hz, 2H), 6.89 (d,  $J = 8.6$  Hz, 2H), 5.60 (dt,  $J = 15.3, 6.7$  Hz, 1H), 5.45 (dt,  $J = 15.3, 6.9$  Hz, 1H), 3.85 (s, 3H), 3.69 (t,  $J = 6.3$  Hz, 2H), 2.79 (ddd,  $J = 14.9, 9.5, 5.8$  Hz, 1H), 2.68 (ddd,  $J = 13.9, 9.4, 6.8$  Hz, 1H), 2.33 (q,  $J = 6.4$  Hz, 2H), 2.10 (d,  $J = 7.1$  Hz, 2H), 1.91–1.70 (m, 2H), 1.67–1.39 (m, 5H), 1.03–0.80 (m, 2H);  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  157.9, 134.3, 133.9, 129.4 (2C), 126.4, 114.0 (2C), 71.3, 62.2, 55.4, 39.5, 37.2, 36.1, 32.7, 31.3, 25.5.

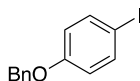


**(4-Methoxyphenyl)dec-3-ene-1,8-diyl dimethanesulfonate (5.1)** was prepared according to Method H. The following amounts of reagents were used: diol **5.28** (0.59 g, 2.1 mmol, 1.0 equiv),  $\text{MsCl}$  (0.41 mL, 5.3 mmol, 2.5 equiv),  $\text{Et}_3\text{N}$  (0.73 mL, 5.3 mmol, 2.5 equiv), DMAP (51 mg, 0.42 mmol, 20 mol %), and  $\text{CH}_2\text{Cl}_2$  (11 mL). Purification by column chromatography (0–50% EtOAc/hexanes) afforded the title compound as a yellow oil (0.76 g, 1.7 mmol, 83% yield, 2:1 dr). For clarity, the  $^1\text{H}$  and  $^{13}\text{C}$  of the major and minor diastereomers have been tabulated individually. **TLC**  $R_f = 0.4$  (50% EtOAc/hexanes, CAM stain); **HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{19}\text{H}_{30}\text{O}_7\text{S}_2\text{Na}$ , 457.1331; found 457.1314.

**Major Diastereomer:**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.11 (d,  $J = 8.5$  Hz, 2H), 6.83 (d,  $J = 8.7$  Hz, 2H), 5.54 (dt,  $J = 10.7, 7.4$  Hz, 1H), 5.38 (dt,  $J = 10.8, 7.2$  Hz, 1H), 4.75 (quint,  $J = 6.0$  Hz, 1H), 4.20 (t,  $J = 6.8$  Hz, 2H), 3.78 (s, 3H), 2.99 (d,  $J = 1.8$  Hz, 6H), 2.76–2.58 (m, 2H), 2.49 (q,  $J = 6.8$  Hz, 2H), 2.18–1.88 (m, 4H), 1.75 (dtd,  $J = 9.4, 6.2, 3.9$  Hz, 2H), 1.57–1.39 (m, 2H);  $^{13}\text{C}$

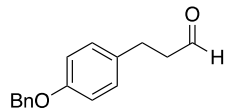
**NMR** (101 MHz, CDCl<sub>3</sub>)  $\delta$  158.2, 133.2, 133.0, 129.5 (2C), 124.0, 114.2 (2C), 83.1, 69.4, 55.5, 39.0, 37.7, 36.6, 34.2, 30.6, 27.6, 27.1, 24.9.

**Minor Diastereomer:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.12 (d,  $J$  = 8.6 Hz, 2H), 6.85 (d,  $J$  = 8.7 Hz, 2H), 5.55 (dt,  $J$  = 15.4, 6.7 Hz, 1H), 5.40 (dt,  $J$  = 15.3, 6.6 Hz, 1H), 4.75 (quint,  $J$  = 6.1 Hz, 1H), 4.22 (t,  $J$  = 6.7 Hz, 2H), 3.79 (s, 3H), 3.00 (d,  $J$  = 1.1 Hz, 6H), 2.76–2.59 (m, 2H), 2.45 (q,  $J$  = 6.7 Hz, 2H), 2.10–1.94 (m, 3H), 1.81–1.66 (m, 2H), 1.49 (dt,  $J$  = 15.7, 7.4 Hz, 1H), 0.96 – 0.79 (m, 2H); <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  158.1, 133.8, 132.9, 129.4 (2C), 124.7, 114.1 (2C), 83.2, 69.6, 55.4, 38.9, 37.6, 36.5, 34.0, 32.5, 32.2, 30.5, 24.5.

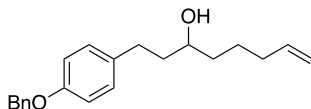


**1-(Benzyloxy)-4-iodobenzene (5.29)** was prepared according to a procedure reported by Fukuyama.<sup>42</sup> To a round bottom flask equipped with a stir bar was added 4-iodophenol (1.1 g, 5.0 mmol, 1.0 equiv), K<sub>2</sub>CO<sub>3</sub> (0.89 g, 6.5 mmol, 1.3 equiv), benzyl bromide (0.65 mL, 5.5 mmol, 1.1 equiv), and DMF (2.5 mL). The reaction flask was fitted with a reflux condenser, and the mixture was heated to 60 °C and allowed to stir overnight. To quench, H<sub>2</sub>O was added at rt and the solution was transferred to a separatory funnel. The aqueous layer was extracted with Et<sub>2</sub>O (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo. Purification by column chromatography (0–10% EtOAc/hexanes) afforded the title compound as a white solid (1.2 g, 3.7 mmol, 74% yield). Analytical data is consistent with literature data.<sup>42</sup> **TLC** R<sub>f</sub> = 0.7 (10% EtOAc/hexanes); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.65 (d,  $J$  = 9.0 Hz, 2H), 7.56–7.47 (m, 4H), 7.47–7.40 (m, 1H), 6.85 (d,  $J$  = 8.9 Hz, 2H), 5.12 (s, 2H).

<sup>42</sup> W. Kurosawa, T. Kan, T. Fukuyama, *J. Am. Chem. Soc.* **2003**, *125*, 8112–8113.

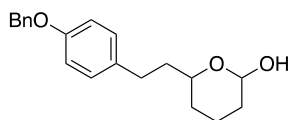


**3-(4-(Benzyloxy)phenyl)propanal (5.30)** was prepared according to Method B. The following amounts of reagents were used: iodide **5.29** (1.0 g, 3.2 mmol, 1.0 equiv), allyl alcohol (0.33 mL, 4.8 mmol, 1.5 equiv), Pd(OAc)<sub>2</sub> (7.0 mg, 32 μmol, 1.0 mol %), BnEt<sub>3</sub>NCl (0.73 g, 3.2 mmol, 1 equiv), NaHCO<sub>3</sub> (0.67 g, 8.0 mmol, 2.5 equiv), and DMF (13 mL). Purification by column chromatography (0–10% EtOAc/hexanes) afforded the title compound as a white solid (0.74 g, 3.1 mmol, 97% yield). **TLC** R<sub>f</sub> = 0.3 (10% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 9.82 (t, *J* = 1.5 Hz, 1H), 7.54–7.34 (m, 5H), 7.24–7.11 (m, 2H), 7.02–6.95 (m, 2H), 5.09 (s, 2H), 2.95 (t, *J* = 7.5 Hz, 2H), 2.75 (t, *J* = 7.7 Hz, 2H); **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 201.5, 157.3, 132.7, 129.2 (2C), 128.5 (2C), 127.9, 127.4 (2C), 114.9 (2C), 70.0,



**1-(4-(Benzyloxy)phenyl)oct-7-en-3-ol (5.31)** was prepared according to Method D. The following amounts of reagents were used: aldehyde **5.30** (0.87 g, 3.6 mmol, 1.0 equiv), Grignard reagent **5.22** (5.0 mL, 1.5 mmol, 1.5 equiv, 1.1 M in THF), and THF (19 mL). The crude reaction mixture was filtered through a pad of silica gel eluting with 100% EtOAc to remove excess magnesium salts. The title compound was afforded as a white solid (1.1 g, 3.5 mmol, 97 % yield). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.47 (d, *J* = 7.3 Hz, 2H), 7.42 (t, *J* = 7.1 Hz, 2H), 7.36 (t, *J* = 7.3 Hz, 1H), 7.16 (d, *J* = 8.3 Hz, 2H), 6.95 (d, *J* = 8.5 Hz, 2H), 5.86 (ddtd, *J* = 16.9, 10.2, 6.7, 1.5 Hz, 1H), 5.11–4.97 (m, 4H), 3.73–3.59 (m, 1H), 2.83–2.72 (m, 1H), 2.66 (ddd, *J* = 14.0, 9.4, 6.8 Hz, 1H), 2.18–2.03 (m, 2H), 1.85–1.66 (m, 3H), 1.64–1.40 (m, 3H); **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 157.1, 138.7, 137.3, 134.6, 129.4 (2C), 128.6 (2C), 127.9, 127.5 (2C), 122.7, 114.9 (2C), 71.2,

70.1, 39.3, 37.0, 33.8, 31.2, 24.9; **HRMS** (TOF MS ES+)  $m/z$ :  $[M+Na]^+$  calcd for  $C_{21}H_{26}O_2Na$ , 333.1830; found 333.1828.

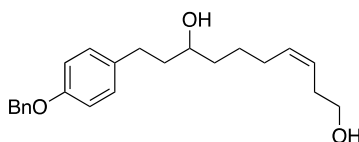


**6-(4-(Benzyloxy)phenethyl)tetrahydro-2H-pyran-2-ol (5.32)** was prepared according to a modified version of Method E. The following amounts of reagents were used: alcohol **5.31** (1.1 g, 3.5 mmol, 1.0 equiv), ozone (excess),  $CH_2Cl_2$  (3.5 mL), MeOH (7.0 mL),  $PPh_3$  (2.2 g, 2.0 g/1.0 mmol of alcohol) and  $NaHCO_3$  (2.2 g, 2.0 g/1.0 g of alcohol). Purification by column chromatography (0–30% EtOAc/hexanes) afforded the title compound as a white solid (0.36 g, 1.2 mmol, 33% yield, 2:1 dr). For clarity, the  $^1H$  and  $^{13}C$  of the major and minor diastereomers have been tabulated individually.

**TLC**  $R_f$  = 0.3 (50% EtOAc/hexanes); **HRMS** (TOF MS ES+)  $m/z$ :  $[M+Na]^+$  calcd for  $C_{20}H_{24}O_3Na$ , 335.1623; found 335.1610.

**Major Diastereomer:**  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.49–7.32 (m, 5H), 7.13 (d,  $J$  = 8.4 Hz, 2H), 6.93 (d,  $J$  = 8.0 Hz, 2H), 5.40–5.32 (m, 1H) 5.06 (s, 2H), 4.73 (d,  $J$  = 9.5 Hz, 1H), 3.44 (dtd,  $J$  = 10.4, 5.3, 2.6 Hz, 1H), 2.83–2.54 (m, 2H), 1.98–1.82 (m, 2H), 1.82–1.58 (m, 2H), 1.58–1.44 (m, 2H), 1.42–1.18 (m, 2H);  $^{13}C$  NMR (101 MHz,  $CDCl_3$ )  $\delta$  157.1, 137.3, 134.6, 129.4 (2C), 128.6 (2C), 127.9, 127.5 (2C), 114.8 (2C), 96.6, 75.6, 70.1, 37.8, 33.0, 30.9, 30.5, 22.2.

**Minor Diastereomer:**  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.49–7.32 (m, 5H), 7.13 (d,  $J$  = 8.4 Hz, 2H), 6.93 (d,  $J$  = 8.0 Hz, 2H), 5.32–5.13 (m, 1H), 5.06 (s, 2H), 4.73 (d,  $J$  = 9.5 Hz, 1H), 4.08–3.92 (m, 1H), 2.83–2.54 (m, 2H), 1.98–1.82 (m, 2H), 1.82–1.58 (m, 2H), 1.58–1.44 (m, 2H), 1.42–1.18 (m, 2H);  $^{13}C$  NMR (101 MHz,  $CDCl_3$ )  $\delta$  157.1, 137.3, 134.7, 129.4 (2C), 128.6 (2C), 127.9, 127.5 (2C), 114.8 (2C), 91.9, 75.6, 68.2, 38.1, 33.0, 31.2, 30.0, 17.5.



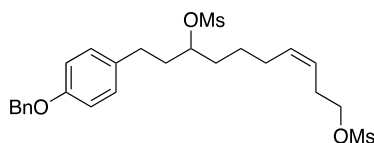
**(4-Benzyloxy)phenyl-dec-3-ene-1,8-diol (5.33)** was prepared according to Method G. The following amounts of reagents were used: lactol **5.32** (56 mg, 0.18 mmol, 1.0 equiv), Wittig salt **5.24** (73 mg, 0.18 mmol, 1.0 equiv), *n*-BuLi (0.30 mL, 0.58 mmol, 3.2 equiv, 2.5 M in hexanes), and THF (0.90 mL). Purification by column chromatography (0–50% yield) afforded the title compound as a white solid (27 mg, 80.  $\mu$ mol, 42% yield). For clarity, the  $^1\text{H}$  and  $^{13}\text{C}$  of the major and minor diastereomers have been tabulated individually.

**TLC**  $R_f$  = 0.2 (50% EtOAc/hexanes,  $\text{KMnO}_4$  stain); **HRMS** (TOF MS ES+)  $m/z$ : calcd for  $\text{C}_{23}\text{H}_{30}\text{O}_3\text{Na}$ , 377.2093; found 377.3075

**Major Diastereomer:**  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ )  $\delta$  7.49 (d,  $J$  = 7.0 Hz, 2H), 7.44 (dd,  $J$  = 8.5, 6.7 Hz, 2H), 7.38 (tt,  $J$  = 7.3, 1.4 Hz, 1H), 7.17 (d,  $J$  = 8.4 Hz, 2H), 6.96 (d,  $J$  = 8.6 Hz, 2H), 5.64–5.57 (m, 1H), 5.49–5.41 (m, 1H), 5.10 (s, 2H), 3.73–3.64 (m, 3H), 2.79 (ddd,  $J$  = 14.8, 9.5, 5.7 Hz, 1H), 2.67 (ddd,  $J$  = 13.9, 9.5, 6.8 Hz, 1H), 2.37 (qt,  $J$  = 6.8, 1.9 Hz, 1H), 2.21–2.05 (m, 2H), 1.87–1.70 (m, 2H), 1.62–1.40 (m, 7H);  $^{13}\text{C}$  NMR (151 MHz,  $\text{CDCl}_3$ )  $\delta$  157.2, 137.3, 134.6, 133.1, 129.4 (2C), 128.7 (2C), 128.0, 127.6 (2C), 125.7, 115.0 (2C), 71.3, 70.2, 62.4, 39.4, 37.2, 31.3, 30.9, 27.4, 25.8.

**Minor Diastereomer:**  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ )  $\delta$  7.49 (d,  $J$  = 7.0 Hz, 2H), 7.44 (dd,  $J$  = 8.5, 6.7 Hz, 2H), 7.38 (tt,  $J$  = 7.3, 1.4 Hz, 1H), 7.17 (d,  $J$  = 8.4 Hz, 2H), 6.96 (d,  $J$  = 8.6 Hz, 2H), 5.64–5.57 (m, 1H), 5.49–5.41 (m, 1H), 5.10 (s, 2H), 3.73–3.64 (m, 3H), 2.79 (ddd,  $J$  = 14.8, 9.5, 5.7 Hz, 1H), 2.67 (ddd,  $J$  = 13.9, 9.5, 6.8 Hz, 1H), 2.32 (qd,  $J$  = 6.3, 1.2 Hz, 1H), 2.21–2.05 (m, 2H), 1.87–1.70 (m, 2H), 1.62–1.40 (m, 7H);  $^{13}\text{C}$  NMR (151 MHz,  $\text{CDCl}_3$ )  $\delta$  157.2, 137.3, 134.6, 133.9, 129.4

(2C), 128.7 (2C), 128.0, 127.6 (2C), 126.4, 115.0 (2C), 71.3, 70.2, 62.2, 39.4, 37.2, 36.1, 32.7, 27.4, 25.5.



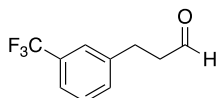
**(4-(Benzyloxy)phenyl)dec-3-ene-1,8-diyl dimethanesulfonate (5.17)** was prepared according to Method H. The following amounts of reagents were used: diol **5.33** (27 mg, 80.  $\mu\text{mol}$ , 1.0 equiv),  $\text{MsCl}$  (20.  $\mu\text{L}$ , 0.20 mmol, 2.5 equiv),  $\text{Et}_3\text{N}$  (30.  $\mu\text{L}$ , 0.20 mmol, 2.5 equiv), and  $\text{CH}_2\text{Cl}_2$  (0.50 mL). The crude reaction mixture was filtered through a pad a silica gel eluting with 100%  $\text{CH}_2\text{Cl}_2$  and the title compound was isolated as a pale yellow oil (35 mg, 68  $\mu\text{mol}$ , 85% yield). For clarity, the  $^1\text{H}$  and  $^{13}\text{C}$  of the major and minor diastereomers have been tabulated individually.

**TLC**  $R_f$  = 0.3 (30%  $\text{EtOAc}$ /hexanes); **HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{25}\text{H}_{34}\text{O}_7\text{S}_2\text{Na}$ , 533.1644; found 533.1658.

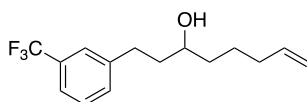
**Major Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.49 (d,  $J$  = 7.3 Hz, 2H), 7.44 (t,  $J$  = 7.5 Hz, 2H), 7.38 (dd,  $J$  = 8.5, 5.9 Hz, 1H), 7.17 (d,  $J$  = 8.4 Hz, 2H), 6.97 (d,  $J$  = 8.5 Hz, 2H), 5.60 (q,  $J$  = 8.5, 7.4 Hz, 1H), 5.51 – 5.40 (m, 1H), 5.10 (d,  $J$  = 3.3 Hz, 2H), 4.81 (quint,  $J$  = 6.0 Hz, 1H), 4.30–4.24 (m, 2H), 3.72 (d,  $J$  = 1.0 Hz, 2H), 3.05 (s, 6H), 2.73 (qdd,  $J$  = 14.3, 9.6, 6.3 Hz, 2H), 2.55 (q,  $J$  = 7.0 Hz, 2H), 2.22–1.94 (m, 2H), 1.90–1.71 (m, 2H), 1.61–1.45 (m, 2H);  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  157.3, 137.2, 133.2, 133.1, 129.4 (2C), 128.7 (2C), 128.0, 127.6 (2C), 123.9, 115.0 (2C), 83.0, 70.1, 69.3, 38.8, 37.6, 36.4, 34.1, 31.7, 27.5, 27.0, 24.8.

**Minor Diastereomer:**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.49 (d,  $J$  = 7.3 Hz, 2H), 7.44 (t,  $J$  = 7.5 Hz, 2H), 7.38 (dd,  $J$  = 8.5, 5.9 Hz, 1H), 7.17 (d,  $J$  = 8.4 Hz, 2H), 6.97 (d,  $J$  = 8.5 Hz, 2H), 5.60 (q,  $J$  = 8.5, 7.4 Hz, 1H), 5.51–5.40 (m, 1H), 5.10 (d,  $J$  = 3.3 Hz, 2H), 4.81 (p,  $J$  = 6.0 Hz, 1H), 4.30–4.24 (m, 2H), 3.19 (d,  $J$  = 1.0 Hz, 1H), 3.05 (s, 6H), 2.73 (qdd,  $J$  = 14.3, 9.6, 6.3 Hz, 2H), 2.50 (q,  $J$  =

7.0 Hz, 2H), 2.22–1.94 (m, 2H), 1.90–1.71 (m, 2H), 1.61–1.45 (m, 2H);  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  157.3, 133.8, 133.2, 133.1, 129.4 (2C), 128.7 (2C), 128.0, 127.6 (2C), 124.7, 115.0 (2C), 83.1, 70.1, 69.6, 38.8, 34.0, 32.5, 32.2, 31.7, 27.5, 27.0, 24.5.

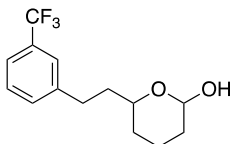


**3-(3-(Trifluoromethyl)phenyl)propanal (5.34)** was prepared according to Method B. The following amounts of reagents were used: 1-iodo-3-(trifluoromethyl)benzene (0.72 mL, 5.0 mmol, 1.0 equiv), allyl alcohol (0.51 mL, 7.5 mmol, 1.5 equiv),  $\text{Pd}(\text{OAc})_2$  (11 mg, 5.0  $\mu\text{mol}$ , 1.0 mol %),  $\text{BnEt}_3\text{NCl}$  (1.1 g, 5.0 mmol, 1.0 equiv),  $\text{NaHCO}_3$  (1.1 g, 13 mmol, 2.5 equiv), and DMF (20. mL). **TLC**  $R_f$  = 0.2 (10% EtOAc/hexanes, CAM stain);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  9.82 (s, 1H), 7.49–7.44 (m, 2H), 7.43–7.37 (m, 2H), 3.01 (t,  $J$  = 7.6 Hz, 2H), 2.82 (t,  $J$  = 7.6 Hz, 2H);  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  200.8, 141.5, 131.9 (q,  $J$  = 1.4 Hz), 131.0 (q,  $J$  = 32.4 Hz), 129.2, 125.2 (q,  $J$  = 3.7 Hz), 124.3 (q,  $J$  = 272.4 Hz), 123.3 (q,  $J$  = 3.7 Hz), 45.1, 27.9;  $^{19}\text{F}$  NMR (565 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.7 (3F); **HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{10}\text{H}_9\text{F}_3\text{Na}$ , 202.0605; found 202.0615.



**1-(3-(Trifluoromethyl)phenyl)oct-7-en-3-ol (5.35)** was prepared according to Method D. The following amounts of reagents were used: aldehyde **5.34** (0.43 g, 2.1 mmol, 1.0 equiv), Grignard reagent **5.22** (6.0 mL, 3.2 mmol, 1.5 equiv, 0.50 M in  $\text{Et}_2\text{O}$ ), and  $\text{Et}_2\text{O}$  (11 mL). Purification by column chromatography (0–20% EtOAc/hexanes) afforded the title compound as a yellow oil (0.44 g, 1.6 mmol, 75% yield). **TLC**  $R_f$  = 0.6 (20% EtOAc/hexanes,  $\text{KMnO}_4$  stain);  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ )  $\delta$  7.47–7.42 (m, 2H), 7.38 (d,  $J$  = 4.8 Hz, 2H), 5.79 (ddt,  $J$  = 16.9, 10.2, 6.7 Hz, 1H), 5.04–4.92 (m, 2H), 3.62 (tt,  $J$  = 7.9, 4.2 Hz, 1H), 2.86 (ddd,  $J$  = 15.0, 10.1, 5.5 Hz, 1H), 2.72 (ddd,

$J = 13.8, 10.0, 6.6$  Hz, 1H), 2.19–2.00 (m, 2H), 1.85–1.66 (m, 3H), 1.62–1.37 (m, 3H);  $^{13}\text{C}$  NMR (151 MHz,  $\text{CDCl}_3$ )  $\delta$  143.2, 138.6, 132.0 (q,  $J = 1.2, 0.7$  Hz), 130.8 (q,  $J = 31.8$  Hz), 128.9, 124.4 (q,  $J = 272.1$  Hz), 125.17 (q,  $J = 3.8$  Hz), 122.79 (q,  $J = 3.8$  Hz), 114.8, 71.1, 40.0, 37.1, 33.8, 32.0, 25.0;  $^{19}\text{F}$  NMR (565 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.6 (3F); HRMS (TOF MS CI+)  $m/z$ :  $[\text{M}+\text{NH}_4]^+$  calcd for  $\text{C}_{15}\text{H}_{19}\text{F}_3\text{ONH}_4$  290.1732, found 290.1728.



**6-(3-(Trifluoromethyl)phenethyl)tetrahydro-2H-pyran-2-ol (5.36)** was prepared according to Method E. The following amount of reagents were used: alkene **5.35** (0.72 g, 2.6 mmol, 1.0 equiv), ozone (excess),  $\text{CH}_2\text{Cl}_2$  (20 mL),  $\text{PPh}_3$  (1.7 g, 2.0 g/1.0 g of alcohol),  $\text{NaHCO}_3$  (1.7 g, 2.0 g/1.0 g of alcohol). Purification by column chromatography (0–30% EtOAc/hexanes) to afford the title compound as a white solid (0.47 g, 1.7 mmol, 65% yield, 2:1 dr). For clarity, the  $^1\text{H}$  and  $^{13}\text{C}$  of the major and minor diastereomers have been tabulated individually.

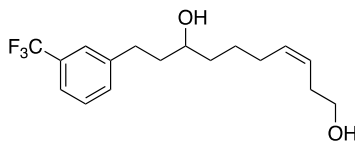
**TLC**  $R_f = 0.2$  (20% EtOAc/hexanes, CAM Stain); **HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{14}\text{H}_{17}\text{F}_3\text{O}_2\text{Na}$ , 297.1078; found 297.1077.

**Major Diastereomer:**  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ )  $\delta$  7.46–7.41 (m, 2H), 7.40–7.33 (m, 2H), 4.70 (ddd,  $J = 9.0, 6.4, 2.1$  Hz, 1H), 3.56 (br s, 1H), 3.41 (dddd,  $J = 10.5, 8.1, 4.4, 2.0$  Hz, 1H), 2.91–2.63 (m, 2H), 1.98–1.42 (m, 6H), 1.41–1.15 (m, 2H);  $^{13}\text{C}$  NMR (151 MHz,  $\text{CDCl}_3$ )  $\delta$  143.1, 132.0 (q,  $J = 1.1$  Hz), 130.72 (q,  $J = 32.1$  Hz), 128.9, 125.3 (q,  $J = 3.8$  Hz), 124.4 (q,  $J = 272.6$  Hz), 122.78 (q,  $J = 3.9$  Hz), 96.7, 75.5, 37.5, 33.0, 31.6, 30.5, 22.1;  $^{19}\text{F}$  NMR (565 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.6 (3F).

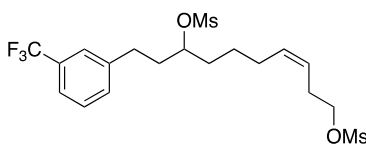
**Minor Diastereomer:**  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ )  $\delta$  7.46–7.41 (m, 2H), 7.40–7.33 (m, 2H), 5.34 (s, 1H), 4.02–3.90 (m, 1H), 3.56 (br s, 1H), 2.91–2.63 (m, 2H), 1.98–1.42 (m, 6H), 1.41–1.15 (m,



2H);  $^{13}\text{C}$  NMR (151 MHz,  $\text{CDCl}_3$ )  $\delta$  143.3, 131.9 (q,  $J = 1.1$  Hz), 130.69 (q,  $J = 31.5$  Hz), 128.8, 125.3 (q,  $J = 3.3$  Hz), 124.4 (q,  $J = 272.6$  Hz), 122.75 (q,  $J = 3.9$  Hz), 92.0, 68.0, 37.8, 31.2 30.0, 22.1, 17.5;  $^{19}\text{F}$  NMR (565 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.6 (3F).



**(3-(Trifluoromethyl)phenyl)dec-3-ene-1,8-diol (5.37)** was prepared according to Method G. The following amounts of reagents were used: lactol **5.36** (0.47 g, 1.7 mmol, 1.0 equiv), Wittig salt **5.24** (0.69 g, 1.7 mmol, 1.0 equiv), *n*-BuLi (2.2 mL, 5.4 mmol, 3.2 equiv, 2.5 M in hexanes), and THF (8.5 mL). Purification by column chromatography (0–5% MeOH/ $\text{CH}_2\text{Cl}_2$ ) afforded a mixture of the title compound and triphenylphosphine oxide. This mixture was carried onto the next step without further purification.



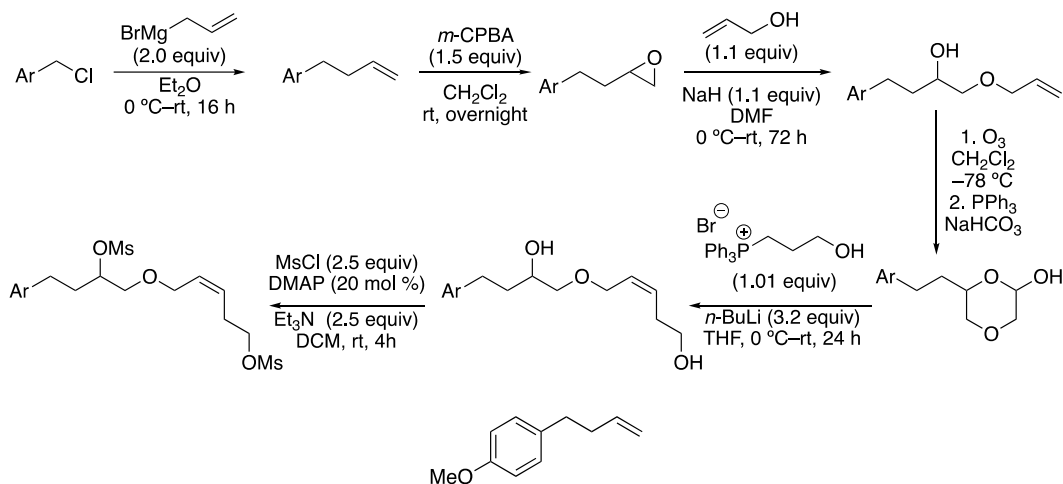
**(3-(Trifluoromethyl)phenyl)dec-3-ene-1,8-diyl dimethanesulfonate (5.18)** was prepared according to Method H. The following amounts of reagents were used: diol **5.37** (0.34 g, 1.1 mmol, 1.0 equiv), methanesulfonyl chloride (0.21 mL, 2.7 mmol, 2.5 equiv), DMAP (26 mg, 0.22 mmol, 20. mol %),  $\text{Et}_3\text{N}$  (0.38 mL, 2.7 mmol, 2.5 equiv), and  $\text{CH}_2\text{Cl}_2$  (5.4 mL). Purification by column chromatography (0–30% EtOAc/hexanes) afforded the title compound as a pale yellow oil (0.40 g, 0.94 mmol, 78% yield, 2:1 cis:trans). For clarity, the  $^1\text{H}$  and  $^{13}\text{C}$  of the major and minor diastereomers have been tabulated individually.

**TLC**  $R_f = 0.6$  (50% EtOAc/hexanes, CAM Stain); **HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{19}\text{H}_{27}\text{F}_3\text{O}_6\text{S}_2\text{Na}$ , 495.1099; found 495.1080.

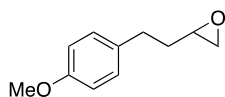
**Major Diastereomer:**  $^1\text{H NMR}$  (600 MHz,  $\text{CDCl}_3$ )  $\delta$  7.47 (dd,  $J = 6.6, 1.7$  Hz, 2H), 7.42–7.39 (m, 2H), 5.59–5.51 (m, 1H), 5.44–5.36 (m, 1H), 5.30 (s, 1H), 4.82–4.74 (m, 1H), 4.28–4.16 (m, 2H), 3.03 (s, 3H), 3.00 (s, 3H), 2.84 (ddd,  $J = 14.0, 10.2, 6.0$  Hz, 1H), 2.77 (ddd,  $J = 14.2, 10.1, 6.4$  Hz, 1H), 2.50 (q,  $J = 7.0$  Hz, 1H), 2.16–1.97 (m, 4H), 1.85–1.69 (m, 2H), 1.49 (dddd,  $J = 20.3, 17.1, 13.5, 6.7$  Hz, 2H);  $^{13}\text{C NMR}$  (151 MHz,  $\text{CDCl}_3$ )  $\delta$  141.9, 133.6, 132.9, 130.7 (q,  $J = 31.5$  Hz), 129.0, 125.0 (q,  $J = 3.9$  Hz), 124.8, 124.2 (q,  $J = 272.4$ ), 123.1 (q,  $J = 3.9$  Hz), 82.5, 69.3, 60.4, 38.7, 35.9 (2C), 34.0, 31.1, 26.9, 24.7;  $^{19}\text{F NMR}$  (565 MHz,  $\text{CDCl}_3$ )  $\delta$  –62.5 (3F).

**Minor Diastereomer:**  $^1\text{H NMR}$  (600 MHz,  $\text{CDCl}_3$ )  $\delta$  7.47 (dd,  $J = 6.6, 1.7$  Hz, 2H), 7.42–7.39 (m, 2H), 5.59–5.51 (m, 1H), 5.44–5.36 (m, 1H), 5.30 (s, 1H), 4.82–4.74 (m, 1H), 4.28–4.16 (m, 2H), 3.03 (s, 3H), 3.00 (s, 3H), 2.84 (ddd,  $J = 14.0, 10.2, 6.0$  Hz, 1H), 2.77 (ddd,  $J = 14.2, 10.1, 6.4$  Hz, 1H), 2.44 (q,  $J = 6.7$  Hz, 1H), 2.16–1.97 (m, 4H), 1.85–1.69 (m, 2H), 1.49 (dddd,  $J = 20.3, 17.1, 13.5, 6.7$  Hz, 2H);  $^{13}\text{C NMR}$  (151 MHz,  $\text{CDCl}_3$ )  $\delta$  141.9, 133.6, 131.9, 130.7 (q,  $J = 31.5$  Hz), 129.0, 125.0 (q,  $J = 3.9$  Hz), 124.8, 124.2 (q,  $J = 272.4$ ), 123.1 (q,  $J = 3.9$  Hz), 82.6, 69.6, 60.4, 37.4 (2C), 33.8, 32.4, 32.0, 27.4, 24.5;  $^{19}\text{F NMR}$  (565 MHz,  $\text{CDCl}_3$ )  $\delta$  –62.5 (3F).

**Scheme 5.6** Synthesis of Ether Containing Dimesylate Starting Material



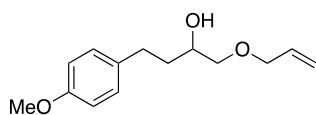
**1-(but-3-en-1-yl)-4-methoxybenzene (5.38)** was prepared according to a procedure reported by Taber.<sup>39</sup> To a flame dried round bottom flask equipped with a stir bar was added 4-methoxybenzyl chloride (1.4 mL, 10. mmol, 1.0 equiv), and THF (50 mL, 0.20 M). The flask was cooled to 0 °C and allylmagnesium bromide (20. mL, 20. mmol, 2.0 equiv, 1.0 M in Et<sub>2</sub>O) was added dropwise. The flask was warmed to rt and allowed to stir overnight. To quench, saturated aq. NH<sub>4</sub>Cl was added dropwise. The biphasic mixture was transferred to a separatory funnel and extracted with EtOAc (x 3). The combined organic layers were washed with H<sub>2</sub>O, brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo. Purification by column chromatography (0–10% EtOAc/hexanes) afforded the desired product as a clear and colorless oil (1.5 g, 9.3 mmol, 93% yield). Analytical data is consistent with literature values.<sup>43</sup> **TLC** *R<sub>f</sub>* = 0.8 (5% EtOAc/hexanes); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.16 (d, *J* = 8.6 Hz, 2H), 6.87 (d, *J* = 8.6 Hz, 2H), 5.90 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.16 – 4.95 (m, 2H), 3.83 (s, 3H), 2.71 (t, *J* = 8.4 Hz, 2H), 2.40 (tdt, *J* = 7.8, 6.6, 1.4 Hz, 2H); **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 158.0, 138.4, 134.2, 129.5 (2C), 115.1, 114.0 (2C), 55.4, 36.0, 34.7.



**2-(4-methoxyphenethyl)oxirane (5.39)** was prepared according to a procedure reported by Taber.<sup>39</sup> A flame dried round bottom flask equipped with a stir bar is charged with alkene **5.38** (1.5 g, 9.3 mmol, 1.0 equiv), CH<sub>2</sub>Cl<sub>2</sub> (93 mL, 0.1 M), and *m*-CPBA (3.4 g, 14 mmol, 1.5 equiv, 70% w/w in H<sub>2</sub>O). The reaction mixture was allowed to stir at rt for 16 h. Upon completion the reaction mixture was diluted with CH<sub>2</sub>Cl<sub>2</sub> and quenched with saturated aq. NaHSO<sub>3</sub>. The biphasic mixture was transferred to a separatory funnel and the layers were separated. The product was

<sup>43</sup> S. Datta, C.-L. Chang, K.-L. Yeh, R.-S. Liu, *J. Am. Chem. Soc.* **2003**, *125*, 9294–9295.

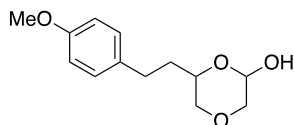
extracted with CH<sub>2</sub>Cl<sub>2</sub> (x 3). The combined organic layers were washed with 1 M NaOH, then brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo. Purification by column chromatography (0–20% EtOAc/hexanes) afforded the title compound as a yellow oil (1.3 g, 7.0 mmol, 75% yield). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.13 (d, *J* = 8.5 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 3.79 (s, 3H), 2.95 (dddd, *J* = 6.4, 5.1, 4.0, 2.8 Hz, 1H), 2.84–2.66 (m, 3H), 2.47 (dd, *J* = 5.0, 2.7 Hz, 1H), 1.92–1.76 (m, 2H); <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 157.9, 133.3, 129.3 (2C), 113.9 (2C), 55.2, 51.7, 47.2, 34.5, 31.3.



**1-(allyloxy)-4-(4-methoxyphenyl)butan-2-ol (5.40)** was prepared according to a procedure reported by Isaacs.<sup>44</sup> In a glovebox, a flame dried round bottom flask equipped with a stir bar was charged with NaH (66 mg, 2.8 mol, 1.1 equiv). The flask was sealed with a septum, removed from the glovebox and placed under an atmosphere of N<sub>2</sub>. Next, DMF (13 mL, 0.2 M) was added via syringe and the reaction mixture was cooled to 0 °C. To the resulting suspension was added a solution of epoxide **5.39** (0.45 g, 2.5 mmol, 1.0 equiv) in DMF (2.5 mL, 1.0 M) dropwise via syringe. The reaction mixture was warmed to rt and allowed to stir for 72 h. The reaction was quenched with addition of H<sub>2</sub>O and extracted with EtOAc (x 3). The combined organic layers were washed with excess H<sub>2</sub>O (x 5) and brine. The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo. Purification by column chromatography (0–20% EtOAc/hexanes) afforded the title compound as a pale yellow oil (0.29 g, 1.2 mmol, 50% yield). TLC R<sub>f</sub> = 0.5 (20% EtOAc/hexanes); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.11 (d, *J* = 8.4 Hz, 2H), 6.81 (d, *J* = 8.3

<sup>44</sup> Isaacs, R. C. A.; Thompson, W. J.; Williams, P. D.; Su, D.-S.; Wenkatraman, S.; Embrey, M. W.; Fisher, T. E.; Wai, J. S.; Dubost, D. C.; Ball, R. C.; Choi, E. J.; Pei, T.; Trice, S. L.; Campbell, N.; Maddess, M.; Maligres, P. E.; Shevlin, M.; Song, Z. J.; Steinhuebel, D. P.; Strotman, N. A.; Yin, J. HIV Integrase Inhibitors. US 2010.0087419 A1, **2010**.

Hz, 2H), 5.89 (ddt,  $J = 16.4, 10.8, 5.6$  Hz, 1H), 5.26 (d,  $J = 17.2$  Hz, 1H), 5.18 (d,  $J = 10.4$  Hz, 1H), 4.03–3.97 (m, 2H), 3.76 (s, 3H), 3.44 (dd,  $J = 9.6, 3.1$  Hz, 1H), 3.30 (t,  $J = 9.0$  Hz, 1H), 2.83–2.70 (m, 1H), 2.68–2.49 (m, 2H), 1.81–1.62 (m, 2H);  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  157.9, 134.6, 134.1, 129.4 (2C), 117.3, 113.9 (2C), 74.6, 72.3, 69.7, 55.3, 35.1, 30.9; HRMS (TOF MS ES+)  $m/z$ :  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{14}\text{H}_{20}\text{O}_3$ , 259.1310; found 259.1322.



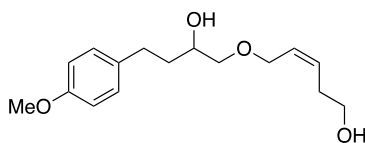
**6-(4-methoxyphenethyl)-1,4-dioxan-2-ol (5.41)** was prepared according to Method E. The following amounts of reagents were used: alkene **5.40** (0.16 g, 0.67 mmol, 1.0 equiv), ozone (excess),  $\text{CH}_2\text{Cl}_2$  (25 mL),  $\text{PPh}_3$  (0.32 g, 2.0 g/1.0 g of alcohol),  $\text{NaHCO}_3$  (0.32 g, 2.0 g/1.0 g of alcohol). Purification by column chromatography (0–70% EtOAc/hexanes) afforded the desired product as a white solid (79 mg, 0.33 mmol, 50% yield, 1.5:1 dr). For clarity, the  $^1\text{H}$  and  $^{13}\text{C}$  are tabulated individually.

**m.p.** = 106–108 °C; **TLC**  $R_f$  = 0.5 (60% EtOAc/hexanes); **HRMS** (TOF MS  $\text{CI}^+$ )  $m/z$ :  $[\text{M}]^+$  calcd for  $\text{C}_{13}\text{H}_{18}\text{O}_4$ , 238.1200; found 238.1205.

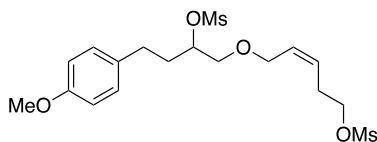
**Major Diastereomer:**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.09 (d,  $J = 8.4$  Hz, 2H), 6.82 (d,  $J = 8.6$  Hz, 2H), 5.04 (d,  $J = 6.2$  Hz, 1H), 4.14 (dddd,  $J = 10.7, 7.9, 4.7, 2.8$  Hz, 1H), 3.78 (s, 3H), 3.73 (ddd,  $J = 11.9, 6.4, 1.8$  Hz, 2H), 3.62 (dd,  $J = 11.8, 2.0$  Hz, 1H), 3.33 (dd,  $J = 11.5, 10.4$  Hz, 1H), 3.27 (s, 1H), 2.78–2.51 (m, 2H), 1.84–1.52 (m, 2H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  158.0, 133.7, 129.4 (2C), 114.0 (2C), 89.8, 71.4, 69.3, 66.6, 55.4, 33.4, 30.3.

**Minor Diastereomer:**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.09 (d,  $J = 8.4$  Hz, 2H), 6.82 (d,  $J = 8.6$  Hz, 2H), 4.87 (ddd,  $J = 9.0, 6.7, 2.6$  Hz, 1H), 3.83 (dd,  $J = 11.2, 2.6$  Hz, 1H), 3.78 (s, 3H), 3.73 (ddd,  $J = 11.9, 6.4, 1.8$  Hz, 2H), 3.62 (dd,  $J = 11.8, 2.0$  Hz, 1H), 3.27 (s, 1H), 3.20–3.06 (m, 1H),

2.78–2.51 (m, 2H), 1.84–1.52 (m, 2H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  158.0, 133.6, 129.4 (2C), 114.0 (2C), 92.3, 73.9, 70.2, 69.8, 55.4, 33.0, 30.4.



**(2-Hydroxy-4-(4-methoxyphenyl)butoxy)pent-3-en-1-ol (5.42)** was prepared according to Method G. The following amounts of reagents were used: lactol **5.41** (0.21 g, 0.88 mmol, 1.0 equiv), Wittig salt **5.24** (0.36 g, 0.89 mmol, 1.0 equiv), *n*-BuLi (1.2 mL, 2.8 mmol, 3.2 equiv, 2.5 M in hexanes), and THF (4.4 mL). Purification by column chromatography afforded a mixture of the title compound with triphenylphosphine oxide. This mixture was carried onto the next step without further purification.



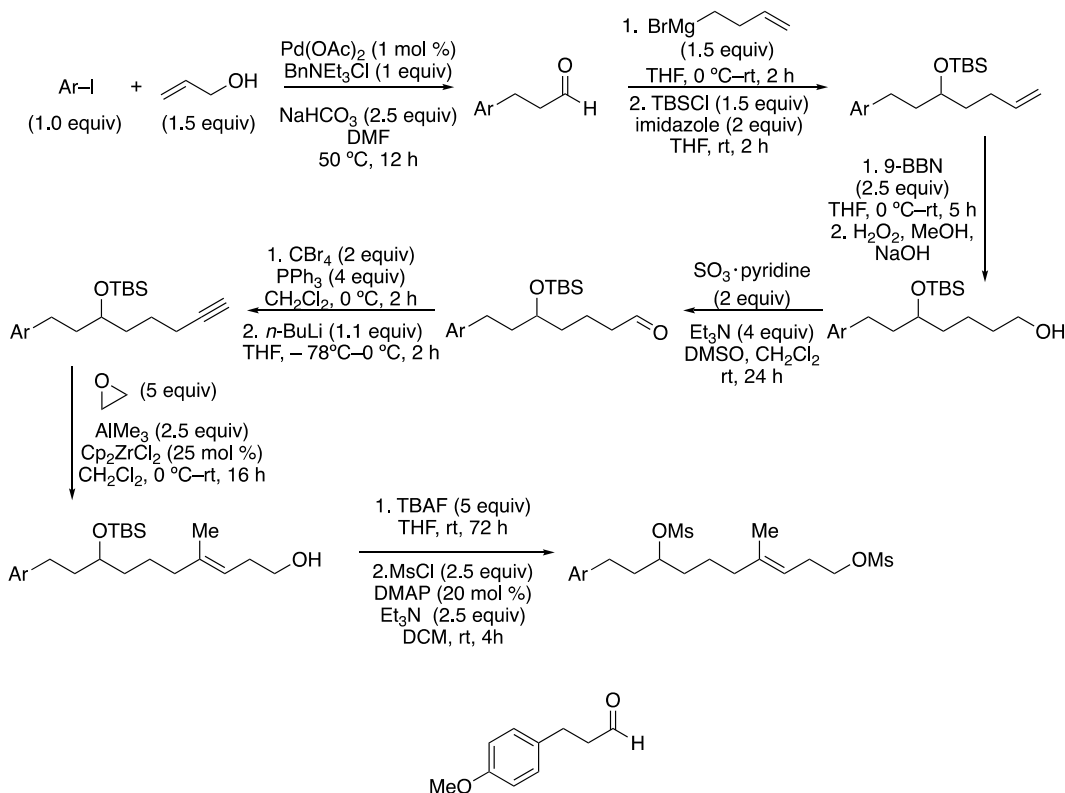
**(4-Methoxyphenyl)-1-((5-((methylsulfonyl)oxy)pent-2-en-1-yl)oxy)butan-2-yl methanesulfonate (5.19)** was prepared according to Method H. The following amounts of reagents were used: diol **5.42** (0.29 g, 1.0 mmol, 1.0 equiv),  $\text{MsCl}$  (0.20 mL, 2.6 mmol, 2.5 equiv), DMAP (25 mg, 0.21 mmol, 20 mol %),  $\text{Et}_3\text{N}$  (0.36 mL, 2.6 mmol, 2.5 equiv), and DCM (5.2 mL). Purification by column chromatography (0–60% EtOAc/hexanes) afforded the desired product as a pale yellow oil (197 mg, 0.45 mmol, 44% yield, 2:1 dr). For clarity, the  $^1\text{H}$  and  $^{13}\text{C}$  are tabulated individually. **TLC**  $R_f$  = 0.6 (60% EtOAc/hexanes); **HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M}+\text{NH}_4]^+$  calcd for  $\text{C}_{18}\text{H}_{32}\text{NO}_8\text{S}_2$  454.1569, found 454.1582.

**Major Diastereomer:**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.11 (d,  $J$  = 8.6 Hz, 2H), 6.83 (d,  $J$  = 8.7 Hz, 2H), 5.75–5.54 (m, 2H), 4.80 (dddd,  $J$  = 9.1, 7.8, 4.5, 1.2 Hz, 1H), 4.23 (dt,  $J$  = 9.7, 6.6 Hz, 2H), 4.15–3.92 (m, 2H), 3.77 (s, 3H), 3.64–3.52 (m, 2H), 3.06 (s, 3H), 2.99 (s, 3H), 2.81–2.60 (m,

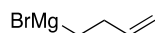
2H), 2.57–2.44 (m, 2H), 1.95 (td,  $J = 14.4, 9.8, 6.7, 4.9$  Hz, 2H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  158.0, 132.7, 129.3 (2C), 128.2, 127.4, 114.0 (2C), 81.7, 71.7, 68.9, 66.6, 55.3, 38.7, 37.4, 33.6, 30.3, 27.8.

**Minor Diastereomer:**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.11 (d,  $J = 8.6$  Hz, 2H), 6.83 (d,  $J = 8.7$  Hz, 2H), 5.75–5.54 (m, 2H), 4.80 (dddd,  $J = 9.1, 7.8, 4.5, 1.2$  Hz, 1H), 4.23 (dt,  $J = 9.7, 6.6$  Hz, 2H), 4.15–3.92 (m, 2H), 3.77 (s, 3H), 3.64–3.52 (m, 2H), 3.06 (s, 3H), 2.99 (s, 3H), 2.81–2.60 (m, 2H), 2.57–2.44 (m, 2H), 1.95 (td,  $J = 14.4, 9.8, 6.7, 4.9$  Hz, 2H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  158.0, 132.7, 129.7, 129.4 (2C), 127.4, 114.1 (2C), 81.7, 71.7, 68.8, 66.6, 55.3, 38.7, 37.5, 33.6, 30.3, 27.8.

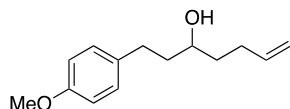
**Scheme 5.7** Synthesis of Trisubstituted Alkene Dimesylate Starting Material



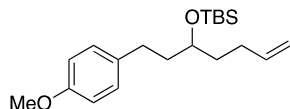
**3-(4-Methoxyphenyl)propanal (5.25)** was prepared according to Method B. Refer above for analytical data.



**But-3-en-1-ylmagnesium bromide (5.43)** was prepared according to Method C. The following amounts of reagents were used: 4-bromo-butene (4.1 mL, 40. mmol, 1.0 equiv), Mg<sup>0</sup> (1.4 g, 60. mmol, 1.5 equiv), and THF (20 mL). The resulting Grignard reagent was titrated to be 1.3 M.<sup>35</sup>



**1-(4-methoxyphenyl)hept-6-en-3-ol (5.44)** was prepared according to a modified version of Method D. The following amounts of reagents were used: aldehyde **5.25** (2.6 g, 16 mmol, 1.0 equiv), Grignard reagent **5.43** (19 mL, 24 mmol, 1.5 equiv, 1.3 M in THF), and THF (80 mL). The crude reaction mixture was passed through a plug of silica gel eluting with 100% EtOAc to remove any remaining magnesium salts and was carried into the next step without further purification.

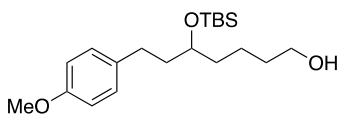


**tert-Butyl((1-(4-methoxyphenyl)hept-6-en-3-yl)oxy)dimethylsilane (5.45)** was prepared according a procedure reported by Jia.<sup>45</sup> To a flame dried round bottom flask equipped with a stir bar was added alcohol **5.44** (3.5 g, 16 mmol, 1.0 equiv), imidazole (2.2 g, 32 mmol, 2.0 equiv), *tert*-butyldimethylsilyl chloride (3.6 g, 24 mmol, 1.5 equiv), and CH<sub>2</sub>Cl<sub>2</sub> (81 mL, 0.20 M). The reaction mixture was allowed to stir at rt for 24 h. After the reaction was complete, H<sub>2</sub>O was added and the mixture was transferred to a separatory funnel. The mixture was extracted with CH<sub>2</sub>Cl<sub>2</sub> (x 3). The combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated in vacuo. Purification by column chromatography (0-30% EtOAc/hexanes) afforded the title compound as a pale yellow oil (4.6 g, 13 mmol, 86% yield). **TLC R<sub>f</sub>** = 0.8 (20% EtOAc/hexanes);

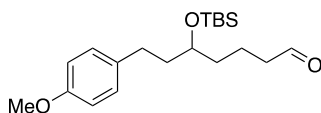
<sup>45</sup> Zhou, S.; Jia, Y. *Org. Lett.* **2014**, *16*, 3416–3418.



**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.17 (d, *J* = 7.9 Hz, 2H), 6.90 (d, *J* = 8.1 Hz, 2H), 5.99–5.83 (m, 1H), 5.17–4.98 (m, 2H), 3.84 (s, 4H), 2.80–2.58 (m, 2H), 2.29–2.13 (m, 2H), 1.83 (aq, *J* = 7.8, 7.3, 6.6 Hz, 2H), 1.69 (aq, *J* = 7.9, 7.4, 6.7 Hz, 2H), 1.05–1.02 (m, 9H), 0.16 (dd, *J* = 3.3, 1.5 Hz, 6H); **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 158.0, 139.1, 134.9, 129.4 (2C), 114.6, 114.1 (2C), 71.6, 55.4, 39.5, 36.5, 31.0, 29.9, 26.2 (3C), 18.4, -4.1 (2C); **HRMS** (TOF MS CI+) *m/z*: [M+H]<sup>+</sup> calcd for C<sub>20</sub>H<sub>34</sub>O<sub>2</sub>SiH 335.2406, found 335.2402.

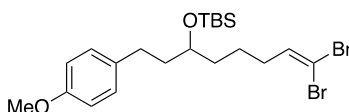


**5-((*tert*-butyldimethylsilyl)oxy)-7-(4-methoxyphenyl)heptan-1-ol (5.46)** was prepared according a procedure reported by Hartwig.<sup>46</sup> To a flame dried round bottom flask equipped with a stir bar was added silyl protected alcohol **5.45** (4.6 g, 14 mmol, 1.0 equiv) and THF (36 mL, 0.20 M). The reaction mixture was cooled to 0 °C and 9-BBN (69 mL, 35 mmol, 1.5 equiv, 0.50 M in THF) was added dropwise via syringe. The mixture was warmed to rt and allowed to stir for at least 5 h. Then MeOH (42 mL, 3.0 mL/mmol), H<sub>2</sub>O<sub>2</sub> (14 mL, 30%, 1.0 mL/mmol), and NaOH (42 mL, 1.0 M, 3.0 mL/mmol) were added, and the reaction stirred for at least 3 h. Once complete, H<sub>2</sub>O (10 mL) was added. The reaction mixture was then extracted with EtOAc (x 3) and combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated in vacuo. Purification by column chromatography (0–30% EtOAc/hexanes) afforded a mixture of the title compound and unreacted 9-BBN as a pale yellow oil. This mixture was carried into the next step without further purification.



<sup>46</sup> Stanley, L. M.; Hartwig, J. F. *J. Am. Chem. Soc.* **2009**, *131*, 8971–8983.

**5-((*tert*-butyldimethylsilyl)oxy)-7-(4-methoxyphenyl)heptanal (5.47)** was prepared according to a procedure reported by Nicolaou.<sup>47</sup> To a flame dried round bottom flask equipped with a stir bar was added alcohol **5.46** (1.1 g, 3.0 mmol, 1.0 equiv), SO<sub>3</sub>·pyridine (0.96 g, 6.0 mmol, 2.0 equiv), Et<sub>3</sub>N (1.7 mL, 12 mmol, 4.0 equiv), DMSO (3 mL), and CH<sub>2</sub>Cl<sub>2</sub> (3 mL). The reaction mixture was allowed to stir at rt for 24 h. Once complete, saturated aq. NH<sub>4</sub>Cl (10 mL) was added. The reaction mixture was then extracted with CH<sub>2</sub>Cl<sub>2</sub> (x 3) and combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated in vacuo.<sup>48</sup> Purification by column chromatography (0–10% EtOAc/hexanes) afforded the title compound as a yellow oil (0.80 g, 2.3 mmol, 76% yield). **TLC** R<sub>f</sub> = 0.6 (20% EtOAc/hexanes, CAM stain); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 9.76 (t, *J* = 1.7 Hz, 1H), 7.08 (d, *J* = 8.7 Hz, 2H), 6.82 (d, *J* = 8.6 Hz, 2H), 3.78 (s, 3H), 3.72 (p, *J* = 5.7 Hz, 1H), 2.57 (qt, *J* = 13.8, 8.1 Hz, 2H), 2.46–2.37 (m, 2H), 1.95–1.82 (m, 2H), 1.79–1.59 (m, 2H), 1.59–1.46 (m, 2H), 0.91 (s, 9H), 0.05 (d, *J* = 5.6 Hz, 6H); **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 202.6, 157.9, 134.7, 129.3 (2C), 113.9 (2C), 71.6, 55.4, 44.1, 42.1, 39.2, 36.4, 30.8, 26.1 (3C), 18.0, -4.3 (2C); **HRMS** (TOF MS ES+) *m/z*: [M+H]<sup>+</sup> calcd for C<sub>20</sub>H<sub>34</sub>O<sub>3</sub>SiH, 351.2355; found 351.2349.



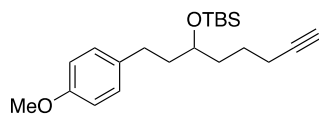
***tert*-butyl((8,8-dibromo-1-(4-methoxyphenyl)oct-7-en-3-yl)oxy)dimethylsilane (5.48)** was prepared according to a procedure reported by Fürstner.<sup>49</sup> To a flame dried round bottom flask equipped with a stir bar was added aldehyde **5.47** (0.31 g, 0.88 mmol, 1.0 equiv), CBr<sub>4</sub> (0.58 g,

<sup>47</sup> Nicolaou, K. C.; Peng, X.-S.; Sun, Y.-P.; Polet, D.; Zou, B.; Lim, C. S.; Chen, D. Y. K. *J. Am. Chem. Soc.* **2009**, *131*, 10587–10597.

<sup>48</sup> Note: We observed that the yield of the Parikh-Doering oxidation decreases with increasing scale. We typically performed this reaction on less than 5.0 mmol of primary alcohol.

<sup>49</sup> Wölfl, B.; Mata, G.; Fürstner, A. *Chem. Eur. J.* **2019**, *25*, 255–259.

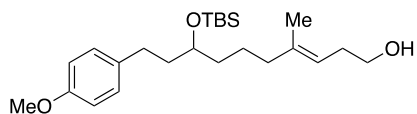
1.8 mmol, 2.0 equiv), and CH<sub>2</sub>Cl<sub>2</sub> (4.4 mL, 0.20 M). The reaction mixture was cooled to 0 °C and then PPh<sub>3</sub> (0.92 g, 3.5 mmol, 4.0 equiv) was added dropwise as a solution in CH<sub>2</sub>Cl<sub>2</sub> (5.8 mL, 0.60 M). The reaction was allowed to stir at 0 °C for 2 h. To quench, hexanes was added and the reaction mixture was filtered through a pad of silica gel eluting with 50% Et<sub>2</sub>O in hexanes. The filtrate was concentrated and carried into the next step without further purification. The resulting crude reaction mixture was carried into the next step without further purification (0.38 g, 0.70 mmol, 84% yield). **TLC** R<sub>f</sub> = 0.6 (20% EtOAc/hexanes, CAM stain); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.12 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 6.40 (t, *J* = 7.2 Hz, 1H), 3.80 (s, 3H), 3.78–3.71 (m, 1H), 2.70–2.45 (m, 3H), 2.45–2.37 (m, 1H), 2.16–2.08 (m, 2H), 1.90 (dtt, *J* = 9.0, 6.2, 2.6 Hz, 2H), 1.75 (tdd, *J* = 8.5, 5.7, 1.2 Hz, 2H), 0.95 (s, 9H), 0.11–0.06 (m, 6H); **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 157.9, 138.8, 134.7, 129.3 (2C), 113.9 (2C), 89.0, 71.6, 55.4, 39.3, 36.4, 33.3, 30.9, 26.1 (3C), 23.6, 18.3, -4.2 (2C).



**tert-butyl((1-(4-methoxyphenyl)oct-7-yn-3-yl)oxy)dimethylsilane (5.49)** was prepared according to a procedure reported by Ghosh.<sup>50</sup> The unpurified dibromide **5.48** (1.1 g, 2.1 mmol, 1.0 equiv) was dissolved in THF (10 mL, 0.20 M) and the mixture was cooled to –78 °C. Then *n*-BuLi (2.3 mL, 3.2 mmol, 1.5 equiv, 1.4 M in hexanes) was added dropwise and the mixture was allowed to slowly warm to 0 °C over 2 h. To quench, saturated aq. NH<sub>4</sub>Cl was added dropwise and the biphasic mixture was transferred to separatory funnel. The organic layer was extracted with EtOAc (x 3). The combined organic layers were washed sequentially with H<sub>2</sub>O and brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated in vacuo. Purification by column chromatography (0–10%

<sup>50</sup> Chandrasekhar, B.; Athe, S.; Reddy, P. P.; Ghosh, S. *Org. Biomol. Chem.* **2015**, *13*, 115–124.

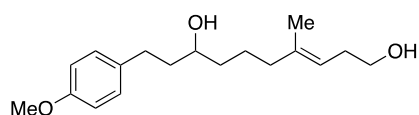
EtOAc/hexanes) afforded the title compound as a pale yellow oil (0.43 g, 1.2 mmol, 53% yield). **TLC**  $R_f$  = 0.6 (20% EtOAc/hexanes, CAM Stain);  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.10 (d,  $J$  = 8.8 Hz, 2H), 6.84 (d,  $J$  = 8.6 Hz, 2H), 3.80 (s, 3H), 3.74 (p,  $J$  = 5.4 Hz, 1H), 2.69–2.49 (m, 2H), 2.25–2.15 (m, 2H), 1.96 (t,  $J$  = 2.6 Hz, 1H), 1.74 (ddd,  $J$  = 9.1, 7.9, 5.7 Hz, 2H), 1.67–1.55 (m, 3H), 0.92 (s, 10H), 0.07 (d,  $J$  = 2.8 Hz, 6H);  **$^{13}\text{C NMR}$**  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  157.8, 134.8, 129.3 (2C), 113.9 (2C), 84.6, 71.6, 68.5, 55.4, 39.4, 36.1, 30.9, 26.1 (3C), 24.4, 18.8, 18.3, -4.2 (2C).



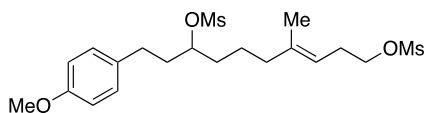
**(E)-8-((tert-butyldimethylsilyloxy)-10-(4-methoxyphenyl)-4-methyldec-3-en-1-ol (5.50)** was prepared according to a procedure reported by Sigman.<sup>51</sup> A flame dried round bottom flask equipped with a stir bar is charged with  $\text{Cp}_2\text{ZrCl}_2$  (31 mg, 0.11 mmol, 25 mol %) and  $\text{CH}_2\text{Cl}_2$  (0.21 mL, 2.0 M). The flask was cooled to 0 °C and  $\text{AlMe}_3$  (0.54 mL, 1.1 mmol, 2.5 equiv, 2.0 M in PhMe) was added dropwise via syringe and the mixture was allowed to stir for 30 mins. Then, a solution of alkyne **5.49** (150 mg, 0.43 mmol, 1.0 equiv) in  $\text{CH}_2\text{Cl}_2$  (0.45 mL, 0.25 M) was added via syringe. The reaction mixture was warmed to rt and allowed to stir overnight. Then the mixture was cooled to 0 °C and ethylene oxide (1.1 mL, 2.2 mmol, 5.0 equiv, 2.5–3.3 M in THF) was added in one portion via syringe. The mixture was allowed to stir for an additional 3 h. To quench, the reaction mixture was cooled to 0 °C and 1 M HCl was added dropwise. The mixture was then passed through a pad of Celite eluting with 100%  $\text{CH}_2\text{Cl}_2$ . The biphasic mixture was transferred to a separatory funnel and extracted with  $\text{CH}_2\text{Cl}_2$  (x 3). The combined organic layers were washed with 1 M HCl and then brine. Then the organic layer was dried over  $\text{Na}_2\text{SO}_4$ , filtered, and concentrated in vacuo. Purification by column chromatography (0–30% EtOAc/hexanes) afforded

<sup>51</sup> Patel, H. H.; Sigman, M. S. *J. Am. Chem. Soc.* **2016**, *138*, 14226–14229.

the desired product as a pale yellow oil (69 mg, 0.17 mmol, 40% yield, >20:1 dr). **TLC**  $R_f$  = 0.3 (30% EtOAc/hexanes);  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.10 (d,  $J$  = 8.4 Hz, 2H), 6.83 (d,  $J$  = 8.4 Hz, 2H), 5.14 (t,  $J$  = 7.2 Hz, 1H), 3.79 (s, 3H), 3.71 (t,  $J$  = 5.0 Hz, 1H), 3.66–3.60 (m, 3H), 3.58 (t,  $J$  = 5.3 Hz, 1H), 2.75–2.46 (m, 2H), 2.30 (q,  $J$  = 6.8 Hz, 2H), 2.00 (d,  $J$  = 9.0 Hz, 2H), 1.80–1.70 (m, 4H), 1.46 (qd,  $J$  = 11.9, 7.2 Hz, 4H), 0.92 (s, 9H), 0.07 (d,  $J$  = 4.4 Hz, 6H);  **$^{13}\text{C NMR}$**  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  157.8, 138.8, 134.9, 129.3 (2C), 120.0, 113.9 (2C), 71.9, 69.3, 62.6, 55.4, 40.0, 36.7, 31.7, 31.0, 26.1 (3C), 23.6, 18.3, 16.2, -4.2 (2C).



**(E)-10-(4-methoxyphenyl)-4-methyldec-3-ene-1,8-diol (5.51)** was prepared according to a procedure reported by Knochel.<sup>52</sup> To a flame dried round bottom flask equipped with a stir bar was added silylprotected alcohol **5.50** (69 mg, 0.17 mmol, 1.0 equiv) and THF (0.85 mL, 0.20 M). Then, tetrabutyl ammonium fluoride (TBAF, 0.85 mL, 0.85 mmol, 5.0 equiv, 1.0 M in THF) was added dropwise at rt. The reaction mixture was allowed to stir at rt for 72 h. The reaction was quenched with  $\text{H}_2\text{O}$ . The biphasic mixture was transferred to a separatory funnel and the product was extracted with EtOAc (x 3). The combined organic layers were washed with brine, dried over  $\text{Na}_2\text{SO}_4$ , filtered, and concentrated in vacuo. Purification by column chromatography (0–30% EtOAc/hexanes) afforded the title compound as a pale yellow oil (40 mg, 0.14 mmol, 80% yield).



**(E)-10-(4-methoxyphenyl)-4-methyldec-3-ene-1,8-diyl dimethanesulfonate (5.20)** was prepared according to Method H. The following amounts of reagents were used: diol **5.51** (40 mg,

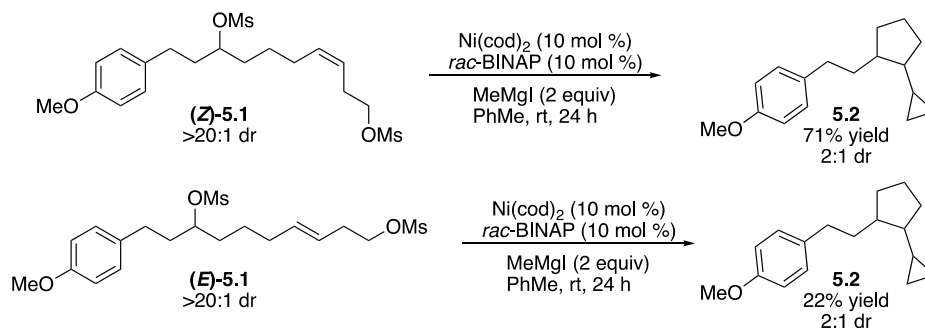
<sup>52</sup> Seel, S.; Thaler, T.; Takatsu, K.; Zhang, C.; Zipse, H.; Straub, B. F.; Mayer, P.; Knochel, P. *J. Am. Chem. Soc.* **2011**, *133*, 4774–4777.

0.14 mmol, 1.0 equiv), MsCl (30.  $\mu$ L, 0.35 mmol, 2.5 equiv), DMAP (3.4 mg, 28  $\mu$ mol, 20 mol %), Et<sub>3</sub>N (50.  $\mu$ L, 0.35 mmol, 2.5 equiv), and CH<sub>2</sub>Cl<sub>2</sub> (0.70 mL). Purification by column chromatography (0–50% EtOAc/hexanes) afforded the desired product as a pale yellow oil (27 mg, 61  $\mu$ mol, 44% yield). **TLC** R<sub>f</sub> = 0.4 (50% EtOAc/hexanes, CAM stain); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.11 (d, *J* = 8.3 Hz, 2H), 6.83 (d, *J* = 8.4 Hz, 2H), 5.12 (t, *J* = 6.8 Hz, 1H), 4.76 (h, *J* = 6.5, 6.0 Hz, 1H), 4.18 (t, *J* = 6.9 Hz, 2H), 3.78 (s, 3H), 3.06–2.92 (m, 6H), 2.78–2.58 (m, 2H), 2.46 (q, *J* = 7.0 Hz, 1H), 2.09–1.89 (m, 4H), 1.80–1.65 (m, 2H), 1.60–1.43 (m, 3H), 1.09 (d, *J* = 1.4 Hz, 3H); **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>)  $\delta$  158.2, 138.9, 133.0, 129.4 (2C), 118.5, 114.1 (2C), 83.2, 69.6, 55.4, 39.2, 38.9, 37.6, 36.5, 34.1, 30.5, 28.2, 23.0, 16.2.

## 5.4.7 Synthesis and Characterization Data for Mechanistic Experiments

### 5.4.7.1 Mechanistic Experiment with Single Alkene Diastereomer

**Scheme 5.8** Results from Single Alkene Diastereomer Experiment



The control reaction was performed according to Method A. Two separate reactions were set up in 7-mL dram vials. The following amounts of reagents were used:

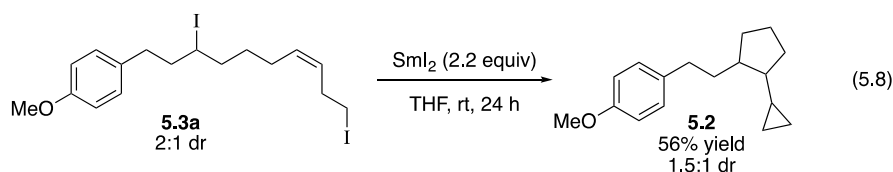
**Vial 1:** (*Z*)-**5.1** (61 mg, 0.14 mmol, 1.0 equiv), Ni(cod)<sub>2</sub> (3.9 mg, 14  $\mu$ mol, 10 mol %), *rac*-BINAP (8.7 mg, 14  $\mu$ mol, 10 mol %), MeMgI (0.11 mL, 0.28 mmol, 2.0 equiv, 2.9 M in Et<sub>2</sub>O), PhMe (0.70 mL), AD-mix- $\beta$  (196 mg, 1.4 g/mol substrate), *t*-BuOH (0.50 mL), and H<sub>2</sub>O (0.50 mL). Purification by column chromatography (0–10% EtOAc/hexanes) afforded the desired product **5.2** (24 mg, 98  $\mu$ mol, 71% yield, 2:1 dr).

**Vial 2:** (*E*)-**5.1** (17 mg, 40  $\mu$ mol, 1.0 equiv), Ni(cod)<sub>2</sub> (1.1 mg, 4.0  $\mu$ mol, 10 mol %), *rac*-BINAP (2.5 mg, 4.0  $\mu$ mol, 10 mol %), MeMgI (30.  $\mu$ L, 80.  $\mu$ mol, 2.0 equiv, 2.9 M in Et<sub>2</sub>O), PhMe (0.20 mL), AD-mix- $\beta$  (56 mg, 1.4 g/mol substrate), *t*-BuOH (0.30 mL), and H<sub>2</sub>O (0.30 mL). Purification by column chromatography (0–10% EtOAc/hexanes) afforded the desired product **5.2** (2.1 mg, 8.5  $\mu$ mol, 22% yield, 2:1 dr).

#### 5.4.7.2 Separation of Alkene Diastereomers on Silver Impregnated Silica

AgNO<sub>3</sub> (10 g) was dissolved in H<sub>2</sub>O (2.5 mL) and MeCN (100 mL) in a 500 mL erlenmeyer flask. The stock solution was poured into a 500 mL round bottom flask that contained 100 g of silica gel and a stir bar. The slurry was allowed to stir at rt for 2 h and then was concentrated in vacuo in the dark. The silica gel was dried on a high vacuum manifold overnight and resulted in a cream colored powder. The silica gel was then used to separate the alkene diastereomers.<sup>53</sup>

#### 5.4.7.3 SmI<sub>2</sub> Control Reaction



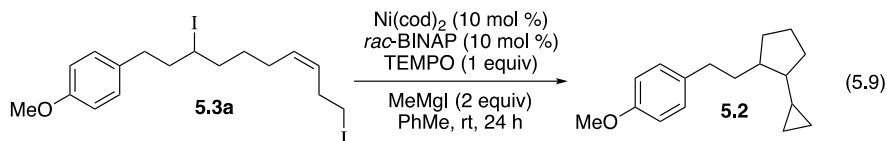
**Preparation of SmI<sub>2</sub> in THF:** SmI<sub>2</sub> was prepared as a solution in THF according to a procedure reported by Procter.<sup>30</sup> In a glovebox, Sm metal (0.60 g, 4.0 mmol, 2.0 equiv) was added to a flame dried Schlenk flask equipped with a stir bar. The flask was sealed with a septum, removed from the glovebox, and placed under an N<sub>2</sub> atmosphere. Then freshly washed 1,2-diiodoethane (0.56 g, 2.0 mmol, 1.0 equiv) was added. The flask was evacuated and backfilled with N<sub>2</sub> (x 3). Then THF (20 mL) was added via syringe and the resulting yellow solution was evacuated until the solvent began to bubble and backfilled with N<sub>2</sub> (x 3). Then the mixture was allowed to stir overnight at rt

<sup>53</sup> (a) see ref: 28. (b) The silver impregnated silica gel is sensitive to light. All preparations of the silica gel were performed in the dark. The column was performed by wrapping the glassware in aluminum foil and in the dark.

in the dark and resulted in a dark blue solution. The SmI<sub>2</sub> was titrated according to a procedure reported by Hilmersson and used immediately.<sup>54</sup>

**Control Reaction with SmI<sub>2</sub>:** To a flame dried round bottom flask equipped with a stir bar was added diiodide **5.3a** (27 mg, 54 μmol, 1.0 equiv) and THF (0.27 mL). Then SmI<sub>2</sub> (3.0 mL, 0.12 mmol, 2.2 equiv, 0.042 M in THF) was added dropwise via syringe. The reaction mixture was stirred at rt overnight. MeOH was added to quench and the reaction mixture was filtered through a pad of silica gel eluting with 100% Et<sub>2</sub>O. Purification by column chromatography (100% hexanes) afforded the cascade product **5.2** (7.4 mg, 30 μmol, 56% yield, 1.5:1 dr). Refer above for characterization data.

#### 5.4.7.4 TEMPO Control Reaction



The mechanistic experiment with TEMPO as an additive was performed according to a modified version of Method A. The following amounts of reagents were used: diiodide **5.3a** (25 mg, 50. μmol, 1.0 equiv), Ni(cod)<sub>2</sub> (1.4 mg, 5.0 μmol, 10. mol %), *rac*-BINAP (3.1 mg, 5.0 μmol, 10. mol %), MeMgI (30. μL, 0.10 mmol, 2.0 equiv, 2.9 M in Et<sub>2</sub>O), TEMPO (7.8 mg, 50. μmol, 1.0 equiv), and PhMe (0.25 mL). The reaction was quenched with MeOH and filtered through a pad of silica gel eluting with 100% Et<sub>2</sub>O. A 48% <sup>1</sup>H NMR yield of **5.2** was observed compared to PhTMS as an internal standard.

<sup>54</sup> Dahlén, A.; Hilmersson, G. *Eur. J. Inorg. Chem.* **2004**, 15, 3020–3024.



### 5.4.8 Competition Experiment

The competition experiment was performed according to a modified version of Method A. Two separate reactions were set up for each additive in different 7-mL dram vials. The following amounts of reagents were used:

**Vial 1:** dimesylate **5.1** (47 mg, 0.11 mmol, 1.0 equiv), secondary mesylate **5.9** (45 mg, 0.11 mmol, 1.0 equiv), Ni(cod)<sub>2</sub> (3.1 mg, 11 μmol, 10. mol %), *rac*-BINAP (6.8 mg, 11 μmol, 10. mol %), MeMgI (0.11 mL, 0.33 mmol, 3.0 equiv, 3.0 M in Et<sub>2</sub>O), and PhMe (0.55 mL). The crude reaction mixture was concentrated to remove PhMe and was diluted to 300 μM in DCM and analyzed by GCMS. All area percent values were obtained from GCMS spectrum.

**Vial 2:** dimesylate **5.1** (43 mg, 0.10 mmol, 1.0 equiv), primary mesylate **5.12** (36 mg, 0.10 mmol, 1.0 equiv), Ni(cod)<sub>2</sub> (2.8 mg, 10. μmol, 10. mol %), *rac*-BINAP (6.2 mg, 10. μmol, 10. mol %), MeMgI (0.10 mL, 0.30 mmol, 3.0 equiv, 3.0 M in Et<sub>2</sub>O), and PhMe (0.50 mL). The crude reaction mixture was concentrated to remove PhMe and was diluted to 300 μM in DCM and analyzed by GCMS. All area percent values were obtained from GCMS spectrum.

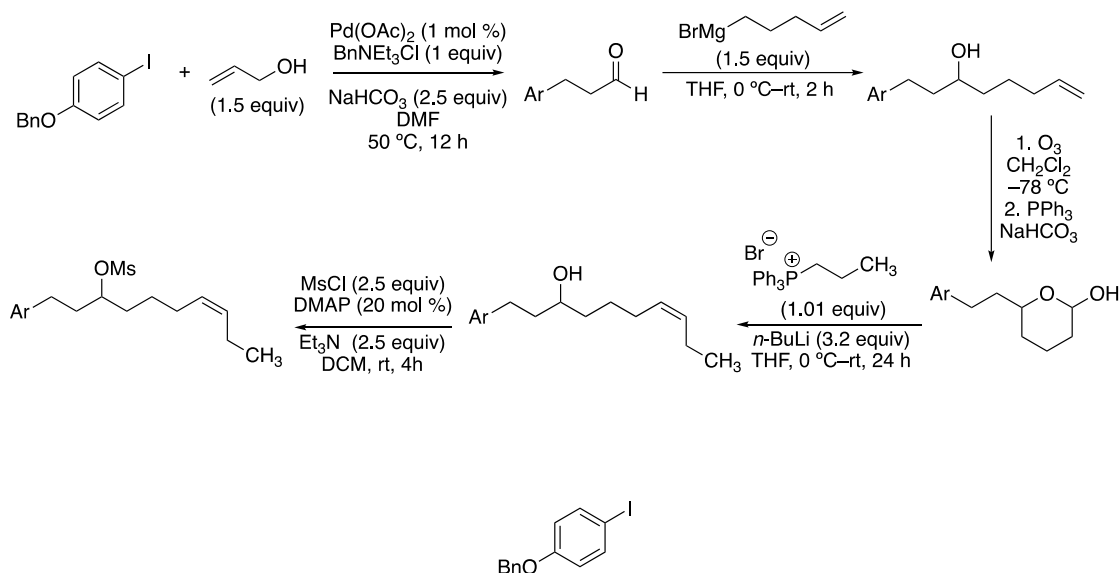
**Table 5.3.** Area Percent Values and Ratio Calculations for the Competition Experiment

Compound	Area	Area Percent	
PhTMS	16627132	16.69231113	
Trimethoxybenzene	33349020	33.47974972	<b>Ratio</b>
Product ( <b>5.2</b> )	30001111	30.11871676	1.52815348
Products from 2° mesylate ( <b>5.10, 5.11</b> )	19632263	19.70922239	
<b>Total</b>	99609526		

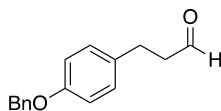
Compound	Area	Area Percent	
PhTMS	28142418.73	16.81324325	
Trimethoxybenzene	48364556.51	28.89463983	
Product <b>2</b>	37642540.71	22.48894096	
Products from 1° mesylate ( <b>5.14</b> )	5668865.774	3.386774253	<b>Ratio</b>
Kumada ( <b>5.13</b> )	8612848	5.145609901	2.63571594
Iodide ( <b>5.52</b> )	38951221.83	23.27079181	

## 5.4.8.1 Synthesis and Characterization Data for Competition Experiment

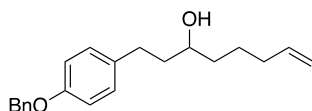
Scheme 5.9 Synthesis of Secondary Mesylate 5.9 for Competition Experiment



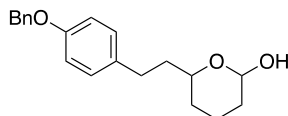
1-(Benzyloxy)-4-iodobenzene (5.29) refer above for synthesis and characterization data.



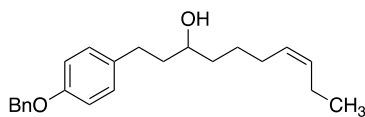
3-(4-(Benzyloxy)phenyl)propanal (5.30) refer above for synthesis and characterization data.



1-(4-(Benzyloxy)phenyl)oct-7-en-3-ol (5.31) refer above for synthesis and characterization data.

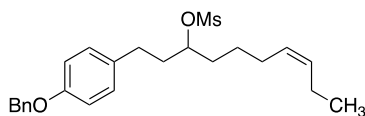


6-(4-(Benzyloxy)phenethyl)tetrahydro-2H-pyran-2-ol (5.32) refer above for synthesis and characterization data.



**(4-(benzyloxy)phenyl)dec-7-en-3-ol (5.53)** was prepared according to Method G. The following amounts of reagents were used: lactol **5.32** (0.10 g, 0.32 mmol, 1.0 equiv), propyltriphenylphosphonium bromide (0.13 g, 0.32 mmol, 1.0 equiv), *n*-BuLi (0.40 mL, 0.96 mmol, 3.0 equiv, 2.5 M in hexanes), and THF (1.6 mL). Purification by column chromatography (0–50% EtOAc/hexanes) afforded the title compound as a white solid (59 mg, 0.17 mmol, 55% yield, 2:1 dr). **TLC**  $R_f$  = 0.3 (30% EtOAc/hexanes, KMnO<sub>4</sub> stain); **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.45–7.40 (m, 4H, both diastereomers), 7.37 (ddd,  $J$  = 9.9, 5.6, 1.6 Hz, 4H, both diastereomers), 7.33–7.28 (m, 2H, both diastereomers), 7.11 (d,  $J$  = 8.5 Hz, 4H, both diastereomers), 6.90 (d,  $J$  = 8.5 Hz, 4H, both diastereomers), 4.68 (br s, 2H, both diastereomers), 5.47–5.25 (m, 4H, both diastereomers), 5.03 (s, 4H, both diastereomers), 3.60 (tt,  $J$  = 8.1, 4.0 Hz, 2H, both diastereomers), 2.72 (ddd,  $J$  = 14.9, 9.6, 5.7 Hz, 2H, both diastereomers), 2.60 (ddd,  $J$  = 13.9, 9.5, 6.8 Hz, 2H, both diastereomers), 2.09–1.93 (m, 8H, both diastereomers), 1.81–1.63 (m, 4H, both diastereomers), 1.54–1.32 (m, 8H, both diastereomers), 0.95 (td,  $J$  = 7.4, 4.4 Hz, 6H, both diastereomers); **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  157.1 (2C, both diastereomers), 137.3 (2C, both diastereomer), 134.6 (2C, both diastereomers), 132.5 (minor diastereomer), 132.1 (major diastereomer), 129.4 (4C, both diastereomers), 128.9 (2C, both diastereomers), 128.7 (4C, both diastereomers), 128.0 (2C, both diastereomer), 127.6 (4C, both diastereomers), 114.9 (4C, both diastereomers), 71.3 (2C, both diastereomers), 70.1 (2C, both diastereomers), 39.4 (2C, both diastereomers), 37.2 (major diastereomers), 37.1 (minor diastereomer), 31.2 (2C, both diastereomers), 27.1 (2C, both diastereomers), 25.8 (major diastereomer), 25.7 (minor diastereomers), 20.6 (2C, both

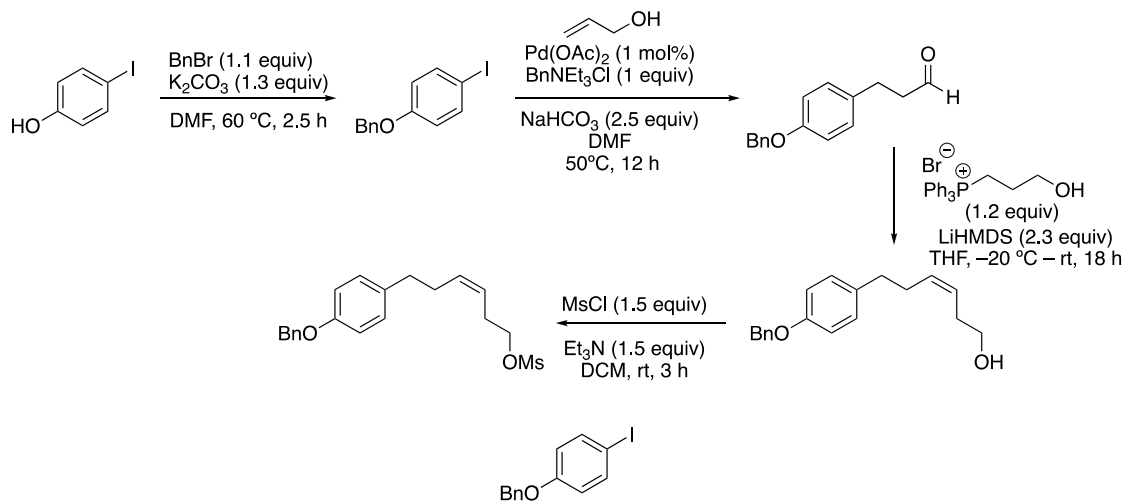
diastereomers), 14.5 (major diastereomer), 14.1 (minor diastereomer); **HRMS** (TOF MS ES+)  $m/z$ :  $[M+Na]^+$  calcd for  $C_{23}H_{30}O_2Na$ , 361.2144; found 361.2129.



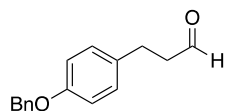
**(Z)-1-(4-(benzyloxy)phenyl)dec-7-en-3-yl methanesulfonate (5.9)** was prepared according to Method H. The following amounts of reagents were used: alcohol **5.53** (0.12 g, 0.34 mmol, 1.0 equiv), MsCl (40.  $\mu$ L, 0.51 mmol, 1.5 equiv),  $Et_3N$  (70.  $\mu$ L, 0.51 mmol, 1.5 equiv), and  $CH_2Cl_2$  (1.7 mL). Purification by column chromatography (0–30% EtOAc/hexanes) afforded the title compound as a yellow oil (124 mg, 0.30 mmol, 87% yield). **TLC**  $R_f$  = 0.8 (50% EtOAc/hexanes, CAM Stain);  **$^1H$  NMR** (600 MHz,  $CDCl_3$ )  $\delta$  7.42 (d,  $J$  = 6.9 Hz, 4H, both diastereomers), 7.38 (t,  $J$  = 7.7 Hz, 4H, both diastereomers), 7.34–7.29 (m, 2H, both diastereomers), 7.11 (d,  $J$  = 8.5 Hz, 4H, both diastereomers), 6.90 (d,  $J$  = 8.5 Hz, 4H, both diastereomers), 5.54–5.21 (m, 4H, both diastereomers), 5.04 (s, 4H, both diastereomers), 4.75 (quint,  $J$  = 6.0 Hz, 2H, both diastereomers), 2.98 (s, 6H, both diastereomers), 2.75–2.58 (m, 4H, both diastereomers), 2.11–1.91 (m, 12H, both diastereomers), 1.80–1.68 (m, 4H, both diastereomers), 1.53–1.39 (m, 4H, both diastereomers), 0.96 (td,  $J$  = 7.4, 4.1 Hz, 6H, both diastereomers);  **$^{13}C$  NMR** (151 MHz,  $CDCl_3$ )  $\delta$  157.35 (major diastereomer), 157.34 (minor diastereomer), 137.2 (2C, both diastereomers), 133.4 (minor diastereomer), 133.1 (major diastereomer), 132.7 (2C, both diastereomers), 129.4 (4C, both diastereomers), 128.7 (4C, both diastereomers), 128.3 (minor diastereomer), 128.2 (major diastereomer), 128.1 (2C, both diastereomers), 127.6 (4C, both diastereomers), 115.1 (4C, both diastereomers), 83.43 (minor diastereomer), 84.36 (major diastereomer), 70.2 (2C, both diastereomers), 38.9 (2C both diastereomers), 36.49 (major diastereomer), 36.47 (minor diastereomer), 34.13 (major diastereomer), 34.00 (minor diastereomer), 32.24 (minor

diastereomer), 30.51 (minor diastereomer), 30.50 (major diastereomer), 26.8 (major diastereomer), 25.7 (minor diastereomer), 25.1 (major diastereomer), 24.9 (minor diastereomer), 20.69 (major diastereomers), 14.47 (major diastereomer), 14.06 (minor diastereomer); **HRMS** (TOF MS ES+)  $m/z$ :  $[M+Na]^+$  calcd for  $C_{24}H_{32}O_4SNa$ , 439.1919; found 439.1915.

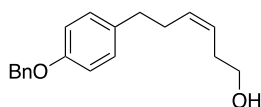
**Scheme 5.10** Synthesis of Primary Mesylate **5.12** for Competition Experiment



**1-(Benzyloxy)-4-iodobenzene (5.29)** refer above for synthesis and characterization data.



**3-(4-(Benzyloxy)phenyl)propanal (5.30)** refer above for synthesis and characterization data.

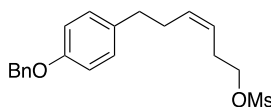


**(4-(benzyloxy)phenyl)hex-3-en-1-ol (5.54)** was prepared according to a procedure reported by Liu.<sup>55</sup> To a flame dried round bottom flask was added Wittig salt **5.24** (0.45 g, 1.1 mmol, 1.2 equiv) and THF (2.4 mL). The flask was cooled to  $-20\text{ }^{\circ}\text{C}$ <sup>56</sup> and LiHMDS (0.36 mg, 2.2 mmol, 2.3 equiv)

<sup>55</sup> Wang, Y.; Jiang, M.; Liu, J.-T. *Adv. Synth. Catal.* **2016**, 358, 1322–1327.

<sup>56</sup> A  $-20\text{ }^{\circ}\text{C}$  bath was prepared using ice water and NaCl.

was added as a solution in PhMe (1.0 M) dropwise via syringe. The dark orange suspension was allowed to stir at  $-20\text{ }^{\circ}\text{C}$  for 1 h, then aldehyde **5.30** (0.27 g, 1.1 mmol, 1.0 equiv) was added as a solution in THF (1.0 mL) dropwise. The reaction mixture was allowed to stir at  $-20\text{ }^{\circ}\text{C}$  for 2 h and then slowly allowed to warm to rt overnight. To quench, saturated aq  $\text{NH}_4\text{Cl}$  was added dropwise. The biphasic mixture was transferred to a separatory funnel and extracted with EtOAc ( $\times 3$ ). The combined organic layers were washed with  $\text{H}_2\text{O}$  and brine, dried over  $\text{Na}_2\text{SO}_4$ , filtered, and concentrated in vacuo. Purification by column chromatography (0–20% EtOAc/hexanes) afforded the title compound as a white solid (140 mg, 0.50 mmol, 45% yield, 1.5:1 dr). **TLC**  $R_f = 0.3$  (30% EtOAc/hexanes, CAM Stain);  **$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.50–7.29 (m, 10H, both diastereomers), 7.11 (dd,  $J = 8.7, 7.1$  Hz, 4H, both diastereomers), 6.93 (d,  $J = 8.4$  Hz, 4H, both diastereomers), 5.66–5.53 (m, 2H, both diastereomers), 5.49–5.32 (m, 2H, both diastereomers), 5.06 (s, 4H, both diastereomers), 3.59 (dt,  $J = 10.5, 6.4$  Hz, 4H, both diastereomers), 2.65 (td,  $J = 7.6, 4.9$  Hz, 4H, both diastereomers), 2.32 (ddt,  $J = 37.5, 12.4, 6.8$  Hz, 8H, both diastereomers);  **$^{13}\text{C}$  NMR** (101 MHz,  $\text{CDCl}_3$ )  $\delta$  157.2 (2C, both diastereomers), 137.4 (2C, both diastereomers), 137.4 (minor diastereomer), 133.3 (major diastereomer), 132.2 (2C, both diastereomers), 129.5 (4C, both diastereomers), 128.7 (4C, both diastereomers), 128.0 (2C, both diastereomer), 127.6 (4C, both diastereomers), 126.8 (major diastereomer), 126.0 (minor diastereomer), 114.9 (4C, both diastereomers), 70.2 (2C, both diastereomers), 62.3 (minor diastereomer), 62.1 (major diastereomer), 36.1 (2C, both diastereomers), 35.1 (major diastereomer), 34.8 (minor diastereomer), 30.9 (minor diastereomer), 29.6 (major diastereomer); **HRMS** (TOF MS ES+)  $m/z$ :  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{19}\text{H}_{22}\text{O}_2\text{Na}$ , 305.1518; found 305.1521.



**(4-(benzyloxy)phenyl)hex-3-en-1-yl methanesulfonate (5.12)** was prepared according to Method H. The following amounts of reagents were used: alcohol **5.54** (140 mg, 0.50 mmol, 1.0 equiv), MsCl (60.  $\mu$ L, 0.74 mmol, 1.5 equiv), Et<sub>3</sub>N (0.10 mL, 0.74 mmol, 1.5 equiv) and CH<sub>2</sub>Cl<sub>2</sub> (2.5 mL). Purification by column chromatography (0–30% EtOAc/hexanes) afforded the title compound as a yellow oil (120 mg, 0.33 mmol, 68% yield, 1.5:1 dr). **TLC** *R<sub>f</sub>* = 0.5 (50% EtOAc/hexanes, CAM Stain); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.43 (d, *J* = 7.2 Hz, 4H, both diastereomers), 7.38 (t, *J* = 7.2 Hz, 4H, both diastereomers), 7.34–7.28 (m, 2H, both diastereomers), 7.08 (dd, *J* = 7.2, 4.8 Hz, 4H, both diastereomers), 6.90 (d, *J* = 8.5 Hz, 4H, both diastereomers), 5.66–5.53 (m, 2H, both diastereomers), 5.43–5.31 (m, 2H, both diastereomers), 5.04 (s, 4H, both diastereomers), 4.18 (t, *J* = 6.8 Hz, 2H, both diastereomers), 4.05 (t, *J* = 6.9 Hz, 2H, both diastereomers), 2.95 (s, 3H, major diastereomer), 2.94 (s, 3H, minor diastereomer), 2.62 (t, *J* = 7.6 Hz, 4H, both diastereomers), 2.46–2.25 (m, 8H, both diastereomers); **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>)  $\delta$  157.23 (minor diastereomer), 157.17 (major diastereomer), 137.34 (major diastereomer), 137.31 (minor diastereomer), 134.2 (major diastereomer), 134.1 (minor diastereomer), 134.0 (major diastereomer), 133.0 (minor diastereomer), 129.6 (2C, minor diastereomer), 129.5 (2C, major diastereomers), 128.7 (4C, both diastereomers), 128.01 (minor diastereomer), 128.00 (major diastereomer), 127.59 (2C, minor diastereomer), 127.58 (2C, minor diastereomer), 124.4 (major diastereomer), 123.7 (minor diastereomer), 114.9 (2C, minor diastereomer), 114.8 (2C, major diastereomer), 70.2 (2C, both diastereomers), 69.6 (major diastereomer), 69.3 (minor diastereomer), 37.56 (minor diastereomer), 37.53 (minor diastereomer), 34.89 (major diastereomer), 34.81 (major diastereomer), 34.6 (major diastereomer), 32.5 (minor diastereomer), 29.6 (major diastereomer), 27.43 (minor diastereomer); **HRMS** (TOF MS ES+) *m/z*: [M+Na]<sup>+</sup> calcd for C<sub>20</sub>H<sub>24</sub>O<sub>4</sub>SNa, 378.1739; found 378.1747.

APPENDIX A.1

$^1\text{H}$ ,  $^{13}\text{C}$ , COSY, NOE NMR SPECTRA FOR CHAPTER 1



**1H spectrum**



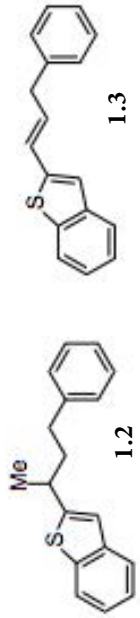
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 SFO1: 500.130761 MHz  
 SOLVENT: CDCl3  
 NS: 2  
 DS: 2  
 SWH: 8012.820 Hz  
 FIDRES: 0.186677 Hz  
 AQ: 2.939571 sec  
 RG: 7.1  
 DM: 62.400 MHz  
 DE: 6.00 MHz  
 TE: 296.2 K  
 T1: 0.10000000 sec  
 T1RHO: 0.00000000 sec  
 T2: 0.00000000 sec  
 T2RHO: 0.00000000 sec  
 MCHRG: 0.03500000 sec

===== CHANNEL f1 =====  
 NUC1: 1H  
 P1: 7.50 MHz  
 PL1: 1.40 dB  
 SFO1: 500.225015 MHz

F2 - Processing parameters  
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 SF: 500.2200000 MHz  
 WDW: 0  
 SSB: 0  
 LB: 0.00 Hz  
 GB: 0  
 PC: 1.00

1D NMR plot parameters  
 CX: 22.80 cm  
 CY: 15.00 cm  
 CZ: 0.00 cm  
 FI: 450.00 ppm  
 F2: -0.500 ppm  
 FZ: -250.11 Hz  
 PH0CH: 0.41667 ppm/cm  
 HZCH: 208.42500 Hz/cm

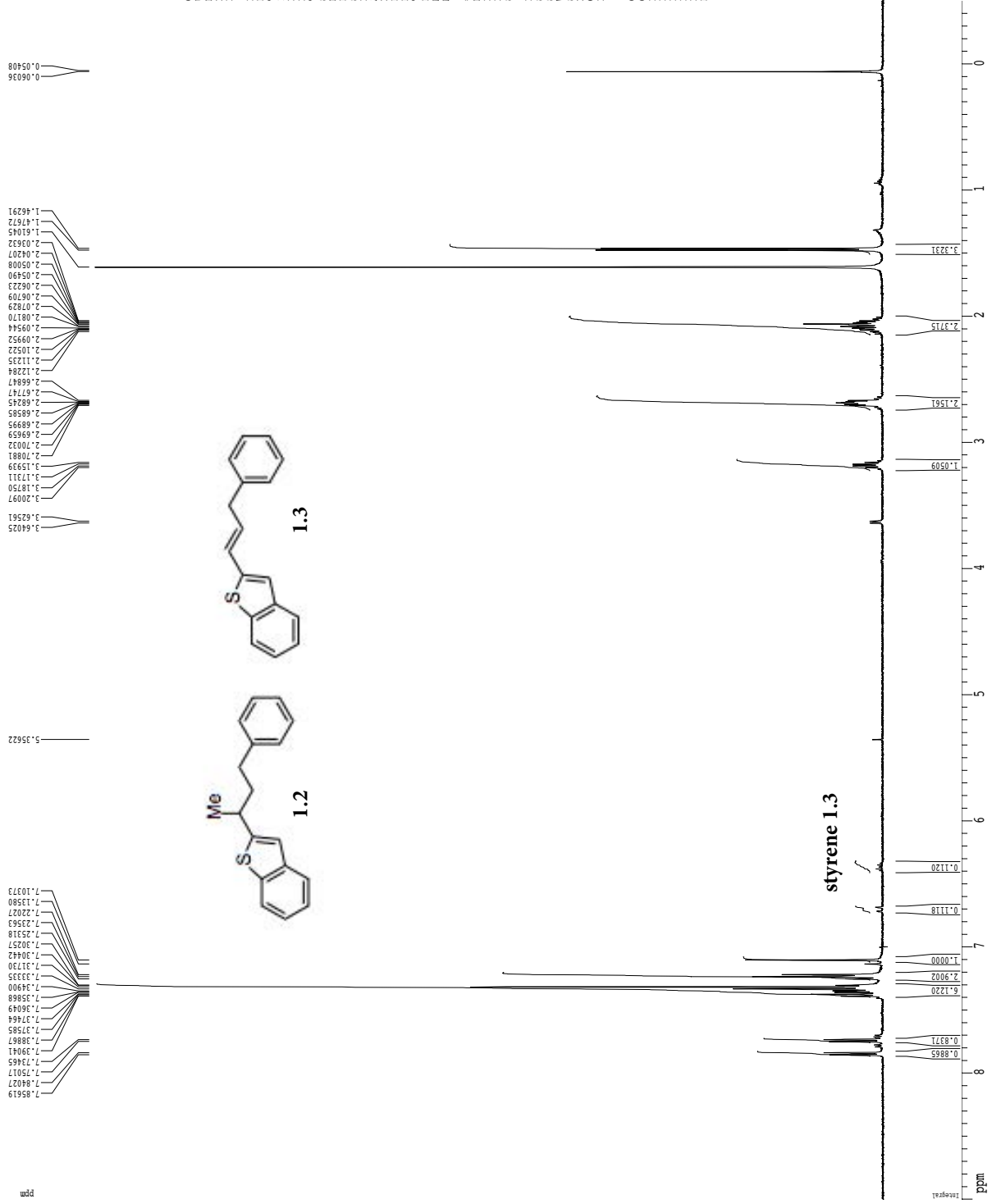


**1.2**

**styrene 1.3**



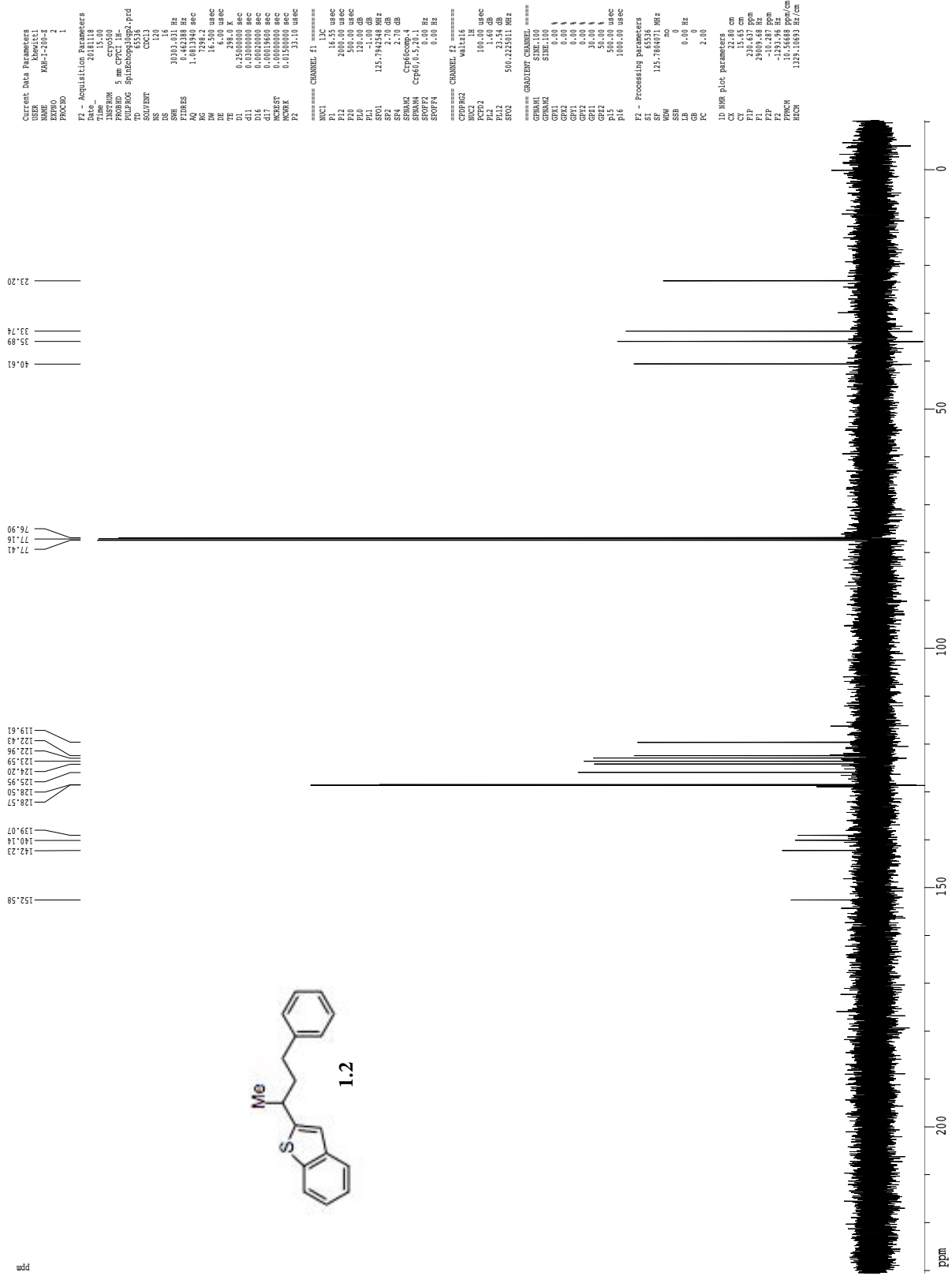
1H spectrum



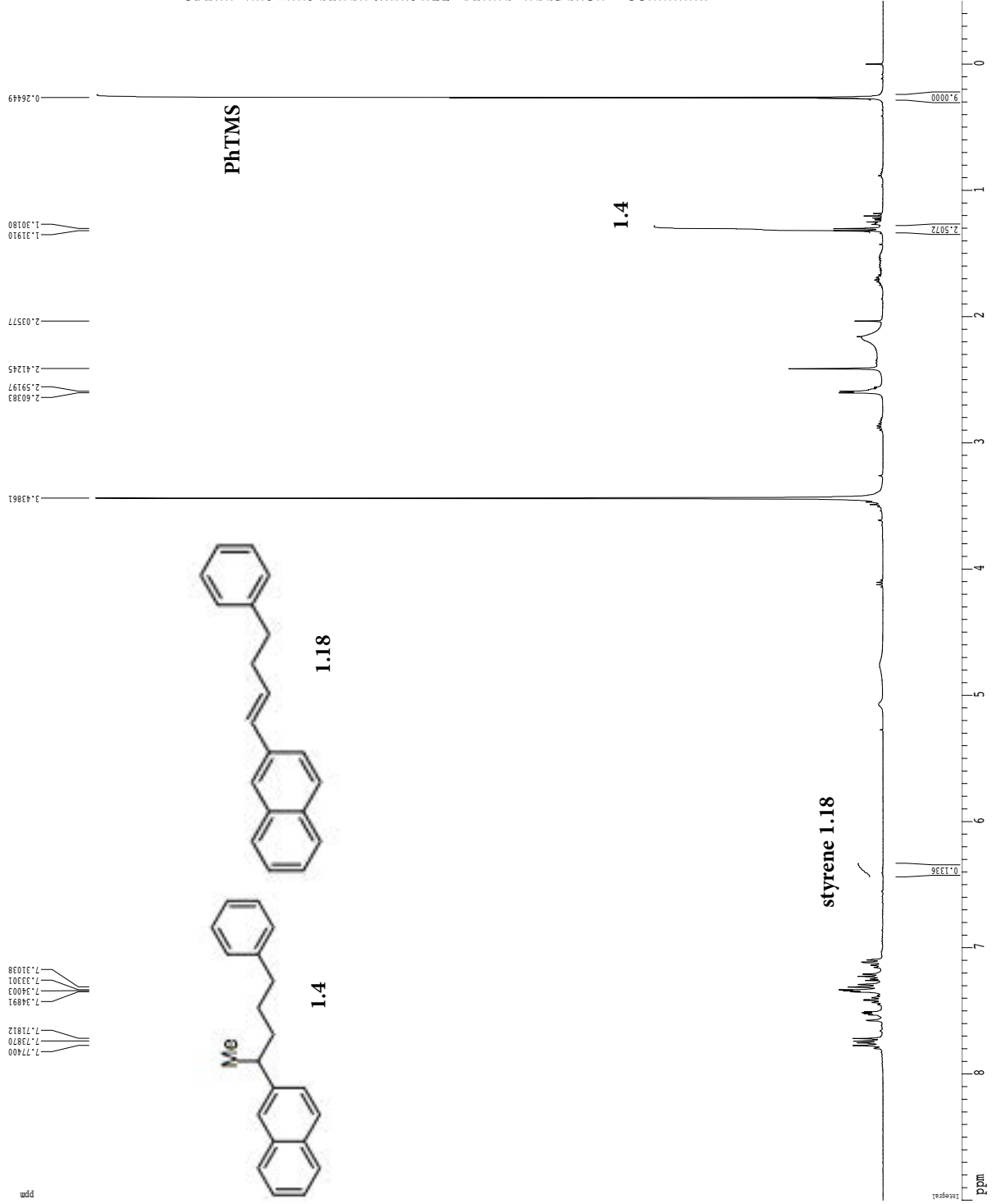
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PULPROG: zg30  
TD: 48074  
SOLVENT: CDCl3  
DS: 9  
SHE: 8012.820 Hz  
FIDRES: 0.166677 Hz  
AQ: 2.3998677 sec  
RG: 62.400 usec  
DE: 6.400 usec  
TE: 298.2 K  
T1: 0.10000000 sec  
MCRET: 0.00000000 sec  
MORCK: 0.00500000 sec  
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NUC1: 13C  
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PL1: -6.00 dB  
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SI: 65536  
SF: 498.6500000 MHz  
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SSB: 0.00 Hz  
GB: 0  
PC: 1.00  
LD: WDR  
ID: WDR  
CQ: 22.80 cm  
CY: 15.00 cm  
FIP: 9.000 ppm  
FL: 4487.85 Hz  
F2: -246.3 Hz  
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P2: 0.41667 ppm/cm  
PCMC: 207.77084 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling

81

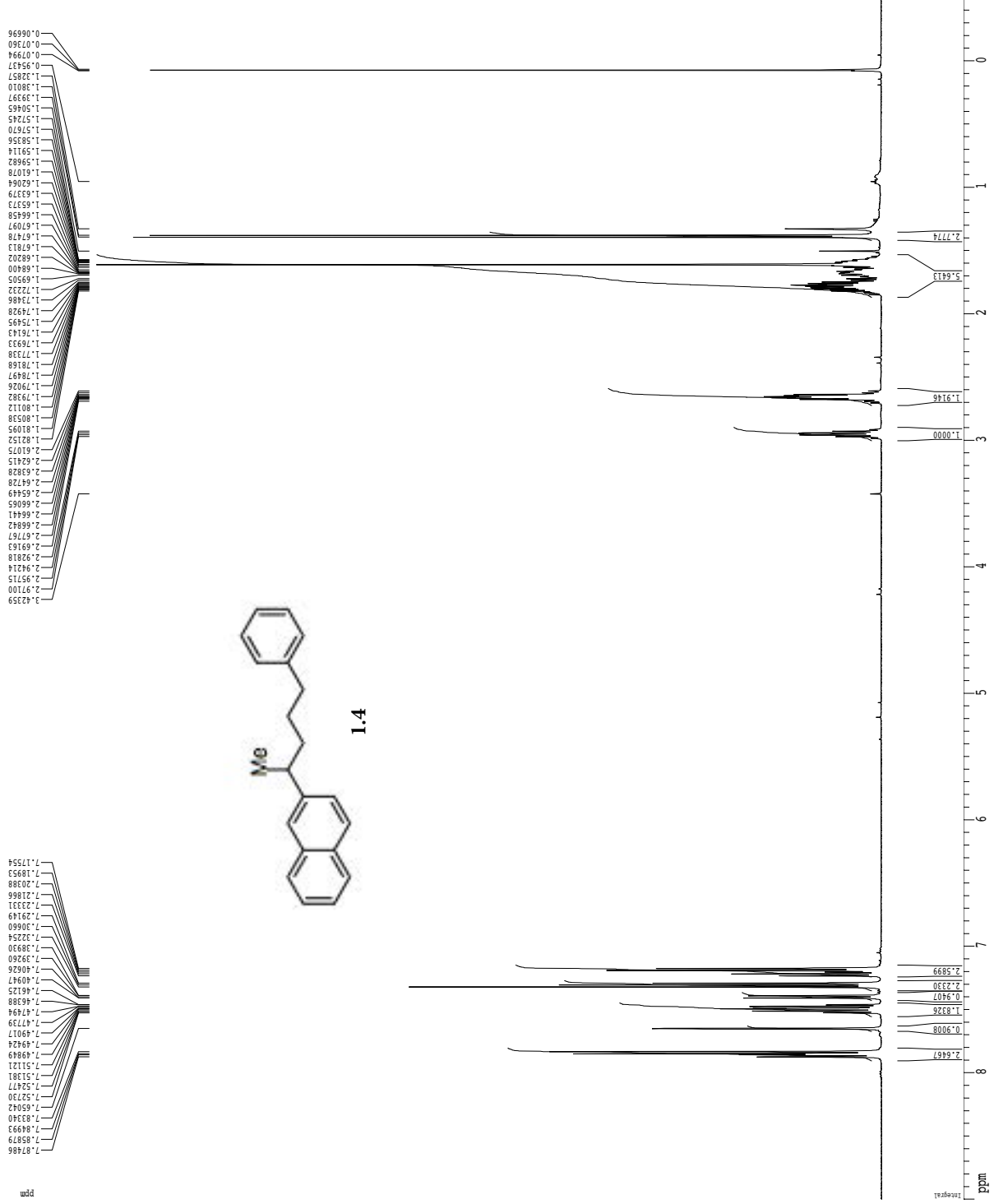


**<sup>1</sup>H spectrum**



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 Time 9:26  
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 TD 65536  
 SFO1 400.1228009 MHz  
 SOLVENT CDCl3  
 NS 2  
 DS 2  
 SWH 6410.256 Hz  
 FIDRES 0.097813 Hz  
 AQ 5.1118379 sec  
 RG 327.5  
 DW 78.400 usec  
 DE 4.50 usec  
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 TMRK 0.01500000 sec  
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 BECN 1667.72866 Hz/cm

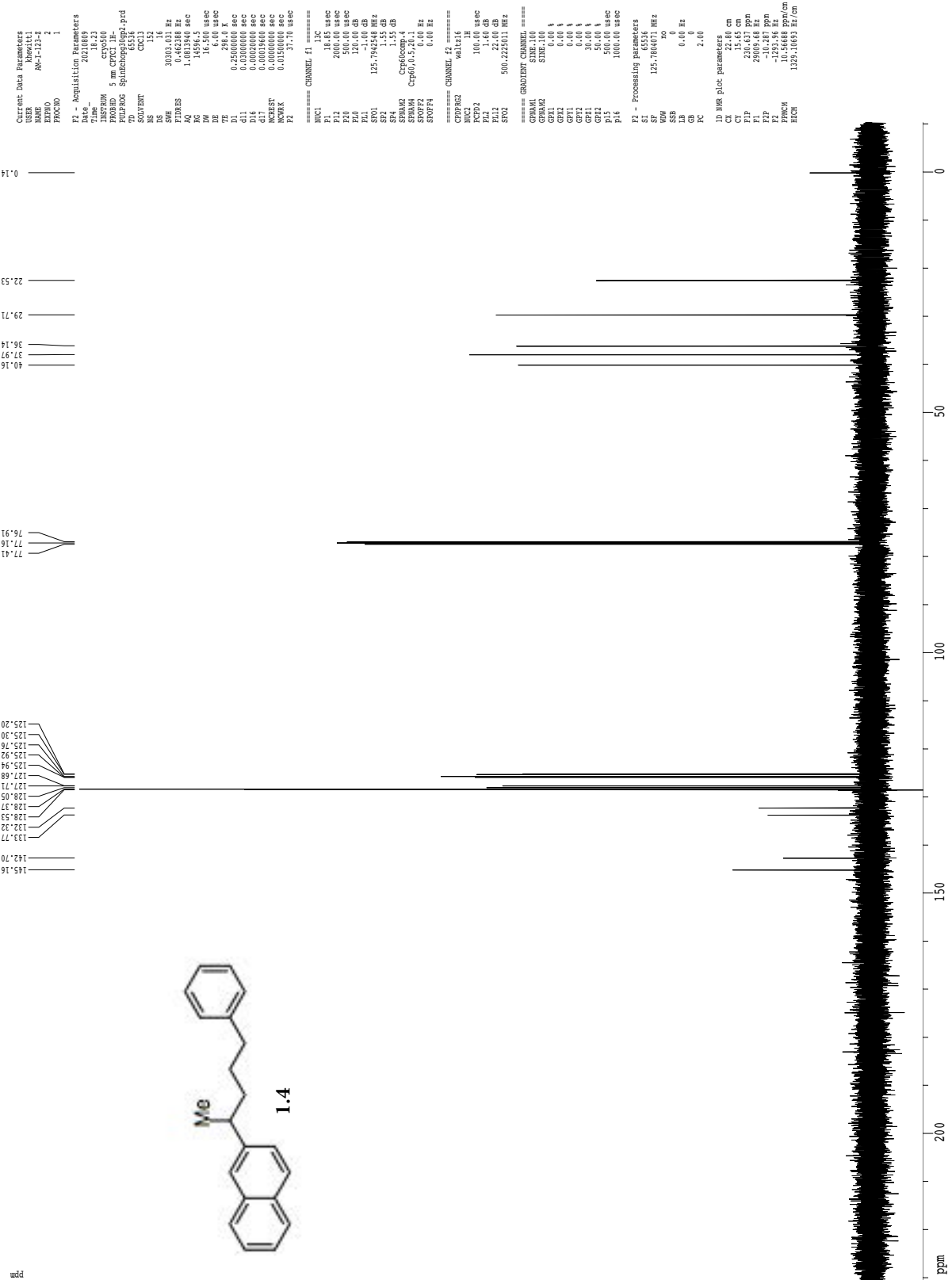
1H spectrum

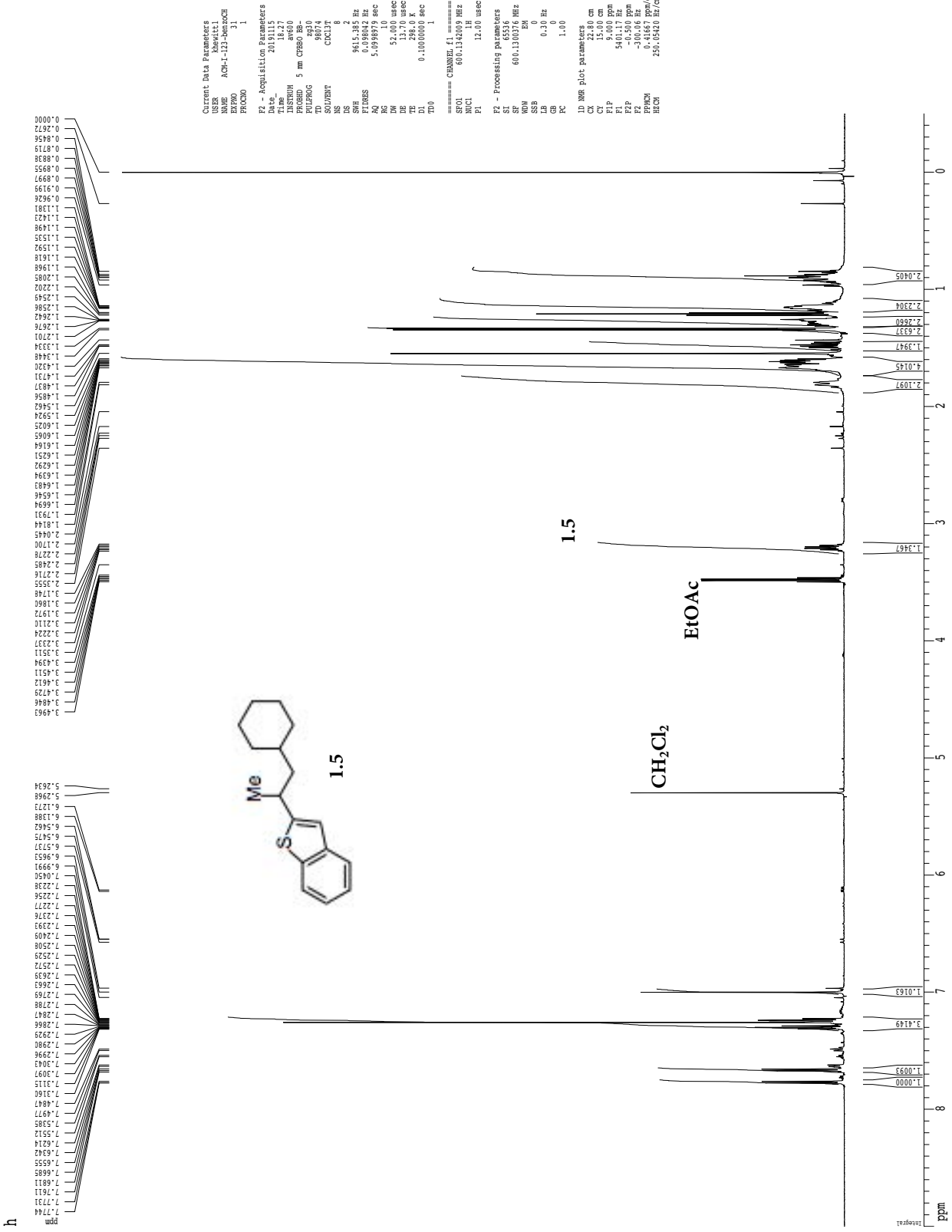


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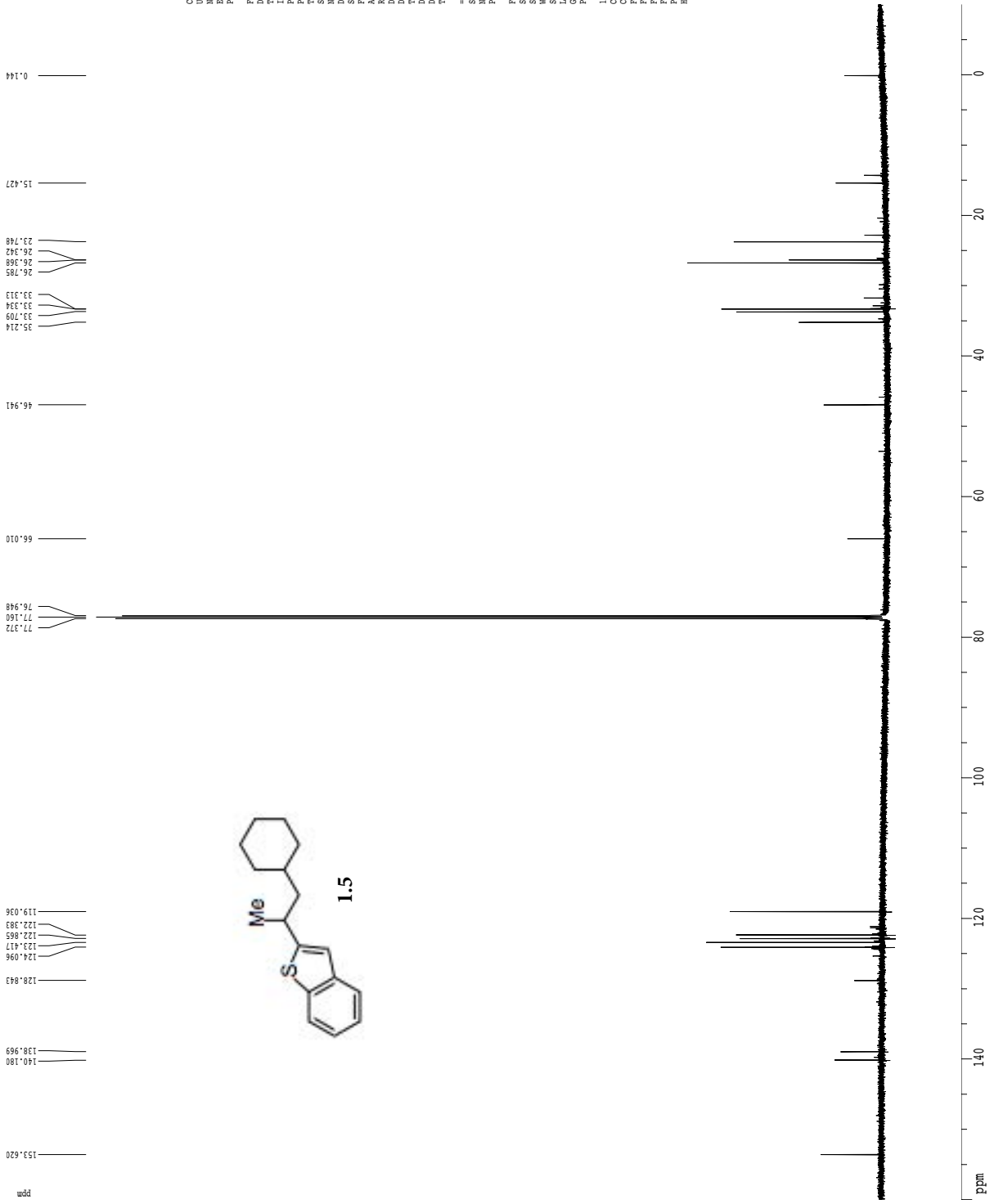
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 SOLVENT: CDCl3  
 DS: 9  
 SFO: 500.136260 MHz  
 SFE: 8012.820 Hz  
 FIDRES: 0.166677 Hz  
 AQ: 2.3398677 sec  
 RG: 327.500  
 DD: 62.400 usec  
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Z-restored spin-echo 13C spectrum with 1H decoupling



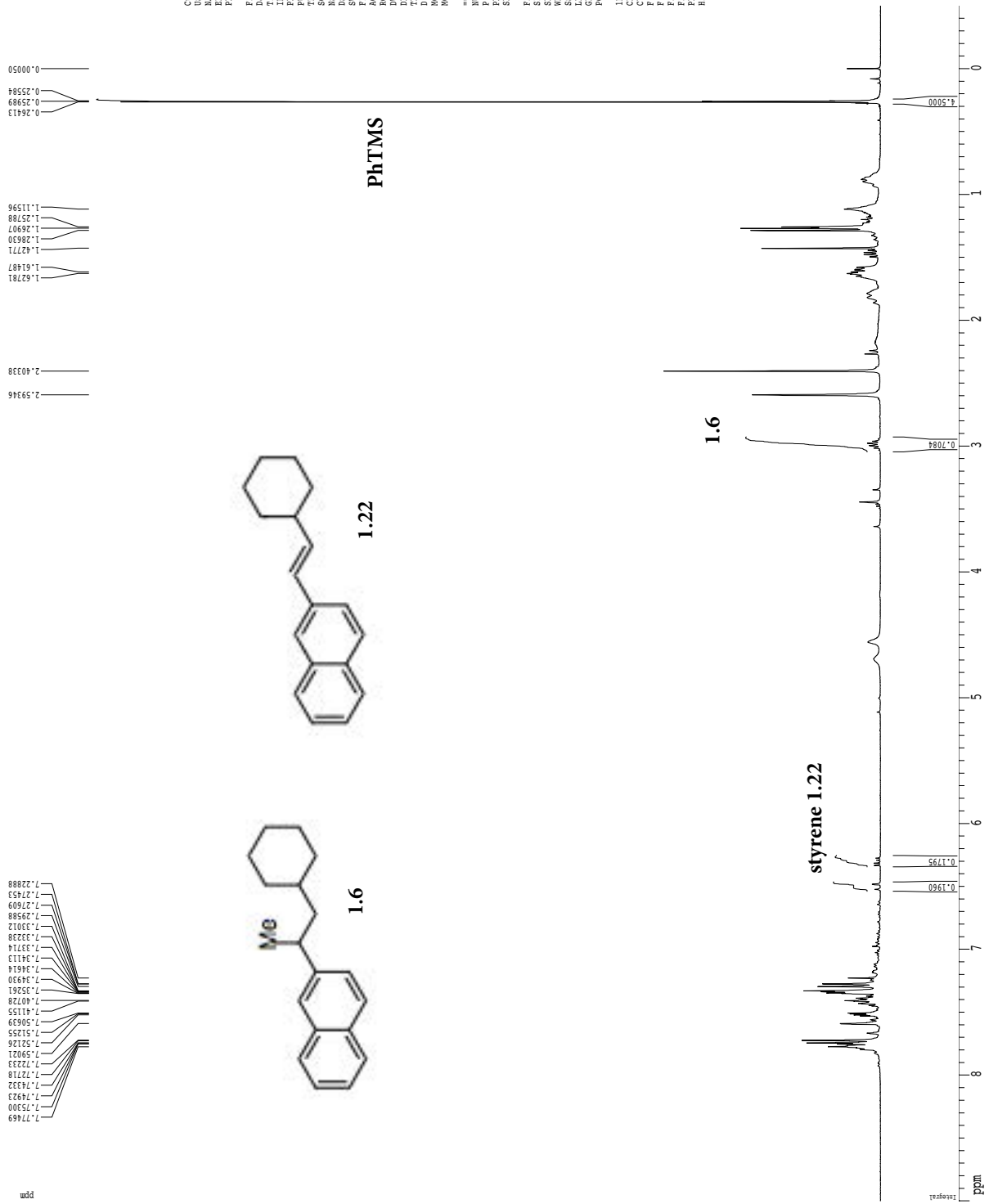


**<sup>13</sup>C spectrum with <sup>1</sup>H decoupling**

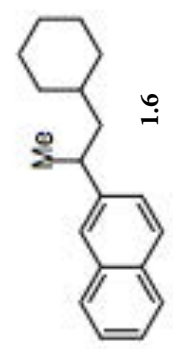
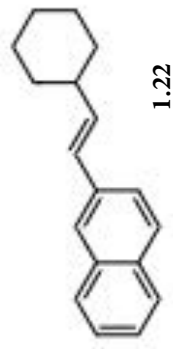




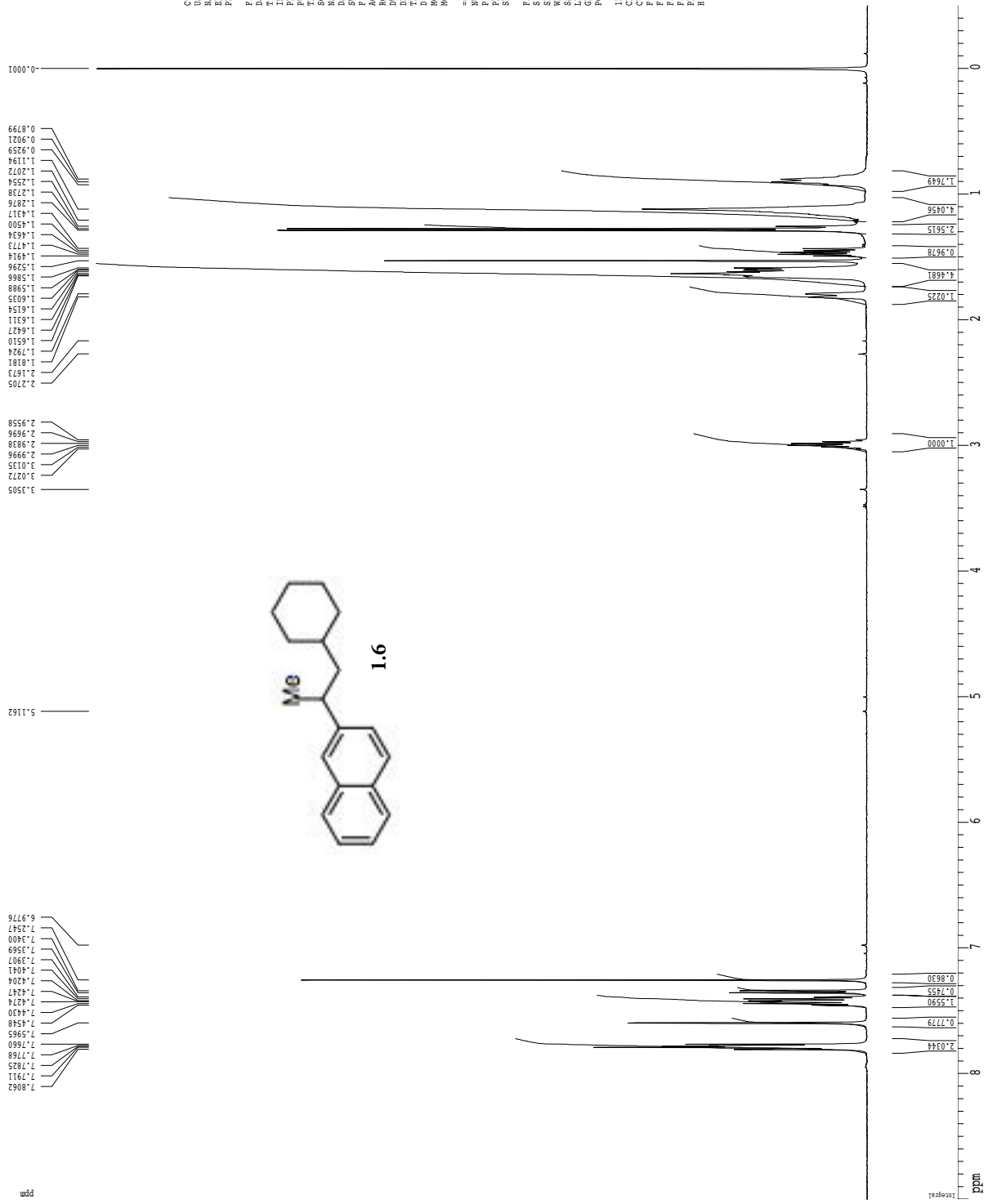
1H spectrum



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 AQ: 5.1118579 sec  
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 GB: 0.00 Hz  
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 F2: -2000.00 Hz  
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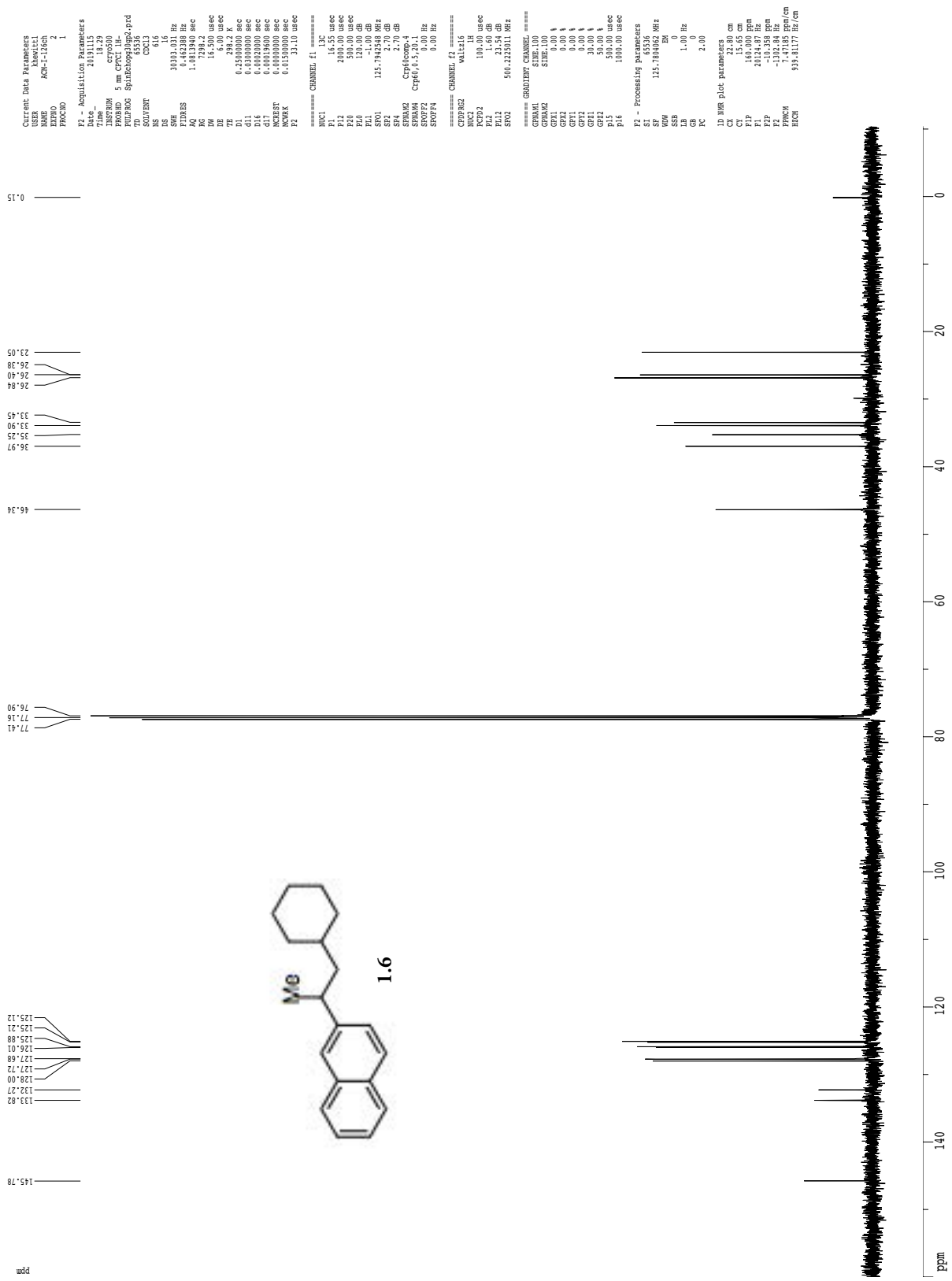
1H spectrum



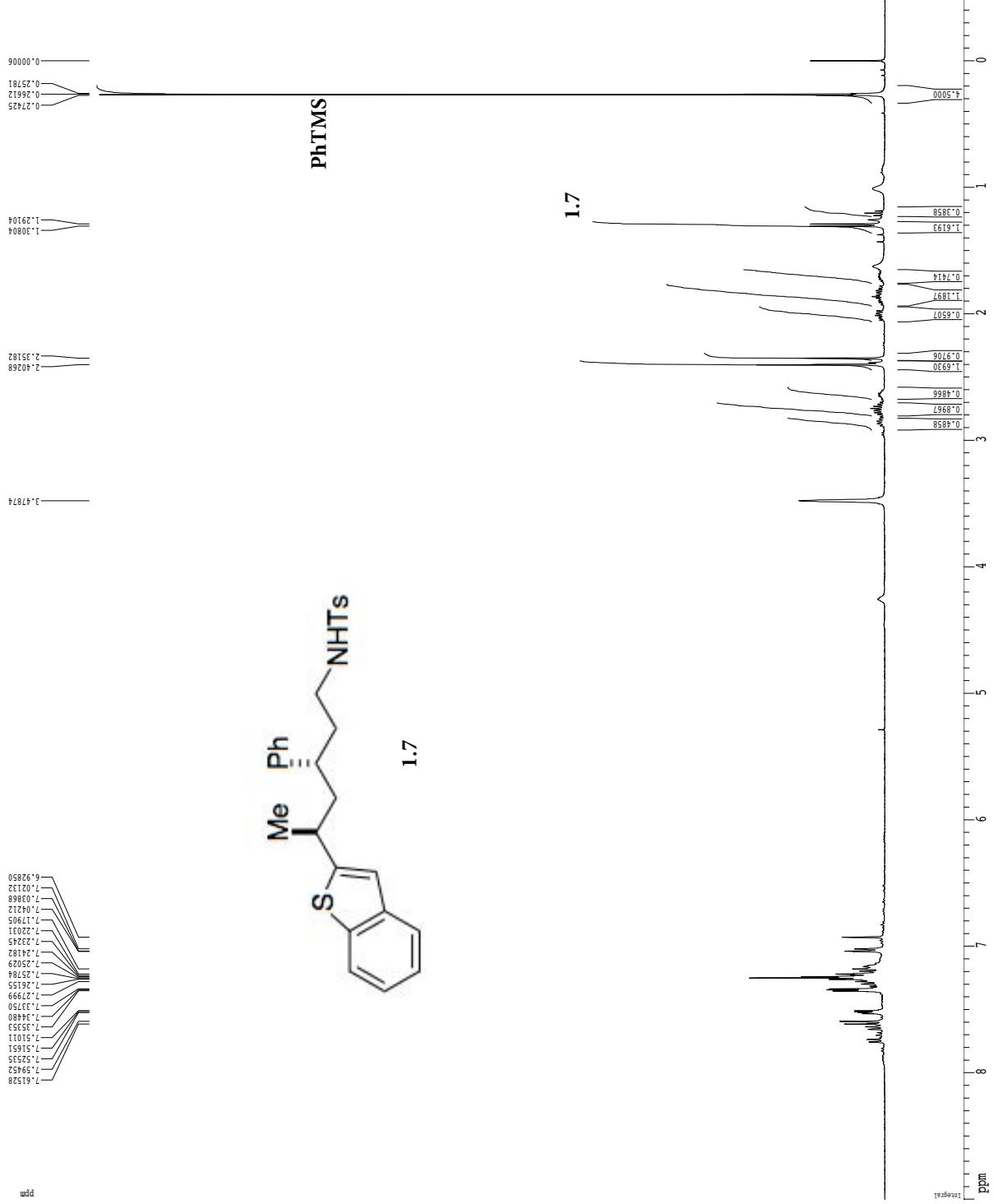
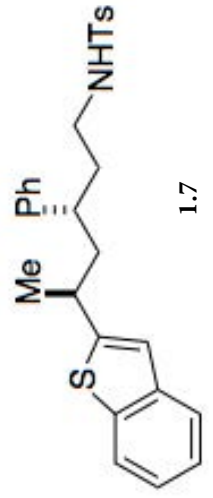
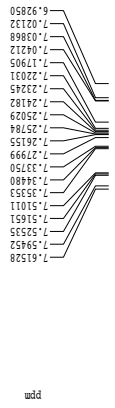
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 PULPROG: zg30  
 TD: 8178  
 SOLVENT: CDCl3  
 NS: 0  
 DS: 0  
 SFO: 8012.820 Hz  
 FIDRES: 0.09804 Hz  
 AQ: 5.199877 sec  
 RG: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
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Z-restored spin-echo <sup>13</sup>C spectrum with <sup>1</sup>H decoupling

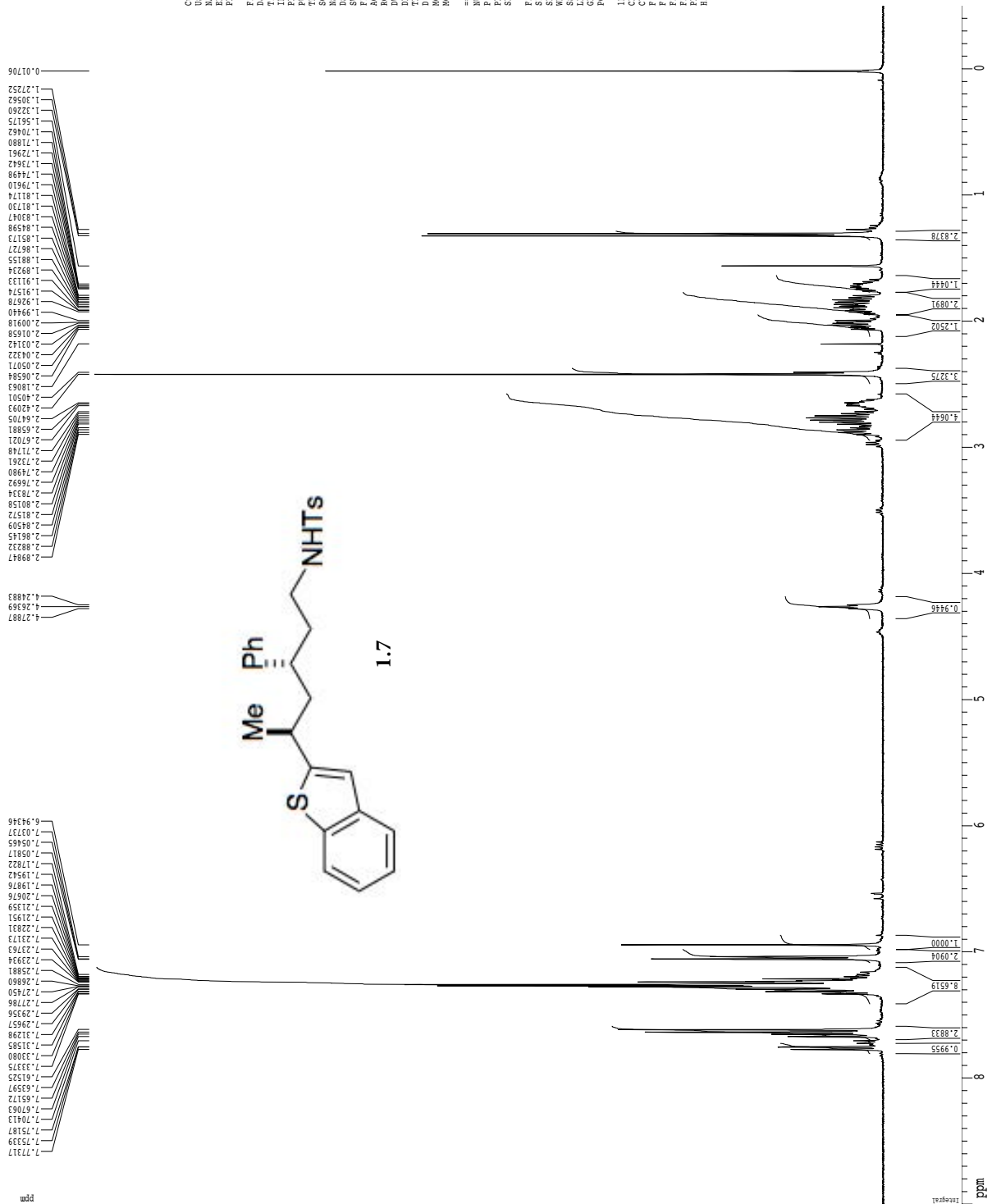


1H spectrum



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 PROCNO 1  
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 SOLVENT CDCl3  
 DS 9  
 SFO 2  
 SFE 6410.256 Hz  
 FIDRES 0.166673 Hz  
 AQ 2.399729 sec  
 RG 327.5  
 DS 78.000 usec  
 DE 4.50 usec  
 TE 298.2 K  
 T1 1.000000 sec  
 T2 0.000000 sec  
 T3 0.000000 sec  
 MCHRES 0.0150000 Hz  
 HZRES 0.0150000 Hz  
 ===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.1328009 MHz  
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 PC 2.00  
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 CY 22.80 cm  
 CV 15.00 cm  
 FIP 9.000 ppm  
 FL 3600.17 Hz  
 F2 2000.00 Hz  
 F3 -2000.00 Hz  
 PPMCM 0.41667 ppm/cm  
 HZCM 166.72086 Hz/cm

**1H spectrum**



Current Data Parameters  
 USER KMH-222-2  
 NAME KMH-222-2  
 EXPO 1  
 PROC 1

F2 - Acquisition Parameters  
 Date\_ 20190329  
 Time 15:37  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/13  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 DS 9  
 SFO 6410.258 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.339229 sec  
 RG 327.5  
 DW 78.000 usec  
 DE 4.450 usec  
 TE 298.1 K  
 T1 0.100000 sec  
 T1RHO 0.000000 sec  
 MCRST 0.000000 sec  
 MCRCK 0.0150000 sec

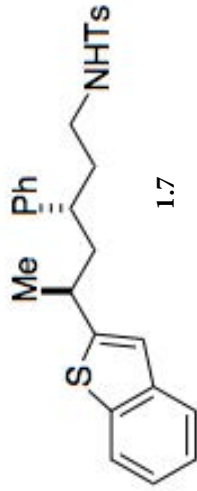
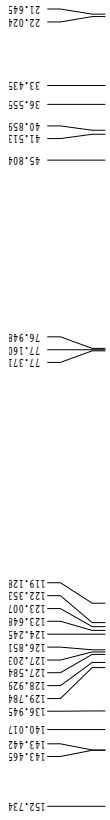
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.1328009 MHz

F2 - Processing Parameters  
 SI 65536  
 SF 400.1300175 MHz  
 WDW no  
 GB 0  
 CB 0  
 PC 2.00

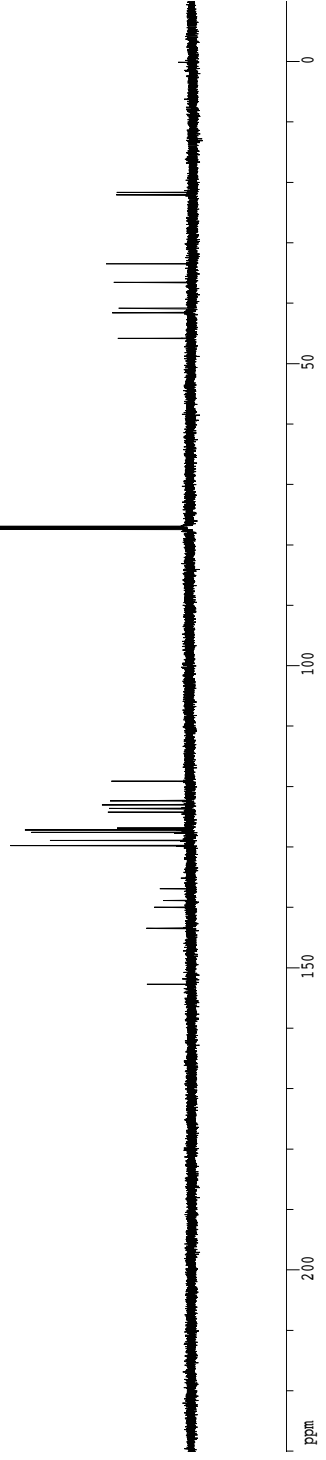
F3 NMR file parameters  
 CY 22.80 cm  
 F1 15.00 cm  
 FIP 9.000 ppm  
 F2 3600.17 Hz  
 F3 2000.00 Hz  
 PPM1 0.41667 ppm/cm  
 PPM2 166.72084 Hz/cm

c13.c

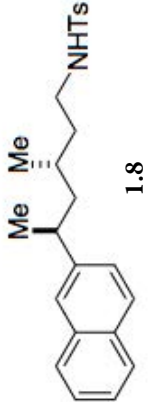
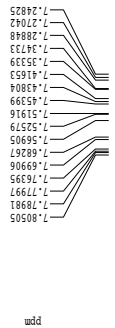
ppm



Current Data Parameters  
USER khewitt1  
NAME KM-1-222-5-C  
EXPNO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20101113  
Time 14:11  
INSTRUM av600  
PROBHD 5 mm CPBBO BB-  
PULPROG zgpg30  
PCPDPR 4  
SOLVENT CDCl3  
NS 446  
DS 4  
SHF 502.11882 MHz  
NUC1 13C  
P1 10.10 usec  
SFO1 150.9194080 MHz  
NUC2 13C  
P2 1.00 usec  
F3 - Processing parameters  
SI 65536  
SF 150.9027858 MHz  
WDW no  
SSB 0  
GB 0  
PC 1.00  
D0 0.00 Hz  
D1 0.4000000 sec  
D2 0.0300000 sec  
TD 1  
===== CHANNEL f1 =====  
SFO1 150.9194080 MHz  
NUC1 13C  
P1 10.10 usec  
F3 - Processing parameters  
SI 65536  
SF 150.9027858 MHz  
WDW no  
SSB 0  
GB 0  
PC 1.00  
D0 0.00 Hz  
D1 0.4000000 sec  
D2 0.0300000 sec  
TD 1  
===== CHANNEL f2 =====  
SFO2 150.9194080 MHz  
NUC2 13C  
P2 1.00 usec  
F3 - Processing parameters  
SI 65536  
SF 150.9027858 MHz  
WDW no  
SSB 0  
GB 0  
PC 1.00  
D0 0.00 Hz  
D1 0.4000000 sec  
D2 0.0300000 sec  
TD 1



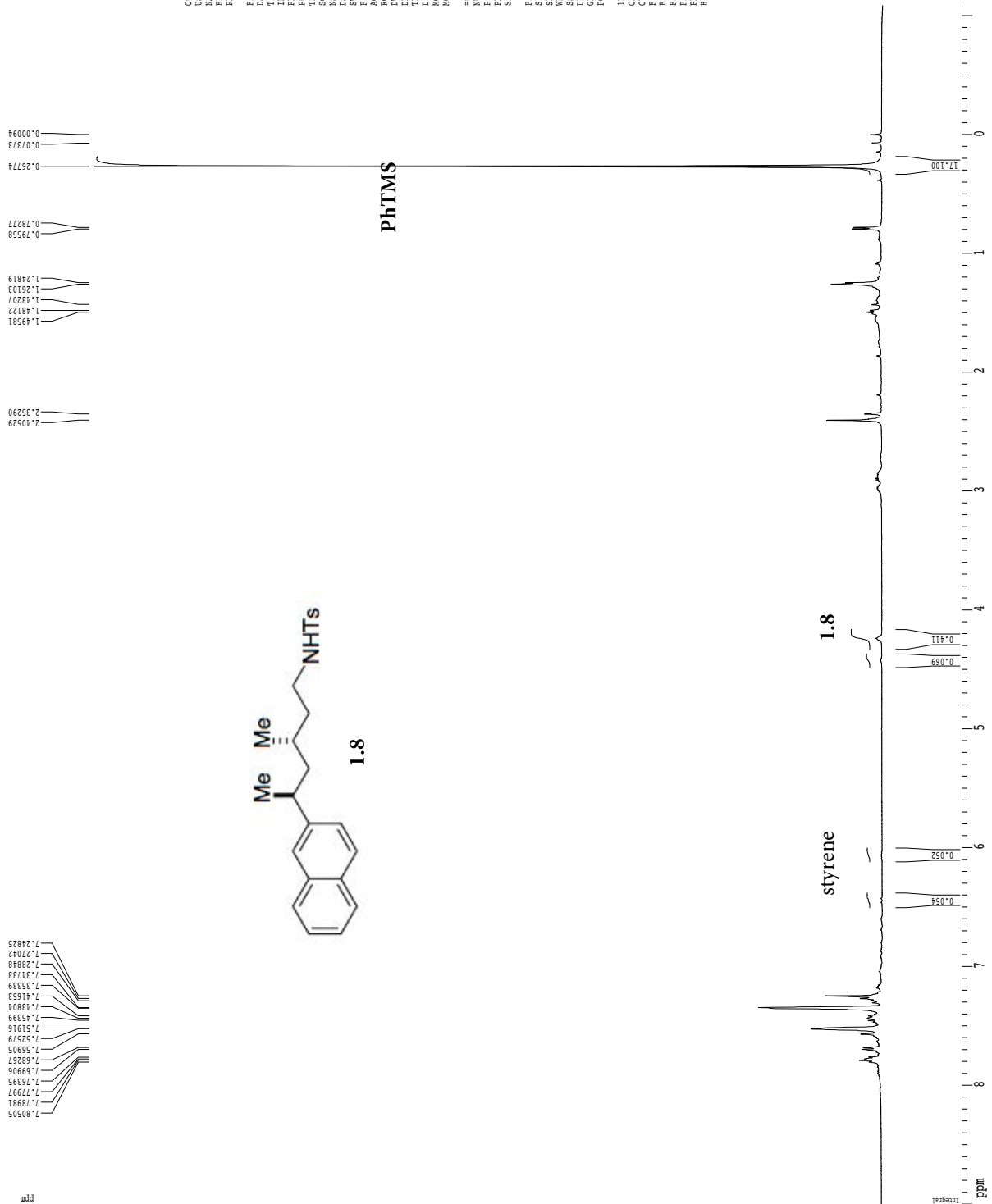
1H spectrum



PhTMS

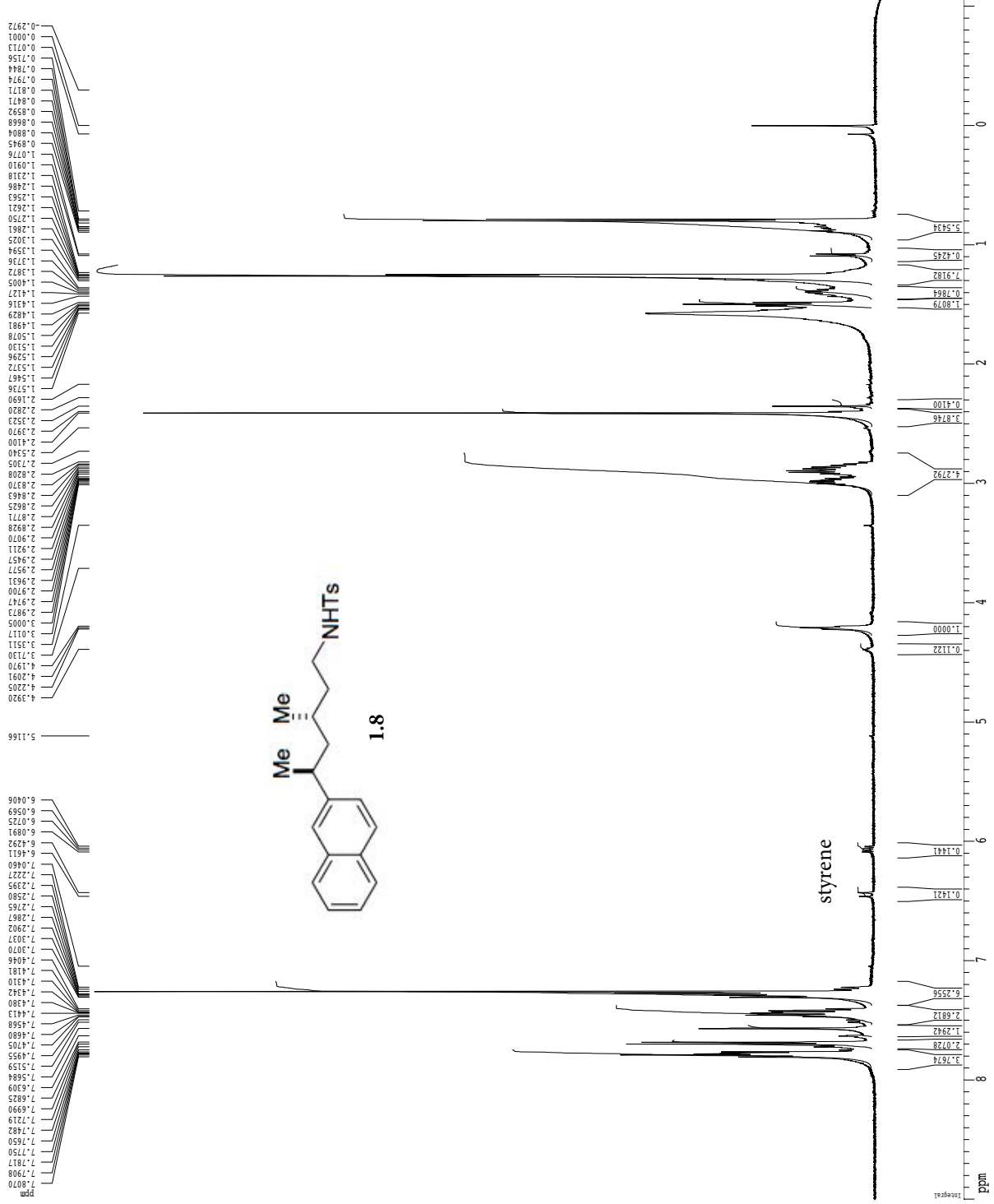
styrene

1.8



Current Data Parameters  
 USER: cshelmer  
 NAME: CSH-4-247-Grude  
 EXNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20210110  
 Time: 11:36  
 SYSTEM: cryo-1  
 PROBHD: 5 mm CRYO-H  
 PULPROG: zgpg30  
 TD: 81728  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 8012.820 Hz  
 FIDRES: 0.098044 Hz  
 AQ: 5.1998774 sec  
 RG: 62.400 usec  
 DE: 6.000 usec  
 TE: 298.2 K  
 T1: 1.0000000 sec  
 T2: 0.0000000 sec  
 T3: 0.0000000 sec  
 T4: 0.0000000 sec  
 T5: 0.0000000 sec  
 T6: 0.0000000 sec  
 T7: 0.0000000 sec  
 T8: 0.0000000 sec  
 T9: 0.0000000 sec  
 T10: 0.0000000 sec  
 T11: 0.0000000 sec  
 T12: 0.0000000 sec  
 T13: 0.0000000 sec  
 T14: 0.0000000 sec  
 T15: 0.0000000 sec  
 T16: 0.0000000 sec  
 T17: 0.0000000 sec  
 T18: 0.0000000 sec  
 T19: 0.0000000 sec  
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 T88: 0.0000000 sec  
 T89: 0.0000000 sec  
 T90: 0.0000000 sec  
 T91: 0.0000000 sec  
 T92: 0.0000000 sec  
 T93: 0.0000000 sec  
 T94: 0.0000000 sec  
 T95: 0.0000000 sec  
 T96: 0.0000000 sec  
 T97: 0.0000000 sec  
 T98: 0.0000000 sec  
 T99: 0.0000000 sec  
 T100: 0.0000000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 9.75 usec  
 PL1: 1.66 dB  
 SFO1: 500.2235015 MHz  
 F2 - Processing Parameters  
 SI: 65536  
 SF: 500.2200370 MHz  
 WDW: no  
 SSB: 0  
 GB: 0.00 Hz  
 PC: 1.00  
 ID: NMR file parameters  
 CY: 22.86 cm  
 CX: 15.00 cm  
 FIP: 9.000 ppm  
 FL: 450.198 Hz  
 F2: 111.825 MHz  
 F3: -541.86 Hz  
 PPMCM: 0.44225 ppm/cm  
 HZCM: 221.22217 Hz/cm

1H spectrum

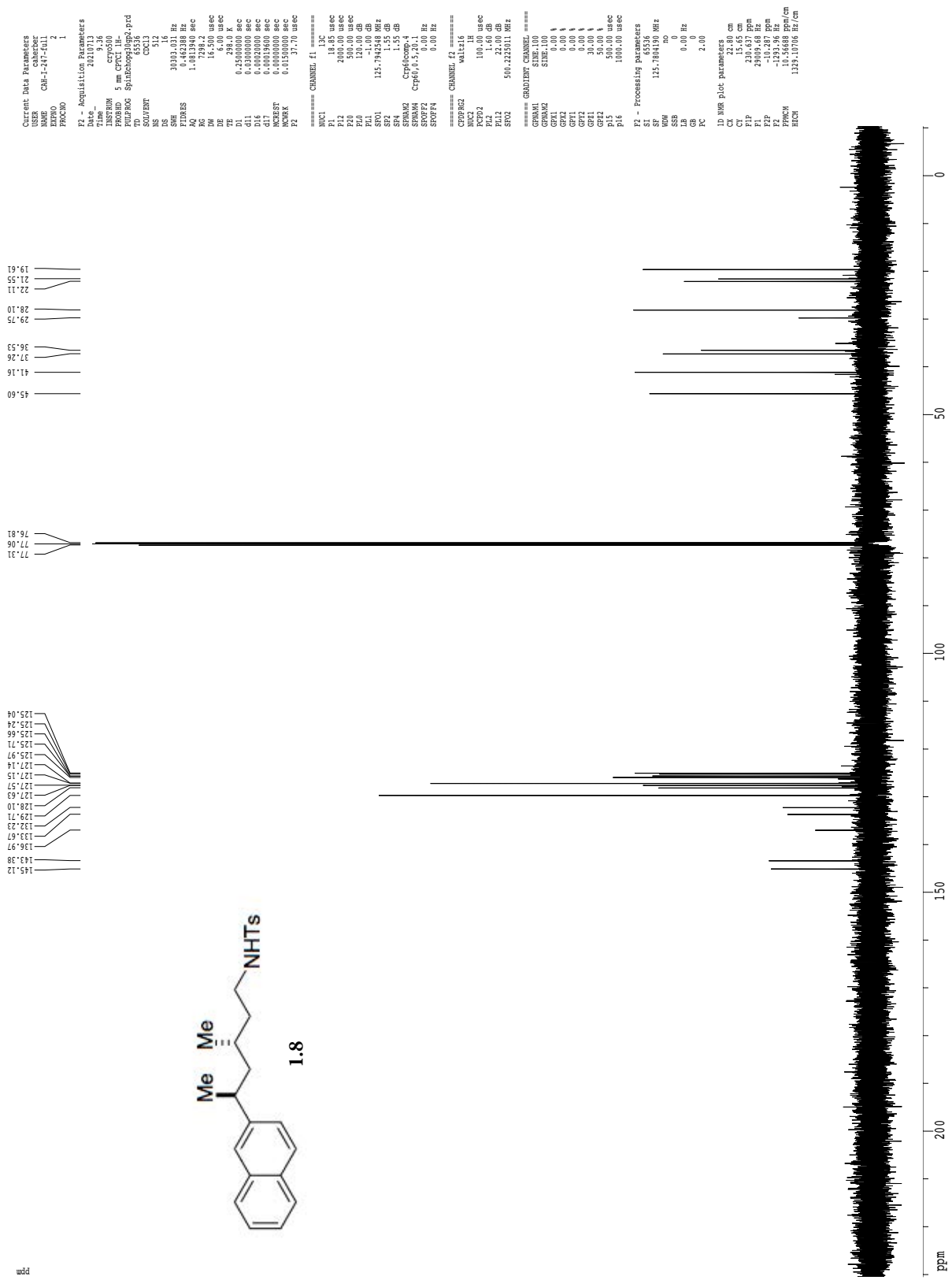
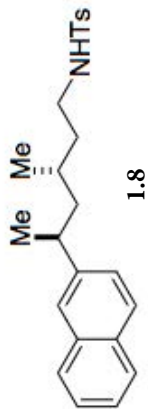


Current Data Parameters  
USER: cubehere  
NAME: CUB-4-217-4ull  
EXPO: 1  
PROCNO: 1  
F2 - Acquisition Parameters  
Date\_: 20210113  
Time: 9:25  
INSTRUM: cryo-1H  
PROBHD: 5 mm CRYO-1H  
PULPROG: zg30  
TD: 8178  
SOLVENT: CDCl3  
DS: 9  
SHE: 8012.820 Hz  
FIDRES: 0.09804 Hz  
AQ: 5.1998774 sec  
RG: 62.400 usec  
DE: 6.00 usec  
TE: 298.2 K  
NUC1: 1H  
MAGNET: 0.1000000 sec  
MCORE: 0.0150000 sec  
===== CHANNEL f1 =====  
NUC1: 1H  
P1: 9.75 usec  
PL1: 1.66 dB  
SFO1: 500.2235015 MHz  
F2 - Processing Parameters  
SI: 65536  
SF: 500.2200315 MHz  
WDW: no  
SSB: 0.00 Hz  
GB: 0  
PC: 1.00  
ID: NMR file parameters  
CY: 22.80 cm  
C1: 15.00 cm  
F1P: 9.000 ppm  
F1: 4501.98 Hz  
F2: -546.38 Hz  
PRGM: 0.44177 ppm/cm  
HZCM: 220.98083 Hz/cm

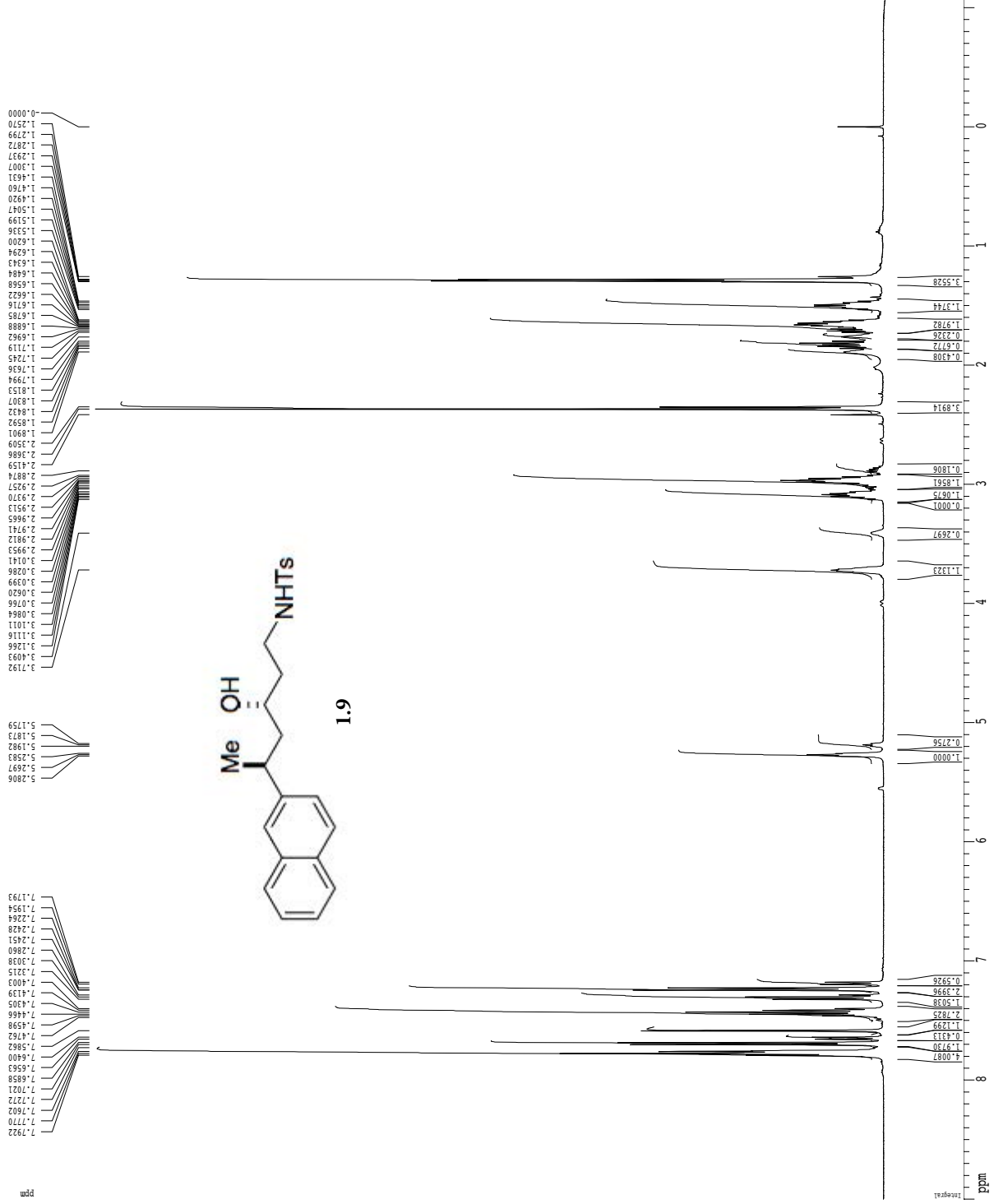


Z-restored spin-echo 13C spectrum with 1H decoupling

81



1H spectrum



Current Data Parameters  
 USER: cubehar  
 EXNO: CUH-4-239-full  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20210623  
 Time: 11.35  
 PROBNM: 077130  
 PROCNO: 5  
 PULPROG: zgpg30  
 TD: 8178  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 500.136260 MHz  
 SHF: 8012.820 Hz  
 FIDRES: 0.098044 Hz  
 AQ: 5.1998774 sec  
 RG: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 INJRES: 0.100000 sec  
 ACQRES: 0.000000 sec  
 SCANS: 1  
 MONRES: 0.0150000 sec

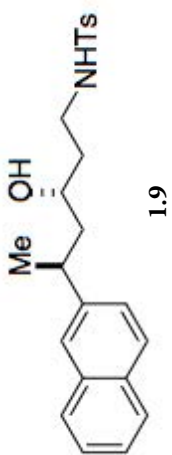
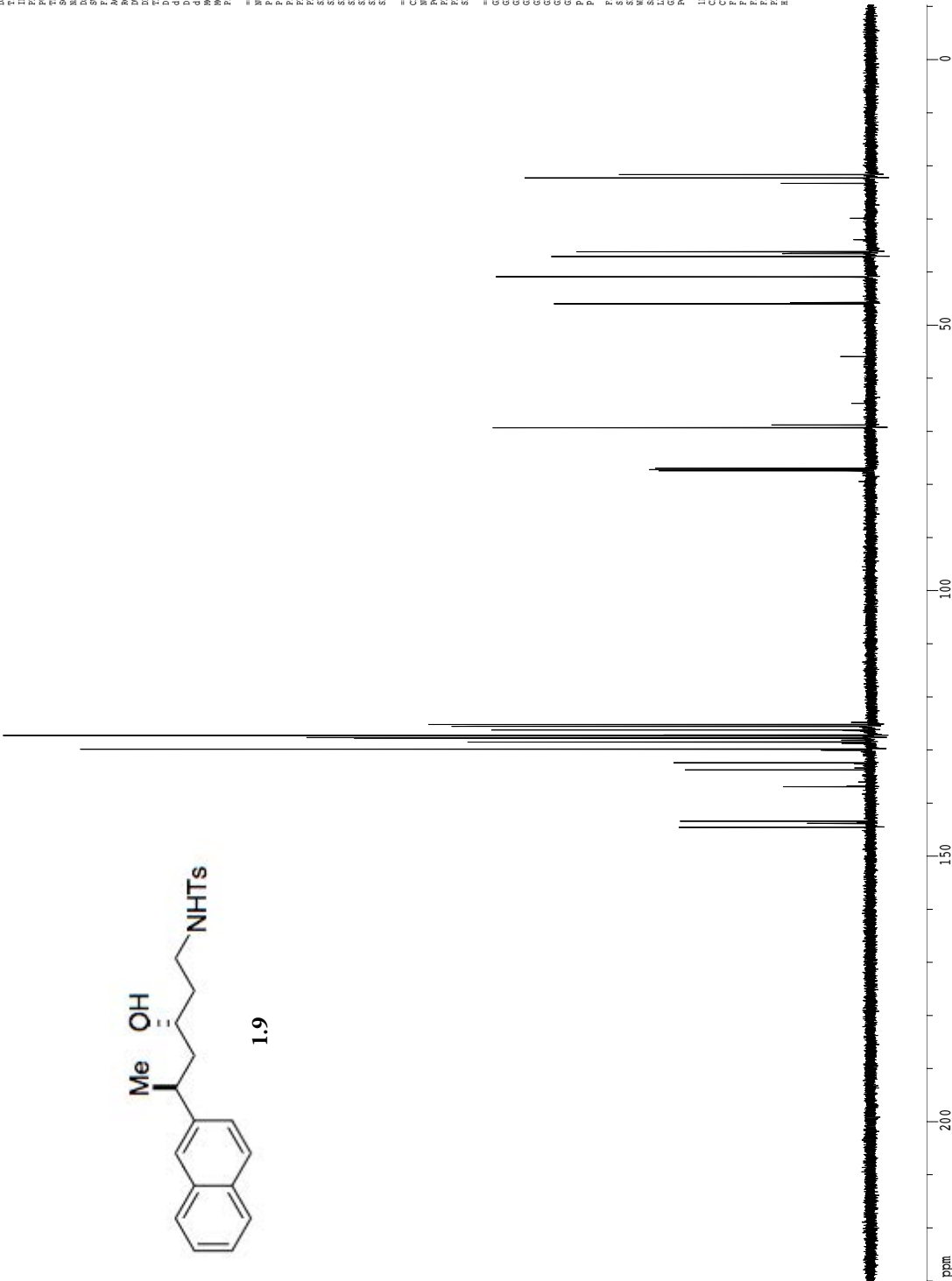
===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 9.75 usec  
 PL1: 1.66 dB  
 SFO1: 500.2235015 MHz

F2 - Processing Parameters  
 SI: 65536  
 SF: 500.2200379 MHz  
 WDM: no  
 LB: 0.00 Hz  
 GB: 0  
 PC: 1.00

LD NMR P1/2 Parameters  
 CY: 22.80 cm  
 CV: 15.00 cm  
 FIP: 9.000 ppm  
 FL: 4301.98 Hz  
 F2: 500.136260 MHz  
 F3: -546.74 Hz  
 PPMCM: 0.44232 ppm/cm  
 HZCM: 221.25967 Hz/cm

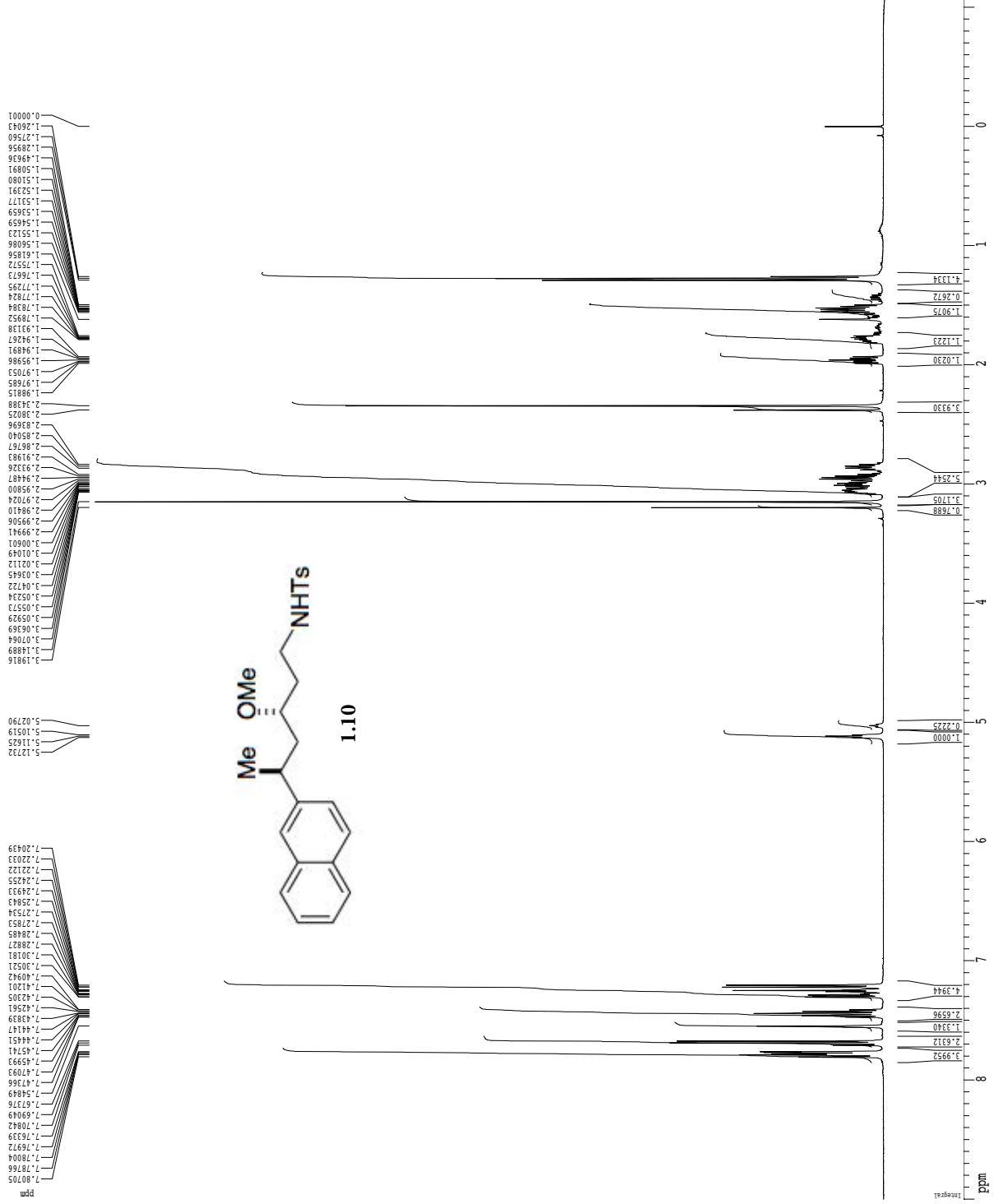
Z-restored spin-echo 13C spectrum with 1H decoupling

21.56  
 21.58  
 22.26  
 23.25  
 24.86  
 25.46  
 36.15  
 36.50  
 37.02  
 37.20  
 40.87  
 45.69  
 45.96  
 55.90  
 68.72  
 69.28  
 76.91  
 77.16  
 77.41  
 124.49  
 124.68  
 124.80  
 124.92  
 125.04  
 125.16  
 125.28  
 125.40  
 125.52  
 125.64  
 125.76  
 125.88  
 126.00  
 126.12  
 126.24  
 126.36  
 126.48  
 126.60  
 126.72  
 126.84  
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 127.08  
 127.20  
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 134.04  
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 134.64  
 134.76  
 134.88  
 135.00  
 135.12



Current Data Parameters  
 USER cahabecb  
 NAME CM-1-239-4111  
 EXPTNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 201202  
 Time 11:41  
 INSTRUM spect  
 PULPROG zgpg30  
 FIDRES 5.000000  
 TD 65536  
 SFO 125.764548 MHz  
 SOLVENT CDCl3  
 NS 96  
 DS 4  
 SWH 3040.4011 Hz  
 F2RES 0.462388 Hz  
 AQ 1.185658 sec  
 RG 4456  
 SFO2 125.764548 MHz  
 DM 16.500 usec  
 DE 8.00 usec  
 DI 0.2500000 usec  
 DC 2.0000000 usec  
 DD 0.000000000 usec  
 DL 0.000000000 usec  
 DT 0.000000000 usec  
 FWHM 0.000000000 usec  
 AVEF 0.000000000 usec  
 ACQF 0.000000000 usec  
 SCALED 1.55 dB  
 KWREK 0.015000000 usec  
 F2 37.70 usec  
 ===== CHANNEL f1 =====  
 NUCL1 13C  
 P1 13.00 usec  
 PL1 0.00 dB  
 PC1 200.00 usec  
 P2 500.00 usec  
 PL2 120.00 dB  
 PC2 120.00 usec  
 P3 125.764548 MHz  
 SFO3 125.764548 MHz  
 GPC1 1.55 dB  
 SFO4 0.00 Hz  
 SFO5 0.00 Hz  
 SFO6 0.00 Hz  
 ===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 SFO7 500.1362600 MHz  
 PCPD2 100.00 usec  
 PL2 1.90 dB  
 PL12 22.00 dB  
 SFO2 500.1223011 MHz  
 ===== GRADIENT CHANNEL =====  
 GRAM1 SINE100  
 SFO1 500.1362600 MHz  
 GPC1 6.00 V  
 GPC2 0.00 V  
 GPC3 0.00 V  
 GPC4 0.00 V  
 GPC5 30.00 V  
 GPC6 50.00 V  
 GPC7 100.00 usec  
 F2 - Processing parameters  
 SI 32768  
 SF 125.764548 MHz  
 WDW no  
 SSB 0  
 GB 0 Hz  
 CB 0 Hz  
 PC 2.00  
 ID\_NMR p1c1 parameters  
 CX 22.88 cm  
 CY 15.95 cm  
 CZ 15.95 cm  
 F1 2000.00 MHz  
 F2 100.00 MHz  
 F3 -10.281 MHz  
 F4 -1292.96 MHz  
 GPRG0 100.00 usec/cm  
 GPRG1 100.00 usec/cm  
 GPRG2 1323.16535 usec/cm

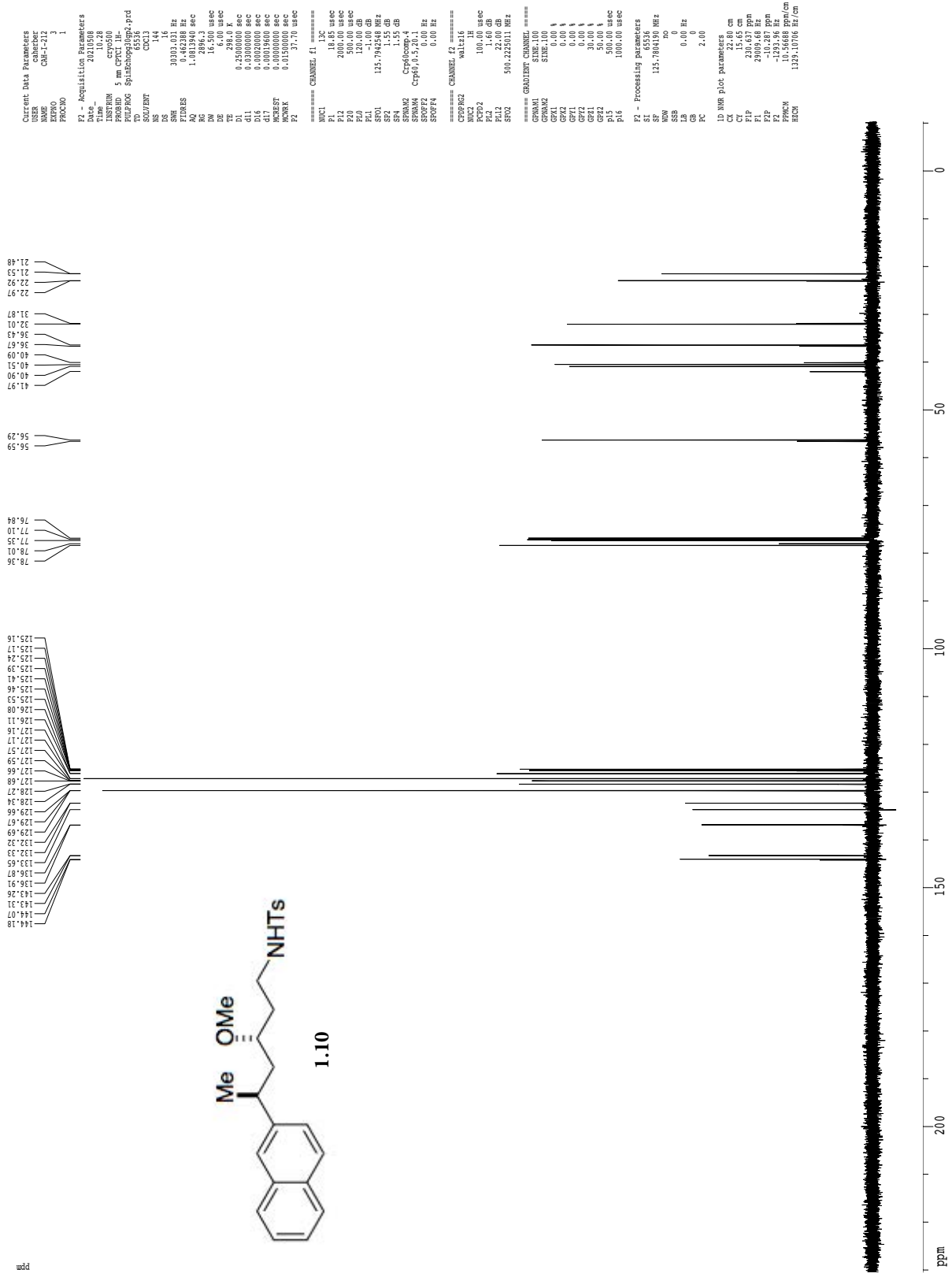
1H spectrum



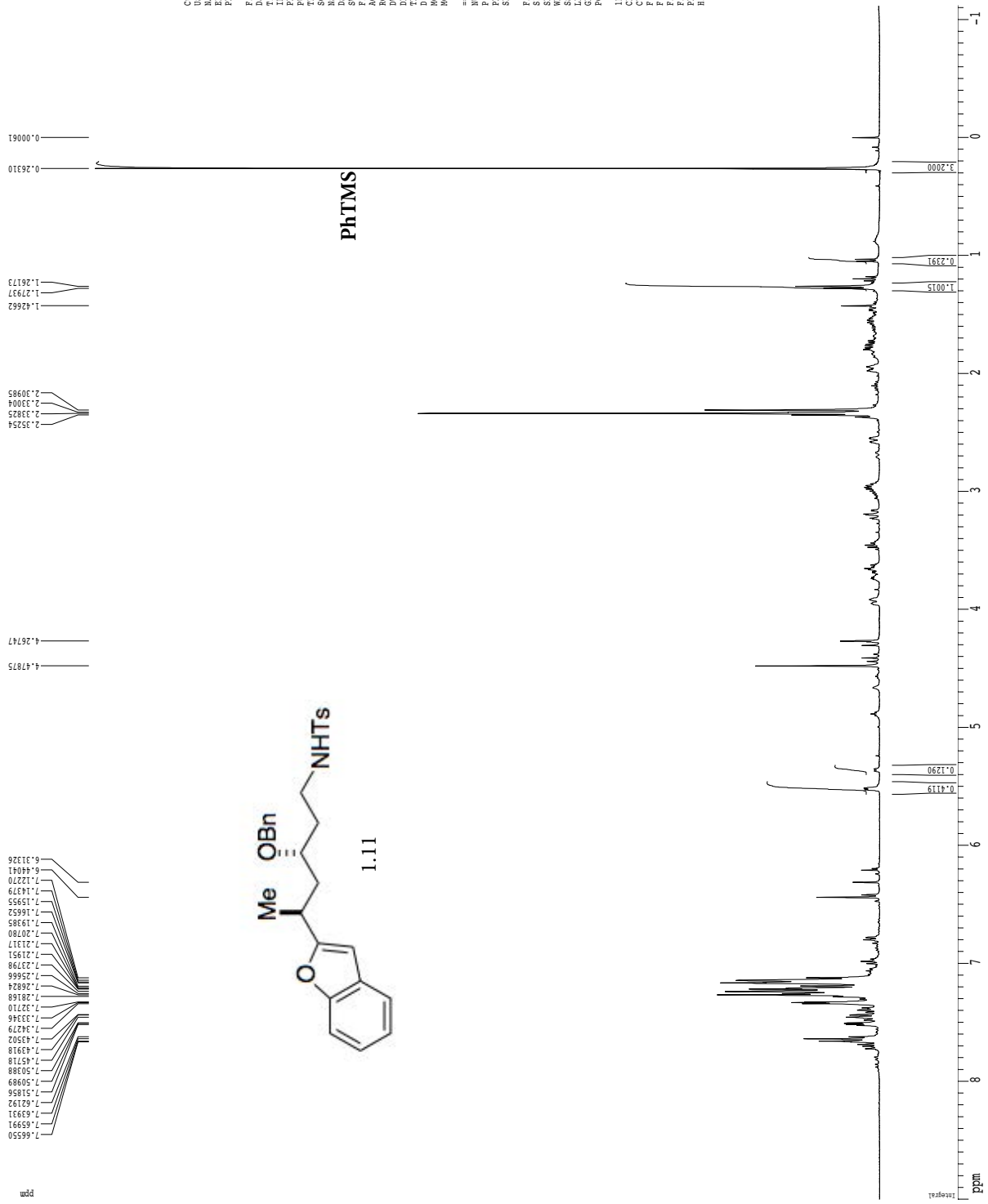
Current Data Parameters  
 USER: chembe  
 NAME: CNF-212  
 EXNO: 2  
 PROCN: 1

F2 - Acquisition Parameters  
 Date\_: 20210508  
 Time: 10.24  
 PROBHD: 5 mm CPY-1H  
 PULPROG: zg30  
 TD: 8178  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 8012.826 Hz  
 FIDRES: 0.098044 Hz  
 AQ: 5.1998774 sec  
 RG: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 1.0000000 sec  
 T2: 0.0000000 sec  
 T3: 0.0000000 sec  
 T4: 0.0000000 sec  
 T5: 0.0000000 sec  
 T6: 0.0000000 sec  
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 T96: 0.0000000 sec  
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 T100: 0.0000000 sec

Z-restored spin-echo 13C spectrum with 1H decoupling

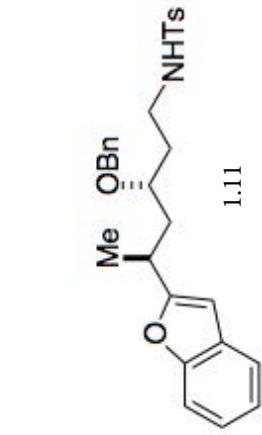
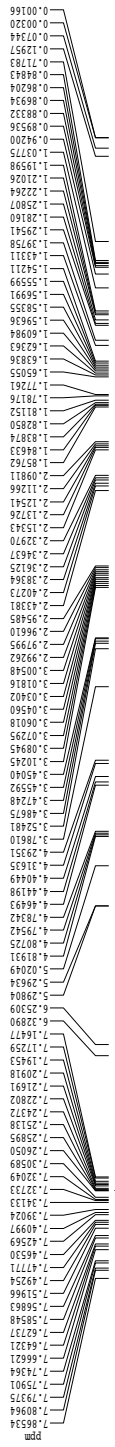


1H spectrum

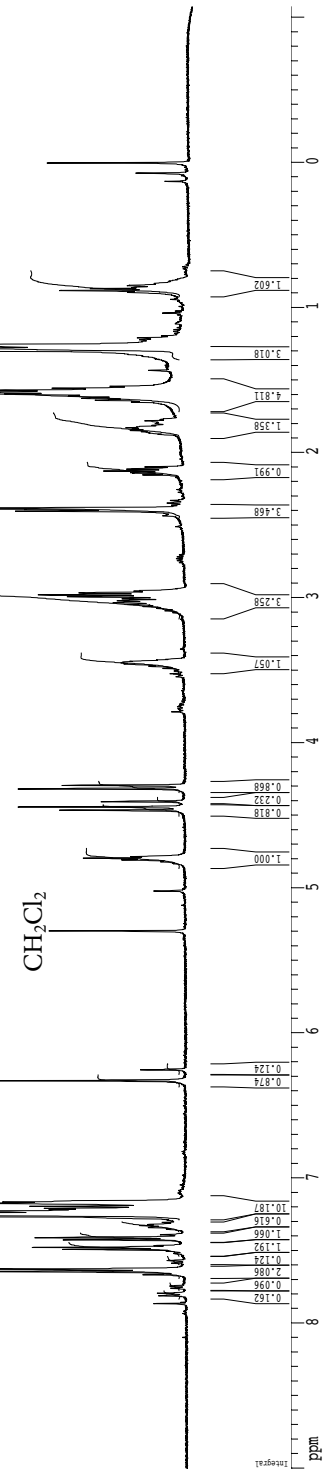


Current Data Parameters  
 USER: chember  
 NAME: CNH-4-233-47c1d  
 EXPNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20210604  
 Time: 15.44  
 PROBNM: 47c1d  
 PULPROG: zgpg30  
 PCPRG2: 5 mm QNP H7 ZG  
 TD: 65536  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 500.136099 MHz  
 SWH: 6410.258 Hz  
 FIDRES: 0.097813 Hz  
 AQ: 5.1118579 sec  
 RG: 327.5  
 DD: 78.000 usec  
 DE: 4.50 usec  
 TE: 298.2 K  
 T1: 0.100000 sec  
 T1RHO: 0.000000 sec  
 T2: 0.000000 sec  
 T2RHO: 0.000000 sec  
 MCRST: 0.000000 sec  
 MCRBK: 0.00500000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 1H  
 P1: 12.00 usec  
 PL1: -1.66 dB  
 SFO1: 400.1326009 MHz  
 F2 - Processing Parameters  
 SI: 65536  
 SF: 400.1300427 MHz  
 WDM: no  
 LB: 0.00 Hz  
 GB: 0  
 PC: 2.00  
 ID: NMR file parameters  
 C2: 22.80 cm  
 C1: 15.00 cm  
 F1P: 9.000 ppm  
 F1: 360.117 Hz  
 F2: -446.91 Hz  
 F2: -446.91 Hz  
 FREQM: 0.44372 ppm/cm  
 HZCM: 177.54126 Hz/cm

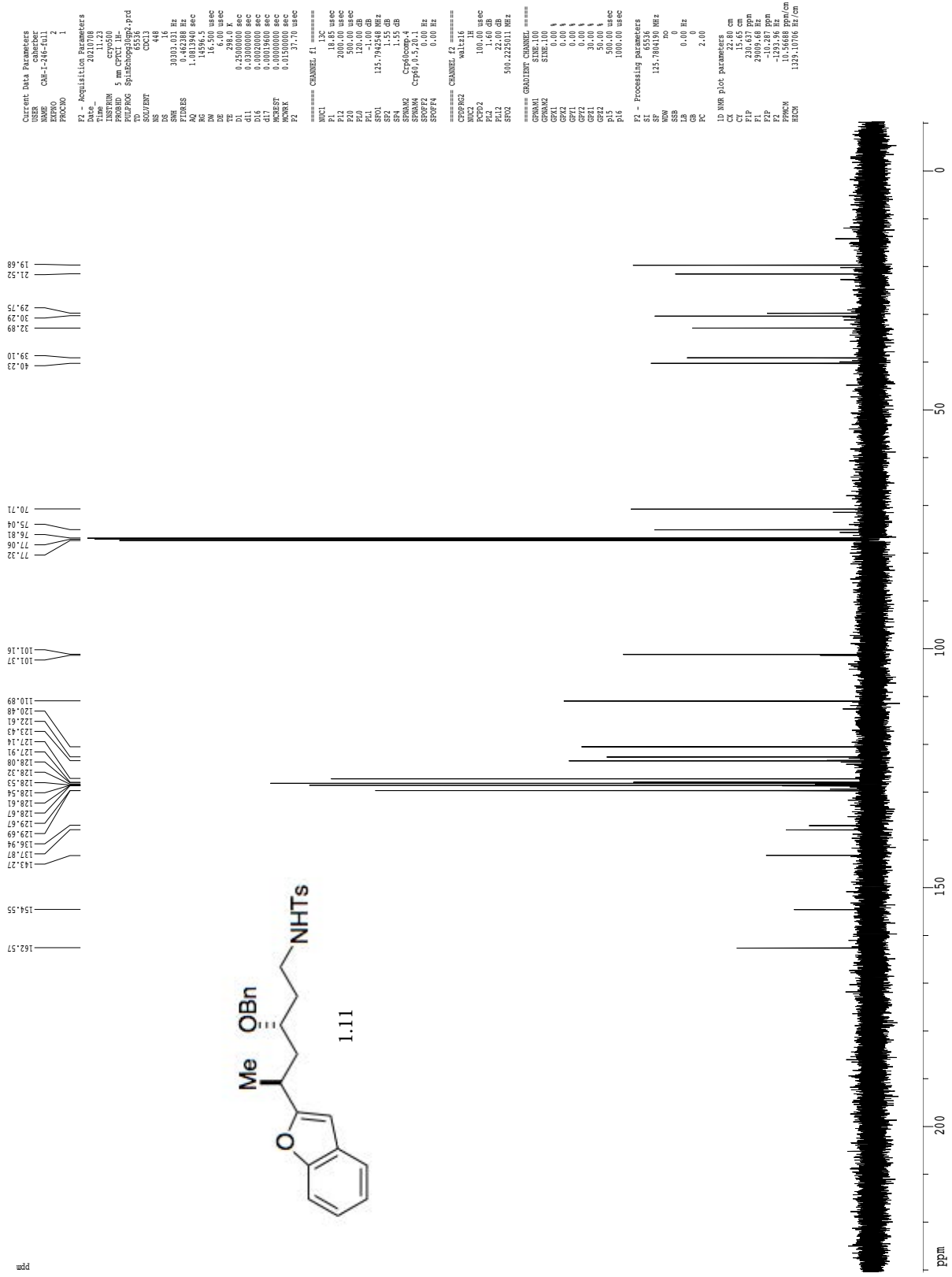
1H spectrum



Current Data Parameters  
 USER: cshen  
 NAME: CSH-4-24-6-4-11  
 EXPNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_ : 2010708  
 Time : 11.14  
 PROBHD: cryo-7  
 PULPROG: zgpg30  
 TD: 8178  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 8012.820 Hz  
 FIDRES: 0.09604 Hz  
 AQ: 5.1998774 sec  
 RG: 655  
 DD: 62.400 usec  
 DE: 6.000 usec  
 TE: 298.2 K  
 T1: 0.10000000 sec  
 T2: 0.00000000 sec  
 T3: 0.00000000 sec  
 MCHRG1: 0.00000000 sec  
 MCHRG2: 0.00000000 sec  
 MCHRG3: 0.00000000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 9.75 usec  
 PL1: 1.66 dB  
 SFO1: 500.2235015 MHz  
 F2 - Processing parameters  
 SI: 65536  
 SF: 500.2200313 MHz  
 WDW: no  
 SSB: 0  
 GB: 0  
 PC: 1.00  
 LD MRB file parameters  
 CY: 22.86 cm  
 CV: 15.06 cm  
 FIP: 9.000 ppm  
 FL: 4501.98 Hz  
 F2: 500.2200313 MHz  
 F3: -536.14 Hz  
 PPMCH: 0.44175 ppm/cm  
 HZCH: 220.97008 Hz/cm

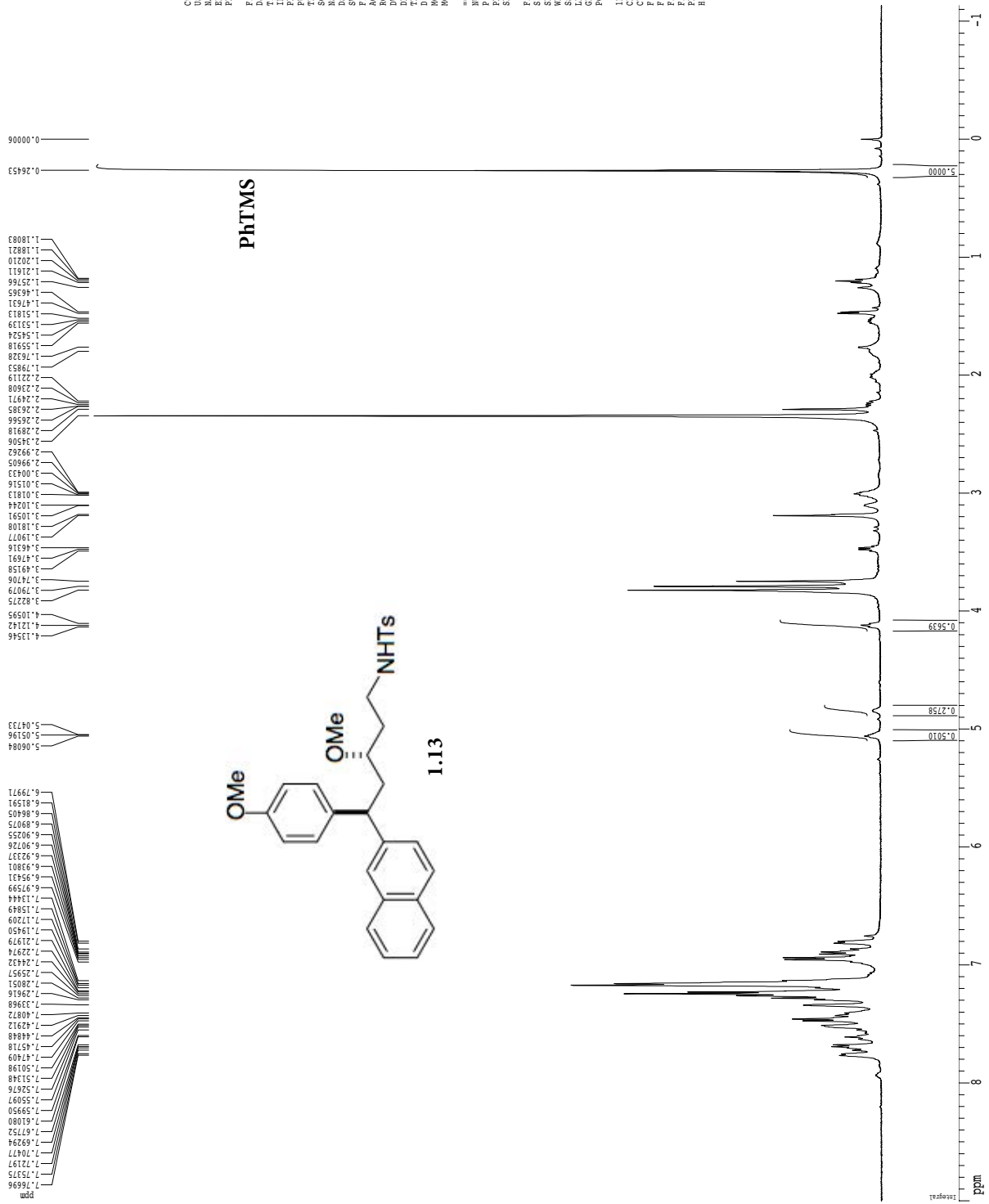


Z-restored spin-echo 13C spectrum with 1H decoupling





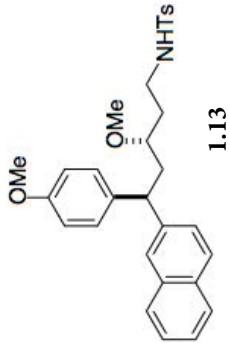
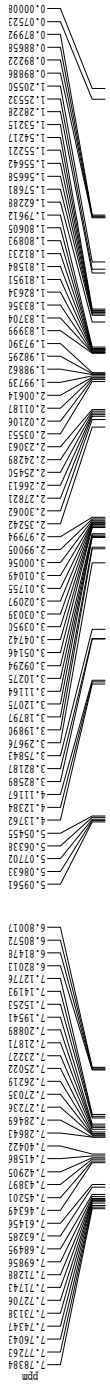
1H spectrum



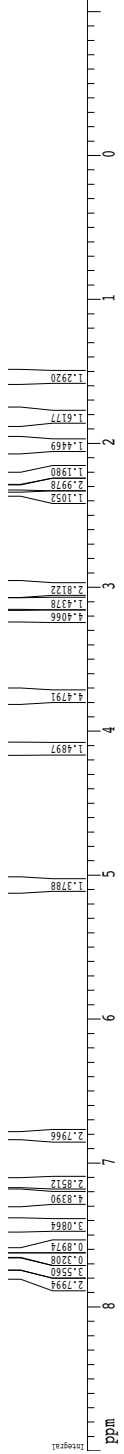
Current Data Parameters  
 USER: chember  
 NAME: C01-4-284-Grude  
 EXPO: 1  
 PROCNO: 1

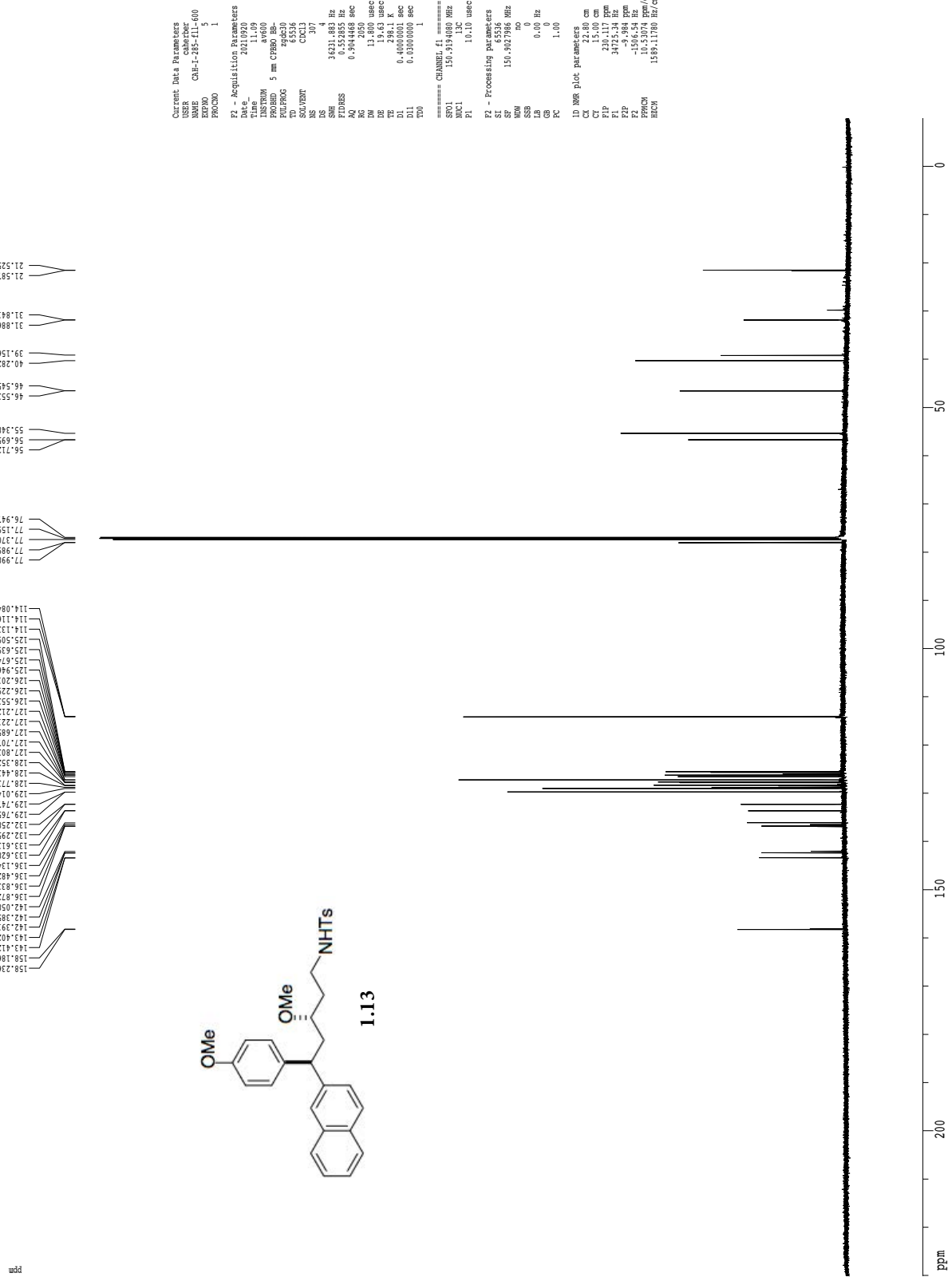
F2 - Acquisition Parameters  
 Date\_: 20210917  
 Time: 10.29  
 INSTR: spect  
 PROCNO: 5  
 PULPROG: zg30  
 TD: 8178  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 5  
 SHF: 8012.820 Hz  
 FIDRES: 0.09804 Hz  
 AQ: 5.1998774 sec  
 RG: 327.5  
 DD: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 1.000000 sec  
 T2: 0.000000 sec  
 T3: 0.000000 sec  
 T4: 0.000000 sec  
 T5: 0.000000 sec  
 T6: 0.000000 sec  
 T7: 0.000000 sec  
 T8: 0.000000 sec  
 T9: 0.000000 sec  
 T10: 0.000000 sec  
 T11: 0.000000 sec  
 T12: 0.000000 sec  
 T13: 0.000000 sec  
 T14: 0.000000 sec  
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 T88: 0.000000 sec  
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 T93: 0.000000 sec  
 T94: 0.000000 sec  
 T95: 0.000000 sec  
 T96: 0.000000 sec  
 T97: 0.000000 sec  
 T98: 0.000000 sec  
 T99: 0.000000 sec  
 T100: 0.000000 sec

1H

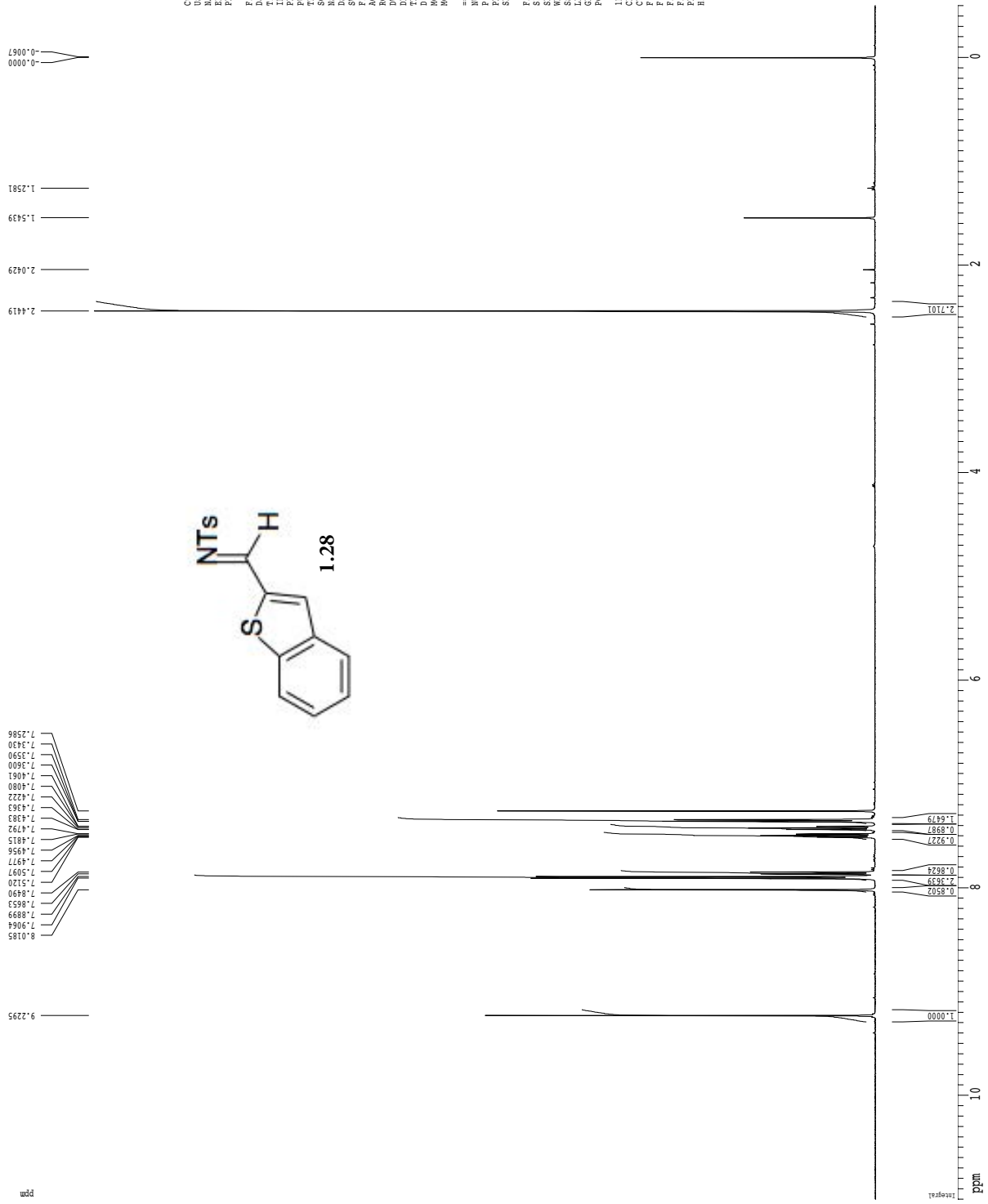


Current Data Parameters  
USER: chemist  
NAME: CN1-1.495-11.400  
EXPNO: 4  
PROCNO: 1  
F2 - Acquisition Parameters  
Date\_: 20110920  
Time: 10.59  
INSTRUM: av600  
PROBHD: 5 mm CPBBO  
PULPROG: zgpg30  
TD: 65536  
SOLVENT: CDCl3  
NS: 9  
DS: 9  
SWH: 9615.385 Hz  
FIDRES: 0.498842 Hz  
AQ: 5.0989979 sec  
RG: 327.500  
DM: 52.400 uSsec  
DE: 1.423 uSsec  
TE: 298.0 K  
DQ: 0.10000001 sec  
TD0: 1  
===== CHANNEL f1 =====  
NUC1: 13C  
P1: 9.50 uSsec  
F2 - Processing parameters  
SI: 32768  
SF: 600.1300407 MHz  
WDW: no  
SSB: 0  
GB: 0  
PC: 1.00  
LD: NMR plot parameters  
CX: 22.80 cm  
CY: 15.00 cm  
FLP: 9.000 ppm  
FZ: 541.133 Hz  
PZ: 1.000 cm  
F2: -617.47 Hz  
PRNCH: 0.44206 ppm/cm  
RECN: 245.23141 Hz/cm





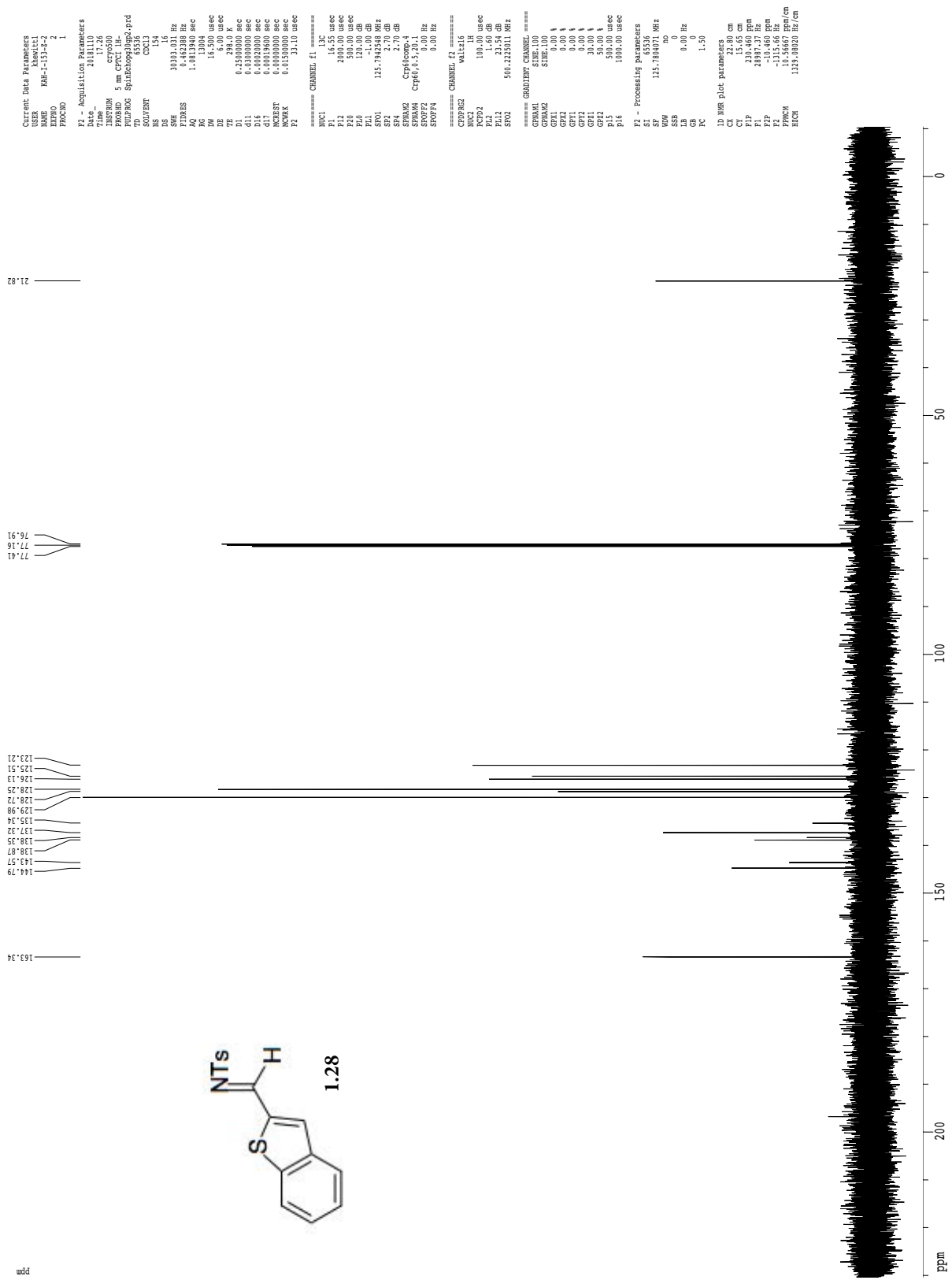
1H spectrum



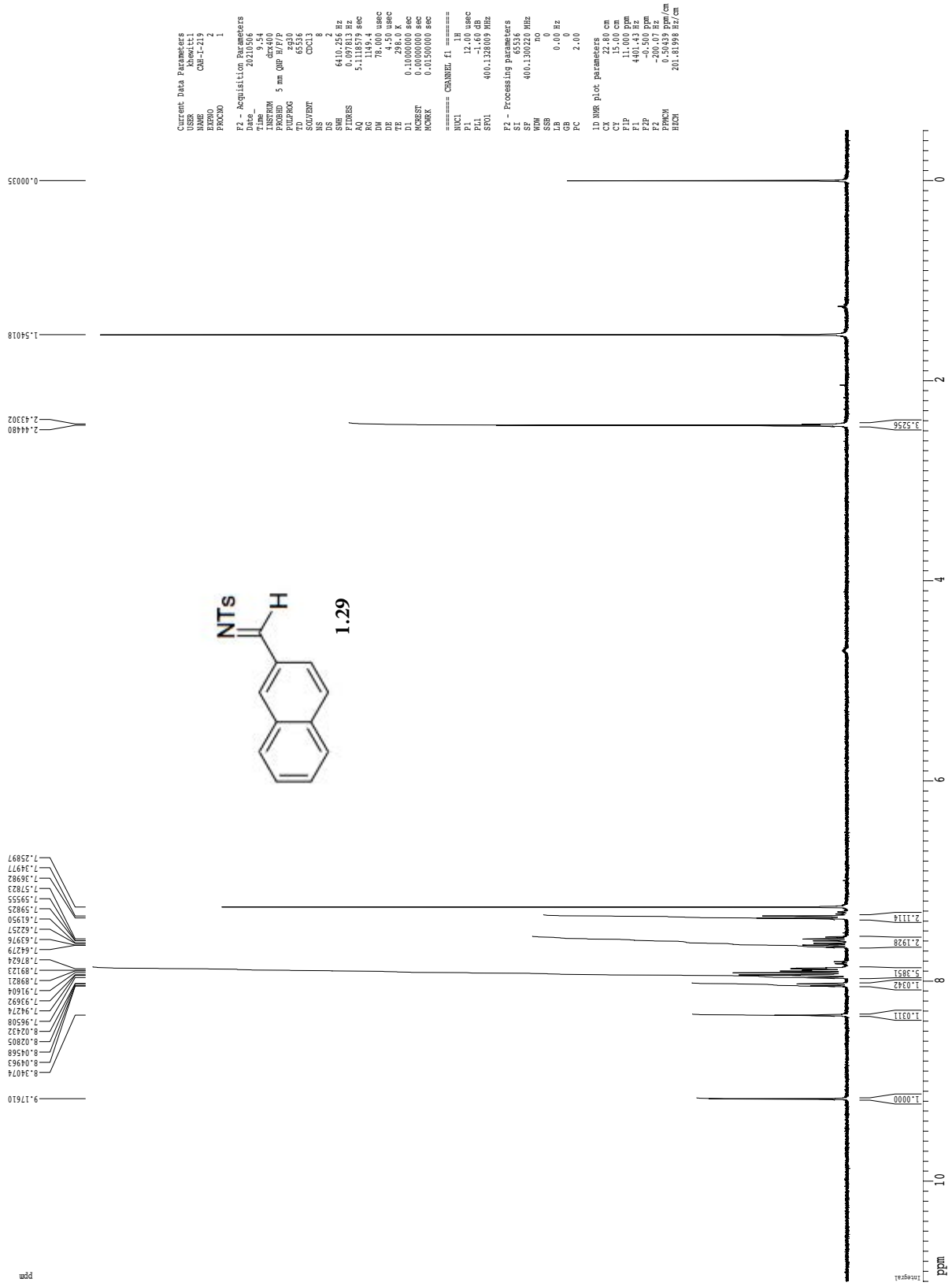
Current Data Parameters  
 USER: Komettt  
 NAME: R0H-152-R-2  
 EXNO: 1  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20181110  
 Time: 17.24  
 INSTR: spect  
 PROBHD: 5 mm CPY-1H  
 PULPROG: zg30  
 TD: 48074  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 8012.820 Hz  
 FIDRES: 0.166677 Hz  
 AQ: 2.3998677 sec  
 RG: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 0.10000000 sec  
 T2: 0.00000000 sec  
 T3: 0.00000000 sec  
 T4: 0.00000000 sec  
 T5: 0.00000000 sec  
 T6: 0.00000000 sec  
 T7: 0.00000000 sec  
 T8: 0.00000000 sec  
 T9: 0.00000000 sec  
 T10: 0.00000000 sec  
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 T94: 0.00000000 sec  
 T95: 0.00000000 sec  
 T96: 0.00000000 sec  
 T97: 0.00000000 sec  
 T98: 0.00000000 sec  
 T99: 0.00000000 sec  
 T100: 0.00000000 sec

Z-restored spin-echo 13C spectrum with 1H decoupling



1H spectrum



Current Data Parameters  
USER: Komettt  
NAME: CNR-F-219  
EXNO: 2  
PROCNO: 1

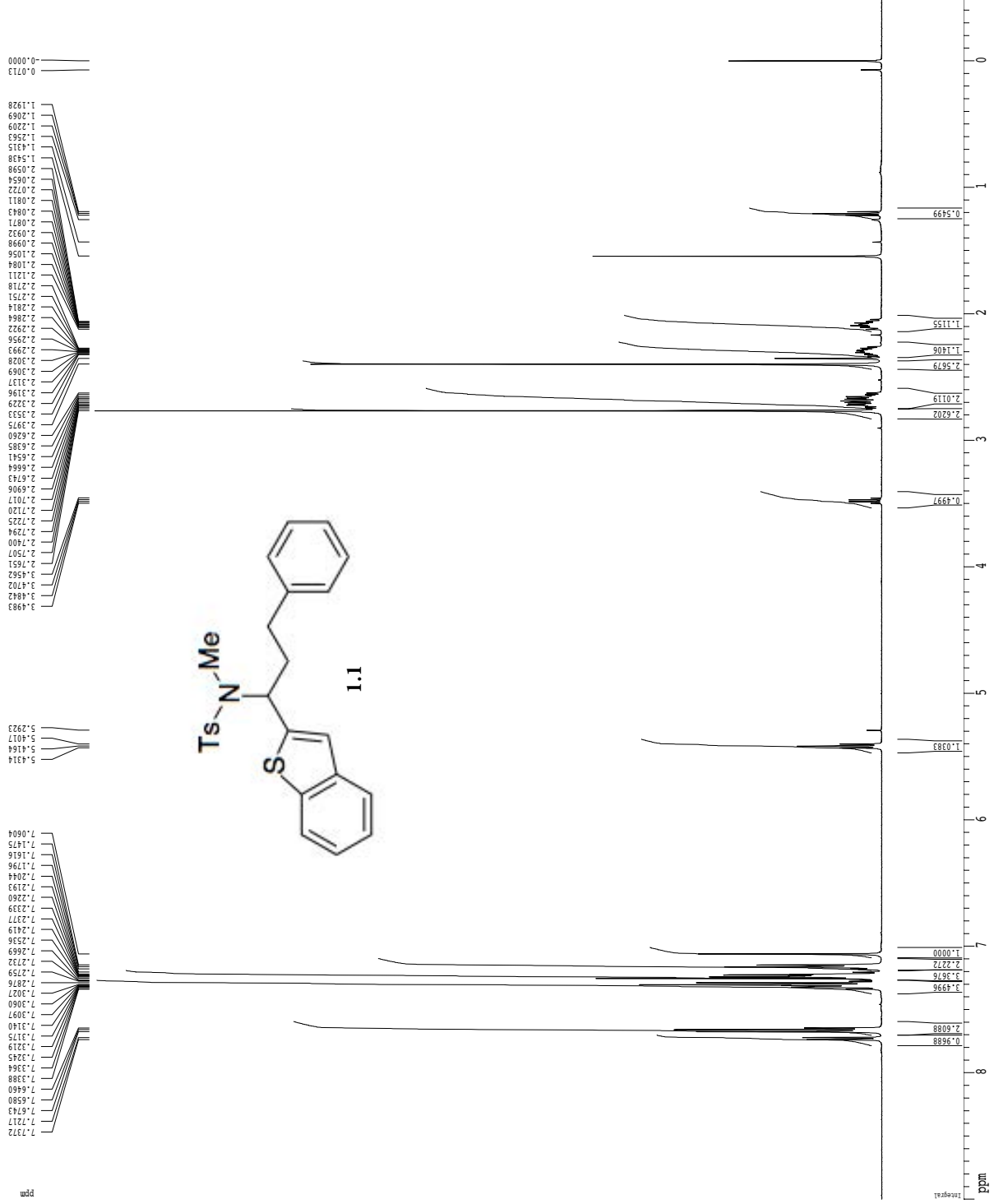
F2 - Acquisition Parameters  
Date\_: 20210506  
Time: 9.54  
PROBHD: 5 mm QNP 1H/1  
PULPROG: zg30  
TD: 65536  
SOLVENT: CDCl3  
DS: 9  
SHE: 6410.256 Hz  
FIDRES: 0.097813 Hz  
AQ: 5.1118579 sec  
RG: 384  
DQ: 78.000 usec  
DE: 4.50 usec  
TE: 298.2 K  
0.1000000 sec  
MORST: 0.0000000 sec  
MORSE: 0.0000000 sec  
MORSEK: 0.0050000 sec

===== CHANNEL f1 =====  
NUC1: 1H  
P1: 12.00 usec  
PL1: -1.66 dB  
SFO1: 400.1328009 MHz

F2 - Processing Parameters  
SI: 65536  
SF: 400.1300220 MHz  
WDW: no  
SSB: 0  
GB: 0.00 Hz  
PC: 2.00

ID: NMR PLOT Parameters  
CY: 22.80 cm  
CY: 15.00 cm  
FIP: 11.000 ppm  
FL: 4400.48 Hz  
F2: 200.00 MHz  
F2: -200.00 Hz  
PRGCM: 0.50439 ppm/cm  
HZCM: 201.81998 Hz/cm

1H spectrum



Current Data Parameters  
 USER: KAWA  
 NAME: KAW-1172-2  
 EKWO: 1  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20181107  
 Time: 21.08  
 SYSTEM: cryo-10  
 PROBHD: 5 mm CRYO-1H  
 PULPROG: zg30  
 TD: 48074  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 500.136260 MHz  
 SFE: 8012.820 Hz  
 FIDRES: 0.166677 Hz  
 AQ: 2.3998677 sec  
 RG: 327.500  
 DD: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 1.000000 sec  
 T2: 0.000000 sec  
 T3: 0.000000 sec  
 MCHRES: 0.30150000 sec  
 MCORE: 0.01500000 sec

===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 1.50 usec  
 PL1: 1.66 dB  
 SFO1: 500.2235015 MHz

F2 - Processing Parameters  
 SI: 65536  
 SF: 500.2200355 MHz  
 WDW: no  
 SSB: 0  
 GB: 0  
 PC: 1.00

ID: NMR file parameters  
 CY: 22.86 cm  
 CV: 15.06 cm  
 FIP: 9.000 ppm  
 FL: 4501.98 Hz  
 F2: 500.136260 MHz  
 F3: -250.11 Hz  
 PPMCH: 0.41667 ppm/cm  
 HZCH: 206.44502 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling

Current Data Parameters  
 USER Rhoett11  
 NMW 131-172-2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 21.12  
 Time 21:12  
 INSTRUM spect  
 P1 0.05000000 sec  
 FREQ 125.7603889 MHz  
 TO F2PROC 0.00000000 sec  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 4  
 SWH 30033.031 Hz  
 FIDRES 0.4623888 Hz  
 RG 1.146823 sec  
 GC 1.00000000 sec  
 DM 16.500 usec  
 DE 9.00 usec  
 DI 0.25000000 sec  
 D1 0.03000000 sec  
 D11 0.03000000 sec  
 D12 0.03000000 sec  
 D13 0.03000000 sec  
 D14 0.03000000 sec  
 D15 0.03000000 sec  
 D16 0.03000000 sec  
 D17 0.03000000 sec  
 D18 0.03000000 sec  
 D19 0.03000000 sec  
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 D95 0.03000000 sec  
 D96 0.03000000 sec  
 D97 0.03000000 sec  
 D98 0.03000000 sec  
 D99 0.03000000 sec  
 D100 0.03000000 sec

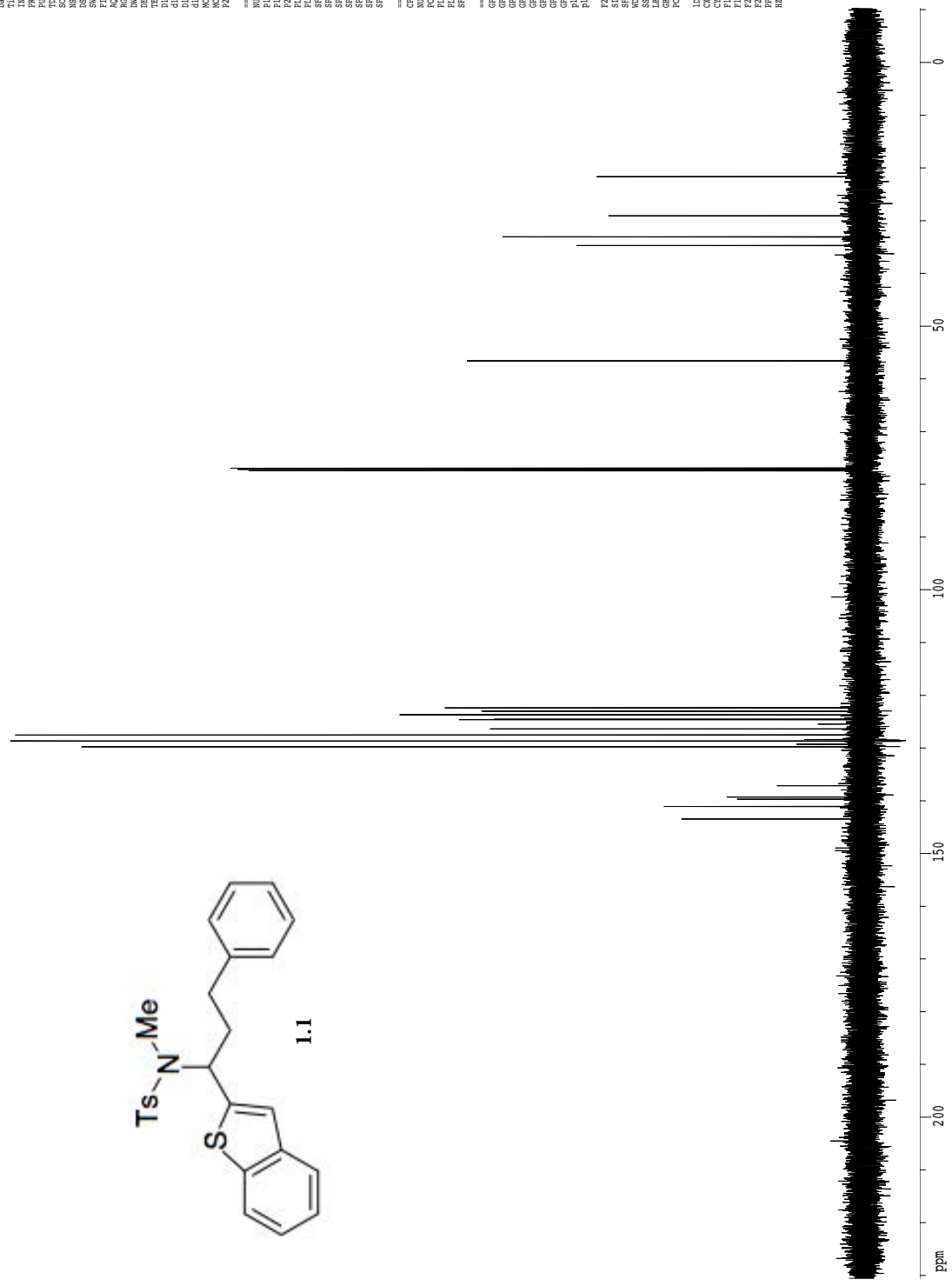
\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUCL1 13C  
 P1 1.00000000 usec  
 F12 2000.00000000 MHz  
 F2 2000.00000000 MHz  
 F20 500.00000000 MHz  
 F21 120.00000000 dB  
 SFO1 125.7603889 MHz  
 SFO2 2.70000000 MHz  
 SFO3 Cmpfoco  
 SFO4 Cmpfof2  
 SFO5 Cmpfof1  
 SFO6 Cmpfof3  
 SFO7 Cmpfof4  
 SFO8 Cmpfof5  
 SFO9 Cmpfof6  
 SFO10 Cmpfof7  
 SFO11 Cmpfof8  
 SFO12 Cmpfof9  
 SFO13 Cmpfof10  
 SFO14 Cmpfof11  
 SFO15 Cmpfof12  
 SFO16 Cmpfof13  
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 SFO95 Cmpfof92  
 SFO96 Cmpfof93  
 SFO97 Cmpfof94  
 SFO98 Cmpfof95  
 SFO99 Cmpfof96  
 SFO100 Cmpfof97

\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
 CPDPRG2 waltz16  
 P1 1.00000000 usec  
 F12 2000.00000000 MHz  
 F2 2000.00000000 MHz  
 F20 500.00000000 MHz  
 F21 120.00000000 dB  
 SFO1 125.7603889 MHz  
 SFO2 2.70000000 MHz  
 SFO3 Cmpfoco  
 SFO4 Cmpfof2  
 SFO5 Cmpfof1  
 SFO6 Cmpfof3  
 SFO7 Cmpfof4  
 SFO8 Cmpfof5  
 SFO9 Cmpfof6  
 SFO10 Cmpfof7  
 SFO11 Cmpfof8  
 SFO12 Cmpfof9  
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 SFO88 Cmpfof85  
 SFO89 Cmpfof86  
 SFO90 Cmpfof87  
 SFO91 Cmpfof88  
 SFO92 Cmpfof89  
 SFO93 Cmpfof90  
 SFO94 Cmpfof91  
 SFO95 Cmpfof92  
 SFO96 Cmpfof93  
 SFO97 Cmpfof94  
 SFO98 Cmpfof95  
 SFO99 Cmpfof96  
 SFO100 Cmpfof97

\*\*\*\*\* GRADIENT CHANNEL \*\*\*\*\*  
 GRAMA SINE-100  
 GRAB1 0.00000000 sec  
 GRAB2 0.00000000 sec  
 GRAB3 0.00000000 sec  
 GRAB4 0.00000000 sec  
 GRAB5 0.00000000 sec  
 GRAB6 0.00000000 sec  
 GRAB7 0.00000000 sec  
 GRAB8 0.00000000 sec  
 GRAB9 0.00000000 sec  
 GRAB10 0.00000000 sec  
 GRAB11 0.00000000 sec  
 GRAB12 0.00000000 sec  
 GRAB13 0.00000000 sec  
 GRAB14 0.00000000 sec  
 GRAB15 0.00000000 sec  
 GRAB16 0.00000000 sec  
 GRAB17 0.00000000 sec  
 GRAB18 0.00000000 sec  
 GRAB19 0.00000000 sec  
 GRAB20 0.00000000 sec  
 GRAB21 0.00000000 sec  
 GRAB22 0.00000000 sec  
 GRAB23 0.00000000 sec  
 GRAB24 0.00000000 sec  
 GRAB25 0.00000000 sec  
 GRAB26 0.00000000 sec  
 GRAB27 0.00000000 sec  
 GRAB28 0.00000000 sec  
 GRAB29 0.00000000 sec  
 GRAB30 0.00000000 sec  
 GRAB31 0.00000000 sec  
 GRAB32 0.00000000 sec  
 GRAB33 0.00000000 sec  
 GRAB34 0.00000000 sec  
 GRAB35 0.00000000 sec  
 GRAB36 0.00000000 sec  
 GRAB37 0.00000000 sec  
 GRAB38 0.00000000 sec  
 GRAB39 0.00000000 sec  
 GRAB40 0.00000000 sec  
 GRAB41 0.00000000 sec  
 GRAB42 0.00000000 sec  
 GRAB43 0.00000000 sec  
 GRAB44 0.00000000 sec  
 GRAB45 0.00000000 sec  
 GRAB46 0.00000000 sec  
 GRAB47 0.00000000 sec  
 GRAB48 0.00000000 sec  
 GRAB49 0.00000000 sec  
 GRAB50 0.00000000 sec  
 GRAB51 0.00000000 sec  
 GRAB52 0.00000000 sec  
 GRAB53 0.00000000 sec  
 GRAB54 0.00000000 sec  
 GRAB55 0.00000000 sec  
 GRAB56 0.00000000 sec  
 GRAB57 0.00000000 sec  
 GRAB58 0.00000000 sec  
 GRAB59 0.00000000 sec  
 GRAB60 0.00000000 sec  
 GRAB61 0.00000000 sec  
 GRAB62 0.00000000 sec  
 GRAB63 0.00000000 sec  
 GRAB64 0.00000000 sec  
 GRAB65 0.00000000 sec  
 GRAB66 0.00000000 sec  
 GRAB67 0.00000000 sec  
 GRAB68 0.00000000 sec  
 GRAB69 0.00000000 sec  
 GRAB70 0.00000000 sec  
 GRAB71 0.00000000 sec  
 GRAB72 0.00000000 sec  
 GRAB73 0.00000000 sec  
 GRAB74 0.00000000 sec  
 GRAB75 0.00000000 sec  
 GRAB76 0.00000000 sec  
 GRAB77 0.00000000 sec  
 GRAB78 0.00000000 sec  
 GRAB79 0.00000000 sec  
 GRAB80 0.00000000 sec  
 GRAB81 0.00000000 sec  
 GRAB82 0.00000000 sec  
 GRAB83 0.00000000 sec  
 GRAB84 0.00000000 sec  
 GRAB85 0.00000000 sec  
 GRAB86 0.00000000 sec  
 GRAB87 0.00000000 sec  
 GRAB88 0.00000000 sec  
 GRAB89 0.00000000 sec  
 GRAB90 0.00000000 sec  
 GRAB91 0.00000000 sec  
 GRAB92 0.00000000 sec  
 GRAB93 0.00000000 sec  
 GRAB94 0.00000000 sec  
 GRAB95 0.00000000 sec  
 GRAB96 0.00000000 sec  
 GRAB97 0.00000000 sec  
 GRAB98 0.00000000 sec  
 GRAB99 0.00000000 sec  
 GRAB100 0.00000000 sec

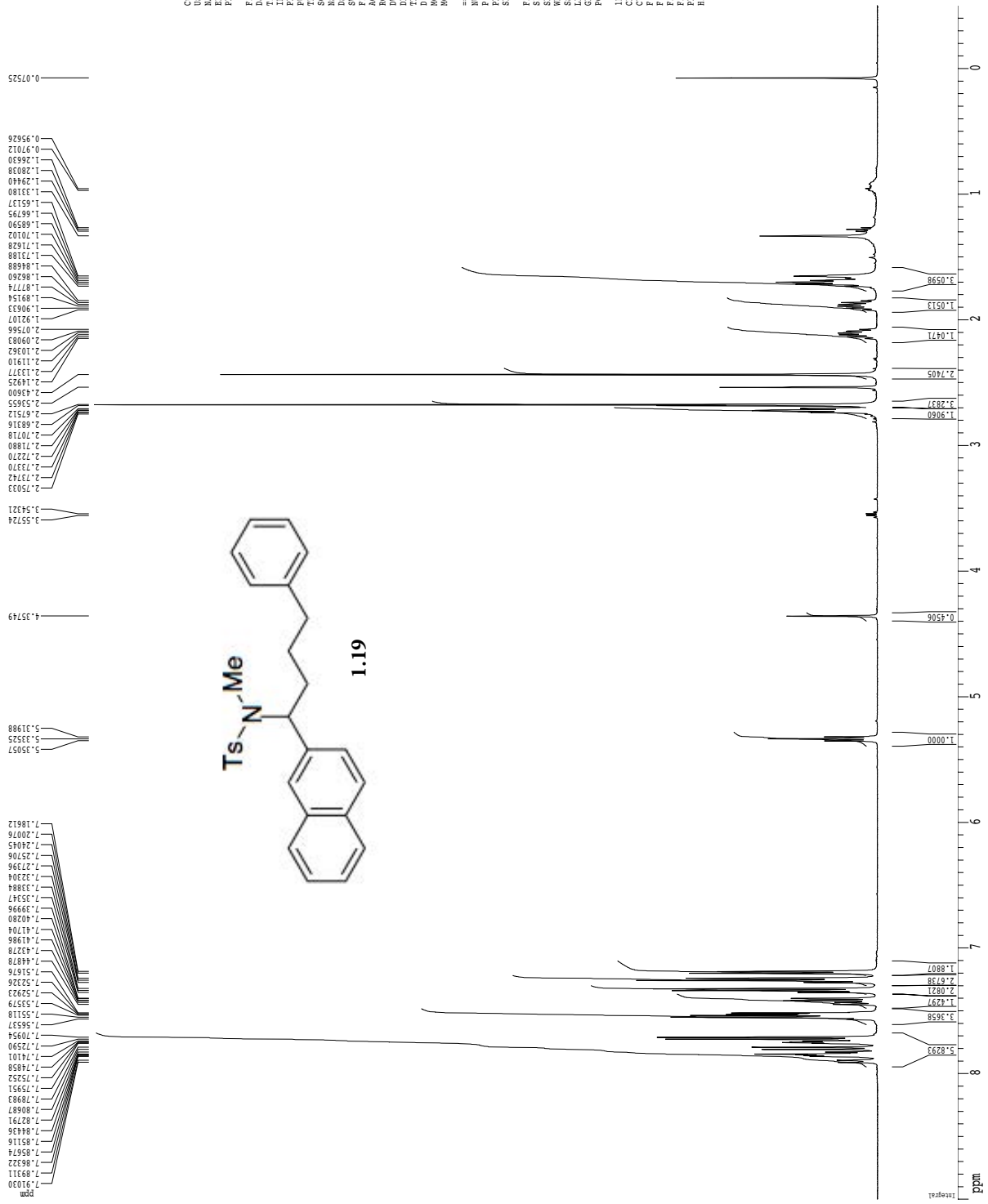
F2 - Processing parameters  
 SI 32768  
 SF 125.7603889 MHz  
 WDW no  
 GB 0  
 SB 0  
 SC 0  
 RC 2.50

1D NMR plot parameters  
 CX 22.80 cm  
 CY 15.60 cm  
 CZ 15.60 cm  
 F1 2000.00000000 MHz  
 F2 125.7603889 MHz  
 F3 -10.287 ppm  
 F4 -129.288 Hz  
 F5 1.10000000 cm  
 F6 1.10000000 cm  
 F7 1325.1652 Hz/cm  
 F8





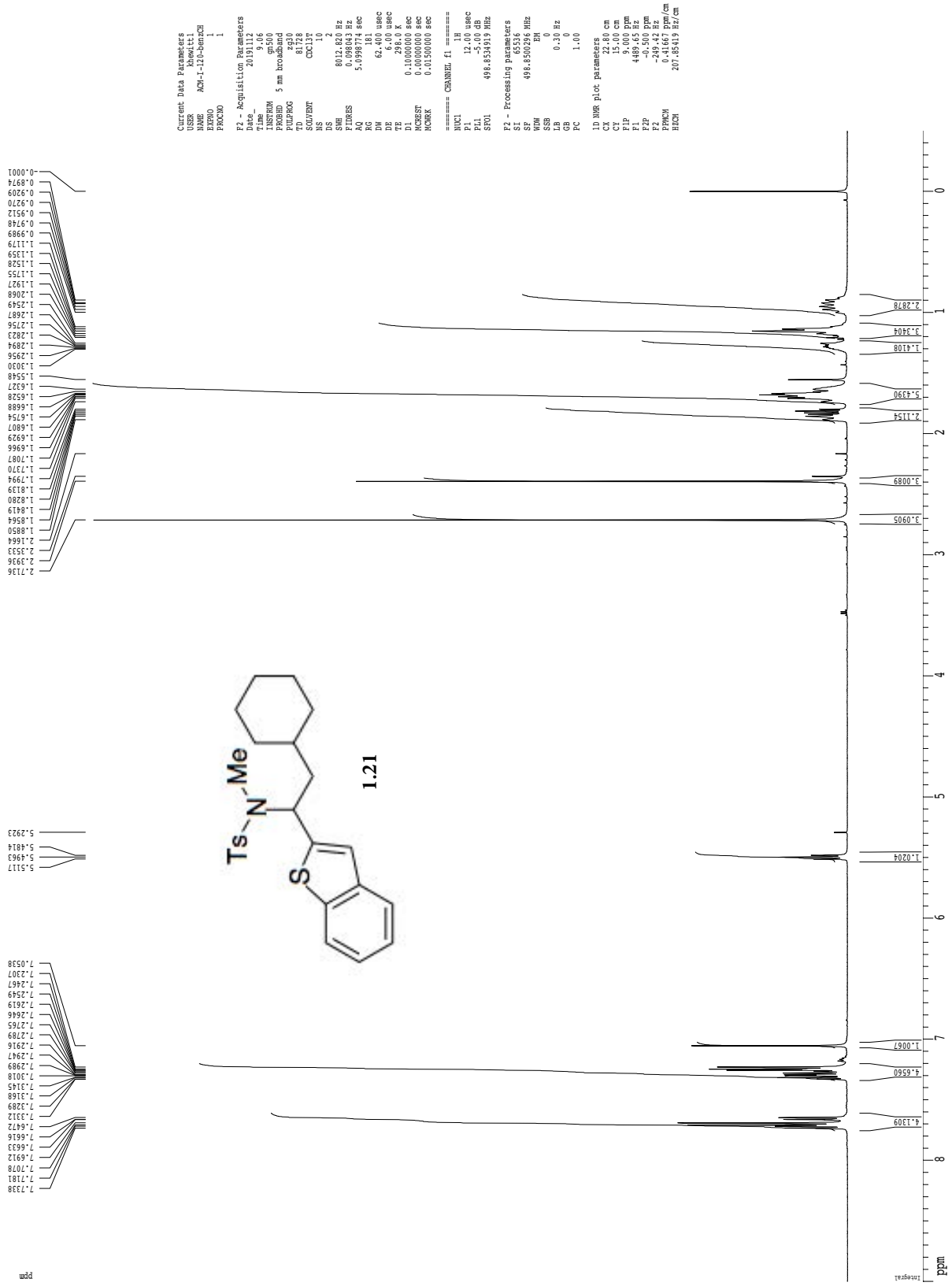
1H spectrum



Current Data Parameters  
USER: Komett  
NAME: AM-1-04-2  
EXPNO: 1  
PROCNO: 1  
P2 - Acquisition Parameters  
Date\_: 20210809  
Time: 18.13  
INSTRUM: spect  
PROBHD: 5 mm CPY-1H  
PULPROG: zg30  
TD: 48074  
SOLVENT: CDCl3  
DS: 9  
SFO: 500.136260 MHz  
SHE: 8012.820 Hz  
FIDRES: 0.166677 Hz  
AQ: 2.3398677 sec  
RG: 62.400 usec  
DE: 6.00 usec  
TE: 298.2 K  
NUC1: 1H  
MAGNET: 0.1600000 sec  
MCORE: 0.0150000 sec  
===== CHANNEL f1 =====  
NUC1: 1H  
P1: 9.75 usec  
PL1: 1.66 dB  
SFO1: 500.2235015 MHz  
P2 - Processing parameters  
SI: 65536  
SF: 500.2200000 MHz  
WDW: no  
SSB: 0  
GB: 0.00 Hz  
PC: 1.00  
ID: NMR file parameters  
CY: 22.80 cm  
C1: 15.00 cm  
F1P: 9.000 ppm  
F1: 43001.98 Hz  
F2: 250.13 Hz  
F2: -250.13 Hz  
PRGCM: 0.41667 ppm/cm  
HZCM: 206.44500 Hz/cm



1H spectrum



Current Data Parameters  
 USER Kowalewski  
 NAME ACH-1-120-H0402CH  
 EXNO 1  
 PROCNO 1

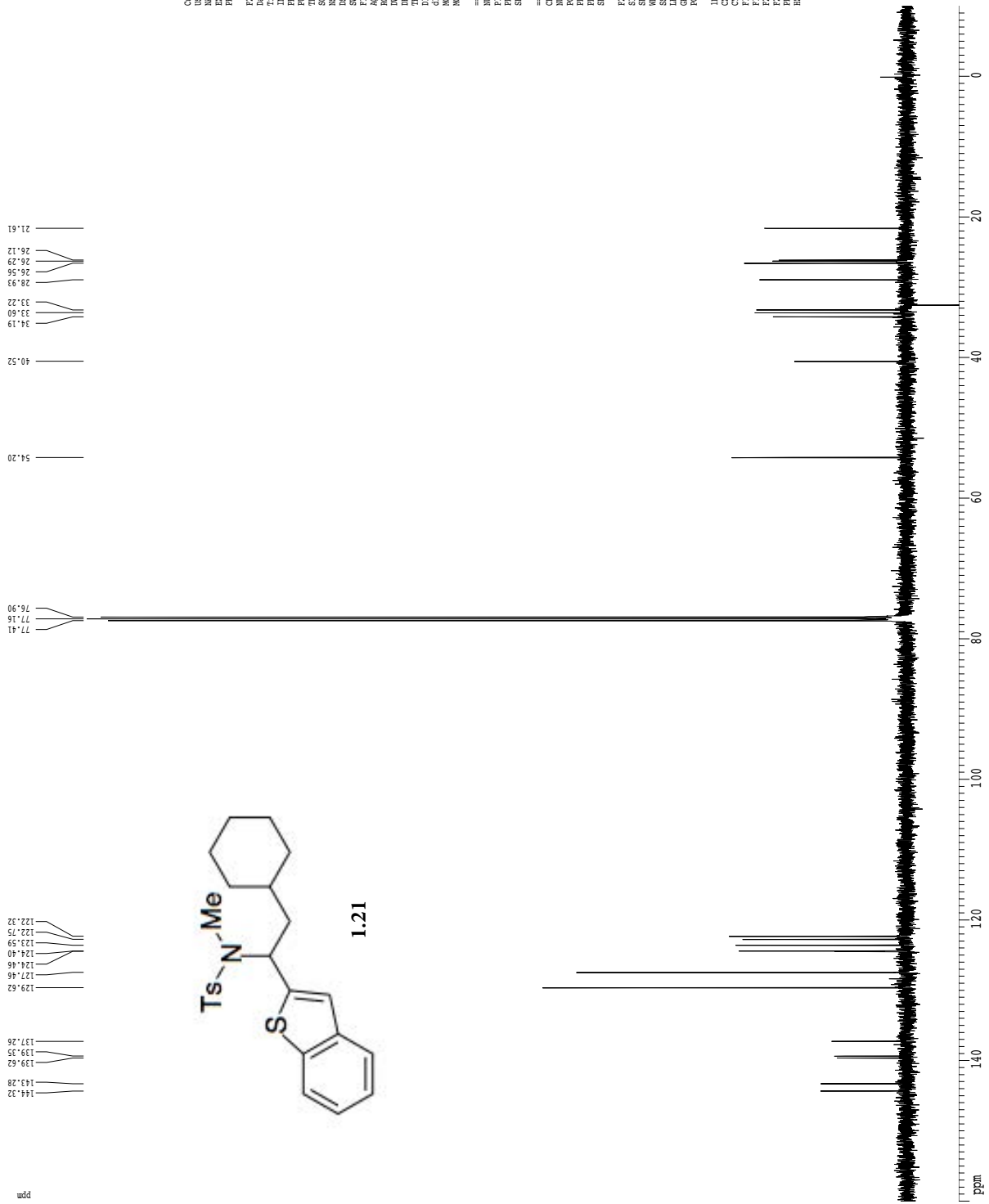
F2 - Acquisition Parameters  
 Date\_ 20191112  
 Time 9:06  
 INSTR spect  
 PROBHD 5 mm bbo-5130  
 PULPROG zg30  
 TD 8178  
 SOLVENT CDCl3  
 DS 0  
 SFO 8012.820 Hz  
 FIDRES 0.098044 Hz  
 AQ 5.1998774 sec  
 RG 62.400 usec  
 DE 6.00 usec  
 TE 298.2 K  
 T1 1.0000000 sec  
 T2 0.0000000 sec  
 T3 0.0000000 sec  
 WDEXT 0.0050000 sec  
 WDSK 0.0050000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -5.00 dB  
 SFO1 498.854919 MHz

F2 - Processing Parameters  
 SI 65536  
 SF 498.8500296 MHz  
 WDM EM  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

DJ VWR PLOT Parameters  
 CY 22.80 cm  
 CX 15.00 cm  
 FIP 9.000 ppm  
 FL 4480.65 Hz  
 F2 240.42 Hz  
 F3 240.42 Hz  
 PPMCM 0.41667 ppm/cm  
 HZCM 207.85419 Hz/cm

<sup>13</sup>C spectrum with <sup>1</sup>H decoupling



```

Current Data Parameters
NAME      ACH-1-13C-bmncph
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20191112
Time      9:07
INSTRUM   spect
PROBHD    5 mm bbo
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         544
DS         4
SWH        30303.031 Hz
FIDRES     0.462388 Hz
AQ         1.0813940 sec
RG         682
DE         16.50 usec
TE         288.0 K
D1         0.25000000 sec
DELTA     0.00000000 sec
MCREST    0.00000000 sec
MCPRER    0.01500000 sec

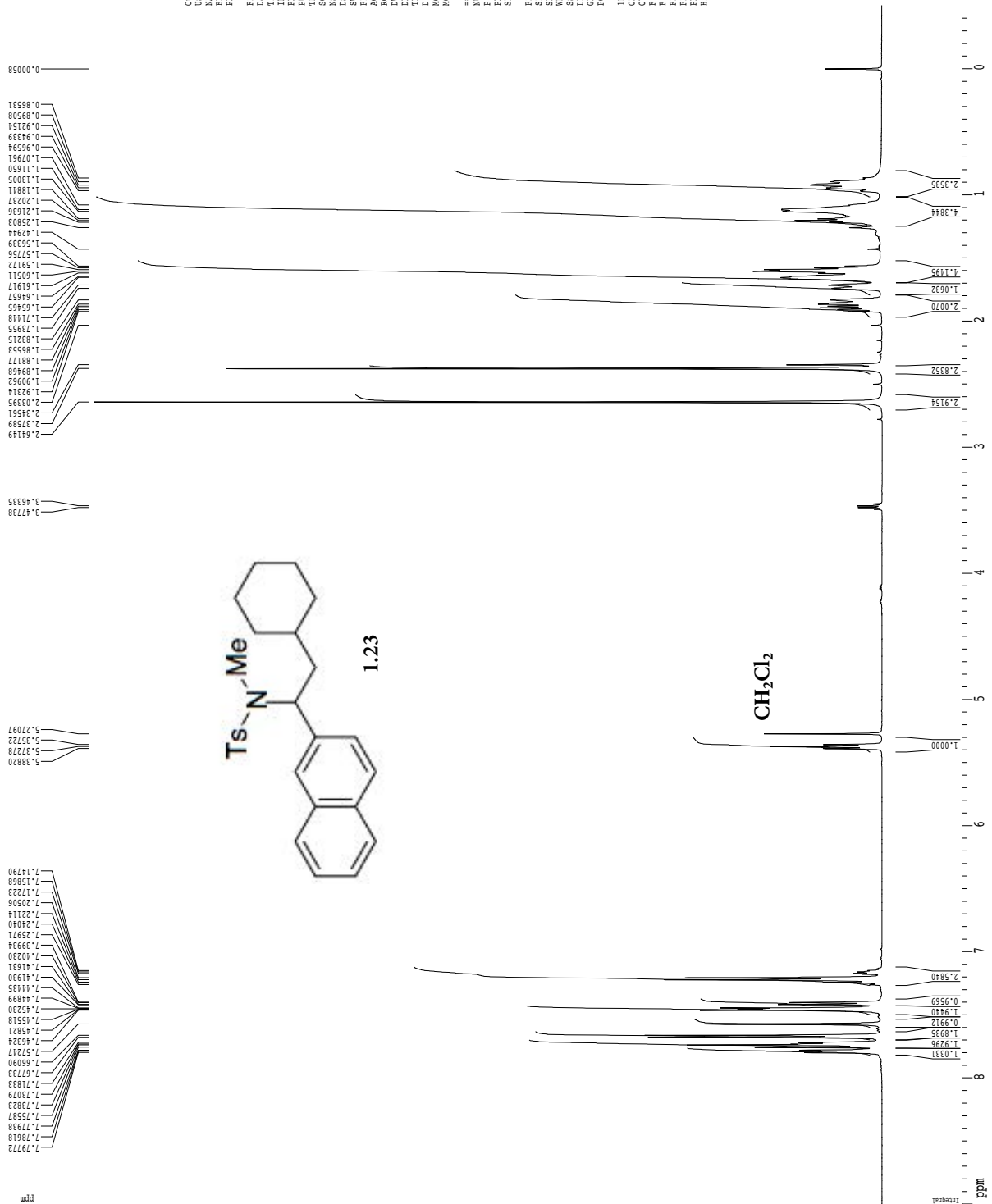
===== CHANNEL f1 =====
NUC1       13C
P1         10.00 usec
PL1        -6.00 dB
SFO1       125.4497000 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2         2.00 dB
SFO2       498.4524943 MHz

F2 - Processing parameters
SI         32768
SF         125.4452003 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         2.00

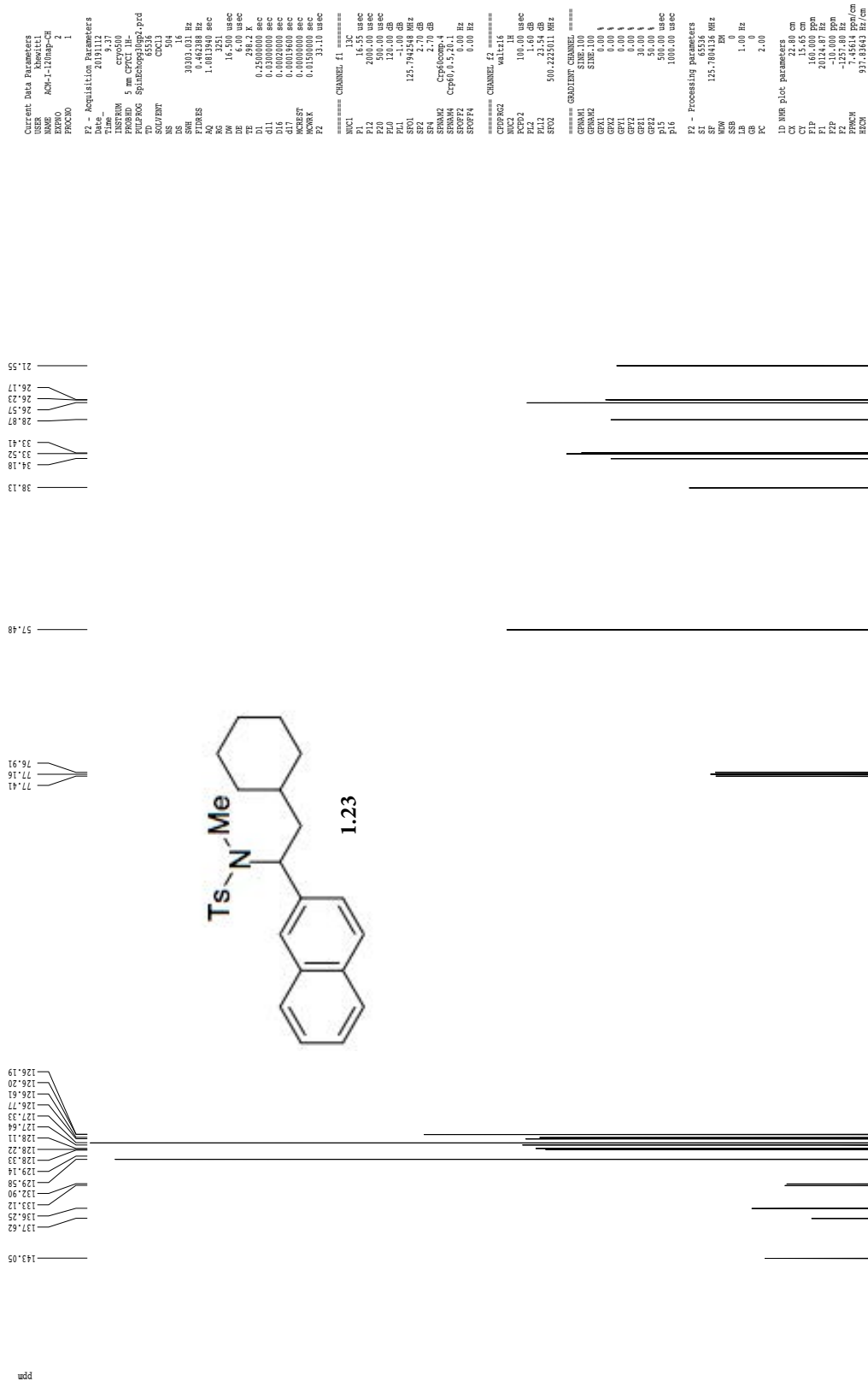
ID NMR plot parameters
X         12.50 cm
Y         12.50 cm
F1P       160.000 ppm
F2P       20069.75 Hz
P2P       -10.000 ppm
SFO1      125.4497000 MHz/cm
SFO2      498.4524943 MHz/cm
RECNO     935.25188 Hz/cm
    
```

1H spectrum

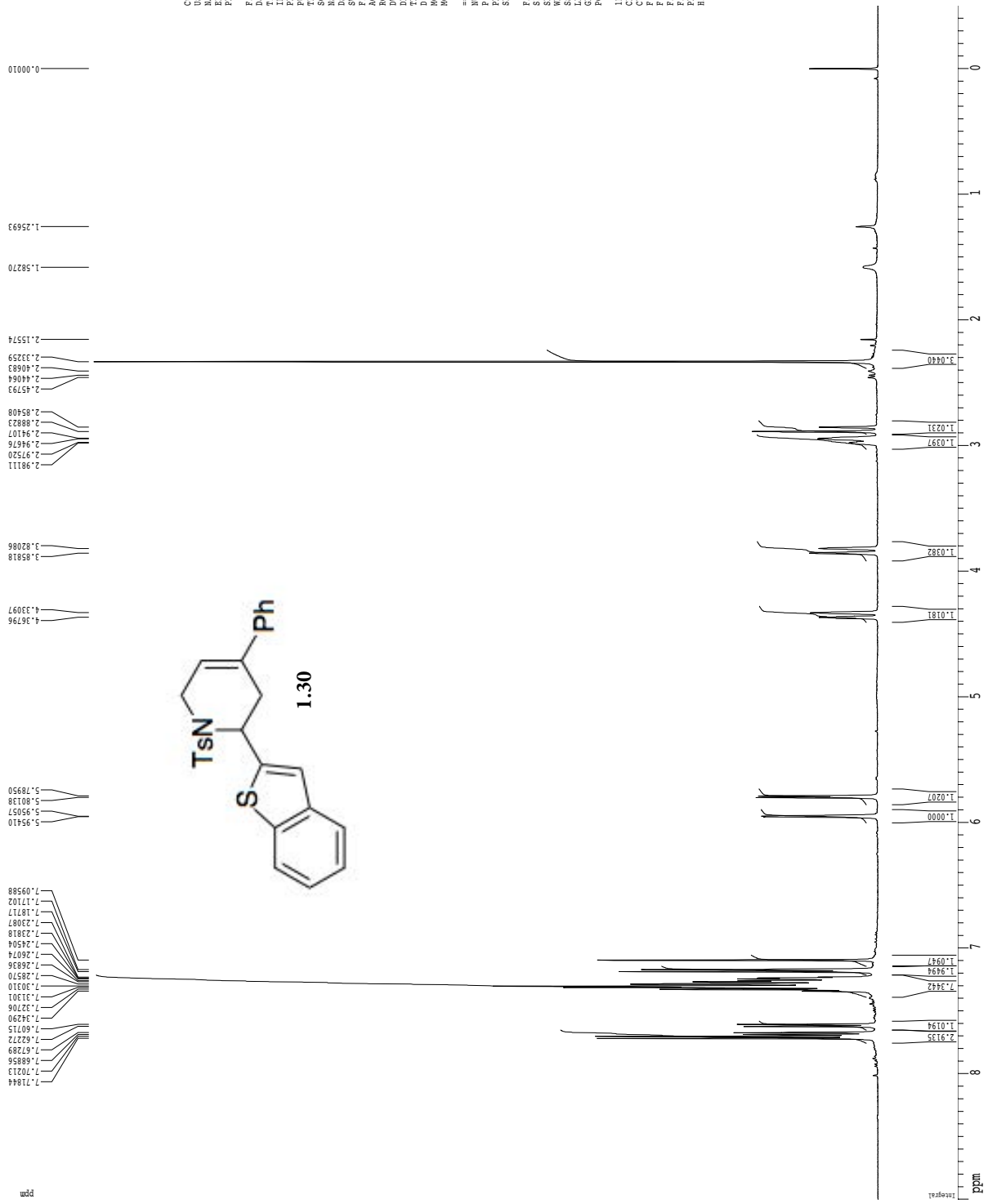


Current Data Parameters  
USER: khawatt  
NAME: ACM-1-130nap-CH  
EXPNO: 1  
PROCNO: 1  
F2 - Acquisition Parameters  
Date\_: 20191112  
Time: 9:24  
PROBHD: cryo1h  
PULPROG: zgpg30  
TD: 8172  
SOLVENT: CHCl3  
DS: 0  
SHE: 8012.820 Hz  
FIDRES: 0.09804 Hz  
AQ: 5.1998774 sec  
RG: 62.400 usec  
DE: 6.00 usec  
TE: 298.2 K  
NUC1: 13C  
NUC2: 1H  
MAGNET: 0.1000000 sec  
HWDW: 0.0150000 sec  
MORPH: 0.0150000 sec  
===== CHANNEL f1 =====  
NUC1: 1H  
P1: 1.50 usec  
PL1: 1.68 dB  
SFO1: 500.2335015 MHz  
F2 - Processing Parameters  
SI: 65536  
SF: 500.2200411 MHz  
WDW: EM  
SSB: 0  
GB: 0  
PC: 1.00  
LD WDR PLOE Parameters  
CY: 22.80 cm  
CX: 15.00 cm  
FIP: 9.000 ppm  
FL: 4501.500 Hz  
FZ: -250.11 Hz  
PRGM: 0.41667 ppm/cm  
HZCM: 208.44502 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



# 1H spectrum



Current Data Parameters  
 USER khmett  
 NAME KAN-1-23-1-2  
 EXNO 3  
 PROCD 1

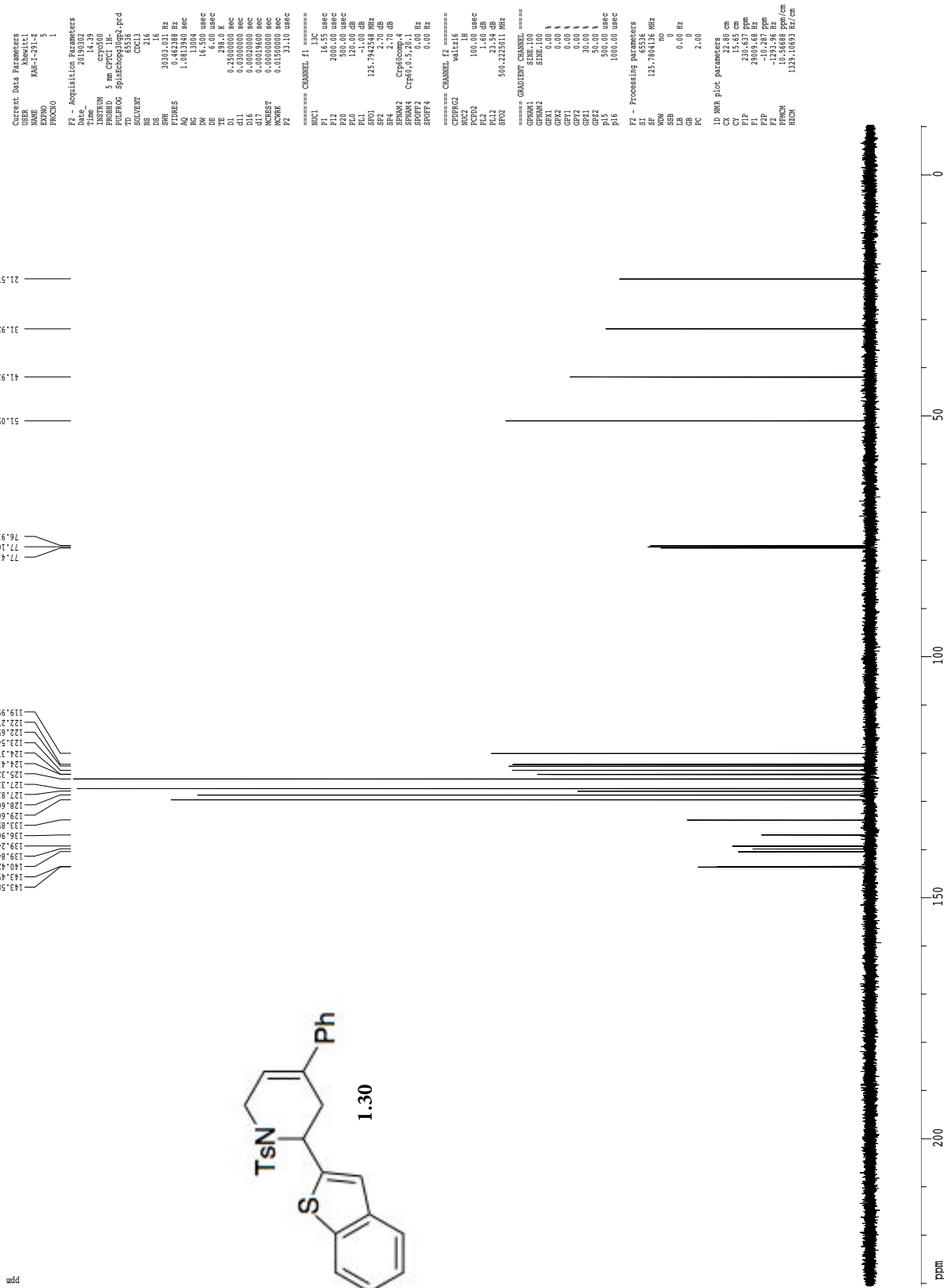
F2 - Acquisition Parameters  
 Date\_ 20190302  
 Time 14.24  
 PROBN C7V10  
 PULPROG zgpg30  
 PROCNO 5  
 TD 48074  
 SOLVENT CDCl3  
 DS 9  
 SFO 8012.820 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.3398677 sec  
 RG 62.400 usec  
 DE 6.00 usec  
 TE 298.2 K  
 INJ 0.100000 sec  
 MCBST 0.000000 sec  
 MCORE 0.0150000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 1.50 usec  
 PL1 1.66 dB  
 SFO1 500.2235015 MHz

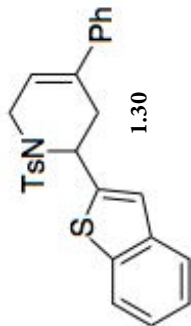
F2 - Processing Parameters  
 SI 65536  
 SF 500.2200426 MHz  
 WDM no  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

D0 NMR File Parameters  
 CY 22.80 cm  
 F1 15.00 cm  
 FIP 9.000 ppm  
 FL 4300.98 Hz  
 F2 -2560.1 Hz  
 PPGM 0.41667 ppm/cm  
 HZCM 206.44502 Hz/cm

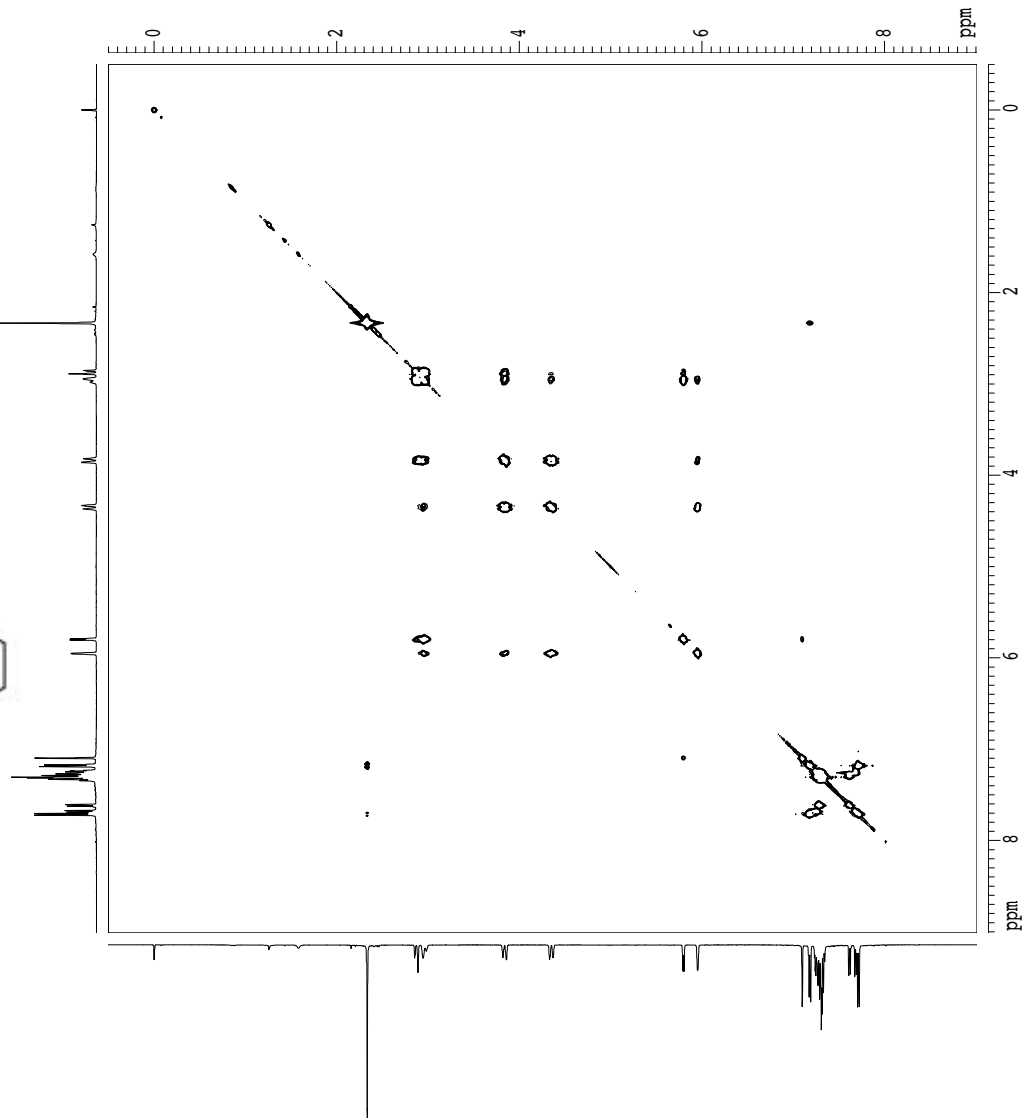
Z-restored spin-echo <sup>13</sup>C spectrum with <sup>1</sup>H decoupling







gcosy60



```

Current Data Parameters
USER      Khevit11
NAME      KAH-1-291-2
EXPNO     4
PROCNO    1

F2 - Acquisition Parameters
Date_     20190302
Time      14.26
INSTRUM   cryo500
PROBHD    5 mm cryoProbe
PULPROG   zgpg30
TD         2048
SOLVENT   CDCl3
NS         1
DS         1
AQ         8013.86 Hz
FIDRES    3.912510 Hz
AQ         0.1278452 sec
RG         114
DM         62.400 usec
DE         298.0 usec
TE         298.0 usec
d0         0.40000300 sec
d1         1.40000000 sec
d13        0.40000300 sec
d16        0.40000000 sec
d10        0.40000480 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
PC         0.00 usec
SFO1       500.225015 MHz

===== GRAB CHANNEL =====
GRAB1     sine,100
GRAB2     sine,100
GR3        0.00 usec
GR4        0.00 usec
GR5        0.00 usec
GR6        0.00 usec
GR7        17.00 usec
GR8        0.00 usec
P16        1000.00 usec

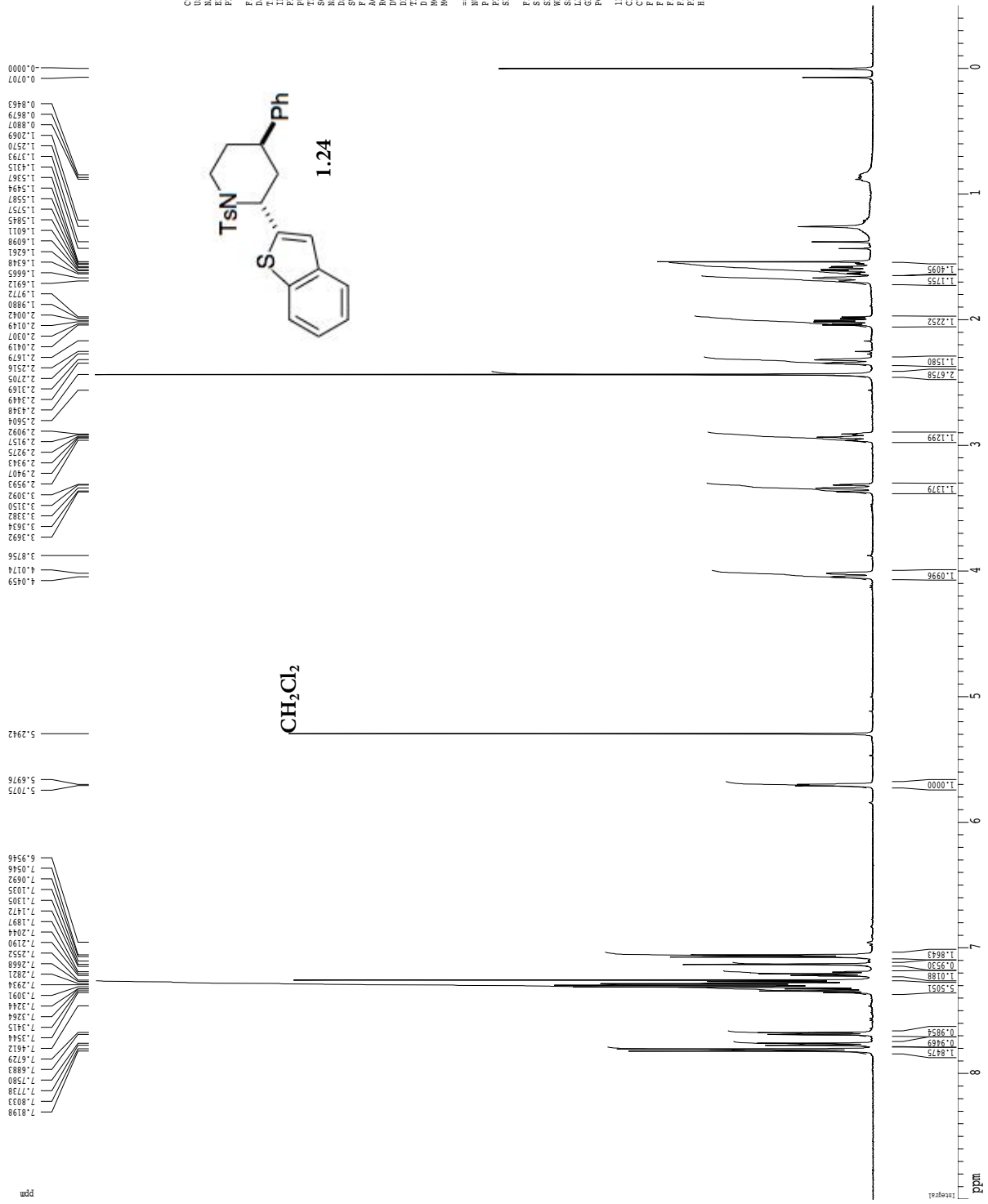
F1 - Acquisition parameters
NUC0       1
P0         5.12 usec
SFO0       500.225 MHz
SFO1       500.225 MHz
FIDRES    15.650040 Hz
SN         16.018 ppm
F0         0.00000000 usec

F2 - Processing parameters
SI         1024
SF         500.2200426 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0.00 Hz
PC         1.00

F1 - Processing parameters
SI         1024
SF         500.2200426 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0.00 Hz

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO     5.011 ppm
F2PO      45.011 ppm
F2PHI     0.500 ppm
F2PHI     -250.16 Hz
F1PLO     9.011 ppm
F1PO      4507.45 Hz
F1PHI     0.500 ppm
F1PHI     -250.16 Hz
F2PRCM    0.63407 ppm/cm
F2PRCM    317.17413 Hz/cm
F1PRCM    0.63407 ppm/cm
F1PRCM    317.17413 Hz/cm
  
```

1H spectrum



Current Data Parameters  
 USER KAH-1-214-2  
 NAME KAH-1-214-2  
 EXNO 1  
 PROXY 1

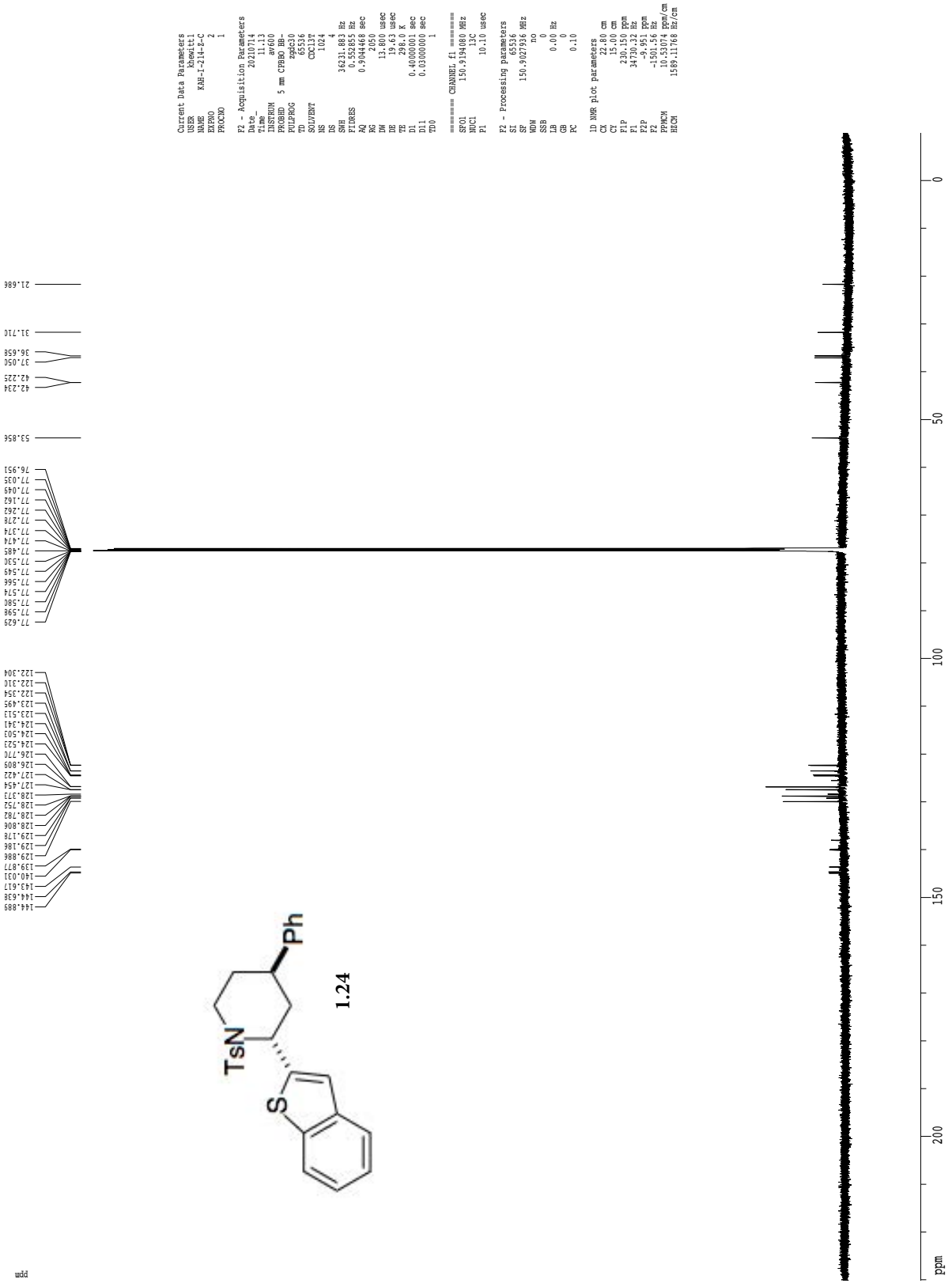
F2 - Acquisition Parameters  
 Date\_ 20181204  
 Time 11.04  
 PROXY 62700  
 PULPROG 5 mm CPC1H3  
 TD 48074  
 SOLVENT CHCl3  
 DS 9  
 SFO 8012.820 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.3398677 sec  
 RG 62.400 usec  
 DE 6.00 usec  
 TE 298.2 K  
 0.1600000 sec  
 0.0000000 sec  
 0.01500000 sec

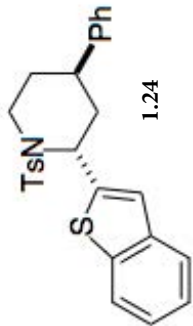
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 1.50 usec  
 PL1 1.66 dB  
 SFO1 500.2235015 MHz

F2 - Processing Parameters  
 SI 65536  
 SF 500.2200349 MHz  
 NO 4  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

DJ VWR Plc Parameters  
 CY 22.80 cm  
 CV 15.00 cm  
 FIP 9.000 ppm  
 FL 4501.98 Hz  
 F2 -250.14 Hz  
 F2 0.44667 ppm/cm  
 HZCN 206.44502 Hz/cm

c13.c





```

Current Data Parameters
=====
USER      Khowdt11
NAME      KAH-1-214-2
EXPNO     2
PROCNO    1

Date_     20181204
Time      11.06
INSTRUM   cryp500
PROBHD    5 mm cryo
PULPROG   zgpg30
SOLVENT   cosy@p60
TD         2048
SOLVENT   CDCl3
NS         1
DS         1
AQ         8013.84 Hz
RG         3.912510 Hz
FIDRES    0.1278452 sec
RG         362
AQ         62.400 usec
RG         288.0 usec
TE         0.00003300 sec
d0         1.00000000 sec
d1         0.00003000 sec
d13        0.00020000 sec
d16        0.00020000 sec
d10        0.00014000 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
PL1        0.00 dB
SFO1       500.225015 MHz

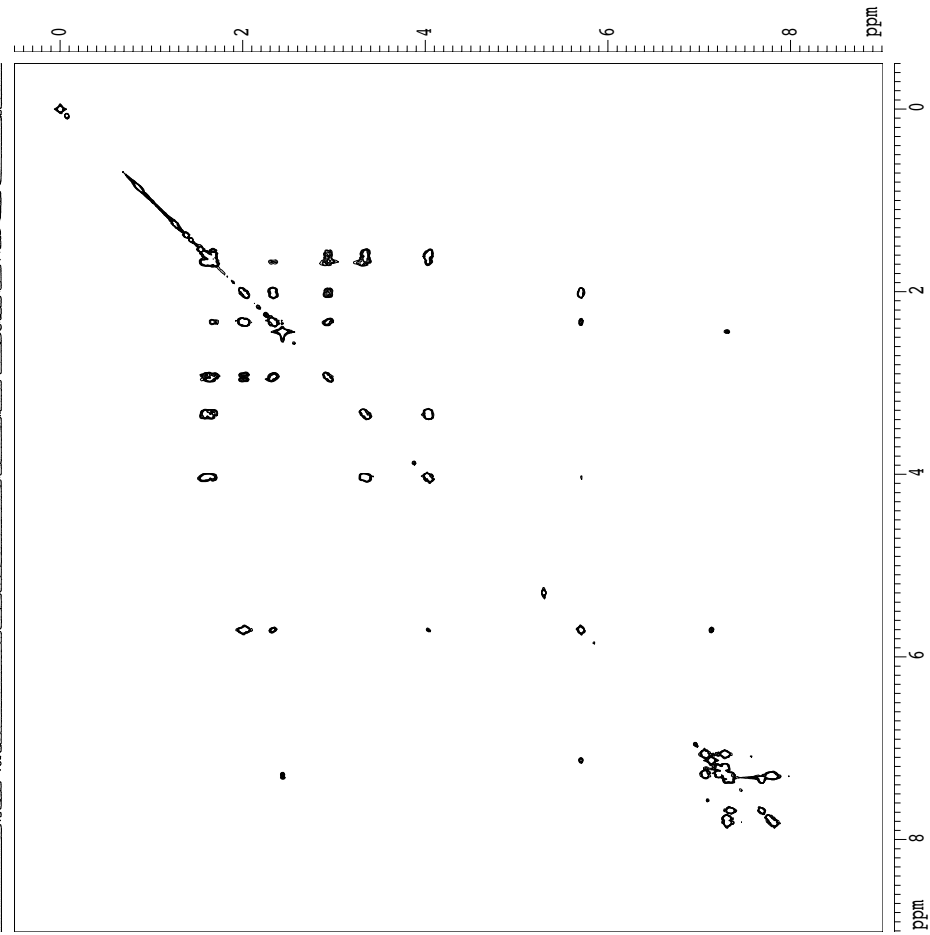
===== GRADIENT CHANNEL =====
GGRAD1     sine.100
GGRAD2     sine.100
GPR1       0.00 kHz
GPR2       0.00 kHz
GPR3       0.00 kHz
GPR4       0.00 kHz
GPR5       17.00 kHz
GPR6       17.00 kHz
P16        1000.00 usec

F1 - Acquisition parameters
=====
NUC0       13C
P0         5.10 usec
PL0        0.00 dB
SFO0       125.760150 MHz
FIDRES     500.225015 MHz
F2RES      15.650040 Hz
SM         16.018 ppm
PRMOB     VE

F2 - Processing parameters
=====
SI         1024
SF         500.2200349 MHz
WDW        EM
SSB        0
LB         0.00 Hz
GB         0.00 Hz
PC         1.00

F1 - Processing parameters
=====
SI         1024
SF         500.2200349 MHz
WDW        EM
SSB        0
LB         0.00 Hz
GB         0.00 Hz

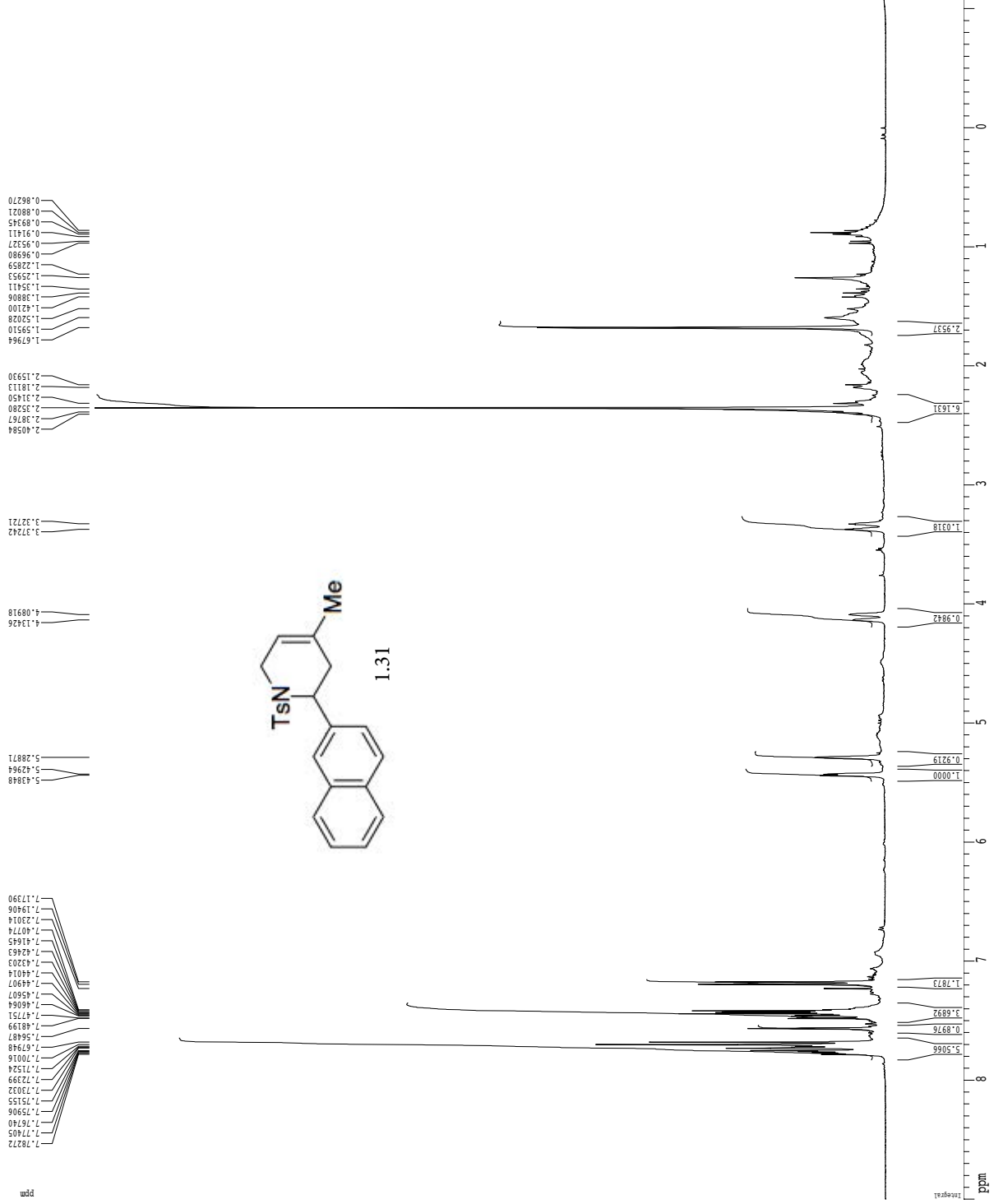
2D NMR Plot parameters
=====
CX2        15.00 cm
CY1        15.00 cm
F2PLO      9.011 ppm
F2PO       0.000000 Hz
F2PHI      0.000000 Hz
F2PHI2     -250.28 Hz
F1PLO      9.011 ppm
F1PO       0.000000 Hz
F1PHI      450.73 Hz
F1PHI2     -0.500000 Hz
F2PRMCM    0.63407 ppm/cm
F2PRMCM2   0.63407 ppm/cm
F1PRMCM    317.17413 Hz/cm
F1PRMCM2   317.17413 Hz/cm
    
```



gnoe



1H spectrum



Current Data Parameters  
 USER chembebe  
 NAME CN1-221  
 EXPNO 1  
 PROCNO 1

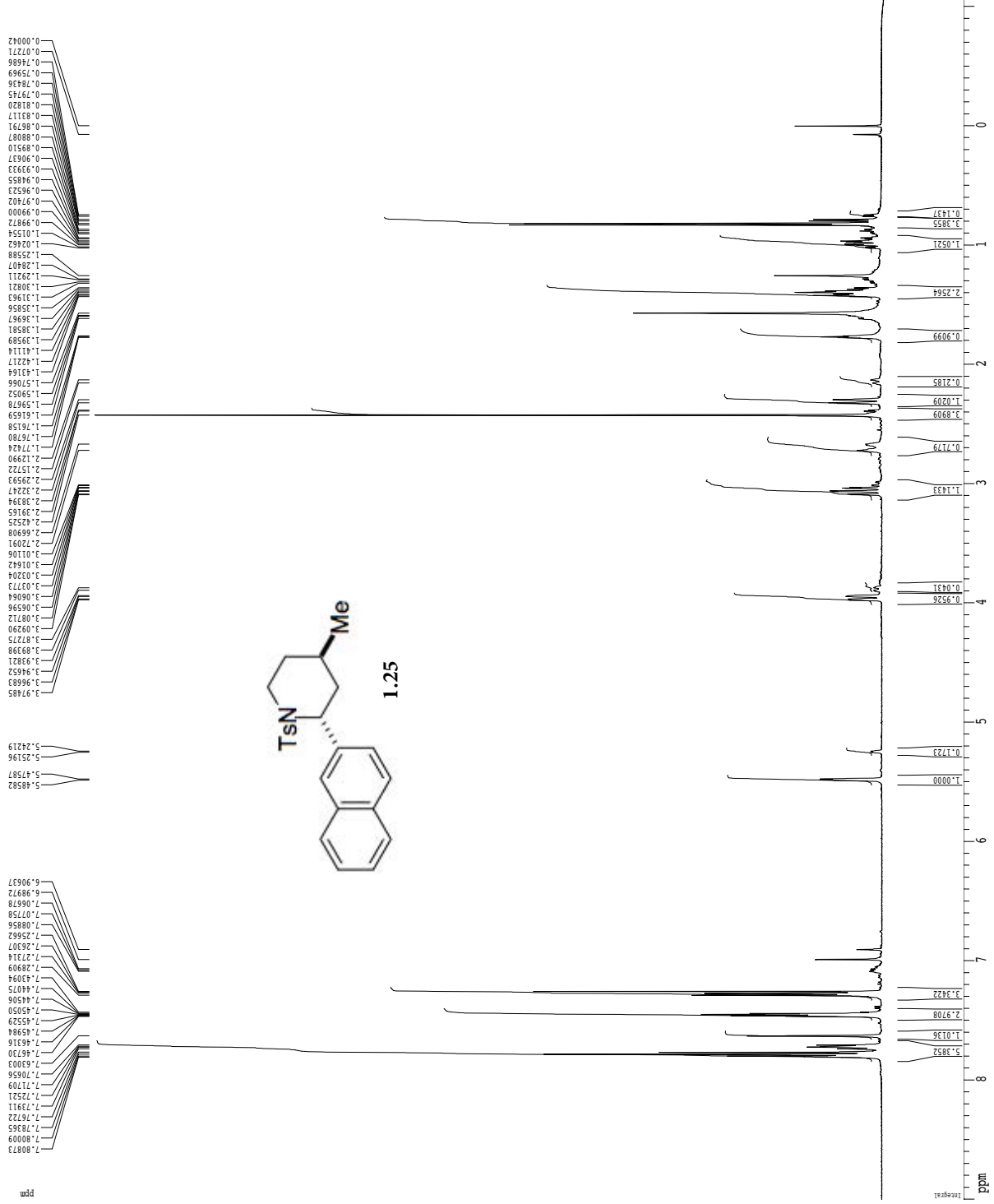
F2 - Acquisition Parameters  
 Date\_ 20210525  
 Time 11:17  
 PROBNM 111111  
 PULPROG zgpg30  
 PROCNO 5 nm QNP HET-7  
 TD 65536  
 SOLVENT CDCl3  
 DS 9  
 SFO 6410.258 Hz  
 FIDRES 0.097813 Hz  
 AQ 5.1118579 sec  
 RG 327.500  
 DW 78.000 usec  
 DE 4.50 usec  
 TE 298.2 K  
 0.16000000 sec  
 0.00000000 sec  
 0.00000000 sec  
 0.00000000 sec  
 0.00000000 sec  
 0.00000000 sec  
 0.00000000 sec  
 0.00000000 sec

===== CHANNEL f1 =====  
 NUC1 13  
 P1 12.00 usec  
 PL1 -1.66 dB  
 SFO1 400.1328009 MHz

F2 - Processing Parameters  
 SI 65536  
 SF 400.1300313 MHz  
 WDM no  
 LB 0.00 Hz  
 GB 0  
 PC 2.00

LD NMR P1 of Parameters  
 CY 22.80 cm  
 CV 15.00 cm  
 FIP 9.000 ppm  
 FL 3600.17 Hz  
 F2 437.51 Hz  
 F2 437.51 Hz  
 PPMCH 0.44269 ppm/cm  
 HZCH 177.13513 Hz/cm

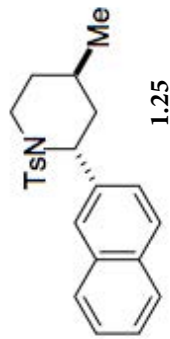
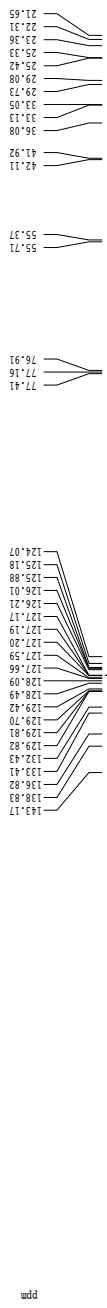
1H spectrum



Current Data Parameters  
 USER: cshen  
 NAME: CNH-4-21-full  
 EXPO: 1  
 PROCNO: 1

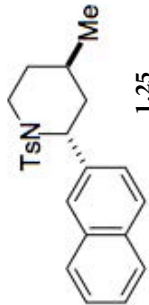
F2 - Acquisition Parameters  
 Date\_: 20210707  
 Time: 11:46  
 INSTR: spect  
 PROBNM: 4-21  
 PULPROG: zg30  
 TD: 8178  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 500.136260 MHz  
 SFE: 8012.820 Hz  
 FIDRES: 0.098044 Hz  
 AQ: 5.1998774 sec  
 RG: 62.400  
 DD: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 0.10000000 sec  
 T2: 0.00000000 sec  
 T3: 0.00000000 sec  
 T4: 0.00000000 sec  
 T5: 0.00000000 sec  
 T6: 0.00000000 sec  
 T7: 0.00000000 sec  
 T8: 0.00000000 sec  
 T9: 0.00000000 sec  
 T10: 0.00000000 sec  
 T11: 0.00000000 sec  
 T12: 0.00000000 sec  
 T13: 0.00000000 sec  
 T14: 0.00000000 sec  
 T15: 0.00000000 sec  
 T16: 0.00000000 sec  
 T17: 0.00000000 sec  
 T18: 0.00000000 sec  
 T19: 0.00000000 sec  
 T20: 0.00000000 sec  
 T21: 0.00000000 sec  
 T22: 0.00000000 sec  
 T23: 0.00000000 sec  
 T24: 0.00000000 sec  
 T25: 0.00000000 sec  
 T26: 0.00000000 sec  
 T27: 0.00000000 sec  
 T28: 0.00000000 sec  
 T29: 0.00000000 sec  
 T30: 0.00000000 sec  
 T31: 0.00000000 sec  
 T32: 0.00000000 sec  
 T33: 0.00000000 sec  
 T34: 0.00000000 sec  
 T35: 0.00000000 sec  
 T36: 0.00000000 sec  
 T37: 0.00000000 sec  
 T38: 0.00000000 sec  
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 T40: 0.00000000 sec  
 T41: 0.00000000 sec  
 T42: 0.00000000 sec  
 T43: 0.00000000 sec  
 T44: 0.00000000 sec  
 T45: 0.00000000 sec  
 T46: 0.00000000 sec  
 T47: 0.00000000 sec  
 T48: 0.00000000 sec  
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 T50: 0.00000000 sec  
 T51: 0.00000000 sec  
 T52: 0.00000000 sec  
 T53: 0.00000000 sec  
 T54: 0.00000000 sec  
 T55: 0.00000000 sec  
 T56: 0.00000000 sec  
 T57: 0.00000000 sec  
 T58: 0.00000000 sec  
 T59: 0.00000000 sec  
 T60: 0.00000000 sec  
 T61: 0.00000000 sec  
 T62: 0.00000000 sec  
 T63: 0.00000000 sec  
 T64: 0.00000000 sec  
 T65: 0.00000000 sec  
 T66: 0.00000000 sec  
 T67: 0.00000000 sec  
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 T91: 0.00000000 sec  
 T92: 0.00000000 sec  
 T93: 0.00000000 sec  
 T94: 0.00000000 sec  
 T95: 0.00000000 sec  
 T96: 0.00000000 sec  
 T97: 0.00000000 sec  
 T98: 0.00000000 sec  
 T99: 0.00000000 sec  
 T100: 0.00000000 sec

Z-restored spin-echo 13C spectrum with 1H decoupling

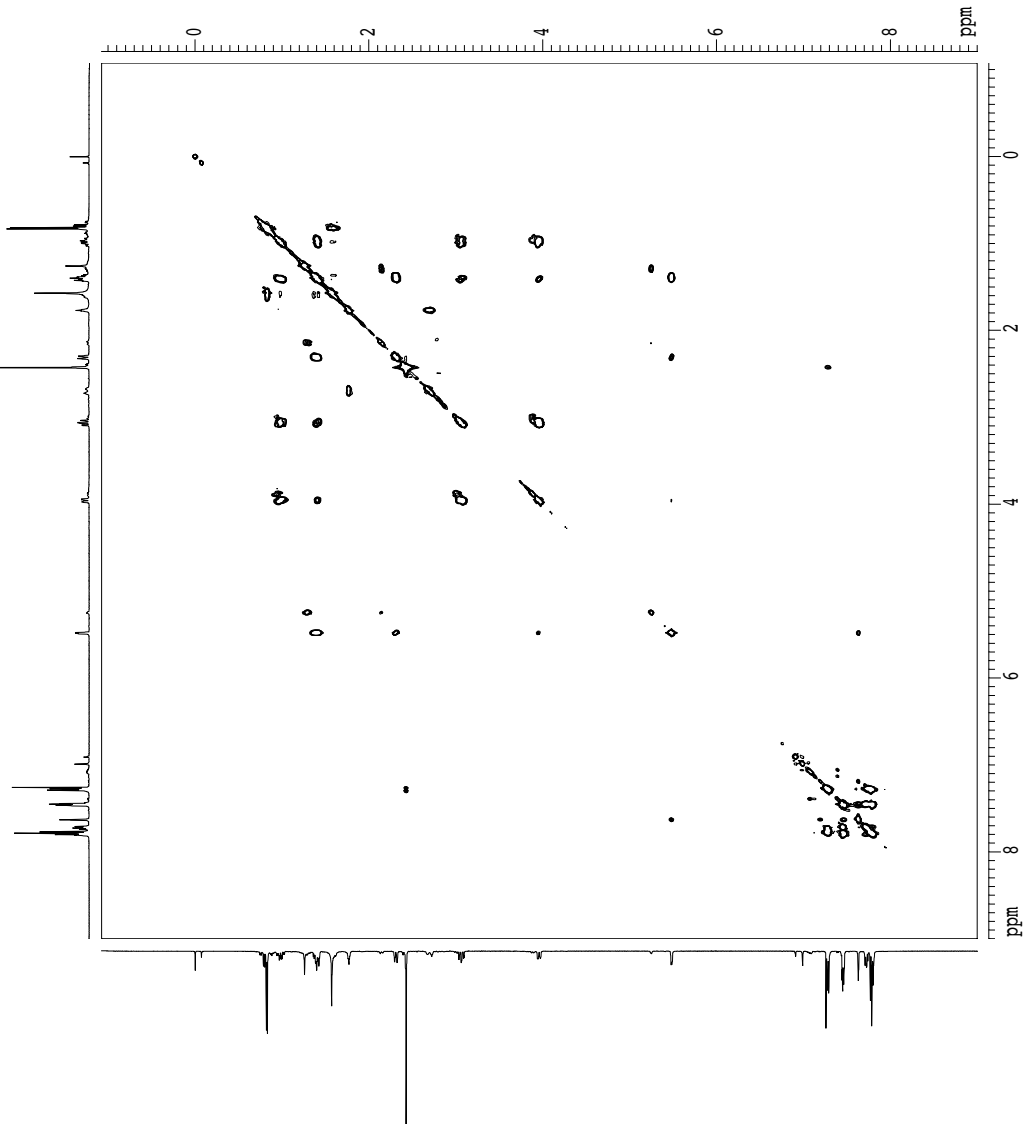


Current Data Parameters  
 USER cahebec  
 NAME CNH-7-21-011  
 DATE 2006  
 PROCNO 1  
 F2 - Acquisition Parameters  
 TIME 21.59  
 INSTRUM spect  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 4  
 SFO 30.81301 Hz  
 FIDRES 0.462388 Hz  
 AQ 1.18672 sec  
 RG 328.2 Hz  
 DW 16.500 usec  
 DE 6.00 usec  
 DI 0.2500000 sec  
 D1 0.0300000 sec  
 D11 0.0000000 sec  
 D12 0.0000000 sec  
 DC 0.0100000 sec  
 MCKEY 0.0000000 sec  
 MCHK 0.0000000 sec  
 F2 37.70 usec  
 ===== CHANNEL f1 =====  
 NUC1 13C  
 P1 14.50 usec  
 F1 125.760311 MHz  
 P2 2000.00 usec  
 F2 500.136050 MHz  
 P3 120.00 dB  
 P4 120.00 dB  
 SFO1 125.760311 MHz  
 SFO2 125.760311 MHz  
 SFO3 125.760311 MHz  
 SFO4 125.760311 MHz  
 SFO5 125.760311 MHz  
 SFO6 125.760311 MHz  
 SFO7 125.760311 MHz  
 SFO8 125.760311 MHz  
 SFO9 125.760311 MHz  
 SFO10 125.760311 MHz  
 SPAN30 Cp06,0.5,20,1  
 SFOFF2 0.00 Hz  
 SFOFF4 0.00 Hz  
 ===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 P1 1.00 usec  
 F1 500.136050 MHz  
 P2 100.00 usec  
 F2 14.60 MHz  
 P3 22.00 dB  
 P4 22.00 dB  
 SFO1 500.136050 MHz  
 SFO2 500.136050 MHz  
 ===== GRADIENT CHANNEL =====  
 GPM1 STE-100  
 STE-100  
 GA1 0.00 A  
 GA2 0.00 A  
 GA3 0.00 A  
 GA4 0.00 A  
 GA5 0.00 A  
 GA6 0.00 A  
 GA7 30.00 A  
 GA8 30.00 A  
 GA9 30.00 A  
 GA10 30.00 A  
 GA11 30.00 A  
 GA12 30.00 A  
 GA13 1000.00 usec  
 GA14 1000.00 usec  
 GA15 1000.00 usec  
 GA16 1000.00 usec  
 F2 - Processing parameters  
 SI 125.760311 MHz  
 SF 125.760311 MHz  
 WDW no  
 SSB 0  
 GB 0  
 CB 0  
 PC 2.00  
 LD MR J-CZ parameters  
 CX 22.80 cm  
 CY 15.45 cm  
 CZ 15.45 cm  
 F1 2.000844 ppm  
 F2 2.000844 ppm  
 F3 -10.487 ppm  
 F4 -10.487 ppm  
 F5 -10.487 ppm  
 F6 -10.487 ppm  
 F7 -10.487 ppm  
 F8 -10.487 ppm  
 F9 -10.487 ppm  
 F10 -10.487 ppm  
 F11 -10.487 ppm  
 F12 -10.487 ppm  
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 F14 -10.487 ppm  
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 F19 -10.487 ppm  
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 F88 -10.487 ppm  
 F89 -10.487 ppm  
 F90 -10.487 ppm  
 F91 -10.487 ppm  
 F92 -10.487 ppm  
 F93 -10.487 ppm  
 F94 -10.487 ppm  
 F95 -10.487 ppm  
 F96 -10.487 ppm  
 F97 -10.487 ppm  
 F98 -10.487 ppm  
 F99 -10.487 ppm  
 F100 -10.487 ppm





gcosy60

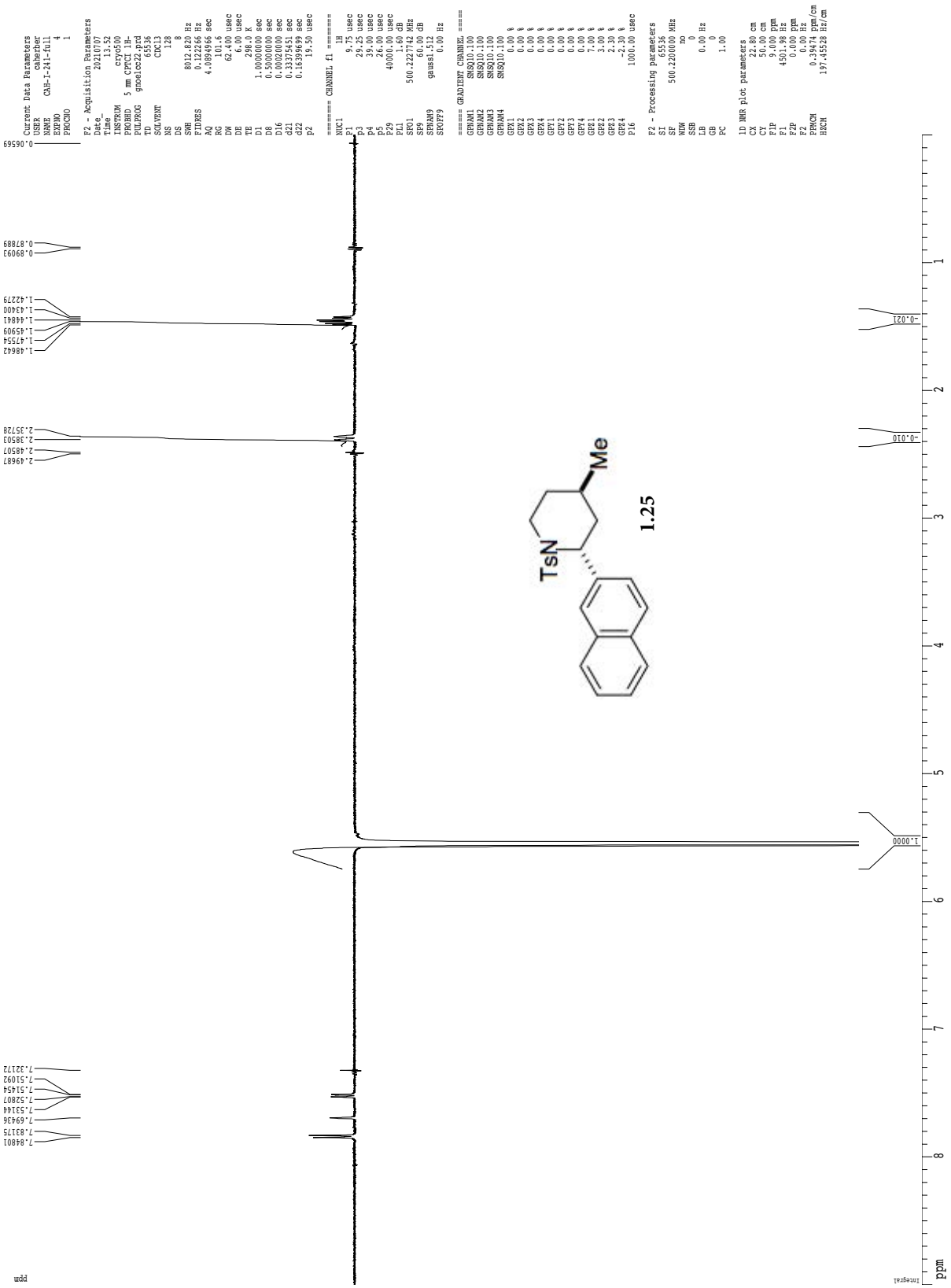


```

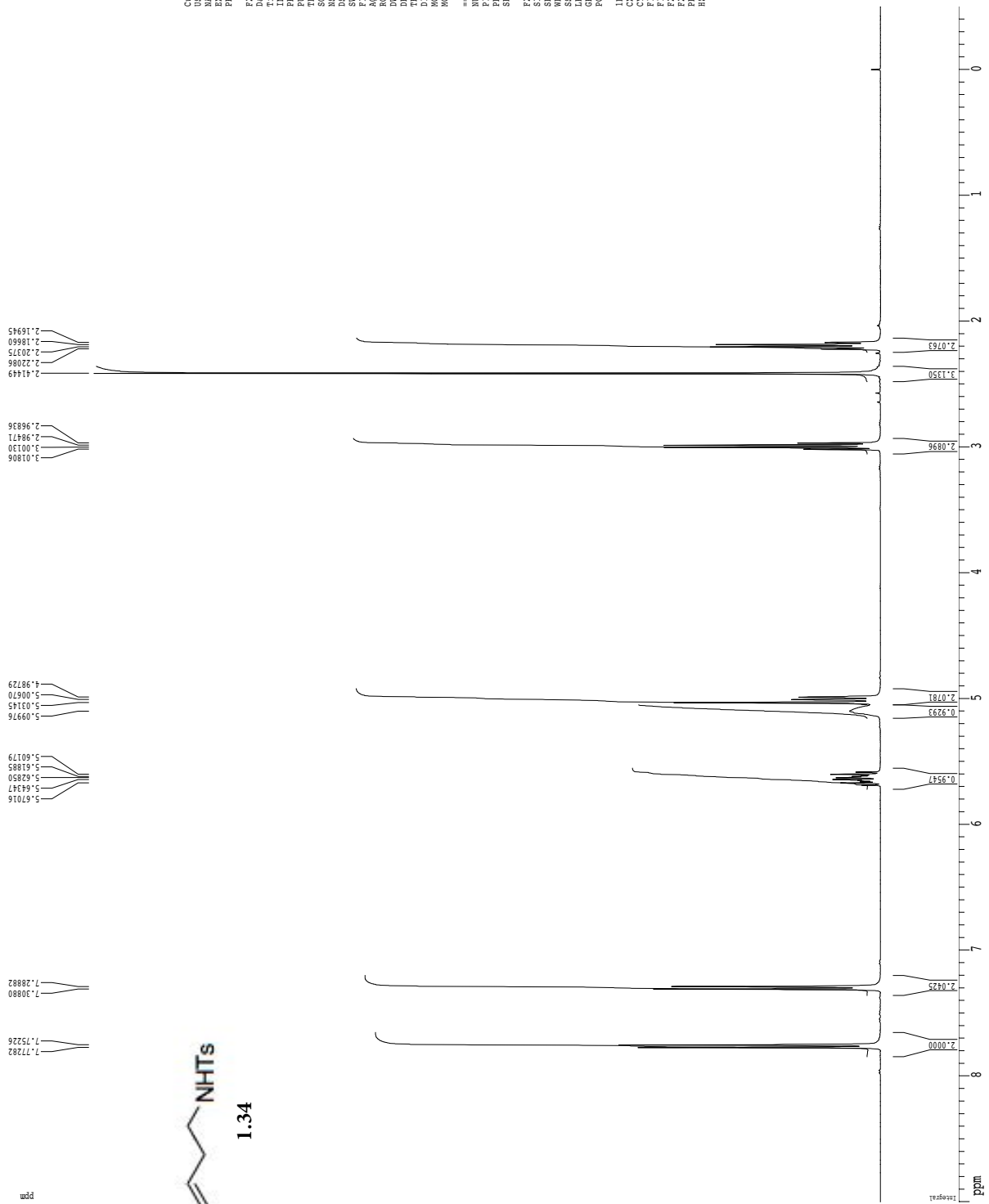
Current Data Parameters
=====
NAME          gcosy60
EXPNO         2
PROCNO        1
=====
Date_         20070709
Time         11:49
INSTRUM      cryo500
PROBHD       5 mm CPTCI 1H-
PULPROG      cosyprg60.prd
TD           2048
SFO          500.131
RG           655
AQ           0.00012480 sec
RG           655
DE           6.400 usec
TE           298.0 K
DO          0.0000300 sec
D1          1.0000000 sec
D13         0.0000300 sec
D16         0.00020000 sec
IN0         0.00012480 sec
===== CHANNEL f1 =====
NUC1         13
P1           9.75 usec
PL1          1.60 dB
SFO1         500.2235015 MHz
===== GRABBER CHANNEL =====
CPDPRG2      SPSIO100
SFO2         500.131
CPDPRG1      SPSIO100
GPA1         0.00 %
GPA2         0.00 %
GPI1         0.00 %
GPI2         0.00 %
GPD1         17.00 %
GPD2         17.00 %
PL6          1000.00 usec
=====
F1 - Acquisition parameters
=====
WDW          EM
SSB          0
GB           0
PC           1.00
=====
F2 - Processing parameters
=====
SI           1024
SF           500.2200327 MHz
WDW          SINE
SSB          0
GB           0
PC           1.00
=====
F1 - Processing parameters
=====
SI           1024
SF           500.2200327 MHz
WDW          SINE
SSB          0
GB           0
PC           1.00
=====
2D NMR plot parameters
=====
CX2          15.00 cm
CX1          15.00 cm
F2FLO        9.000 ppm
F2FID        48.000 ppm
F2PHI        -51.075 deg
F2H1         -537.60 Hz
F1FLO        9.000 ppm
F1FID        4501.98 Hz
F1PHI        -51.075 deg
F2F2MCH1     0.67165 ppm/cm
F2F2MCH2     335.97217 Hz/cm
F1F2MCH1     0.67165 ppm/cm
F1F2MCH2     335.97217 Hz/cm

```

gnoe



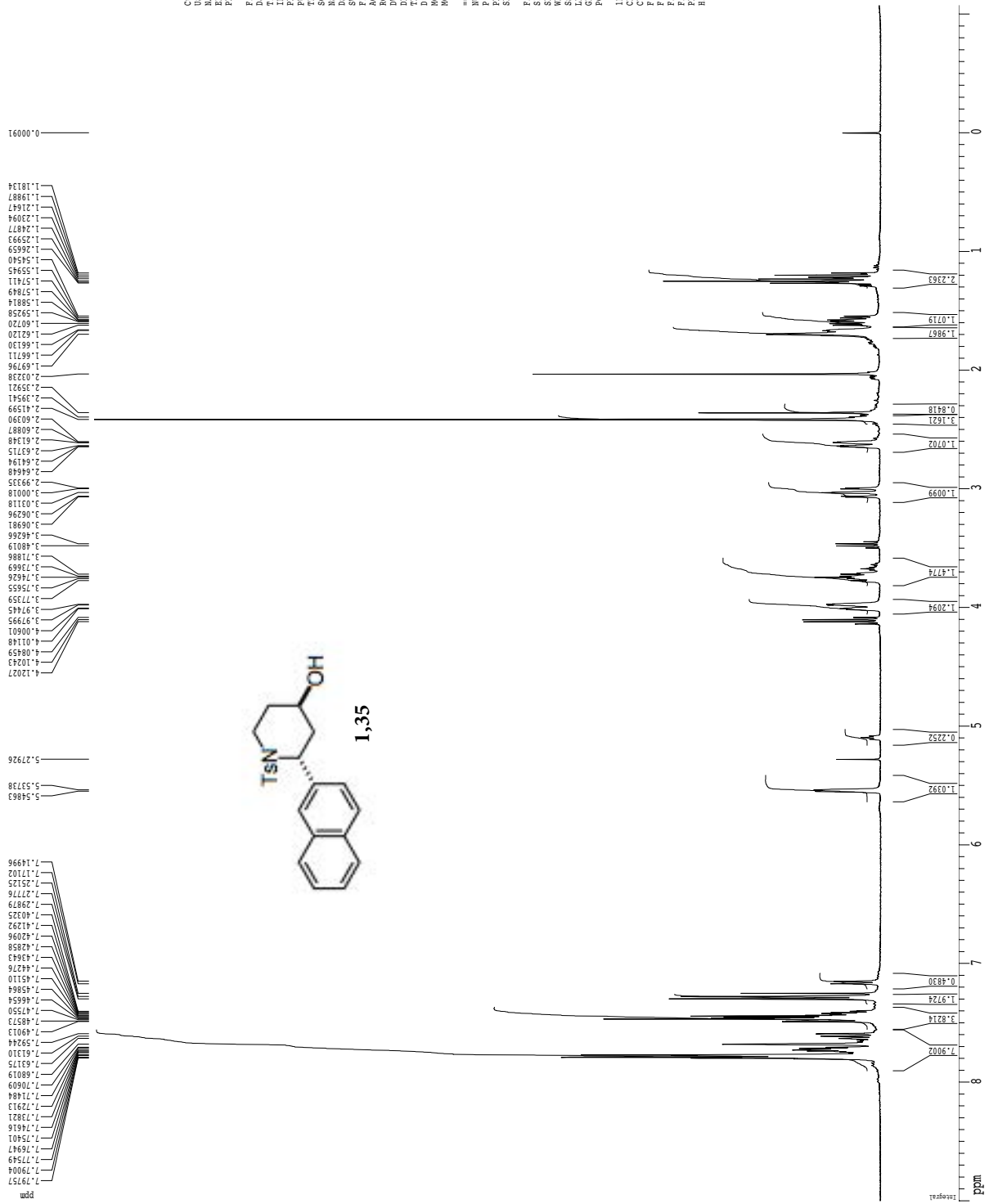
1H spectrum



Current Data Parameters  
 USER kshel11  
 NAME KNE-1-102E  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 2018022  
 Time 12.03  
 Date\_ 201802  
 Time 12.03  
 PULPROG zgpg30  
 PROCNO 5 nm QNP zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 DS 2  
 SWH 6410.256 Hz  
 FIDRES 0.166673 Hz  
 AQ 2.999929 sec  
 RG 327.5  
 DW 78.000 ns  
 DE 4.50 ns  
 TE 297.2 K  
 T1 1.000000 sec  
 T2 0.000000 sec  
 T3 0.000000 sec  
 T4 0.000000 sec  
 T5 0.000000 sec  
 T6 0.000000 sec  
 T7 0.000000 sec  
 T8 0.000000 sec  
 T9 0.000000 sec  
 T10 0.000000 sec  
 T11 0.000000 sec  
 T12 0.000000 sec  
 T13 0.000000 sec  
 T14 0.000000 sec  
 T15 0.000000 sec  
 T16 0.000000 sec  
 T17 0.000000 sec  
 T18 0.000000 sec  
 T19 0.000000 sec  
 T20 0.000000 sec  
 T21 0.000000 sec  
 T22 0.000000 sec  
 T23 0.000000 sec  
 T24 0.000000 sec  
 T25 0.000000 sec  
 T26 0.000000 sec  
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 T34 0.000000 sec  
 T35 0.000000 sec  
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 T37 0.000000 sec  
 T38 0.000000 sec  
 T39 0.000000 sec  
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 T41 0.000000 sec  
 T42 0.000000 sec  
 T43 0.000000 sec  
 T44 0.000000 sec  
 T45 0.000000 sec  
 T46 0.000000 sec  
 T47 0.000000 sec  
 T48 0.000000 sec  
 T49 0.000000 sec  
 T50 0.000000 sec  
 T51 0.000000 sec  
 T52 0.000000 sec  
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 T54 0.000000 sec  
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 T57 0.000000 sec  
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 T60 0.000000 sec  
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 T62 0.000000 sec  
 T63 0.000000 sec  
 T64 0.000000 sec  
 T65 0.000000 sec  
 T66 0.000000 sec  
 T67 0.000000 sec  
 T68 0.000000 sec  
 T69 0.000000 sec  
 T70 0.000000 sec  
 T71 0.000000 sec  
 T72 0.000000 sec  
 T73 0.000000 sec  
 T74 0.000000 sec  
 T75 0.000000 sec  
 T76 0.000000 sec  
 T77 0.000000 sec  
 T78 0.000000 sec  
 T79 0.000000 sec  
 T80 0.000000 sec  
 T81 0.000000 sec  
 T82 0.000000 sec  
 T83 0.000000 sec  
 T84 0.000000 sec  
 T85 0.000000 sec  
 T86 0.000000 sec  
 T87 0.000000 sec  
 T88 0.000000 sec  
 T89 0.000000 sec  
 T90 0.000000 sec  
 T91 0.000000 sec  
 T92 0.000000 sec  
 T93 0.000000 sec  
 T94 0.000000 sec  
 T95 0.000000 sec  
 T96 0.000000 sec  
 T97 0.000000 sec  
 T98 0.000000 sec  
 T99 0.000000 sec  
 T100 0.000000 sec

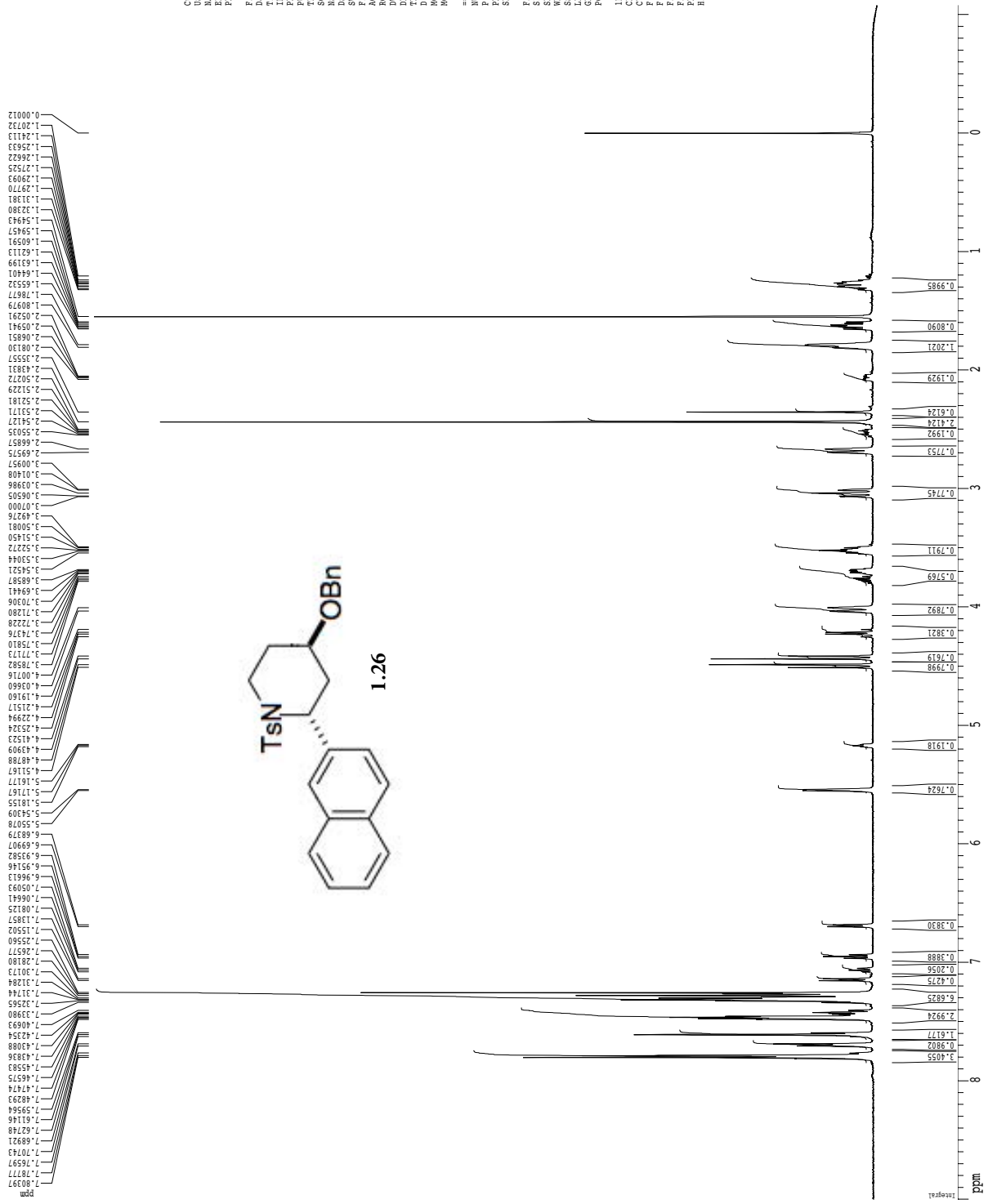
1H spectrum



Current Data Parameters  
 USER: chembe1  
 NAME: CNF-201  
 EXPNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20210330  
 Time: 18.04  
 PROBHD: 5 mm QNP HET  
 PULPROG: zg30  
 TD: 65536  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 6410.256 Hz  
 FIDRES: 0.097813 Hz  
 AQ: 5.1118579 sec  
 RG: 78.000  
 DD: 78.000 usec  
 DE: 4.50 usec  
 TE: 298.2 K  
 T1: 0.10000000 sec  
 T2: 0.00000000 sec  
 T3: 0.00000000 sec  
 MCHRES: 0.00150000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -1.60 dB  
 SFO1: 400.1326009 MHz  
 F2 - Processing Parameters  
 SI: 65536  
 SF: 400.1300245 MHz  
 WDW: no  
 GB: 0.00 Hz  
 PC: 2.00  
 ID: NMR PLOT Parameters  
 CY: 22.80 cm  
 CX: 15.00 cm  
 FIP: 9.000 ppm  
 F1: 3600.17 Hz  
 F2: 426.71 Hz  
 F3: -426.71 Hz  
 PPMCM: 0.44173 ppm/cm  
 HZCM: 176.74925 Hz/cm



1H spectrum



Current Data Parameters  
 USER: cshelmer  
 NAME: CSH-4-236-4ull  
 EXPO: 1  
 PROCNO: 1

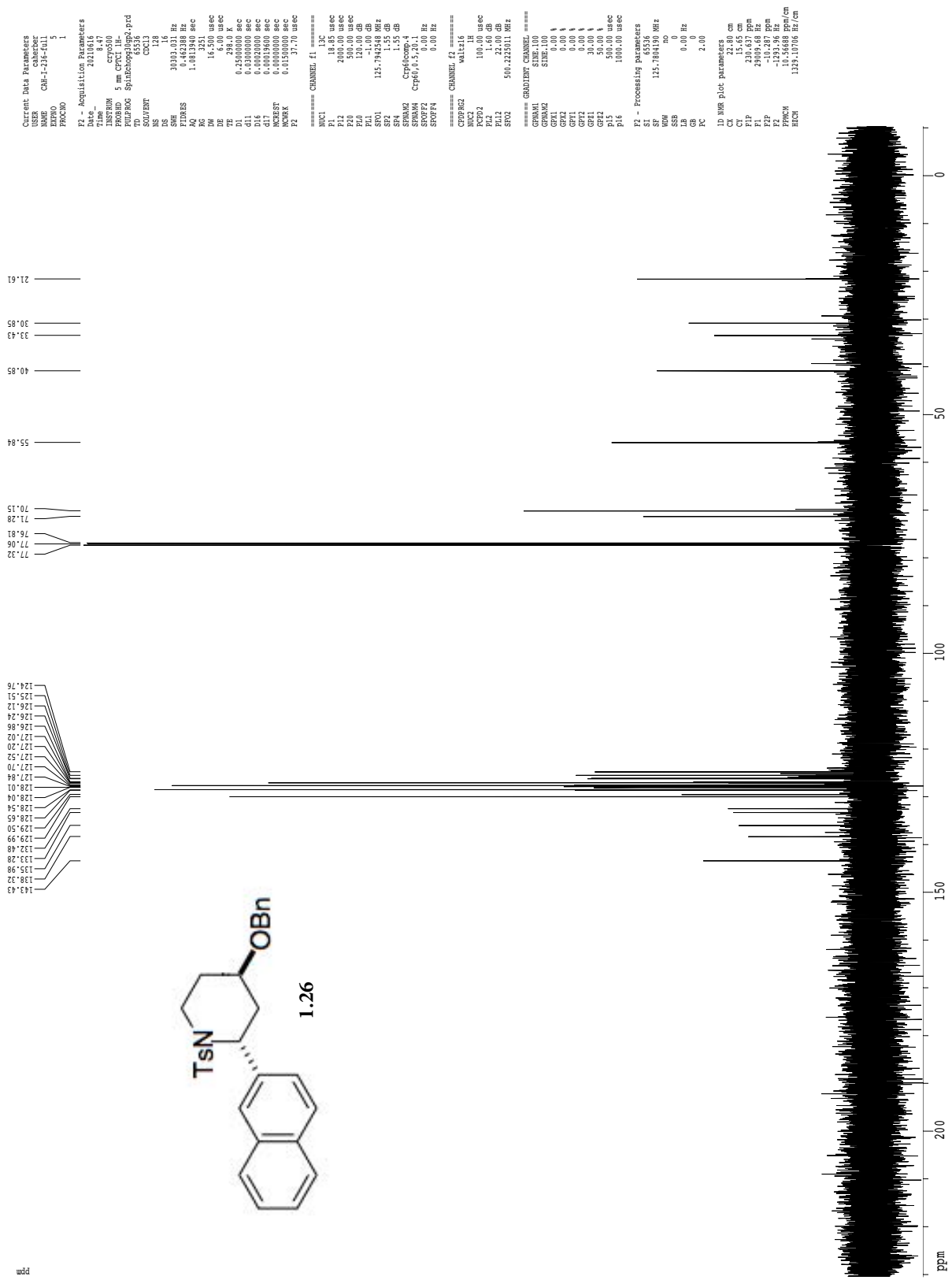
F2 - Acquisition Parameters  
 Date\_: 20210616  
 Time: 7:56  
 INSTR: cryo1h0  
 PROBNM: 5 mm CRYO-1H  
 PULPROG: zgpg30  
 TD: 48074  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 8012.820 Hz  
 SFE: 0.166677 Hz  
 AQ: 2.3998677 sec  
 RG: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 0.10000000 sec  
 T2: 0.00000000 sec  
 T3: 0.00000000 sec  
 MCHRG: 0.01500000 sec

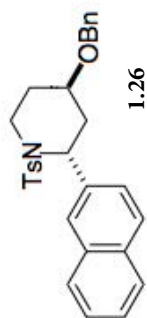
===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 9.75 usec  
 PL1: 1.66 dB  
 SFO1: 500.2235015 MHz

F2 - Processing Parameters  
 SI: 65536  
 SF: 500.2200329 MHz  
 NDM: no  
 LSF: 0.00 Hz  
 GB: 0  
 PC: 1.00

ID: NMR file parameters  
 CY: 22.80 cm  
 CV: 15.00 cm  
 PIP: 9.000 ppm  
 FI: 4500.798 Hz  
 F2: 537.75 Hz  
 PPMCM: 0.44189 ppm/cm  
 HZCM: 221.03986 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling





gcosy60

Current Data Parameters  
 USRP 2006  
 CHANNEL CAM-1-426-FU1  
 EXPRO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20060606  
 Time\_ 8:03  
 INSTRUM cryo600  
 PROBRG 5 mm CPXI 1H-  
 PULPROG zgpg30.prd  
 TD 2048  
 SFO 500.136261 MHz  
 AQUEVENT CXC11  
 NS 16  
 DS 16  
 SWH 4734.849 Hz  
 FIDRES 2.311938 Hz  
 AQ 0.22162188 sec  
 SFO2 500.136261 MHz  
 DM 105.600 usec  
 DE 6.00 usec  
 TE 298.0 K  
 d0 0.0000300 sec  
 d1 0.0000300 sec  
 d13 0.0000300 sec  
 d16 0.0002000 sec  
 INO 0.0002120 sec

===== CHANNEL F1 =====  
 NUCL1 15N  
 P1 9.75 usec  
 PL1 1.60 dB  
 SFO1 500.2221588 MHz

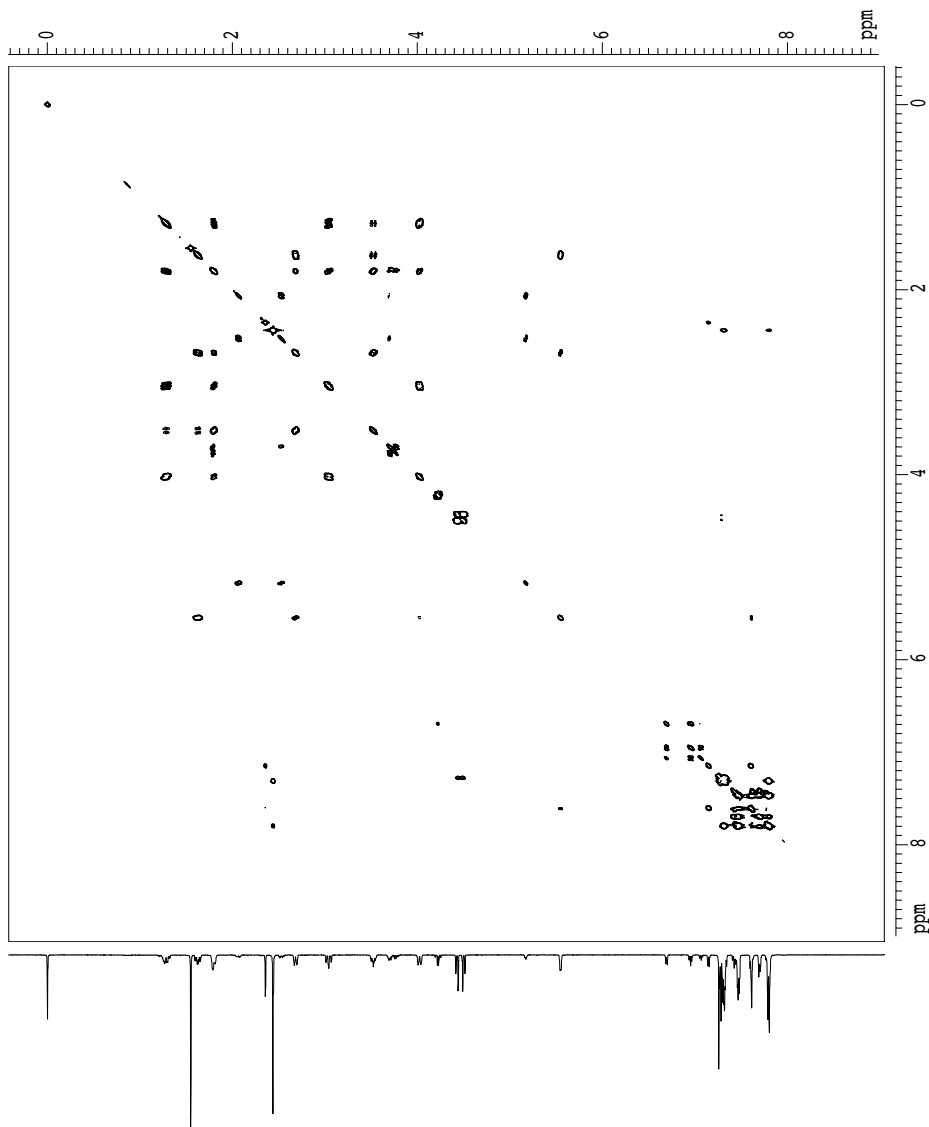
===== GRABBER CHANNEL =====  
 CHANNEL1 15N  
 CPBRM2 SWS10.100  
 GPX1 0.00 %  
 GPX2 0.00 %  
 GPT1 0.00 %  
 GPZ1 0.00 %  
 GPZ2 17.00 %  
 P16 1000.00 usec

F1 - Acquisition Parameters  
 NS0 512  
 TD 512  
 SFO1 500.2222 MHz  
 FIDRES 9.47751 Hz  
 SN 9.465 ppm  
 FMODE QF

F2 - Processing Parameters  
 SI 1024  
 SF 500.2200329 MHz  
 WDW SINE  
 SSB 0  
 LB 0 Hz  
 GB 0  
 PC 1.00

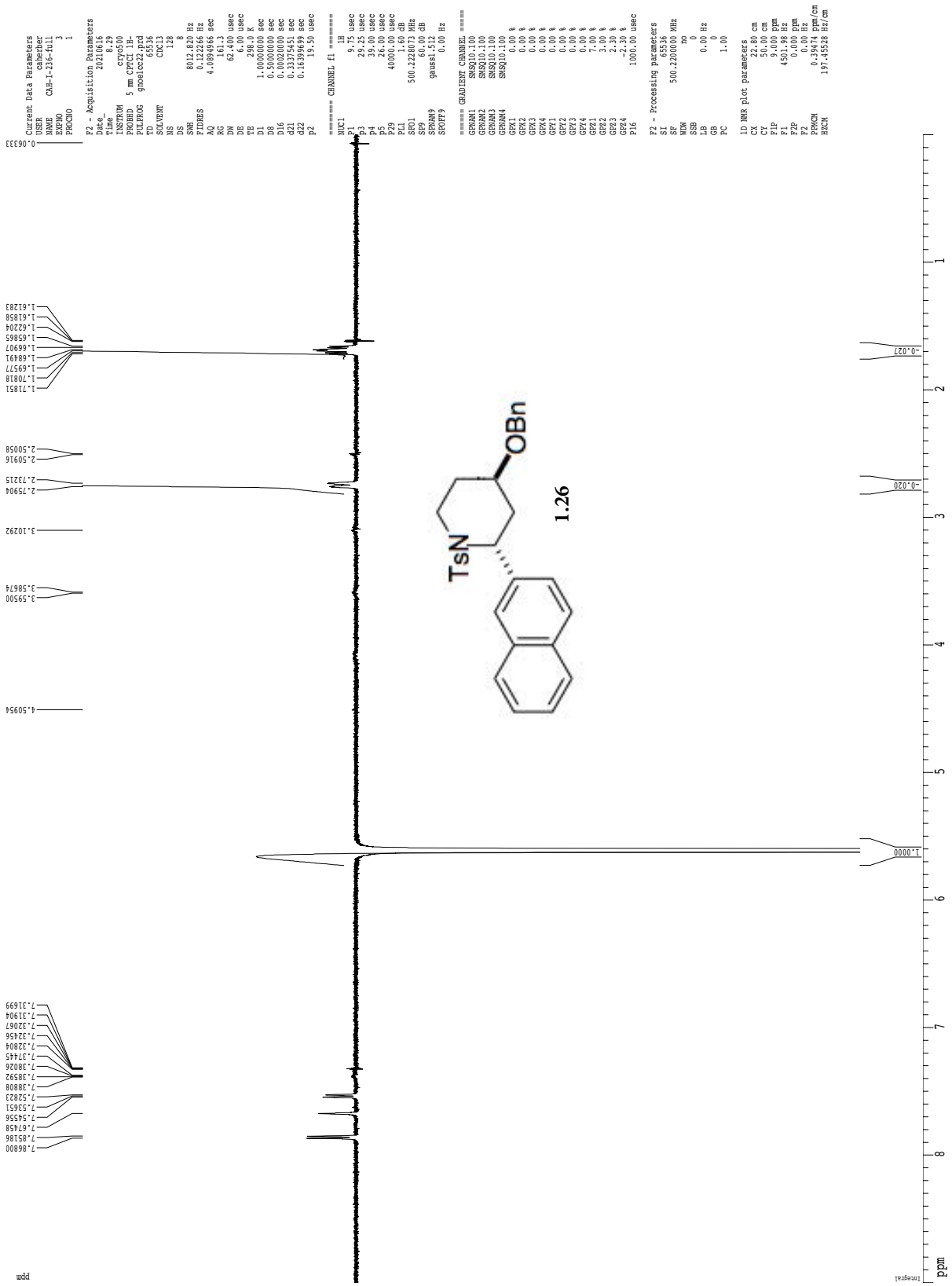
F1 - Processing Parameters  
 SI 1024  
 SF 500.2200329 MHz  
 WDW SINE  
 SSB 0  
 LB 0 Hz  
 GB 0  
 PC 1.00

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 FZPLO 9.048 ppm  
 FZPHI 48.667 Hz  
 FZPHI -20.417 ppm  
 FZHI -208.63 Hz  
 FZLO 9.048 ppm  
 FZLO 4526.21 Hz  
 FZHI -20.417 ppm  
 FZHI -20.417 ppm  
 FZPHI 0.63104 ppm/cm  
 FZPHI 315.65659 Hz/cm  
 FZPHI 0.63104 ppm/cm  
 FZPHI 315.65659 Hz/cm

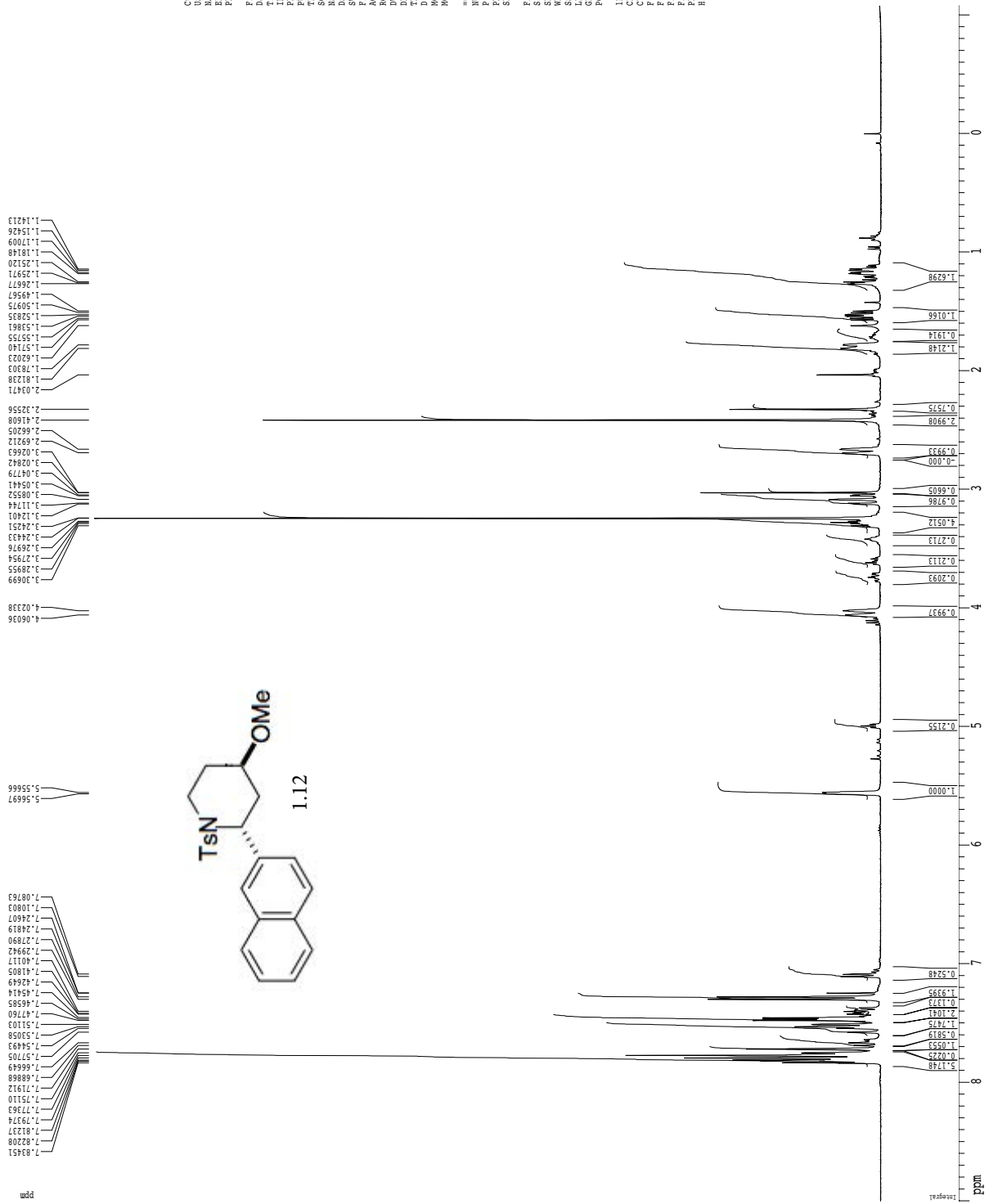




gnoe

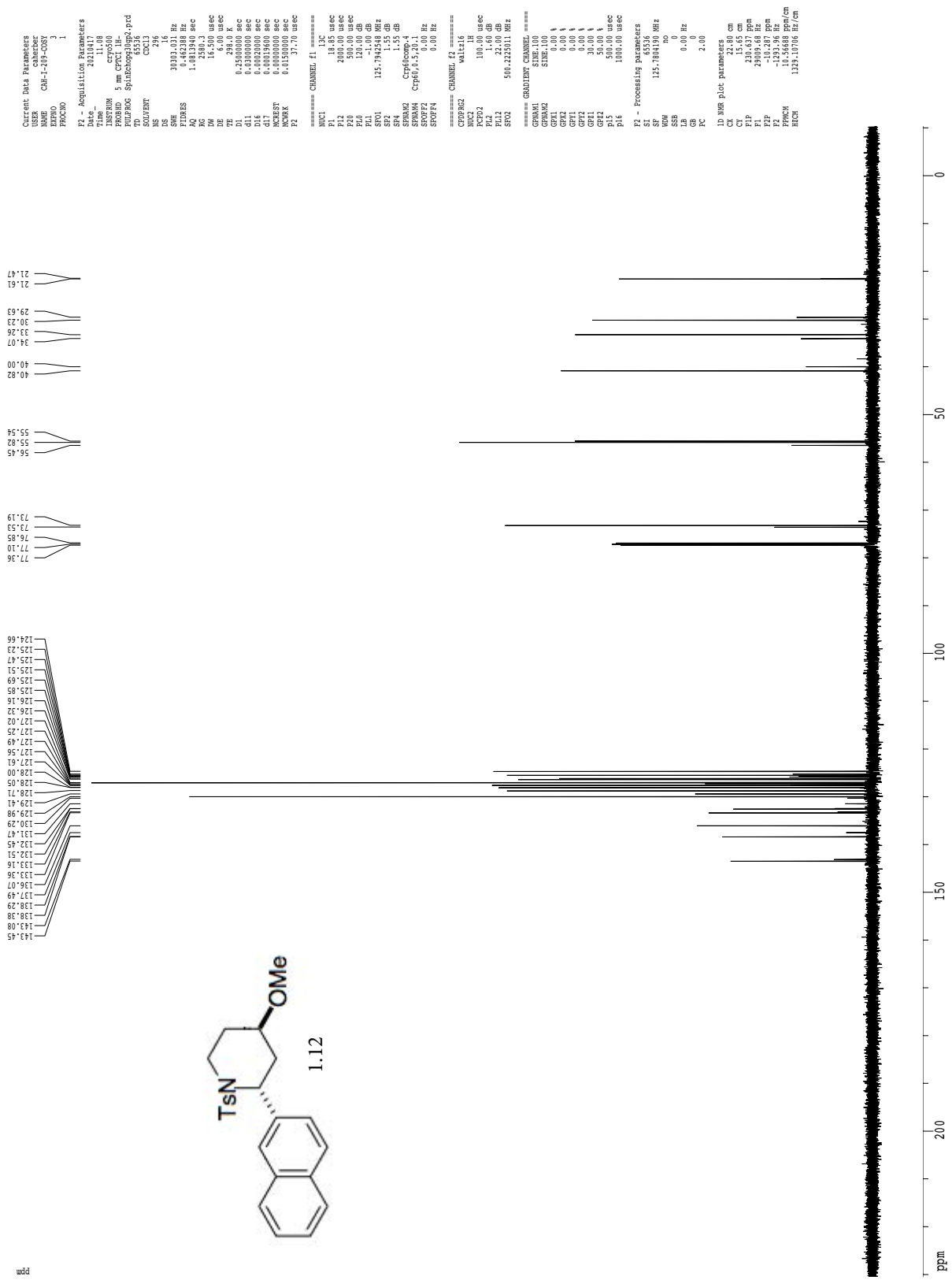


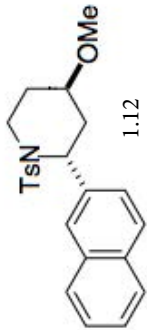
1H spectrum



Current Data Parameters  
 USER: chembebe  
 NAME: CNM-209  
 EXPNO: 1  
 PROCNO: 1  
 P2 - Acquisition Parameters  
 Date\_: 20210415  
 Time: 17.05  
 PROBHD: 5 mm QNP 1H/13  
 PULPROG: zgpg30  
 TD: 65536  
 SOLVENT: CDCl3  
 DS: 2  
 SFO: 400.1300267 MHz  
 SWH: 6410.258 Hz  
 FIDRES: 0.097813 Hz  
 AQ: 5.1118577 sec  
 RG: 327.5  
 DD: 78.000 usec  
 DE: 4.50 usec  
 TE: 298.2 K  
 T1: 0.1000000 sec  
 T2: 0.0000000 sec  
 T3: 0.0000000 sec  
 MCHRG1: 0.0000000 sec  
 MCHRG2: 0.0000000 sec  
 MCHRG3: 0.0000000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -1.68 dB  
 SFO1: 400.1326009 MHz  
 P2 - Processing parameters  
 SI: 65536  
 SF: 400.1300267 MHz  
 WDM: no  
 LB: 0.00 Hz  
 GB: 0  
 PC: 2.00  
 ID: NMR file parameters  
 CP: 22.80 cm  
 CY: 15.00 cm  
 P1P: 9.000 ppm  
 F1: 3600.17 Hz  
 F2: 430.96 Hz  
 F3: 430.96 Hz  
 PPM1: 0.44198 ppm/cm  
 HZCM: 176.84187 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling





1.12

gcosy60

```

Current Data Parameters
=====
USER      cahester
NAME      CAR-1-209-COSY
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
=====
Date_     20210417
Time      10:51
INSTRUM   cryso0
PROBHD    5
PULPROG   zgpg30
TD         2688
SOLVENT   CDCl3
NS         1
DS         4
SWH        8012.620 Hz
FIDRES    0.1278452 Hz
AQ         3.912510 Hz
RG         35.9
AQ         67.400 usec
DE         1.0000000 usec
TE         298.0 K
DO         0.00003300 sec
D1         1.00000000 sec
d13        0.00003300 sec
d16        0.00003300 sec
d19        0.00012600 sec
DNU

===== CHANNEL f1 =====
NUC1       1H
P1         9.75 usec
PC         1.00
SFO1       500.22035015 MHz

===== GRADIENT CHANNEL =====
GPRM1     SSGU0.100
GPRM2     SSGU0.100
GPX2      0.00
GPY2      0.00
GPZ2      0.00
GPT1      0.00
GPT2      0.00
GPT3      17.00
GPT4      17.00
PT6        1000.00 usec

F1 - Acquisition parameters
=====
NUC1       1H
P1         9.75 usec
PC         1.00
SFO1       500.22035 MHz
FIDRES    0.1278452 Hz
AQ         3.912510 Hz
RG         35.9
AQ         67.400 usec
DE         1.0000000 usec
TE         298.0 K
DO         0.00003300 sec
D1         1.00000000 sec
d13        0.00003300 sec
d16        0.00003300 sec
d19        0.00012600 sec
DNU

===== CHANNEL f2 =====
NUC2       13C
P2         10.00 usec
PC         1.00
SFO2       101.25321 MHz
FIDRES    0.1278452 Hz
AQ         3.912510 Hz
RG         35.9
AQ         67.400 usec
DE         1.0000000 usec
TE         298.0 K
DO         0.00003300 sec
D1         1.00000000 sec
d13        0.00003300 sec
d16        0.00003300 sec
d19        0.00012600 sec
DNU

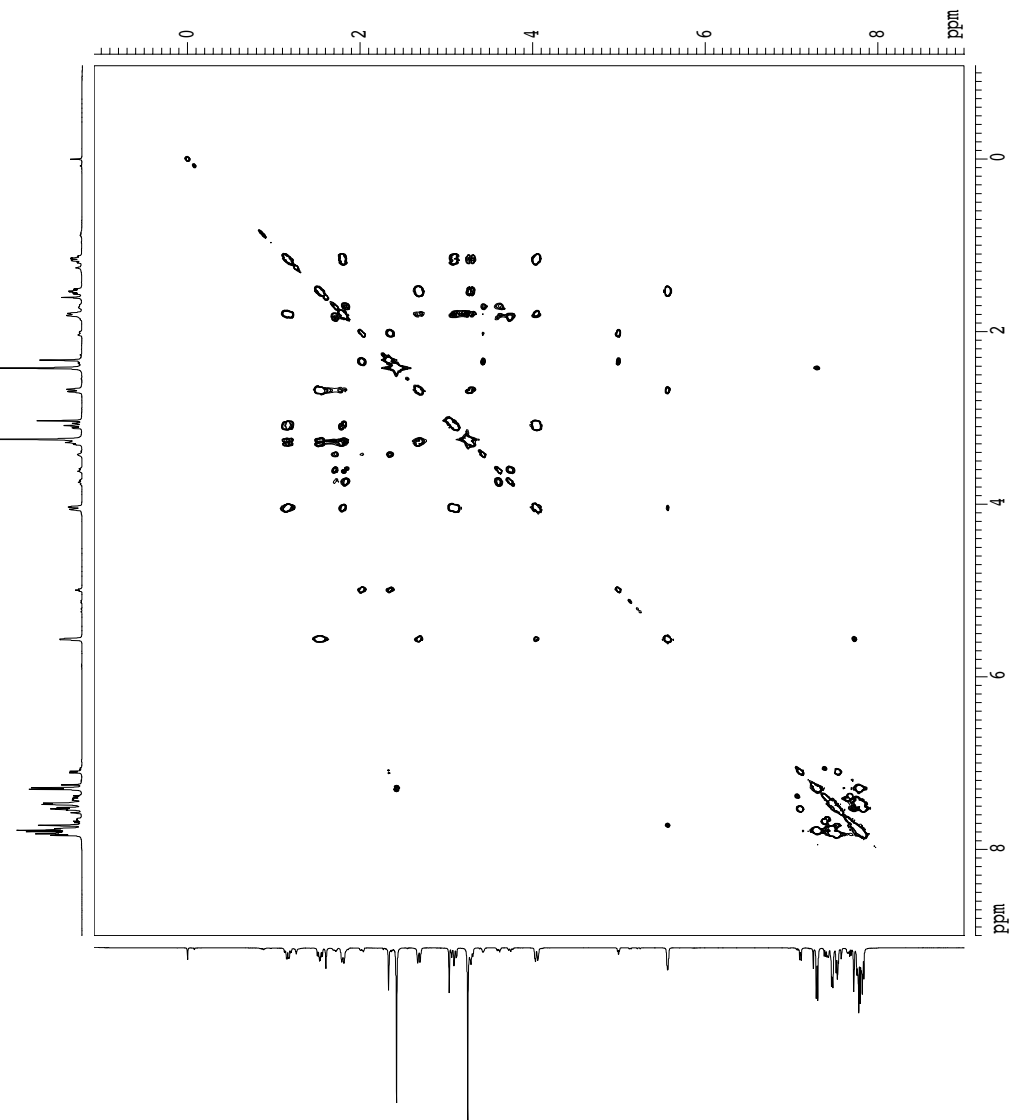
===== GRADIENT CHANNEL =====
GPRM1     SSGU0.100
GPRM2     SSGU0.100
GPX2      0.00
GPY2      0.00
GPZ2      0.00
GPT1      0.00
GPT2      0.00
GPT3      17.00
GPT4      17.00
PT6        1000.00 usec

F2 - Processing parameters
=====
SI         1024
SF         500.220352 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
=====
SI         1024
SF         500.220352 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
=====
CX2        15.00 cm
CX1        15.00 cm
F2PLO     5.000 ppm
F2PO      451.082 Hz
F2PHI     -541.03 Hz
F1PLO     9.000 ppm
F1PO      4501.98 Hz
F1PHI     -1.082 Hz
F2PRCM    0.67211 ppm/cm
F2PRCH    336.20662 Hz/cm
F1PRCM    0.67211 ppm/cm
F1PRCH    336.20662 Hz/cm

```



gnoe

ppm

7.8550  
7.7944  
7.7920  
7.60549  
7.60223  
7.58845  
7.58570

3.32462  
3.32022  
3.31597

2.76929  
2.76333  
2.75266  
2.73916  
2.73603

1.63414  
1.62304  
1.61089  
1.59986  
1.58888  
1.57991

Current Data Parameters  
USER cahleber  
NAME GNH-L-209-noe  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210417

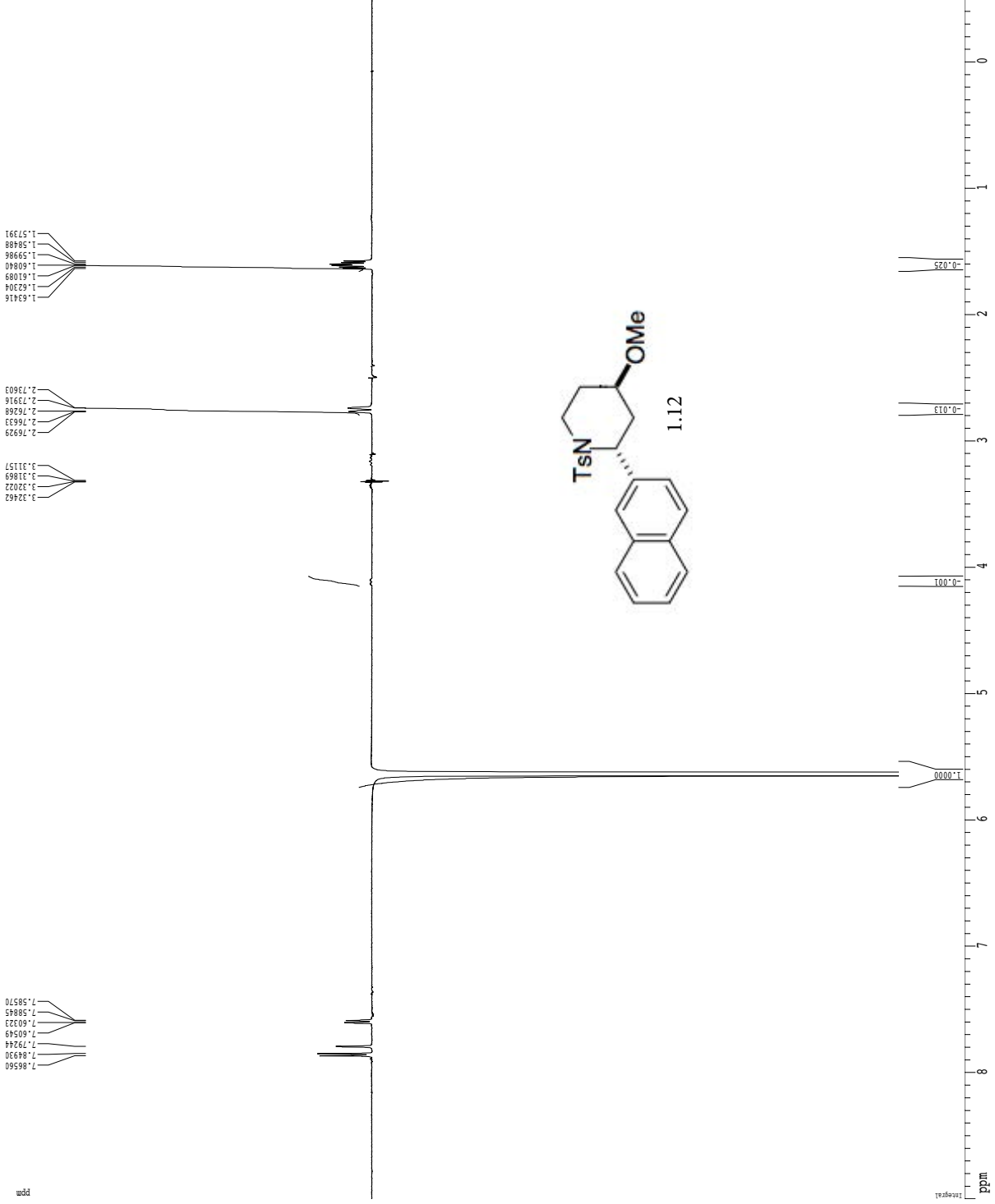
Time 14.16  
INSTRUM spect  
PROBHD 5 mm CPCT 1H  
PULPROG zgpg30c22.prd  
TD 65536  
SOLVENT CCL3  
NS 128  
DS 8  
SWH 8012.820 Hz  
FIDRES 0.122246 Hz  
AQ 4.0894966 sec  
RG 320  
DM 62.00 usec  
DE 6.00 usec  
TE 298.0 K  
DL 1.0000000 sec  
C1 0.8000000 sec  
D16 0.0020000 sec  
d21 0.33375451 sec  
d22 0.16398699 sec  
P2 19.50 usec

===== CHANNEL f1 =====  
NUC1 1H  
P1 9.75 usec  
P2 29.25 usec  
P3 29.00 usec  
P4 29.00 usec  
E29 40000.00 usec  
FL1 1.66 dB  
SFO1 500.2228398 MHz  
GAMMA 1H  
SFOB9 900.00 dB  
SFOFF9 0.00 Hz

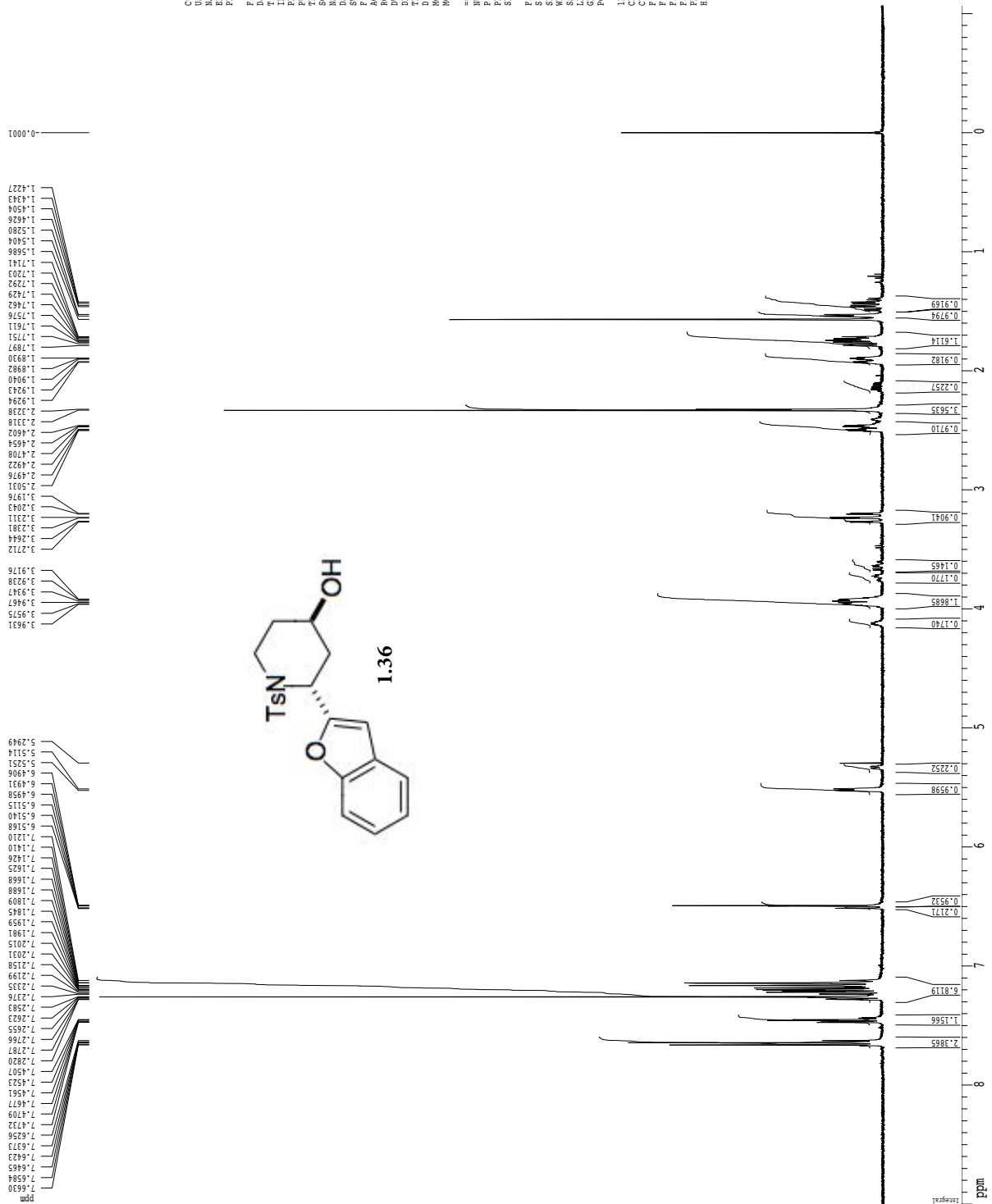
===== CHANNEL =====  
SFO10 500.1362730 MHz  
SFOB10 900.00 dB  
SFOFF10 0.00 Hz  
SFO11 500.1362730 MHz  
SFOB11 900.00 dB  
SFOFF11 0.00 Hz  
SFO12 500.1362730 MHz  
SFOB12 900.00 dB  
SFOFF12 0.00 Hz  
SFO13 500.1362730 MHz  
SFOB13 900.00 dB  
SFOFF13 0.00 Hz  
SFO14 500.1362730 MHz  
SFOB14 900.00 dB  
SFOFF14 0.00 Hz  
SFO15 500.1362730 MHz  
SFOB15 900.00 dB  
SFOFF15 0.00 Hz

F2 - Processing parameters  
SI 65536  
SF 500.2200000 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

ID NMR Plot parameters  
CX 22.88 cm  
CY 50.00 cm  
FID 16384.000 pps  
F1P 480.000 pps  
F2P -0.500 pps  
FZ -250.11 Hz  
PPHM 0.41667 ppm/cm  
HZCM 208.42500 Hz/cm



1H spectrum



Current Data Parameters  
 USER chembee  
 NAME CNR-1-215  
 EXPOV 2  
 PROCNO 1

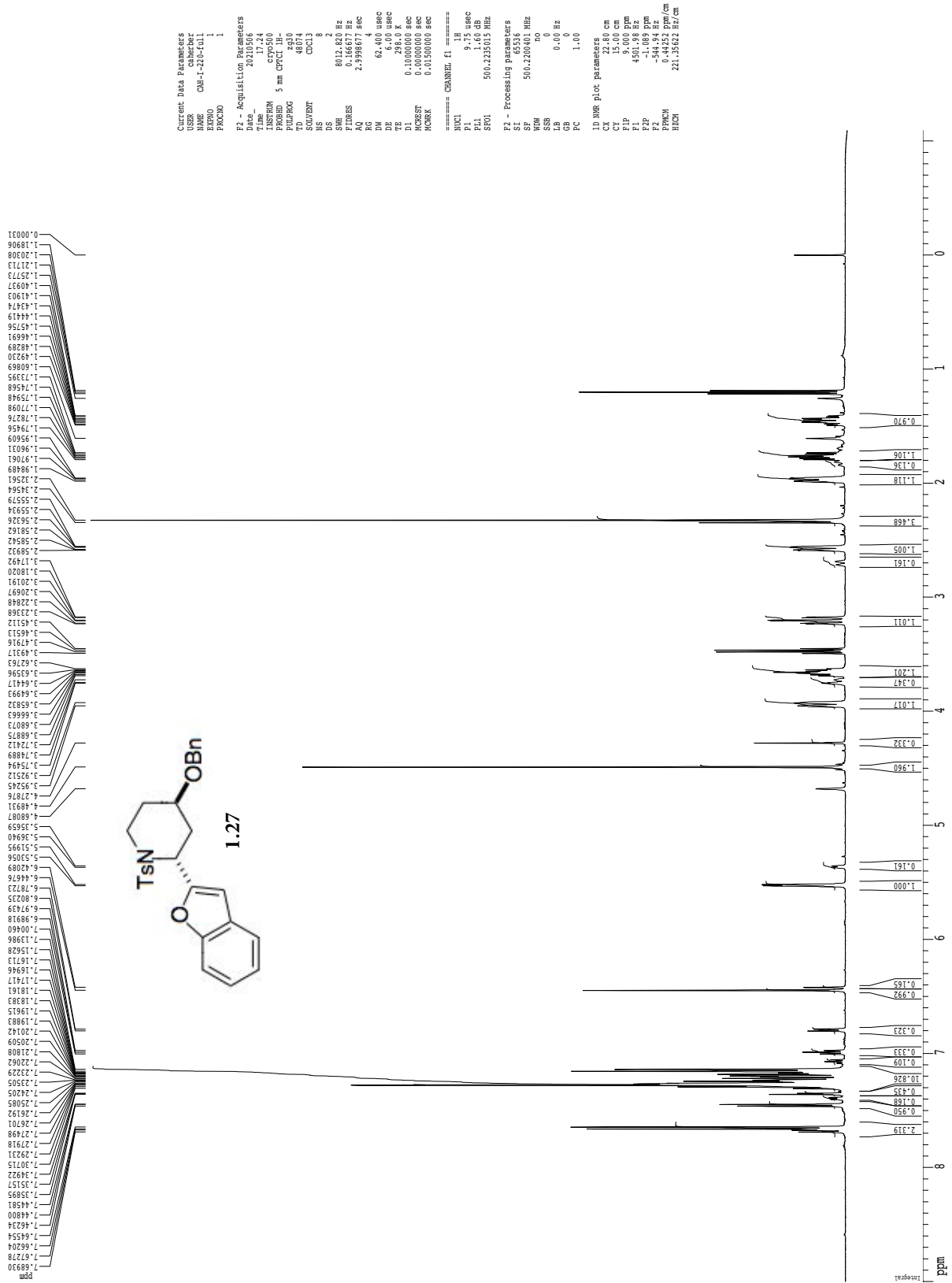
F2 - Acquisition Parameters  
 Date\_ 20210503  
 Time 13:54  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/1  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 DS 9  
 SFO 6410.258 Hz  
 FIDRES 0.09781 Hz  
 AQ 5.1118579 sec  
 RG 327.5  
 DD 78.006 usec  
 DE 4.50 usec  
 TE 298.2 K  
 Z 1  
 MCBEST 0.000000 sec  
 MCHRG 0.000000 sec  
 MCHRGK 0.0150000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -1.60 dB  
 SFO1 400.1328009 MHz

F2 - Processing Parameters  
 SI 65536  
 SF 400.1300223 MHz  
 NVM no  
 LSF 0.00 Hz  
 GB 0  
 PC 2.00

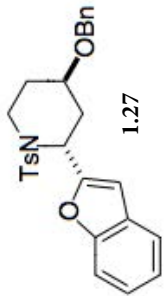
1D NMR P1/QF Parameters  
 CQ 22.80 cm  
 CY 15.00 cm  
 FIP 9.000 ppm  
 F1 3600.117 Hz  
 F2 426.568 Hz  
 F3 444.449 ppm/cm  
 HZCM 176.65485 Hz/cm

1H spectrum

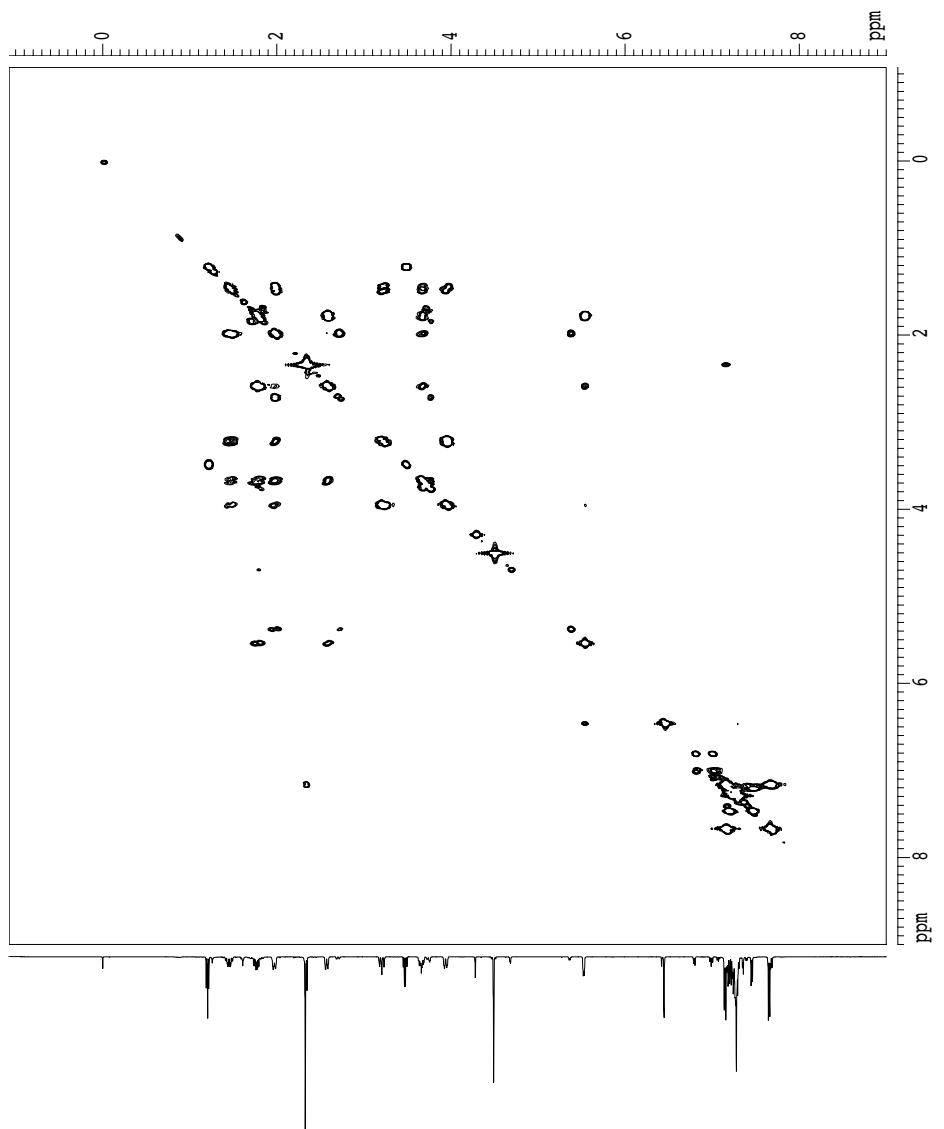








gc0sy60



```

Current Data Parameters
=====
USER          NAME          CNB-L-27b-f01
EXPNO         2
PROCNO        1

F2 - Acquisition Parameters
=====
Date_         20100606
Time          17.29
INSTRUM       cryo500
PROBHD        5 mm CPTCI IH-
PULPROG       zgpg30
AQ            0.0000300 sec
RG            62.400 usec
DE            6.00 usec
TE            298.0 K
AQ            0.0000300 sec
RG            62.400 usec
DE            6.00 usec
D13           0.0000300 sec
D16           0.0002000 sec
IN0           0.00012480 sec

===== CHANNEL f1 =====
NUC1          13
P1            9.75 usec
PL1           1.60 dB
SFO1          500.220325 MHz

===== GSDPTPR CHANNEL =====
GNAME         SWSQ130
GPRMG         SWSQ130.00
GPA1          0.00 %
GPA2          0.00 %
GPA3          0.00 %
GPA4          0.00 %
GPA5          17.00 %
GPA6          17.00 %
P16           1000.00 usec

F1 - Acquisition parameters
=====
NUC1          13
P1            9.75 usec
PL1           1.60 dB
SFO1          500.220325 MHz
FIDRES        29.787437 Hz
SN            16.018 ppm
FWD0DE        QF

F2 - Processing parameters
=====
SI            1024
SF            500.220325 MHz
WDW           SINE
SSB           0
GB            0
PC            1.00

F1 - Processing parameters
=====
SI            1024
SF            500.220325 MHz
WDW           SINE
SSB           0
GB            0
PC            1.00

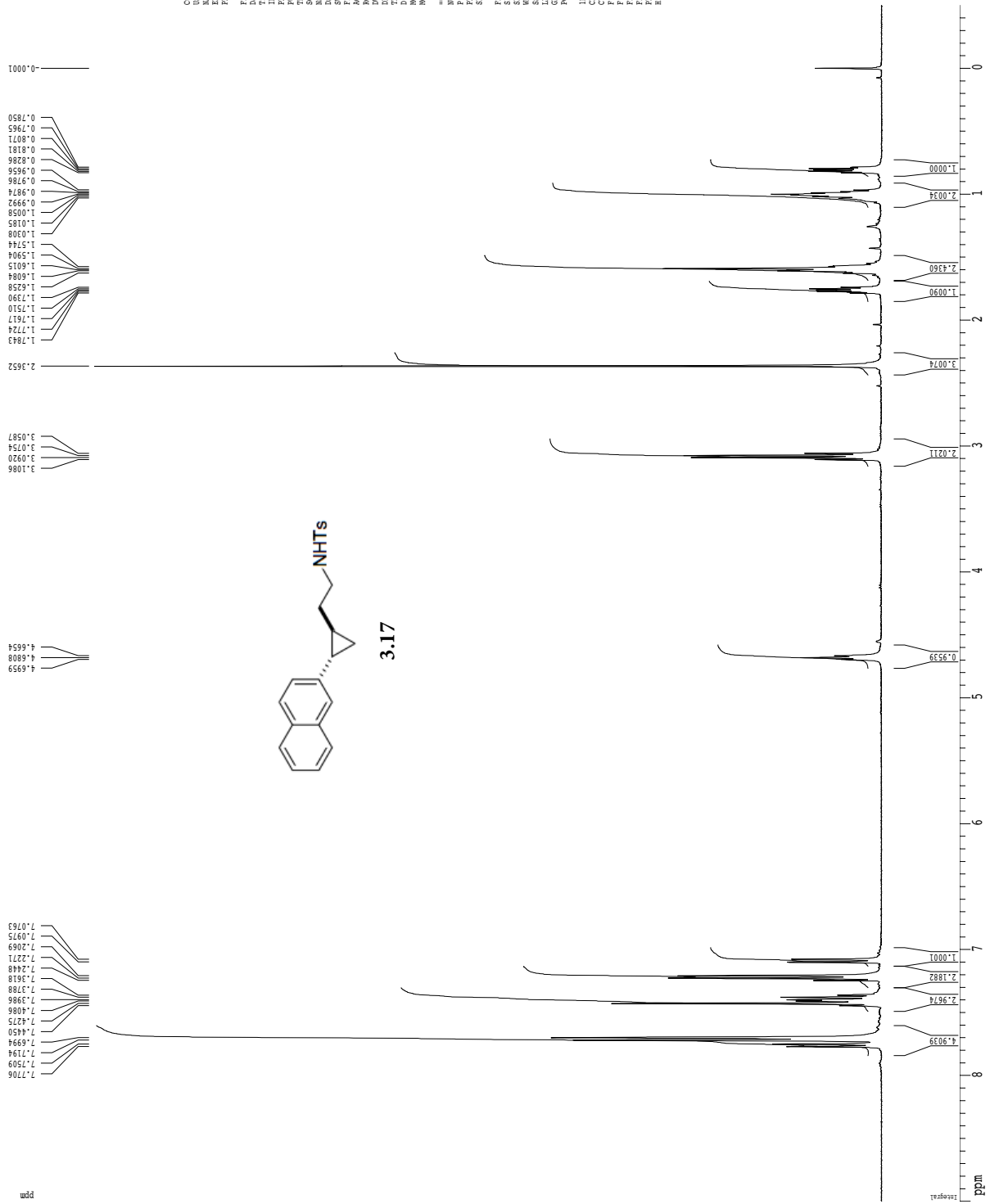
2D NMR plot parameters
=====
CX2           15.00 cm
F2PLO         9.000 ppm
F2P20         49.000 ppm
F2P40         21.075 ppm
F2P60         -537.73 Hz
F2P80         9.000 ppm
F2P100        4501.98 Hz
F2P120        -537.73 ppm
F2P140        0.67167 ppm/cm
F2P160        335.98087 Hz/cm
F2P180        0.67167 ppm/cm
F2P200        335.98087 Hz/cm
  
```



APPENDIX A.2

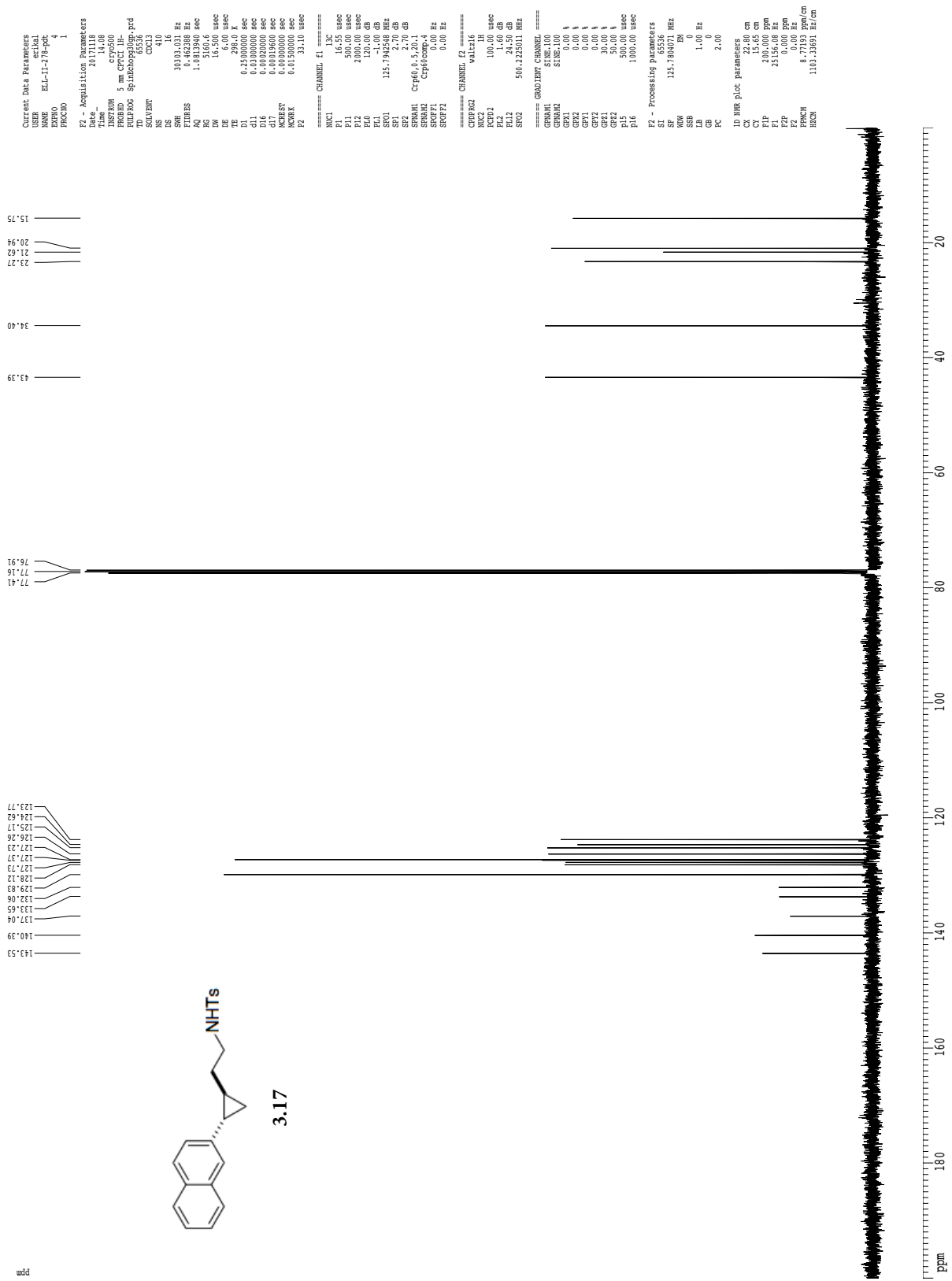
$^1\text{H}$ ,  $^{13}\text{C}$ , COSY, NOE NMR SPECTRA, SFC TRACES FOR CHAPTER 3

**<sup>1</sup>H spectrum**



Current Data Parameters  
 USER uc164  
 NAME ELI-111-2016-isolated  
 EXPNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20160819  
 Time 19:42  
 INSTRUM dx400  
 PROBRD 5 mm QNP H<sup>1</sup>/F<sup>2</sup>  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT OCCLP  
 DS 2  
 SWH 6410.256 Hz  
 FIDRES 0.188673 Hz  
 AQ 4.1913 sec  
 RG 161.3 sec  
 SN 78.000 usac  
 LB 0.300 Hz  
 TE 296.2 K  
 D1 0.10000000 sec  
 ACQRES 0.30000000 sec  
 AORIG 0.10000000 sec  
 ===== CHANNEL f1 =====  
 NU1 1  
 PR 1  
 P1 12.00 usec  
 PL -1.10 dB  
 SFO1 400.126009 MHz  
 F2 - Processing parameters  
 SI 65536  
 SF 400.126009 MHz  
 SW 65536  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 2.00  
 ID NMR plot parameters  
 CT 15.00 cm  
 P1 9.000 ppm  
 P2 30.000 ppm  
 F2 200.000 ppm  
 F2 -200.06 Hz  
 PRON 0.41167 ppm/cm  
 HSCN 160.78000 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters  
 USER: etikal  
 NAME: ELL-II-278-pdt  
 EXPNO: 3  
 PROCNO: 1

F2 - Acquisition Parameters

Date\_ 20101016  
 Time 11:31  
 INSTRUM: cryo500  
 PROBHD: 5 mm CPTCI 1H-  
 PULPROG: cosygp60.prd  
 TD: 2048  
 SOLVENT: CDCl3  
 NS: 2  
 DS: 16  
 SFO1: 4731.618 Hz  
 FIDRES: 7.311938 Hz  
 AQ: 0.2163188 sec  
 RG: 203.2  
 DW: 105.600 usec  
 DE: 6.00 usec  
 TE: 298.0 K  
 d0: 0.00000300 sec  
 D1: 1.00000000 sec  
 D11: 0.00000000 sec  
 D16: 0.00020000 sec  
 INU: 0.00021120 sec

==== CHANNEL f1 =====  
 NUC1: 1H  
 P1: 7.50 usec  
 PL1: 1.60 dB  
 SFO1: 500.2221599 MHz

==== GRADIENT CHANNEL =====

GPRAM1: size,100  
 GPC1: 0.00 %  
 GPC2: 0.00 %  
 GPC3: 0.00 %  
 GPC4: 0.00 %  
 GPC5: 17.00 %  
 GPC6: 17.00 %  
 GPC7: 100.00 usec  
 P16

F1 - Acquisition Parameters

NUC0: 1  
 TD: 512  
 SFO1: 500.2222 MHz  
 FIDRES: 9.247751 Hz  
 SW: 9.485 ppm  
 FROUOE: undefiined

F2 - Processing Parameters

SI: 1024  
 SF: 500.220340 MHz  
 WDW: SINE  
 SSB: 0  
 LB: 0.00 Hz  
 GB: 0  
 PC: 1.00

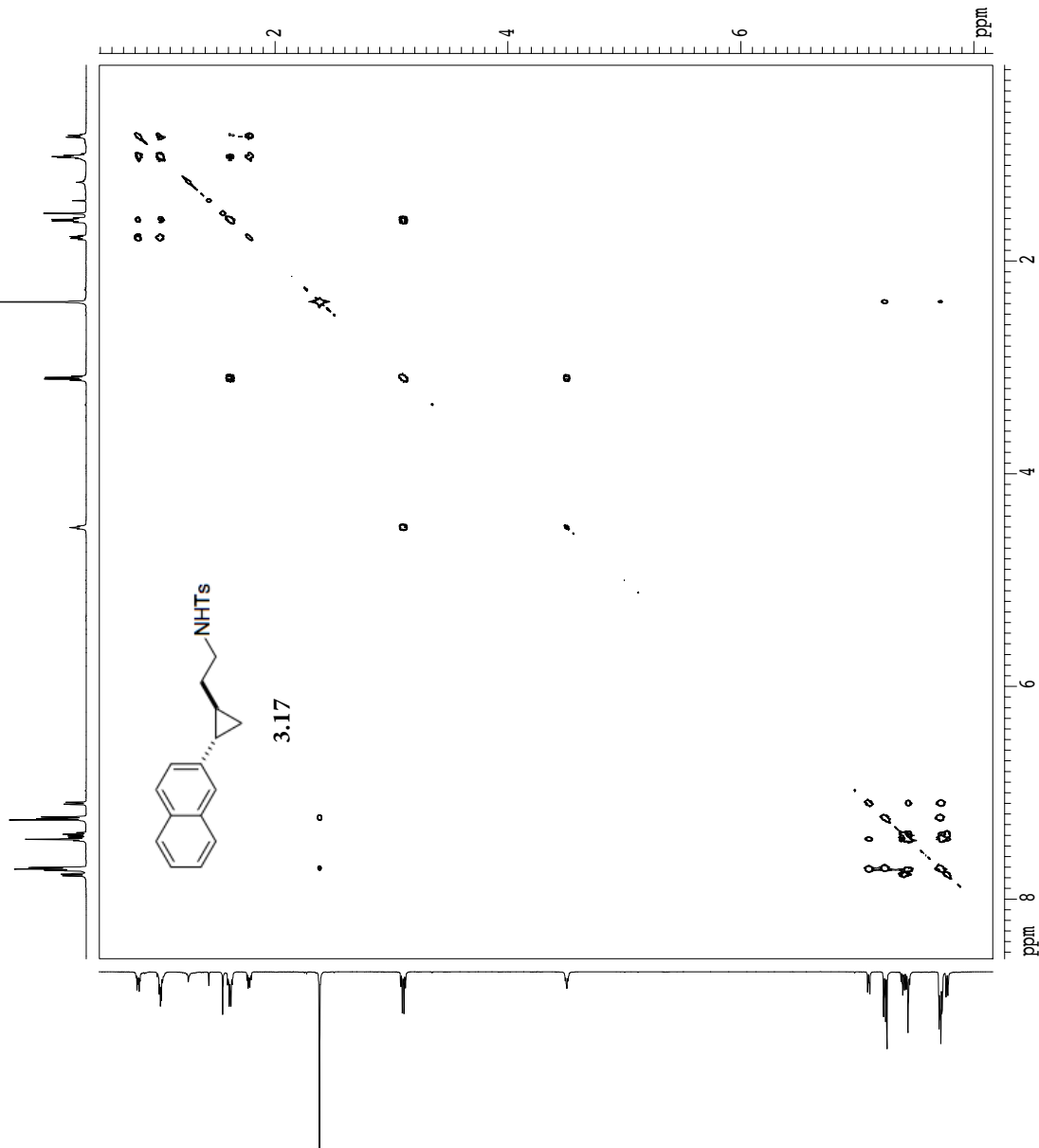
F1 - Processing Parameters

SI: 1024  
 MC2: OF  
 SF: 500.220340 MHz  
 WDW: SINE  
 SSB: 0  
 LB: 0.00 Hz  
 GB: 0

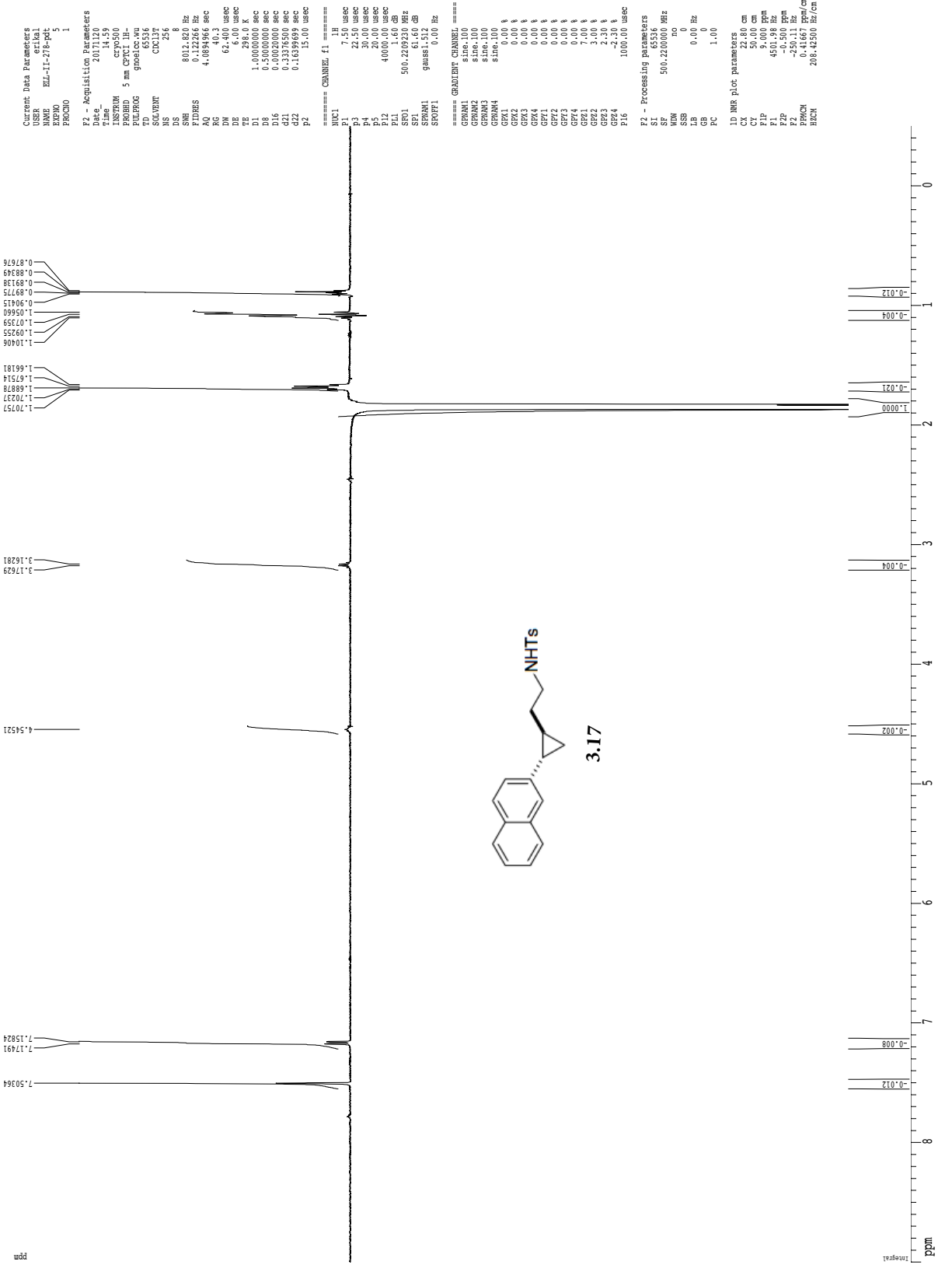
2D NMR plot parameters

CX2: 15.00 cm  
 CX1: 15.00 cm  
 F2PLO: 8.561 ppm  
 FZLO: 4282.25 Hz  
 F2PHI: 0.158 ppm  
 F2H1: 7.115 Hz  
 F2H2: 7.115 Hz  
 F1LO: 4083.48 Hz  
 F1PHI: 0.491 ppm  
 F1H1: 245.61 Hz  
 F2FPMCM: 0.56017 ppm/cm  
 F2HPCM: 280.20686 Hz/cm  
 F1FPMCM: 0.51148 ppm/cm  
 F1HPCM: 255.85446 Hz/cm

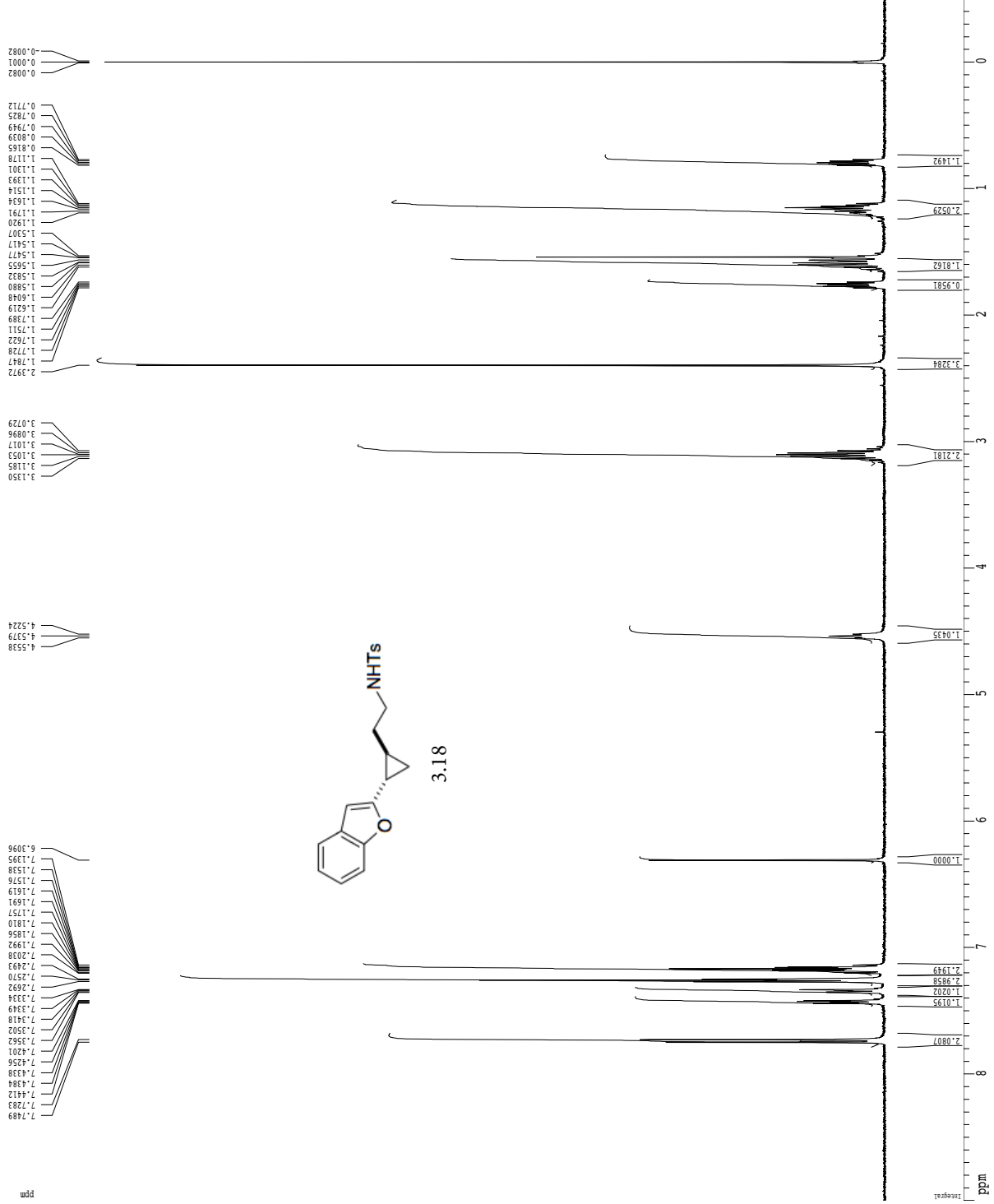
gc05y60



gnoe



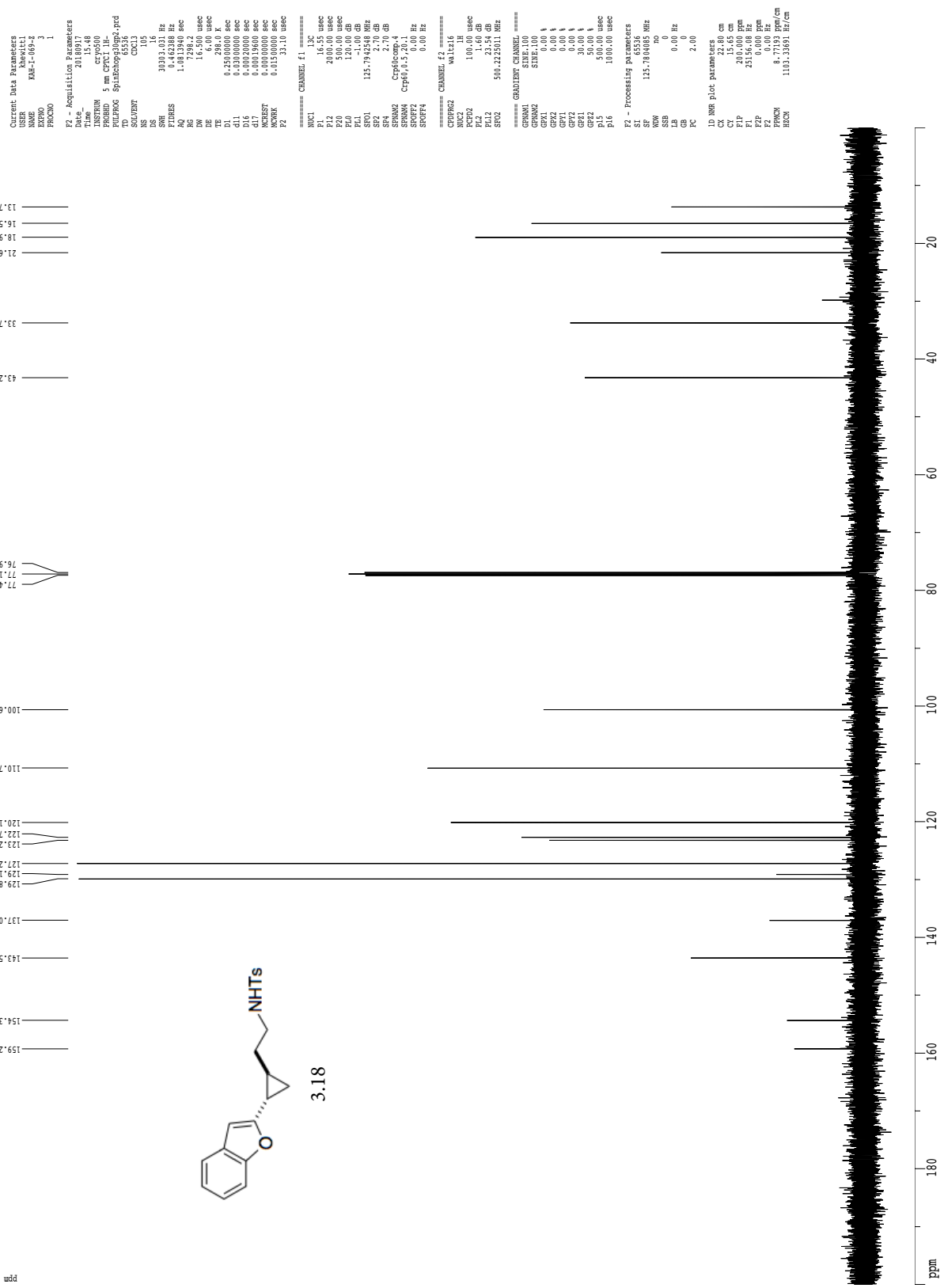
1H spectrum

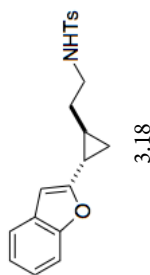


Current Data Parameters  
 USER KMH-IT-111 check  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20190725  
 Time 12.04  
 Operator  
 PULPROG zgpg30  
 PROCNO 3846  
 TD 32768  
 SOLVENT CDCl3  
 NS 2  
 DS 0  
 SWH 6410.256 Hz  
 FIDRES 0.166672 Hz  
 AQ 2.939239 sec  
 RG 327.5  
 DW 78.000 usec  
 DE 4.50 usec  
 TE 298.2 K  
 D1 0.100000 sec  
 MCHRES 0.100000 sec  
 MCNTR 0.0550000 sec  
 ===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.132809 MHz  
 F2 - Processing Parameters  
 SI 65536  
 SF 400.130022 MHz  
 SD 0  
 ASB 0 Hz  
 GB 0  
 PC 2.00  
 ID\_NMR File Parameters  
 CX 22.80 cm  
 CY 15.40 cm  
 F1 9.000 ppm  
 F2 500.137 Hz  
 F3 0.000 ppm  
 F4 -200.90 Hz  
 PPMCM 0.41667 ppm/cm  
 RECH 166.72086 Hz/cm

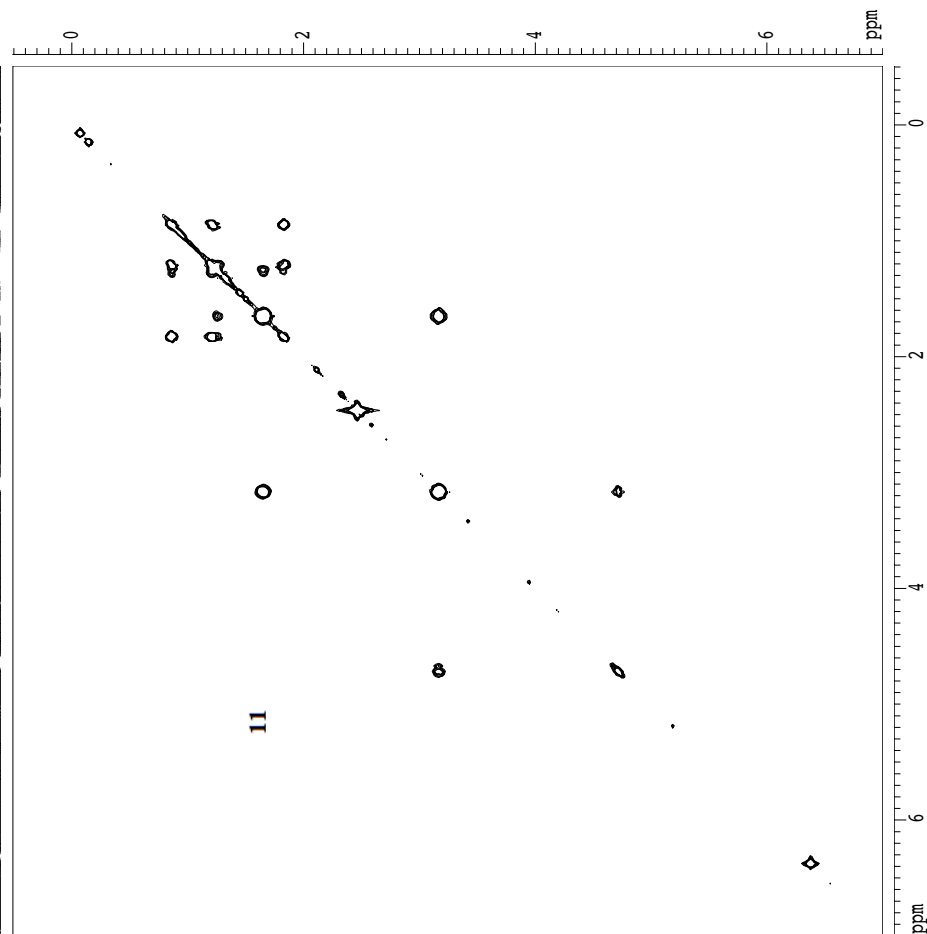


Z-restored spin-echo 13C spectrum with 1H decoupling





gcosy60



```

Current Data Parameters
=====
USRR      Khesiti1
NAME      KNH-1-062-2
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
=====
Date_     20180417
Time      15:35
PULPROG   zgpg30
PROCPRG   cpgm
PULPROG   5 mm Cgpg30
PILARPG   cosyg60_frd
TD         2448
SOLVENT   CDCl3
NS         1
DS         1
SWH        8032.500 Hz
FIDRES     3.912510 Hz
AQ         0.127852 sec
RG         256
INSTRUM   spect
PROBHD    5 mm QNP1H
TE         298.0 K
D0         0.0000300 sec
d1         1.0000000 sec
d13        0.0000300 sec
d16        0.0000000 sec
d18        0.0000000 sec
d20        0.0001280 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
PL1        0.00 dB
SFO1       500.225015 MHz

===== GRAB F1 =====
GPRM1     size=100
GRAB1     size=100
GPRM2     size=100
GPRM3     size=100
GPRM4     size=100
GPRM5     size=100
GPRM6     size=100
GPRM7     size=100
GPRM8     size=100
GPRM9     size=100
GPRM10    size=100
GPRM11    size=100
GPRM12    size=100
GPRM13    size=100
GPRM14    size=100
GPRM15    size=100
GPRM16    size=100
GPRM17    size=100
GPRM18    size=100
GPRM19    size=100
GPRM20    size=100
GPRM21    size=100
GPRM22    size=100
GPRM23    size=100
GPRM24    size=100
GPRM25    size=100
GPRM26    size=100
GPRM27    size=100
GPRM28    size=100
GPRM29    size=100
GPRM30    size=100

===== CHANNEL f2 =====
NUC2       13C
P2         12.00 usec
PL2        0.00 dB
SFO2       100.628150 MHz

===== GRAB F2 =====
GPRM1     size=100
GRAB1     size=100
GPRM2     size=100
GPRM3     size=100
GPRM4     size=100
GPRM5     size=100
GPRM6     size=100
GPRM7     size=100
GPRM8     size=100
GPRM9     size=100
GPRM10    size=100
GPRM11    size=100
GPRM12    size=100
GPRM13    size=100
GPRM14    size=100
GPRM15    size=100
GPRM16    size=100
GPRM17    size=100
GPRM18    size=100
GPRM19    size=100
GPRM20    size=100
GPRM21    size=100
GPRM22    size=100
GPRM23    size=100
GPRM24    size=100
GPRM25    size=100
GPRM26    size=100
GPRM27    size=100
GPRM28    size=100
GPRM29    size=100
GPRM30    size=100

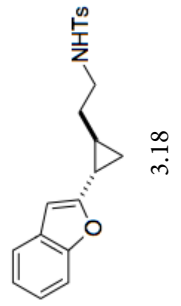
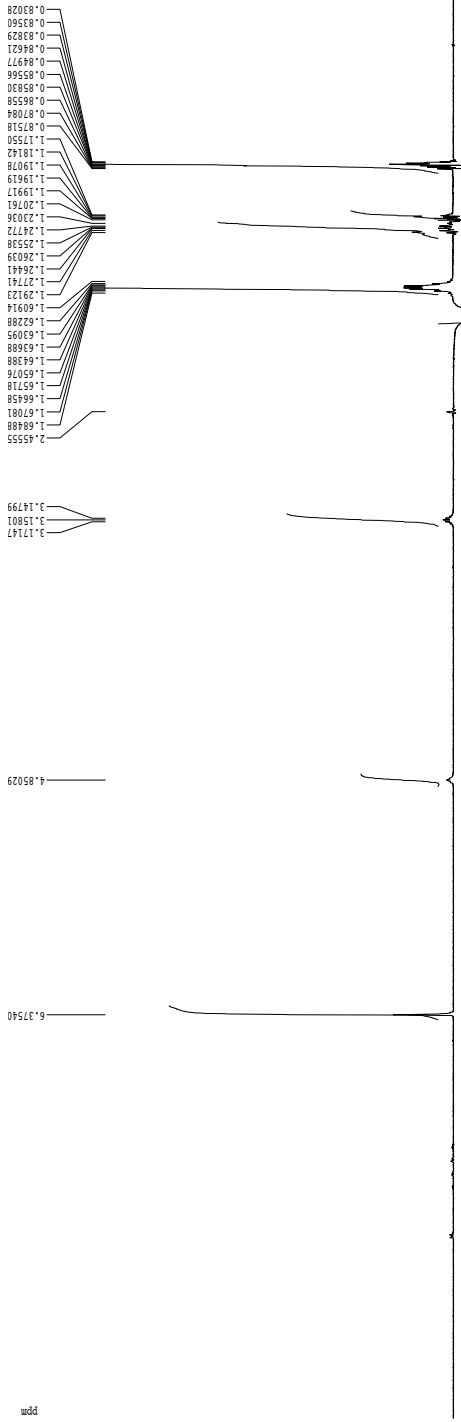
===== Processing parameters =====
SI         1634
SF         500.2200000 MHz
WDW        EM
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

===== Processing parameters =====
SI         1024
SF         500.2200000 MHz
WDW        EM
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot Parameters
=====
CX2        15.000 cm
CX1        15.000 cm
F2FLO      7.000 ppm
F2FID      38.000 ppm
F2AQ       -10.500 ppm
F2F1       -254.47 Hz
F1FLO      7.000 ppm
F1FID      3801.54 Hz
F1F1       -10.500 ppm
F1F2       -10.500 ppm
F2F2MCH    0.50658 ppm/cm
F2F1MCH    250.40663 Hz/cm
F1F2MCH    0.50658 ppm/cm
F1F1MCH    250.40663 Hz/cm
  
```

gnoe

ppm



Current Data Parameters  
USER khewitt1  
NAME KAH-1-069-2  
EXPNO 5  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20180917  
Time 20.12  
PROBHD 5 mm CPCT 1H  
PULPROG gnoe1cc22.prd  
TD 65536  
SOLVENT CDCl3  
DS 8  
SS 8  
SMB 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0899966 sec  
RG 327.500  
DE 62.000 usec  
TE 288.0 K  
D1 1.0000000 sec  
D2 0.0000000 sec  
D3 0.0000000 sec  
dZ1 0.33375000 sec  
dZ2 0.16398699 sec  
P2 15.00 usec

==== CHANNEL f1 =====  
NUC1 1H  
P1 7.50 usec  
P2 22.50 usec  
P3 20.00 usec  
P4 20.00 usec  
P5 20.00 usec  
PZ9 40000.00 usec  
PL1 1.60 dB  
SFO1 500.2200124 MHz  
SFO2 500.0000000 MHz  
SFO3 500.0000000 MHz  
SFO4 500.0000000 MHz  
SFO5 500.0000000 MHz  
SFO6 500.0000000 MHz  
SFO7 500.0000000 MHz  
SFO8 500.0000000 MHz  
SFO9 500.0000000 MHz  
SFO10 500.0000000 MHz  
SFO11 500.0000000 MHz  
SFO12 500.0000000 MHz  
SFO13 500.0000000 MHz  
SFO14 500.0000000 MHz  
SFO15 500.0000000 MHz  
SFO16 500.0000000 MHz  
SFO17 500.0000000 MHz  
SFO18 500.0000000 MHz  
SFO19 500.0000000 MHz  
SFO20 500.0000000 MHz  
SFO21 500.0000000 MHz  
SFO22 500.0000000 MHz  
SFO23 500.0000000 MHz  
SFO24 500.0000000 MHz  
SFO25 500.0000000 MHz  
SFO26 500.0000000 MHz  
SFO27 500.0000000 MHz  
SFO28 500.0000000 MHz  
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SFO33 500.0000000 MHz  
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SFO35 500.0000000 MHz  
SFO36 500.0000000 MHz  
SFO37 500.0000000 MHz  
SFO38 500.0000000 MHz  
SFO39 500.0000000 MHz  
SFO40 500.0000000 MHz  
SFO41 500.0000000 MHz  
SFO42 500.0000000 MHz  
SFO43 500.0000000 MHz  
SFO44 500.0000000 MHz  
SFO45 500.0000000 MHz  
SFO46 500.0000000 MHz  
SFO47 500.0000000 MHz  
SFO48 500.0000000 MHz  
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SFO76 500.0000000 MHz  
SFO77 500.0000000 MHz  
SFO78 500.0000000 MHz  
SFO79 500.0000000 MHz  
SFO80 500.0000000 MHz  
SFO81 500.0000000 MHz  
SFO82 500.0000000 MHz  
SFO83 500.0000000 MHz  
SFO84 500.0000000 MHz  
SFO85 500.0000000 MHz  
SFO86 500.0000000 MHz  
SFO87 500.0000000 MHz  
SFO88 500.0000000 MHz  
SFO89 500.0000000 MHz  
SFO90 500.0000000 MHz  
SFO91 500.0000000 MHz  
SFO92 500.0000000 MHz  
SFO93 500.0000000 MHz  
SFO94 500.0000000 MHz  
SFO95 500.0000000 MHz  
SFO96 500.0000000 MHz  
SFO97 500.0000000 MHz  
SFO98 500.0000000 MHz  
SFO99 500.0000000 MHz  
SFO100 500.0000000 MHz

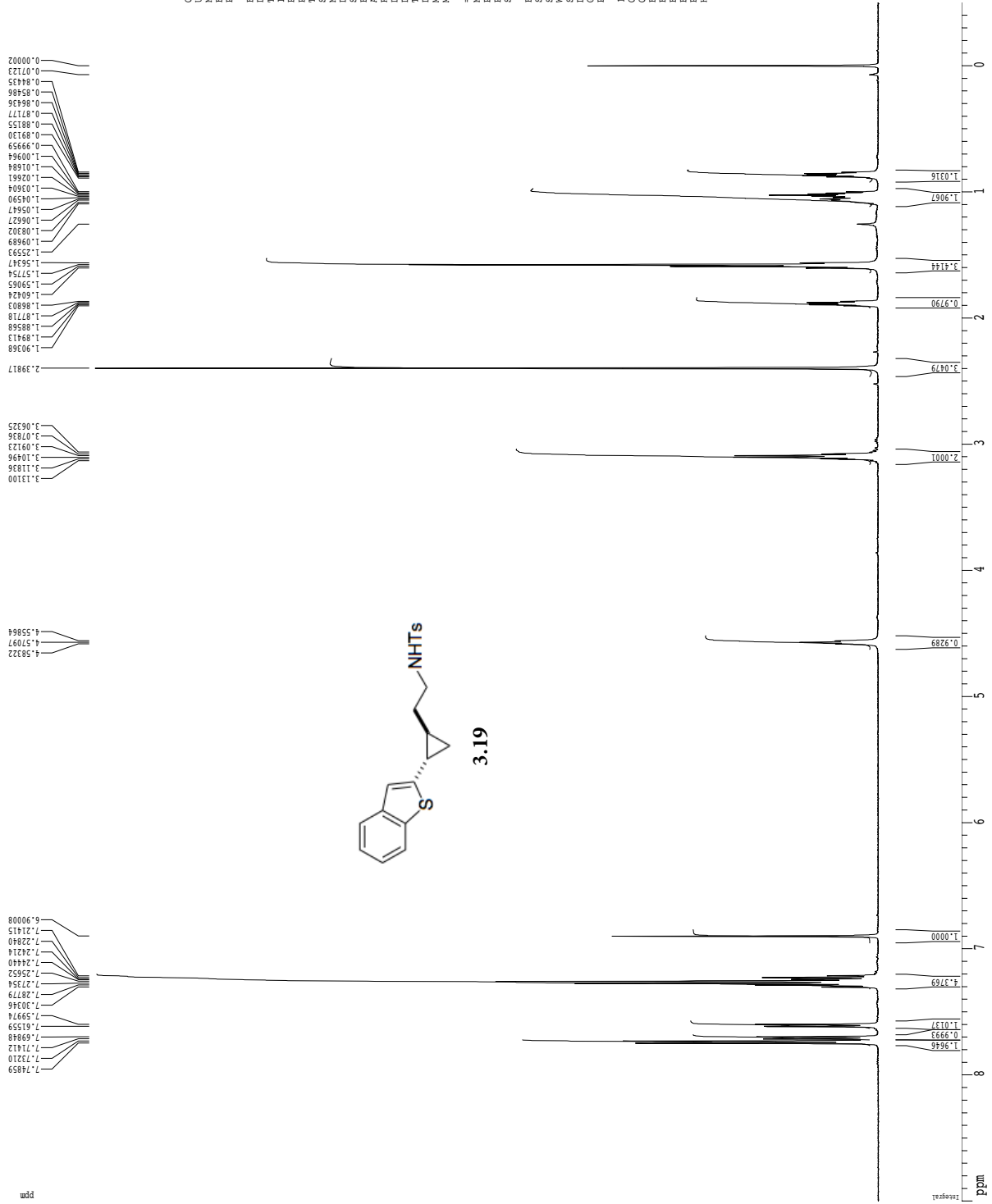
==== CHANNEL f2 =====  
NUC2 13C  
P1 7.50 usec  
P2 22.50 usec  
P3 20.00 usec  
P4 20.00 usec  
P5 20.00 usec  
PZ9 40000.00 usec  
PL1 1.60 dB  
SFO1 500.2200124 MHz  
SFO2 500.0000000 MHz  
SFO3 500.0000000 MHz  
SFO4 500.0000000 MHz  
SFO5 500.0000000 MHz  
SFO6 500.0000000 MHz  
SFO7 500.0000000 MHz  
SFO8 500.0000000 MHz  
SFO9 500.0000000 MHz  
SFO10 500.0000000 MHz  
SFO11 500.0000000 MHz  
SFO12 500.0000000 MHz  
SFO13 500.0000000 MHz  
SFO14 500.0000000 MHz  
SFO15 500.0000000 MHz  
SFO16 500.0000000 MHz  
SFO17 500.0000000 MHz  
SFO18 500.0000000 MHz  
SFO19 500.0000000 MHz  
SFO20 500.0000000 MHz  
SFO21 500.0000000 MHz  
SFO22 500.0000000 MHz  
SFO23 500.0000000 MHz  
SFO24 500.0000000 MHz  
SFO25 500.0000000 MHz  
SFO26 500.0000000 MHz  
SFO27 500.0000000 MHz  
SFO28 500.0000000 MHz  
SFO29 500.0000000 MHz  
SFO30 500.0000000 MHz  
SFO31 500.0000000 MHz  
SFO32 500.0000000 MHz  
SFO33 500.0000000 MHz  
SFO34 500.0000000 MHz  
SFO35 500.0000000 MHz  
SFO36 500.0000000 MHz  
SFO37 500.0000000 MHz  
SFO38 500.0000000 MHz  
SFO39 500.0000000 MHz  
SFO40 500.0000000 MHz  
SFO41 500.0000000 MHz  
SFO42 500.0000000 MHz  
SFO43 500.0000000 MHz  
SFO44 500.0000000 MHz  
SFO45 500.0000000 MHz  
SFO46 500.0000000 MHz  
SFO47 500.0000000 MHz  
SFO48 500.0000000 MHz  
SFO49 500.0000000 MHz  
SFO50 500.0000000 MHz  
SFO51 500.0000000 MHz  
SFO52 500.0000000 MHz  
SFO53 500.0000000 MHz  
SFO54 500.0000000 MHz  
SFO55 500.0000000 MHz  
SFO56 500.0000000 MHz  
SFO57 500.0000000 MHz  
SFO58 500.0000000 MHz  
SFO59 500.0000000 MHz  
SFO60 500.0000000 MHz  
SFO61 500.0000000 MHz  
SFO62 500.0000000 MHz  
SFO63 500.0000000 MHz  
SFO64 500.0000000 MHz  
SFO65 500.0000000 MHz  
SFO66 500.0000000 MHz  
SFO67 500.0000000 MHz  
SFO68 500.0000000 MHz  
SFO69 500.0000000 MHz  
SFO70 500.0000000 MHz  
SFO71 500.0000000 MHz  
SFO72 500.0000000 MHz  
SFO73 500.0000000 MHz  
SFO74 500.0000000 MHz  
SFO75 500.0000000 MHz  
SFO76 500.0000000 MHz  
SFO77 500.0000000 MHz  
SFO78 500.0000000 MHz  
SFO79 500.0000000 MHz  
SFO80 500.0000000 MHz  
SFO81 500.0000000 MHz  
SFO82 500.0000000 MHz  
SFO83 500.0000000 MHz  
SFO84 500.0000000 MHz  
SFO85 500.0000000 MHz  
SFO86 500.0000000 MHz  
SFO87 500.0000000 MHz  
SFO88 500.0000000 MHz  
SFO89 500.0000000 MHz  
SFO90 500.0000000 MHz  
SFO91 500.0000000 MHz  
SFO92 500.0000000 MHz  
SFO93 500.0000000 MHz  
SFO94 500.0000000 MHz  
SFO95 500.0000000 MHz  
SFO96 500.0000000 MHz  
SFO97 500.0000000 MHz  
SFO98 500.0000000 MHz  
SFO99 500.0000000 MHz  
SFO100 500.0000000 MHz

F2 - Processing parameters  
SI 65536  
SF 500.2200000 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

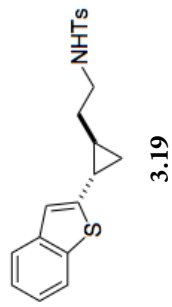
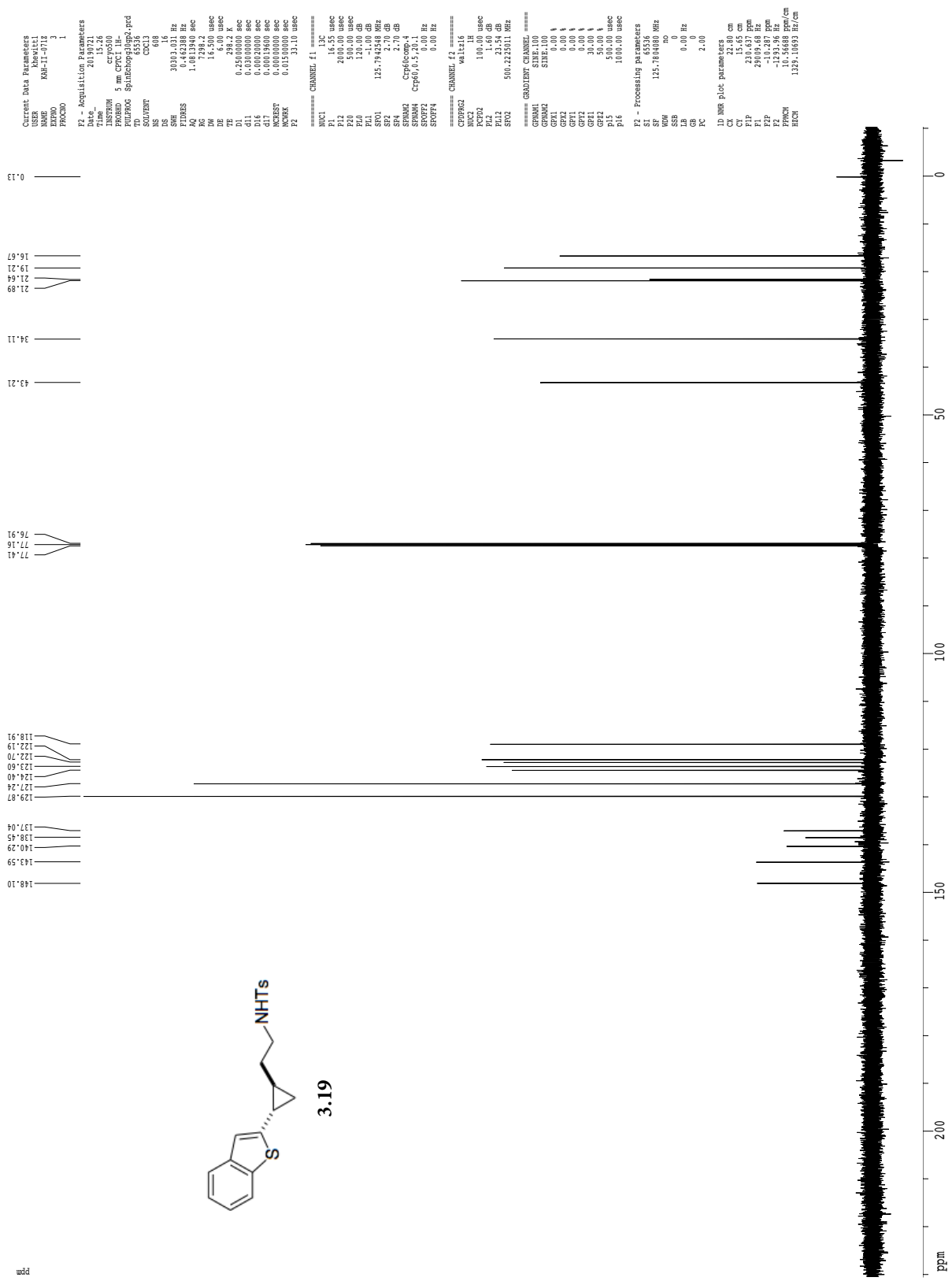
ID NMR plot parameters  
CX 22.80 cm  
CY 50.00 cm  
CZ 50.00 cm  
F1 450.00 ppm  
F2 -0.500 ppm  
F3 -250.11 Hz  
PRGCM 0.41667 ppm/cm  
HCN 208.42500 Hz/cm



1H spectrum



Z-restored spin-echo 13C spectrum with 1H decoupling



```

Current Data Parameters
USER      khwitll
NAME      KM-17-0712
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Time      411.15.26
INSTRUM   spect
PROBHD    5 mm QNP1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         64
DS         16
SWH        30103.031 Hz
FIDRES     0.462288 Hz
AQ          1.729812 sec
RG          327.500
DE          16.500 usec
TE          300.2 K
D1          0.25000000 sec
d11         0.03000000 sec
d12         0.00000000 sec
d13         0.00000000 sec
d14         0.00000000 sec
d15         0.00000000 sec
d16         0.00000000 sec
d17         0.00000000 sec
d18         0.00000000 sec
d19         0.00000000 sec
d20         0.00000000 sec
d21         0.00000000 sec
d22         0.00000000 sec
d23         0.00000000 sec
d24         0.00000000 sec
d25         0.00000000 sec
d26         0.00000000 sec
d27         0.00000000 sec
d28         0.00000000 sec
d29         0.00000000 sec
d30         0.00000000 sec
d31         0.00000000 sec
d32         0.00000000 sec
d33         0.00000000 sec
d34         0.00000000 sec
d35         0.00000000 sec
d36         0.00000000 sec
d37         0.00000000 sec
d38         0.00000000 sec
d39         0.00000000 sec
d40         0.00000000 sec
d41         0.00000000 sec
d42         0.00000000 sec
d43         0.00000000 sec
d44         0.00000000 sec
d45         0.00000000 sec
d46         0.00000000 sec
d47         0.00000000 sec
d48         0.00000000 sec
d49         0.00000000 sec
d50         0.00000000 sec
d51         0.00000000 sec
d52         0.00000000 sec
d53         0.00000000 sec
d54         0.00000000 sec
d55         0.00000000 sec
d56         0.00000000 sec
d57         0.00000000 sec
d58         0.00000000 sec
d59         0.00000000 sec
d60         0.00000000 sec
d61         0.00000000 sec
d62         0.00000000 sec
d63         0.00000000 sec
d64         0.00000000 sec
d65         0.00000000 sec
d66         0.00000000 sec
d67         0.00000000 sec
d68         0.00000000 sec
d69         0.00000000 sec
d70         0.00000000 sec
d71         0.00000000 sec
d72         0.00000000 sec
d73         0.00000000 sec
d74         0.00000000 sec
d75         0.00000000 sec
d76         0.00000000 sec
d77         0.00000000 sec
d78         0.00000000 sec
d79         0.00000000 sec
d80         0.00000000 sec
d81         0.00000000 sec
d82         0.00000000 sec
d83         0.00000000 sec
d84         0.00000000 sec
d85         0.00000000 sec
d86         0.00000000 sec
d87         0.00000000 sec
d88         0.00000000 sec
d89         0.00000000 sec
d90         0.00000000 sec
d91         0.00000000 sec
d92         0.00000000 sec
d93         0.00000000 sec
d94         0.00000000 sec
d95         0.00000000 sec
d96         0.00000000 sec
d97         0.00000000 sec
d98         0.00000000 sec
d99         0.00000000 sec
d100        0.00000000 sec

===== CHANNEL f1 =====
NUC1        13C
P1          12.00 usec
PL1         0.00 dB
PC1         100.00 usec
P2          200.00 usec
PL2         0.00 dB
PC2         100.00 usec
P3          500.00 usec
PL3         120.00 dB
PC3         100.00 usec
SFO1        125.7642548 MHz
SFO2        2.70 GHz
SFO3        2.70 GHz
SFO4        2.70 GHz
SFO5        2.70 GHz
SFO6        2.70 GHz
SFO7        2.70 GHz
SFO8        2.70 GHz
SFO9        2.70 GHz
SFO10       2.70 GHz
SFO11       2.70 GHz
SFO12       2.70 GHz
SFO13       2.70 GHz
SFO14       2.70 GHz
SFO15       2.70 GHz
SFO16       2.70 GHz
SFO17       2.70 GHz
SFO18       2.70 GHz
SFO19       2.70 GHz
SFO20       2.70 GHz
SFO21       2.70 GHz
SFO22       2.70 GHz
SFO23       2.70 GHz
SFO24       2.70 GHz
SFO25       2.70 GHz
SFO26       2.70 GHz
SFO27       2.70 GHz
SFO28       2.70 GHz
SFO29       2.70 GHz
SFO30       2.70 GHz
SFO31       2.70 GHz
SFO32       2.70 GHz
SFO33       2.70 GHz
SFO34       2.70 GHz
SFO35       2.70 GHz
SFO36       2.70 GHz
SFO37       2.70 GHz
SFO38       2.70 GHz
SFO39       2.70 GHz
SFO40       2.70 GHz
SFO41       2.70 GHz
SFO42       2.70 GHz
SFO43       2.70 GHz
SFO44       2.70 GHz
SFO45       2.70 GHz
SFO46       2.70 GHz
SFO47       2.70 GHz
SFO48       2.70 GHz
SFO49       2.70 GHz
SFO50       2.70 GHz
SFO51       2.70 GHz
SFO52       2.70 GHz
SFO53       2.70 GHz
SFO54       2.70 GHz
SFO55       2.70 GHz
SFO56       2.70 GHz
SFO57       2.70 GHz
SFO58       2.70 GHz
SFO59       2.70 GHz
SFO60       2.70 GHz
SFO61       2.70 GHz
SFO62       2.70 GHz
SFO63       2.70 GHz
SFO64       2.70 GHz
SFO65       2.70 GHz
SFO66       2.70 GHz
SFO67       2.70 GHz
SFO68       2.70 GHz
SFO69       2.70 GHz
SFO70       2.70 GHz
SFO71       2.70 GHz
SFO72       2.70 GHz
SFO73       2.70 GHz
SFO74       2.70 GHz
SFO75       2.70 GHz
SFO76       2.70 GHz
SFO77       2.70 GHz
SFO78       2.70 GHz
SFO79       2.70 GHz
SFO80       2.70 GHz
SFO81       2.70 GHz
SFO82       2.70 GHz
SFO83       2.70 GHz
SFO84       2.70 GHz
SFO85       2.70 GHz
SFO86       2.70 GHz
SFO87       2.70 GHz
SFO88       2.70 GHz
SFO89       2.70 GHz
SFO90       2.70 GHz
SFO91       2.70 GHz
SFO92       2.70 GHz
SFO93       2.70 GHz
SFO94       2.70 GHz
SFO95       2.70 GHz
SFO96       2.70 GHz
SFO97       2.70 GHz
SFO98       2.70 GHz
SFO99       2.70 GHz
SFO100      2.70 GHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        13C
P1          12.00 usec
PL1         0.00 dB
PC1         100.00 usec
P2          200.00 usec
PL2         0.00 dB
PC2         100.00 usec
P3          500.2225011 MHz
PL3         23.54 dB
PC3         100.00 usec
SFO1        500.2225011 MHz

===== GRABVIEW CHANNEL =====
GRABM1     SDEP.100
SDEP1      100.00 usec
SDEP2      100.00 usec
SDEP3      100.00 usec
SDEP4      100.00 usec
SDEP5      100.00 usec
SDEP6      100.00 usec
SDEP7      100.00 usec
SDEP8      100.00 usec
SDEP9      100.00 usec
SDEP10     100.00 usec
SDEP11     100.00 usec
SDEP12     100.00 usec
SDEP13     100.00 usec
SDEP14     100.00 usec
SDEP15     100.00 usec
SDEP16     100.00 usec
SDEP17     100.00 usec
SDEP18     100.00 usec
SDEP19     100.00 usec
SDEP20     100.00 usec
SDEP21     100.00 usec
SDEP22     100.00 usec
SDEP23     100.00 usec
SDEP24     100.00 usec
SDEP25     100.00 usec
SDEP26     100.00 usec
SDEP27     100.00 usec
SDEP28     100.00 usec
SDEP29     100.00 usec
SDEP30     100.00 usec
SDEP31     100.00 usec
SDEP32     100.00 usec
SDEP33     100.00 usec
SDEP34     100.00 usec
SDEP35     100.00 usec
SDEP36     100.00 usec
SDEP37     100.00 usec
SDEP38     100.00 usec
SDEP39     100.00 usec
SDEP40     100.00 usec
SDEP41     100.00 usec
SDEP42     100.00 usec
SDEP43     100.00 usec
SDEP44     100.00 usec
SDEP45     100.00 usec
SDEP46     100.00 usec
SDEP47     100.00 usec
SDEP48     100.00 usec
SDEP49     100.00 usec
SDEP50     100.00 usec
SDEP51     100.00 usec
SDEP52     100.00 usec
SDEP53     100.00 usec
SDEP54     100.00 usec
SDEP55     100.00 usec
SDEP56     100.00 usec
SDEP57     100.00 usec
SDEP58     100.00 usec
SDEP59     100.00 usec
SDEP60     100.00 usec
SDEP61     100.00 usec
SDEP62     100.00 usec
SDEP63     100.00 usec
SDEP64     100.00 usec
SDEP65     100.00 usec
SDEP66     100.00 usec
SDEP67     100.00 usec
SDEP68     100.00 usec
SDEP69     100.00 usec
SDEP70     100.00 usec
SDEP71     100.00 usec
SDEP72     100.00 usec
SDEP73     100.00 usec
SDEP74     100.00 usec
SDEP75     100.00 usec
SDEP76     100.00 usec
SDEP77     100.00 usec
SDEP78     100.00 usec
SDEP79     100.00 usec
SDEP80     100.00 usec
SDEP81     100.00 usec
SDEP82     100.00 usec
SDEP83     100.00 usec
SDEP84     100.00 usec
SDEP85     100.00 usec
SDEP86     100.00 usec
SDEP87     100.00 usec
SDEP88     100.00 usec
SDEP89     100.00 usec
SDEP90     100.00 usec
SDEP91     100.00 usec
SDEP92     100.00 usec
SDEP93     100.00 usec
SDEP94     100.00 usec
SDEP95     100.00 usec
SDEP96     100.00 usec
SDEP97     100.00 usec
SDEP98     100.00 usec
SDEP99     100.00 usec
SDEP100    100.00 usec

F2 - Processing parameters
SI         32768
SF         125.7642548 MHz
WDW        no
SSB        0
GB         0
PC         2.00

ID_NMR Plot parameters
CX         22.80 cm
CY         15.65 cm
CZ         15.65 cm
FL1        24019.64 Hz
FL2        24019.64 Hz
FL3        24019.64 Hz
FPP        -10.287 ppm
FZ         -1293.96 Hz
PCPROG     1.00000000 cm
SECON      1324.10693 Hz/cm
    
```



3.19

gcosy60

Current Data Parameters  
 USER Khevit1  
 NAME RM-11-0712  
 PROCNO 2  
 P2 1

F2 - Acquisition Parameters  
 Date\_ 20190721  
 Time 15:53  
 INSTRUM spect  
 PROBRM 5 mm CPYCI 1H-  
 PULPROG zgpg30.prd  
 TD 2048  
 SOLVENT CDCl3  
 NS 1  
 DS 16  
 SFR 8012.820 Hz  
 FIDRES 3.412510 Hz  
 AQ 0.177852 sec  
 RG 457.1  
 TE 298.1 K  
 DE 6.00 usec  
 TE 298.1 K  
 DO 0.0000300 sec  
 DL 1.0000000 sec  
 CLS 4  
 CL3 0.0000000 sec  
 CL2 0.0000000 sec  
 CL0 0.00012480 sec

===== CHANNEL f1 =====  
 NUCL1 1H  
 P1 7.00 usec  
 PL1 1.60 dB  
 SFO1 500.2235015 MHz

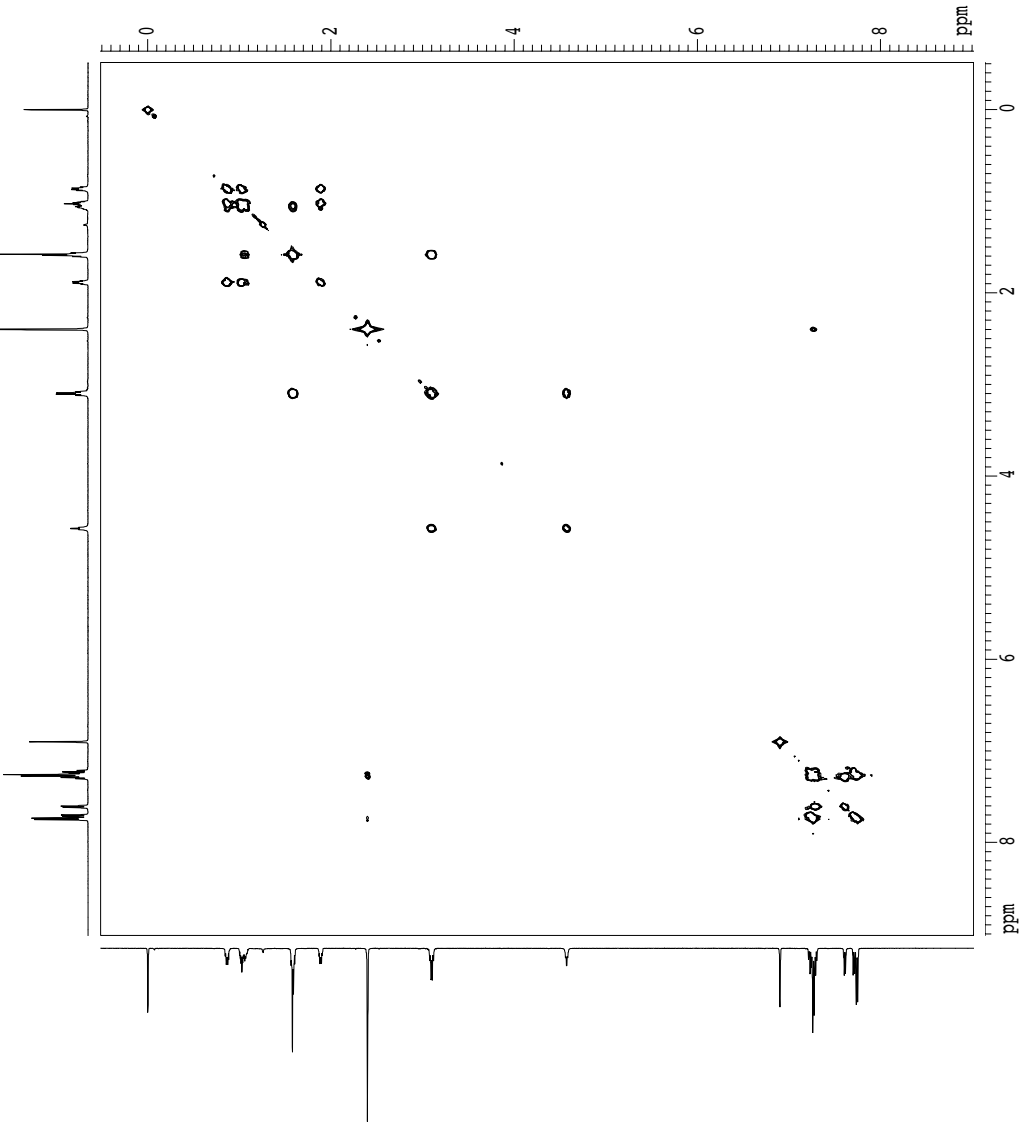
===== GRADIENT CHANNEL =====  
 CHANNEL gnmr1  
 SFO2 500.1313000 MHz  
 GX1 0.00 %  
 GX2 0.00 %  
 GP1 0.00 %  
 GP2 0.00 %  
 GP3 17.00 %  
 GP4 17.00 %  
 G16 1000.00 usec

F1 - Acquisition Parameters  
 NUCL1 13C  
 P1 4.72  
 PL1 19.24  
 SFO1 500.2235 MHz  
 FIDRES 15.450040 Hz  
 SW 16.418 ppm  
 FWHM 0.6

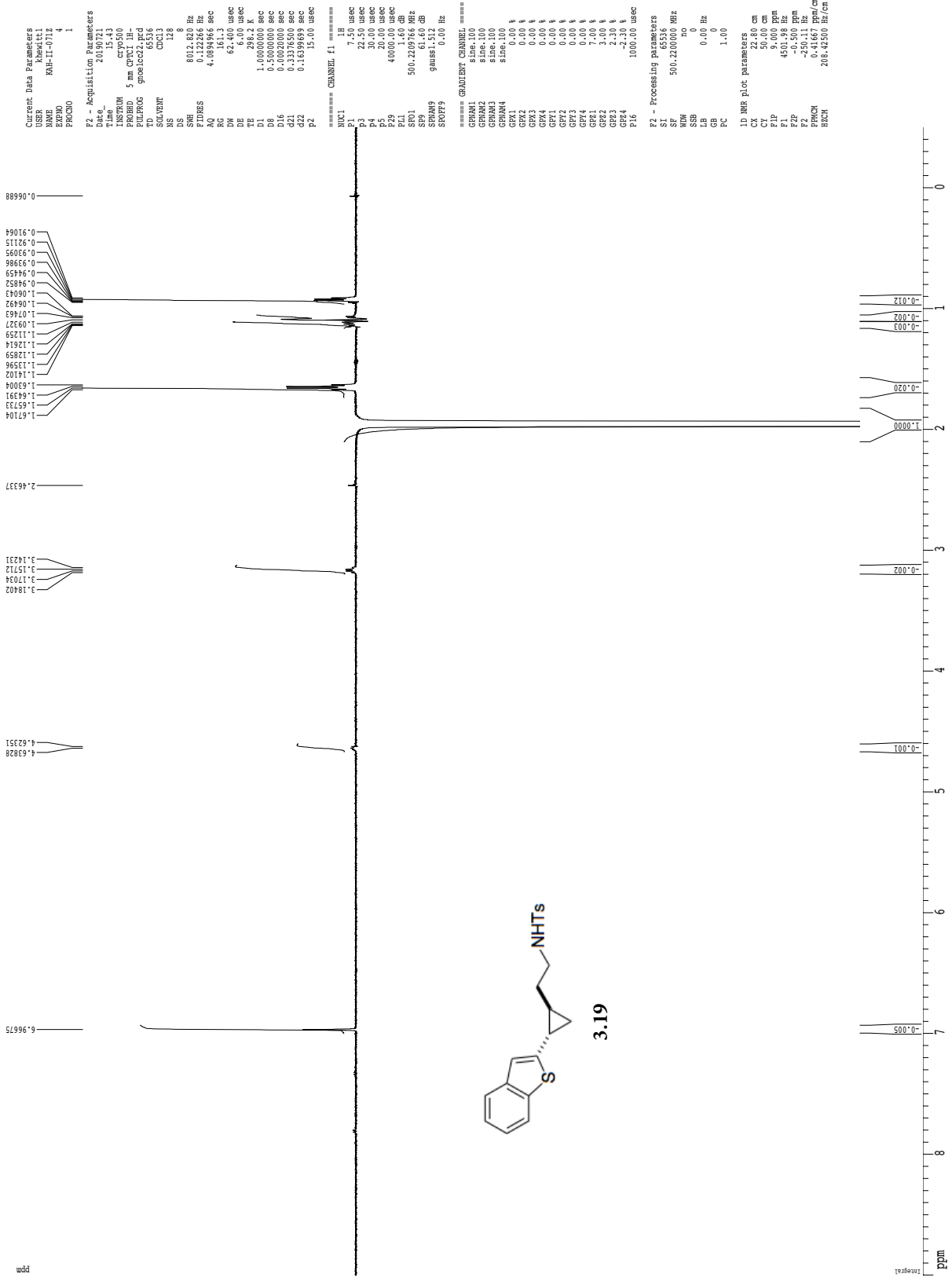
F2 - Processing Parameters  
 SI 1624  
 SF 500.2200333 MHz  
 WMW SINE  
 SSB 0  
 GB 0  
 PC 1.00

F1 - Processing Parameters  
 SI 1024  
 SF 500.2200333 MHz  
 WMW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

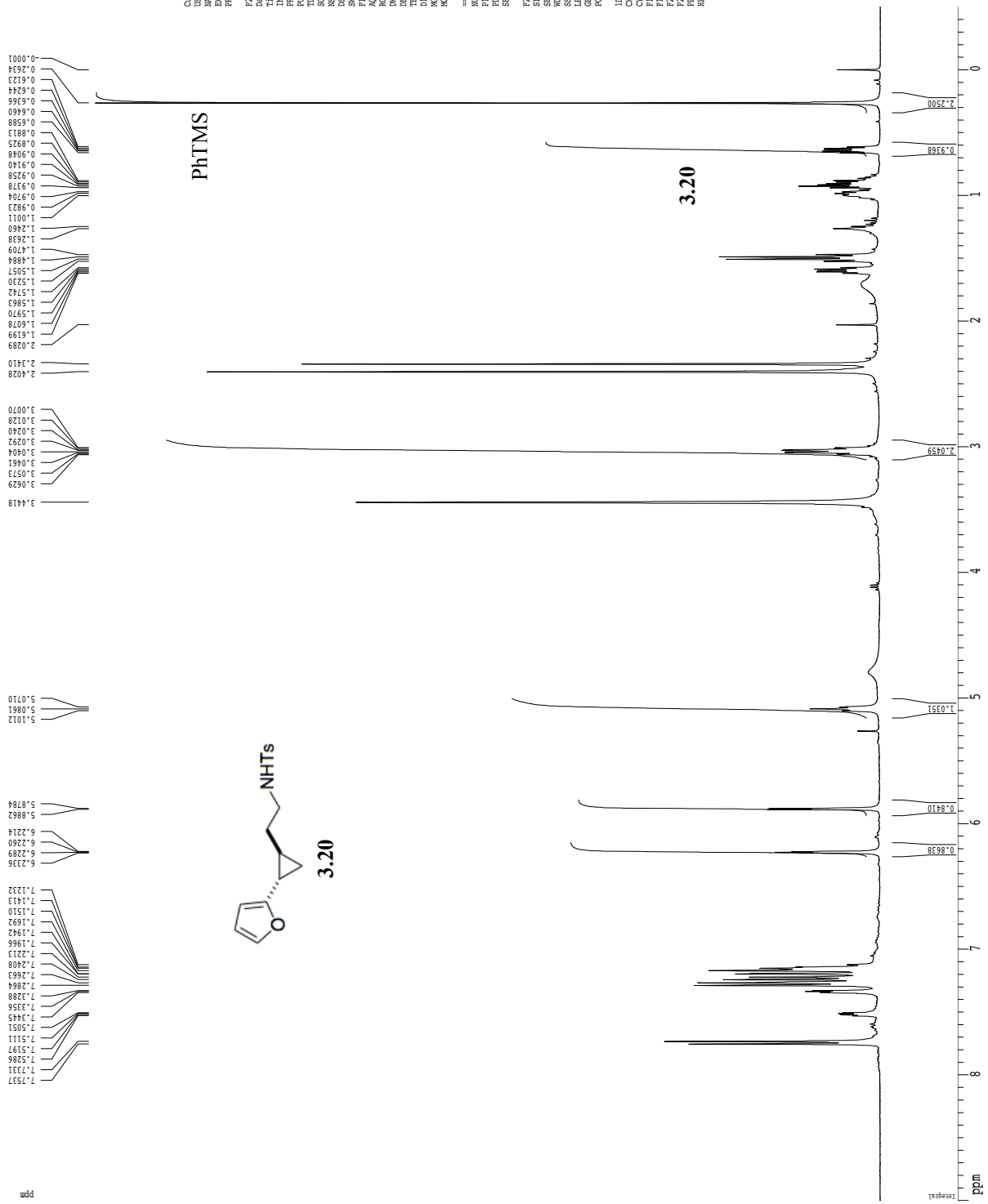
2D NMR plot parameters  
 CX1 15.00 cm  
 CX2 15.00 cm  
 F2PLO 9.014 ppm  
 F2FLO 48.513 ppm  
 F2PHI -20.513 deg  
 F2PI -256.52 Hz  
 F2PLO 9.014 ppm  
 F2FLO 4508.92 Hz  
 F2PHI -20.513 ppm  
 F2PI -256.52 Hz  
 F2PVM 0.63311 ppm/cm  
 F2VM 317.69580 Hz/cm  
 F2PMCH 0.63311 ppm/cm  
 F2VMCH 317.69580 Hz/cm



gnoe



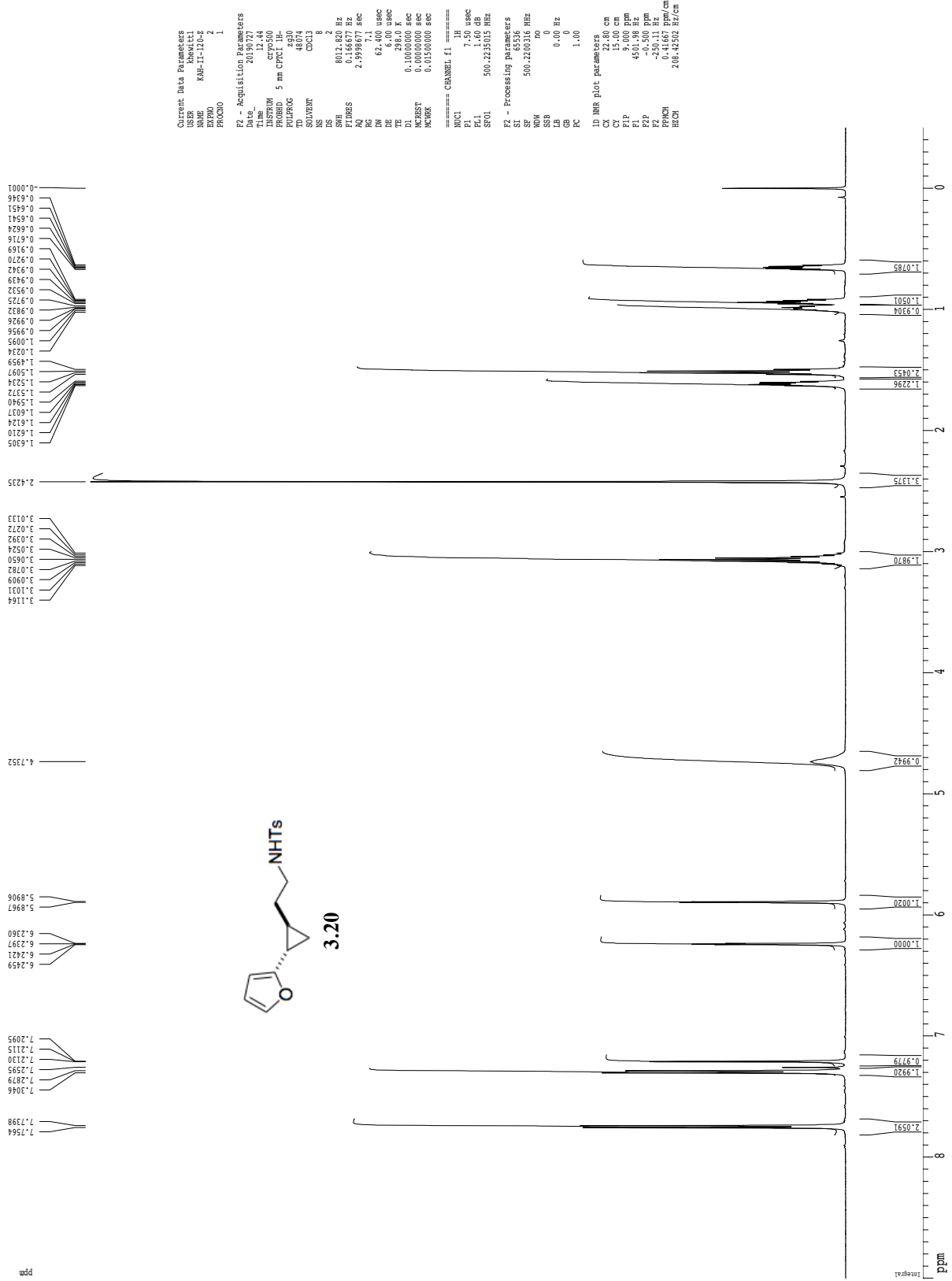
1H spectrum



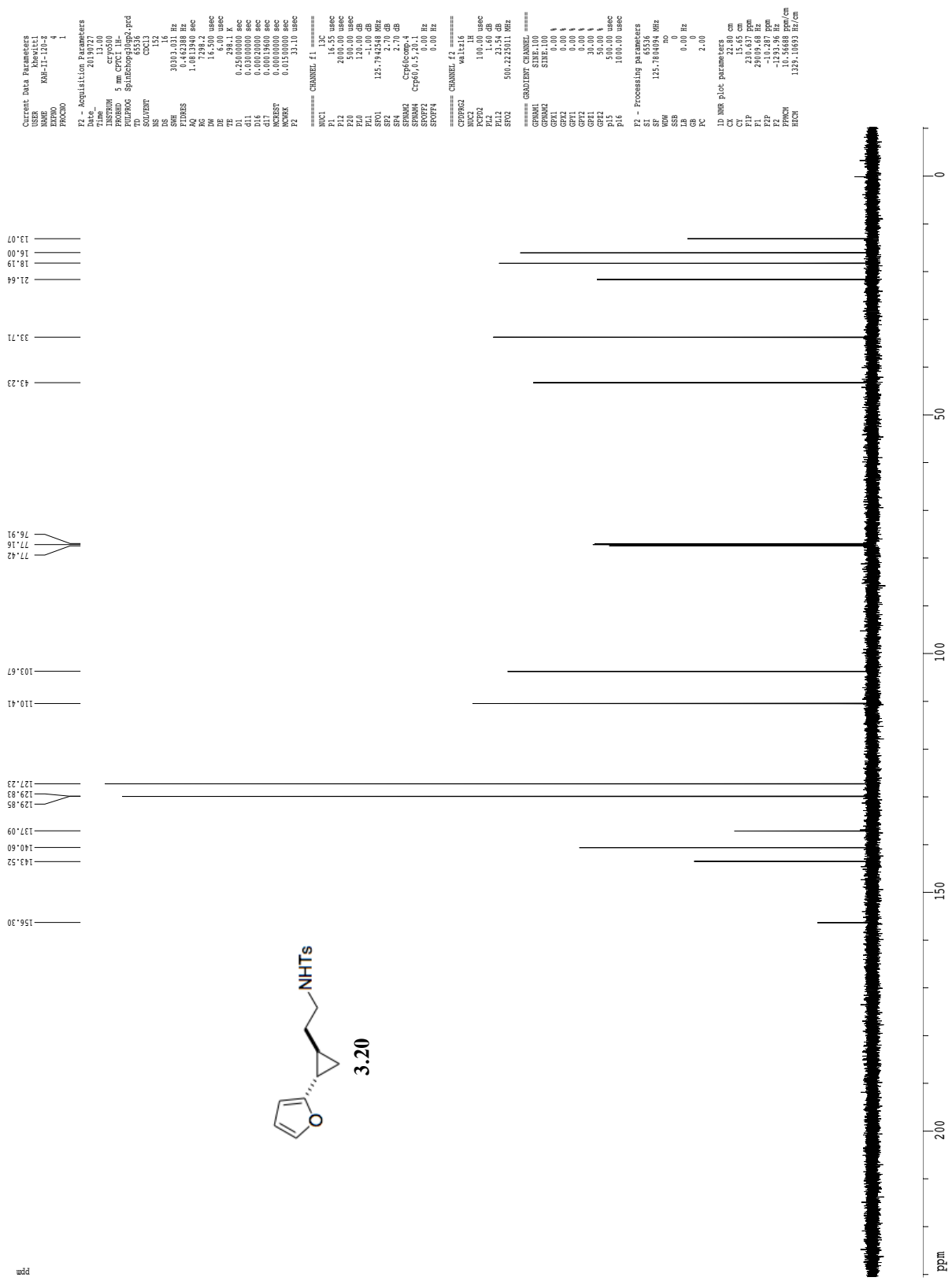
Current Data Parameters  
 USER kkwatt1  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20190725  
 Time 17.53  
 Operator kkwatt1  
 INSTRUM spect  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6410.256 Hz  
 FIDRES 0.166672 Hz  
 AQ 2.9999279 sec  
 RG 327.5  
 DW 78.000 usec  
 DE 4.50 usec  
 TE 298.1 K  
 F0 100.626136 MHz  
 MCHRES 0.1000000 sec  
 MCWRR 0.0550000 sec  
 ===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.132609 MHz  
 F2 - Processing parameters  
 SI 65536  
 SF 400.1300281 MHz  
 SD 32768  
 SSB 0 Hz  
 GB 0 Hz  
 PC 2.00  
 ID\_NMR File Parameters  
 CX 22.80 cm  
 CZ 15.40 cm  
 F1P 9.000 ppm  
 F2P 500.137 Hz  
 F3P 0.000 ppm  
 FZ -200.06 Hz  
 PPRCM 0.41667 ppm/cm  
 RECH 166.72086 Hz/cm

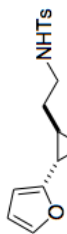


1H spectrum



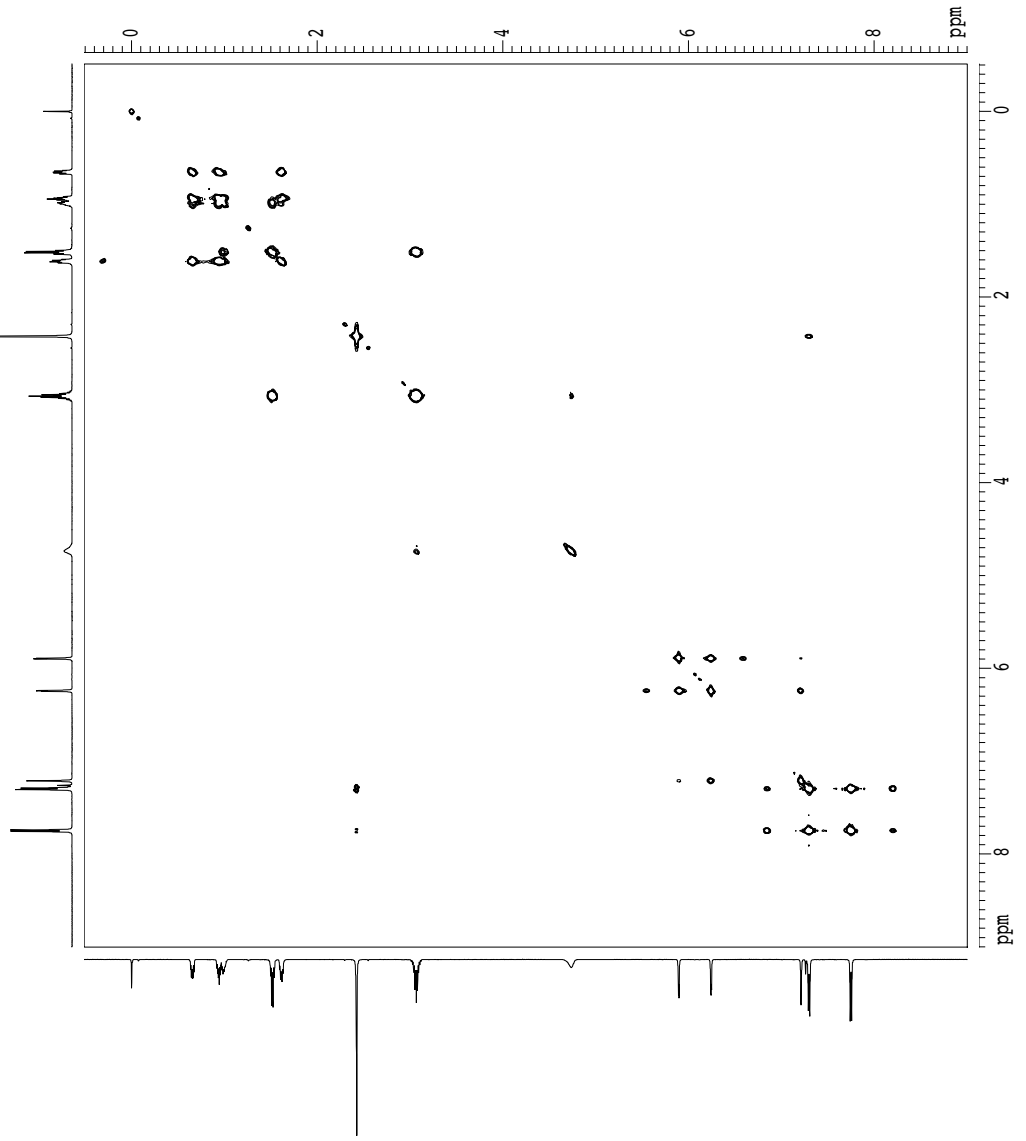
Z-restored spin-echo 13C spectrum with 1H decoupling





3.20

gcosy60



```

Current Data Parameters
=====
USER          khewitt1
NAME          KM-11-120-2
EXPNO        3
PROCNO       1

F2 - Acquisition Parameters
=====
Date_         2019077
Time          12.47
INSTRUM      cryso00
PROBHD       5 mm cryo
PULPROG      cosyprfand
TD           2048
SOLVENT      CDCl3
NS           1
DS           16
AQ           8012.816 Hz
F1FRES      3.912510 Hz
RG           0.12778452 sec
DW           62.400 usec
DE           6.00 usec
d0           2.00 usec
d1           0.0000300 sec
d11          1.00000000 sec
d13          0.0000300 sec
d16          0.0002000 sec
IN0          0.0012460 sec

===== CHANNEL f1 =====
NUC1         1H
P1           7.50 usec
PL1          1.60 dB
SFO1         500.225015 MHz

===== GRADIENT CHANNEL =====
GPRAM1      sine-100
GPRAM2      sine-100
GFX1        0.00 %
GFX2        0.00 %
GFX3        0.00 %
GFX4        0.00 %
GYZ1        17.00 %
GYZ2        17.00 %
P16         1000.00 usec

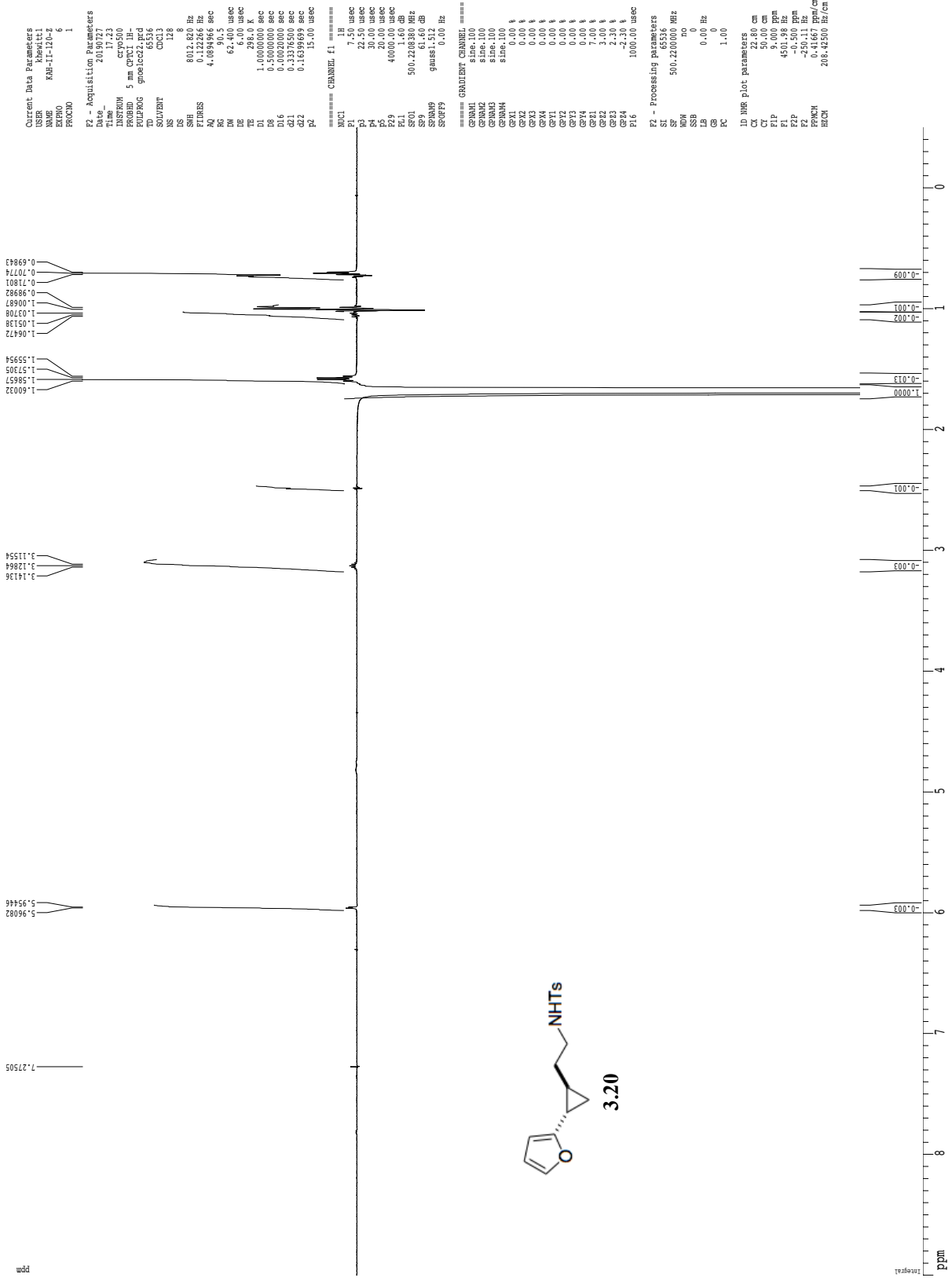
F1 - Acquisition Parameters
=====
NUC0         1
TD           512
SFO0         500.2235 MHz
F1FRES      15.650040 Hz
SMA         16.018 ppm
RG0         0.00 usec

F2 - Processing parameters
=====
SI           1024
SF           500.220315 MHz
SMA         SINE
SSB         0.00 Hz
LB          0.00 Hz
GB          0
PC           1.00

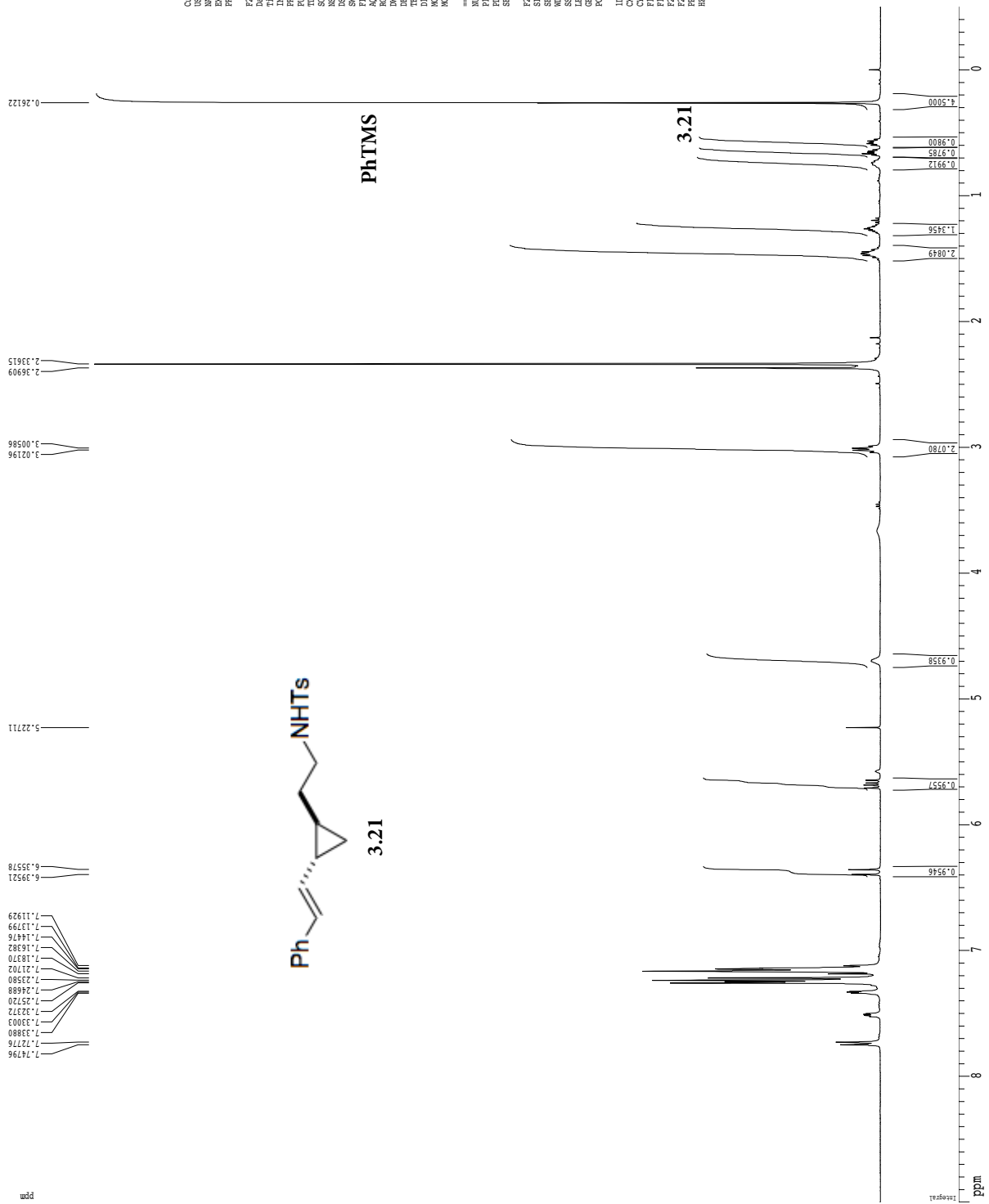
F1 - Processing parameters
=====
SI           1024
SF           500.220315 MHz
SMA         SINE
SSB         0.00 Hz
LB          0.00 Hz
GB          0
PC           1.00

2D NMR plot parameters
=====
CX2         15.00 cm
CX1         15.00 cm
FZ0         4502.93 Hz
F2PHI      -0.509 ppm
F2H1       -254.68 Hz
F1F0       9.002 ppm
F1L0       4502.93 Hz
F1H1       -254.68 Hz
F2P0P0CM   0.63407 ppm/cm
F2P0P1CM   317.17413 Hz/cm
F1P0P0CM   0.63407 ppm/cm
F1P0P1CM   317.17413 Hz/cm
  
```

gnoe



1H spectrum



Current Data Parameters  
 USER kkwatt1  
 NAME KME-1264  
 EXPNO 2  
 PROCNO 1

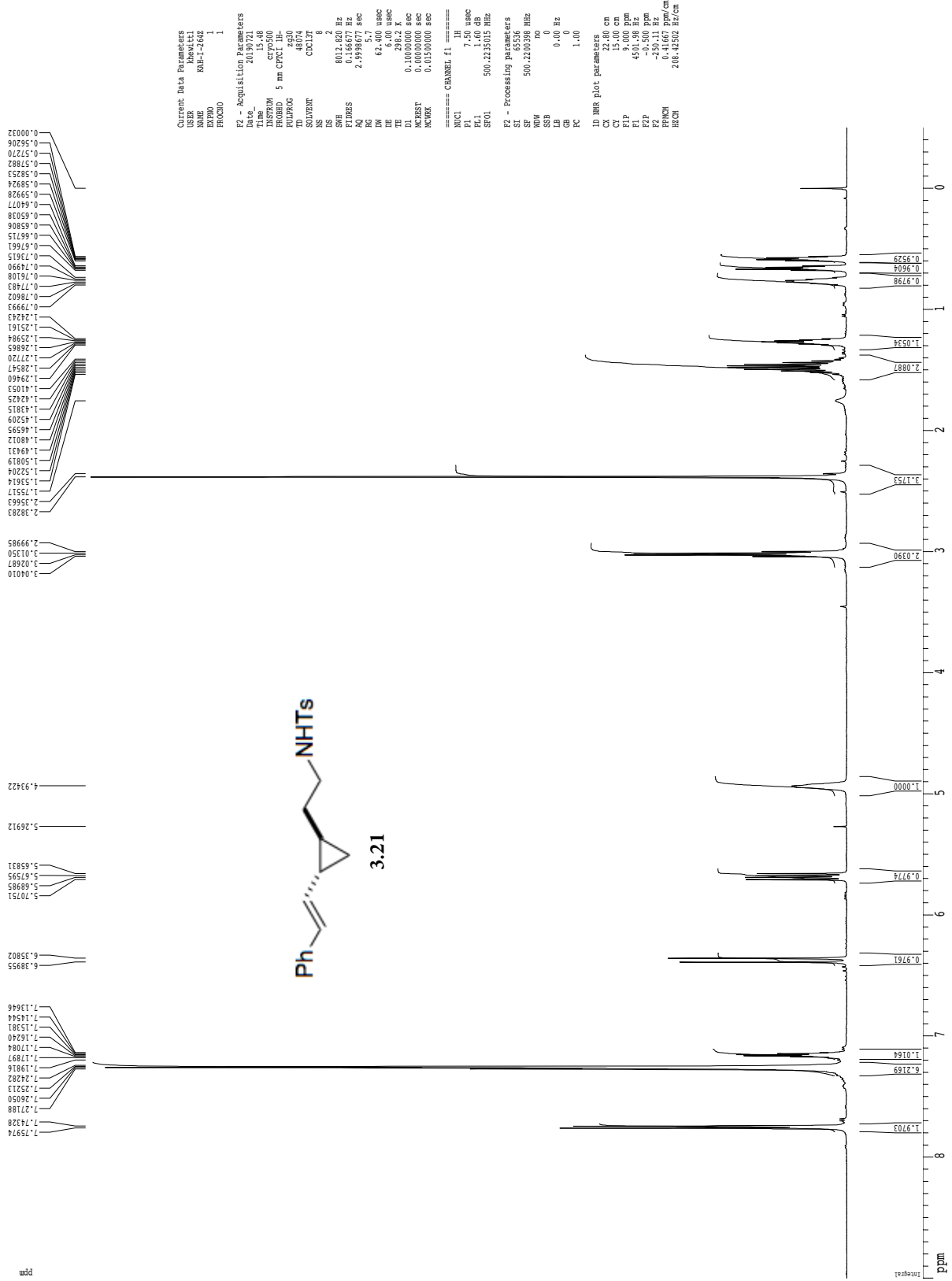
F2 - Acquisition Parameters  
 Date\_ 20190131  
 Time 16:37  
 Operator kkwatt1  
 PULPROG 5 mm QNP H270  
 PCPRG2 5g30  
 TD 38460  
 SOLVENT CDCl3  
 NS 0  
 DS 2  
 SWH 6410.256 Hz  
 FIDRES 0.166672 Hz  
 AQ 2.9999239 sec  
 RG 655  
 DW 78.000 usec  
 DE 4.50 usec  
 TE 298.1 K  
 TC 0.100000 sec  
 MCXST 0.000000 sec  
 MCHRS 0.0500000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.132809 MHz

F2 - Processing Parameters  
 SI 65536  
 SF 400.130614 MHz  
 WDW no  
 SSB 0 Hz  
 GB 0  
 PC 2.00

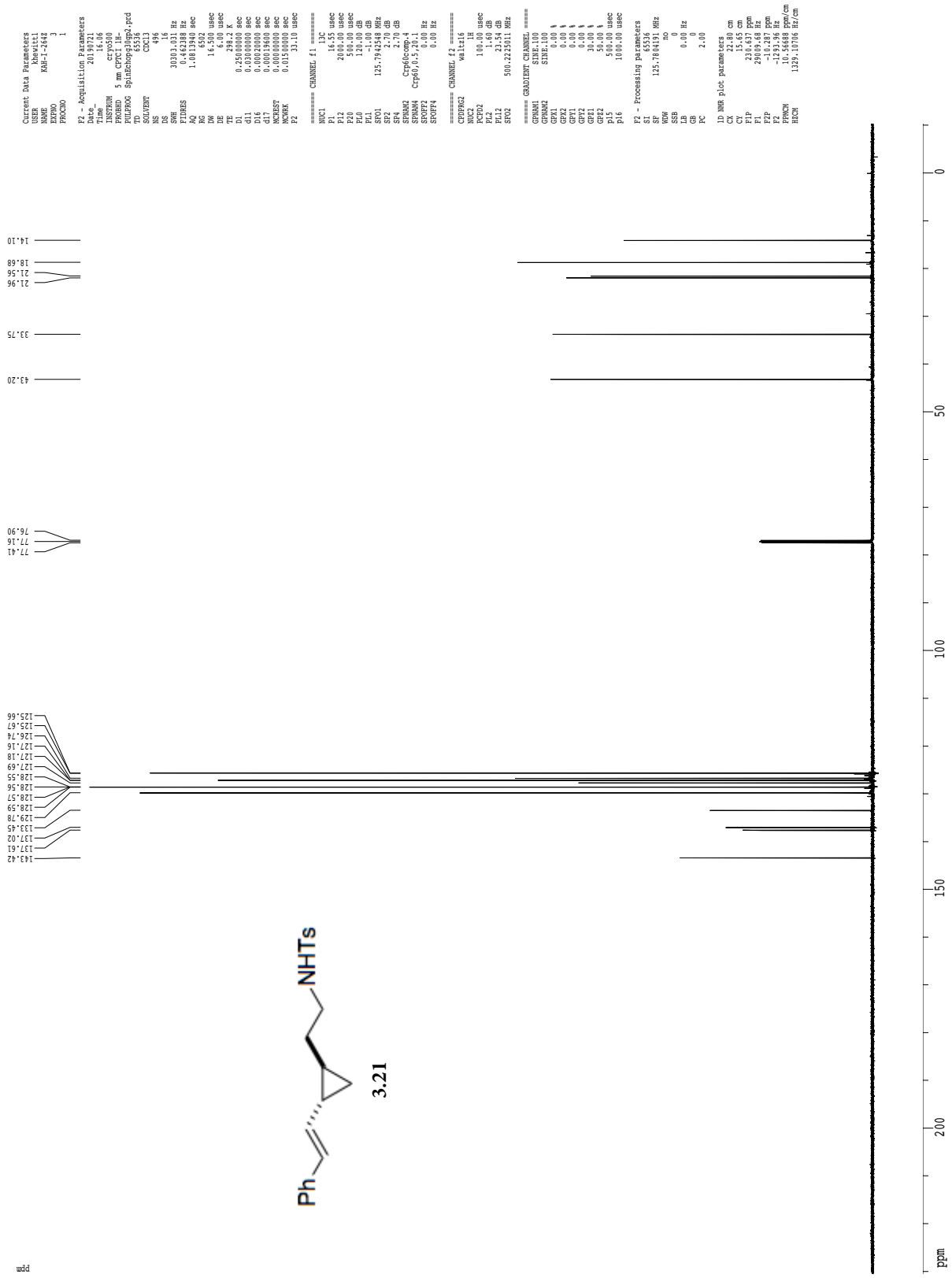
D0 NMR P10c Parameters  
 C1 22.80 cm  
 C2 15.40 cm  
 F1 9.000 ppm  
 F2 500.137 Hz  
 ZF 200.06 ppm  
 F2 -200.06 Hz  
 FFOCM 0.41667 ppm/cm  
 HECM 166.72087 Hz/cm

1H spectrum



Current Data Parameters  
 Name Rawdata1  
 ExpNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20190721  
 Time 15:48  
 Operator  
 PROBHD 5 mm CPYX1 1H  
 PULPROG zg30  
 TD 48074  
 SOLVENT CDCl3  
 NS 0  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.5998677 sec  
 RG 62.400 uspc  
 DE 6.00 uspc  
 TE 298.2 K  
 T1 0.100000 sec  
 T2 0.100000 sec  
 T3 0.100000 sec  
 T4 0.100000 sec  
 T5 0.100000 sec  
 T6 0.100000 sec  
 T7 0.100000 sec  
 T8 0.100000 sec  
 T9 0.100000 sec  
 T10 0.100000 sec  
 T11 0.100000 sec  
 T12 0.100000 sec  
 T13 0.100000 sec  
 T14 0.100000 sec  
 T15 0.100000 sec  
 T16 0.100000 sec  
 T17 0.100000 sec  
 T18 0.100000 sec  
 T19 0.100000 sec  
 T20 0.100000 sec  
 T21 0.100000 sec  
 T22 0.100000 sec  
 T23 0.100000 sec  
 T24 0.100000 sec  
 T25 0.100000 sec  
 T26 0.100000 sec  
 T27 0.100000 sec  
 T28 0.100000 sec  
 T29 0.100000 sec  
 T30 0.100000 sec  
 T31 0.100000 sec  
 T32 0.100000 sec  
 T33 0.100000 sec  
 T34 0.100000 sec  
 T35 0.100000 sec  
 T36 0.100000 sec  
 T37 0.100000 sec  
 T38 0.100000 sec  
 T39 0.100000 sec  
 T40 0.100000 sec  
 T41 0.100000 sec  
 T42 0.100000 sec  
 T43 0.100000 sec  
 T44 0.100000 sec  
 T45 0.100000 sec  
 T46 0.100000 sec  
 T47 0.100000 sec  
 T48 0.100000 sec  
 T49 0.100000 sec  
 T50 0.100000 sec  
 T51 0.100000 sec  
 T52 0.100000 sec  
 T53 0.100000 sec  
 T54 0.100000 sec  
 T55 0.100000 sec  
 T56 0.100000 sec  
 T57 0.100000 sec  
 T58 0.100000 sec  
 T59 0.100000 sec  
 T60 0.100000 sec  
 T61 0.100000 sec  
 T62 0.100000 sec  
 T63 0.100000 sec  
 T64 0.100000 sec  
 T65 0.100000 sec  
 T66 0.100000 sec  
 T67 0.100000 sec  
 T68 0.100000 sec  
 T69 0.100000 sec  
 T70 0.100000 sec  
 T71 0.100000 sec  
 T72 0.100000 sec  
 T73 0.100000 sec  
 T74 0.100000 sec  
 T75 0.100000 sec  
 T76 0.100000 sec  
 T77 0.100000 sec  
 T78 0.100000 sec  
 T79 0.100000 sec  
 T80 0.100000 sec  
 T81 0.100000 sec  
 T82 0.100000 sec  
 T83 0.100000 sec  
 T84 0.100000 sec  
 T85 0.100000 sec  
 T86 0.100000 sec  
 T87 0.100000 sec  
 T88 0.100000 sec  
 T89 0.100000 sec  
 T90 0.100000 sec  
 T91 0.100000 sec  
 T92 0.100000 sec  
 T93 0.100000 sec  
 T94 0.100000 sec  
 T95 0.100000 sec  
 T96 0.100000 sec  
 T97 0.100000 sec  
 T98 0.100000 sec  
 T99 0.100000 sec  
 T100 0.100000 sec  
 ===== CHANNEL f1 =====  
 NUCL 1H  
 P1 7.50 uspc  
 PL1 1.60 dB  
 SFO1 500.235015 MHz  
 F2 - Processing Parameters  
 SI 65536  
 SF 500.235015 MHz  
 DS 4  
 NS 0  
 ISF 0.00 Hz  
 GB 0  
 CB 0  
 PC 1.00  
 ID NMR file parameters  
 CD 22.80 cm  
 CT 15.00 cm  
 FFP 9.000 ppm  
 F1F 4501.98 Hz  
 F2F 250.11 Hz  
 F3F 125.06 Hz  
 F4F 62.53 Hz  
 FPMCN 0.41667 ppm/cm  
 RECN 206.45502 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling





```

Current Data Parameters
=====
USER      Name      khvict1
EXPNO    2
PROCNO   1

F2 - Acquisition Parameters
=====
Date_     20190721
Time      15:51
INSTRUM   cryo500
PROBHD    5 mm cryo
PULPROG   caspchg
TD         2848
SOLVENT   CDCl3
NS         1
DS         1
SWH        8012.600 Hz
FIDRES    3.912510 Hz
AQ         0.1278462 sec
RG         35.9
DM         62.400 usec
DE         286.2 K
TE         0.0000300 sec
d0         0.0000300 sec
d1         1.0000000 sec
d13       0.0000200 sec
d16       0.0002000 sec
d19       0.0001400 sec
ZG0       0.0000000 sec

===== CHANNEL f1 =====
NUC1      1H
P1        7.50 usec
PL        0.00 dB
SFO1      500.225015 MHz

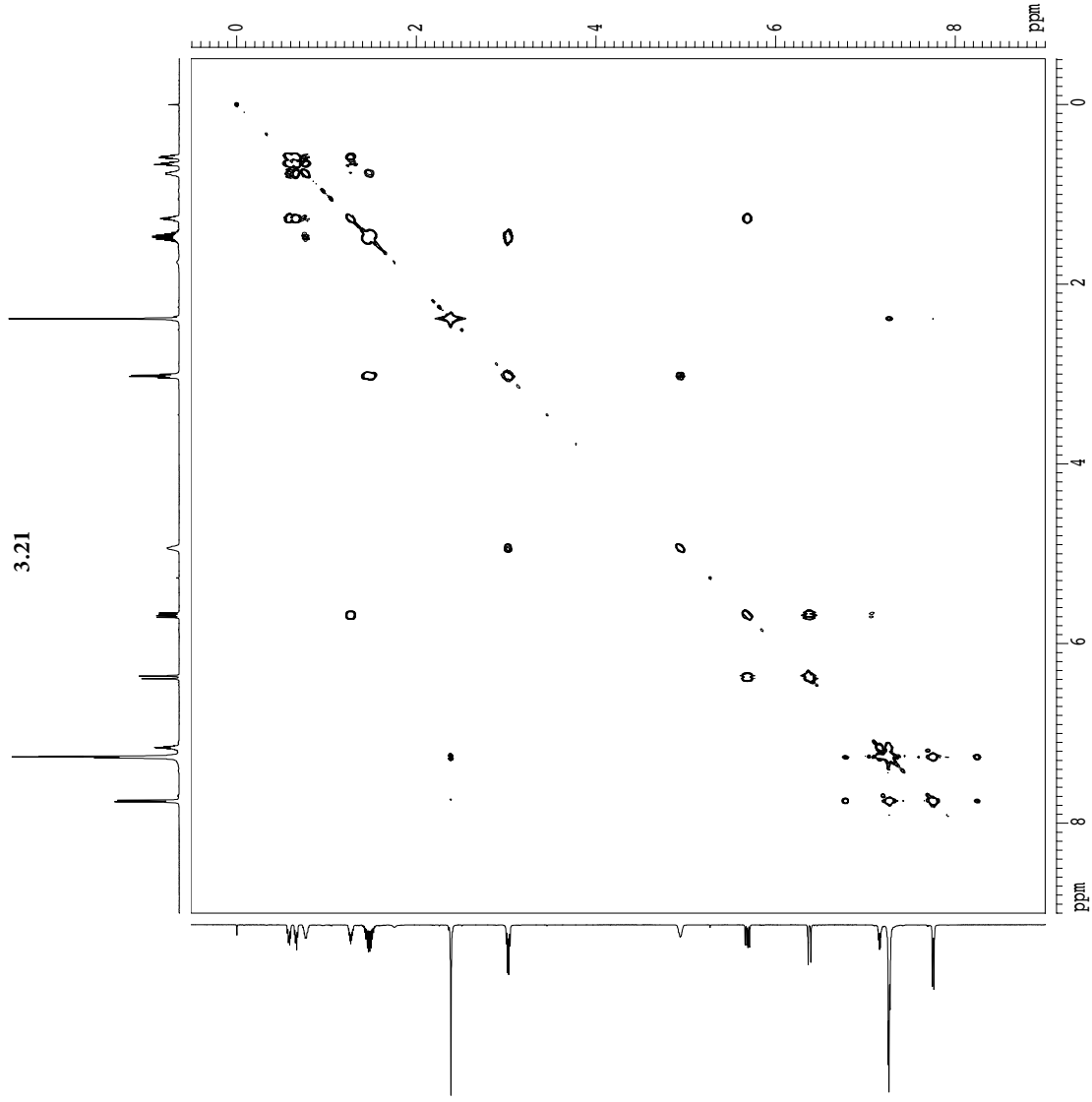
===== GRADIENT CHANNEL =====
GPRM1     size:100
GPRM2     size:100
GPRM3     size:100
GPRM4     size:100
GPRM5     size:100
GPRM6     size:100
GPRM7     size:100
GPRM8     size:100
GPRM9     size:100
GPRM10    size:100
GPRM11    size:100
GPRM12    size:100
GPRM13    size:100
GPRM14    size:100
GPRM15    size:100
GPRM16    size:100
GPRM17    size:100
GPRM18    size:100
GPRM19    size:100
GPRM20    size:100

F1 - Acquisition parameters
=====
NUC1      1H
P1        7.50 usec
PL        0.00 dB
SFO1      500.225015 MHz
FIDRES    3.912510 Hz
AQ         0.1278462 sec
RG         35.9
DM         62.400 usec
DE         286.2 K
TE         0.0000300 sec
d0         0.0000300 sec
d1         1.0000000 sec
d13       0.0000200 sec
d16       0.0002000 sec
d19       0.0001400 sec
ZG0       0.0000000 sec

F2 - Processing parameters
=====
SI         16384
SF         500.2200398 MHz
WDW        EM
SSB        0
GB         0
PC         1.00

F1 - Processing parameters
=====
SI         16384
SF         500.2200398 MHz
WDW        EM
SSB        0
GB         0
PC         1.00

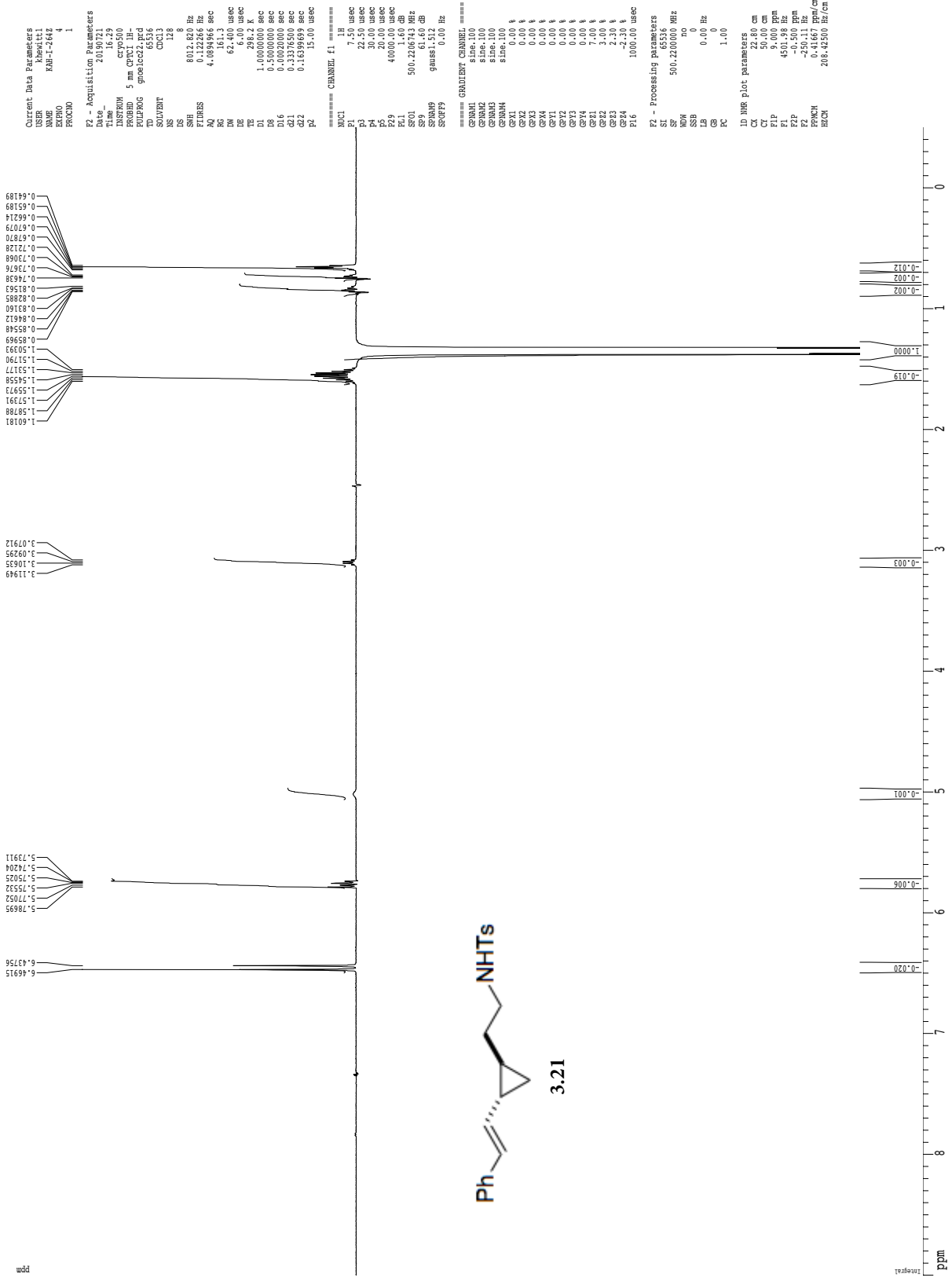
2D NMR plot parameters
=====
CX2        15.00 cm
CX1        15.00 cm
FZPLO      9.001 ppm
FZPLM      450.44 Hz
FZPHI      50.510 ppm
FZPH       -235.17 Hz
F1FLO      9.001 ppm
F1FLO      4502.44 Hz
F1FPHI     -235.17 ppm
F1FPHI     -235.17 ppm
F2FPHI     0.63407 ppm/cm
F2FPHI     317.17413 Hz/cm
F1FPHI     0.63407 ppm/cm
F1FPHI     317.17413 Hz/cm
  
```



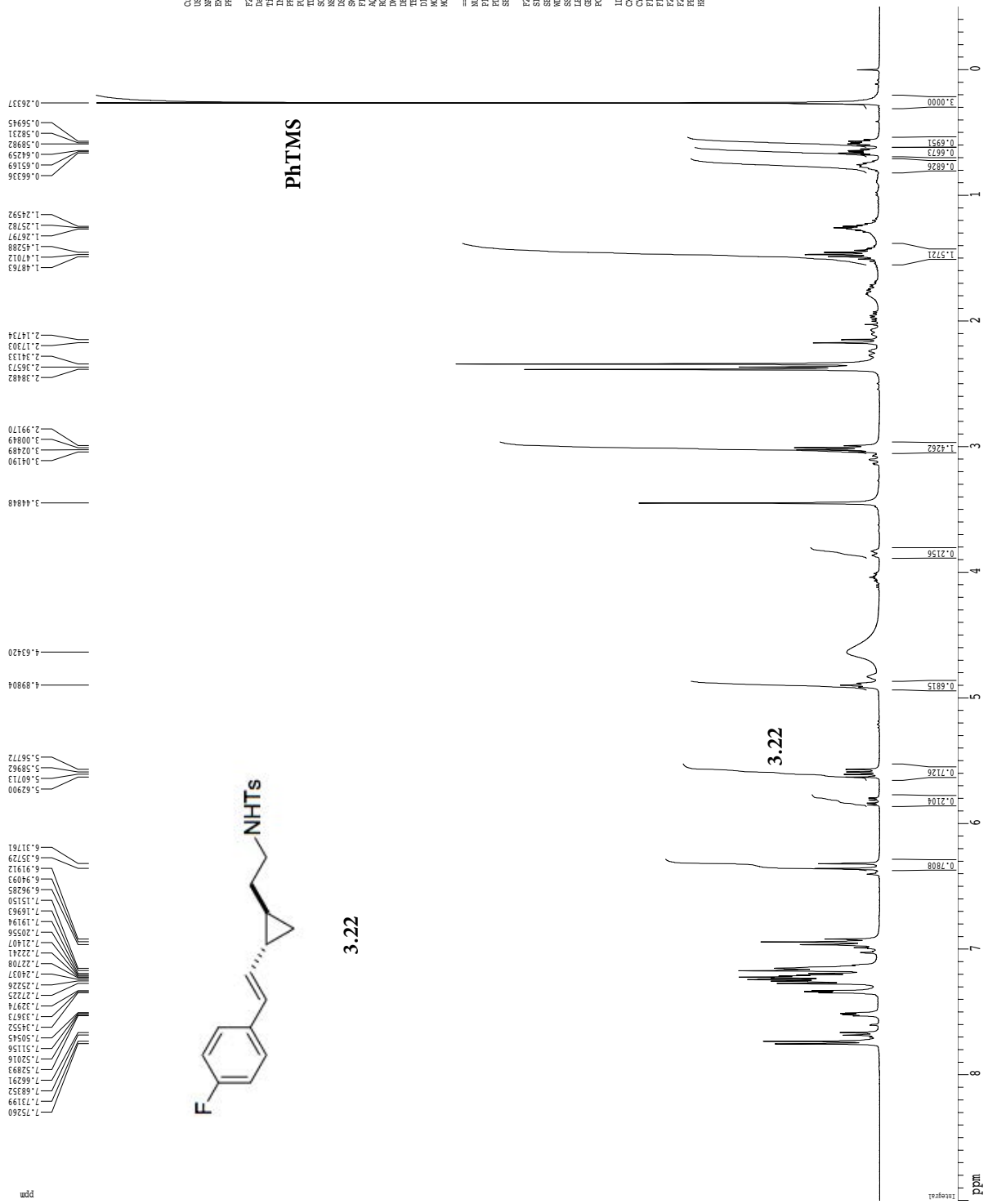
gcosy60



gnoe



1H spectrum



Current Data Parameters  
 USER Rawlett  
 NAME R01-11-17  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20191005  
 Time 16:47  
 Operator  
 PULPROG zgpg30  
 PROBR0 5 mm QNP1H70  
 TD 38460  
 SOLVENT COCL3T  
 NS 8  
 DS 2  
 SWH 6410.256 Hz  
 FIDRES 0.166672 Hz  
 AQ 2.5999239 sec  
 RG 655  
 DW 78.000 usec  
 DE 4.50 usec  
 TE 297.2 K  
 T1 0.10000000 sec  
 MCRST 0.00000000 sec  
 MCRRE 0.01500000 sec

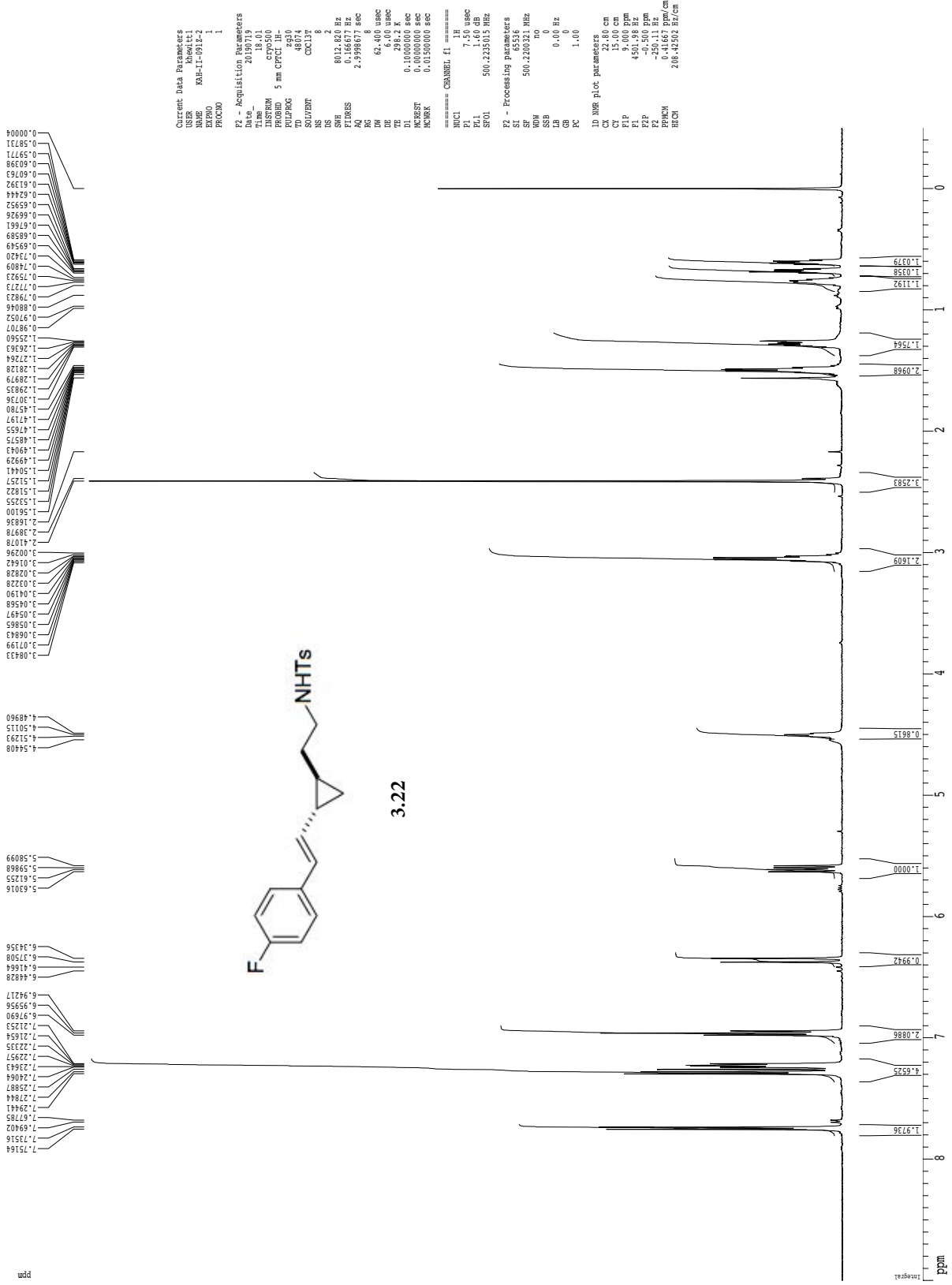
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.1328009 MHz

F2 - Processing Parameters  
 SI 65536  
 SF 400.1300004 MHz  
 SD 0  
 SSB 0 Hz  
 GB 0  
 PC 2.00

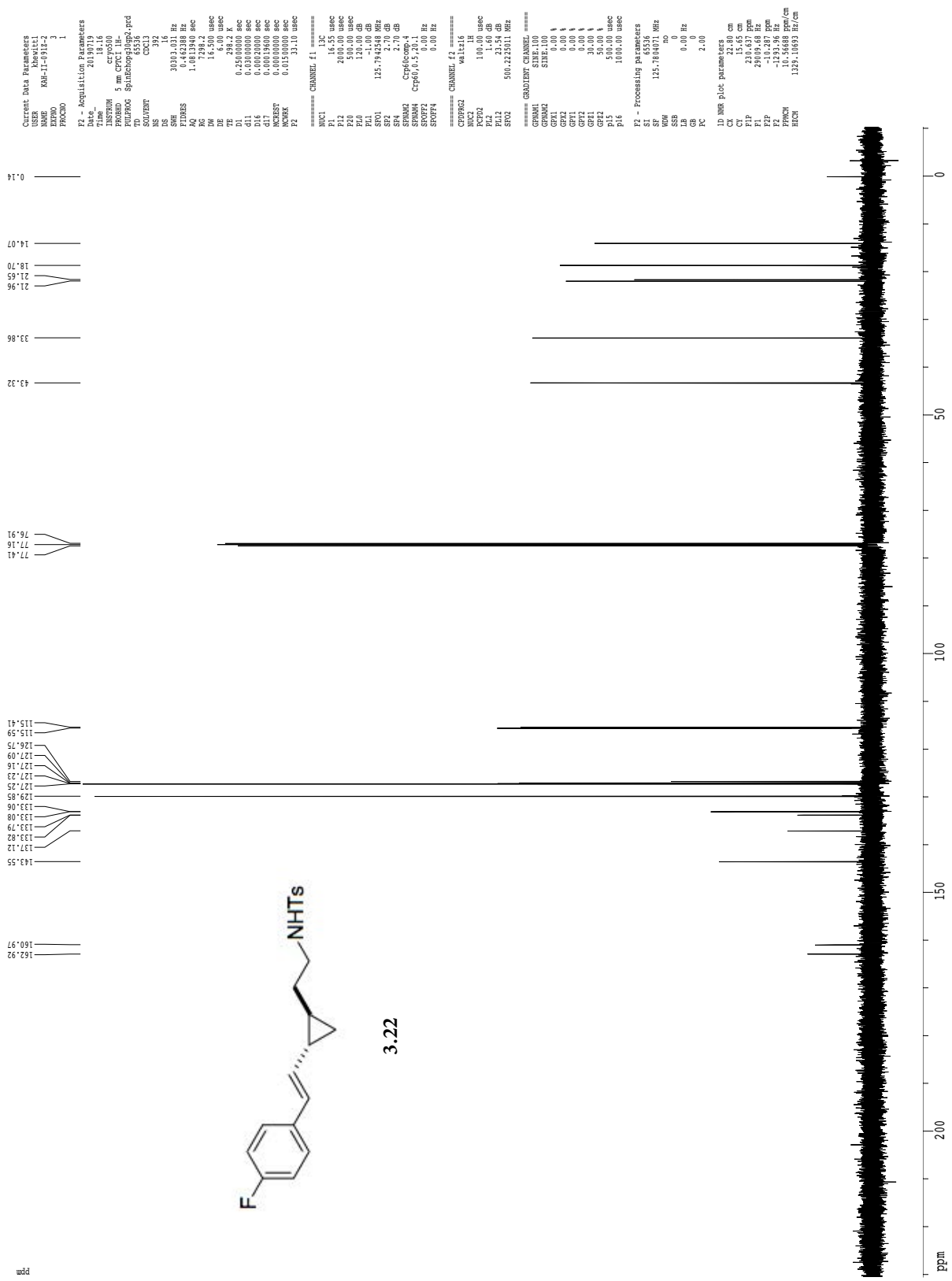
ID: NMR FIDIC Parameters  
 C1 22.80 cm  
 C2 15.40 cm  
 F1P 9.000 ppm  
 F2P 500.137 Hz  
 F3P -200.000 ppm  
 PPMXN 0.41667 ppm/cm  
 HZCXN 166.72086 Hz/cm



1H spectrum



Z-restored spin-echo 13C spectrum with 1H decoupling





gcosy60

3.22

Current Data Parameters  
 USER Name: khoviti1  
 EXPNO: RMH-11-091E-2  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_ Time: 20190719 18:03  
 INSTRUM: cryo500  
 PULPROG: zgpg30  
 PROCNO: 2588  
 TD: 65536  
 SOLVENT: CDCl3  
 NS: 1  
 DS: 4  
 SWH: 8013.630 Hz  
 FIDRES: 3.912510 Hz  
 AQ: 0.1278462 sec  
 RG: 456.1  
 DM: 62.400 usec  
 DE: 1.850 usec  
 TE: 296.2 K  
 D0: 0.0000300 sec  
 d1: 1.0000000 sec  
 d13: 0.0000300 sec  
 D16: 0.0002000 sec  
 ZD0: 0.0001489 sec

===== CHANNEL f1 =====  
 NUC1: 1H  
 P1: 7.50 usec  
 PL1: 0.00 dB  
 SFO1: 500.225015 MHz

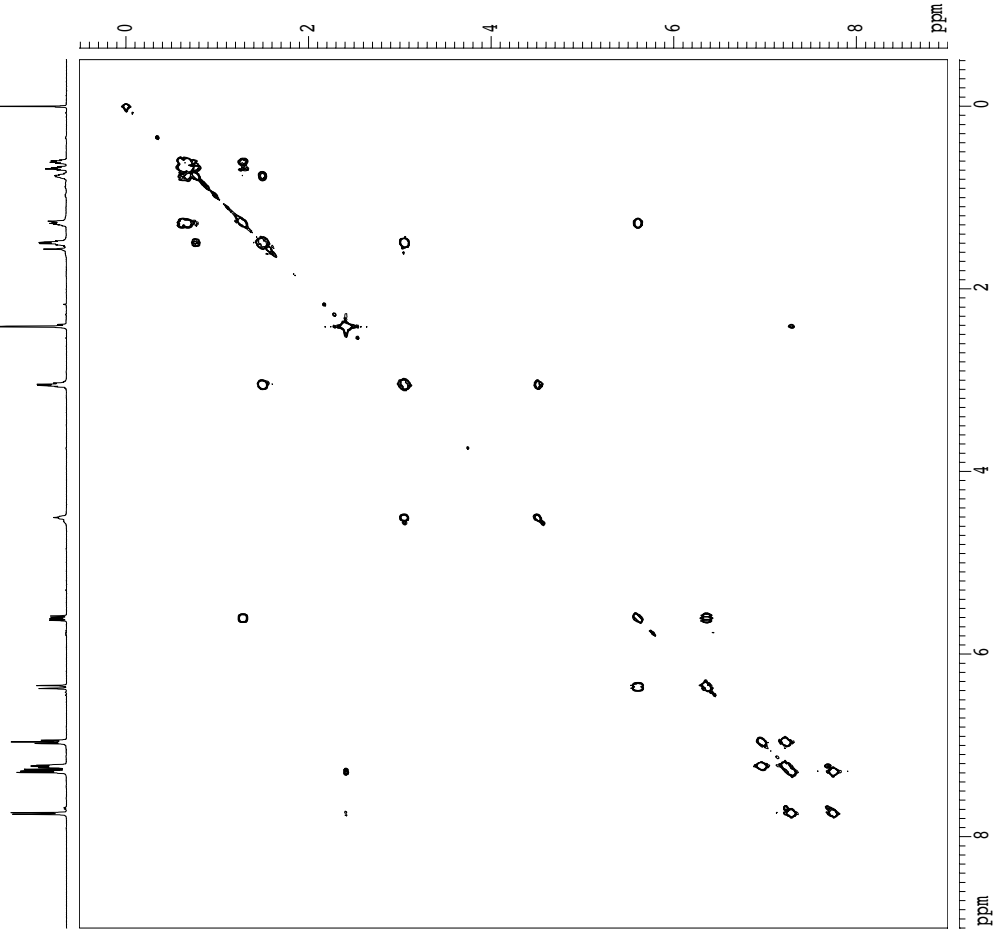
===== GRADIENT CHANNEL =====  
 GPRM1: sine.100  
 GRAM2: sine.100  
 GA2: 0.00 %  
 GP1: 0.00 %  
 GP2: 0.00 %  
 GP3: 17.00 %  
 PL6: 1000.00 usec

F1 - Acquisition parameters  
 MD: 1  
 SI: 16384  
 SF: 500.225015 MHz  
 WDM: 0  
 SSB: 0  
 GB: 0  
 PC: 1.00

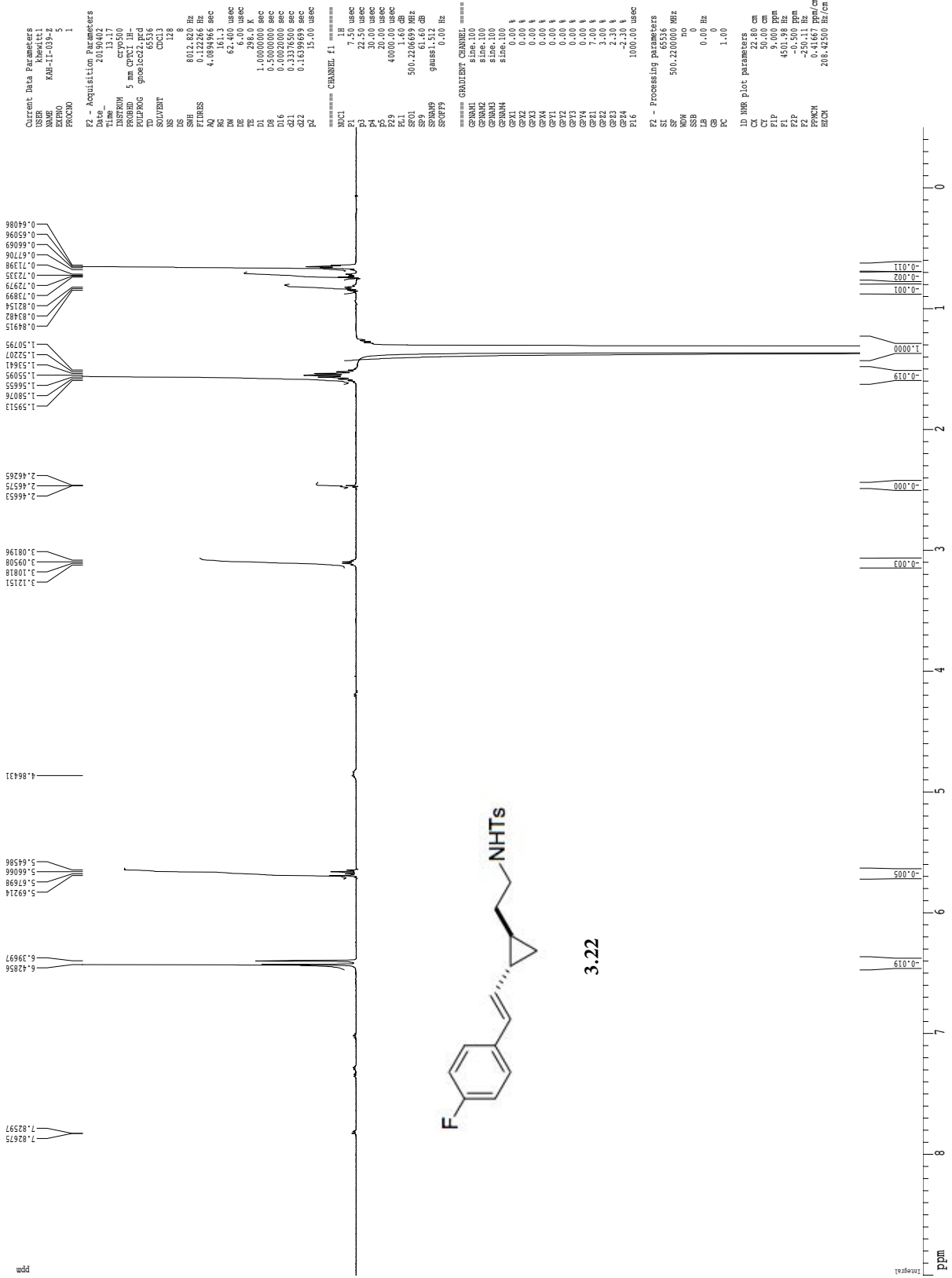
F2 - Processing parameters  
 SI: 16384  
 SF: 500.220021 MHz  
 WDM: 0  
 SSB: 0  
 GB: 0  
 PC: 1.00

F1 - Processing parameters  
 SI: 1024  
 SF: 500.220021 MHz  
 WDM: 0  
 SSB: 0  
 GB: 0

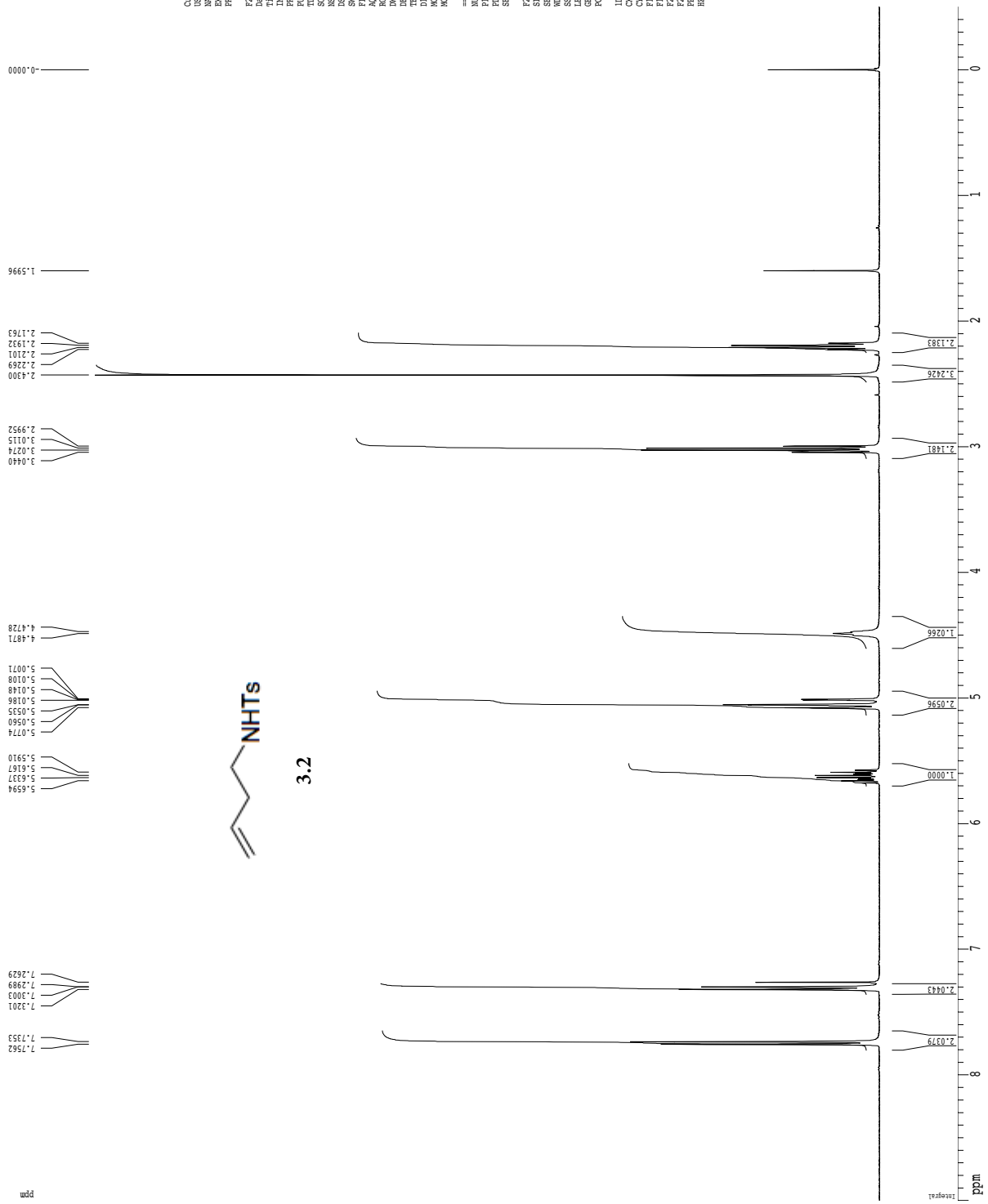
2D NMR plot Parameters  
 CX1: 15.00 cm  
 CX2: 15.00 cm  
 FZPO: 9.001 ppm  
 FZPO2: 9.001 ppm  
 FZPH: 450.510 Hz  
 FZPH2: -255.29 Hz  
 F1F0: 9.001 ppm  
 F1F02: 4502.32 Hz  
 F1F1: 10.510 ppm  
 F1F12: -2.510 ppm  
 F2F0MCH: 0.63407 ppm/cm  
 F2F0CM: 317.17413 Hz/cm  
 F1F0MCH: 0.63407 ppm/cm  
 F1F0CM: 317.17413 Hz/cm



gnoe

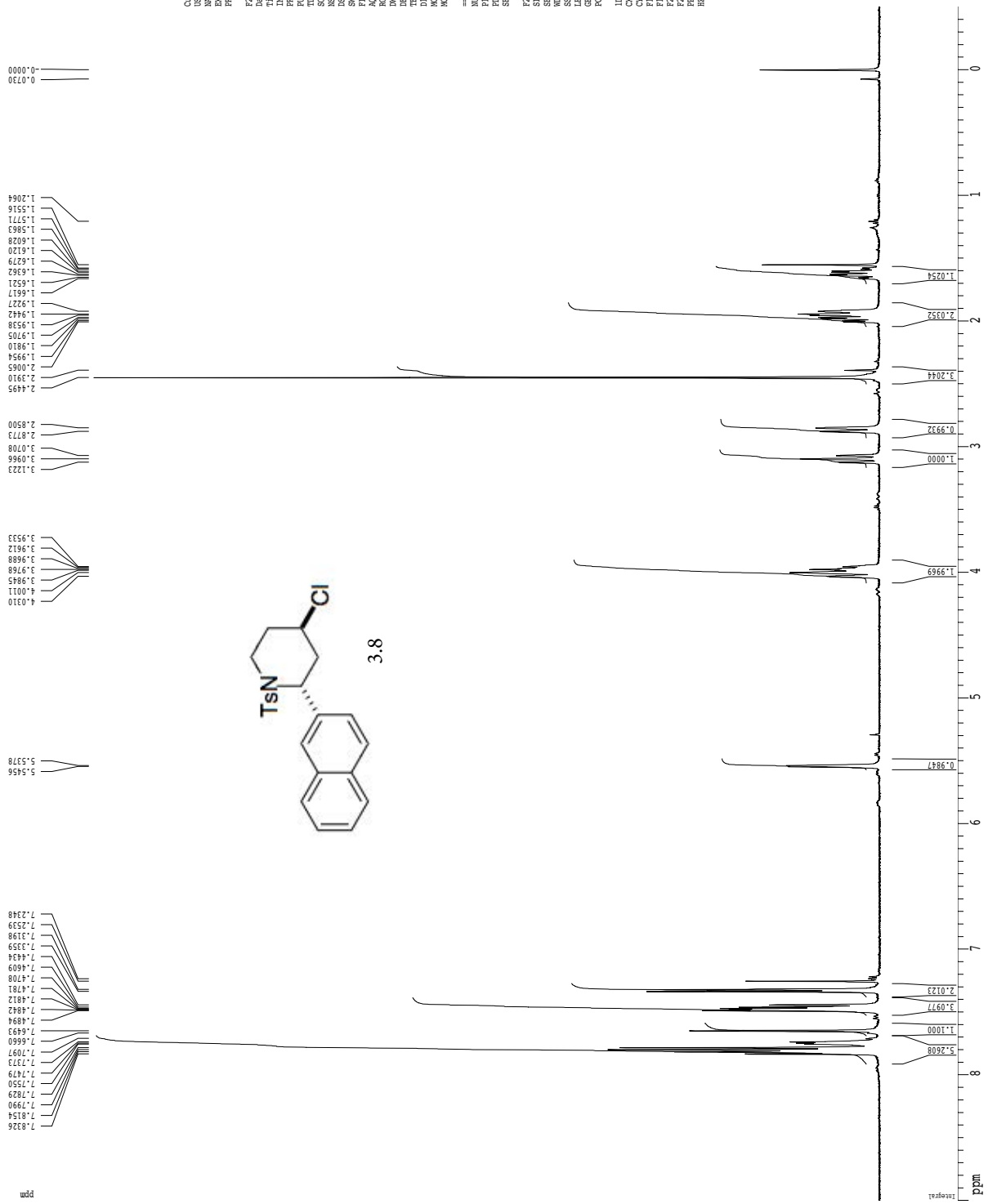


1H spectrum



Current Data Parameters  
 USER: etical  
 NAME: EHL-1V-122  
 EXPNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20190226  
 Time: 10.14  
 SYSTEM: spect  
 PULPROG: zgpg30  
 PROCNO: 38460  
 TD: 65536  
 SOLVENT: CDCl3  
 NS: 2  
 DS: 4  
 SWH: 6410.256 Hz  
 FIDRES: 0.166672 Hz  
 AQ: 2.999299 sec  
 RG: 327.5  
 DM: 78.000 us/cg  
 DE: 4.50 us/cg  
 TE: 298.0 K  
 T1: 0.100000 sec  
 T2: 0.100000 sec  
 T3: 0.100000 sec  
 MCHRG: 0.0500000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -1.10 db  
 SFO1: 400.132809 MHz  
 F2 - Processing Parameters  
 SI: 65536  
 SF: 400.130001 MHz  
 WDW: no  
 SSB: 0 Hz  
 GB: 0  
 PC: 2.00  
 ID: NMR FID Parameters  
 CD: 22.80 cm  
 CF: 15.00 cm  
 FIP: 9.000 ppm  
 F1: 500.137 Hz  
 F2: 125.762 ppm  
 F3: -200.06 Hz  
 FPRCM: 0.41667 ppm/cm  
 RECH: 166.72084 Hz/cm

1H spectrum

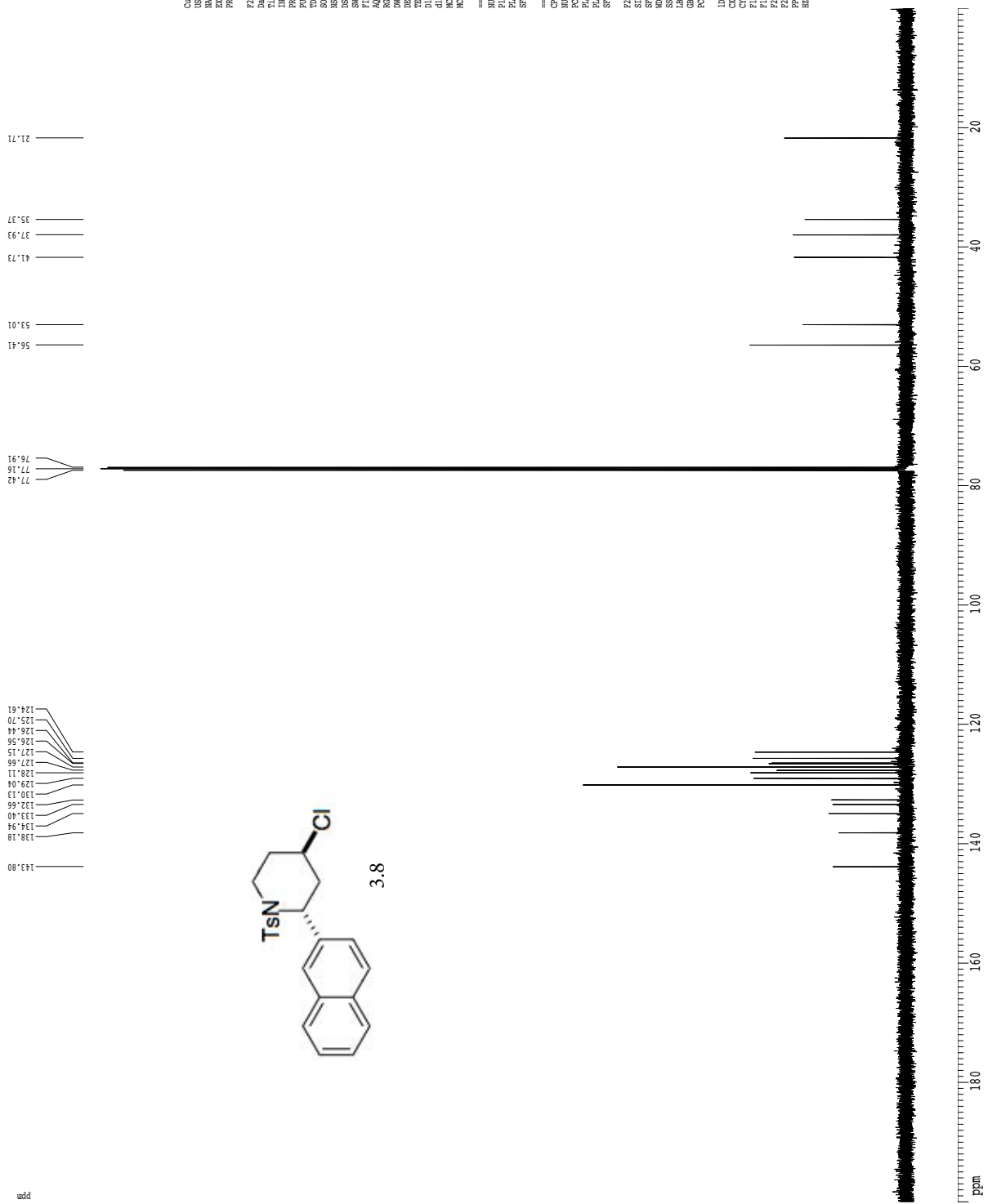


Current Data Parameters  
 USER: EHL-rj-2481  
 NAME: EHL-rj-2481  
 EXPNO: 2  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20171108  
 Time: 19.50  
 INSTRUM: spect  
 PROBHD: 5 mm 1HCOZY  
 PULPROG: zg30  
 TD: 81728  
 SOLVENT: CDCl3  
 NS: 8  
 DS: 2  
 SWH: 8012.820 Hz  
 FIDRES: 0.098042 Hz  
 AQ: 5.0998774 sec  
 RG: 655  
 DW: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.0 K  
 T1: 0.10000000 sec  
 T2: 0.10000000 sec  
 T3: 0.10000000 sec  
 T4: 0.10000000 sec  
 T5: 0.10000000 sec  
 T6: 0.10000000 sec  
 T7: 0.10000000 sec  
 T8: 0.10000000 sec  
 T9: 0.10000000 sec  
 T10: 0.10000000 sec  
 T11: 0.10000000 sec  
 T12: 0.10000000 sec  
 T13: 0.10000000 sec  
 T14: 0.10000000 sec  
 T15: 0.10000000 sec  
 T16: 0.10000000 sec  
 T17: 0.10000000 sec  
 T18: 0.10000000 sec  
 T19: 0.10000000 sec  
 T20: 0.10000000 sec  
 T21: 0.10000000 sec  
 T22: 0.10000000 sec  
 T23: 0.10000000 sec  
 T24: 0.10000000 sec  
 T25: 0.10000000 sec  
 T26: 0.10000000 sec  
 T27: 0.10000000 sec  
 T28: 0.10000000 sec  
 T29: 0.10000000 sec  
 T30: 0.10000000 sec  
 T31: 0.10000000 sec  
 T32: 0.10000000 sec  
 T33: 0.10000000 sec  
 T34: 0.10000000 sec  
 T35: 0.10000000 sec  
 T36: 0.10000000 sec  
 T37: 0.10000000 sec  
 T38: 0.10000000 sec  
 T39: 0.10000000 sec  
 T40: 0.10000000 sec  
 T41: 0.10000000 sec  
 T42: 0.10000000 sec  
 T43: 0.10000000 sec  
 T44: 0.10000000 sec  
 T45: 0.10000000 sec  
 T46: 0.10000000 sec  
 T47: 0.10000000 sec  
 T48: 0.10000000 sec  
 T49: 0.10000000 sec  
 T50: 0.10000000 sec  
 T51: 0.10000000 sec  
 T52: 0.10000000 sec  
 T53: 0.10000000 sec  
 T54: 0.10000000 sec  
 T55: 0.10000000 sec  
 T56: 0.10000000 sec  
 T57: 0.10000000 sec  
 T58: 0.10000000 sec  
 T59: 0.10000000 sec  
 T60: 0.10000000 sec  
 T61: 0.10000000 sec  
 T62: 0.10000000 sec  
 T63: 0.10000000 sec  
 T64: 0.10000000 sec  
 T65: 0.10000000 sec  
 T66: 0.10000000 sec  
 T67: 0.10000000 sec  
 T68: 0.10000000 sec  
 T69: 0.10000000 sec  
 T70: 0.10000000 sec  
 T71: 0.10000000 sec  
 T72: 0.10000000 sec  
 T73: 0.10000000 sec  
 T74: 0.10000000 sec  
 T75: 0.10000000 sec  
 T76: 0.10000000 sec  
 T77: 0.10000000 sec  
 T78: 0.10000000 sec  
 T79: 0.10000000 sec  
 T80: 0.10000000 sec  
 T81: 0.10000000 sec  
 T82: 0.10000000 sec  
 T83: 0.10000000 sec  
 T84: 0.10000000 sec  
 T85: 0.10000000 sec  
 T86: 0.10000000 sec  
 T87: 0.10000000 sec  
 T88: 0.10000000 sec  
 T89: 0.10000000 sec  
 T90: 0.10000000 sec  
 T91: 0.10000000 sec  
 T92: 0.10000000 sec  
 T93: 0.10000000 sec  
 T94: 0.10000000 sec  
 T95: 0.10000000 sec  
 T96: 0.10000000 sec  
 T97: 0.10000000 sec  
 T98: 0.10000000 sec  
 T99: 0.10000000 sec  
 T100: 0.10000000 sec



13C spectrum with 1H decoupling



```

Current Data Parameters
NAME      EUL-11-281
EXPNO    4
PROCNO   1

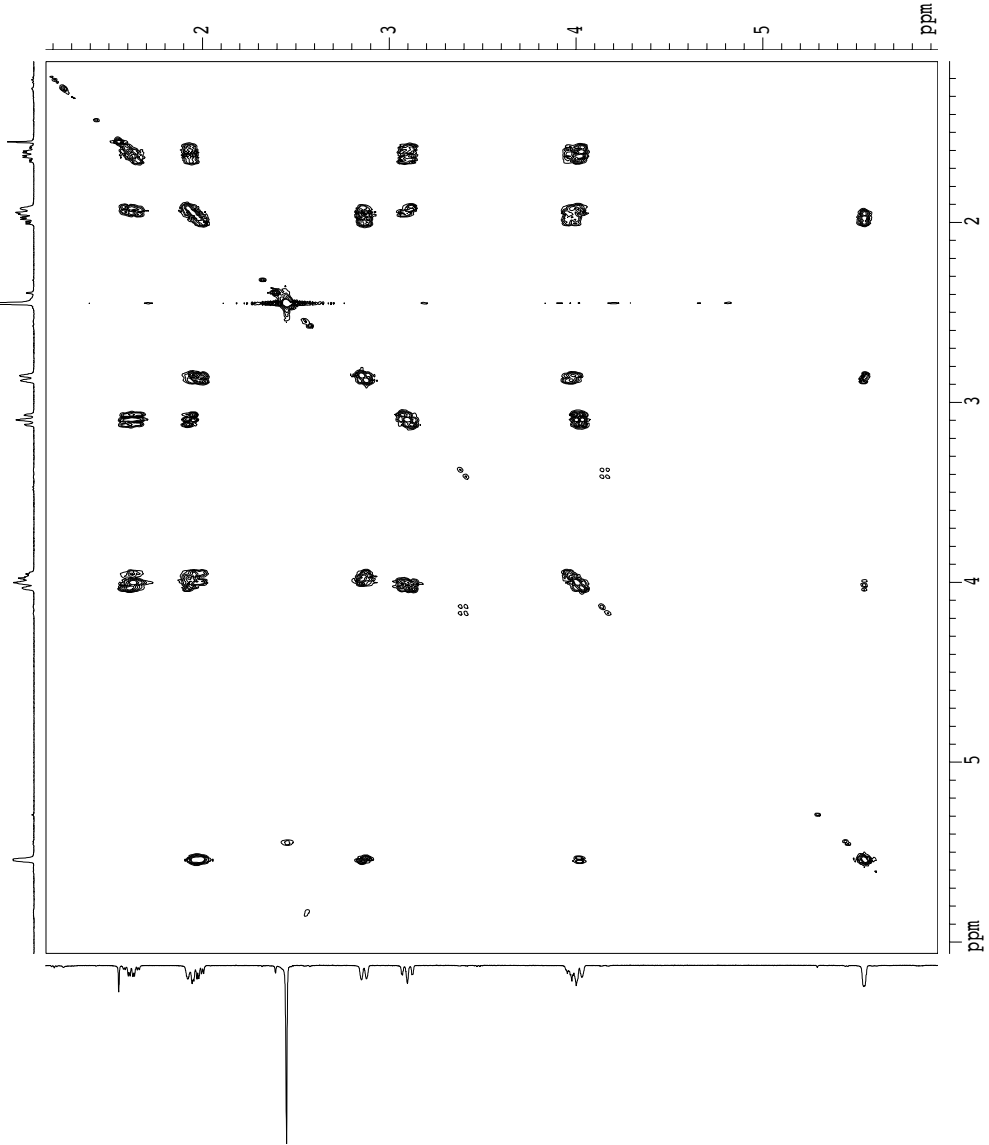
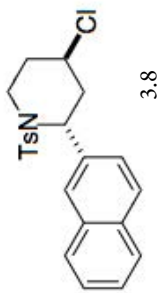
F2 - Acquisition Parameters
Date_    20171108
Time     20.22
INSTRUM  gpc500
PROBHD   5 mm broadband
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        788
DS        4
AQ        30303.031 Hz
F1RES    0.462388 Hz
AQ        1.0813940 sec
RG        3251
DE        16.470 usec
TE        298.2 K
D1        0.25000000 sec
d11       0.02000000 sec
d12       0.02000000 sec
d13       0.02000000 sec
d14       0.02000000 sec
d15       0.02000000 sec
d16       0.02000000 sec
d17       0.02000000 sec
d18       0.02000000 sec
d19       0.02000000 sec
d20       0.02000000 sec
===== CHANNEL f1 =====
NUC1      13C
P1        9.46 usec
PL1       -1.50 dB
SFO1     125.4748777 MHz

===== CHANNEL f2 =====
NUC2      13C
P2        9.46 usec
PL2       -1.50 dB
SFO2     125.4748777 MHz

===== Processing parameters =====
SI        65536
SF        125.4610662 MHz
WDW       no
SSB       no
GB        0
PC        2.00

ID:IMP Plot parameters
CX        22.80 cm
CY        15.45 cm
FLP       200.000 ppm
FIDRES    25072.724 Hz
F2P       0.000 ppm
F3P       0.000 ppm
PPMCK     8.77193 ppm/cm
RECN      1100.53577 Hz/cm
    
```

gcosy60



```
Current Data Parameters
=====
USER          erical
NAME          ELL-II-281
EXPNO         3
PROCNO        1

Date_         20111108
Time          19:53
INSTRUM       gn500
PROBHD        5 mm broadband
PULPROG       cosypp0-pfd
AQ            0.00100000
SOLVENT       CDCl3
NS            2
DS            16
SWH           3594.954 Hz
F2RES        1.753884 Hz
AQ           0.48531880 sec
RG            328.423
WDW           139.200 usec
DE            6.00 usec
TE            298.0 K
d0            0.0000300 sec
d1            1.40000000 sec
d13           0.00000000 sec
d16           0.00025000 sec
d10           0.00027840 sec

===== CHANNEL f1 =====
NUC1          1H
P1            12.00 usec
PL1           -5.80 dB
SFO1          498.9523665 MHz

===== GRADIENT CHANNEL =====
GPROG         SINE.100
GSPRG         SINE.100
GCX2          0.00 %
GCY2          0.00 %
GP11          0.00 %
GP12          0.00 %
GP21          17.00 %
GP22          17.00 %
P16           1000.00 usec

F1 - Acquisition parameters
=====
NUC0          1
TD            256
SFO0          498.9524 MHz
SFRES        14.00000000 MHz
SW           71.99 PPM
FREQ0        7.199 QF

F2 - Processing parameters
=====
SI            1024
WDW           EM
SSB           0
LB            0.00 Hz
GB            0
PC            1.00

F1 - Processing parameters
=====
SI            1024
WDW           EM
SSB           0
LB            0.00 Hz
GB            0
PC            1.00

ZD NMR plot parameters
=====
CZ2           15.00 cm
CZ1           15.00 cm
CZ0           0.00 cm
EZL0          3084.77 Hz
EZFHI         1.106 ppm
EZFLO         551.79 Hz
EZF0          5.936 ppm
FZLO          2981.63 Hz
FZHI          579.85 Hz
FZ0           1.106 ppm
EZFPPMCH     0.33042 ppm/cm
FZPPMCH      164.86508 Hz/cm
EZFPPMCH     0.31824 ppm/cm
FZPPMCH      158.78494 Hz/cm
```

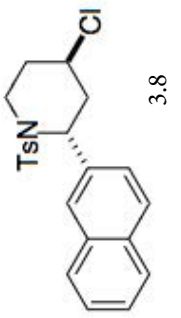
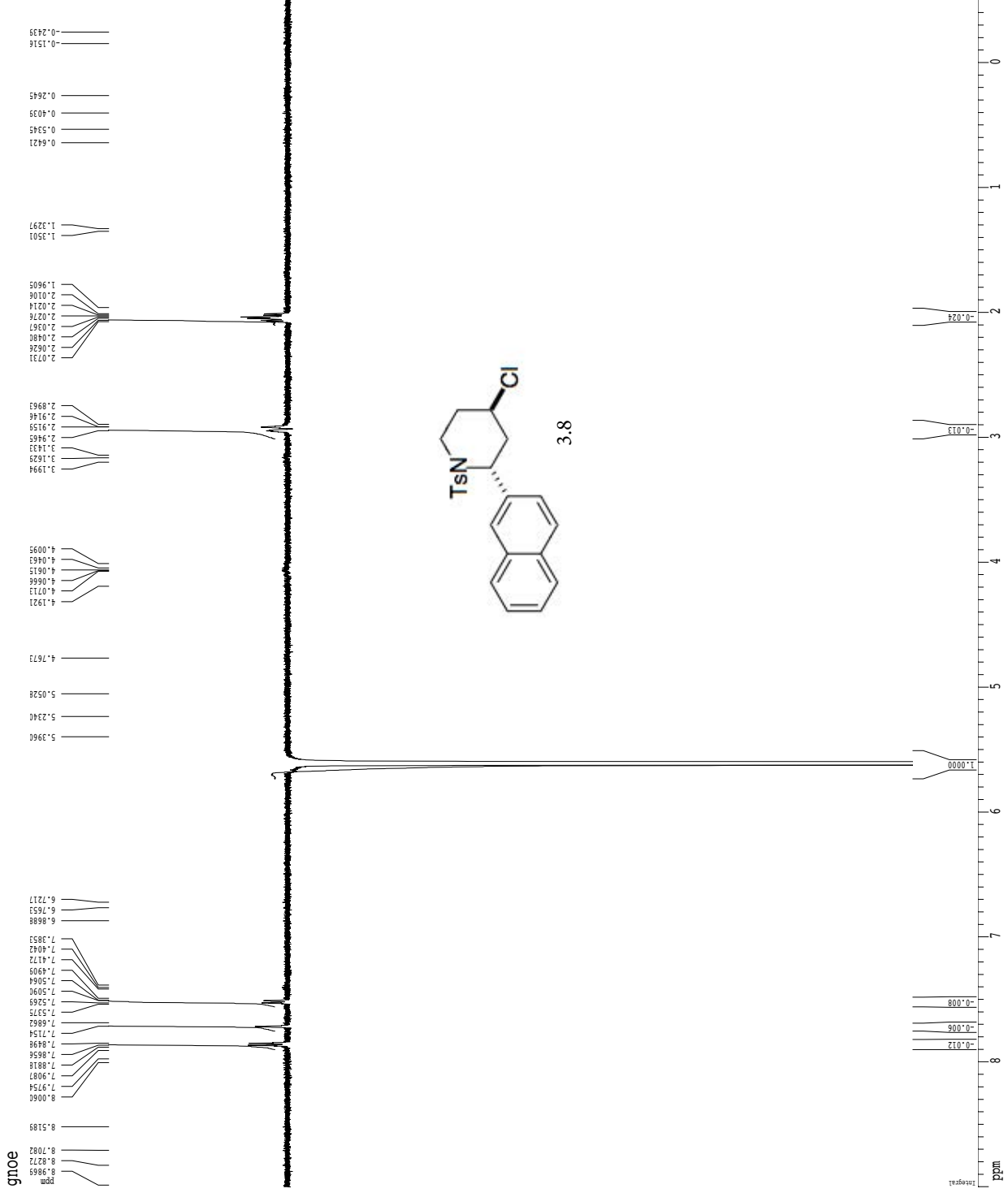
Current Data Parameters  
 USER: actba  
 EXPRN: EHL-11-281  
 PROCNO: 1  
 P2 - Acquisition Parameters  
 Date\_: 20171108  
 Time: 20.50  
 INSTRUM: gpc500  
 PROBHD: 5 mm BBO-1H  
 PULPROG: gpcaccs  
 TD: 65536  
 SOLVENT: CCl3  
 NS: 256  
 SH: 8012.828 Hz  
 FIDRES: 0.122268 Hz  
 AQ: 4.089496 sec  
 RG: 5160.6  
 DE: 62.00 usec  
 TE: 298.2 K  
 D1: 1.0000000 sec  
 D2: 0.5000000 sec  
 D3: 0.3333333 sec  
 DZ: 0.1639969 sec  
 P2: 24.00 usec

==== CHANNEL f1 ====  
 NUC1: 1H  
 P1: 12.00 usec  
 P3: 36.00 usec  
 P4: 48.00 usec  
 P7: 7.00 usec  
 P12: 4000.00 usec  
 P11: -5.88 dB  
 SFO1: 498.527981 MHz  
 SF1: 51.60 dB  
 GPCPM1: gauss1.51  
 SPCP1: 0.00 Hz

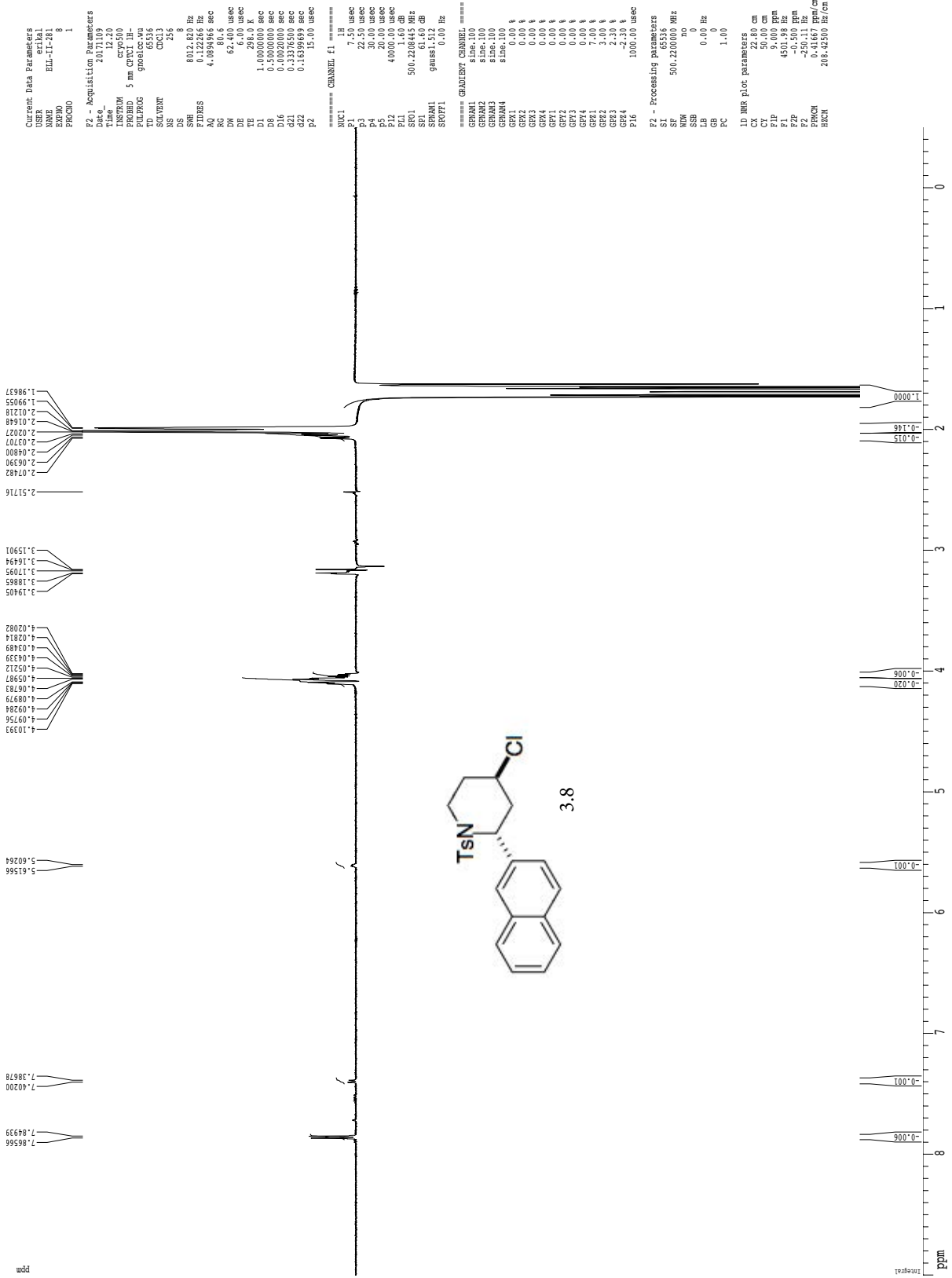
==== GRADIENT CHANNEL ====  
 GRNM1: sine.100  
 GRNV1: sine.100  
 GRNA2: sine.100  
 GRNM4: sine.100  
 GPCX1: 0.00 %  
 GPCX2: 0.00 %  
 GPCX3: 0.00 %  
 GPCX4: 0.00 %  
 GPCX5: 0.00 %  
 GPCX6: 0.00 %  
 GPCX7: 0.00 %  
 GPCX8: 0.00 %  
 GPCX9: 0.00 %  
 GPCX10: 0.00 %  
 GPCX11: 0.00 %  
 GPCX12: 0.00 %  
 GPCX13: 0.00 %  
 GPCX14: 0.00 %  
 GPCX15: 0.00 %  
 GPCX16: 0.00 %  
 P16: 1000.00 usec

P2 - Processing parameters  
 S1: 65536  
 SF: 498.5000000 MHz  
 NS: no  
 SD: no  
 LB: 0.00 Hz  
 GB: 0  
 PC: 1.00

ID: NMR Plot parameters  
 CX: 22.80 cm  
 CY: 50.00 cm  
 F1P: 9.000 ppm  
 F2: 400.618 Hz  
 F3: 101.253 MHz  
 FZ: -249.41 Hz  
 PPRMCK: 0.41667 ppm/cm  
 RECH: 207.85984 Hz/cm



gnoe



Current Data Parameters  
USER erikal  
NAME RLL-11-281  
EXPNO 8  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 2017109  
Time 12:20  
PROBHD 5 mm CPVT-1H  
PULPROG zgpg30c-vu  
TD 65536  
SOLVENT CDCl3  
NS 8  
DS 8  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894966 sec  
RG 62.400  
DE 6.000 usec  
TE 298.0 K  
D1 1.0000000 sec  
d11 0.0300000 sec  
D16 0.0002000 sec  
d21 0.3337500 sec  
d22 0.1639869 sec  
P2 15.00 usec

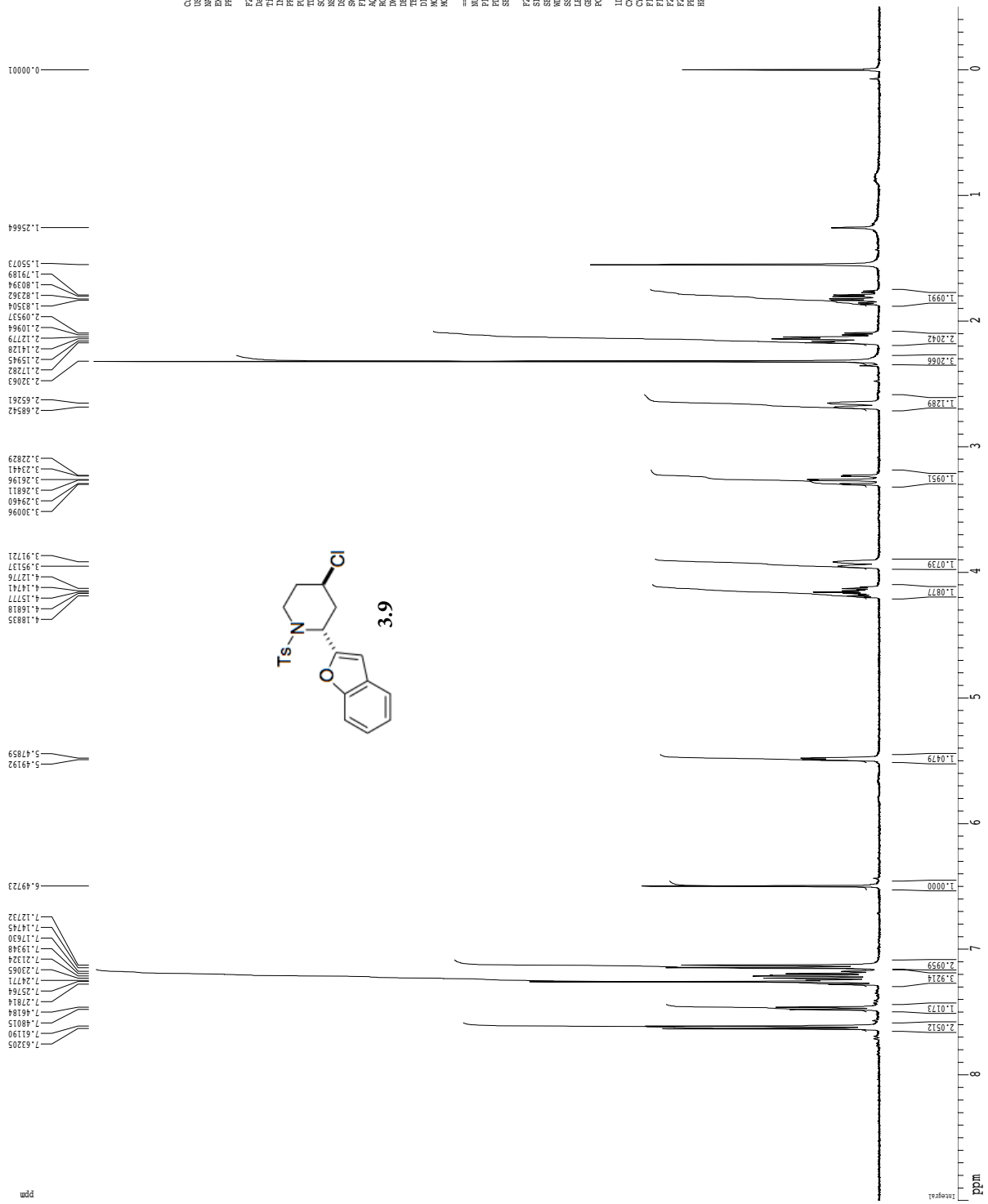
==== CHANNEL f1 =====  
NUC1 1H  
P1 7.50 usec  
P3 22.50 usec  
P4 20.00 usec  
P5 20.00 usec  
P12 40000.00 usec  
PL1 1.60 dB  
SFO1 500.2208445 MHz  
GPROB1 gauss-15  
SFOFF1 0.00 Hz

==== GRADIENT CHANNEL =====  
GUNIT1 sine,100  
GUNIT2 sine,100  
GUNIT3 sine,100  
GUNIT4 sine,100  
GGR1 0.00 V  
GGR2 0.00 V  
GGR3 0.00 V  
GGR4 0.00 V  
GFF1 0.00 V  
GFF2 0.00 V  
GFF3 0.00 V  
GFF4 0.00 V  
GFF5 0.00 V  
GFF6 7.00 V  
GFF7 3.00 V  
GFF8 2.30 V  
GFF9 2.30 V  
GFF10 1000.00 usec

F2 - Processing parameters  
SI 65536  
SF 500.2208445 MHz  
WDW 0  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

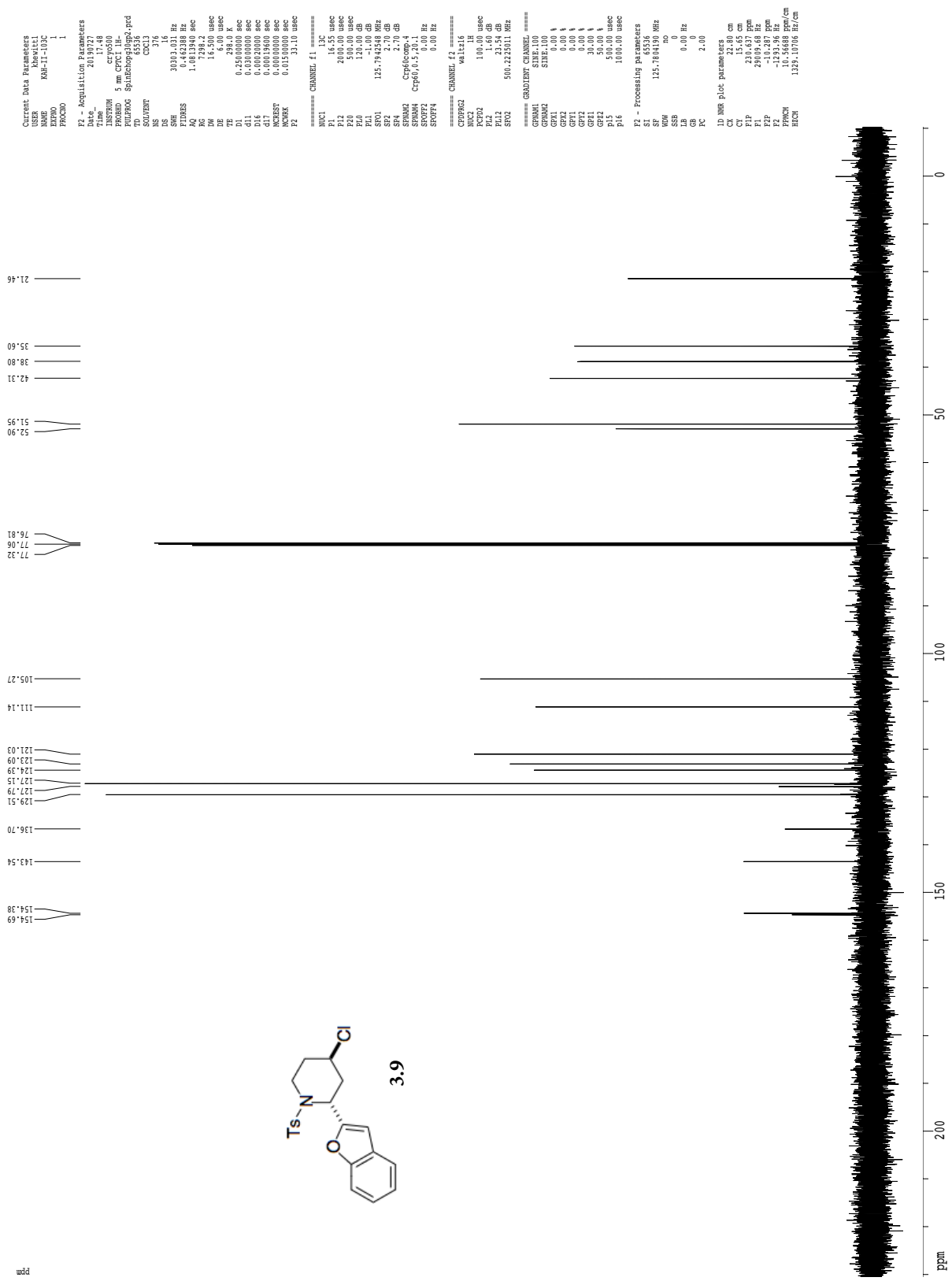
1D NMR Plot parameters  
CX 22.80 cm  
CY 50.00 cm  
CZ 45.00 cm  
F1 650.00 ppm  
F2 -0.500 ppm  
F3 -250.11 Hz  
PRGCM 0-41667 ppm/cm  
HCN 208-4250 Hz/cm

1H spectrum



Current Data Parameters  
 USER KAWA  
 NAME KAWA-018-4  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20180629  
 Time 14.43  
 Operator  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 NS 2  
 DS 0  
 SWH 6410.256 Hz  
 FIDRES 0.166672 Hz  
 AQ 2.9999239 sec  
 RG 384  
 DW 78.000 usec  
 DE 4.50 usec  
 TE 298.0 K  
 T1 0.10000000 sec  
 T2 0.10000000 sec  
 T3 0.10000000 sec  
 MCHRG 0.05500000 sec  
 ===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.132809 MHz  
 F2 - Processing Parameters  
 SI 65536  
 SF 400.130021 MHz  
 SD 0 Hz  
 SSB 0 Hz  
 GB 0 Hz  
 PC 2.00  
 ID NMR File Parameters  
 CF 22.80 cm  
 C1 15.00 cm  
 F1P 9.000 ppm  
 F1 36001.77 Hz  
 F2P -200.00 ppm  
 F2 -200.00 Hz  
 FWHM 0.41667 ppm/cm  
 HECW 166.72086 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



gc05y60

Current Data Parameters  
 USER khewitt1  
 NAME KAH-1-018  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 201809  
 Time 08:00  
 INSTRUM crys500  
 PROBHD 5 mm CPTCI 1H-  
 PULPROG cosygp60.prd  
 TD 2048  
 SOLVENT CDCl3  
 NS 2  
 DS 16  
 SWH 8012.820 Hz  
 FIDRES 3.91251 Hz  
 AQ 0.1278452 sec  
 RG 362  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.0 K  
 d0 0.00000300 sec  
 D1 1.00000000 sec  
 D11 0.00000000 sec  
 D15 0.00020000 sec  
 INU 0.0002488 sec

==== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.50 usec  
 PL1 1.60 dB  
 SFO1 500.225015 MHz

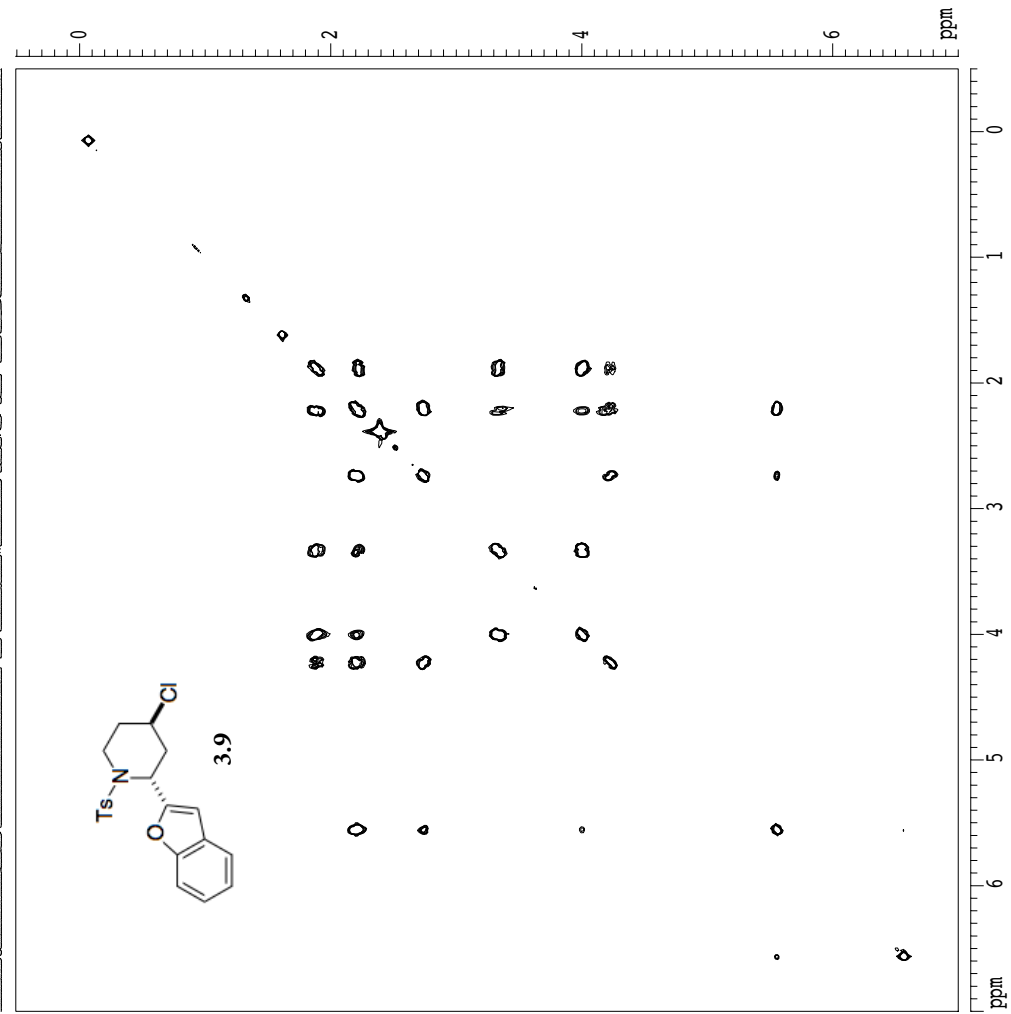
==== GRADIENT CHANNEL =====  
 GPRM1 size:100  
 GEX1 0.00 %  
 GEX2 0.00 %  
 GEV1 0.00 %  
 GEV2 0.00 %  
 GEZ1 17.00 %  
 GEZ2 17.00 %  
 P16 1000.00 usec

F1 - Acquisition parameters  
 ND0 1  
 TD 512  
 SFO1 500.2235 MHz  
 FIDRES 15.650040 Hz  
 SW 16.018 ppm  
 FREQ0Z undefined

F2 - Processing parameters  
 SI 1024  
 SF 500.2200000 MHz  
 WDM 0  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

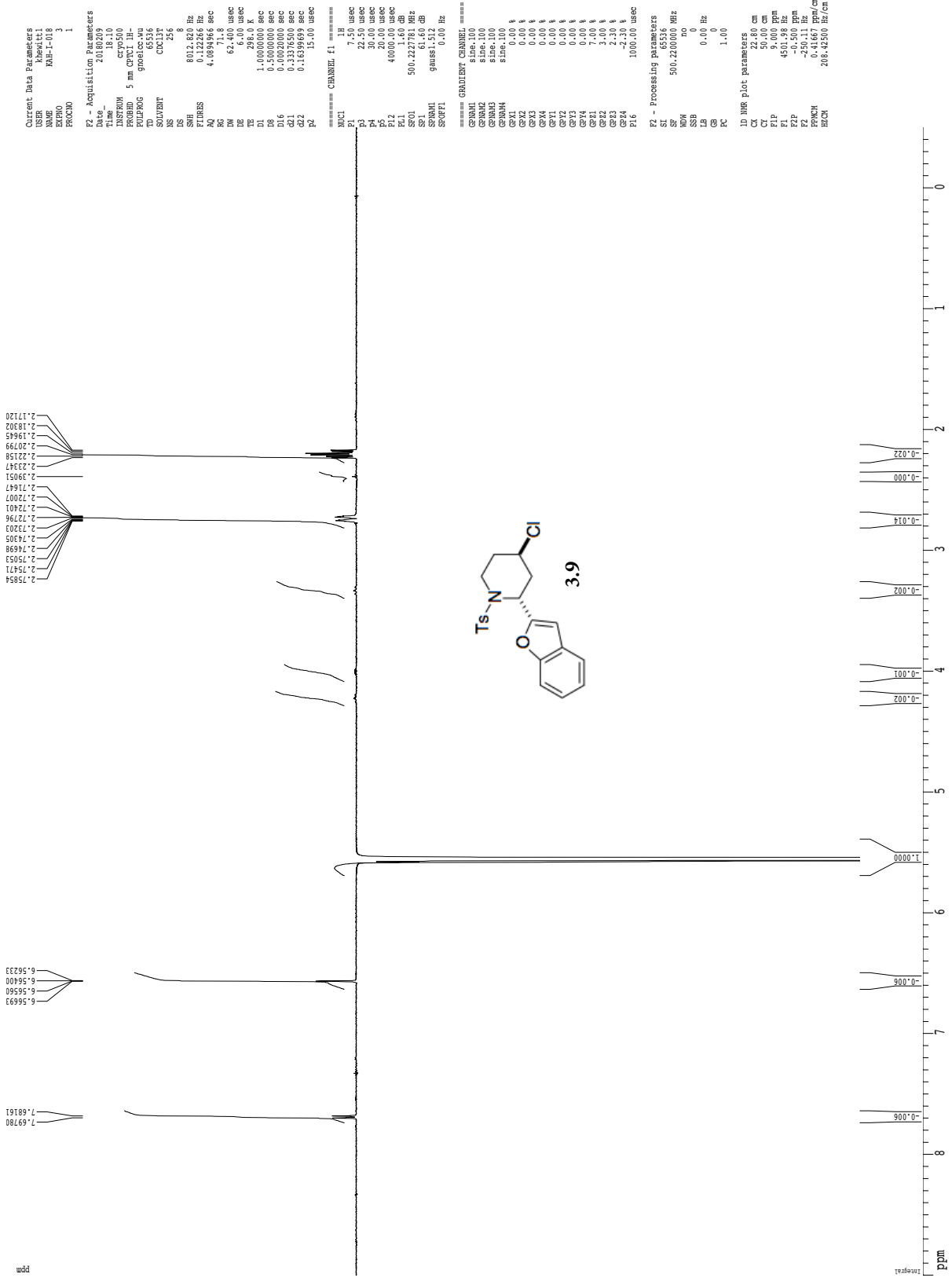
F1 - Processing parameters  
 SI 1024  
 MC2 QF  
 SF 500.2200000 MHz  
 WDM 0  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 7.000 ppm  
 FZLO 3501.54 Hz  
 FZHI -0.500 ppm  
 FZFI -2501.13 Hz  
 FZLO 3501.54 Hz  
 FZFI -2501.13 Hz  
 F1HI -254.47 Hz  
 F1FI -254.47 Hz  
 F2FPMCM 0.50000 ppm/cm  
 FZHECM 250.11000 Hz/cm  
 F1FPMCM 0.50005 ppm/cm  
 F1HECM 250.40063 Hz/cm



gnoe

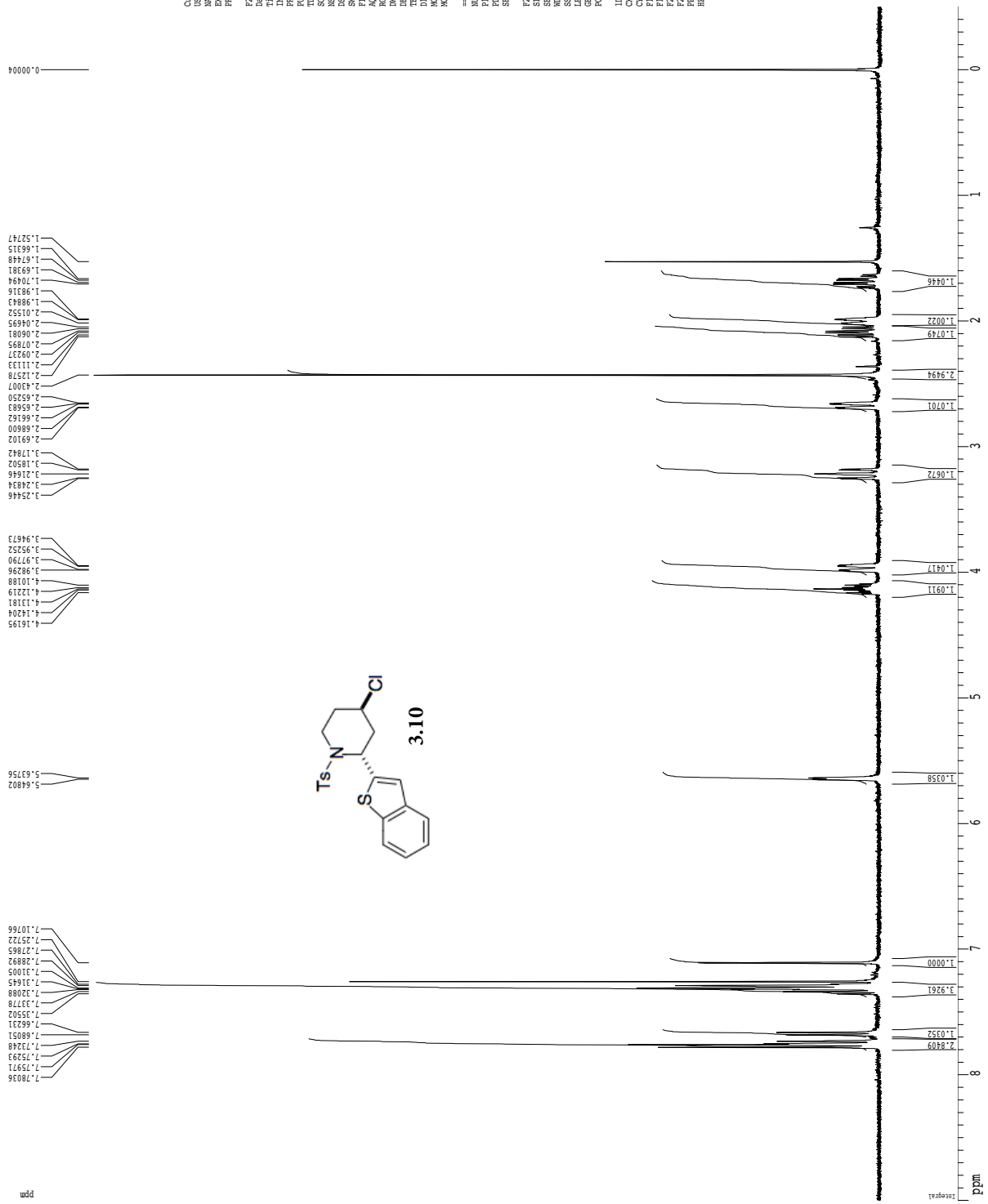
ppm







1H spectrum



Current Data Parameters  
 USER Rawsett1  
 NAME KM-1-057-4  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20180728  
 Time 16:38  
 Operator  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6410.256 Hz  
 FIDRES 0.166672 Hz  
 AQ 2.5959239 sec  
 RG 327.5  
 DW 78.000 usec  
 DE 4.50 usec  
 TE 298.0 K  
 D0 0.10000000 sec  
 MCREST 0.10000000 sec  
 MCXRF 0.03500000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.1328009 MHz

F2 - Processing Parameters  
 SI 65536  
 SF 400.1300225 MHz  
 SD 0.00000000 sec  
 SSB 0.00 Hz  
 GB 0  
 PC 2.00

ID: NMR FIDIC Parameters  
 CQ 22.80 cm  
 CZ 15.00 cm  
 F1P 9.000 ppm  
 F2P 500.137 Hz  
 F3P 200.000 ppm  
 FPMCN 0.41667 ppm/cm  
 RECN 166.72086 Hz/cm



gcossy60

Current Data Parameters  
 USER khevitt1  
 NAME KAH-1-057-2  
 EXPNO 3  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 201008  
 Time 13:00  
 INSTRUM cryo500  
 PROBHD 5 mm CPTCI 1H  
 PULPROG cosygp60.prd  
 TD 2048  
 SOLVENT CDCl3  
 NS 1  
 DS 16  
 SWH 8012.820 Hz  
 FIDRES 3.91251 Hz  
 AQ 0.1278452 sec  
 RG 724.1  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.0 K  
 d0 0.00000300 sec  
 D1 1.00000000 sec  
 D11 0.00000000 sec  
 D15 0.00020000 sec  
 INU 0.0002488 sec

==== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.50 usec  
 PL1 1.60 dB  
 SFO1 500.225015 MHz

==== GRADIENT CHANNEL =====  
 GPRM1 size:100  
 GEX1 0.00 %  
 GEY1 0.00 %  
 GEZ1 0.00 %  
 GEX2 17.00 %  
 GEY2 17.00 %  
 GEZ2 100.00 usec

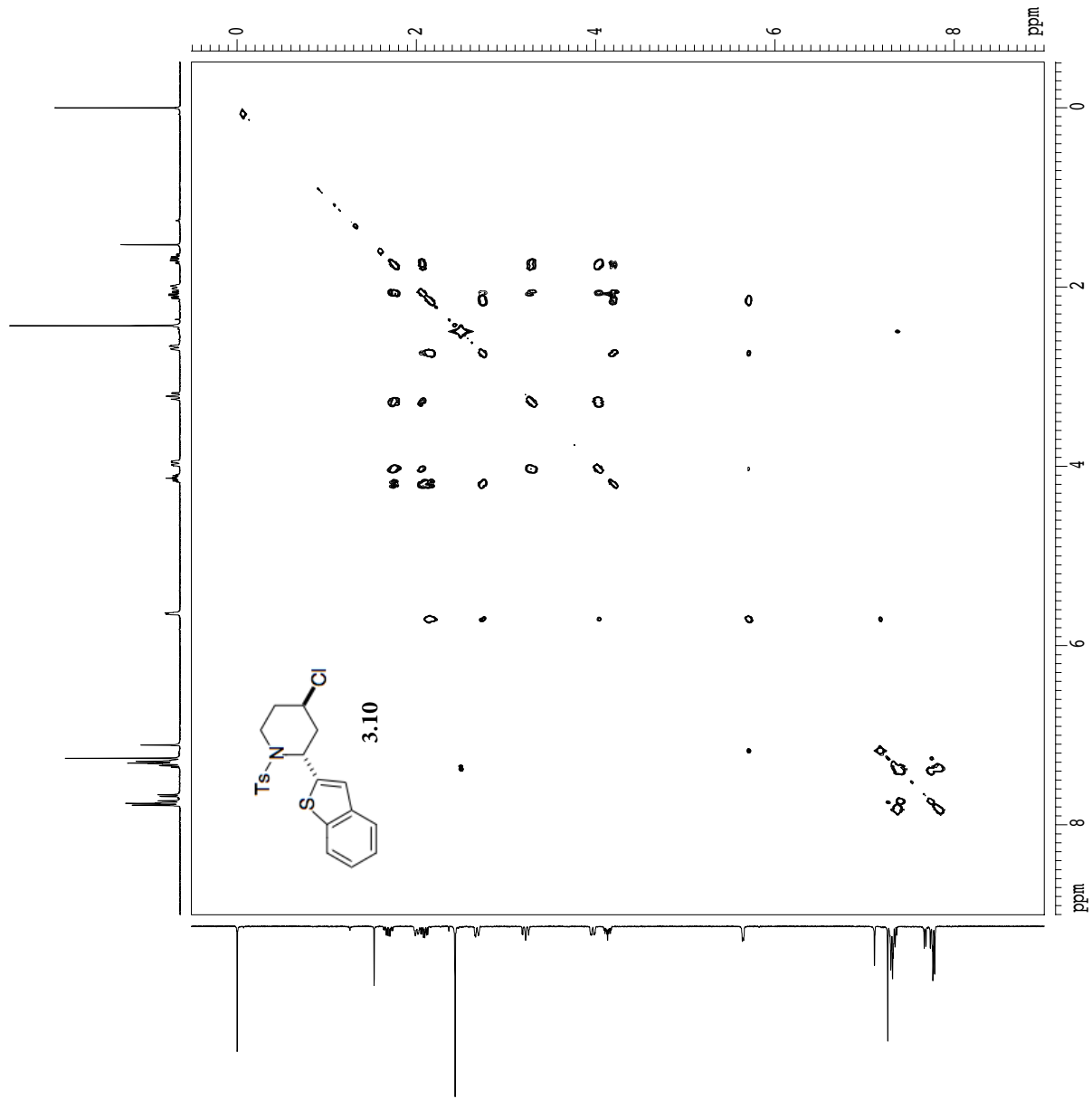
F1 - Acquisition parameters  
 ND0 1  
 TD 512  
 SFO1 500.2235 MHz  
 FIDRES 15.650040 Hz  
 SW 16.018 Ppm  
 FREQ0E QF

F2 - Processing parameters  
 SI 1024  
 SF 500.2200000 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 1024  
 MC2 QF  
 SF 500.2200000 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

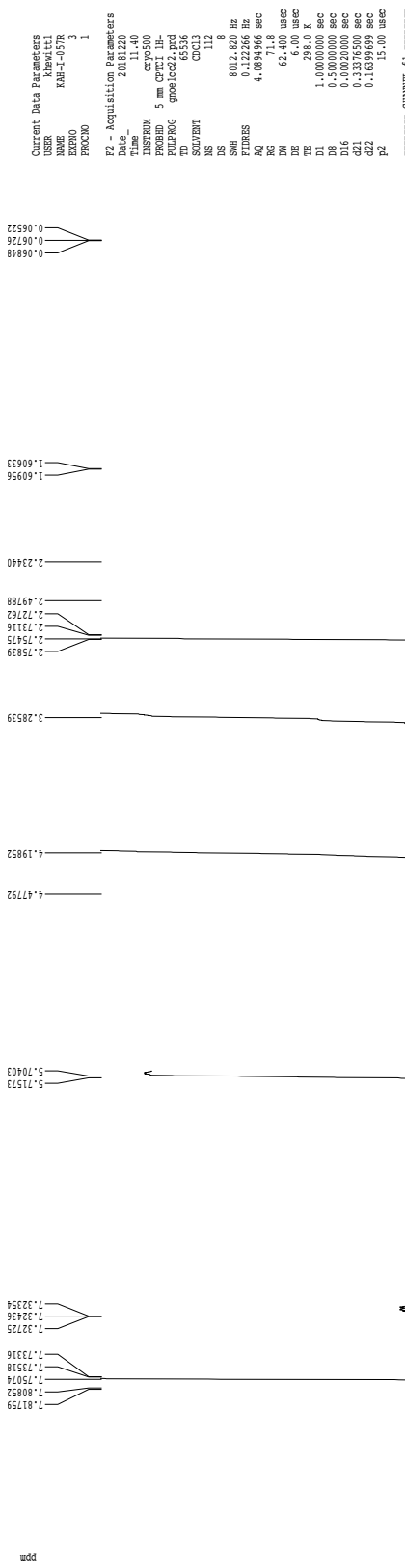
2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 9.002 Ppm  
 FZLO 4502.14 Hz  
 FZPHI -0.509 Ppm  
 FZHI -254.47 Hz  
 FZLO 4502.14 Hz  
 FZPHI -0.509 Ppm  
 FZHI -254.47 Hz

F2PFCM 0.63407 ppm/cm  
 FZPFCM 317.17416 Hz/cm  
 FIPFCM 0.63407 ppm/cm  
 FIIPFCM 317.17416 Hz/cm

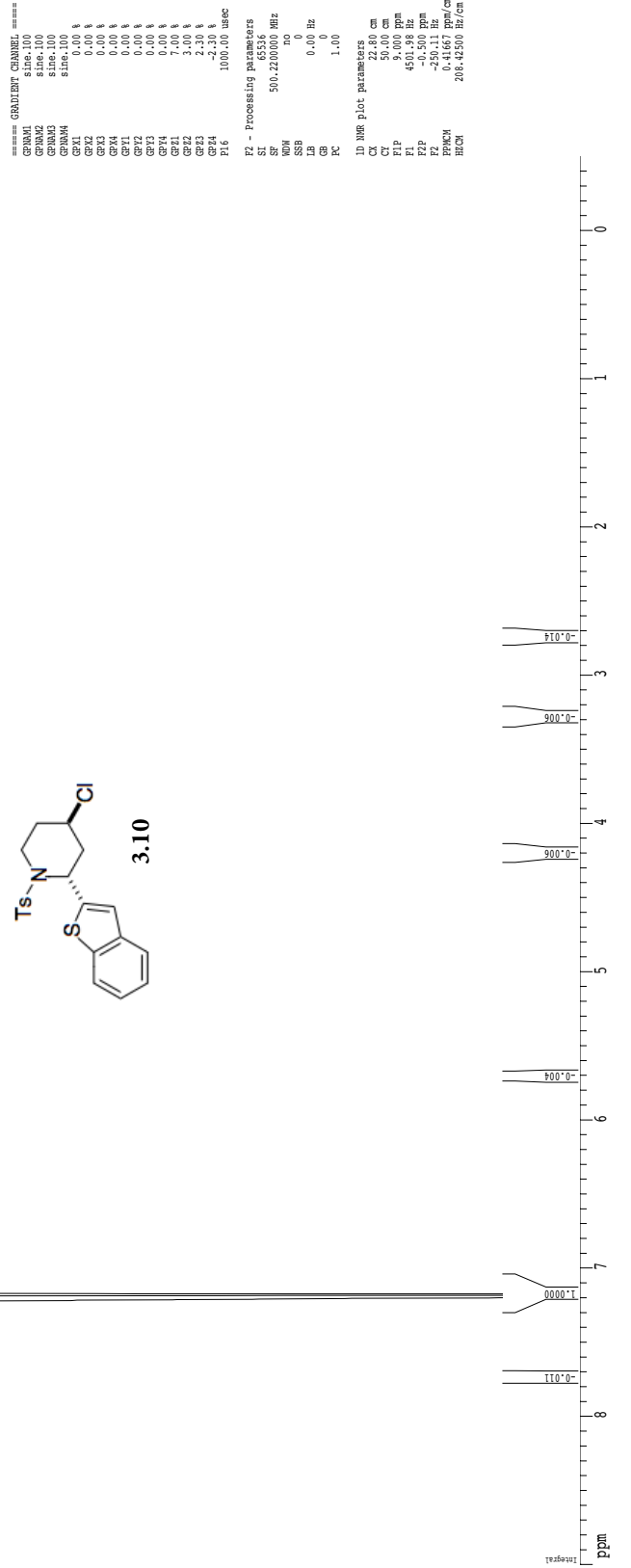
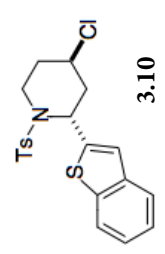




gnoe

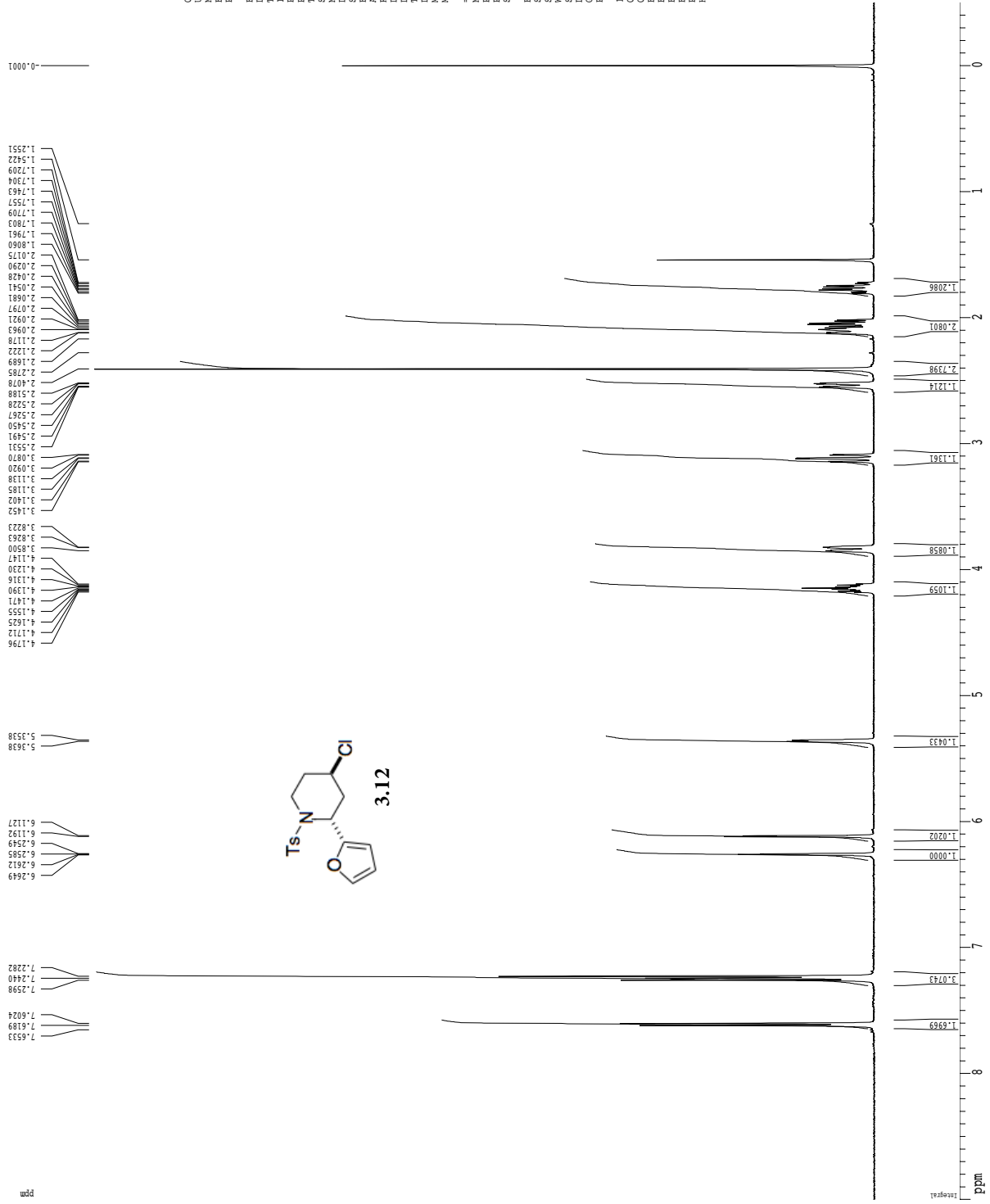


```
==== CHANNEL f1 =====
NUC1 1H
P1 7.50 usec
P2 22.50 usec
P3 20.00 usec
P4 20.00 usec
P5 20.00 usec
P6 20.00 usec
P7 20.00 usec
P8 20.00 usec
P9 20.00 usec
P10 20.00 usec
P11 20.00 usec
P12 20.00 usec
P13 20.00 usec
P14 20.00 usec
P15 20.00 usec
P16 20.00 usec
P17 20.00 usec
P18 20.00 usec
P19 20.00 usec
P20 20.00 usec
P21 20.00 usec
P22 20.00 usec
P23 20.00 usec
P24 20.00 usec
P25 20.00 usec
P26 20.00 usec
P27 20.00 usec
P28 20.00 usec
P29 20.00 usec
P30 20.00 usec
P31 20.00 usec
P32 20.00 usec
P33 20.00 usec
P34 20.00 usec
P35 20.00 usec
P36 20.00 usec
P37 20.00 usec
P38 20.00 usec
P39 20.00 usec
P40 20.00 usec
P41 20.00 usec
P42 20.00 usec
P43 20.00 usec
P44 20.00 usec
P45 20.00 usec
P46 20.00 usec
P47 20.00 usec
P48 20.00 usec
P49 20.00 usec
P50 20.00 usec
P51 20.00 usec
P52 20.00 usec
P53 20.00 usec
P54 20.00 usec
P55 20.00 usec
P56 20.00 usec
P57 20.00 usec
P58 20.00 usec
P59 20.00 usec
P60 20.00 usec
P61 20.00 usec
P62 20.00 usec
P63 20.00 usec
P64 20.00 usec
P65 20.00 usec
P66 20.00 usec
P67 20.00 usec
P68 20.00 usec
P69 20.00 usec
P70 20.00 usec
P71 20.00 usec
P72 20.00 usec
P73 20.00 usec
P74 20.00 usec
P75 20.00 usec
P76 20.00 usec
P77 20.00 usec
P78 20.00 usec
P79 20.00 usec
P80 20.00 usec
P81 20.00 usec
P82 20.00 usec
P83 20.00 usec
P84 20.00 usec
P85 20.00 usec
P86 20.00 usec
P87 20.00 usec
P88 20.00 usec
P89 20.00 usec
P90 20.00 usec
P91 20.00 usec
P92 20.00 usec
P93 20.00 usec
P94 20.00 usec
P95 20.00 usec
P96 20.00 usec
P97 20.00 usec
P98 20.00 usec
P99 20.00 usec
P100 20.00 usec
=====
```



```
==== GRABDET CHANNEL =====
GRAB1 1H
GRAB2 13C
GRAB3 13C
GRAB4 13C
GRAB5 13C
GRAB6 13C
GRAB7 13C
GRAB8 13C
GRAB9 13C
GRAB10 13C
GRAB11 13C
GRAB12 13C
GRAB13 13C
GRAB14 13C
GRAB15 13C
GRAB16 13C
GRAB17 13C
GRAB18 13C
GRAB19 13C
GRAB20 13C
GRAB21 13C
GRAB22 13C
GRAB23 13C
GRAB24 13C
GRAB25 13C
GRAB26 13C
GRAB27 13C
GRAB28 13C
GRAB29 13C
GRAB30 13C
GRAB31 13C
GRAB32 13C
GRAB33 13C
GRAB34 13C
GRAB35 13C
GRAB36 13C
GRAB37 13C
GRAB38 13C
GRAB39 13C
GRAB40 13C
GRAB41 13C
GRAB42 13C
GRAB43 13C
GRAB44 13C
GRAB45 13C
GRAB46 13C
GRAB47 13C
GRAB48 13C
GRAB49 13C
GRAB50 13C
GRAB51 13C
GRAB52 13C
GRAB53 13C
GRAB54 13C
GRAB55 13C
GRAB56 13C
GRAB57 13C
GRAB58 13C
GRAB59 13C
GRAB60 13C
GRAB61 13C
GRAB62 13C
GRAB63 13C
GRAB64 13C
GRAB65 13C
GRAB66 13C
GRAB67 13C
GRAB68 13C
GRAB69 13C
GRAB70 13C
GRAB71 13C
GRAB72 13C
GRAB73 13C
GRAB74 13C
GRAB75 13C
GRAB76 13C
GRAB77 13C
GRAB78 13C
GRAB79 13C
GRAB80 13C
GRAB81 13C
GRAB82 13C
GRAB83 13C
GRAB84 13C
GRAB85 13C
GRAB86 13C
GRAB87 13C
GRAB88 13C
GRAB89 13C
GRAB90 13C
GRAB91 13C
GRAB92 13C
GRAB93 13C
GRAB94 13C
GRAB95 13C
GRAB96 13C
GRAB97 13C
GRAB98 13C
GRAB99 13C
GRAB100 13C
=====
```

1H spectrum



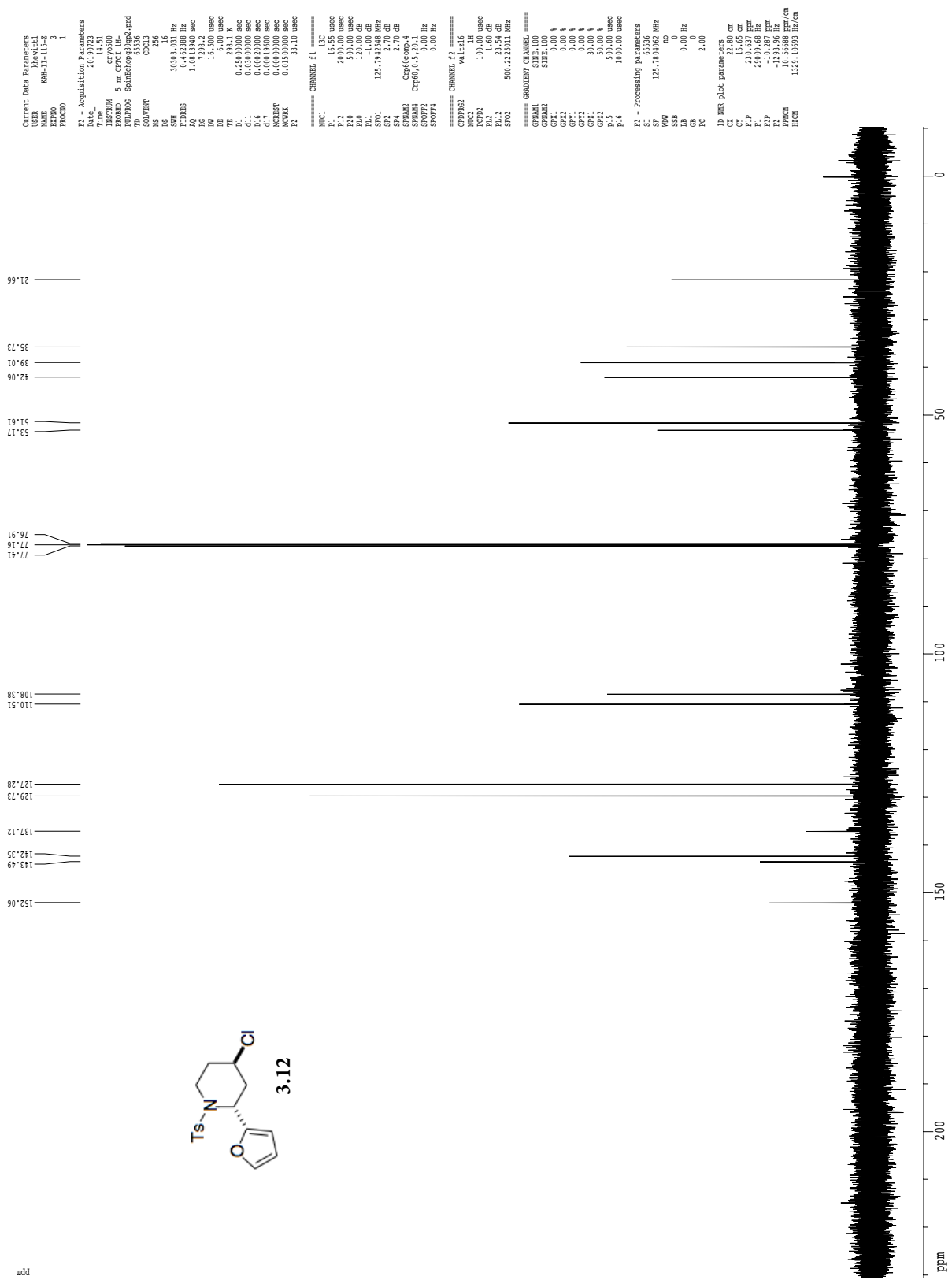
Current Data Parameters  
 USER Rawdata11  
 NAME KMF-11-5-4  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20190723  
 Time 14:34  
 Operator c...  
 PULPROG 5 mm CPMAS 1H  
 SFO1 500.235015 MHz  
 TD 65536  
 FIDRES 0.166677 Hz  
 AQ 2.5998677 sec  
 RG 62.400 us/ac  
 DE 6.400 us/ac  
 TE 298.2 K  
 F2 - Processing Parameters  
 SI 500.235015 MHz  
 SF 65536  
 NDM no  
 NSB 0 Hz  
 GB 0  
 PC 1.00

1D NMR file parameters  
 CF 22.80 cm  
 C1 15.00 cm  
 F1P 9.000 ppm  
 F1 4501.98 Hz  
 F2P 250.11 Hz  
 F2 4501.98 Hz  
 PPM0 0.41667 ppm/cm  
 RECH 208.45502 Hz/cm

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 7.50 us/ac  
 PL1 1.60 dB  
 SFO1 500.235015 MHz

Z-restored spin-echo <sup>13</sup>C spectrum with <sup>1</sup>H decoupling





gcossy60

Current Data Parameters  
USER khewitt1  
NAME KAH-1-134-2  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters

Date\_ 20100515  
Time 18:22  
INSTRUM crys500  
PROBHD 5 mm CPTCI 1H  
PULPROG cosygp60.prd  
TD 2048  
SOLVENT CDCl3  
NS 1  
DS 16  
SWH 8012.820 Hz  
FIDRES 3.91251 Hz  
AQ 0.1278452 sec  
RG 406.4  
DW 62.400 usec  
DE 6.00 usec  
TE 298.0 K  
d0 0.00000300 sec  
D1 1.00000000 sec  
D11 0.00000000 sec  
D15 0.00020000 sec  
D16 0.00020000 sec  
IN0 0.0002488 sec

==== CHANNEL f1 =====

NUC1 1H  
P1 7.50 usec  
PL1 1.60 dB  
SFO1 500.225015 MHz

==== GRADIENT CHANNEL =====

GPRAM1 size:100  
GEX1 0.00 %  
GEX2 0.00 %  
GEV1 0.00 %  
GEV2 0.00 %  
GZ1 17.00 %  
GZ2 17.00 %  
P16 1000.00 usec

F1 - Acquisition parameters

ND0 1  
TD 512  
SFO1 500.2235 MHz  
FIDRES 15.650040 Hz  
SW 16.018 Ppm  
FROUSE QF

F2 - Processing parameters

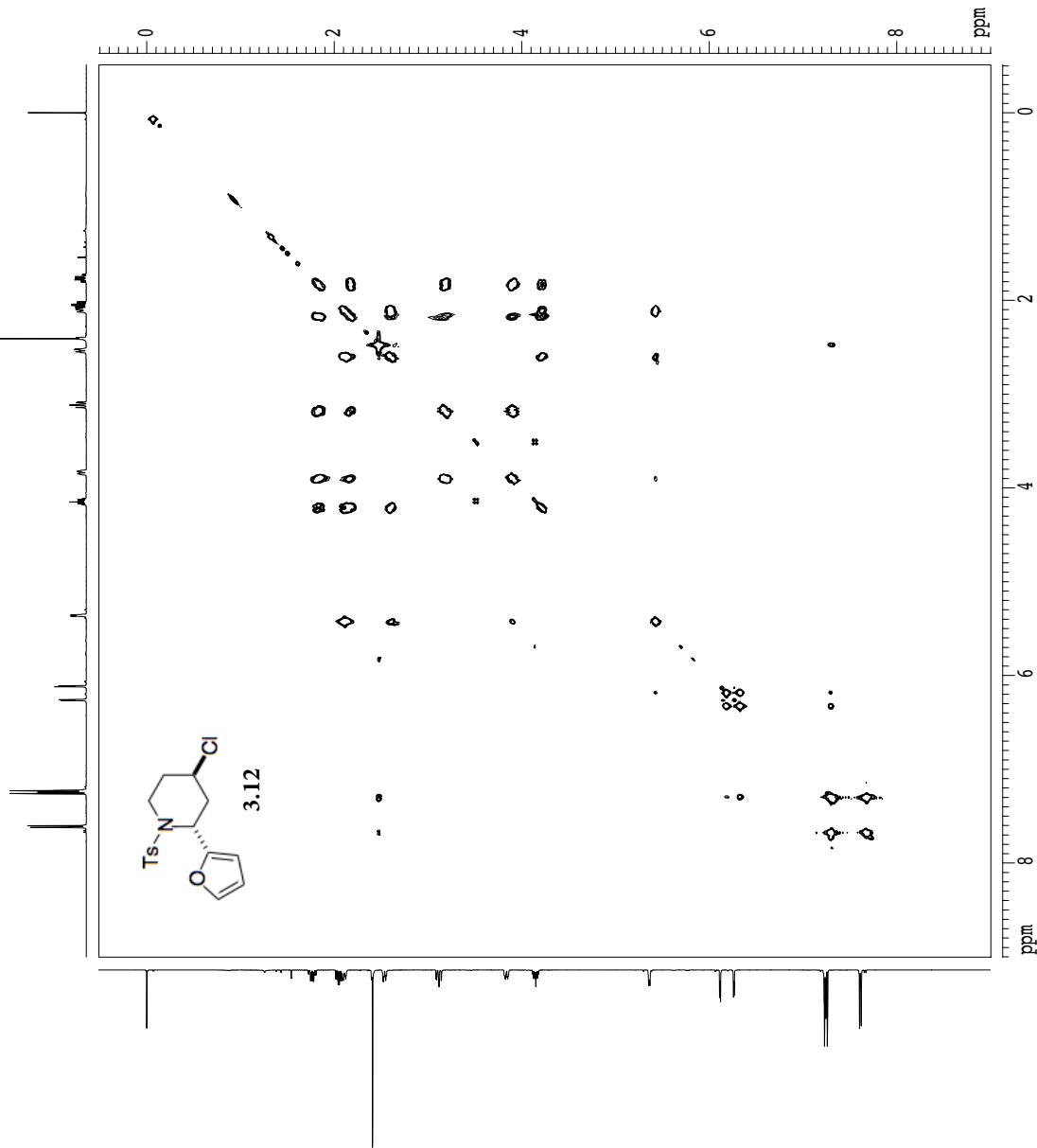
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SF 500.2200000 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

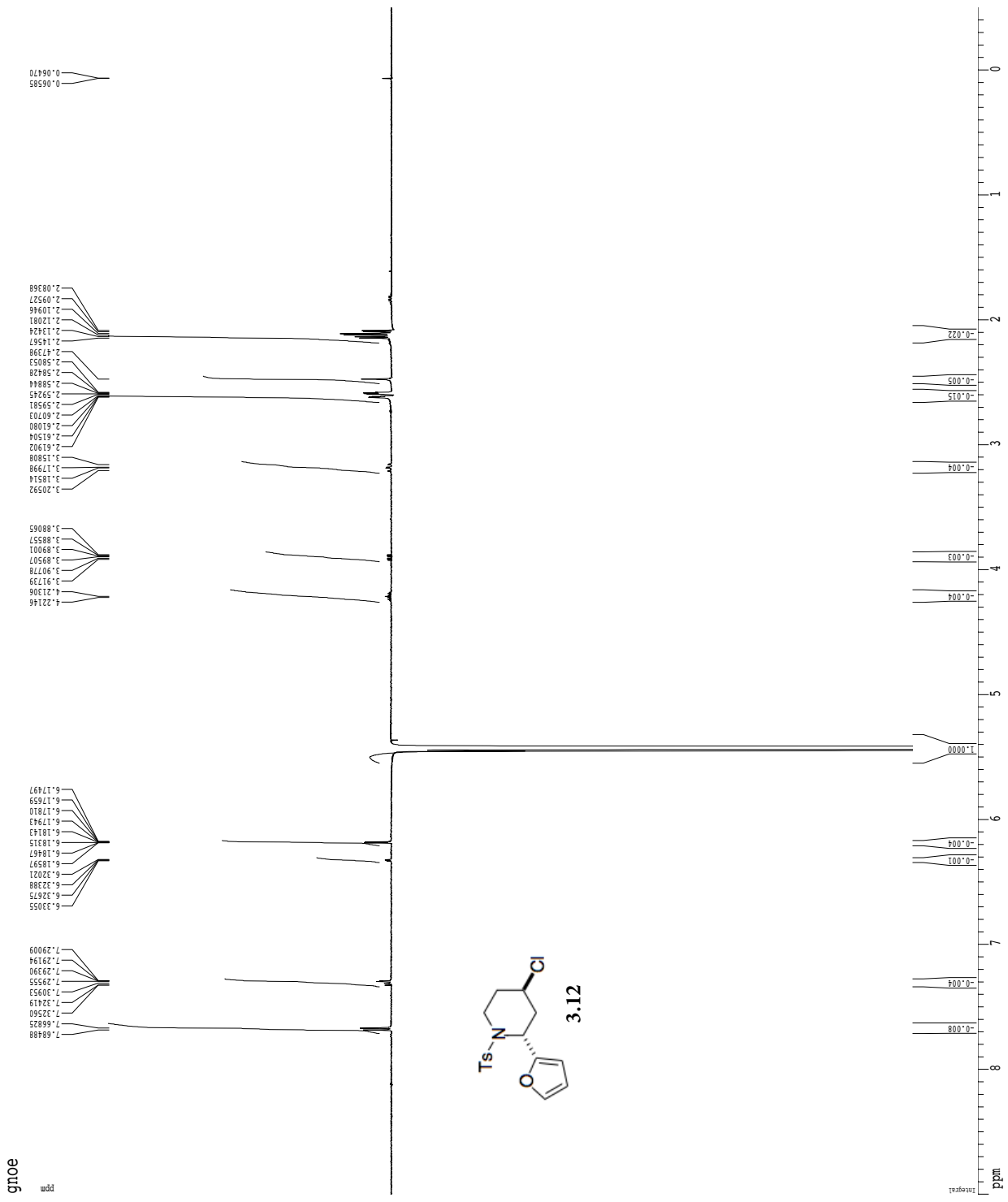
F1 - Processing parameters

SI 1024  
MC2 QF  
SF 500.2200000 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0

2D NMR plot parameters

CX2 15.00 cm  
CX1 15.00 cm  
F2PLO 9.002 Ppm  
FZLO 4502.14 Hz  
FZPHI -0.509 Ppm  
FZHI -254.47 Hz  
FZLO 4502.14 Ppm  
FZHI -0.509 Ppm  
F1PHI -254.47 Hz  
F1HI -254.47 Hz  
F2FPMCM 0.63407 Ppm/cm  
F2HPCM 317.17416 Hz/cm  
F1FPMCM 0.63407 Ppm/cm  
F1HPCM 317.17416 Hz/cm





gnoe

ppm

1.54109  
1.74331  
1.75211  
1.80776  
1.82012  
1.84538  
2.08171  
2.09298  
2.10417  
2.10841  
2.11402  
2.11992  
2.12842  
2.14115  
2.15566  
2.15973  
2.16998  
2.17591  
2.18098  
2.18585  
2.46294  
2.58254  
2.58623  
2.59018  
2.59401  
2.60839  
2.61650  
2.62072  
2.62711  
3.15911  
3.15931  
3.17676  
3.18238  
3.20556  
3.20867

6.17614  
6.18254  
6.88172  
6.88228  
7.28973

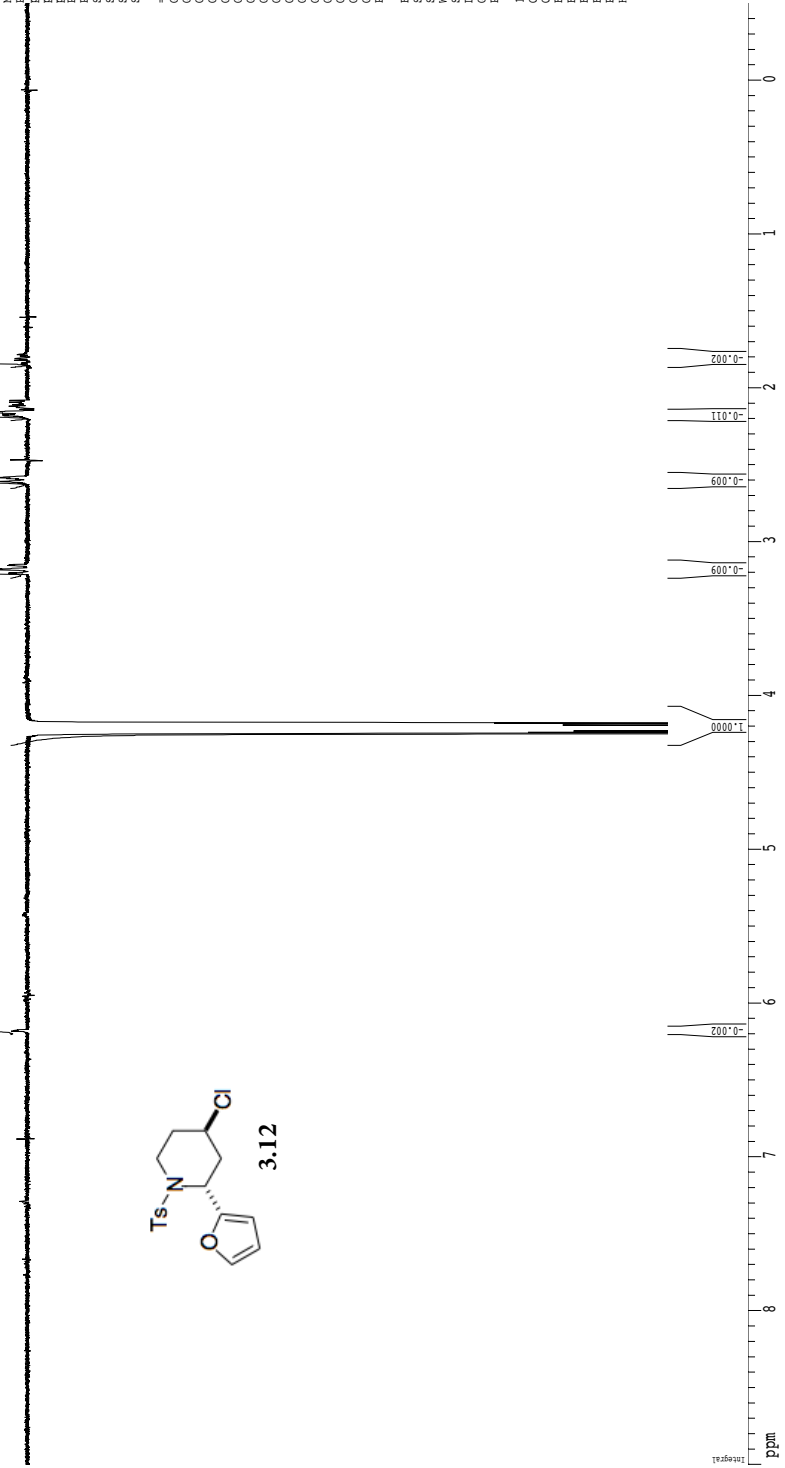
Current Data Parameters  
USER: khwatt1  
NAME: RM-1-115-2  
EXPNO: 2  
PROCNO: 1

F2 - Acquisition Parameters  
Date\_: 20190723  
Time: 14.49  
PROBHD: 5 mm CPCT 1H  
PULPROG: zgpg3022.prd  
TD: 65536  
SOLVENT: CDCl3  
NS: 128  
DS: 8  
SWH: 8012.820 Hz  
FIDRES: 0.122266 Hz  
AQ: 4.0894966 sec  
RG: 62.400  
DM: 6.000 usec  
DE: 4.000 usec  
TE: 298.2 K  
D1: 1.0000000 sec  
d11: 0.1000000 sec  
D16: 0.0000000 sec  
dL1: 0.3337500 sec  
dL2: 0.1639869 sec  
dL3: 15.000 usec

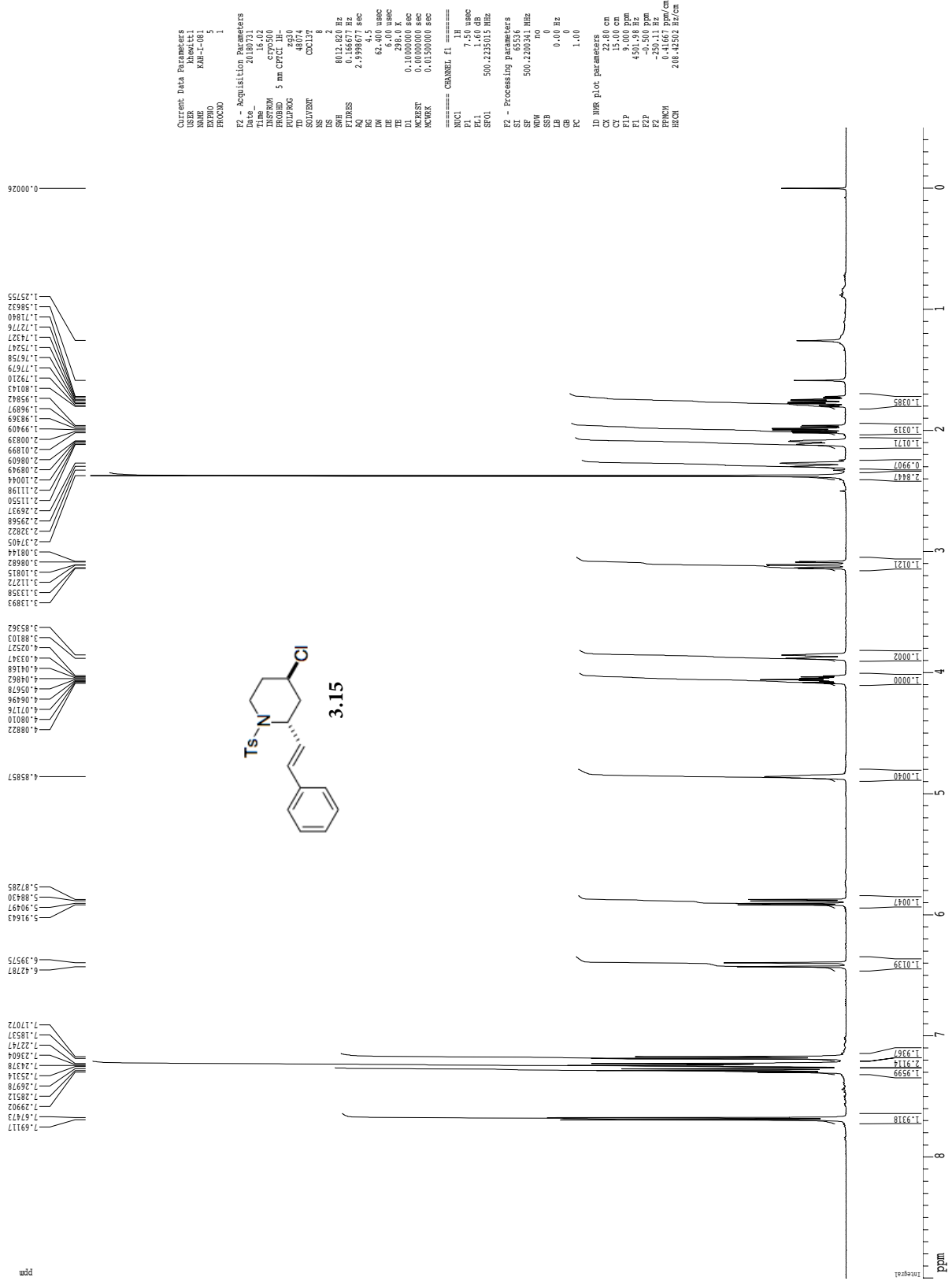
==== CHANNEL f1 =====  
NUC1: 1H  
P1: 7.50 usec  
PL1: 22.50 usec  
PC: 20.00 usec  
PD: 20.00 usec  
PR: 40000.00 usec  
PZ: 40000.00 usec  
RF1: 1.60 dB  
SFO1: 500.2221064 MHz  
WDW: EM  
SSB: gauss15  
GAMMA9: 90.00 Hz  
SFOFF9: 0.00 Hz

==== GRADIENT CHANNEL =====  
GAMMA0: sine.100  
GAMMA1: sine.100  
GAMMA2: sine.100  
GAMMA3: sine.100  
GAMMA4: sine.100  
GAMMA5: sine.100  
GAMMA6: sine.100  
GAMMA7: sine.100  
GAMMA8: sine.100  
GAMMA9: sine.100  
GAMMA10: sine.100  
GAMMA11: sine.100  
GAMMA12: sine.100  
GAMMA13: sine.100  
GAMMA14: sine.100  
GAMMA15: sine.100  
GAMMA16: sine.100  
GAMMA17: sine.100  
GAMMA18: sine.100  
GAMMA19: sine.100  
GAMMA20: sine.100  
GAMMA21: sine.100  
GAMMA22: sine.100  
GAMMA23: sine.100  
GAMMA24: sine.100  
GAMMA25: sine.100  
GAMMA26: sine.100  
GAMMA27: sine.100  
GAMMA28: sine.100  
GAMMA29: sine.100  
GAMMA30: sine.100

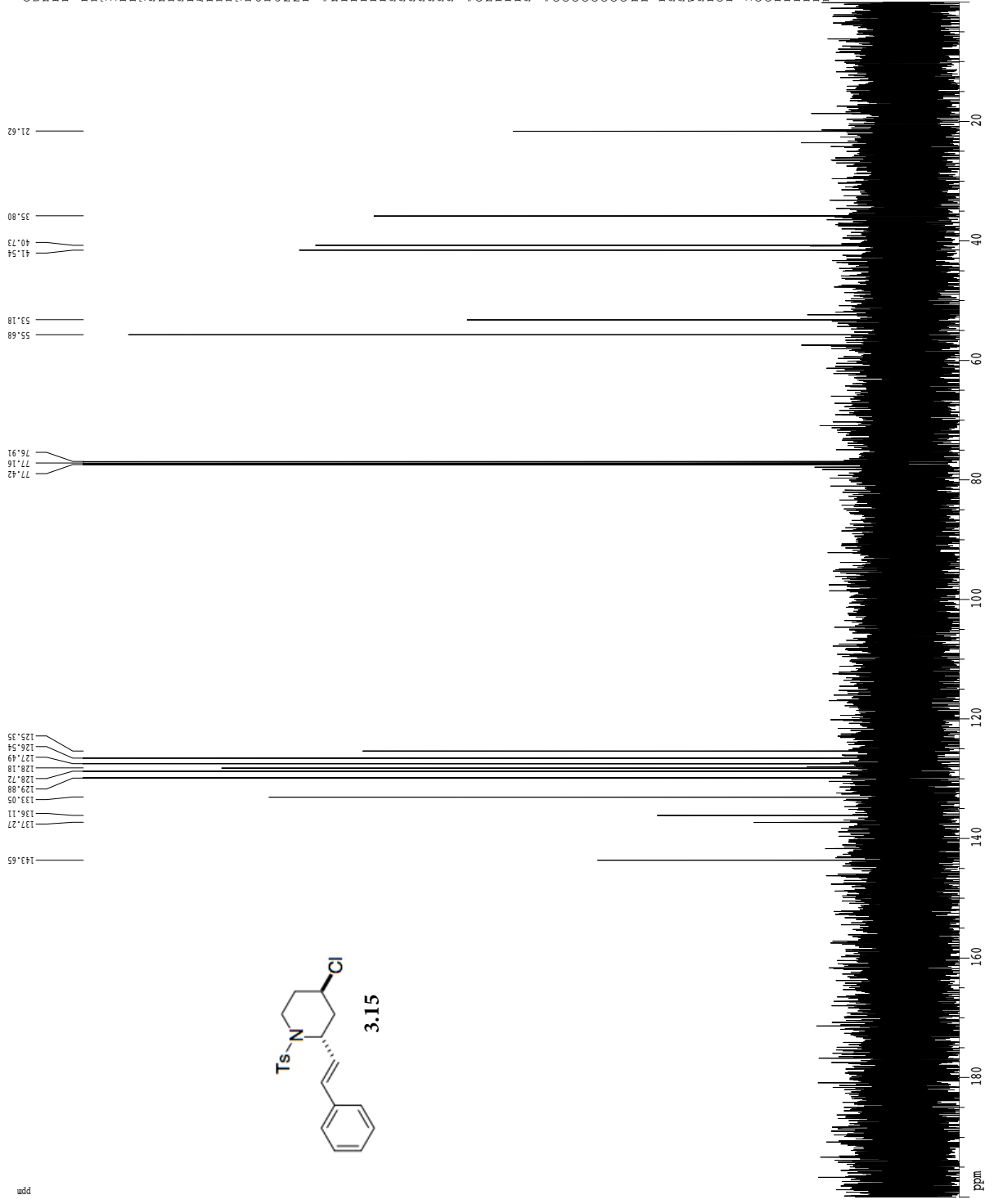
F2 - Processing parameters  
SI: 65536  
SF: 500.2200000 MHz  
WDW: EM  
SSB: 0  
LB: 0.00 Hz  
GB: 0  
PC: 1.00  
ID NMR plot parameters  
CX: 22.80 cm  
CY: 50.00 cm  
CZ: 50.00 cm  
F1P: 450.00 ppm  
F2P: -0.500 ppm  
F3P: -250.11 Hz  
PPHM: 0.41667 ppm/cm  
RECN: 208.42500 Hz/cm



1H spectrum



# Z-restored spin-echo 13C spectrum with 1H decoupling



gcosy60

```

Current Data Parameters
USER      khewitt1
NAME      KAH-1-071-2
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20100818
Time      13:06:00
INSTRUM   crysov0
PROBHD    5 mm CPTCI 1H-
PULPROG   cosygp60.prd
TD         2048
SOLVENT   CDCl3
NS         1
DS         16
SFO1       8012.827 Hz
FIDRES     3.91251 Hz
AQ         0.1278457 sec
RG         362
DW         62.400 usec
DE         6.00 usec
TE         298.0 K
d0         0.00000300 sec
d1         1.00000000 sec
d11        0.00000000 sec
d15        0.00020000 sec
d16        0.00020000 sec
IN0        0.0002489 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
PL1        1.60 dB
SFO1       500.225015 MHz

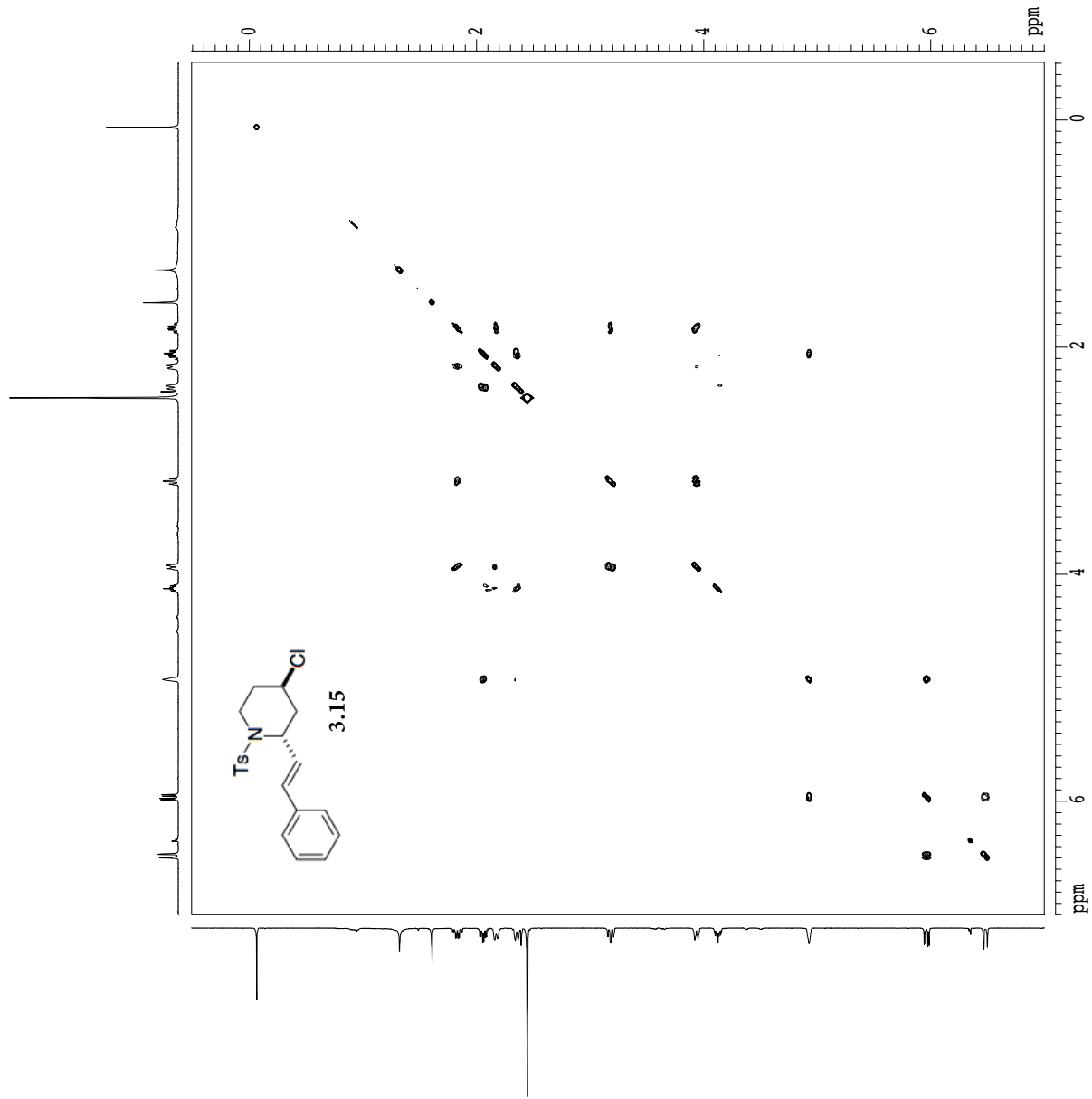
===== GRADIENT CHANNEL =====
GPRAM1     size:100
GEX1       0.00 %
GEX2       0.00 %
GEV1       0.00 %
GEV2       0.00 %
GZ1        17.00 %
GZ2        17.00 %
P16        1000.00 usec

F1 - Acquisition parameters
NU0        1
TD          512
SFO1       500.2235 MHz
FIDRES     15.650040 Hz
SW         16.018 Ppm
FROUZE     QF

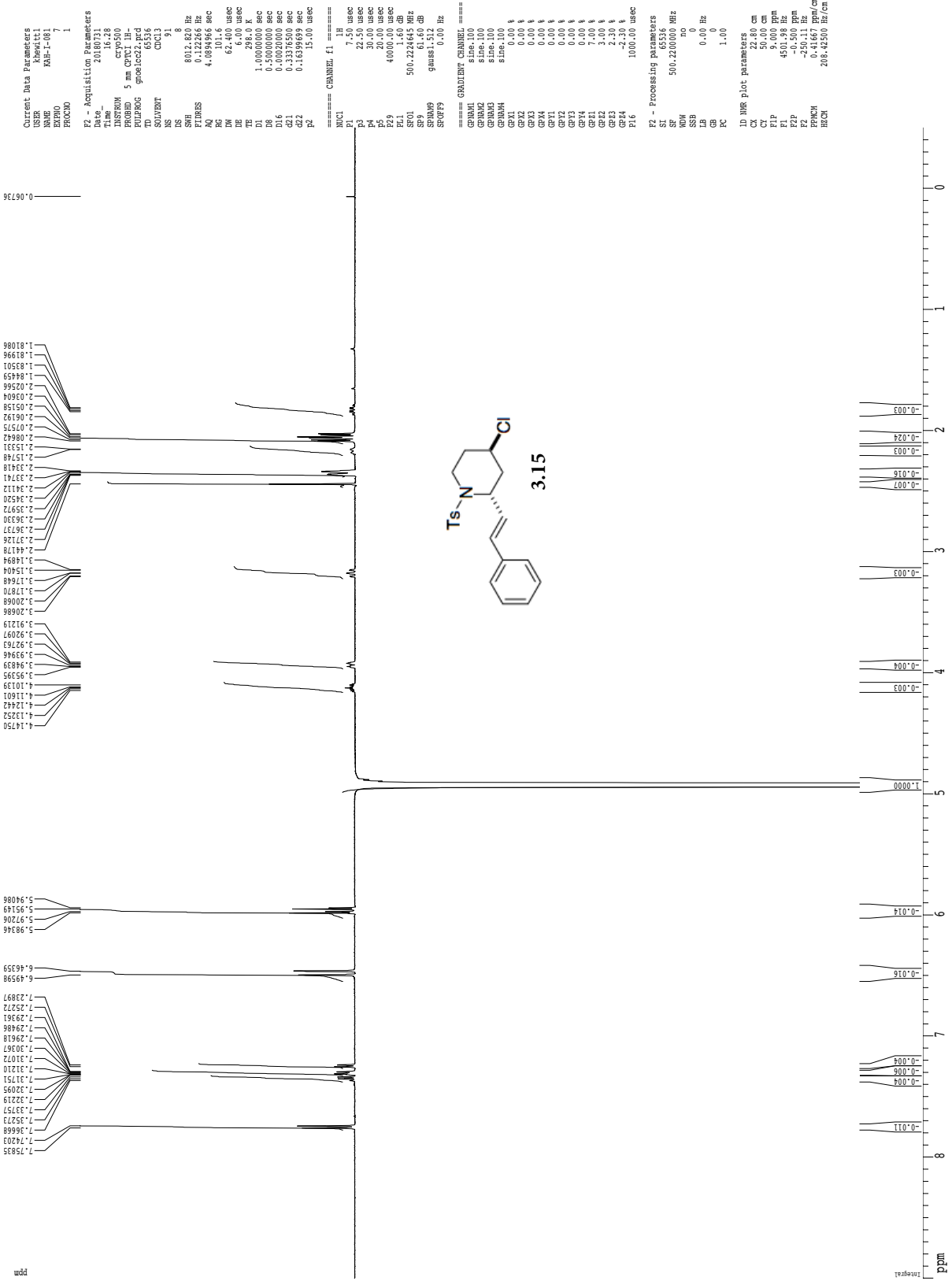
F2 - Processing parameters
SI         1024
SF         500.2200000 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        QF
SF         500.2200000 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      7.000 ppm
FZLO       3501.54 Hz
F2PHI      -0.509 Ppm
FZHI       -254.47 Hz
F1LO       3501.54 Hz
F1PHI      -0.509 Ppm
F1HI       -254.47 Hz
F2FPMCM    0.50058 ppm/cm
F2HCM      250.40063 Hz/cm
F1FPMCM    0.50058 ppm/cm
F1HCM      250.40063 Hz/cm
  
```

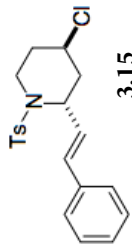
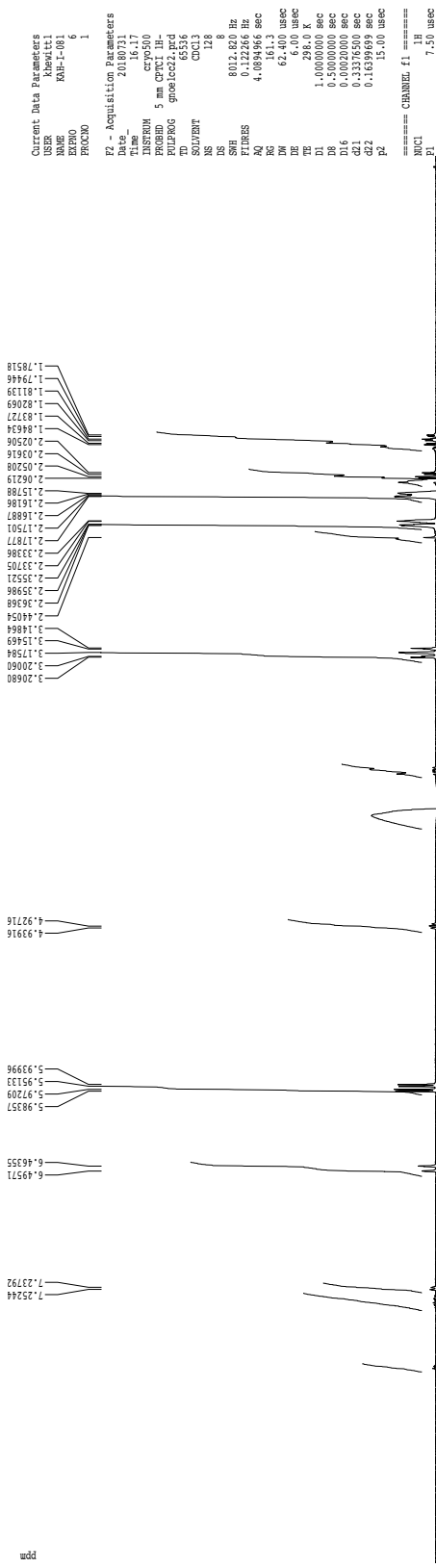


gnoe



gnoe

ppm

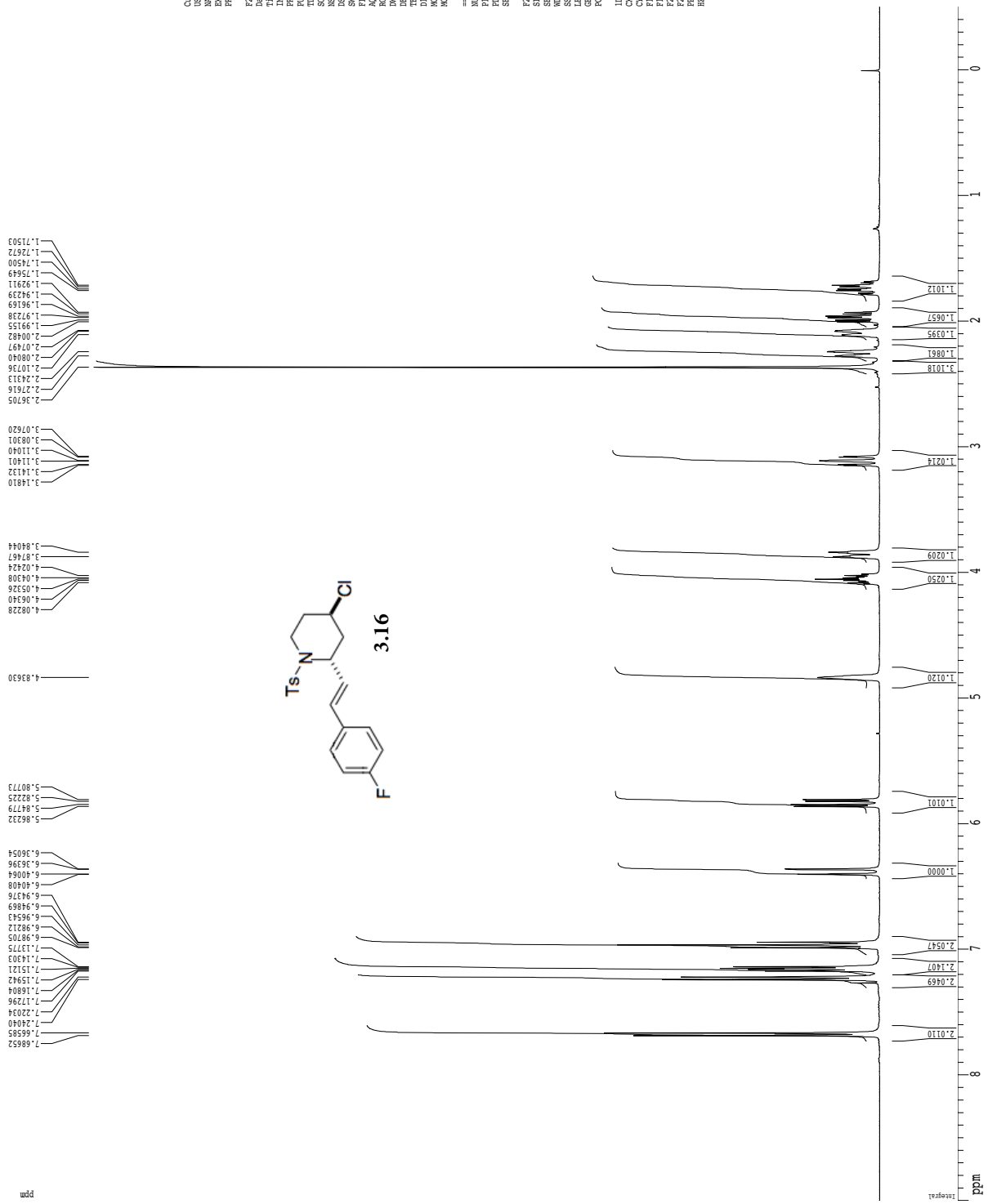


```
Current Data Parameters
USER      khawitt1
NAME      RNH-1-081
EXPNO     6
PROCNO    1
=====
F2 - Acquisition Parameters
Date_     20180731
Time      16.17
PROBHD    5 mm CPCT 1H
PULPROG   zgpg30
SOLVENT   gnoe
TD         65536
SOLVENT   CDCl3
NS         128
DS         8
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.089996 sec
RG         655.36
WDW         EM
SSB         0
LB         62.400 Hz
GB         0
TE         298.0 K
D1         1.0000000 sec
d11        0.3337500 sec
d12        0.3337500 sec
d2         0.1639869 sec
d22        15.00 usec
=====
===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
PL1        22.50 dB
PC1        20.00 usec
=====
===== CHANNEL f2 =====
NUC2       13C
P2         40000.00 usec
PL2        1.60 dB
PC2        20.00 usec
=====
===== GRABDT CHANNEL =====
GRAB1      sine.100
GRAB2      sine.100
GRAB3      sine.100
GRAB4      sine.100
GRAB5      sine.100
GRAB6      sine.100
GRAB7      sine.100
GRAB8      sine.100
GRAB9      sine.100
GRAB10     sine.100
=====
F2 - Processing parameters
SI         65536
SF         500.2200000 MHz
WDW         EM
SSB         0
LB         0.00 Hz
GB         0
PC         1.00
=====
ID NMR plot parameters
CX         22.80 cm
CY         50.00 cm
TI         1.00 ppm
F1P        450.00 ppm
F2P        0.000 ppm
FZ         0.00 Hz
PPHMM      0.38974 ppm/cm
RECN       197.45268 Hz/cm
```



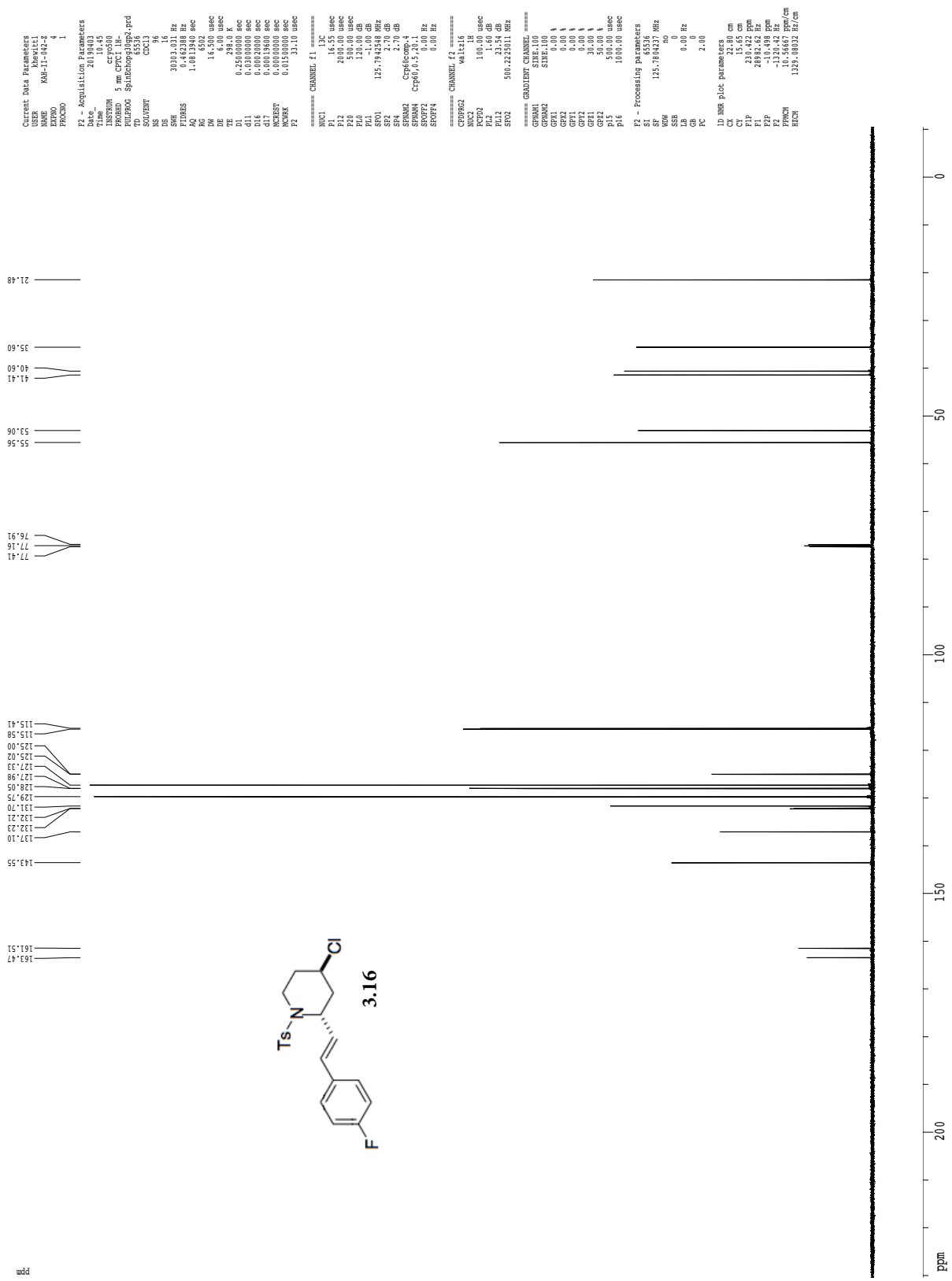


1H spectrum



Current Data Parameters  
 USER kkwatt1  
 NAME KMF-17-042-2  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20191003  
 Time 10.13  
 Operator  
 PULPROG zgpg30  
 PROBR0 5 mm QNP HET  
 TD 38460  
 SOLVENT CDCl3  
 NS 0  
 DS 2  
 SWH 6410.256 Hz  
 FIDRES 0.166672 Hz  
 AQ 2.9999239 sec  
 SFO1 400.132809 MHz  
 DQ 78.000 usec  
 DE 4.50 usec  
 TE 298.1 K  
 T1 0.100000 sec  
 MCHRS1 0.100000 sec  
 MCHRS2 0.1500000 sec  
 ===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.132809 MHz  
 F2 - Processing Parameters  
 SI 65536  
 SF 400.1300175 MHz  
 WDW no  
 SSB 0 Hz  
 GB 0  
 PC 2.00  
 ID\_NMR File Parameters  
 CX 22.80 cm  
 CZ 15.40 cm  
 FIP 9.000 ppm  
 F1F 3601.17 Hz  
 F2F 200.000 ppm  
 F3F -200.000 Hz  
 PPRCM 0.41667 ppm/cm  
 RECH 166.72084 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters  
 USER khsctt1  
 NAME RAH-II-042-Z  
 EXPNO 3  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 201003  
 Time 00:30  
 INSTRUM crys500  
 PROBHD 5 mm CPTCI 1H-  
 PULPROG cosygp60.prd  
 TD 2048  
 SOLVENT CDCl3  
 NS 1  
 DS 16  
 SFO1 500.225015 MHz  
 FIDRES 3.91251 Hz  
 AQ 0.1278452 sec  
 RG 28.5  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.0 K  
 d0 0.00000300 sec  
 D1 1.00000000 sec  
 D11 0.00000000 sec  
 D16 0.00020000 sec  
 INU 0.0002488 sec

==== CHANNEL f1 =====

NUC1 1H  
 P1 7.50 usec  
 PL1 1.60 dB  
 SFO1 500.225015 MHz

==== GRADIENT CHANNEL =====

GPRAM1  
 GPRAM2  
 GPC1 0.00 %  
 GPC2 0.00 %  
 GPC3 0.00 %  
 GPC4 0.00 %  
 GPC5 17.00 %  
 GPC6 17.00 %  
 P16 1000.00 usec

F1 - Acquisition parameters

ND0 1  
 TD 512  
 SFO1 500.2235 MHz  
 FIDRES 15.650040 Hz  
 SW 16.018 Ppm  
 FROUSE QF

F2 - Processing parameters

SI 1024  
 SF 500.2200300 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

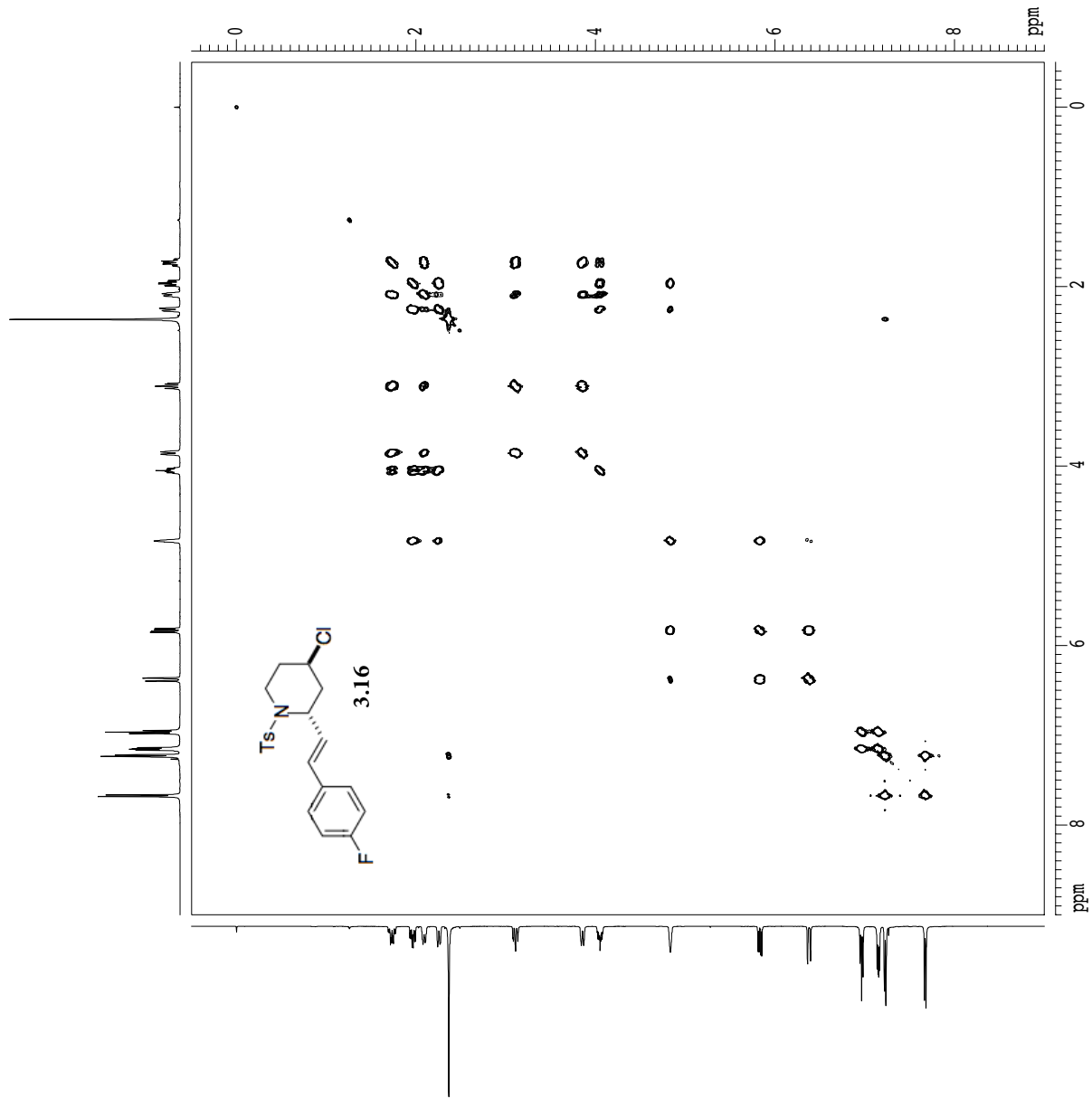
F1 - Processing parameters

SI 1024  
 MC2 QF  
 SF 500.2200300 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

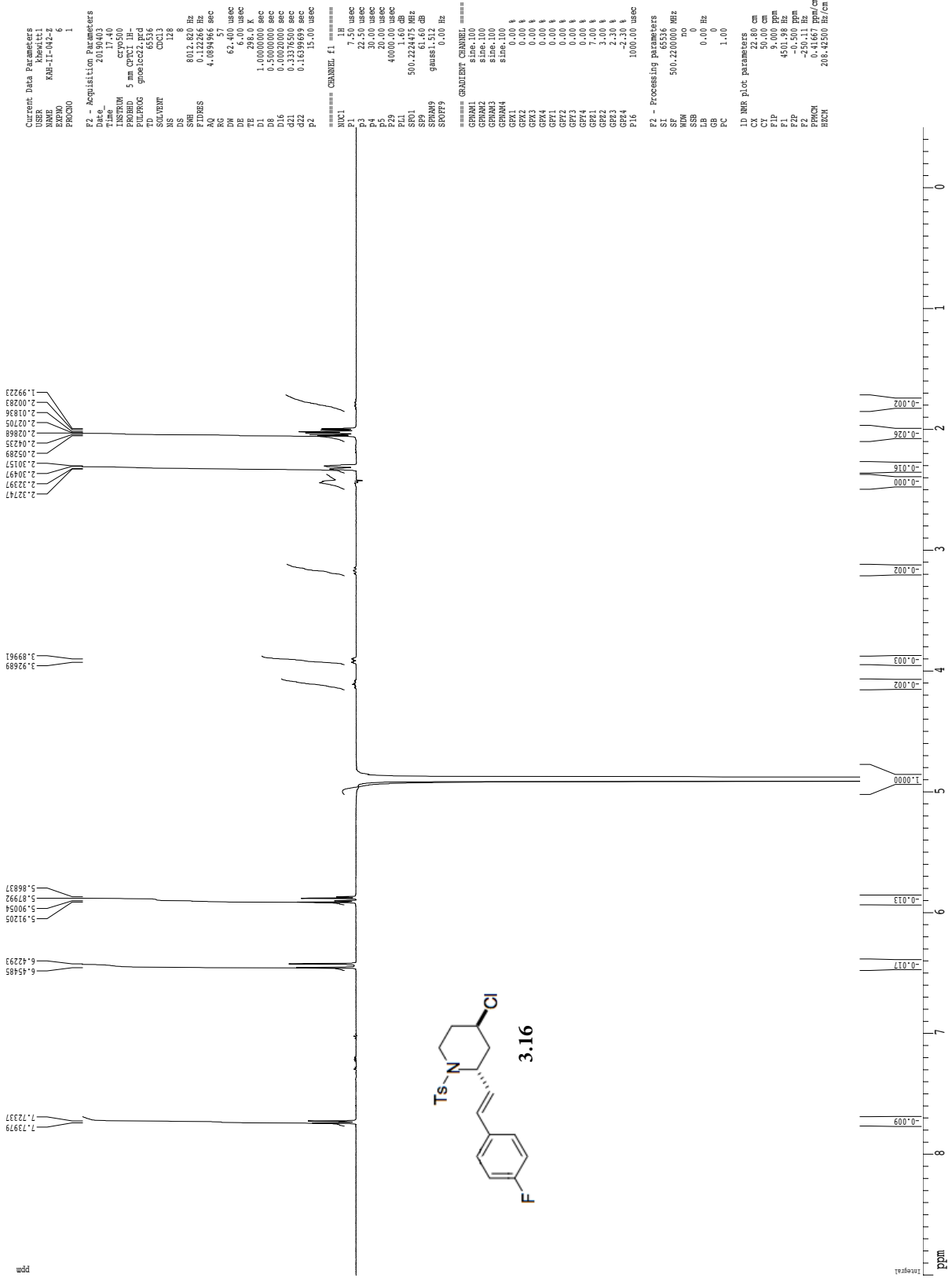
2D NMR plot parameters

CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 9.000 Ppm  
 FZLO 4501.98 Hz  
 FZPH -0.500 Ppm  
 FZHI -250.11 Hz  
 FZLO 4501.98 Hz  
 FZPH -0.500 Ppm  
 FZHI -250.11 Hz  
 F2PFCM 0.6333 Ppm/cm  
 FZPFCM 316.8603 Hz/cm  
 F1PFCM 0.6333 Ppm/cm  
 F1PFCM 316.8603 Hz/cm

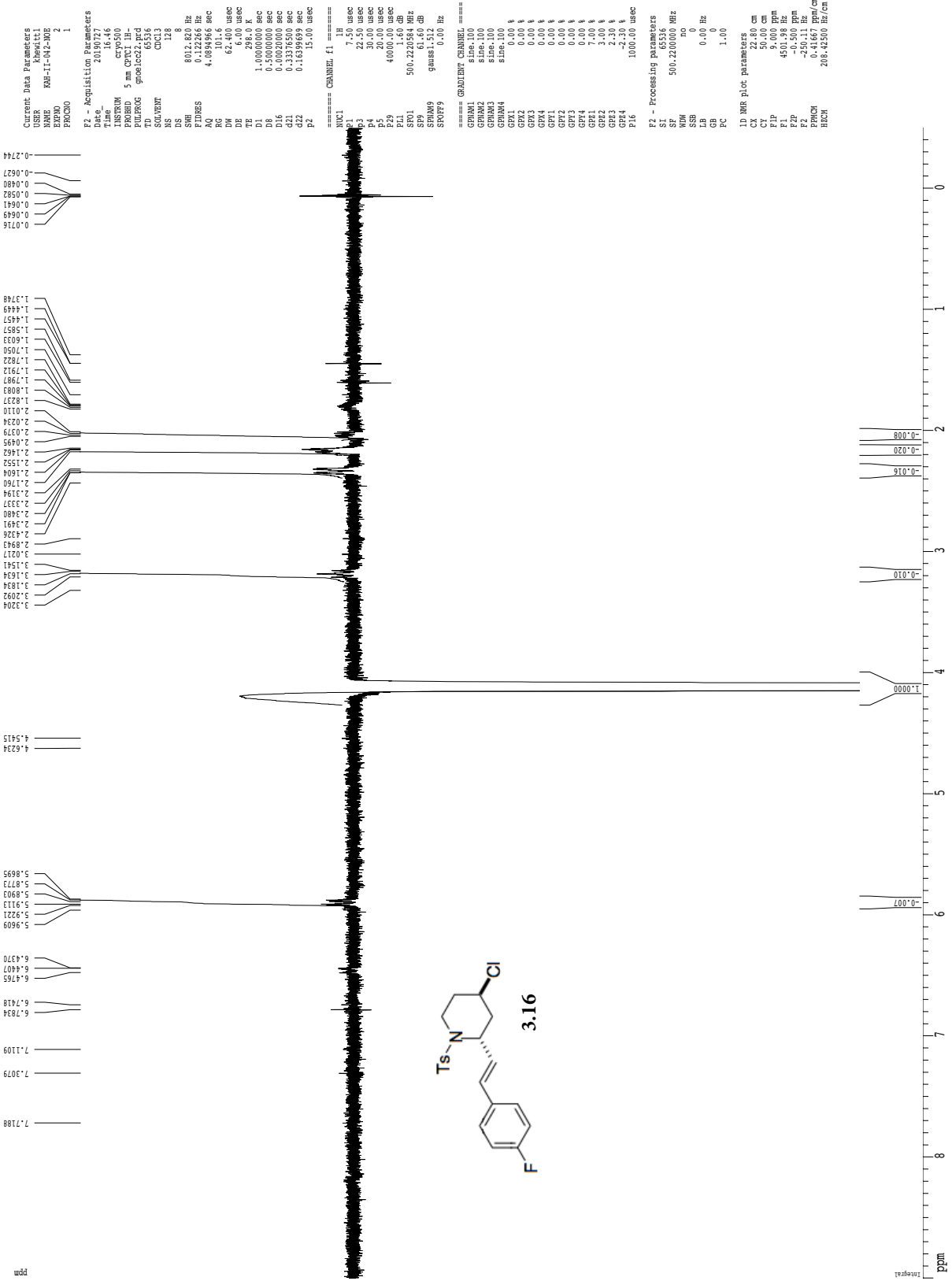
gc05y60



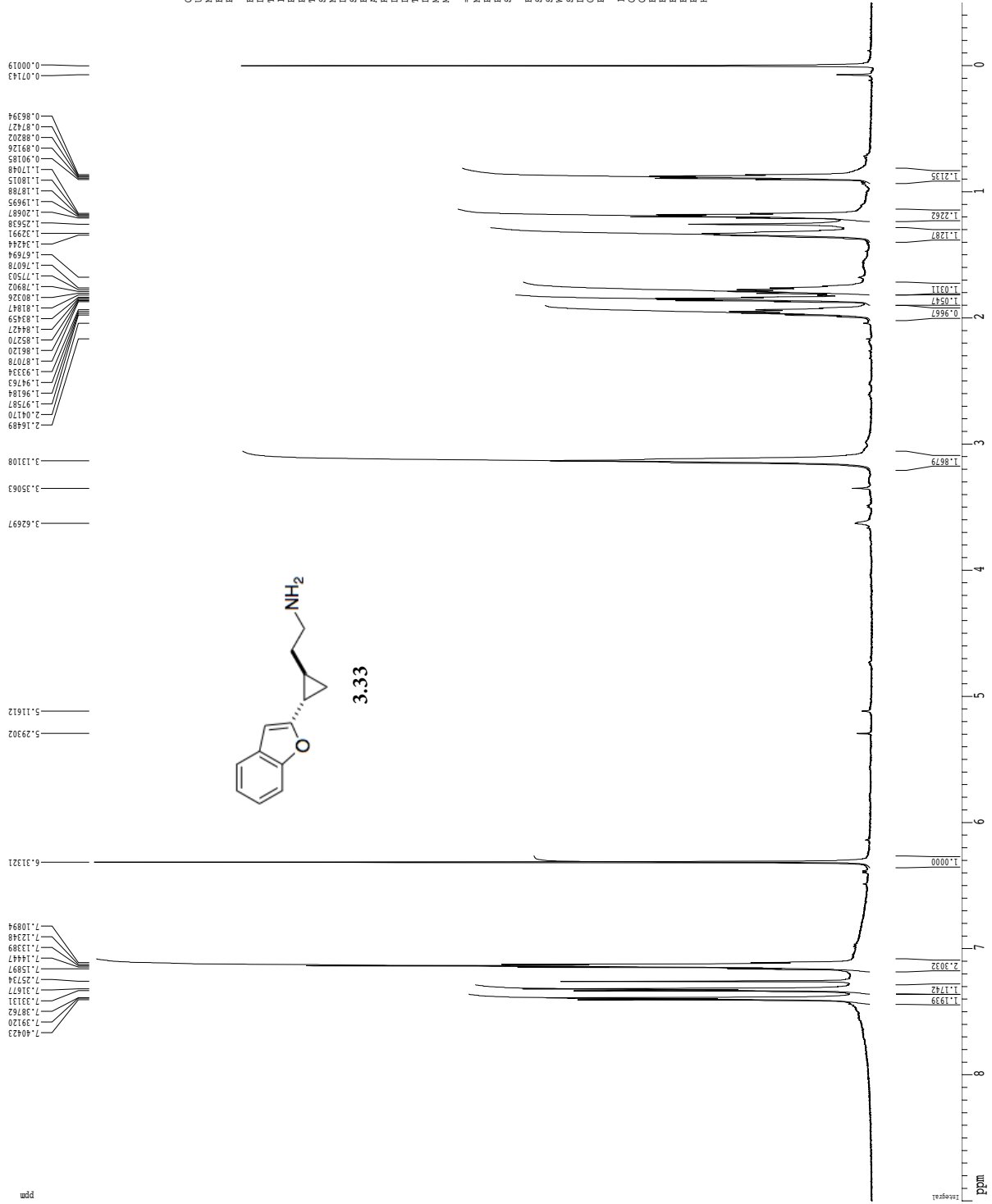
gnoe



gnoe

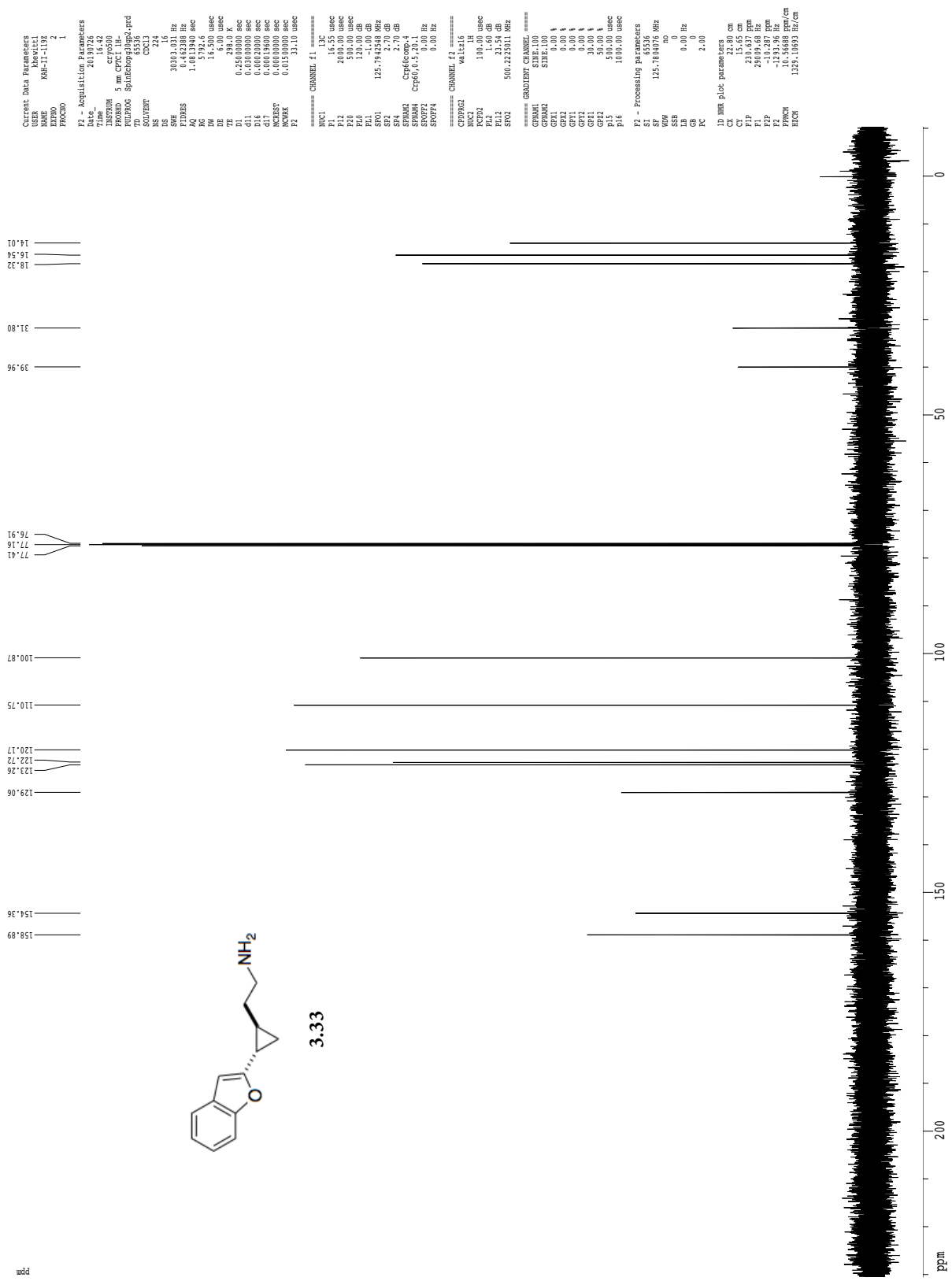


1H spectrum



Current Data Parameters  
 USER: Rawlett  
 NAME: RM-1-192  
 EXPNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_ : 20190726  
 Time : 16.36  
 Operator :  
 PULPROG : zgpg30  
 PROBRN : 5  
 TD : 65536  
 SFO1 : 500.136261 MHz  
 SOLVENT : CDCl3  
 NS : 6  
 DS : 2  
 SWH : 8012.820 Hz  
 FIDRES : 0.166677 Hz  
 AQ : 2.5998677 sec  
 RG : 327.5  
 DE : 62.400 usec  
 TE : 298.2 K  
 D0 : 0.10000000 sec  
 MCHRES : 0.10000000 sec  
 MCNMRK : 0.01500000 sec  
 ===== CHANNEL f1 =====  
 NUC1 : 13C  
 P1 : 1.50 usec  
 PL1 : 1.90 dB  
 SFO1 : 500.2235015 MHz  
 F2 - Processing parameters  
 SI : 65536  
 SF : 500.2203332 MHz  
 WDW : no  
 SSB : 0  
 CB : 0.00 Hz  
 GB : 0  
 PC : 1.00  
 ID: NMR file parameters  
 CD : 22.80 cm  
 C1 : 15.00 cm  
 F1P : 9.000 ppm  
 F1R : 4501.78 Hz  
 F2P : -250.11 Hz  
 F2R : 0.41667 ppm/cm  
 FREQM : 208.44502 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



gc05y60

Current Data Parameters  
USER khewitt1  
NAME KAH-II-119 COSY-2  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 201905  
Time 11:11  
INSTRUM crys500  
PROBHD 5 mm CPTCI 1H  
PULPROG cosygp60.prd  
TD 2048  
SOLVENT CDCl3  
NS 1  
DS 16  
SWH 8012.871 Hz  
FIDRES 3.91251 Hz  
AQ 0.1278452 sec  
RG 287.4  
DW 62.400 usec  
DE 6.00 usec  
TE 298.0 K  
d0 0.00000300 sec  
D1 1.00000000 sec  
D11 0.00000000 sec  
D15 0.00020000 sec  
D16 0.00020000 sec  
D17 0.00020000 sec  
D18 0.00020000 sec  
D19 0.00020000 sec  
D20 0.00020000 sec  
D21 0.00020000 sec  
D22 0.00020000 sec  
D23 0.00020000 sec  
D24 0.00020000 sec  
D25 0.00020000 sec  
D26 0.00020000 sec  
D27 0.00020000 sec  
D28 0.00020000 sec  
D29 0.00020000 sec  
D30 0.00020000 sec  
D31 0.00020000 sec  
D32 0.00020000 sec  
D33 0.00020000 sec  
D34 0.00020000 sec  
D35 0.00020000 sec  
D36 0.00020000 sec  
D37 0.00020000 sec  
D38 0.00020000 sec  
D39 0.00020000 sec  
D40 0.00020000 sec  
D41 0.00020000 sec  
D42 0.00020000 sec  
D43 0.00020000 sec  
D44 0.00020000 sec  
D45 0.00020000 sec  
D46 0.00020000 sec  
D47 0.00020000 sec  
D48 0.00020000 sec  
D49 0.00020000 sec  
D50 0.00020000 sec  
D51 0.00020000 sec  
D52 0.00020000 sec  
D53 0.00020000 sec  
D54 0.00020000 sec  
D55 0.00020000 sec  
D56 0.00020000 sec  
D57 0.00020000 sec  
D58 0.00020000 sec  
D59 0.00020000 sec  
D60 0.00020000 sec  
D61 0.00020000 sec  
D62 0.00020000 sec  
D63 0.00020000 sec  
D64 0.00020000 sec  
D65 0.00020000 sec  
D66 0.00020000 sec  
D67 0.00020000 sec  
D68 0.00020000 sec  
D69 0.00020000 sec  
D70 0.00020000 sec  
D71 0.00020000 sec  
D72 0.00020000 sec  
D73 0.00020000 sec  
D74 0.00020000 sec  
D75 0.00020000 sec  
D76 0.00020000 sec  
D77 0.00020000 sec  
D78 0.00020000 sec  
D79 0.00020000 sec  
D80 0.00020000 sec  
D81 0.00020000 sec  
D82 0.00020000 sec  
D83 0.00020000 sec  
D84 0.00020000 sec  
D85 0.00020000 sec  
D86 0.00020000 sec  
D87 0.00020000 sec  
D88 0.00020000 sec  
D89 0.00020000 sec  
D90 0.00020000 sec  
D91 0.00020000 sec  
D92 0.00020000 sec  
D93 0.00020000 sec  
D94 0.00020000 sec  
D95 0.00020000 sec  
D96 0.00020000 sec  
D97 0.00020000 sec  
D98 0.00020000 sec  
D99 0.00020000 sec  
D100 0.00020000 sec

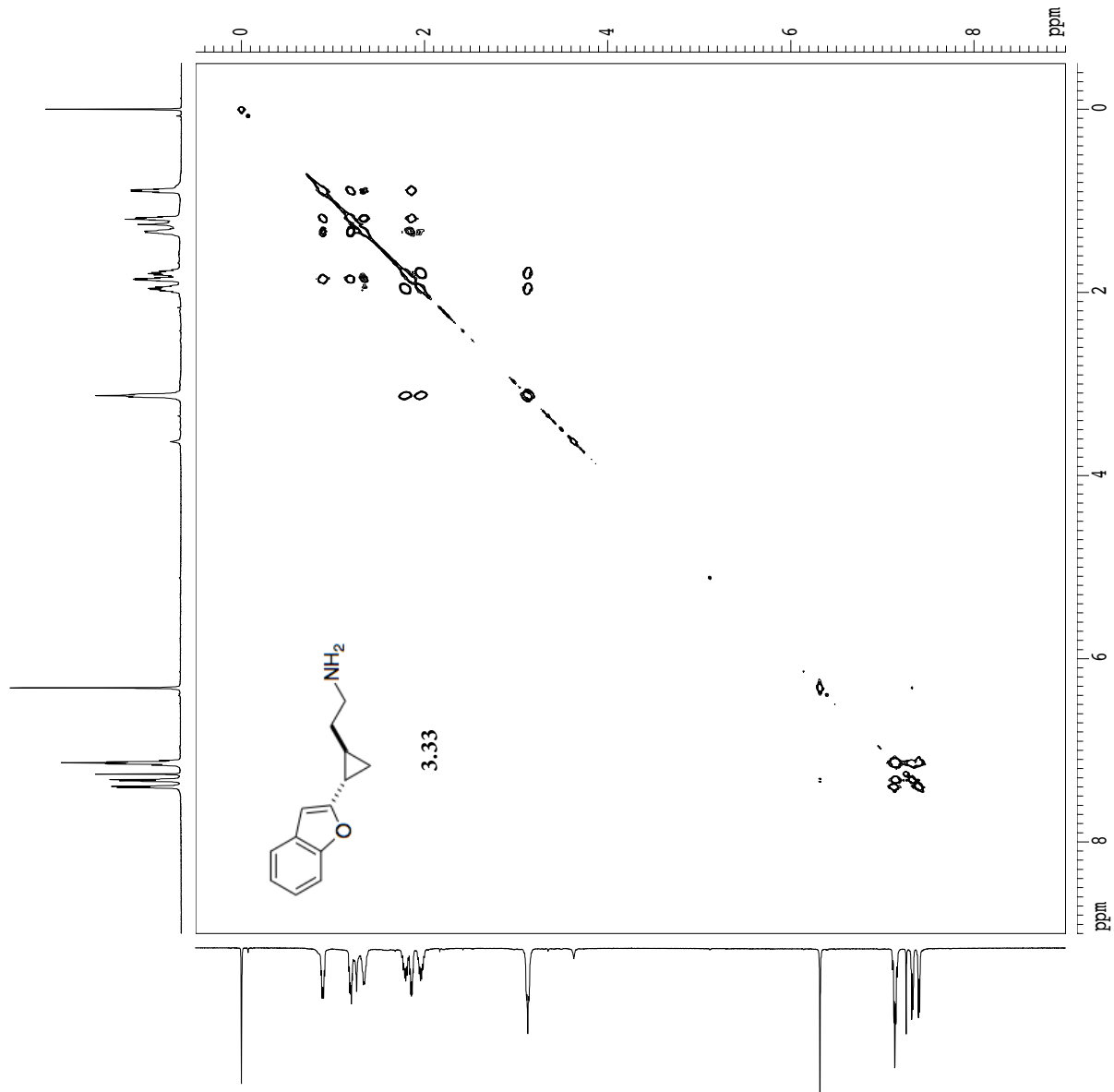
==== CHANNEL f1 =====  
NUC1 1H  
P1 7.50 usec  
PL1 1.60 dB  
SFO1 500.225015 MHz  
===== GRADIENT CHANNEL =====  
GPRM1 size:100  
GPRM2 size:100  
GEX1 0.00 %  
GEX2 0.00 %  
GEV1 0.00 %  
GEV2 0.00 %  
GEZ1 17.00 %  
GEZ2 17.00 %  
P16 1000.00 usec

F1 - Acquisition parameters  
ND0 1  
TD 512  
SFO1 500.2235 MHz  
FIDRES 15.450040 Hz  
SW 16.018 Ppm  
FREQ0 0F

F2 - Processing parameters  
SI 1024  
SF 500.220320 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

F1 - Processing parameters  
SI 1024  
MC2 0F  
SF 500.220320 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0

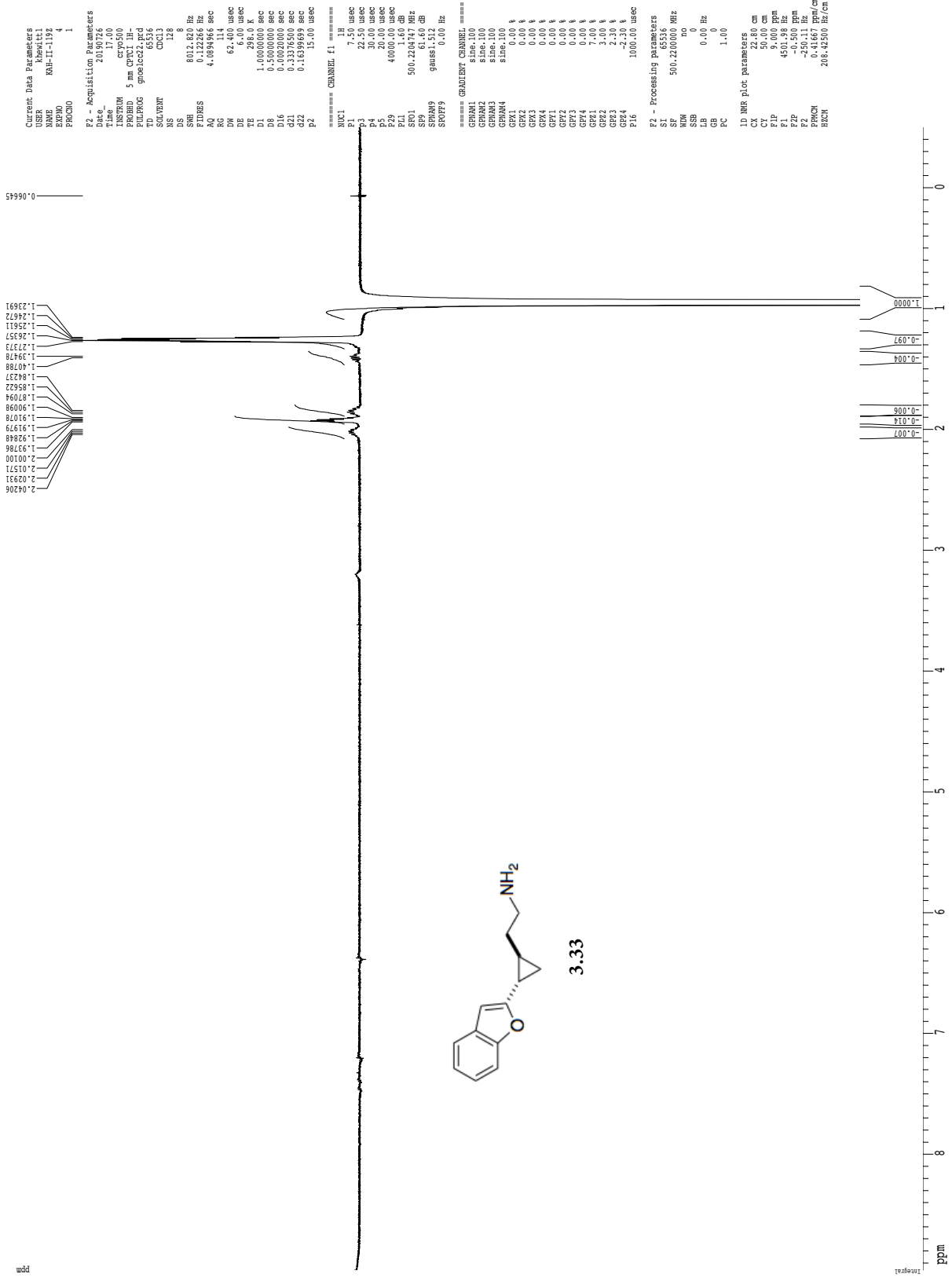
2D NMR plot parameters  
CX2 15.00 cm  
CX1 15.00 cm  
F2PLO 9.000 Ppm  
FZLO 4501.98 Hz  
FZHI -0.500 Ppm  
FZFL -2501.1 Hz  
FZFO 1001.00 Ppm  
FZLO 4501.98 Hz  
FZHI -0.500 Ppm  
FZFL -2501.1 Hz  
FZFO 1001.00 Ppm  
F1HI -2501.1 Hz  
F1FO 0.6333 Ppm/cm  
F2PFCM 0.6333 Hz/cm  
F2HFCM 316.80603 Hz/cm  
F1PFCM 0.6333 Ppm/cm  
F1HFCM 316.80603 Hz/cm



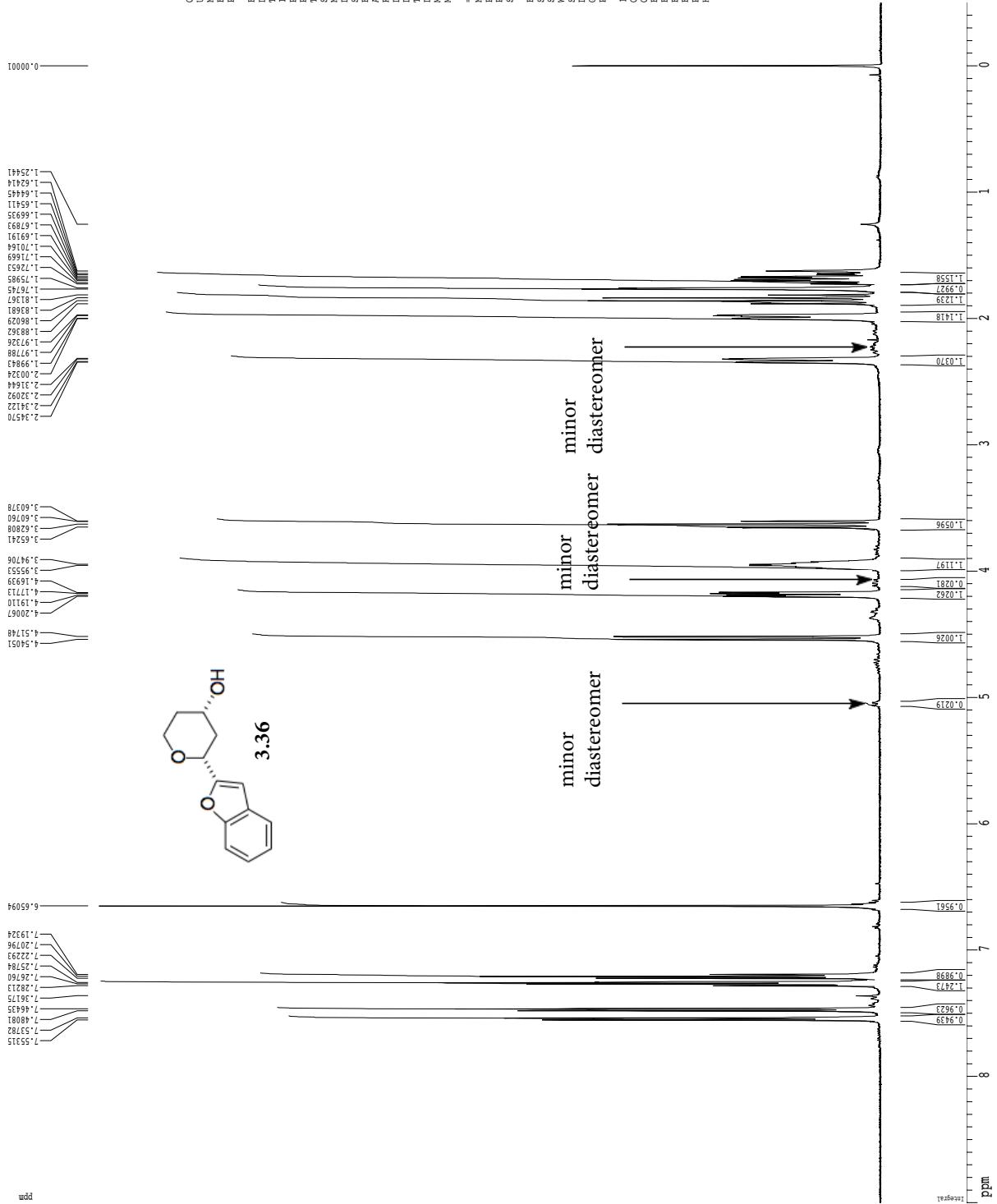


gnoe

ppm

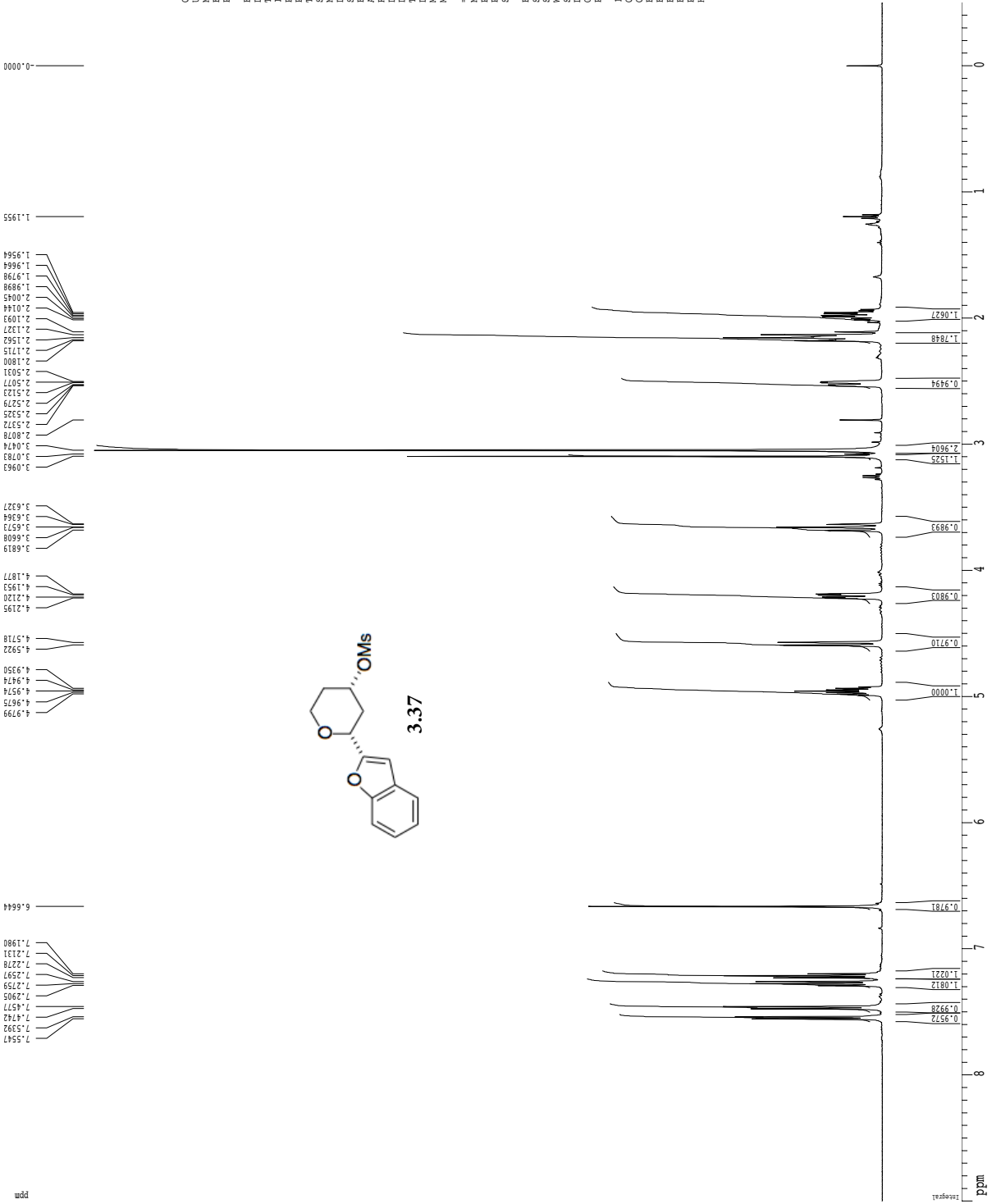


1H spectrum



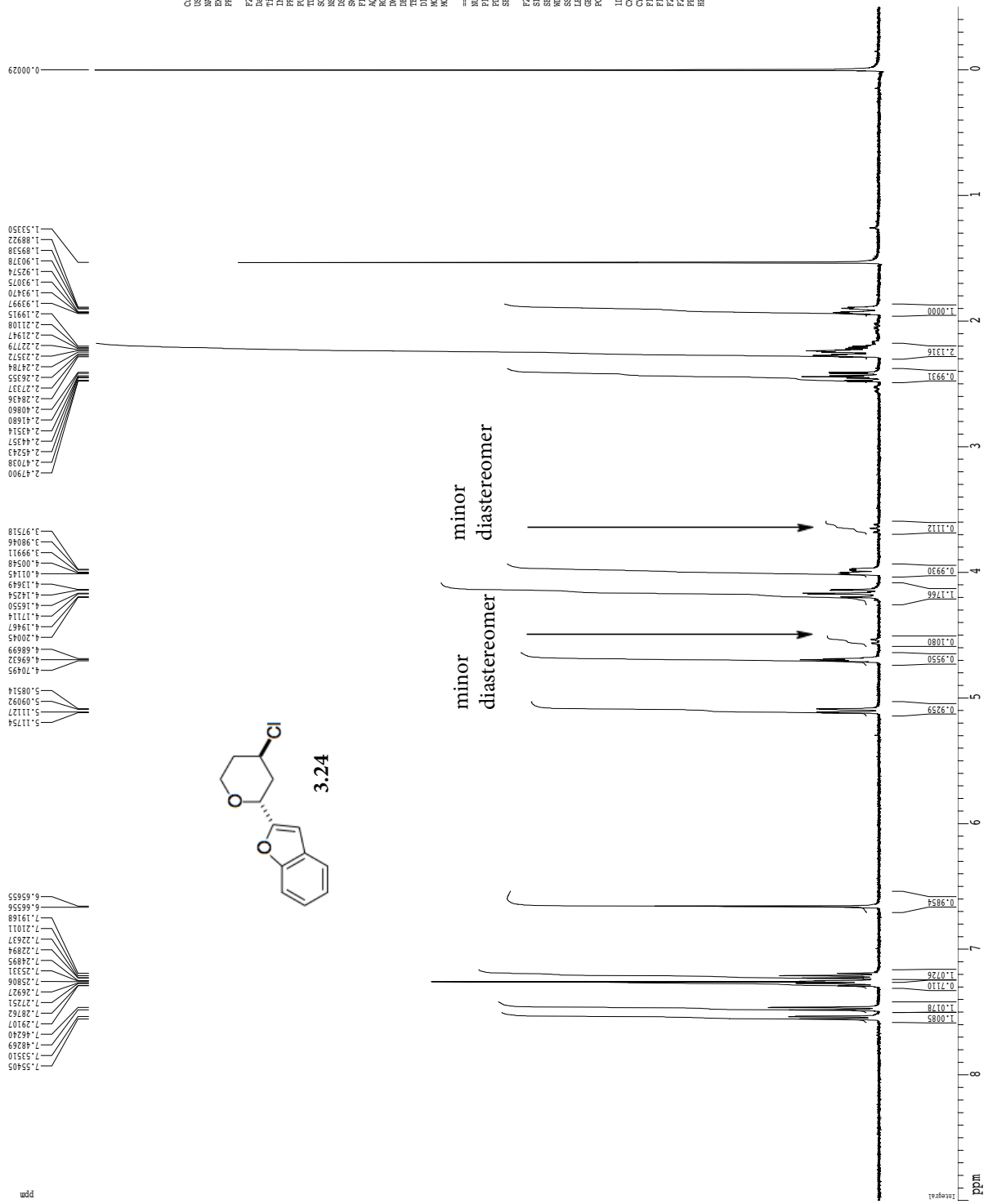
Current Data Parameters  
 USER emilyt  
 NAME LW-11-013 OH  
 EXPNO 10  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20150718  
 Time 17:14  
 INSTRUM czt500  
 PROBRD 5 mm CPYX1-1H  
 PULPROG zg30  
 TD 81728  
 NS 2048  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.098843 Hz  
 AQ 3.093758 sec  
 RG 71  
 DM 62.400 usec  
 DE 6.00 usec  
 DT 0.1000000 sec  
 K 1  
 MCHST 0.0000000 sec  
 MCRRK 0.0150000 sec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.50 usec  
 PL1 1.60 dB  
 SFO1 500.225015 MHz  
 F2 - Processing parameters  
 SI 65536  
 SF 500.2200326 MHz  
 WH 0  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 CB 4.00  
 PC  
 ID NMR plot parameters  
 CX 22.80 cm  
 CY 15.00 cm  
 FID 7.000 ppm  
 F1P -0.500 ppm  
 F2P -250.11 Hz  
 FZ 0.41667 ppm/cm  
 PPMXN 206.45302 Hz/cm

1H spectrum

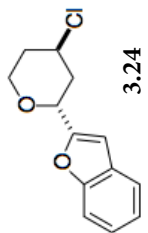


Current Data Parameters  
 USER emilyt  
 NAME LWR-111-022\_0Ms  
 EXPNO 10  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20150719  
 Time 11:08  
 INSTRUM czt450  
 PROBHD 5 mm CPYCT 1H  
 PULPROG zg30  
 TD 81728  
 NS 2  
 DS 2  
 SWH 8032.820 Hz  
 FIDRES 0.0989443 Hz  
 AQ 3.0938674 sec  
 RG 4  
 DM 62.400 usec  
 DE 6.00 usec  
 DT 0.0001000 K  
 MD 0.0000000 sec  
 MCBSST 0.0000000 sec  
 MCRRK 0.0150000 sec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.50 usec  
 PL1 1.50 dB  
 FL1 1.60 dB  
 SFO1 500.225015 MHz  
 F2 - Processing parameters  
 SI 65536  
 SF 500.2200310 MHz  
 WH 0  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 CB 4.00  
 ID NMR Plot parameters  
 CX 22.80 cm  
 CY 15.00 cm  
 FID 657.000 ppm  
 F2 0.0000000 Hz  
 FZP -0.500 ppm  
 FZ -250.11 Hz  
 PPMXN 0.41667 ppm/cm  
 RECIN 208.45302 Hz/cm

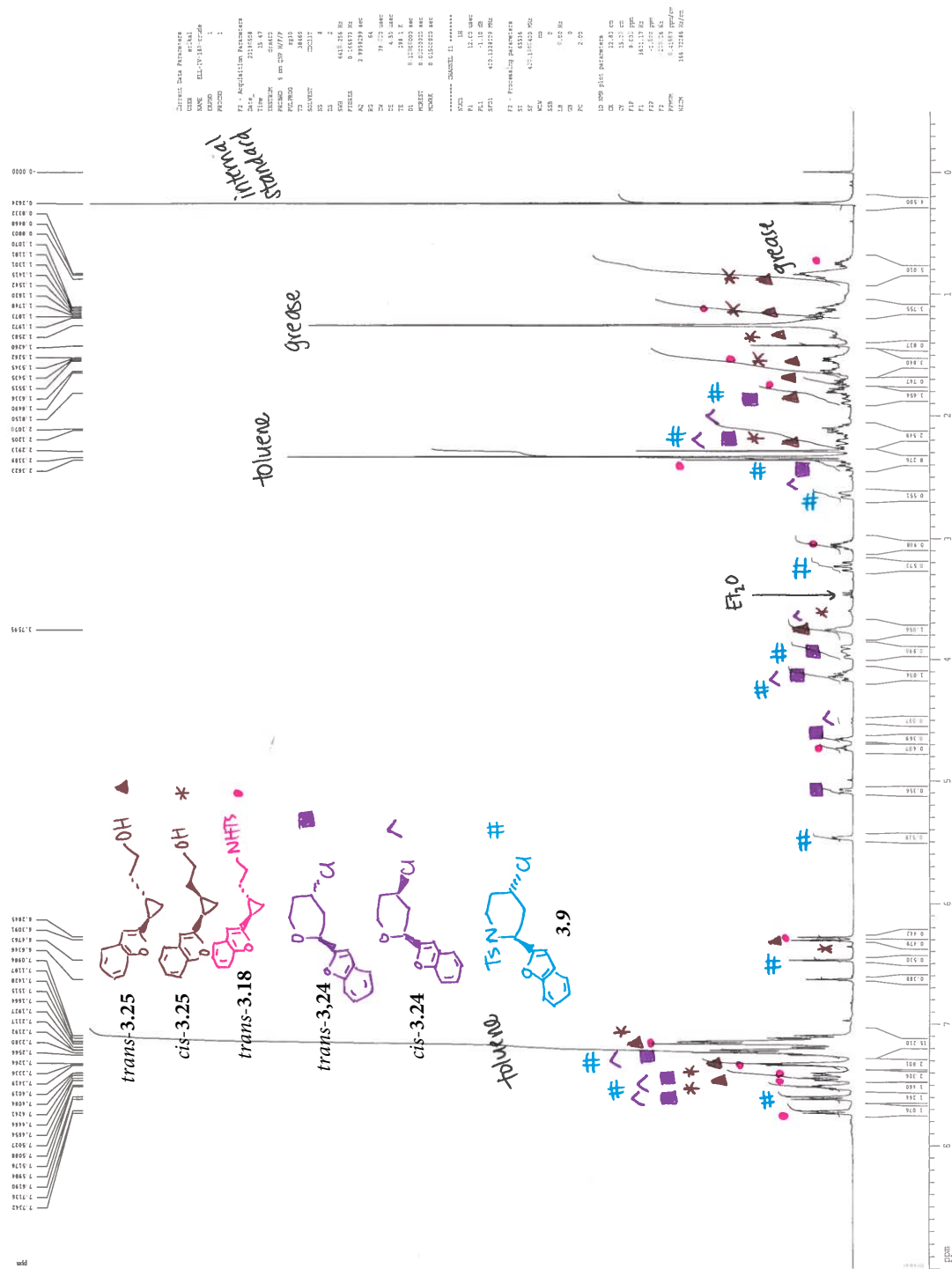
1H spectrum

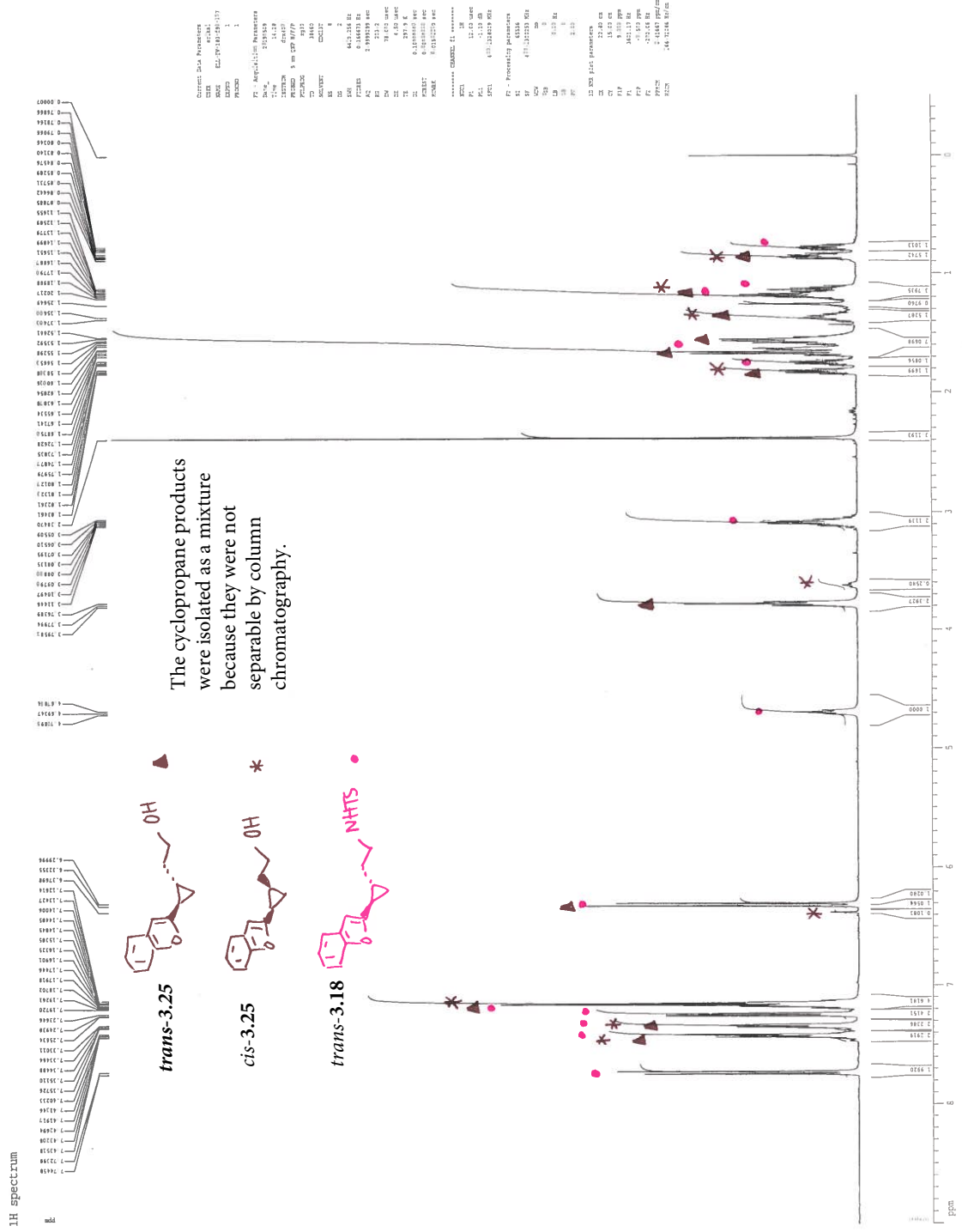


Current Data Parameters  
 USER: ELL-IV-175-Dr  
 NAME: ELL-IV-175-Dr  
 EXPNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20190507  
 Time: 9:58  
 Operator: ELL-IV-175-Dr  
 PULPROG: zgpg30  
 PCPDPRG2: 38460  
 TD: 38460  
 SOLVENT: CDCl3  
 NS: 2  
 DS: 2  
 SWH: 6410.256 Hz  
 FIDRES: 0.166672 Hz  
 AQ: 2.9999399 sec  
 RG: 327.5  
 WGT: 78.000 usec  
 DE: 4.50 usec  
 TE: 297.2 K  
 T1: 0.10000000 sec  
 T2: 0.10000000 sec  
 T3: 0.10000000 sec  
 MCHRG: 0.05500000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -1.10 dB  
 SFO1: 400.1328009 MHz  
 F2 - Processing Parameters  
 SI: 65536  
 SF: 400.1300217 MHz  
 WDW: no  
 SSB: 0 Hz  
 GB: 0 Hz  
 PC: 2.00  
 ID: NMR FIDIC Parameters  
 C1: 22.80 cm  
 C2: 15.00 cm  
 F1P: 9.000 ppm  
 F2P: 500.117 Hz  
 F3P: 200.000 ppm  
 F4P: -200.000 Hz  
 PPRCM: 0.41667 ppm/cm  
 RECH: 166.72086 Hz/cm

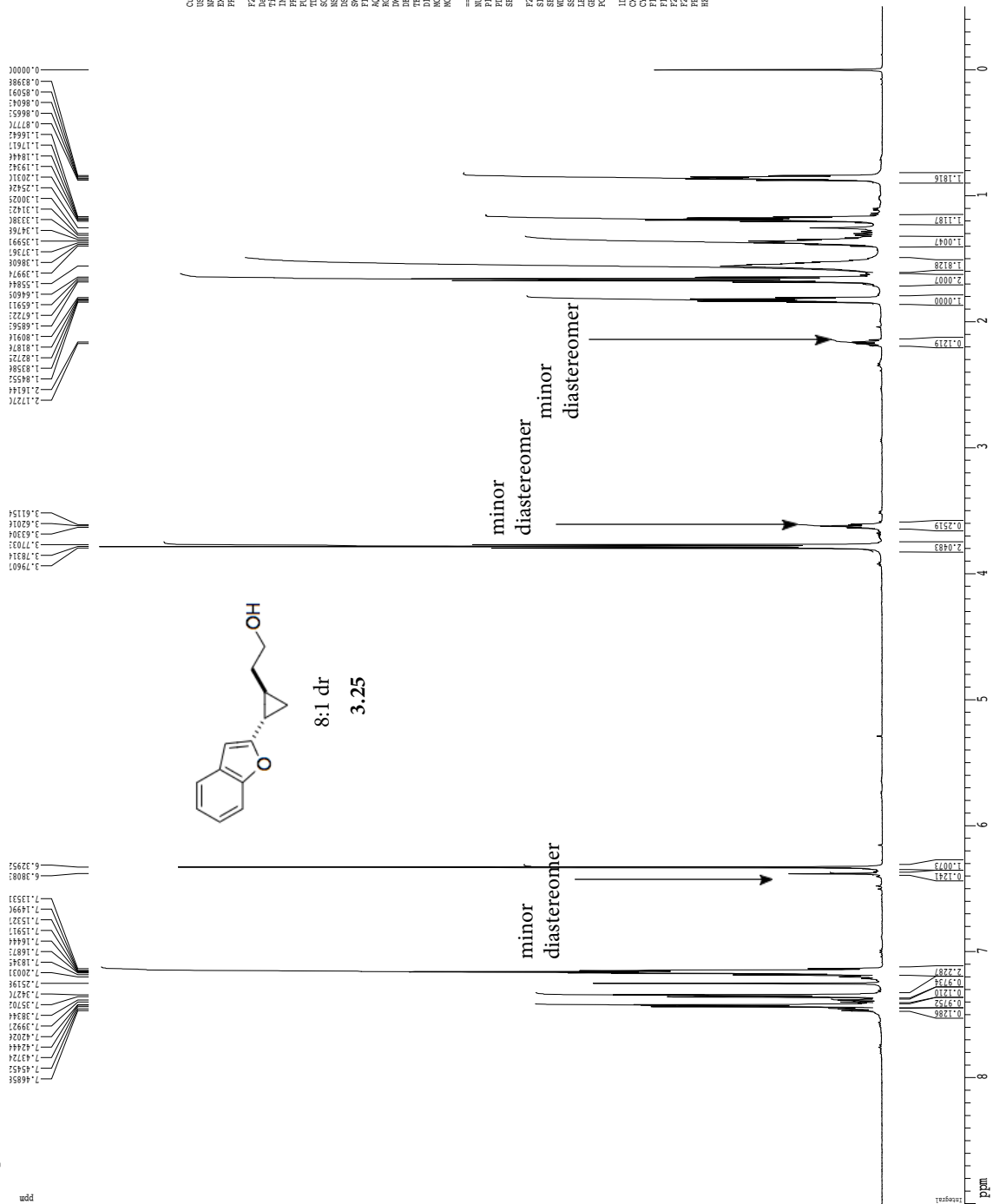


<sup>1</sup>H spectrum

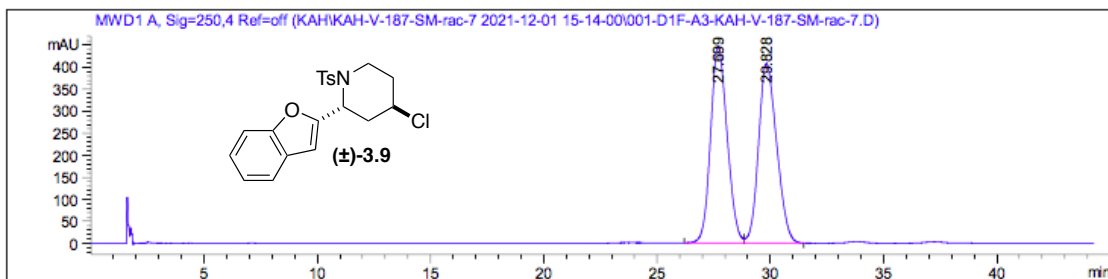




<sup>1</sup>H spectrum



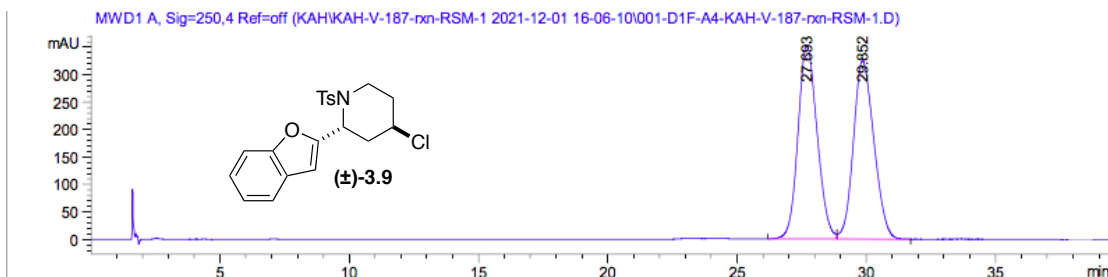
Current Data Parameters  
 Name: JAE-II-041.P001  
 ExpNO: 10  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20150717  
 Time: 14.21  
 Station: ccc1  
 PROBHD: 5 mm CPCLP1H-  
 PULPROG: zgpg30  
 TD: 65536  
 SFO1: 500.136261 MHz  
 AQ: 0.1000000 sec  
 AS: 8  
 DS: 2  
 SWH: 8013.500 Hz  
 FIDRES: 0.1000000 Hz  
 AQ: 5.0949398 sec  
 RG: 10.1  
 BR: 62.000 usec  
 RE: 298.0 K  
 TE: 298.0 K  
 DI: 0.1000000 sec  
 DE: 0.1000000 sec  
 ACQRES: 0.1000000 sec  
 FWHM: 0.1000000 sec  
 ===== CHANNEL f1 =====  
 NU1: 1  
 P1: 7.50 usec  
 PL1: 1.60 dB  
 SFO1: 500.2235015 MHz  
 F2 - Processing parameters  
 SI: 65536  
 SF: 500.2235015 MHz  
 AS: 8  
 SSB: 0  
 LB: 0.30 Hz  
 GB: 0  
 PC: 4.00  
 ID NMR plot parameters  
 CT: 22.00 cm  
 CD: 5.000 mm  
 FIDP: 5.000 ppm  
 FI: 4501.000 Hz  
 F2P: 2.500 ppm  
 F3P: 0.000 ppm  
 FREQCN: 0.41667 ppm/cm  
 HZCN: 2.0042502 Hz/cm



Signal 1: MWD1 A, Sig=250,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.699	VV R	0.7747	2.27162e4	447.14914	50.0210
2	29.828	VB	0.8196	2.26971e4	407.65872	49.9790

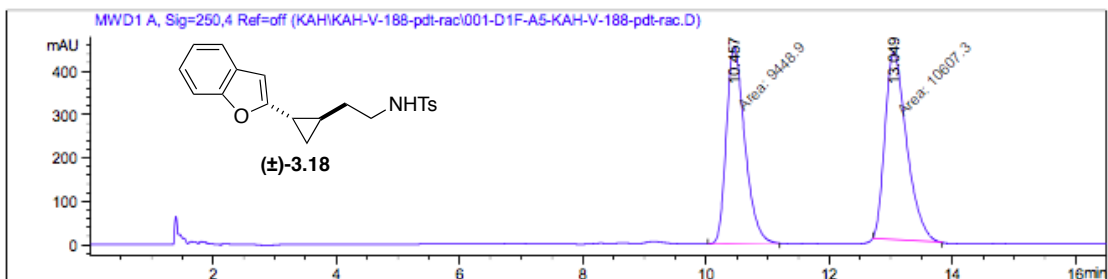
Totals : 4.54133e4 854.80786



Signal 1: MWD1 A, Sig=250,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.693	BV	0.7571	1.81977e4	353.17920	50.3969
2	29.852	VV R	0.8092	1.79111e4	324.90347	49.6031

Totals : 3.61088e4 678.08267

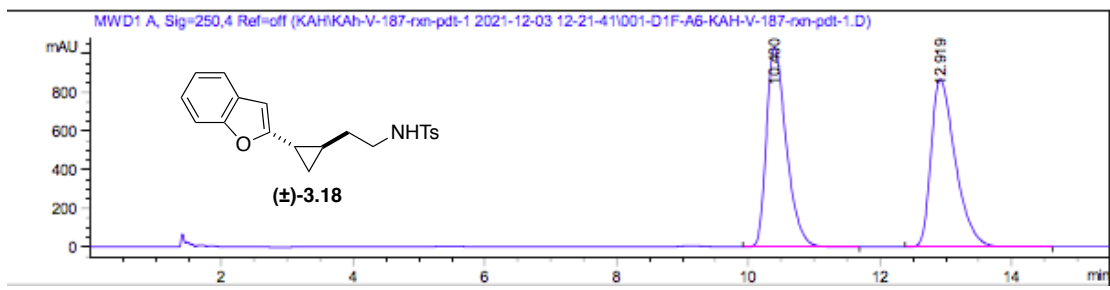


Signal 1: MWD1 A, Sig=250,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.457	MM	0.3468	9448.90332	454.14722	47.1121
2	13.049	MM	0.4096	1.06073e4	431.57236	52.8879

Totals : 2.00562e4 885.71957





Signal 1: MWD1 A, Sig=250,4 Ref=off

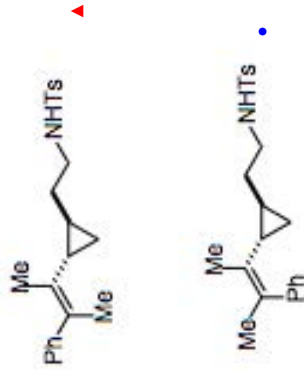
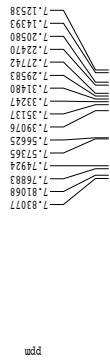
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.400	BB	0.3222	2.15896e4	1031.54016	49.3558
2	12.919	BV R	0.3899	2.21532e4	867.65588	50.6442

Totals : 4.37428e4 1899.19604

### APPENDIX A.3

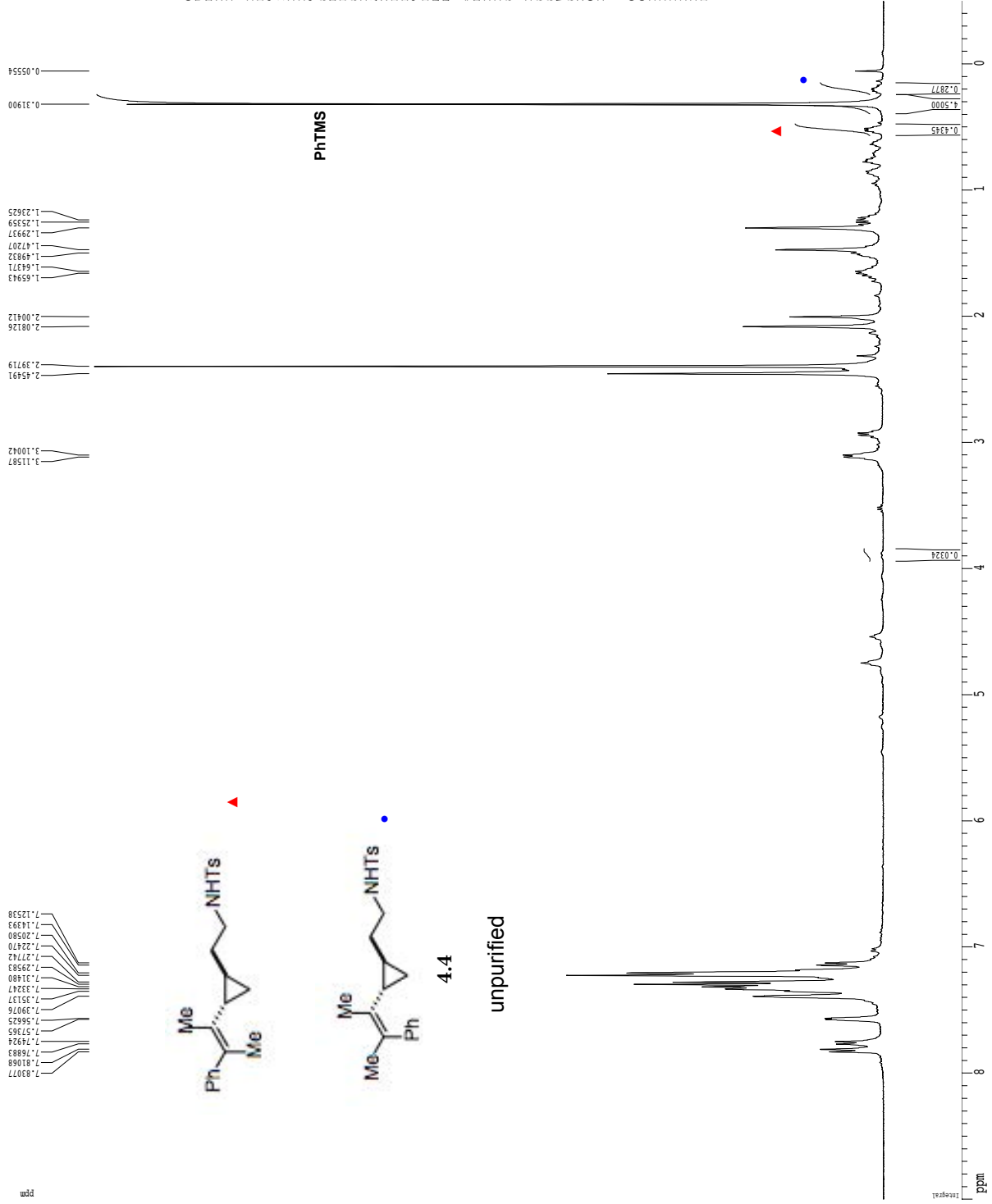
$^1\text{H}$ ,  $^{13}\text{C}$ , COSY, NOE NMR SPECTRA, SFC TRACES FOR CHAPTER 4

1H spectrum



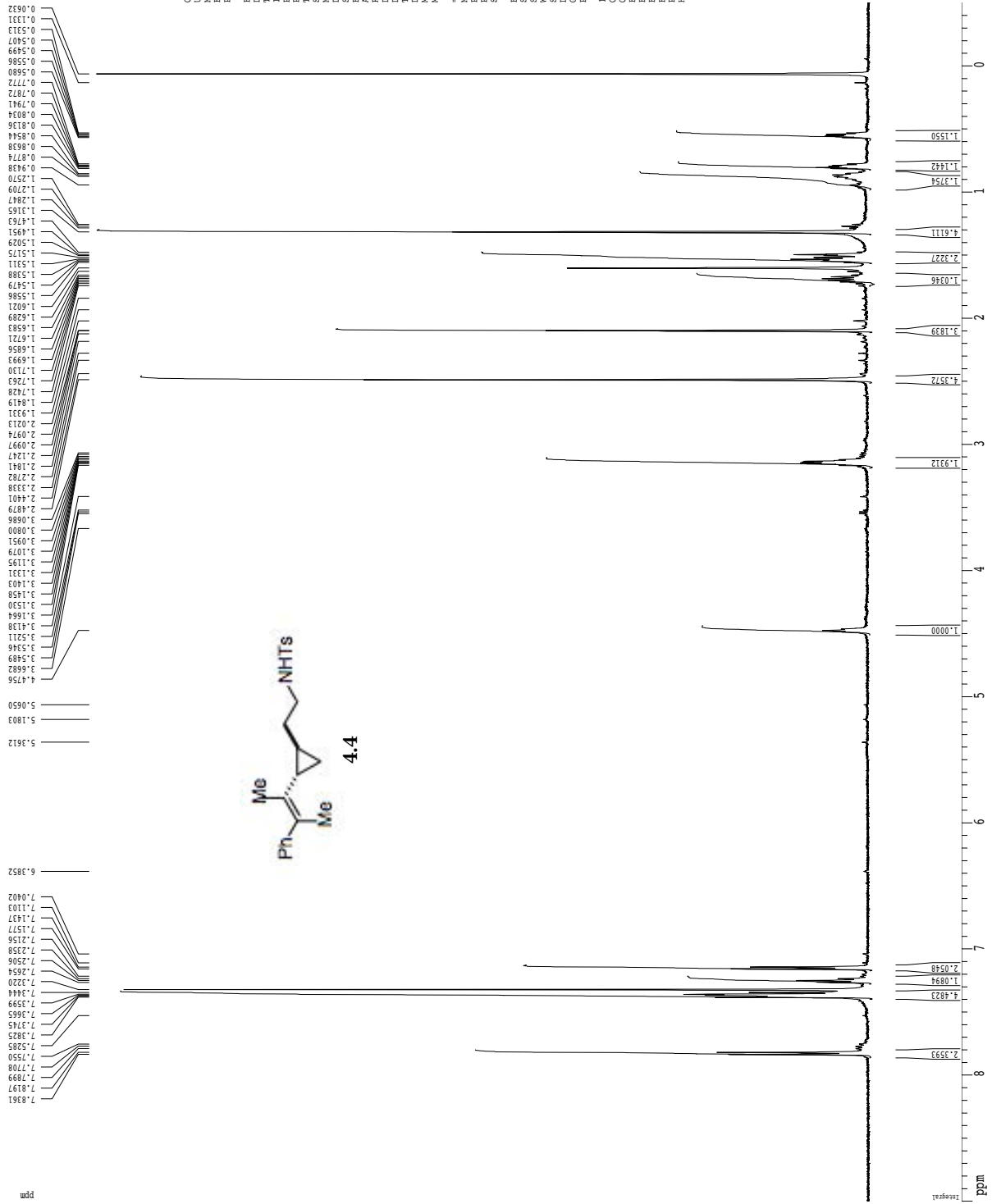
4.4

unpurified



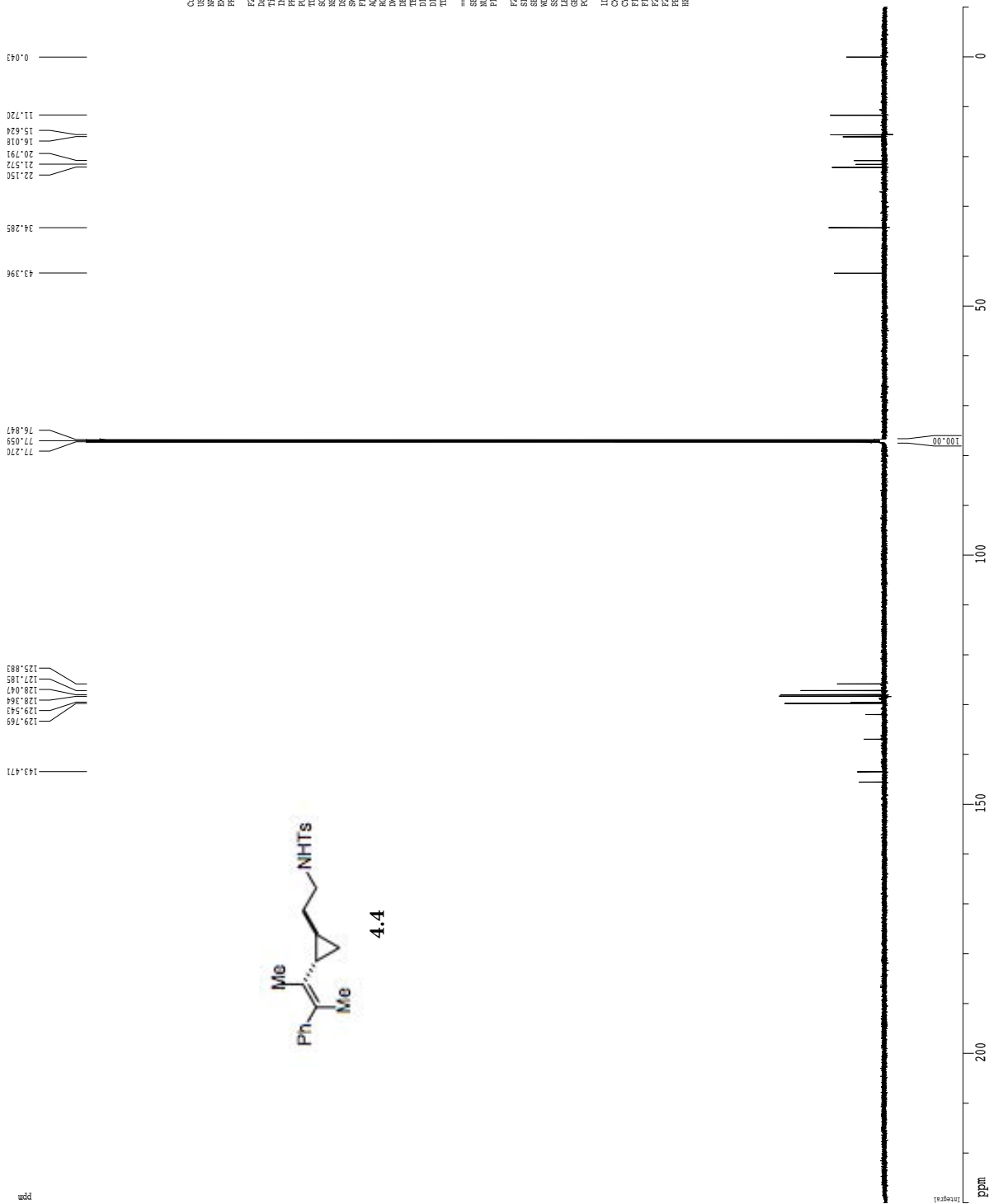
Current Data Parameters  
 USER: Komett  
 NAME: TMT150  
 EXNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20200630  
 Time: 10.11  
 PROBHD: 5 mm QNP 1H/1  
 PULPROG: zg30  
 TD: 65536  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 400.1300175 MHz  
 SFE: 65536  
 FIDRES: 0.097813 Hz  
 AQ: 5.1118579 sec  
 RG: 78.000  
 DE: 4.50 usec  
 TE: 297.2 K  
 T1: 0.10000000 sec  
 T2: 0.00000000 sec  
 T3: 0.00000000 sec  
 MCHRG: 0.01500000 sec  
 MCHRG: 0.01500000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 1H  
 P1: 12.00 usec  
 PL1: -1.10 dB  
 SFO1: 400.1300175 MHz  
 F2 - Processing Parameters  
 SI: 65536  
 SF: 400.1300175 MHz  
 WDM: no  
 LB: 0.00 Hz  
 GB: 0  
 PC: 2.00  
 ID: NMR file parameters  
 CY: 22.80 cm  
 CV: 15.00 cm  
 FIP: 9.000 ppm  
 FL: 3600.17 Hz  
 F2: -200.00 Hz  
 F3: -200.00 Hz  
 PPMCH: 0.41667 ppm/cm  
 HZCH: 166.72084 Hz/cm

1H spectrum



Current Data Parameters  
 USER KMH-III-031-2-CY0  
 KNOW111  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20201117  
 Time\_ 17:55  
 INSTRUM cryo500  
 PROBHD 5 mm CPCL 1H-  
 PULPROG zgpg30  
 SFO 500.136198  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 8012.2 Hz  
 FIDRES 0.164673 Hz  
 AQ 2.9998677 sec  
 RG 62.7  
 DE 6.40 usec  
 TE 298.4 K  
 D1 0.1000000 sec  
 d11 0.0500000 sec  
 ACQPRG 1  
 XCPROG 0.0150000 sec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 1.50 usec  
 PL1 1.60 dB  
 SFO1 500.233015 MHz  
 F2 - Processing Parameters  
 SI 65336  
 SF 500.220000 MHz  
 WDW 0  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00  
 ID NMR pLOT parameters  
 CX 22.80 cm  
 CT 1.50 cm  
 CIP 9.000 Hz  
 F1 450.198 Hz  
 F2 -0.500 ppm  
 FZP 0.000 Hz  
 FWHM 0.4165 Hz/cm  
 HZCX 208.43500 Hz/cm

1H spectrum



Current Data Parameters  
 USER Rhowell  
 SAMPLE KM-11-011-000  
 EXNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20080220  
 Time 11:25  
 INSTRUM av600  
 PROBHD 5 mm CPBBO  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 718  
 SWH 30231.483 Hz  
 FIDRES 0.532485 Hz  
 AQ 0.5944468 sec  
 RG 655  
 DM 13.100 usec  
 DE 19.63 usec  
 TE 298.0 K  
 D1 1.11 sec  
 D11 0.4300000 sec  
 TDD 1  
 ===== CHANNEL f1 =====  
 SFO1 150.9194000 MHz  
 NUC1 13C  
 P1 10.10 usec  
 F2 - Processing parameters  
 SI 65536  
 SF 150.9028085 MHz  
 NQ 1  
 SFO 150.9028085 MHz  
 LB 0.400 Hz  
 GB 0  
 PC 1.00  
 ID NO plot parameters  
 CX 22.80 cm  
 CT 24.30 cm  
 F1 27.15 Hz  
 F2 34715.72 Hz  
 F3 10.00 Hz  
 F4 -10.489 ppm  
 F5 -15.647 Hz  
 F6 10.53074 ppm/cm  
 F7 13.611100 Hz/cm



4.4

gc05y60

```

Current Data Parameters
NAME      khewitt1
EXPNO    2
PROCNO   1

F2 - Acquisition Parameters
Date_    20200117
Time     17.57
INSTRUM  cryo500
PROBHD   5 mm cryo
PULPROG  zgpg30
TD        2048
SOLVENT  CDCl3
NS        1
DS        1
AQ        8032.82 Hz
FIDRES    3.912510 Hz
AQ        0.1278452 sec
RG         812.7
DM         62.400 usec
DE         6.00 usec
TE        299.2 K
d0         0.0000300 sec
d1         1.0000000 sec
d13        0.0000300 sec
d16        0.0002000 sec
IN0        0.0012460 sec

===== CHANNEL f1 =====
NUC1      1H
P1        7.50 usec
PL1       1.60 dB
SFO1      500.225015 MHz

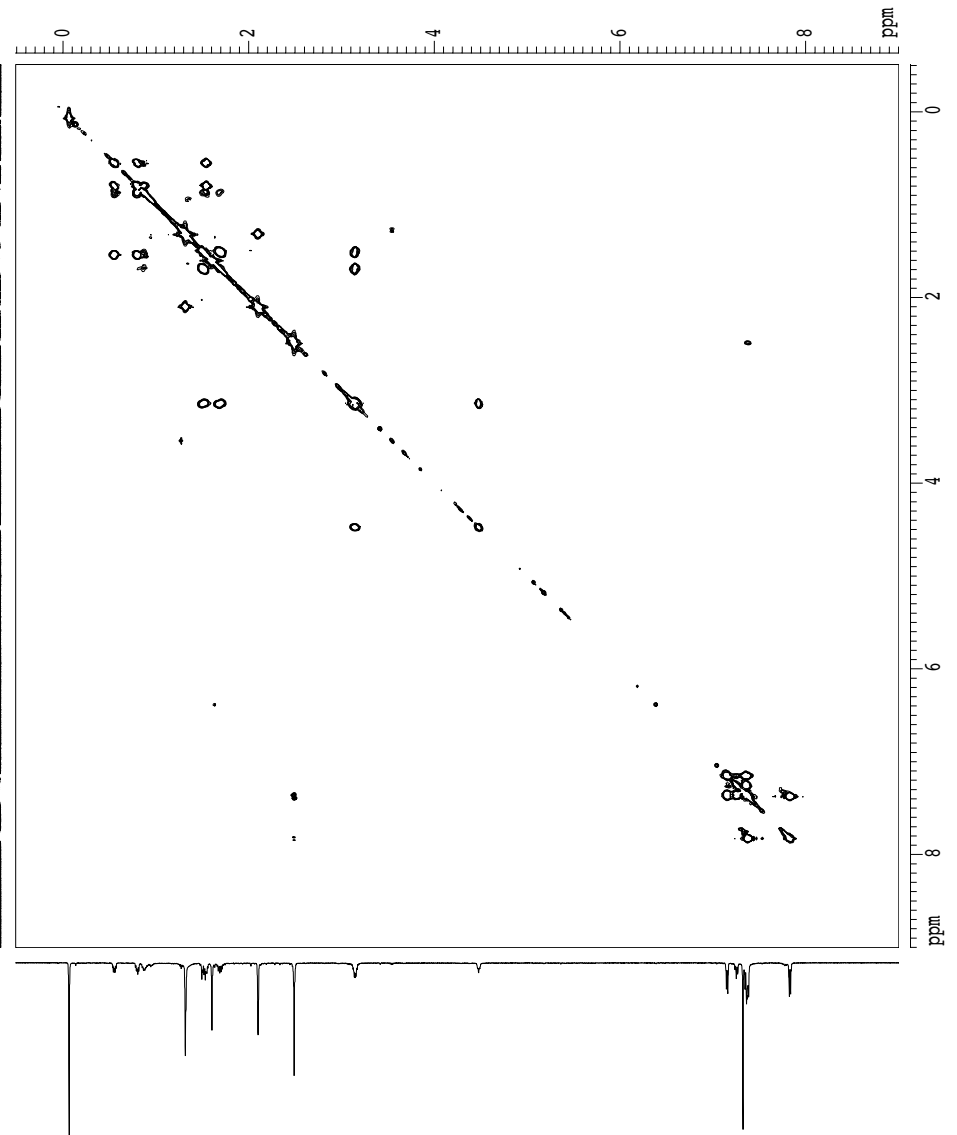
===== GRADIENT CHANNEL =====
GPM1M1    size:100
GPM1M2    size:100
GPR1       0.00 %
GPR2       0.00 %
GPR3       0.00 %
GPR4       0.00 %
GPR5       17.00 %
GPR6       17.00 %
P16        1000.00 usec

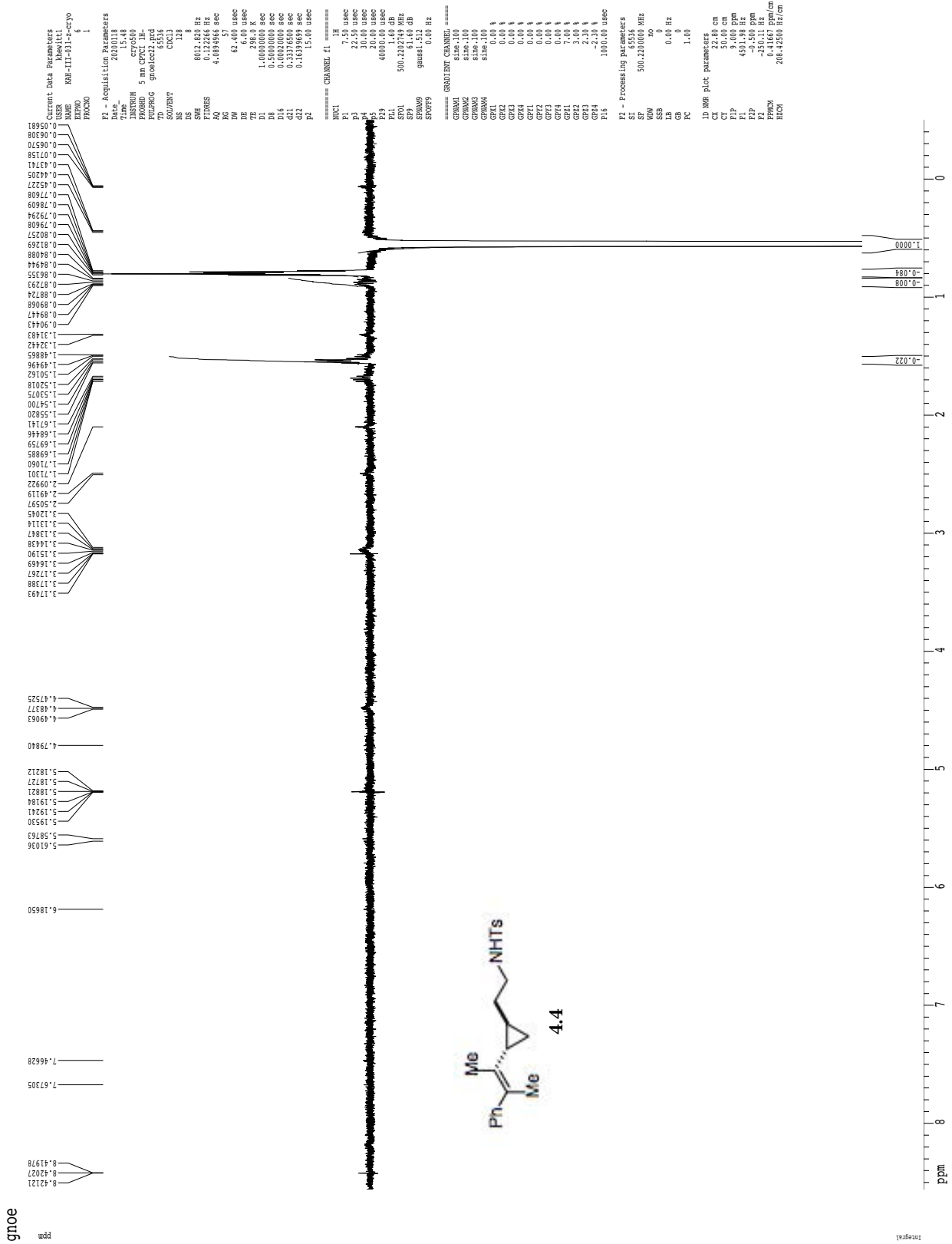
F1 - Acquisition parameters
NU0        1
TD          512
SFO1       500.225 MHz
FIDRES     15.650040 Hz
SMA        16.08 ppm
WDW         GF
SSB         0
LB          0
GB          0
PC          1.00

F2 - Processing parameters
SI         500.220000 MHz
SF         500.220000 MHz
WDW         SINE
SSB         0
LB          0
GB          0
PC          1.00

F1 - Processing parameters
SI         500.220000 MHz
SF         500.220000 MHz
WDW         SINE
SSB         0
LB          0
GB          0

2D NMR plot parameters
CX2        15.00 cm
CY2        15.00 cm
FZ20       450.00 Hz
F2PHI      -0.509 ppm
F2PHI      -254.47 Hz
F1FLO      9.002 ppm
F1FLO      4503.14 Hz
F1F1       4.501 ppm
F1F1       -257.24 Hz
F2F2PACH   0.63407 ppm/cm
F2F2PACH   317.17416 Hz/cm
F1F2PACH   0.63407 ppm/cm
F1F2PACH   317.17416 Hz/cm
  
```





gnoe

ppm

7.15718  
7.14350

1.54746  
1.53942  
1.5309  
1.51040

Current Data Parameters  
USER KheWatt1  
NAME KM-III-031-2-cr-tyo  
PROCNO 7  
P0000 1

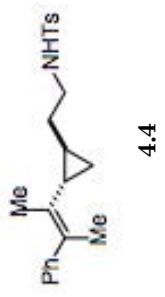
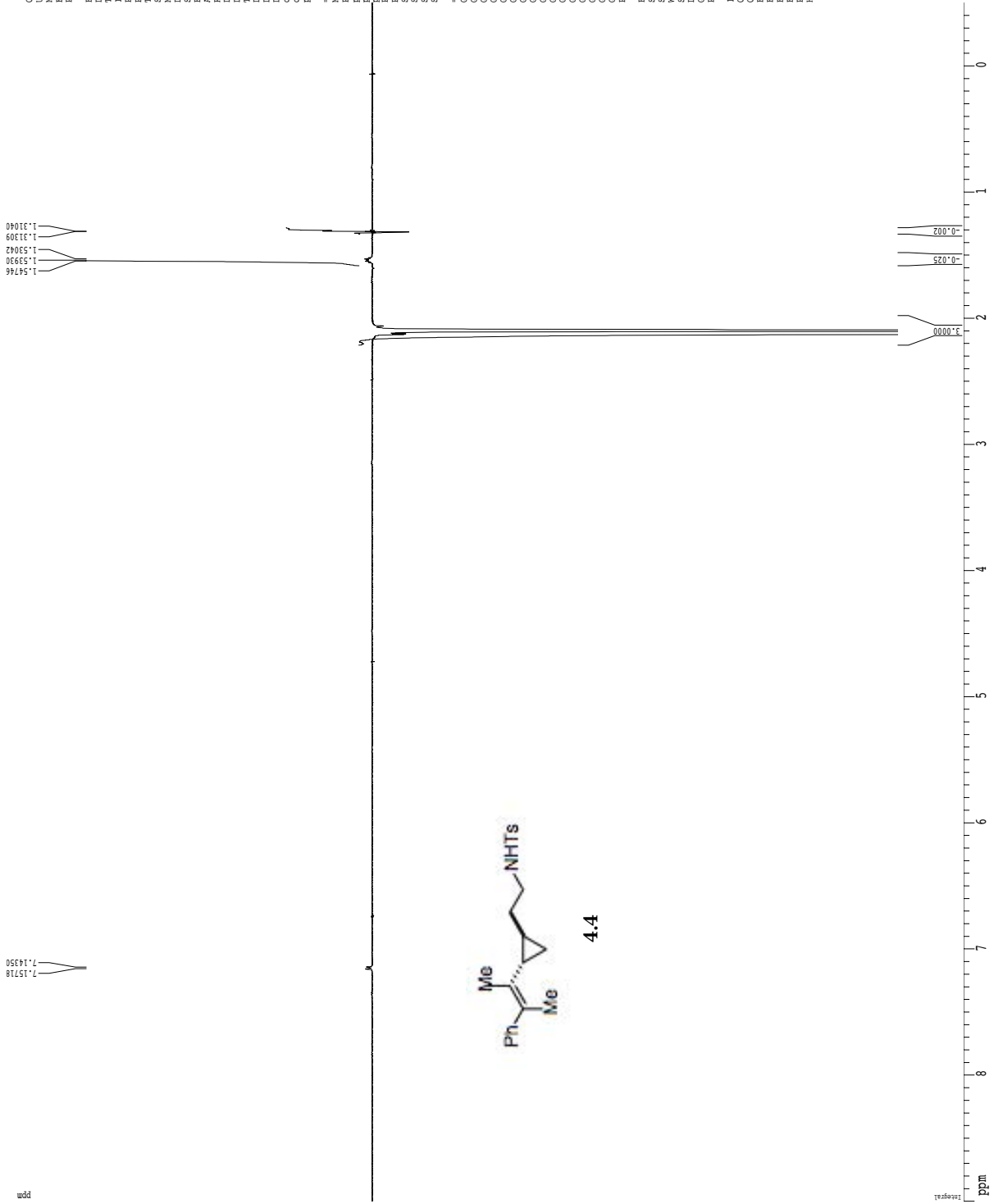
F2 - Acquisition Parameters  
Date\_ 2010-03-08  
Time 16:02  
INSTRUM cryo500  
PROBHD 5 mm CPCLP1H-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 128  
DS 4  
SWH 8012.820 Hz  
FIDRES 0.122464 Hz  
AQ 4.189496 sec  
RG 62.400 us/ac  
DE 6.00 us/ac  
TE 298.2 K  
F1 1.000000 sec  
D8 0.500000 sec  
D16 0.002000 sec  
d21 0.3337500 sec  
d22 0.1633750 sec  
F2 15.00 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 7.50 us/ac  
P3 22.50 us/ac  
P4 30.00 us/ac  
P5 4000.00 us/ac  
P6 4000.00 us/ac  
P7 1.40 dB  
SFO1 500.221095 MHz  
SFO2 125.00 MHz  
SFO3 157.50 MHz  
SFOFF9 0.00 Hz

===== GRADIENT CHANNELS =====  
GRAN1 4.00 MHz  
GRAN2 size:100  
GRAN3 size:100  
GRAN4 size:100  
GR5 size:100  
GR6 0.00 V  
GR7 0.00 V  
GR8 0.00 V  
GR9 0.00 V  
GR10 0.00 V  
GR11 0.00 V  
GR12 0.00 V  
GR13 0.00 V  
GR14 0.00 V  
GR15 1.00 V  
GR16 1.00 V  
GR17 2.30 V  
GR18 2.30 V  
GR19 1000.00 us/ac  
F16

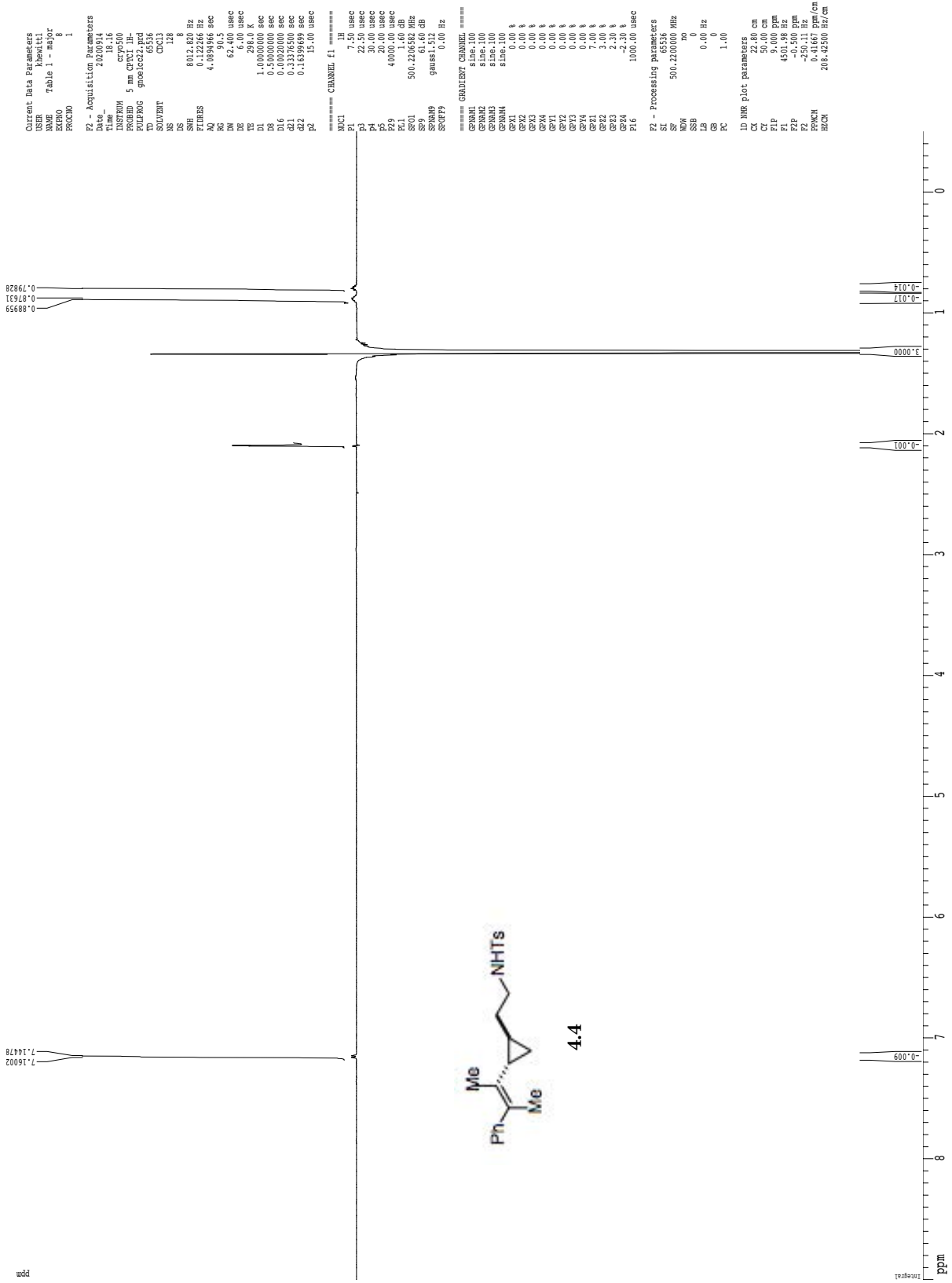
F2 - Processing parameters  
SI 6558  
SF 500.221095 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

ID NAME parameters  
CY 516.00 cm  
PIP 9.400 ppm  
FL 450.198 Hz  
F2 250.11 Hz  
F2 250.11 Hz  
FPMON 0.41667 ppm/cm  
HCON 200.42500 Hz/cm



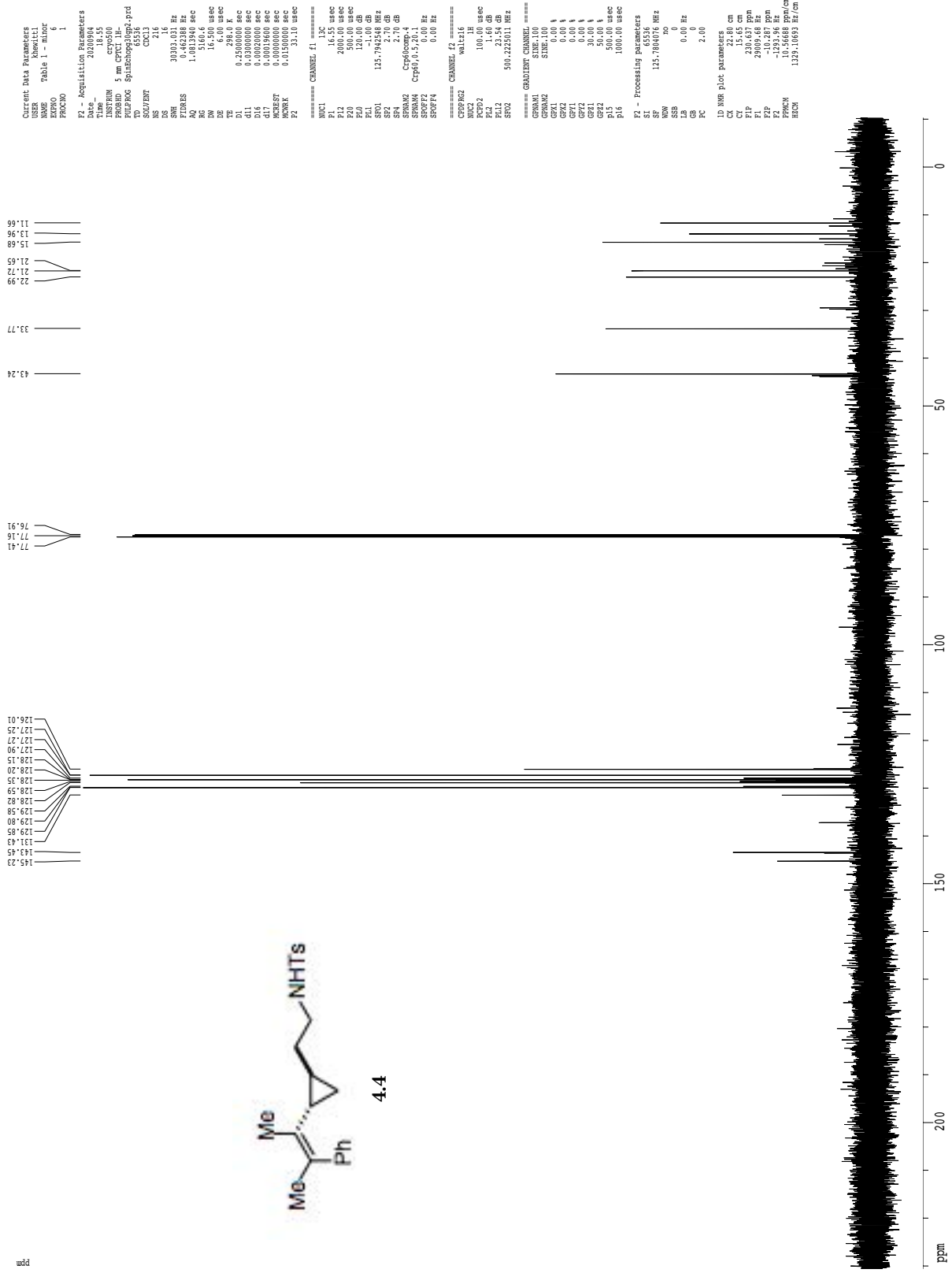


gnoe





Z-restored spin-echo 13C spectrum with 1H decoupling





4.4

gcosy60

```

Current Data Parameters
USER          Khevit1
NAME          Table 1 - minor
EXPNO         5
PROCNO        1

F2 - Acquisition Parameters
Date_         20200904
Time          18.43
INSTRUM       cryo500
PROBHD        5 mm cryo1H/1
PULPROG       zgpg30
TD            2048
SOLVENT       CDCl3
NS            1
DS            16
AQ            8013.85 Hz
FIDRES        3.912510 Hz
RG            0.1278452 sec
AQ            0.1278452 sec
RG            287.4
DM            62.400 usec
DE            288.0 usec
TE            298.0
D0            0.0000300 sec
d1            1.0000000 sec
d13           0.0000300 sec
d16           0.0002000 sec
d10           0.0001400 sec
D10           0.0001400 sec

===== CHANNEL f1 =====
NUC1          1H
P1            7.50 usec
PC            0.00 usec
SFO1          500.225015 MHz

===== GRADIENT CHANNEL =====
GPRM1        sine,100
GPRM2        sine,100
GPRM3        sine,100
GPZ1         0.00 %
GPZ2         0.00 %
GPZ3         0.00 %
GPZ4         0.00 %
GPZ5         17.00 %
GPZ6         17.00 %
P16          1000.00 usec

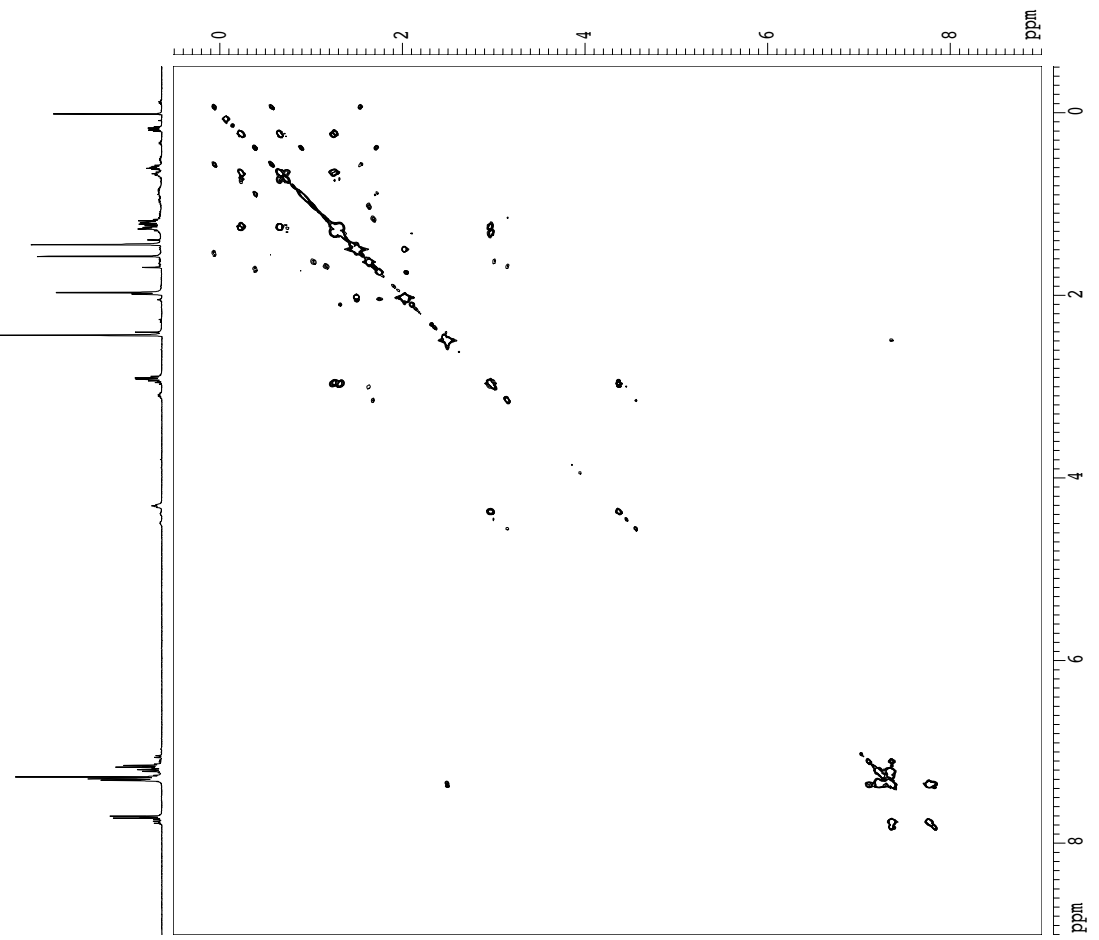
F1 - Acquisition parameters
NUC1          1H
P1            7.50 usec
PC            0.00 usec
SFO1          500.225015 MHz

===== GRADIENT CHANNEL =====
GPRM1        sine,100
GPRM2        sine,100
GPRM3        sine,100
GPZ1         0.00 %
GPZ2         0.00 %
GPZ3         0.00 %
GPZ4         0.00 %
GPZ5         17.00 %
GPZ6         17.00 %
P16          1000.00 usec

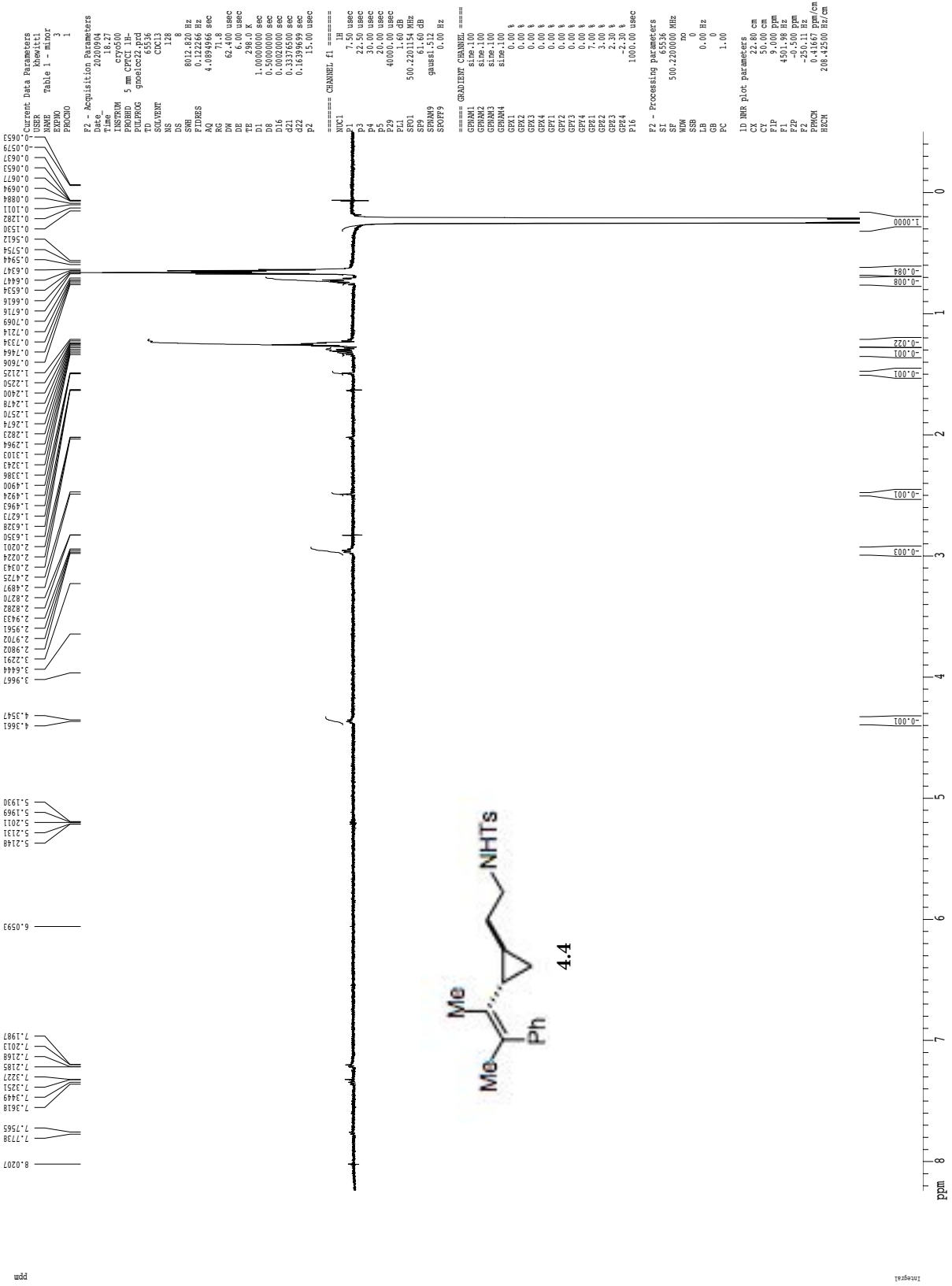
F2 - Processing parameters
SI            1024
SF            500.2200000 MHz
WDW           EM
SSB           0
GB            0.00 Hz
PC            1.00

F1 - Processing parameters
SI            1024
SF            500.2200000 MHz
WDW           EM
SSB           0
GB            0.00 Hz
PC            1.00

2D NMR plot parameters
CX2           15.00 cm
CX1           15.00 cm
F2PLO        5.002 ppm
F2FID        15.002 ppm
F2PHI        40.569 deg
F2HF         -254.47 Hz
F1PLO        9.002 ppm
F1FID        4503.14 Hz
F1PHI        0.509 ppm
F1PC         2.000 ppm
F2PRGCM      0.63407 ppm/cm
F2PRCHM      317.17416 Hz/cm
F1PRGCM      0.63407 ppm/cm
F1PRCHM      317.17416 Hz/cm
  
```

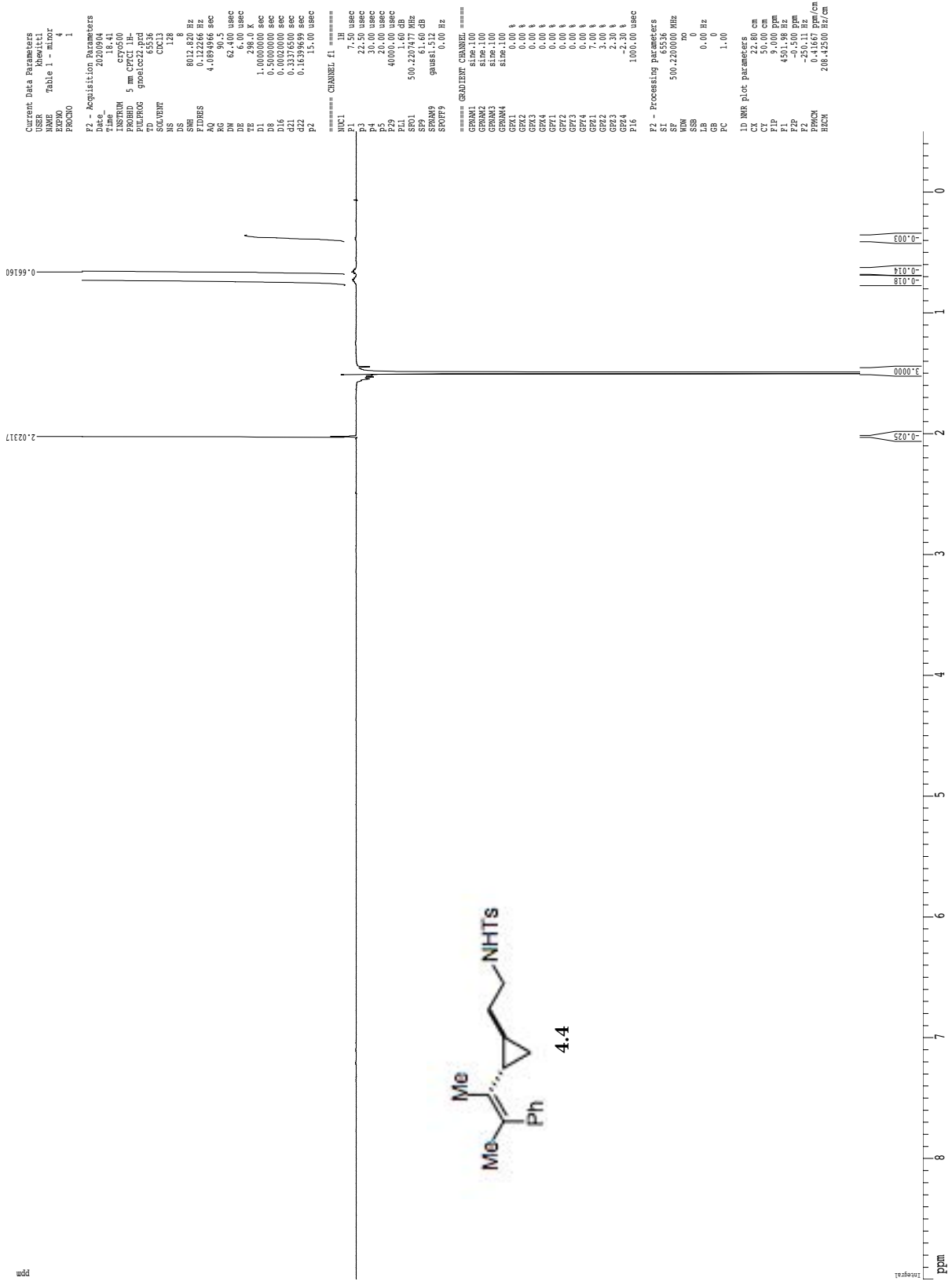


gnoe



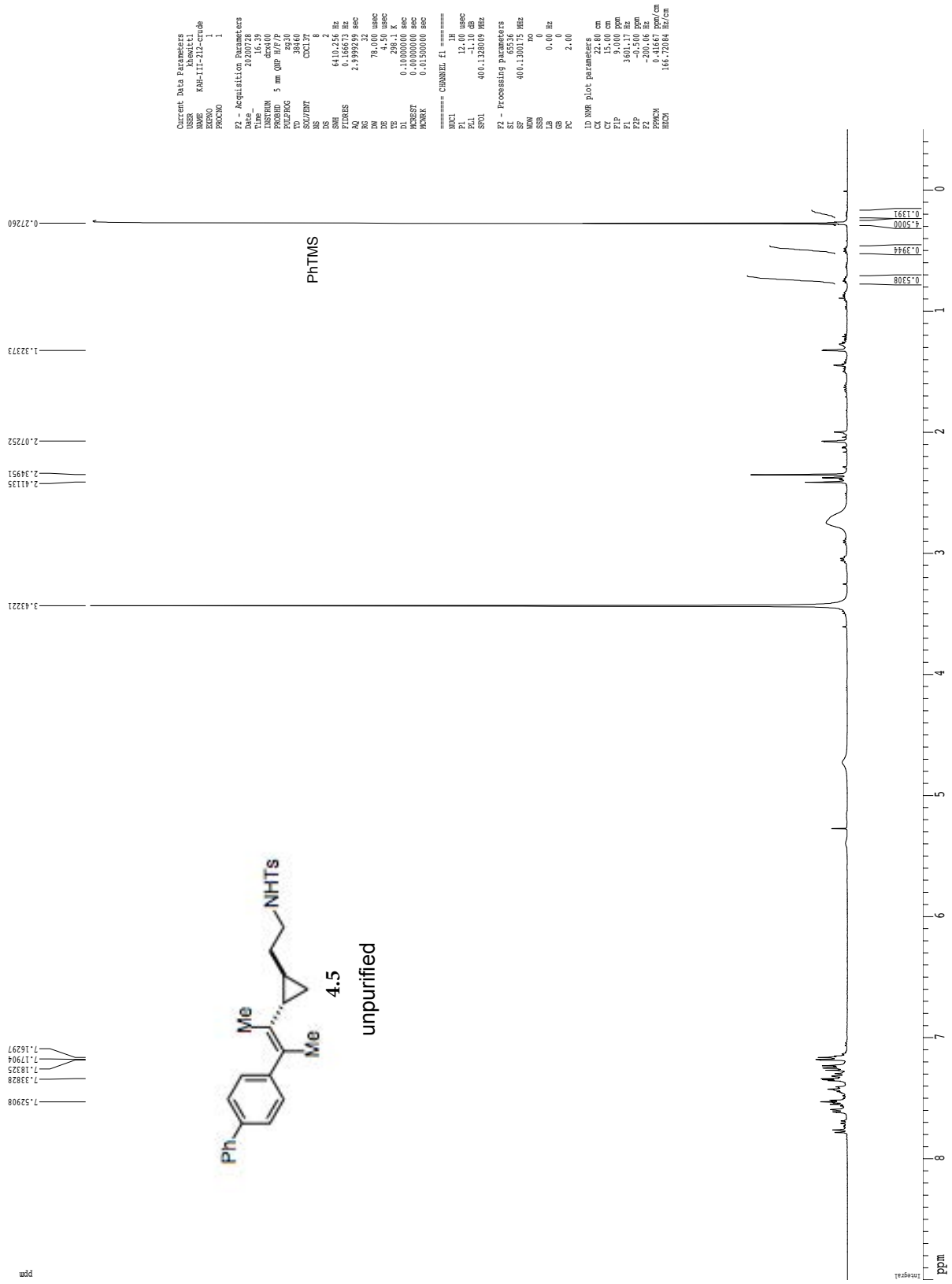
gnoe

100





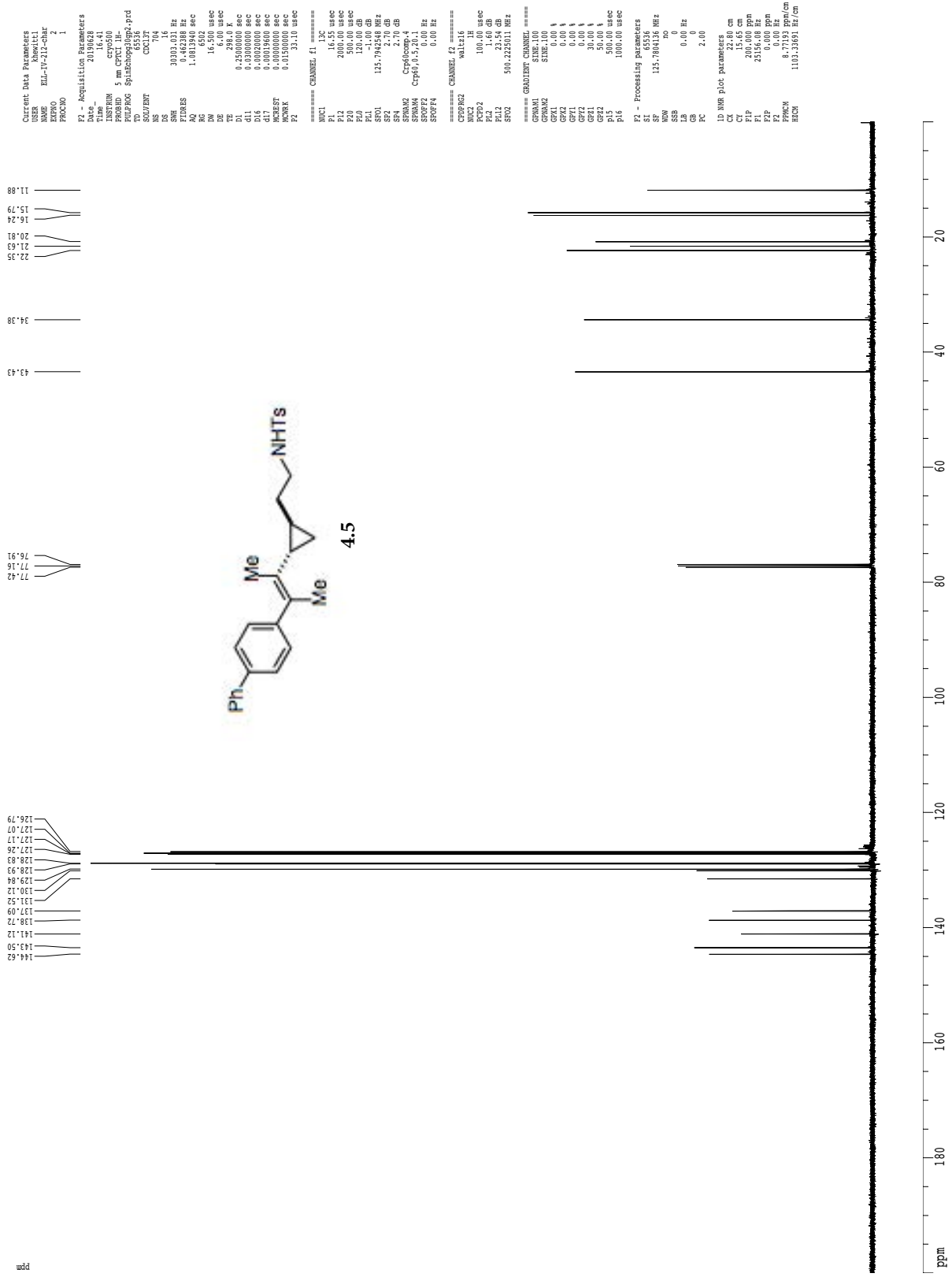
1H spectrum

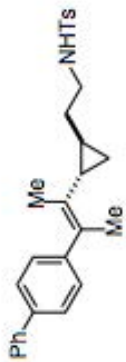






# Z-restored spin-echo 13C spectrum with 1H decoupling





gcosy60

4.5

```

Current Data Parameters
NAME      Khowit1
EXPNO    3
PROCNO   1

F2 - Acquisition Parameters
Date_    20190309
Time     11:35
INSTRUM  spect
PROBHD   5 mm CPXI 1H
PULPROG  zgpg30
TD       2048
SOLVENT  CDCl3
NS       15
DS       4
SWH      4734.849 Hz
FIDRES   2.311938 Hz
AQ       0.2163188 sec
RG       114
WDW      EM
SSB      0
GB       0
TE       298.0 K
DE       0.0000300 sec
d0       1.0000000 sec
d1       0.0000000 sec
d2       0.0000000 sec
d3       0.0000000 sec
d4       0.0000000 sec
d5       0.0000000 sec
d6       0.0000000 sec
===== CHANNEL f1 =====
NUC1     1H
P1       12.00 nsec
PL1      0.00 dB
SFO1     500.2221637 MHz

===== GRAB CHANNEL =====
GRAB1    sine-100
GRAB2    sine-100
GRX2     0.00 %
GRY2     0.00 %
GRZ2     0.00 %
GR42     0.00 %
GR52     17.00 %
GR62     17.00 %
GR72     17.00 %
GR82     17.00 %
GR92     17.00 %
GR16     1000.00 usec

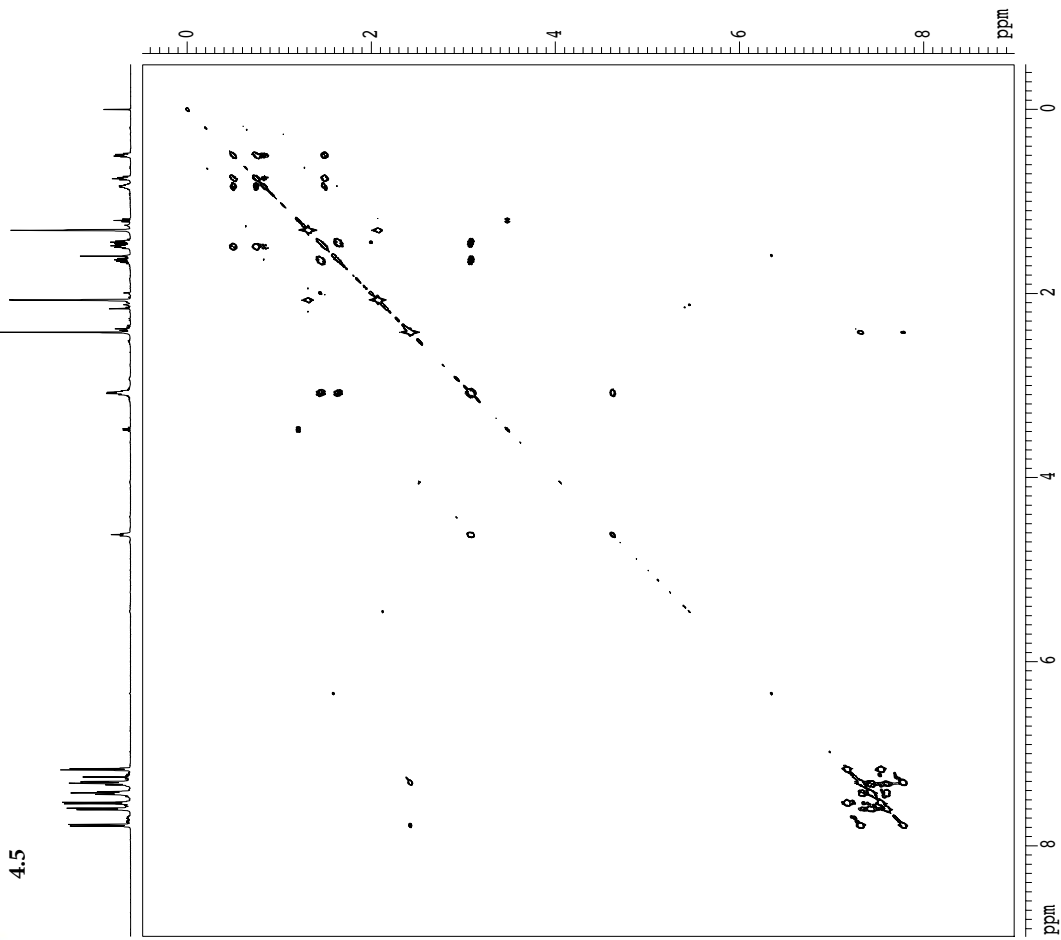
F1 - Acquisition Parameters
NUC1     1H
P1       12.00 nsec
PL1      0.00 dB
SFO1     500.2221637 MHz

===== GRAB CHANNEL =====
GRAB1    sine-100
GRAB2    sine-100
GRX2     0.00 %
GRY2     0.00 %
GRZ2     0.00 %
GR42     0.00 %
GR52     17.00 %
GR62     17.00 %
GR72     17.00 %
GR82     17.00 %
GR92     17.00 %
GR16     1000.00 usec

F2 - Processing parameters
SI       1024
SF       500.2200368 MHz
WDW      EM
SSB      0
GB       0
PC       1.00

F1 - Processing parameters
SI       1024
SF       500.2200368 MHz
WDW      EM
SSB      0
GB       0
PC       1.00

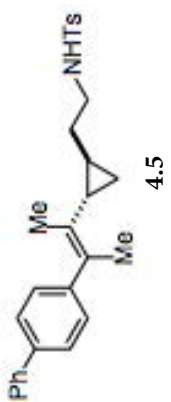
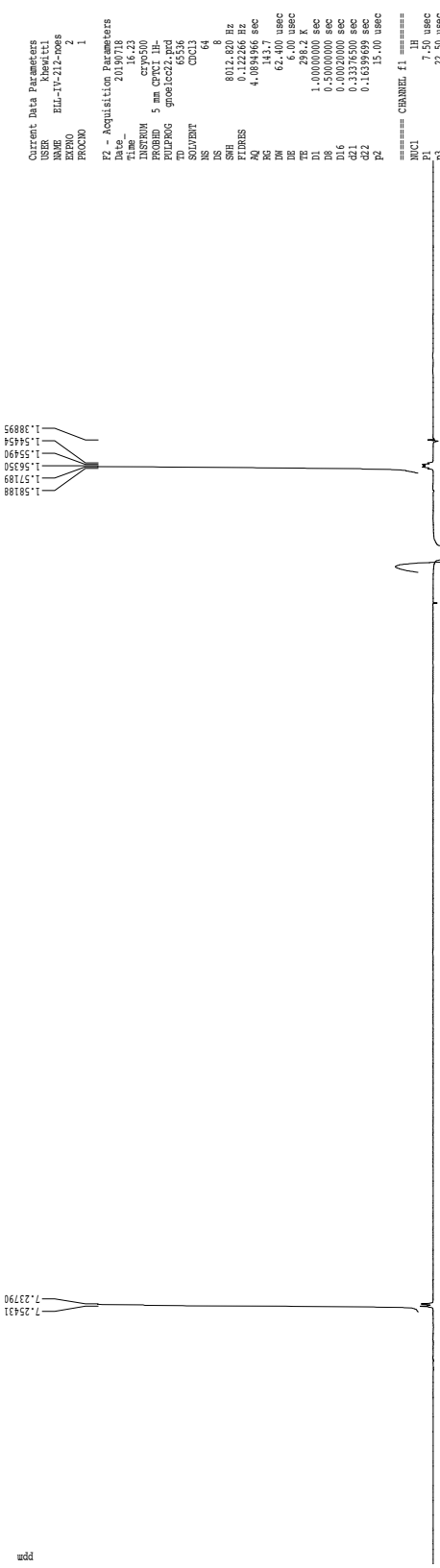
2D NMR plot parameters
CX2      15.00 cm
CX1      15.00 cm
F2FLO    8.583 ppm
F2FID    440.483 ppm
F2F1F2   -241.49 Hz
F1FLO    8.583 ppm
F1FID    4493.36 Hz
F1F1F2   -1.83 ppm
F2PRECH  0.63104 ppm/cm
F2F2CH   315.65656 Hz/cm
F1PRECH  0.63104 ppm/cm
F1F1CH   315.65656 Hz/cm
  
```





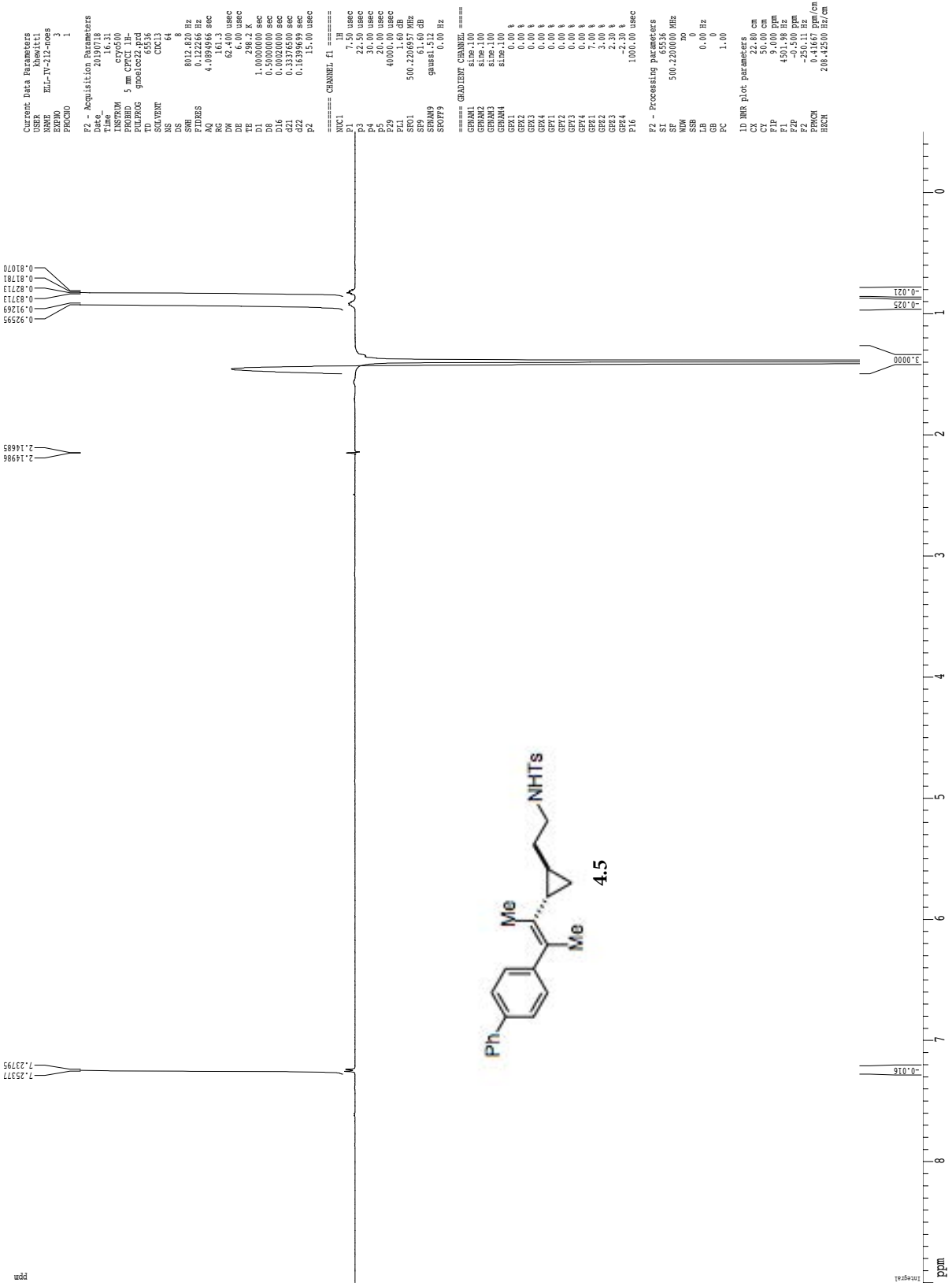
gnoe

ppm

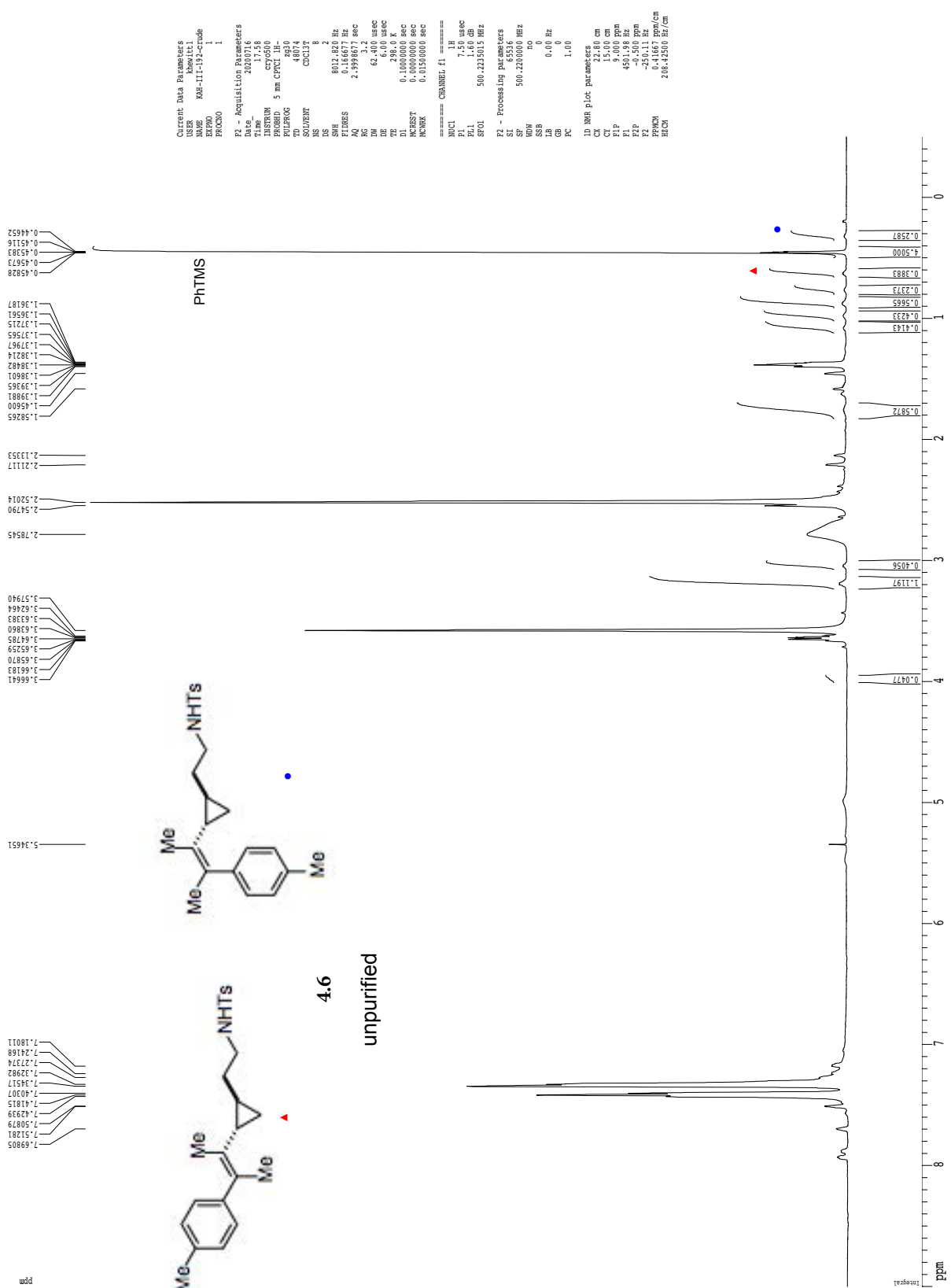


```
Current Data Parameters
USER      khovatt1
NAME      ELL-IV-212-noes
EXPNO     2
PROCNO    1
-----
F2 - Acquisition Parameters
Date_     20190718
Time      16:23
PROBHD    5 mm CPDCT 1H
PULPROG   gnoe1cc22.prd
TD         65536
SOLVENT   CDCl3
NS         6
DS         4
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0893966 sec
RG         1800
DE         62.400 usec
TE         298.2 K
D1         1.0000000 sec
DELTA     0.1800000 sec
D06        0.0020000 sec
d21        0.33375000 sec
d22        0.16398699 sec
P2         15.00 usec
-----
===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
P2         22.50 usec
P3         20.00 usec
P4         20.00 usec
E29        40000.00 usec
PL1        1.66 dB
SFO1       500.221374 MHz
G1         6.00 dB
SFO2       500.136199 MHz
G2         6.00 dB
SFO3       500.136199 MHz
SFO4       500.136199 MHz
SFO5       500.136199 MHz
===== GRADIENT CHANNEL =====
G1A1       1000.00 usec
G1A2       1000.00 usec
G1B1       1000.00 usec
G1B2       1000.00 usec
G1B3       1000.00 usec
G1B4       1000.00 usec
G1B5       1000.00 usec
G1B6       1000.00 usec
G1B7       1000.00 usec
G1B8       1000.00 usec
G1B9       1000.00 usec
G1B10      1000.00 usec
G1B11      1000.00 usec
G1B12      1000.00 usec
G1B13      1000.00 usec
G1B14      1000.00 usec
G1B15      1000.00 usec
G1B16      1000.00 usec
G1B17      1000.00 usec
G1B18      1000.00 usec
G1B19      1000.00 usec
G1B20      1000.00 usec
-----
F2 - Processing parameters
SI         65536
SF         500.221374 MHz
WDW        EM
SSB        0
LB         0.00 Hz
GB         0
PC         1.00
-----
ID NMR plot parameters
CX         22.88 cm
CY         50.00 cm
CP         14.00 cm
FIDRES     0.122266 ppm
F2P        -0.500 ppm
F2         -2.5011 Hz
PPH1M      0.41667 ppm/cm
PPH2M      208.42500 Hz/cm
```

gnoe

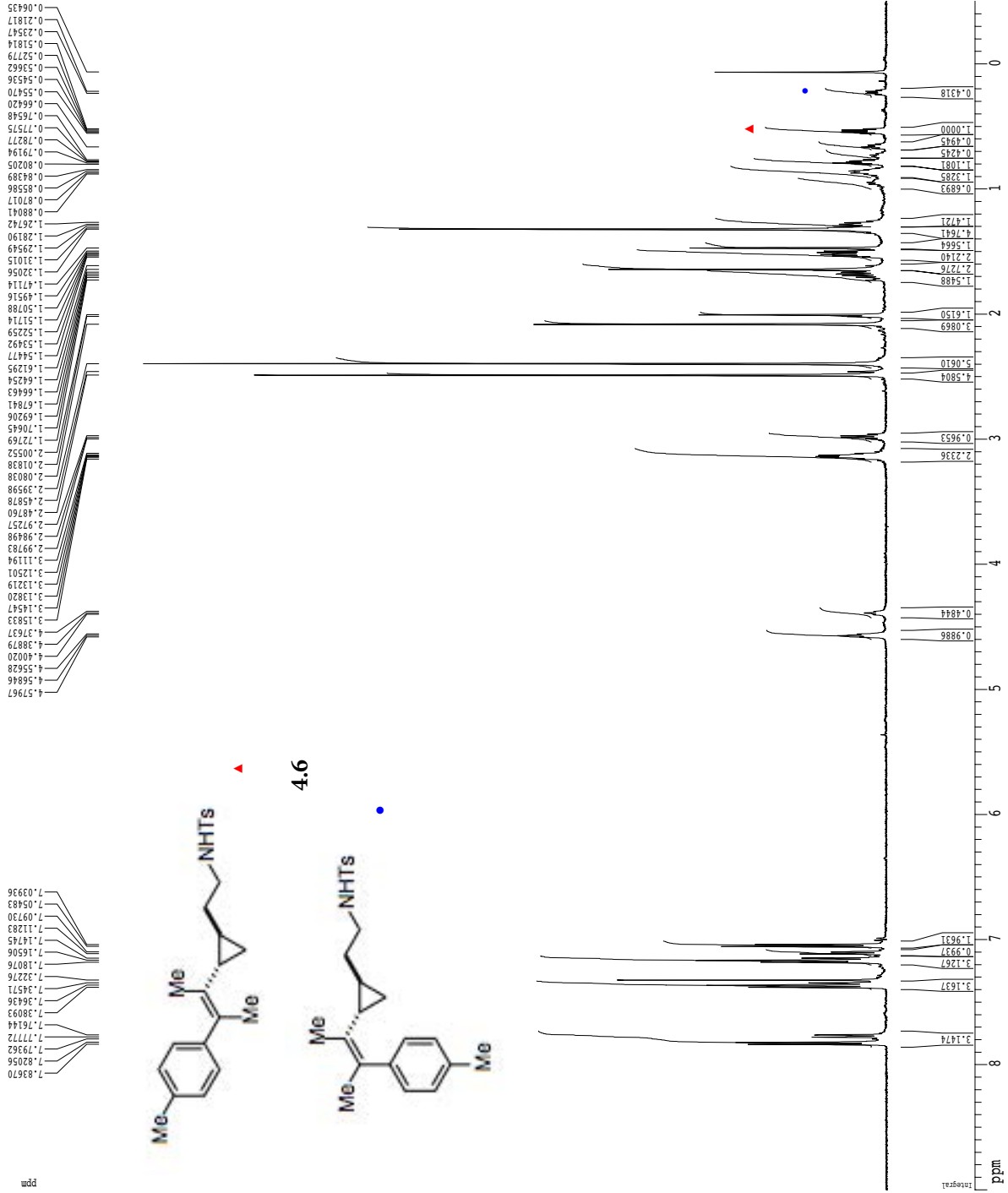


1H spectrum



Current Data Parameters  
 USER: hewitt1  
 NAME: KH-III-192-crude  
 EXPNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20200716  
 Time: 17:38  
 PULPROG: zgpg30  
 PROCNO: 1  
 PULPROG: 5 mm CPZG\_HF-  
 TD: 48074  
 SFO: 400.146  
 AQ: 1.00  
 RG: 2  
 SHF: 801.420 MHz  
 F2: 400.146 MHz  
 F1: 100.626 MHz  
 ZONES: 2  
 NS: 3.2  
 DS: 4  
 SWH: 62.400 MHz  
 FWHM: 2.759867 Hz  
 AQ: 1.00  
 RG: 2  
 DE: 6.00 uSec  
 TE: 300.2  
 DL: 0.1000000 sec  
 ACQRES: 0.4000000 sec  
 PCPRG1: hsqc  
 PCPRG2: 0.0500000 sec  
 ===== CHANNEL f1 =====  
 NUCL1: 1H  
 P1: 7.50 uSec  
 PL1: 0.00 dB  
 SFO1: 500.1325013 MHz  
 F2 - Processing parameters  
 SI: 65536  
 SF: 500.1325000 MHz  
 WDW: EM  
 SSB: 0  
 LB: 0.00 Hz  
 GB: 0  
 PC: 1.00  
 ID: NMR plot parameters  
 CX: 12.80 cm  
 CK: 1.00  
 FLIP: 9.000 gpa  
 F1: 450.138 MHz  
 F2: 49.500 ppm  
 FWHM: 0.41647 ppm/cm  
 HZCEN: 208.42500 Hz/cm

1H spectrum



Current Data Parameters  
 USER khewitt1  
 NAME KAH-III-192-Z  
 EXPNO 9  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20200911  
 Time 13:33  
 INSTRUM gn500  
 PROBHD 5 mm broadband  
 PULPROG zg30  
 TD 48074  
 SOLVENT CDCl3T  
 NS 8  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.9998677 sec  
 RG 912.3  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.4 K  
 D1 0.10000000 sec  
 MCREST 0.00000000 sec  
 MCWRK 0.01500000 sec

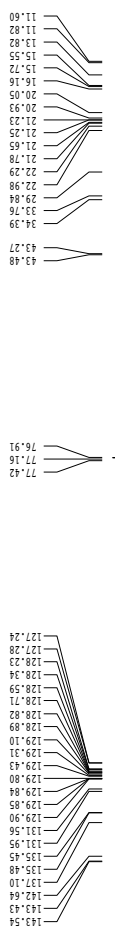
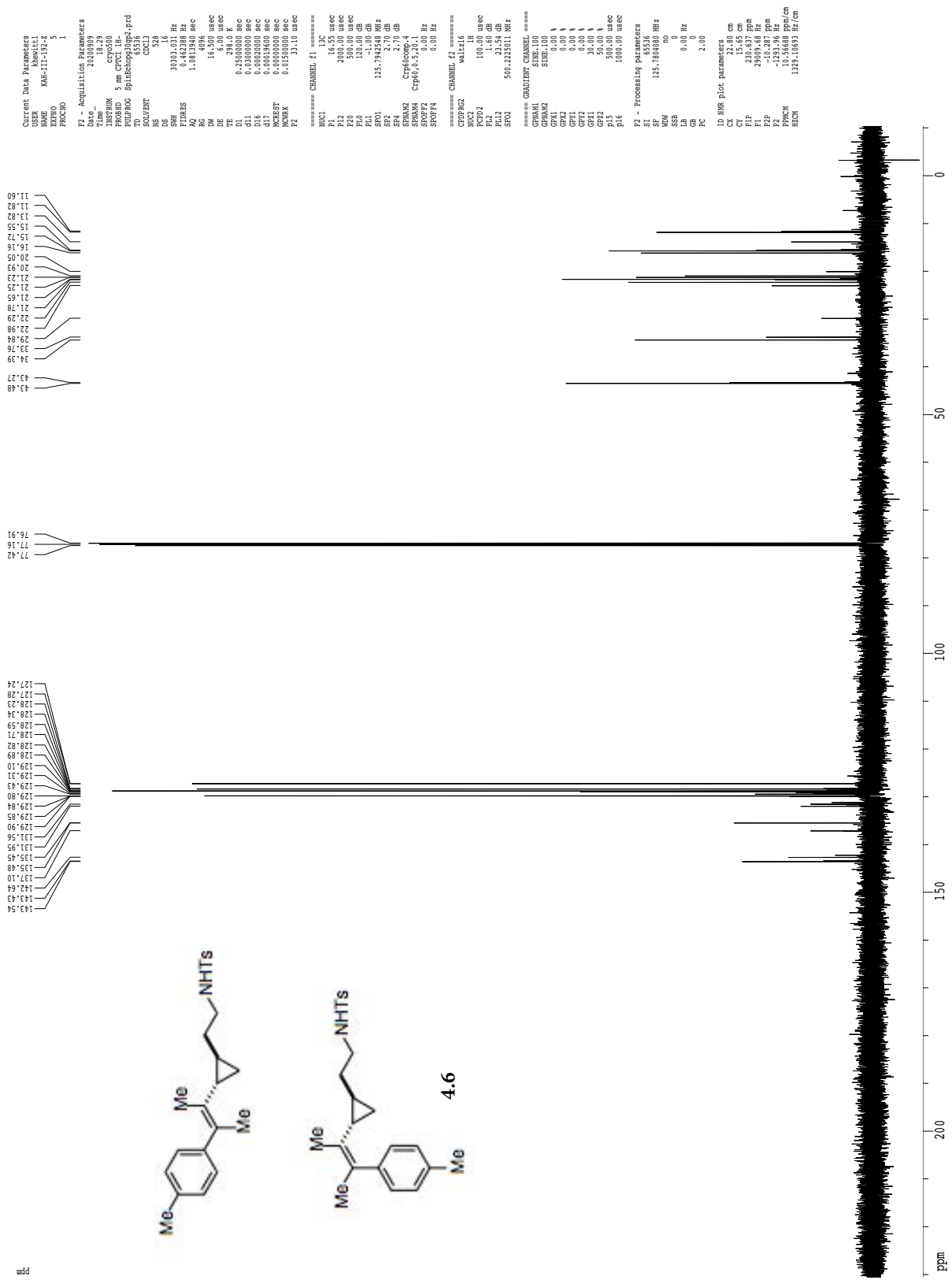
==== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -6.00 dB  
 SF01 498.7534913 MHz

F2 - Processing parameters  
 SI 65536  
 SF 498.7500000 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 12.50 cm  
 FLP 9.000 ppm  
 F1 4488.75 Hz  
 F2P -0.500 ppm  
 F2 -249.38 Hz  
 PPMCM 0.47500 ppm/cm  
 HZCM 236.90625 Hz/cm

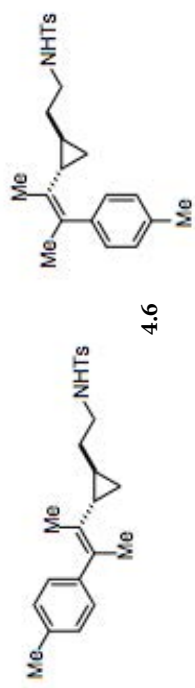


Z-restored spin-echo 13C spectrum with 1H decoupling



```

Current Data Parameters
=====
NAME      RHM-III-192-2
PROCNO    1
PROBHD    5 mm CryoProbe
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         31
DS         4
SWH        30803.401 Hz
FIDRES     0.462388 Hz
AQ         1.1013698 sec
RG          696
DE         16.500 usec
TE         300.2
D1         0.25000000 sec
d11        0.03000000 sec
d12        0.03000000 sec
d13        0.03000000 sec
d14        0.03000000 sec
d15        0.03000000 sec
d16        0.03000000 sec
d17        0.03000000 sec
d18        0.03000000 sec
d19        0.03000000 sec
d20        0.03000000 sec
d21        0.03000000 sec
d22        0.03000000 sec
d23        0.03000000 sec
d24        0.03000000 sec
d25        0.03000000 sec
d26        0.03000000 sec
d27        0.03000000 sec
d28        0.03000000 sec
d29        0.03000000 sec
d30        0.03000000 sec
d31        0.03000000 sec
===== CHANNEL f1 =====
NUC1       13C
P1         12.00 usec
PL1        0.00 dB
PC1        2000.00 usec
P2         500.00 usec
PL2        120.00 dB
PC2        120.00 usec
SFO1       125.7642548 MHz
SFO2       2.70 dB
SFO3       2.70 dB
SFO4       2.70 dB
SFO5       2.70 dB
SFO6       2.70 dB
SFO7       2.70 dB
SFO8       2.70 dB
SFO9       2.70 dB
SFO10      2.70 dB
SFO11      2.70 dB
SFO12      2.70 dB
SFO13      2.70 dB
SFO14      2.70 dB
SFO15      2.70 dB
SFO16      2.70 dB
SFO17      2.70 dB
SFO18      2.70 dB
SFO19      2.70 dB
SFO20      2.70 dB
SFO21      2.70 dB
SFO22      2.70 dB
SFO23      2.70 dB
SFO24      2.70 dB
SFO25      2.70 dB
SFO26      2.70 dB
SFO27      2.70 dB
SFO28      2.70 dB
SFO29      2.70 dB
SFO30      2.70 dB
SFO31      2.70 dB
SFO32      2.70 dB
SFO33      2.70 dB
SFO34      2.70 dB
SFO35      2.70 dB
SFO36      2.70 dB
SFO37      2.70 dB
SFO38      2.70 dB
SFO39      2.70 dB
SFO40      2.70 dB
SFO41      2.70 dB
SFO42      2.70 dB
SFO43      2.70 dB
SFO44      2.70 dB
SFO45      2.70 dB
SFO46      2.70 dB
SFO47      2.70 dB
SFO48      2.70 dB
SFO49      2.70 dB
SFO50      2.70 dB
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
P2         100.00 usec
PL2        0.00 dB
PC2        1.60 dB
PL12       500.2220111 MHz
SFO1       500.2220111 MHz
===== GRADIENT CHANNEL =====
GPMAX1    300.00 V
SFO1       500.2220111 MHz
SFO2       500.2220111 MHz
GPA1       0.00 V
GPA2       0.00 V
GPA3       0.00 V
GPA4       0.00 V
GPA5       0.00 V
GPA6       0.00 V
GPA7       0.00 V
GPA8       0.00 V
GPA9       0.00 V
GPA10      0.00 V
GPA11      0.00 V
GPA12      0.00 V
GPA13      0.00 V
GPA14      0.00 V
GPA15      0.00 V
GPA16      0.00 V
GPA17      0.00 V
GPA18      0.00 V
GPA19      0.00 V
GPA20      0.00 V
GPA21      0.00 V
GPA22      0.00 V
GPA23      0.00 V
GPA24      0.00 V
GPA25      0.00 V
GPA26      0.00 V
GPA27      0.00 V
GPA28      0.00 V
GPA29      0.00 V
GPA30      0.00 V
GPA31      0.00 V
GPA32      0.00 V
GPA33      0.00 V
GPA34      0.00 V
GPA35      0.00 V
GPA36      0.00 V
GPA37      0.00 V
GPA38      0.00 V
GPA39      0.00 V
GPA40      0.00 V
GPA41      0.00 V
GPA42      0.00 V
GPA43      0.00 V
GPA44      0.00 V
GPA45      0.00 V
GPA46      0.00 V
GPA47      0.00 V
GPA48      0.00 V
GPA49      0.00 V
GPA50      0.00 V
===== Processing parameters =====
SI         32768
SF          125.7642548 MHz
WDW        EM
SSB         0
GB          0
PC          2.00
ID_NMR     10
CX          22.80 cm
CY          15.55 cm
CZ          15.55 cm
RG          2500.00 usec
RF1         2.00000000 ppm
RF2         -10.48732000 ppm
RF3         -10.48732000 ppm
RF4         -10.48732000 ppm
RF5         -10.48732000 ppm
RF6         -10.48732000 ppm
RF7         -10.48732000 ppm
RF8         -10.48732000 ppm
RF9         -10.48732000 ppm
RF10        -10.48732000 ppm
RF11        -10.48732000 ppm
RF12        -10.48732000 ppm
RF13        -10.48732000 ppm
RF14        -10.48732000 ppm
RF15        -10.48732000 ppm
RF16        -10.48732000 ppm
RF17        -10.48732000 ppm
RF18        -10.48732000 ppm
RF19        -10.48732000 ppm
RF20        -10.48732000 ppm
RF21        -10.48732000 ppm
RF22        -10.48732000 ppm
RF23        -10.48732000 ppm
RF24        -10.48732000 ppm
RF25        -10.48732000 ppm
RF26        -10.48732000 ppm
RF27        -10.48732000 ppm
RF28        -10.48732000 ppm
RF29        -10.48732000 ppm
RF30        -10.48732000 ppm
RF31        -10.48732000 ppm
RF32        -10.48732000 ppm
RF33        -10.48732000 ppm
RF34        -10.48732000 ppm
RF35        -10.48732000 ppm
RF36        -10.48732000 ppm
RF37        -10.48732000 ppm
RF38        -10.48732000 ppm
RF39        -10.48732000 ppm
RF40        -10.48732000 ppm
RF41        -10.48732000 ppm
RF42        -10.48732000 ppm
RF43        -10.48732000 ppm
RF44        -10.48732000 ppm
RF45        -10.48732000 ppm
RF46        -10.48732000 ppm
RF47        -10.48732000 ppm
RF48        -10.48732000 ppm
RF49        -10.48732000 ppm
RF50        -10.48732000 ppm
RF51        -10.48732000 ppm
RF52        -10.48732000 ppm
RF53        -10.48732000 ppm
RF54        -10.48732000 ppm
RF55        -10.48732000 ppm
RF56        -10.48732000 ppm
RF57        -10.48732000 ppm
RF58        -10.48732000 ppm
RF59        -10.48732000 ppm
RF60        -10.48732000 ppm
RF61        -10.48732000 ppm
RF62        -10.48732000 ppm
RF63        -10.48732000 ppm
RF64        -10.48732000 ppm
RF65        -10.48732000 ppm
RF66        -10.48732000 ppm
RF67        -10.48732000 ppm
RF68        -10.48732000 ppm
RF69        -10.48732000 ppm
RF70        -10.48732000 ppm
RF71        -10.48732000 ppm
RF72        -10.48732000 ppm
RF73        -10.48732000 ppm
RF74        -10.48732000 ppm
RF75        -10.48732000 ppm
RF76        -10.48732000 ppm
RF77        -10.48732000 ppm
RF78        -10.48732000 ppm
RF79        -10.48732000 ppm
RF80        -10.48732000 ppm
RF81        -10.48732000 ppm
RF82        -10.48732000 ppm
RF83        -10.48732000 ppm
RF84        -10.48732000 ppm
RF85        -10.48732000 ppm
RF86        -10.48732000 ppm
RF87        -10.48732000 ppm
RF88        -10.48732000 ppm
RF89        -10.48732000 ppm
RF90        -10.48732000 ppm
RF91        -10.48732000 ppm
RF92        -10.48732000 ppm
RF93        -10.48732000 ppm
RF94        -10.48732000 ppm
RF95        -10.48732000 ppm
RF96        -10.48732000 ppm
RF97        -10.48732000 ppm
RF98        -10.48732000 ppm
RF99        -10.48732000 ppm
RF100       -10.48732000 ppm
=====
  
```



gcosy60

4.6

Current Data Parameters  
 USER kheswtt1  
 NAME FAH-III-192-2  
 RFXNO 1  
 RFXCD0 1

F2 - Acquisition Parameters  
 Date\_ 20200909  
 Time 18:03  
 SYSTEM crysno  
 PROBN 5 mm CPY500  
 PULPROG cosygpg0.pcd  
 TD 2048  
 SOLVENT CDCl3  
 NS 1  
 SH 8012.820 Hz  
 F2PRES 3.912510 Hz  
 AQ 0.1278452 sec  
 RS 128  
 DM 62.400 usec  
 DE 7.00 usec  
 TE 288.2  
 d0 0.00000300 sec  
 d1 1.00000000 sec  
 d13 0.00000300 sec  
 d16 0.00020000 sec  
 INO 0.0012480 sec

==== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.50 usec  
 PL1 1.60 dB  
 SFO1 500.225015 MHz

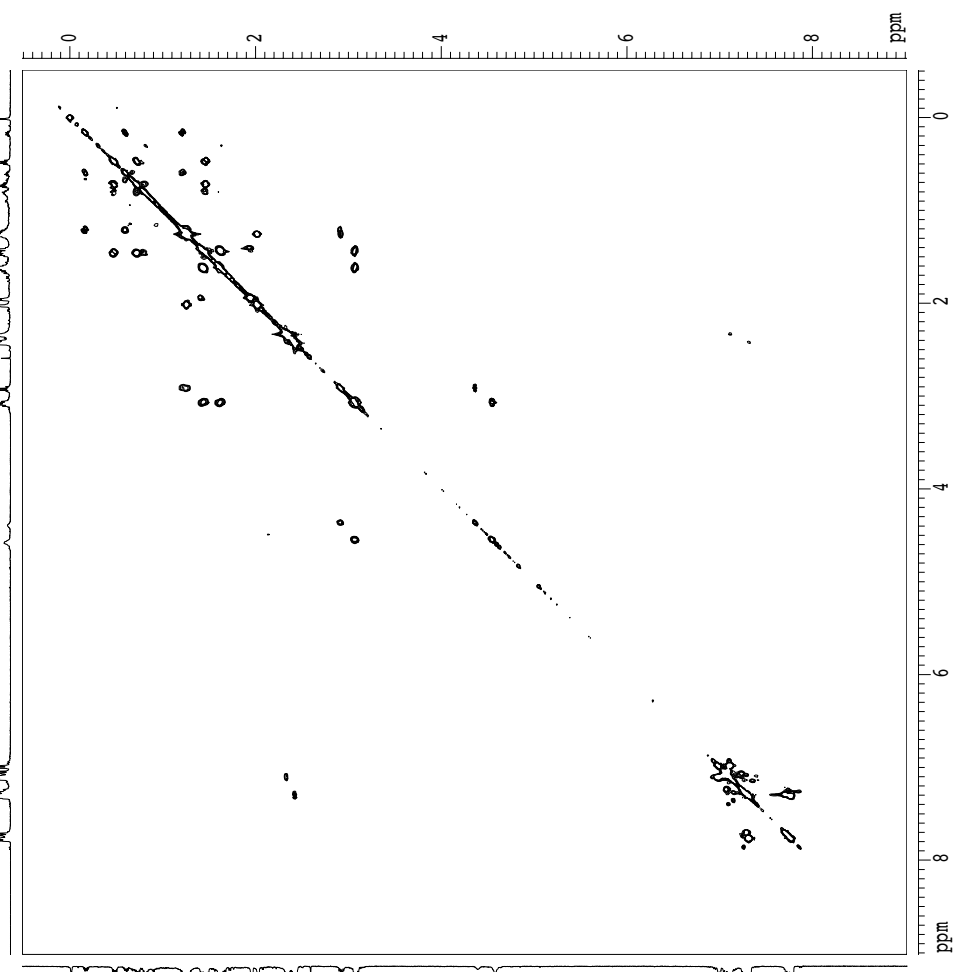
==== GRADIENT CHANNEL =====  
 GENAM1 sine.100  
 GENAM2 sine.100  
 GP1 0.00 %  
 GP2 0.00 %  
 GP3 0.00 %  
 GP4 0.00 %  
 GP5 0.00 %  
 GP6 17.00 %  
 GP7 17.00 %  
 GP8 17.00 %  
 P16 1000.00 usec

F1 - Acquisition parameters  
 ND0 1  
 TD 512  
 SFO1 500.2235 MHz  
 F2PRES 15.650040 Hz  
 SW 16.08 ppm  
 FWHOSE 0

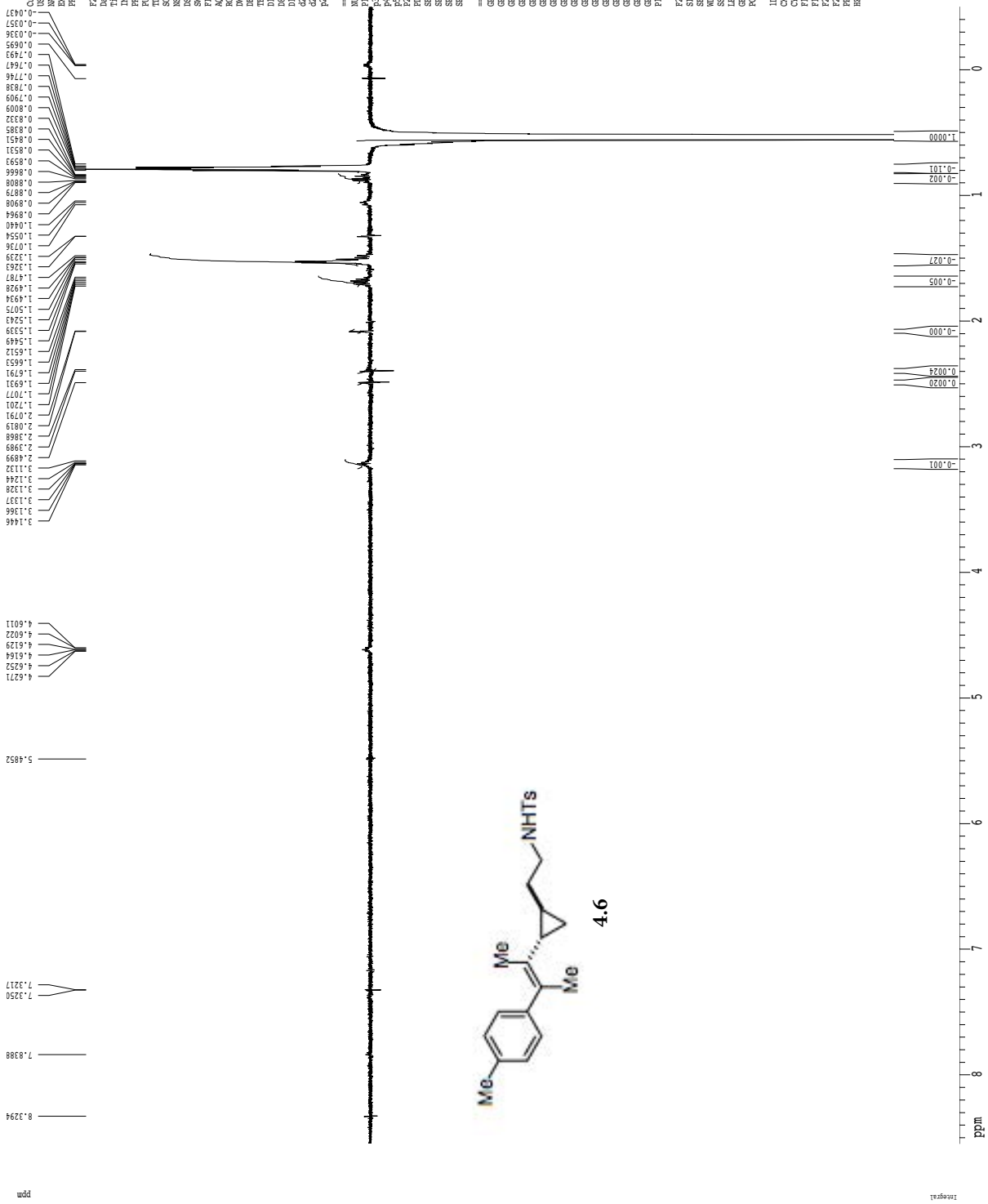
F2 - Processing parameters  
 SI 500.2200326 MHz  
 SF 500.2200326 MHz  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 500.2200326 MHz  
 SF 500.2200326 MHz  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D INMR plot parameters  
 CK1 15.00 cm  
 CK2 15.00 cm  
 F2LO 450.02 ppm  
 F2HI -0.511 ppm  
 F2B1 -255.78 Hz  
 F2LO 9.015 ppm  
 F2HI 4509.66 Hz  
 F2B1 -255.78 Hz  
 F2PACH 0.63511 ppm/cm  
 F2BACH 317.69580 Hz/cm  
 F1PACH 0.63511 ppm/cm  
 F1BACH 317.69580 Hz/cm



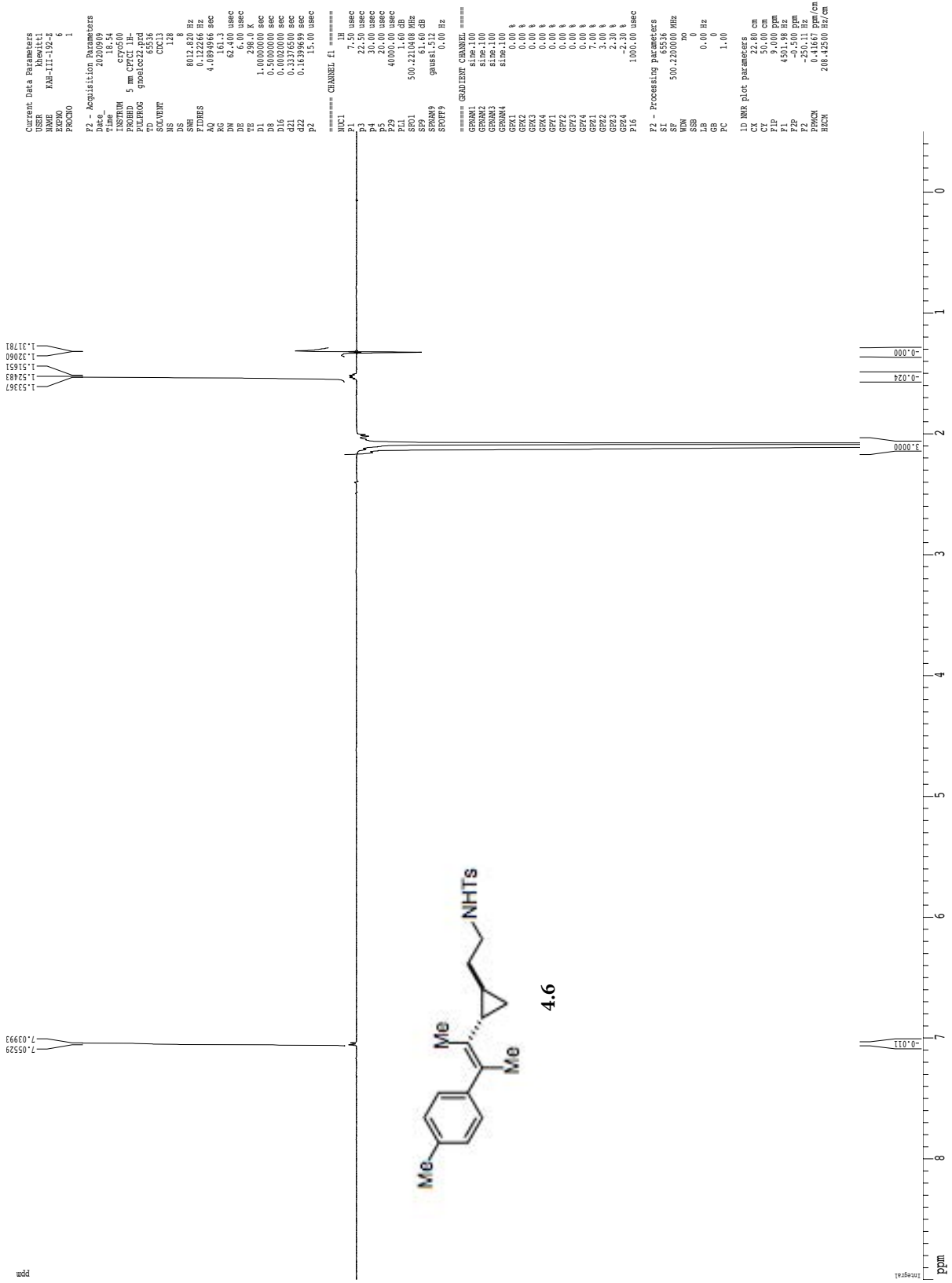
gnoe



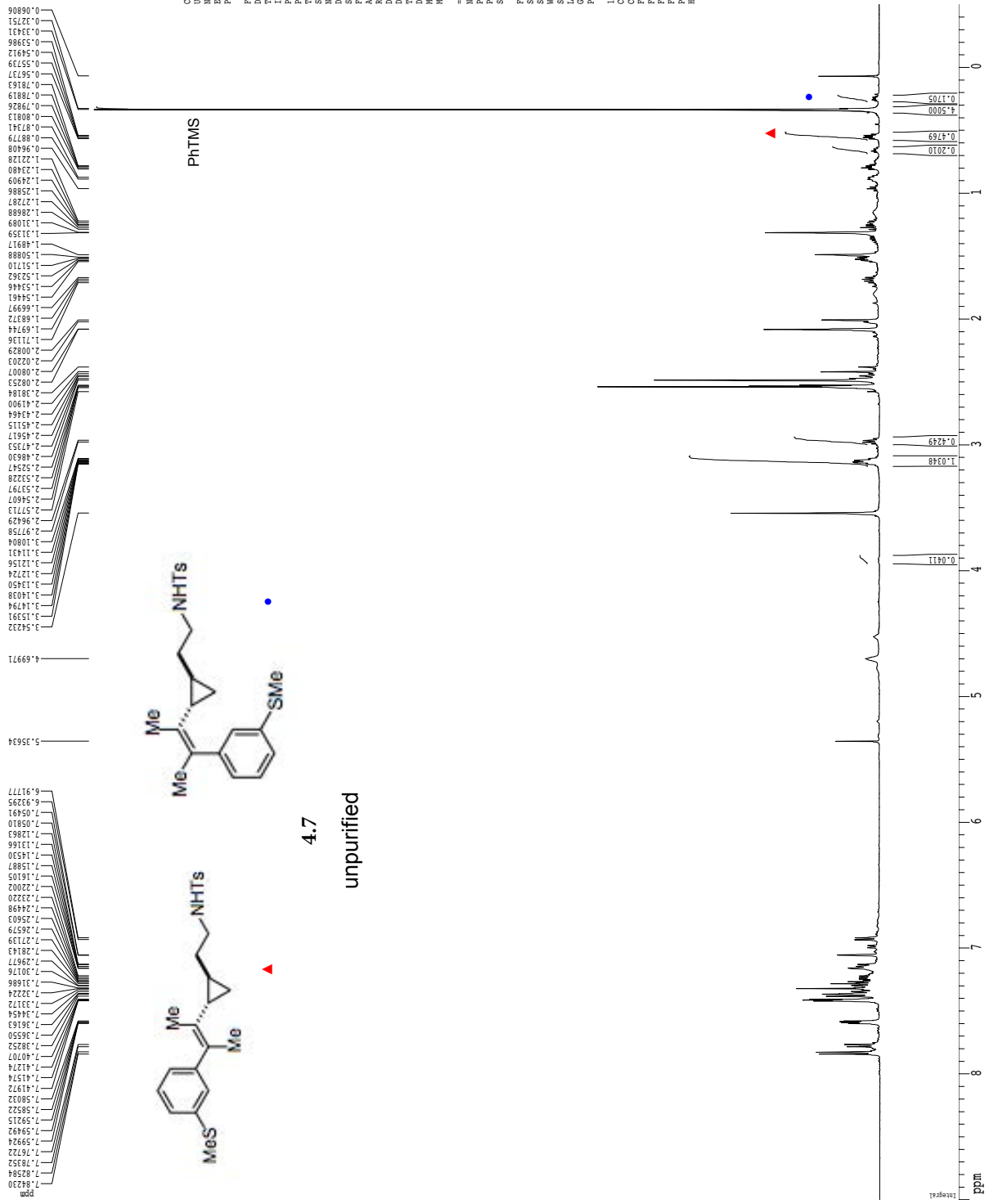
=====  
Date\_ 2020/09/09  
Time 18:27  
USER khawatt1  
NAME RMR-III-192-2  
EXPNO 4  
PROCNO 1  
F2 - Acquisition Parameters  
PROBHD 5 mm CPCT 1H  
PULPROG gnoe1cc22.prd  
TD 65536  
SOLVENT CCL4  
NS 128  
DS 8  
SWH 8012.622 Hz  
FIDRES 0.122266 Hz  
AQ 4.0893966 sec  
RG 327.500  
DM 62.400 usec  
DE 6.000 usec  
TE 298.0 K  
D1 1.0000000 sec  
SFO1 500.2200682 MHz  
D2 0.33375000 sec  
d21 0.33375000 sec  
d22 0.16398699 sec  
P2 15.00 usec  
===== CHANNEL f1 =====  
NUC1 1H  
P1 7.50 usec  
P2 22.50 usec  
PL 0  
PL1 0  
PL2 0  
PL3 0  
PL4 0  
PL5 0  
PL6 0  
PL7 0  
PL8 0  
PL9 0  
PL10 0  
PL11 0  
PL12 0  
PL13 0  
PL14 0  
PL15 0  
PL16 0  
PL17 0  
PL18 0  
PL19 0  
PL20 0  
PL21 0  
PL22 0  
PL23 0  
PL24 0  
PL25 0  
PL26 0  
PL27 0  
PL28 0  
PL29 0  
PL30 0  
PL31 0  
PL32 0  
PL33 0  
PL34 0  
PL35 0  
PL36 0  
PL37 0  
PL38 0  
PL39 0  
PL40 0  
PL41 0  
PL42 0  
PL43 0  
PL44 0  
PL45 0  
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PL72 0  
PL73 0  
PL74 0  
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PL76 0  
PL77 0  
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PL80 0  
PL81 0  
PL82 0  
PL83 0  
PL84 0  
PL85 0  
PL86 0  
PL87 0  
PL88 0  
PL89 0  
PL90 0  
PL91 0  
PL92 0  
PL93 0  
PL94 0  
PL95 0  
PL96 0  
PL97 0  
PL98 0  
PL99 0  
PL100 0  
===== GRADIENT CHANNEL =====  
GUNIT1 line:100  
GUNIT2 line:100  
GUNIT3 line:100  
GUNIT4 line:100  
GUNIT5 line:100  
GUNIT6 line:100  
GUNIT7 line:100  
GUNIT8 line:100  
GUNIT9 line:100  
GUNIT10 line:100  
GUNIT11 line:100  
GUNIT12 line:100  
GUNIT13 line:100  
GUNIT14 line:100  
GUNIT15 line:100  
GUNIT16 line:100  
GUNIT17 line:100  
GUNIT18 line:100  
GUNIT19 line:100  
GUNIT20 line:100  
GUNIT21 line:100  
GUNIT22 line:100  
GUNIT23 line:100  
GUNIT24 line:100  
GUNIT25 line:100  
GUNIT26 line:100  
GUNIT27 line:100  
GUNIT28 line:100  
GUNIT29 line:100  
GUNIT30 line:100  
GUNIT31 line:100  
GUNIT32 line:100  
GUNIT33 line:100  
GUNIT34 line:100  
GUNIT35 line:100  
GUNIT36 line:100  
GUNIT37 line:100  
GUNIT38 line:100  
GUNIT39 line:100  
GUNIT40 line:100  
GUNIT41 line:100  
GUNIT42 line:100  
GUNIT43 line:100  
GUNIT44 line:100  
GUNIT45 line:100  
GUNIT46 line:100  
GUNIT47 line:100  
GUNIT48 line:100  
GUNIT49 line:100  
GUNIT50 line:100  
===== Processing parameters =====  
SI 65536  
SF 500.2200682 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
CB 1.00  
PC 22.88 cm  
CT 50.00 cm  
FL 4.0000000 ppm  
F1 450.1360000 ppm  
F2 -250.11 Hz  
PPHMM 0.41664 ppm/cm  
HPCN 208.42500 Hz/cm

gnoe

ppm

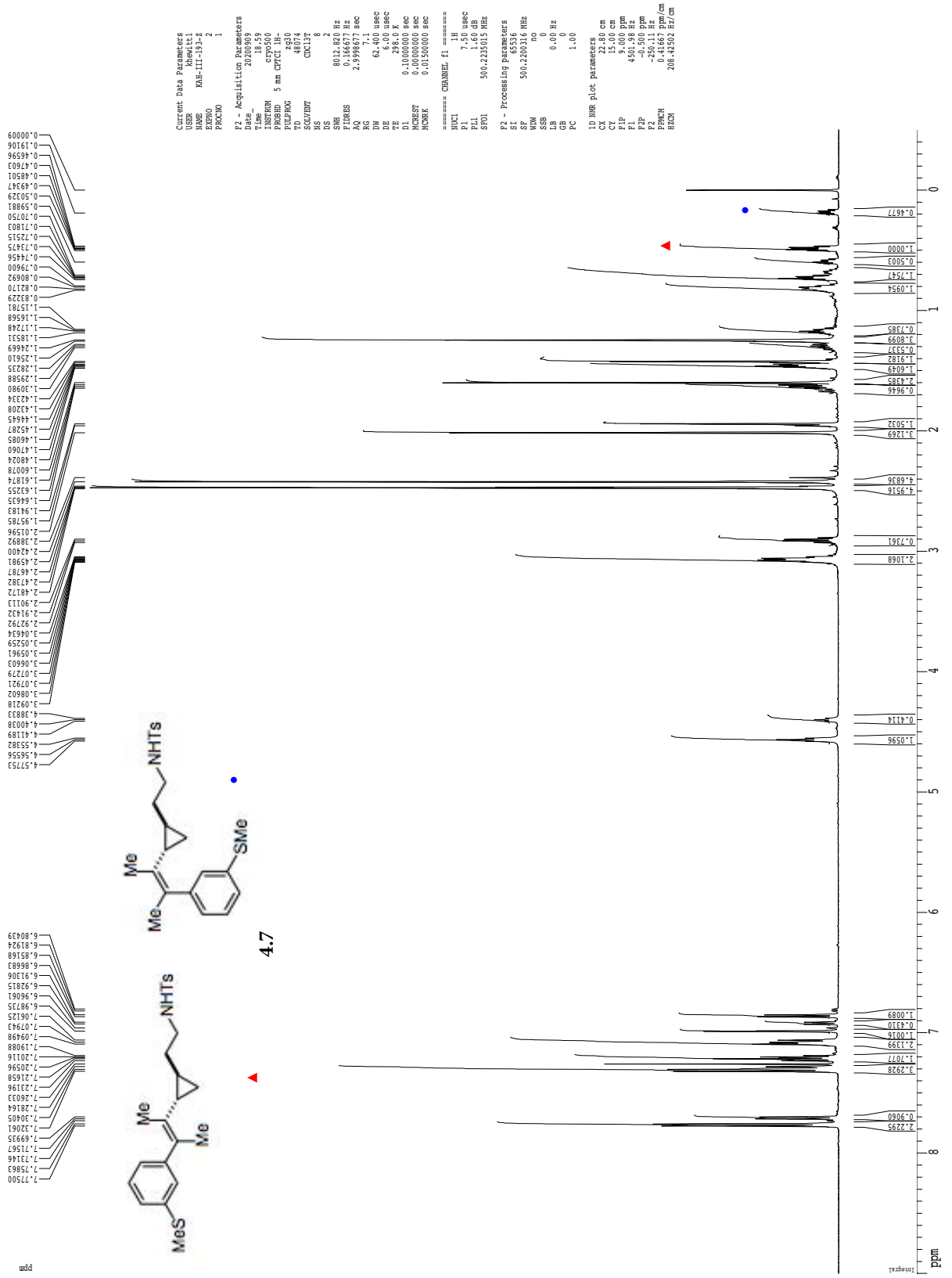


**<sup>1</sup>H spectrum**

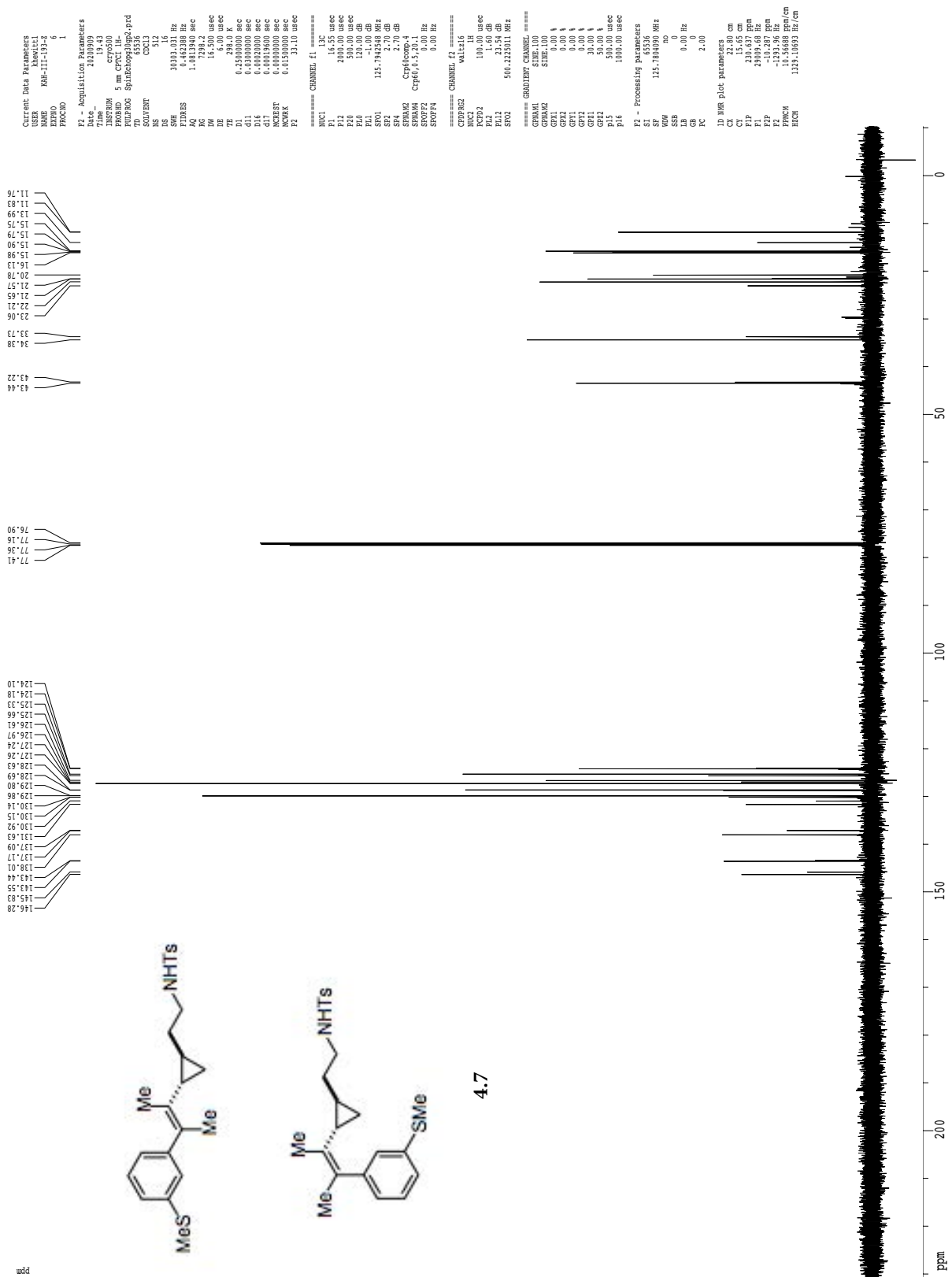


Current Data Parameters  
 USER kweid11  
 NAME KAH-III-193-crude  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20200716  
 Time 18.01  
 C1NAME C-130  
 PROBHD 5 mm CPYCI 1H  
 PULPROG zg30  
 TD 48074  
 SFO 400.146  
 AQC 1.00  
 ACQRES 0.150000 sec  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.146877 Hz  
 AQ 2.7398677 sec  
 RG 7.1  
 DW 62.400 usec  
 DE 6.00 usec  
 DI 20.00 usec  
 D1 0.1000000 sec  
 MCREST 0.0000000 sec  
 MCORR 0.0150000 sec  
 ===== CHANNEL f1 =====  
 NU1 1H  
 P1 7.50 usec  
 PL 0.00 dB  
 SFO1 500.126015 MHz  
 F2 - Processing parameters  
 SI 6556  
 SF 500.126015 MHz  
 WDW 0  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00  
 ID NMR plot parameters  
 CX 22.80 cm  
 CY 22.80 cm  
 FIP 9.000 ppm  
 F1 4501.98 Hz  
 F2 -0.500 ppm  
 F3 0.000 ppm  
 FWHM 0.41661 ppm/cm  
 HCN 208.44516 Hz/cm

# 1H spectrum



Z-restored spin-echo 13C spectrum with 1H decoupling

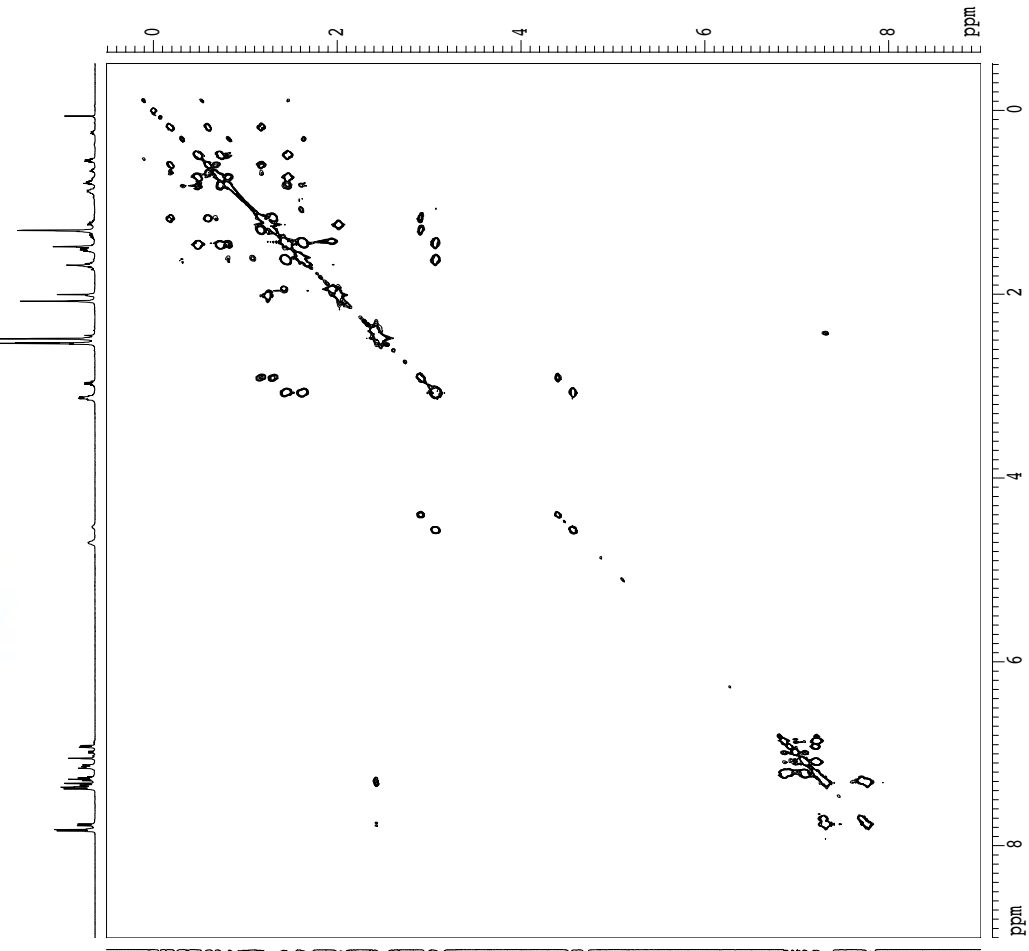




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Current Data Parameters
=====
USR      Khsvit1
EXPNO    KM-III-199-2
PROCNO   1
=====
F2 - Acquisition Parameters
=====
Date_    20080909
Time     14:56:00
INSTRUM  crys500
PROBHD   5 mm CPXI 1H-
PULPROG  zgpg30.prd
TD       2048
SOLVENT  CDCl3
NS       16
DS       6
SWH      8012.820 Hz
FIDRES   3.432510 Hz
AQ       0.1278432 sec
RG       62.400 usec
DE       6.00 usec
TE       298.0 K
D0       0.0000300 sec
d1       1.0000000 sec
d2       0.0000000 sec
d16      0.0002000 sec
d18      0.0001240 sec
IN0      0.0001240 sec
===== CHANNEL f1 =====
NUC1     13C
P1       7.50 usec
PL1     -1.60 dB
SFO1    500.2235015 MHz
===== GRABBER CHANNEL =====
GPNM1    13C
GPNM2    13C
GPA1     0.00 usec
GPA2     0.00 usec
GPF1     0.00 usec
GPF2     0.00 usec
GPR1     17.00 usec
GPR2     17.00 usec
PL6      1000.00 usec
=====
F1 - Acquisition Parameters
=====
NUC1     13C
P1       7.50 usec
PL1     -1.60 dB
SFO1    500.2235015 MHz
===== GRABBER CHANNEL =====
GPNM1    13C
GPNM2    13C
GPA1     0.00 usec
GPA2     0.00 usec
GPF1     0.00 usec
GPF2     0.00 usec
GPR1     17.00 usec
GPR2     17.00 usec
PL6      1000.00 usec
=====
F2 - Processing Parameters
=====
SI       1024
SF       500.2200315 MHz
WDW      SINE
SSB      0
GB       0
PC       1.00
=====
F1 - Processing Parameters
=====
SI       1024
SF       500.2200315 MHz
WDW      SINE
SSB      0
GB       0
PC       1.00
=====
2D NMR plot parameters
=====
CX1      15.00 cm
CX2      15.00 cm
F2FLO    9.002 ppm
F2FHI    48.500 ppm
F2FLO2   -0.500 ppm
F2FHI2   -354.668 Hz
F2FLO3   9.002 ppm
F2FHI3   4502.93 Hz
F2FLO4   -254.668 ppm
F2FHI4   0.63407 ppm/cm
F2FPCN   317.17413 Hz/cm
F2FPCN2  0.63407 ppm/cm
F2FPCN3  317.17413 Hz/cm

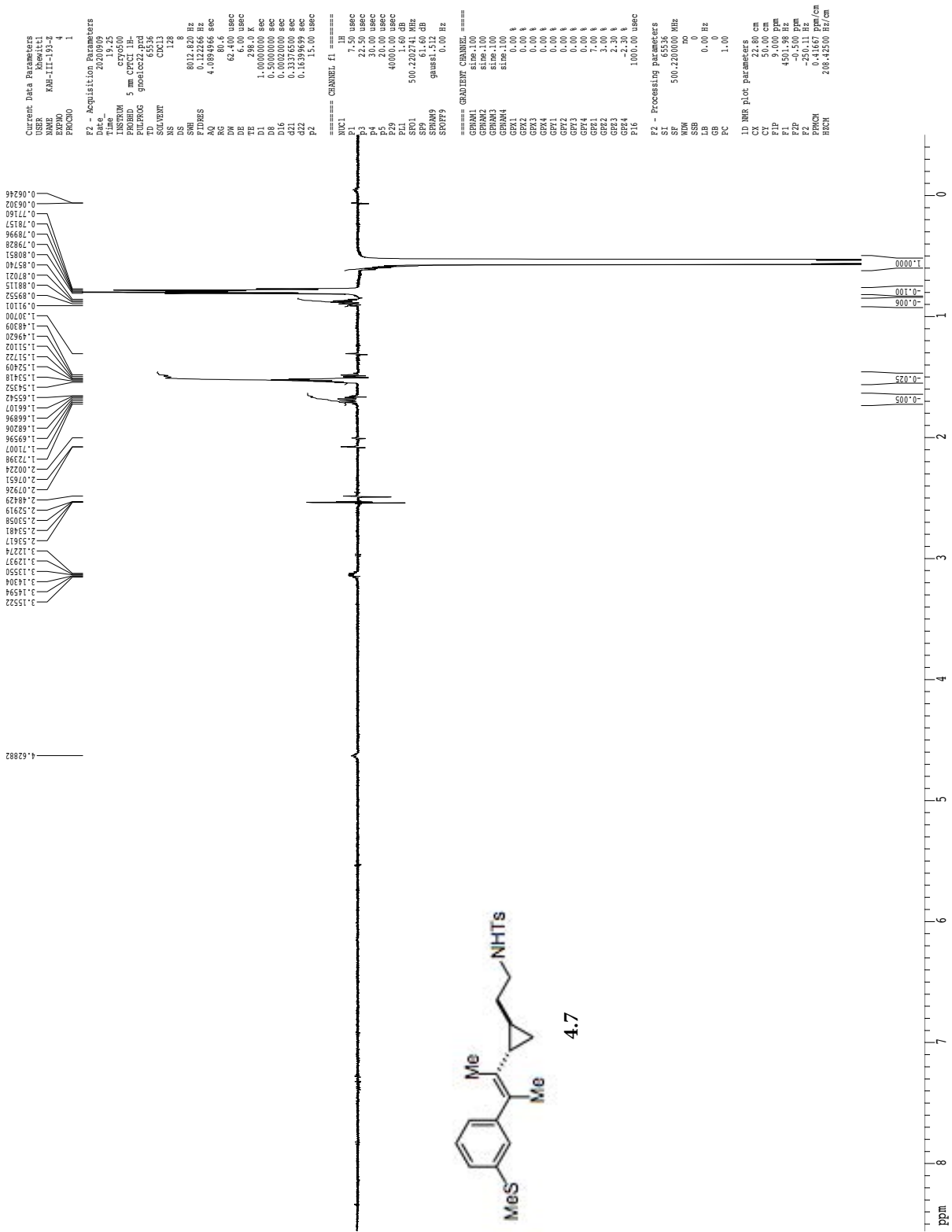
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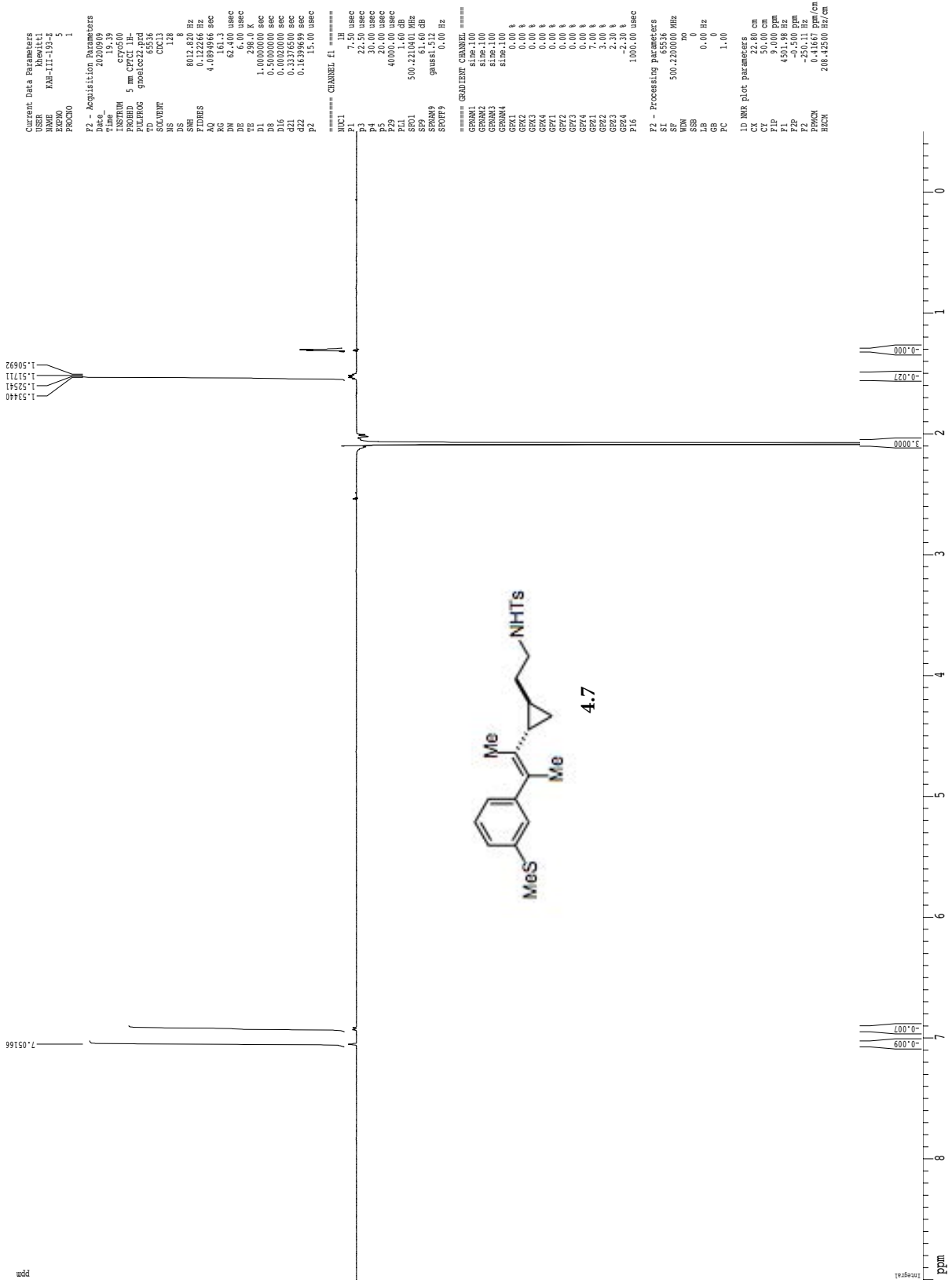


gnoe

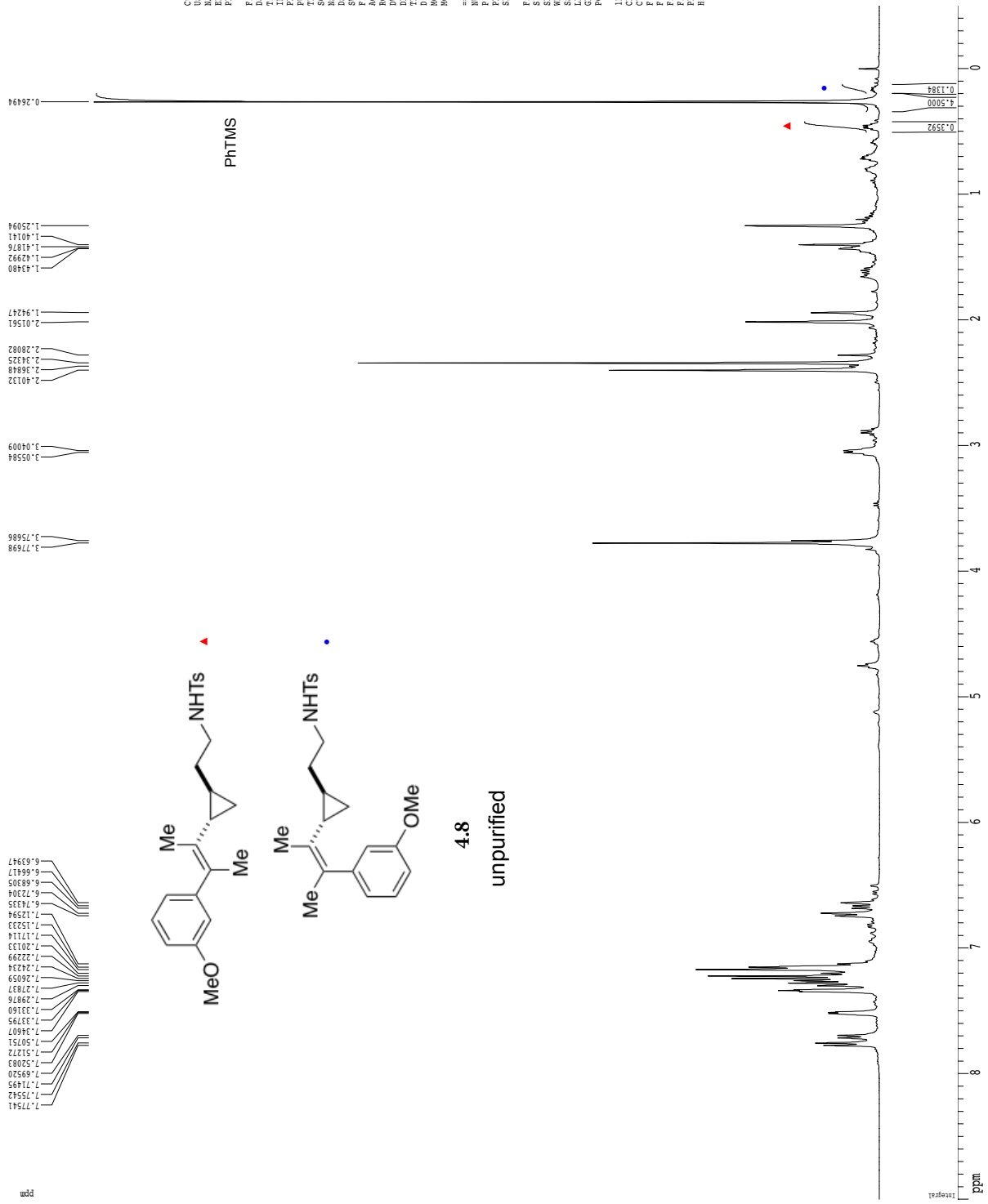
ppm



gnoe



1H spectrum



Current Data Parameters  
USER: null  
NAME: NH-4-57-acide  
EXPNO: 1  
PROCNO: 1

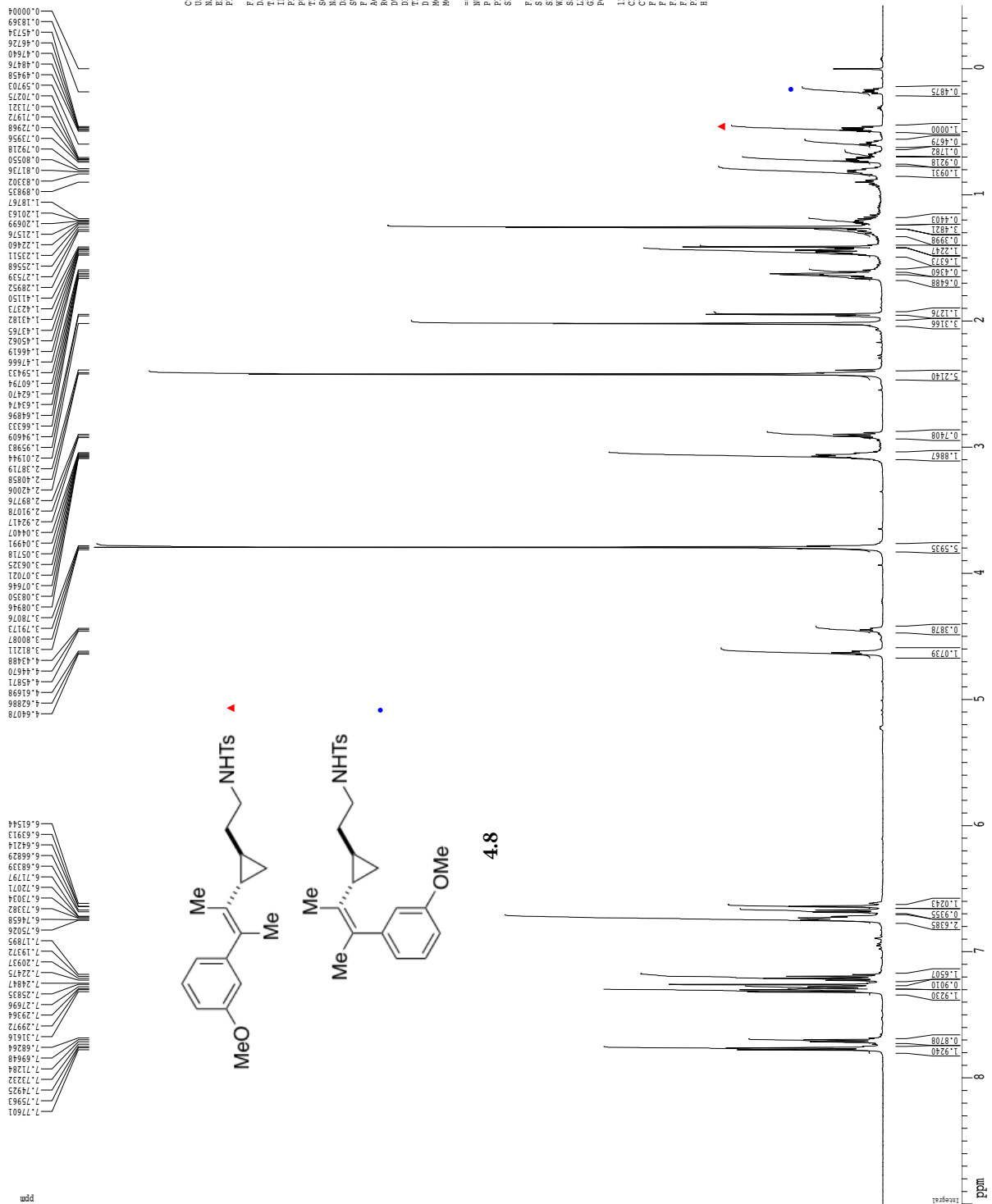
F2 - Acquisition Parameters  
Date\_: 20200922  
Time: 17.05  
PROBHD: 5 mm QNP 1H/1  
PULPROG: zgpg30  
TD: 65536  
SOLVENT: CDCl3  
DS: 9  
SFO: 6410.258 Hz  
FIDRES: 0.097813 Hz  
AQ: 5.1118579 sec  
RG: 327.5  
DSF: 78.000 usec  
DE: 4.50 usec  
TE: 295.1 K  
T1: 0.1900000 sec  
T2: 0.0000000 sec  
T3: 0.0000000 sec  
MORPH: 0.0150000 sec

===== CHANNEL f1 =====  
NUC1: 13C  
P1: 12.00 usec  
PL1: -1.10 dB  
SFO1: 400.1328009 MHz

F2 - Processing Parameters  
SI: 65536  
SF: 400.1300365 MHz  
WDW: EM  
SSB: 0  
GB: 0.30 Hz  
PC: 2.00

ID: NMR file parameters  
CY: 22.80 cm  
CT: 15.00 cm  
FIP: 9.000 ppm  
FL: 3600.17 Hz  
F2: 200.000 MHz  
P1: 12.00 usec  
P2: 0.41667 ppm/cm  
HSCN: 166.72086 Hz/cm

# 1H spectrum



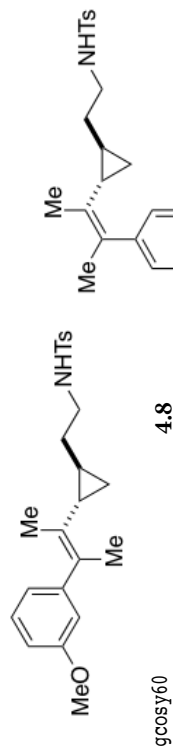
Current Data Parameters  
USER: nhlfbaw  
NAME: NH-157-HANDCOST  
EXPNO: 1  
PROCNO: 1  
Date\_ 20200928  
Time: 17.33  
PROBHD: 5 mm CPYX-1H  
PULPROG: zgpg30  
TD: 8172  
SOLVENT: CDCl3  
DS: 9  
SHE: 8012.820 Hz  
FIDRES: 0.09804 Hz  
AQ: 5.1998774 sec  
RG: 62.400 usec  
DE: 6.000 usec  
TE: 298.0 K  
AQ: 0.16000000 sec  
RG: 62.40000000 sec  
MCOREST: 0.03500000 sec  
MCORRE: 0.03500000 sec

===== CHANNEL f1 =====  
NUC1: 15N  
P1: 1.50 usec  
PL1: 1.66 dB  
SFO1: 500.2335015 MHz

F2 - Processing Parameters  
SI: 65536  
SF: 500.2200322 MHz  
WDW: EM  
SSB: 0  
GB: 0  
PC: 1.00

D0 NMR P1/2 Parameters  
C1: 22.80 cm  
CY: 15.00 cm  
FIP: 9.000 ppm  
F1: 4501.98 Hz  
F2: -250.14 Hz  
P1P1CM: 0.44667 ppm/cm  
H1CN: 206.44502 Hz/cm





Current Data Parameters  
 USER nhrbawi  
 NAME NH-1-57-HandCOSY  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20200928  
 Time 17.36  
 INSTRUM cryo500  
 PROBHD 5 mm CPCL1 1H-  
 PULPROG cosygp0.prd  
 SOLVENT CDCl3T  
 NS 2  
 DS 16  
 SWH 4734.849 Hz  
 FIDRES 2.31198 Hz  
 AQ 0.216118 sec  
 RG 655  
 DM 105.600 usec  
 DE 6.00 usec  
 TE 298.0 K  
 TD 0.0000300 sec  
 DL 1.0000000 sec  
 D1 0.0000000 sec  
 D11 0.0000000 sec  
 D12 0.0000000 sec  
 D13 0.0000000 sec  
 D14 0.0000000 sec  
 D15 0.0000000 sec  
 D16 0.0000000 sec  
 D17 0.0000000 sec  
 D18 0.0000000 sec  
 D19 0.0000000 sec  
 D20 0.0000000 sec  
 D21 0.0000000 sec  
 D22 0.0000000 sec  
 D23 0.0000000 sec  
 D24 0.0000000 sec  
 D25 0.0000000 sec  
 D26 0.0000000 sec  
 D27 0.0000000 sec  
 D28 0.0000000 sec  
 D29 0.0000000 sec  
 D30 0.0000000 sec  
 D31 0.0000000 sec  
 D32 0.0000000 sec  
 D33 0.0000000 sec  
 D34 0.0000000 sec  
 D35 0.0000000 sec  
 D36 0.0000000 sec  
 D37 0.0000000 sec  
 D38 0.0000000 sec  
 D39 0.0000000 sec  
 D40 0.0000000 sec  
 D41 0.0000000 sec  
 D42 0.0000000 sec  
 D43 0.0000000 sec  
 D44 0.0000000 sec  
 D45 0.0000000 sec  
 D46 0.0000000 sec  
 D47 0.0000000 sec  
 D48 0.0000000 sec  
 D49 0.0000000 sec  
 D50 0.0000000 sec  
 D51 0.0000000 sec  
 D52 0.0000000 sec  
 D53 0.0000000 sec  
 D54 0.0000000 sec  
 D55 0.0000000 sec  
 D56 0.0000000 sec  
 D57 0.0000000 sec  
 D58 0.0000000 sec  
 D59 0.0000000 sec  
 D60 0.0000000 sec  
 D61 0.0000000 sec  
 D62 0.0000000 sec  
 D63 0.0000000 sec  
 D64 0.0000000 sec  
 D65 0.0000000 sec  
 D66 0.0000000 sec  
 D67 0.0000000 sec  
 D68 0.0000000 sec  
 D69 0.0000000 sec  
 D70 0.0000000 sec  
 D71 0.0000000 sec  
 D72 0.0000000 sec  
 D73 0.0000000 sec  
 D74 0.0000000 sec  
 D75 0.0000000 sec  
 D76 0.0000000 sec  
 D77 0.0000000 sec  
 D78 0.0000000 sec  
 D79 0.0000000 sec  
 D80 0.0000000 sec  
 D81 0.0000000 sec  
 D82 0.0000000 sec  
 D83 0.0000000 sec  
 D84 0.0000000 sec  
 D85 0.0000000 sec  
 D86 0.0000000 sec  
 D87 0.0000000 sec  
 D88 0.0000000 sec  
 D89 0.0000000 sec  
 D90 0.0000000 sec  
 D91 0.0000000 sec  
 D92 0.0000000 sec  
 D93 0.0000000 sec  
 D94 0.0000000 sec  
 D95 0.0000000 sec  
 D96 0.0000000 sec  
 D97 0.0000000 sec  
 D98 0.0000000 sec  
 D99 0.0000000 sec  
 D100 0.0000000 sec

==== CHANNEL f1 =====

NUC1 1H  
 P1 7.50 usec  
 PL 0.00 dB  
 SFO1 500.2221582 MHz

==== GRADIENT CHANNEL =====

GPRAM1 sine.100  
 GPRAM2 sine.100  
 GP1 0.00 %  
 GP2 0.00 %  
 GP3 0.00 %  
 GP4 0.00 %  
 GP5 0.00 %  
 GP6 0.00 %  
 GP7 0.00 %  
 GP8 0.00 %  
 GP9 0.00 %  
 GP10 0.00 %  
 GP11 0.00 %  
 GP12 0.00 %  
 GP13 0.00 %  
 GP14 0.00 %  
 GP15 0.00 %  
 GP16 0.00 %  
 GP17 0.00 %  
 GP18 0.00 %  
 GP19 0.00 %  
 GP20 0.00 %  
 GP21 0.00 %  
 GP22 0.00 %  
 GP23 0.00 %  
 GP24 0.00 %  
 GP25 0.00 %  
 GP26 0.00 %  
 GP27 0.00 %  
 GP28 0.00 %  
 GP29 0.00 %  
 GP30 0.00 %  
 GP31 0.00 %  
 GP32 0.00 %  
 GP33 0.00 %  
 GP34 0.00 %  
 GP35 0.00 %  
 GP36 0.00 %  
 GP37 0.00 %  
 GP38 0.00 %  
 GP39 0.00 %  
 GP40 0.00 %  
 GP41 0.00 %  
 GP42 0.00 %  
 GP43 0.00 %  
 GP44 0.00 %  
 GP45 0.00 %  
 GP46 0.00 %  
 GP47 0.00 %  
 GP48 0.00 %  
 GP49 0.00 %  
 GP50 0.00 %  
 GP51 0.00 %  
 GP52 0.00 %  
 GP53 0.00 %  
 GP54 0.00 %  
 GP55 0.00 %  
 GP56 0.00 %  
 GP57 0.00 %  
 GP58 0.00 %  
 GP59 0.00 %  
 GP60 0.00 %  
 GP61 0.00 %  
 GP62 0.00 %  
 GP63 0.00 %  
 GP64 0.00 %  
 GP65 0.00 %  
 GP66 0.00 %  
 GP67 0.00 %  
 GP68 0.00 %  
 GP69 0.00 %  
 GP70 0.00 %  
 GP71 0.00 %  
 GP72 0.00 %  
 GP73 0.00 %  
 GP74 0.00 %  
 GP75 0.00 %  
 GP76 0.00 %  
 GP77 0.00 %  
 GP78 0.00 %  
 GP79 0.00 %  
 GP80 0.00 %  
 GP81 0.00 %  
 GP82 0.00 %  
 GP83 0.00 %  
 GP84 0.00 %  
 GP85 0.00 %  
 GP86 0.00 %  
 GP87 0.00 %  
 GP88 0.00 %  
 GP89 0.00 %  
 GP90 0.00 %  
 GP91 0.00 %  
 GP92 0.00 %  
 GP93 0.00 %  
 GP94 0.00 %  
 GP95 0.00 %  
 GP96 0.00 %  
 GP97 0.00 %  
 GP98 0.00 %  
 GP99 0.00 %  
 GP100 0.00 %

F1 - Acquisition Parameters

NUC0 256  
 TD 256  
 SFO1 500.2222 MHz  
 FIDRES 18.45502 Hz  
 SN 9.465 PPM  
 FMODE QF

F2 - Processing parameters

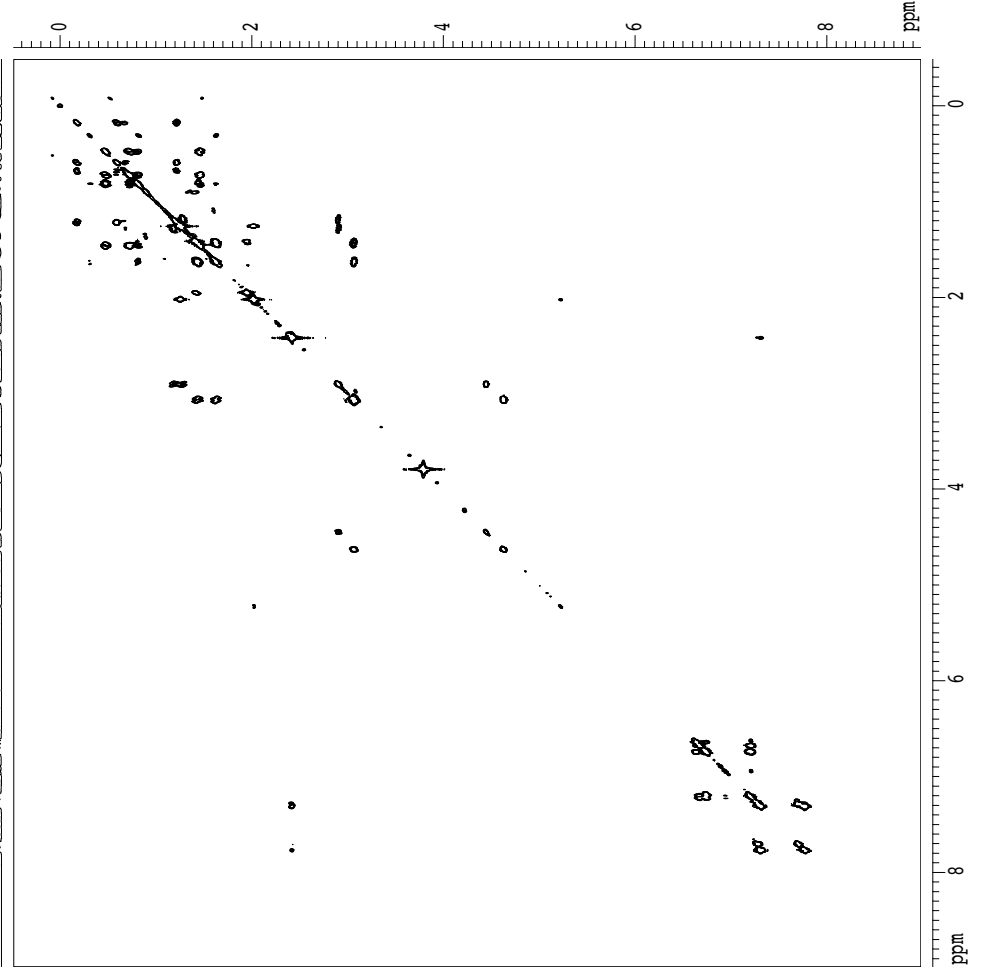
SF 500.2200323 MHz  
 SI 1024  
 SF 500.2200323 MHz  
 NUN SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 1.00

F1 - Processing parameters

SI 1024  
 MC2 500.2200324 MHz  
 SF SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

ZD NMR plot parameters

CV2 15.00 cm  
 CX 1.00 cm  
 FZLO 8.983 ppm  
 FZLO 4493.29 Hz  
 FZPH -0.483 ppm  
 FZPH -241.56 Hz  
 FZLO 8.982 ppm  
 FZLO 4493.23 Hz  
 FZPH -0.483 ppm  
 FZPH -241.52 Hz  
 FZPACH 0.63104 ppm/cm  
 FZPACH 315.65659 Hz/cm  
 FHPACH 0.63104 ppm/cm  
 FHPACH 315.65659 Hz/cm



gnoe

```

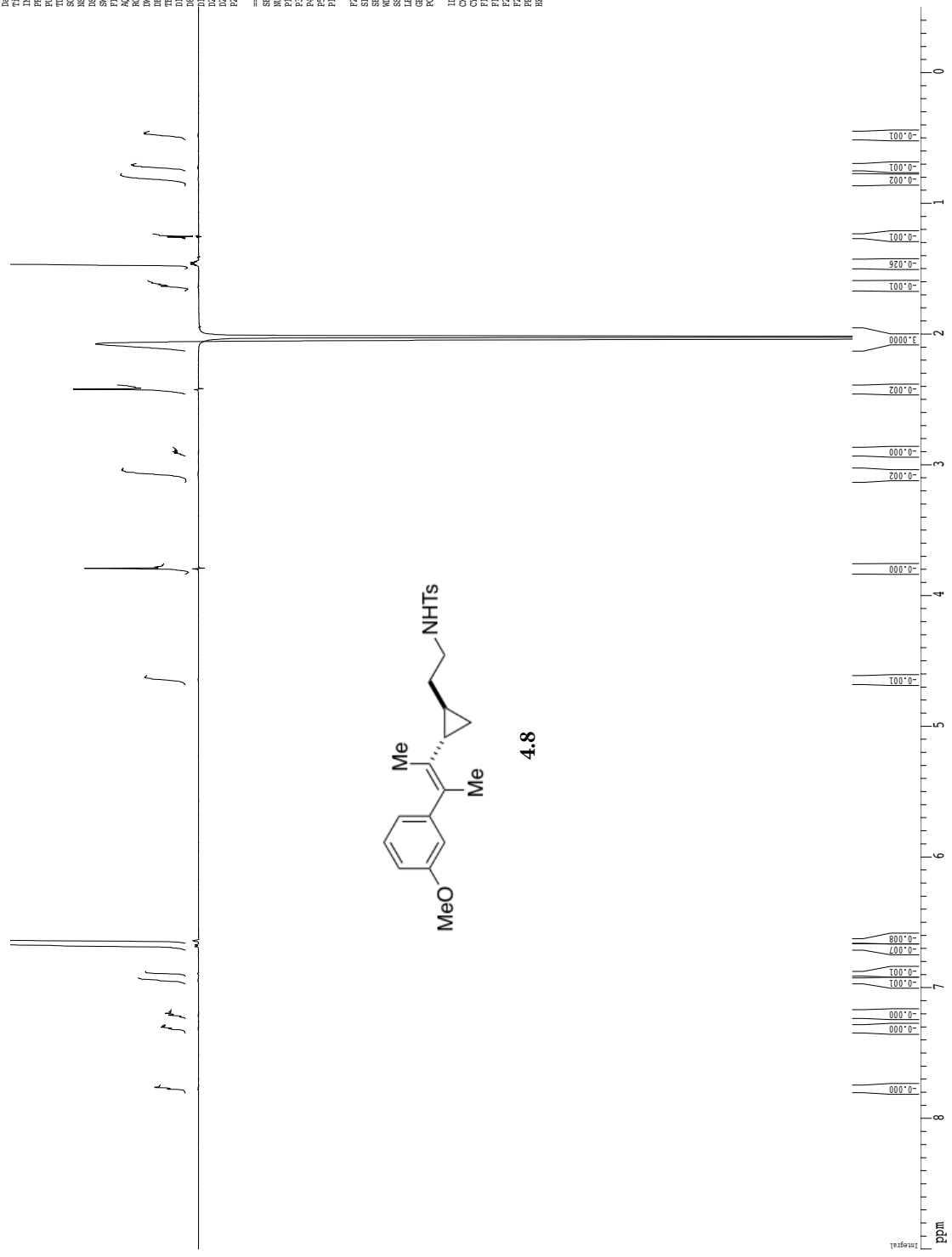
Current Data Parameters
=====
NAME      MF-1-57-600X61
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
=====
Date_     20200928
Time      18.29
INSTRUM   av600
PROBHD    5 mm QNP
PULPROG   zgpg30
TD         98074
SOLVENT   CDCl3
NS         158
DS         4
SWH        9615.388 Hz
FIDRES    0.098044 Hz
AQ         5.093877 sec
RG         32
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

===== CHANNEL f1 =====
SFO1      600.1317416 MHz
NUC1      1H
P1         9.50 usec
P2         28.50 usec
P3         28.50 usec
P4         28.50 usec
P5         28.50 usec
P12        80000.00 usec

F2 - Processing parameters
=====
SI         32768
SF         600.130345 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

ID NMR plot parameters
=====
CX         22.88 cm
CY         9.00 cm
CZ         9.00 cm
FLIP       5401.17 Hz
F1P        -0.500 ppm
F2P        -300.06 Hz
F3P        0.14666 ppm/cm
RGXCM      2531.9249 Hz/cm
  
```



gnoe

ppm

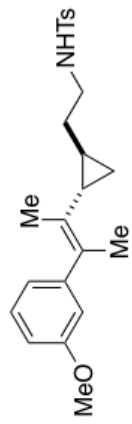
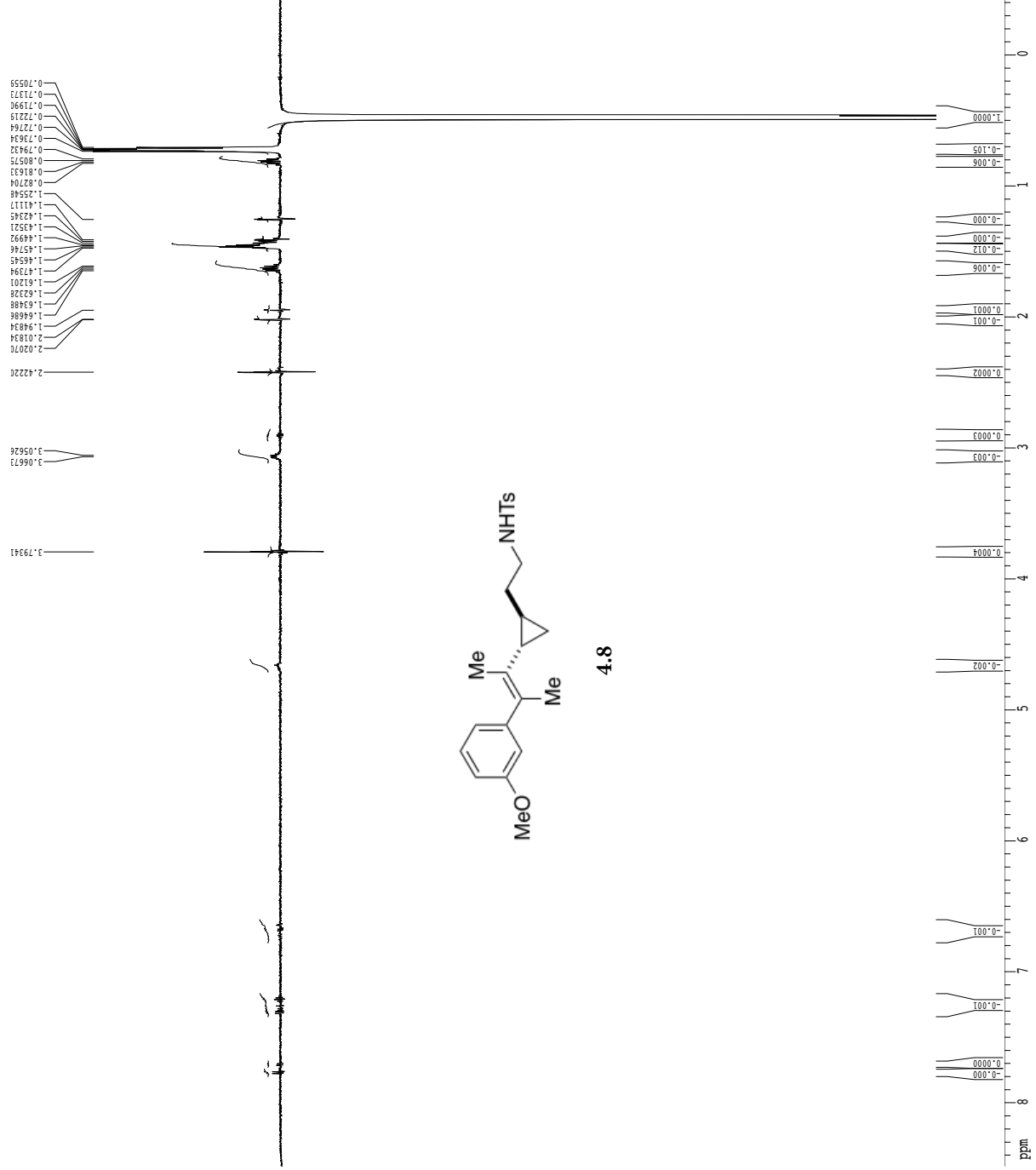
Current Data Parameters  
USER: mlr/bas  
NAME: NP-1-57-600002  
EXPNO: 2  
PROCNO: 1

F2 - Acquisition Parameters  
Date\_ Time: 20201001 17.12  
INSTRUM: av600  
PROBHD: 5 mm QNP1H  
PULPROG: zgpg30  
TD: 98074  
SOLVENT: CHCl3  
NS: 158  
DS: 4  
SWH: 9615.388 Hz  
FIDRES: 0.098042 Hz  
AQ: 5.0998979 sec  
RG: 20.2  
WV: 63.2  
DE: 110.00 usec  
TE: 298.2 K  
D1: 1.0000000 sec  
D8: 0.5000000 sec  
D9: 0.1000000 sec  
D12: 0.1337500 sec  
D22: 0.1539659 sec  
P2: 14.00 usec

==== CHANNEL f1 =====  
SFO1: 600.130130 MHz  
NUC1: 1H  
P1: 9.50 usec  
P3: 24.50 usec  
P4: 19.00 usec  
P5: 24.50 usec  
P12: 80000.00 usec

F2 - Processing parameters  
SI: 32768  
SF: 600.130130 MHz  
WDW: EM  
SSB: 0  
LB: 0.30 Hz  
GB: 0  
PC: 1.00

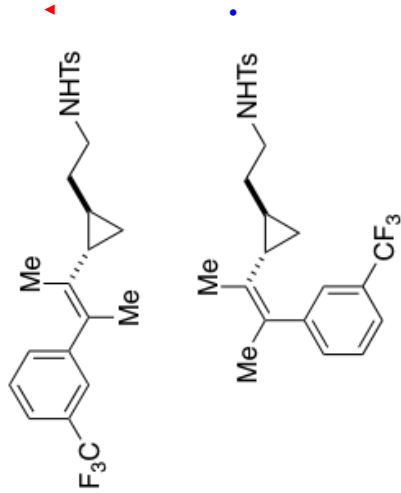
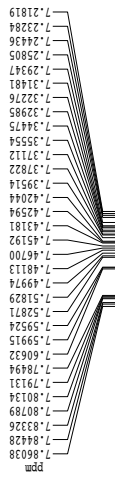
ID MMR Plot parameters  
CX: 22.80 cm  
CY: 14.00 cm  
CZ: 9.00 cm  
F1: 5401.13 Hz  
F2: -0.500 ppm  
F3: -300.06 Hz  
PRWD: 0.4466 ppm/cm  
RGST: 250.0940 Hz/cm



4.8



1H spectrum



4.9  
unpurified

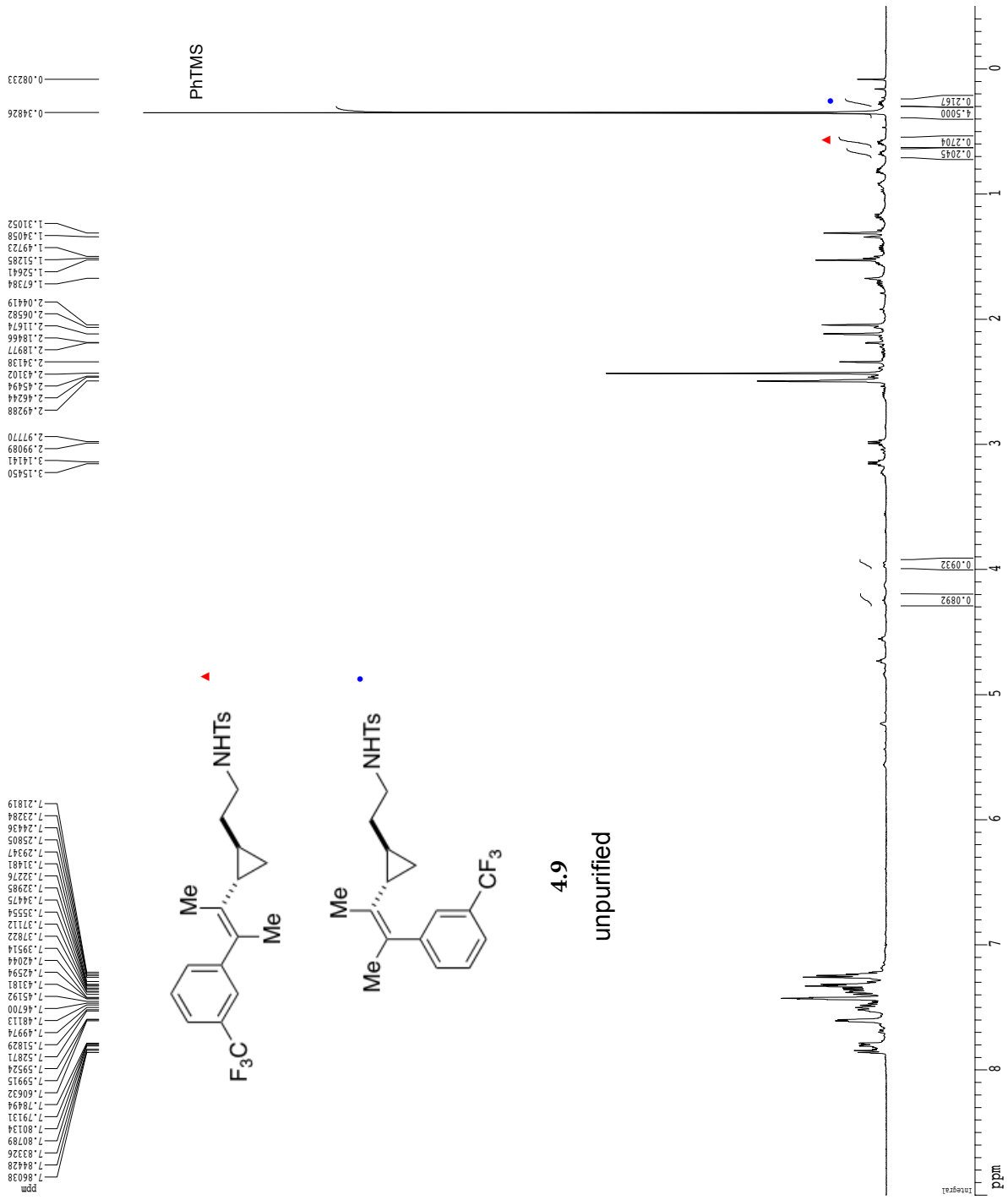
Current Data Parameters  
 USER tthane  
 NAME FATiv134nmr  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20200909  
 Time 11.34  
 INSTRUM gn500  
 PROBHD 5 mm broadband  
 PULPROG zg30  
 TD 81728  
 SOLVENT CDCl3T  
 NS 8  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.098043 Hz  
 AQ 5.0998774 sec  
 RG 114  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 MCREST 0.00000000 sec  
 MCWRK 0.01500000 sec

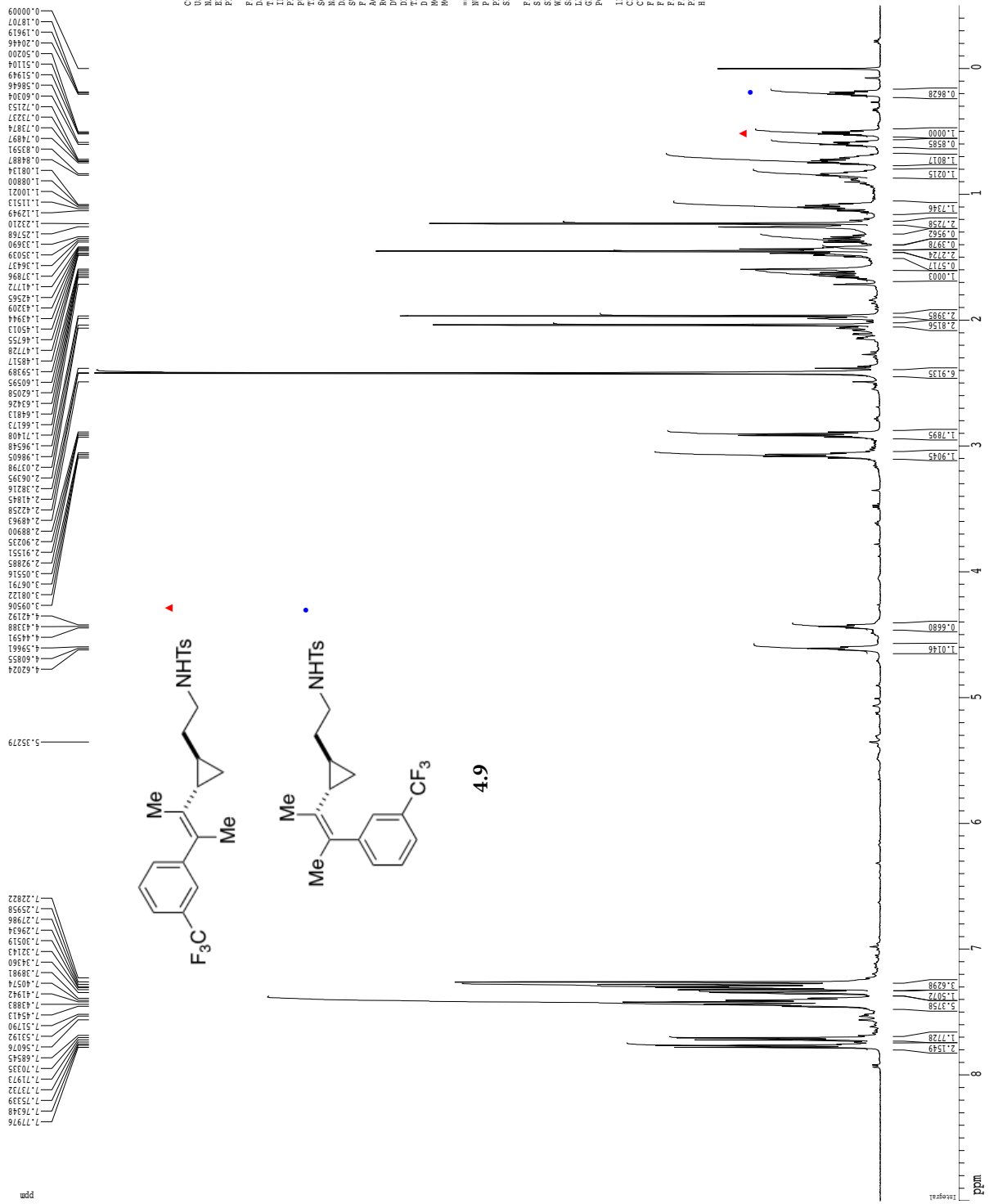
==== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -6.00 dB  
 SF01 498.7534913 MHz

F2 - Processing parameters  
 SI 65536  
 SF 498.7500000 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

ID NMR plot parameters  
 CX 20.00 cm  
 CY 12.50 cm  
 F1P 9.000 ppm  
 F1 4488.75 Hz  
 F2P -0.500 ppm  
 F2 -249.38 Hz  
 PPMCM 0.47500 ppm/cm  
 HZCM 236.90625 Hz/cm



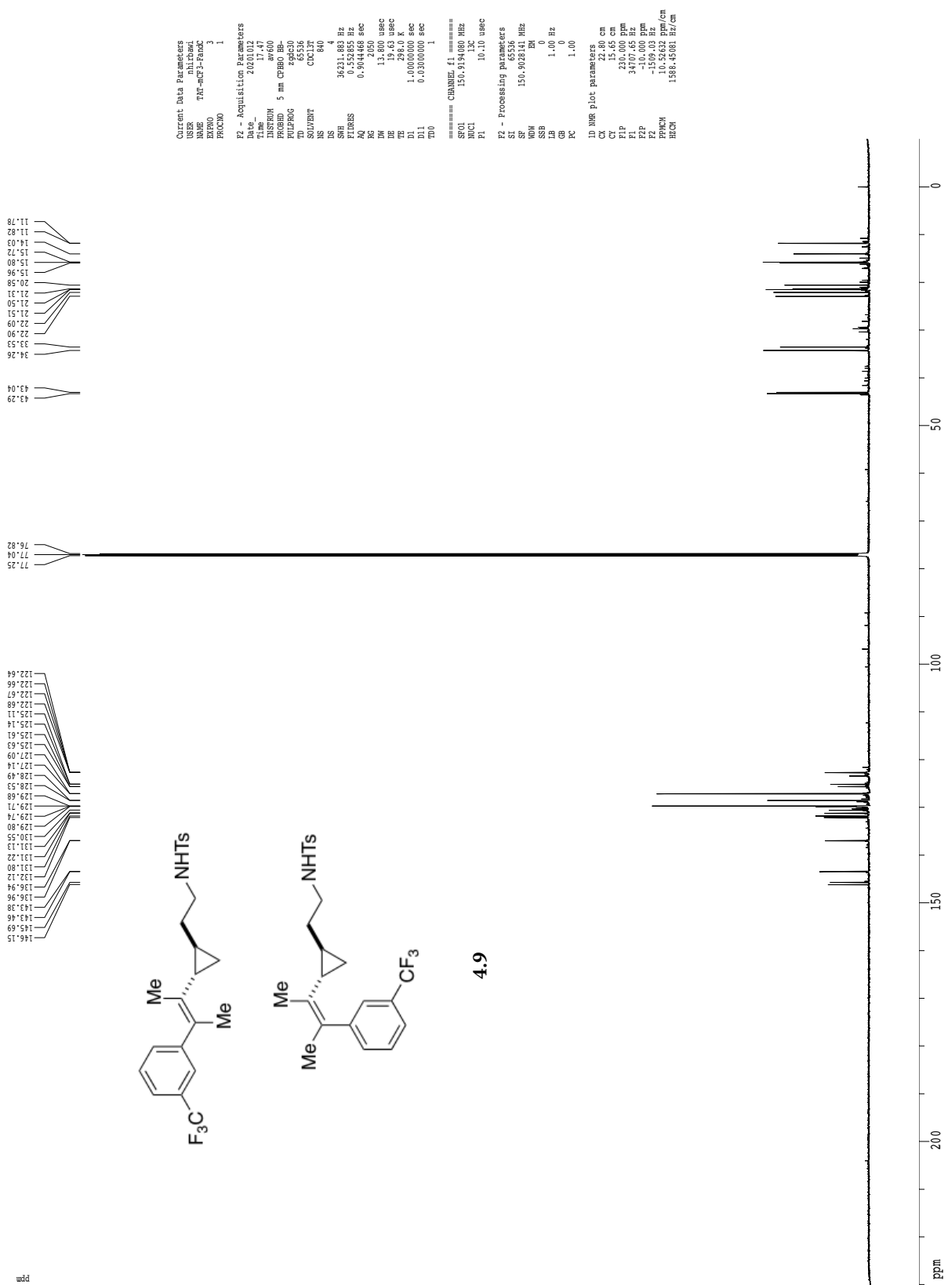
1H spectrum



Current Data Parameters  
 USER: TWT-MC73-HO35C  
 NAME: TWT-MC73-HO35C  
 EXPNO: 1  
 PROCNO: 1

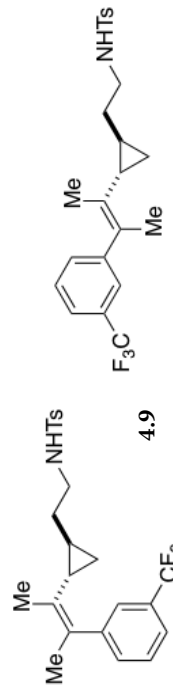
F2 - Acquisition Parameters  
 Date\_: 20201010  
 Time: 13.14  
 PULPROG: zgpg30  
 PROGRAM: 5 nm C131-1H  
 TD: 8178  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 8012.820 Hz  
 FIDRES: 0.09804 Hz  
 AQ: 5.1998774 sec  
 RG: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 0.16000000 sec  
 T2: 0.00000000 sec  
 T3: 0.00000000 sec  
 T4: 0.00000000 sec  
 T5: 0.00000000 sec  
 T6: 0.00000000 sec  
 T7: 0.00000000 sec  
 T8: 0.00000000 sec  
 T9: 0.00000000 sec  
 T10: 0.00000000 sec  
 T11: 0.00000000 sec  
 T12: 0.00000000 sec  
 T13: 0.00000000 sec  
 T14: 0.00000000 sec  
 T15: 0.00000000 sec  
 T16: 0.00000000 sec  
 T17: 0.00000000 sec  
 T18: 0.00000000 sec  
 T19: 0.00000000 sec  
 T20: 0.00000000 sec  
 T21: 0.00000000 sec  
 T22: 0.00000000 sec  
 T23: 0.00000000 sec  
 T24: 0.00000000 sec  
 T25: 0.00000000 sec  
 T26: 0.00000000 sec  
 T27: 0.00000000 sec  
 T28: 0.00000000 sec  
 T29: 0.00000000 sec  
 T30: 0.00000000 sec  
 T31: 0.00000000 sec  
 T32: 0.00000000 sec  
 T33: 0.00000000 sec  
 T34: 0.00000000 sec  
 T35: 0.00000000 sec  
 T36: 0.00000000 sec  
 T37: 0.00000000 sec  
 T38: 0.00000000 sec  
 T39: 0.00000000 sec  
 T40: 0.00000000 sec  
 T41: 0.00000000 sec  
 T42: 0.00000000 sec  
 T43: 0.00000000 sec  
 T44: 0.00000000 sec  
 T45: 0.00000000 sec  
 T46: 0.00000000 sec  
 T47: 0.00000000 sec  
 T48: 0.00000000 sec  
 T49: 0.00000000 sec  
 T50: 0.00000000 sec  
 T51: 0.00000000 sec  
 T52: 0.00000000 sec  
 T53: 0.00000000 sec  
 T54: 0.00000000 sec  
 T55: 0.00000000 sec  
 T56: 0.00000000 sec  
 T57: 0.00000000 sec  
 T58: 0.00000000 sec  
 T59: 0.00000000 sec  
 T60: 0.00000000 sec  
 T61: 0.00000000 sec  
 T62: 0.00000000 sec  
 T63: 0.00000000 sec  
 T64: 0.00000000 sec  
 T65: 0.00000000 sec  
 T66: 0.00000000 sec  
 T67: 0.00000000 sec  
 T68: 0.00000000 sec  
 T69: 0.00000000 sec  
 T70: 0.00000000 sec  
 T71: 0.00000000 sec  
 T72: 0.00000000 sec  
 T73: 0.00000000 sec  
 T74: 0.00000000 sec  
 T75: 0.00000000 sec  
 T76: 0.00000000 sec  
 T77: 0.00000000 sec  
 T78: 0.00000000 sec  
 T79: 0.00000000 sec  
 T80: 0.00000000 sec  
 T81: 0.00000000 sec  
 T82: 0.00000000 sec  
 T83: 0.00000000 sec  
 T84: 0.00000000 sec  
 T85: 0.00000000 sec  
 T86: 0.00000000 sec  
 T87: 0.00000000 sec  
 T88: 0.00000000 sec  
 T89: 0.00000000 sec  
 T90: 0.00000000 sec  
 T91: 0.00000000 sec  
 T92: 0.00000000 sec  
 T93: 0.00000000 sec  
 T94: 0.00000000 sec  
 T95: 0.00000000 sec  
 T96: 0.00000000 sec  
 T97: 0.00000000 sec  
 T98: 0.00000000 sec  
 T99: 0.00000000 sec  
 T100: 0.00000000 sec

13C spectrum with 1H decoupling

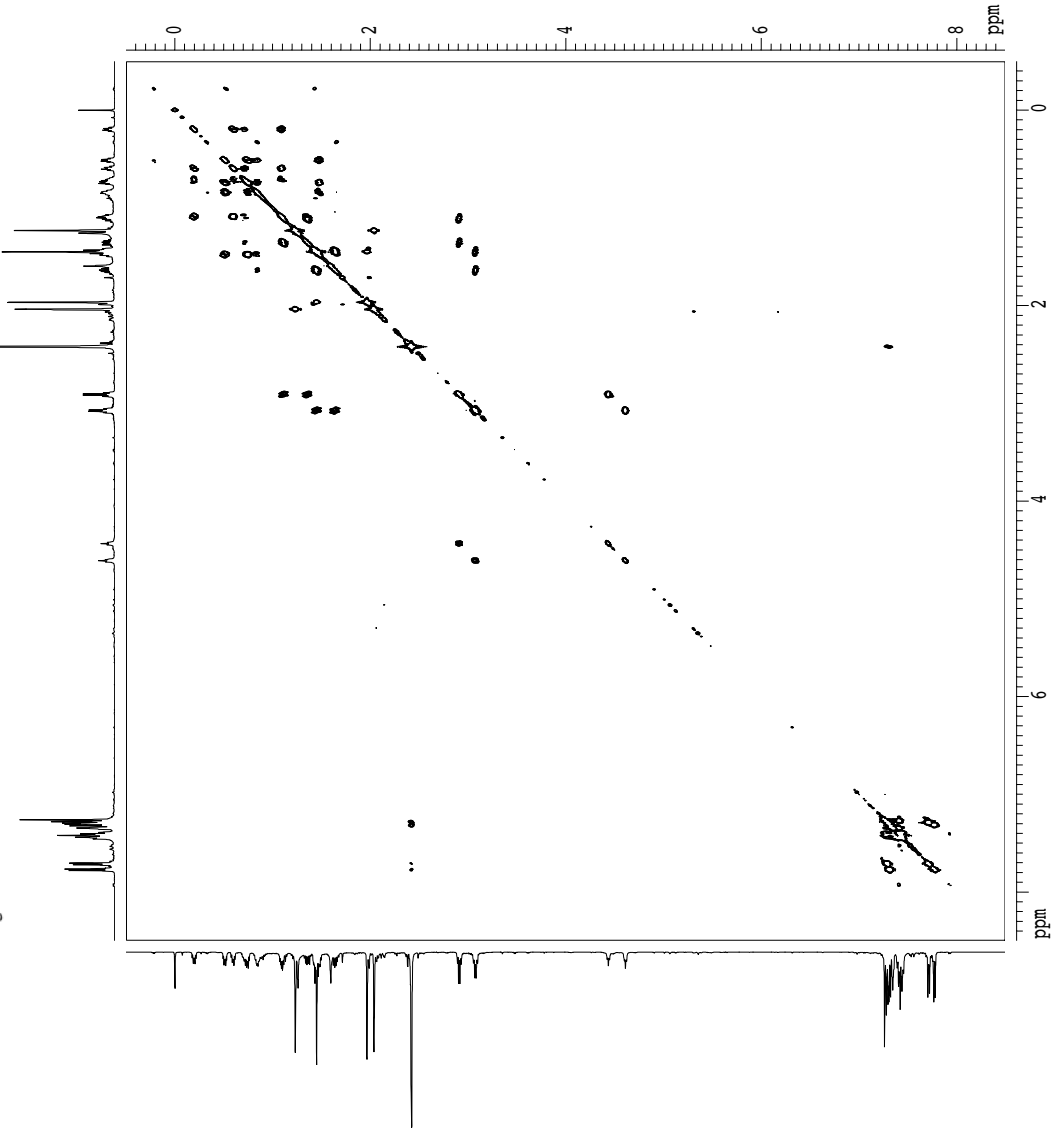


```

Current Data Parameters
=====
NAME          13C-1
EXPNO         3
PROCNO        1
F2 - Acquisition Parameters
=====
Date_         20201012
Time          17.47
INSTRUM       av600
PROBHD        5 mm CPBBO-1
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            640
DS            4
SF            36231.884 Hz
FIDRES       0.555855 Hz
AQ           0.8044466 sec
RG           2050
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
TE           298.2 K
D1            1.0000000 sec
D11           0.0300000 sec
TD0           1
===== CHANNEL f1 =====
SFO1         150.8134080 MHz
NUC1          13C
P1           10.10 usec
F2 - Processing parameters
=====
SI            65536
SF           150.8028144 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC           1.00
ID NMR PLOT parameters
=====
CX           22.89 cm
CY           15.65 cm
FIDP         230.000 ppm
F1P          340.000 Hz
F2P          -1500.00 Hz
PPMVAL       10.52632 ppm/cm
RECH         1588.45084 Hz/cm
    
```



gc05y60



Current Data Parameters  
 USER NAME TAT-MCF3-HCOSYC  
 EXPRO 2  
 PRCKNO 1

F2 - Acquisition Parameters  
 Date\_ 20201010  
 Time\_ 13:17  
 INSTRUM crys500  
 PROBHD 5 mm CPCTC IH-  
 PULPROG zgpg30  
 SOLVENT cosypp60  
 NS 2  
 DS 16  
 SFR 4496.403 Hz  
 FIDRES 0.22559 Hz  
 RG 203.2  
 DW 111.200 usec  
 DE 6.00 usec  
 TE 298.2 K  
 D1 0.000000 sec  
 D11 1.000000 sec  
 d13 0.00000300 sec  
 D16 0.00020000 sec  
 INO 0.0002240 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.50 usec  
 PLL 1.60 dB  
 SFO1 500.220330 MHz

===== CHANNEL CHANNEL =====  
 GPM1M size:100  
 GPM1M2 size:100  
 GPC1 0.00 %  
 GPC2 0.00 %  
 GPF1 0.00 %  
 GPF2 0.00 %  
 GPC1 17.00 %  
 GPC2 17.00 %  
 P16 1000.00 usec

F1 - Acquisition Parameters  
 NU0 256  
 TD 256  
 SFO1 500.222 MHz  
 FIDRES 17.564074 Hz  
 SN 8.393 ppm  
 FWHM 0.7

F2 - Processing parameters  
 SI 1024  
 SF 500.220332 MHz  
 SDE  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 16  
 SF 500.220332 MHz  
 SDE  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CXZ 15.00 cm  
 CIL 15.00 cm  
 FZLO 436.494 ppm  
 FZHI -0.495 ppm  
 FZHL -241.416 Hz  
 FZLO 8.494 ppm  
 FZLO 4246.96 Hz  
 FZHI -241.416 ppm  
 FZHL -241.416 ppm  
 FZPMCH 0.55926 ppm/cm  
 FZPCH 295.76016 Hz/cm  
 FZPMCH 0.55926 ppm/cm  
 FZPCH 295.76016 Hz/cm

gnoe

100

Current Data Parameters  
 USER nhlrbawd  
 NAME TMT-MC72-NMR1S1S  
 EXPNO 2  
 PROCNO 1

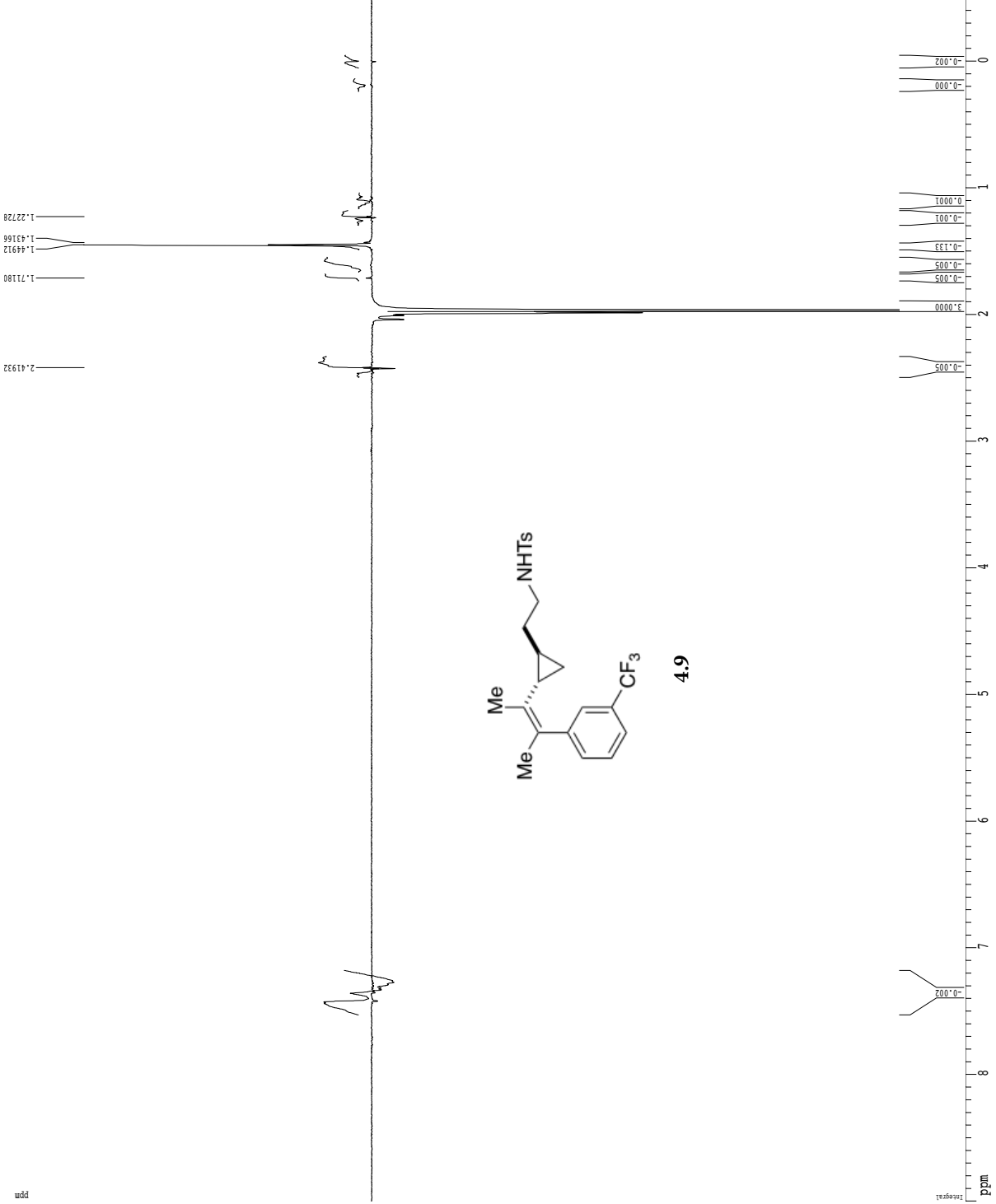
F2 - Acquisition Parameters  
 Date\_ 20201013  
 Time 16:40  
 INSTRUM cryo  
 PROBHD 5 mm CPYCI 1H  
 PULPROG gnoe.coc22.pcd  
 TD 65536  
 CHANID1 158  
 NS 8  
 SH 8012.822 Hz  
 FIDRES 0.122266 Hz  
 AQ 4.103696 sec  
 RG 181.9  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 300.2 K  
 DS 1.000200  
 DB 0.5000000 sec  
 D16 0.00020000 sec  
 dZ1 0.33376500 sec  
 dZ2 0.18398699 sec  
 PC 15.00 usec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 1.00 usec  
 PL 0.00 dB  
 P2 22.50 usec  
 PL 30.00 dB  
 P3 30.00 usec  
 PL 24.00 dB  
 P4 40000.00 usec  
 PL 0.00 dB  
 P5 500.2210144 MHz  
 SFO1 61.66 dB  
 SFO9 0.00 Hz  
 SFO10 0.00 Hz  
 SFO11 0.00 Hz  
 SFO12 0.00 Hz

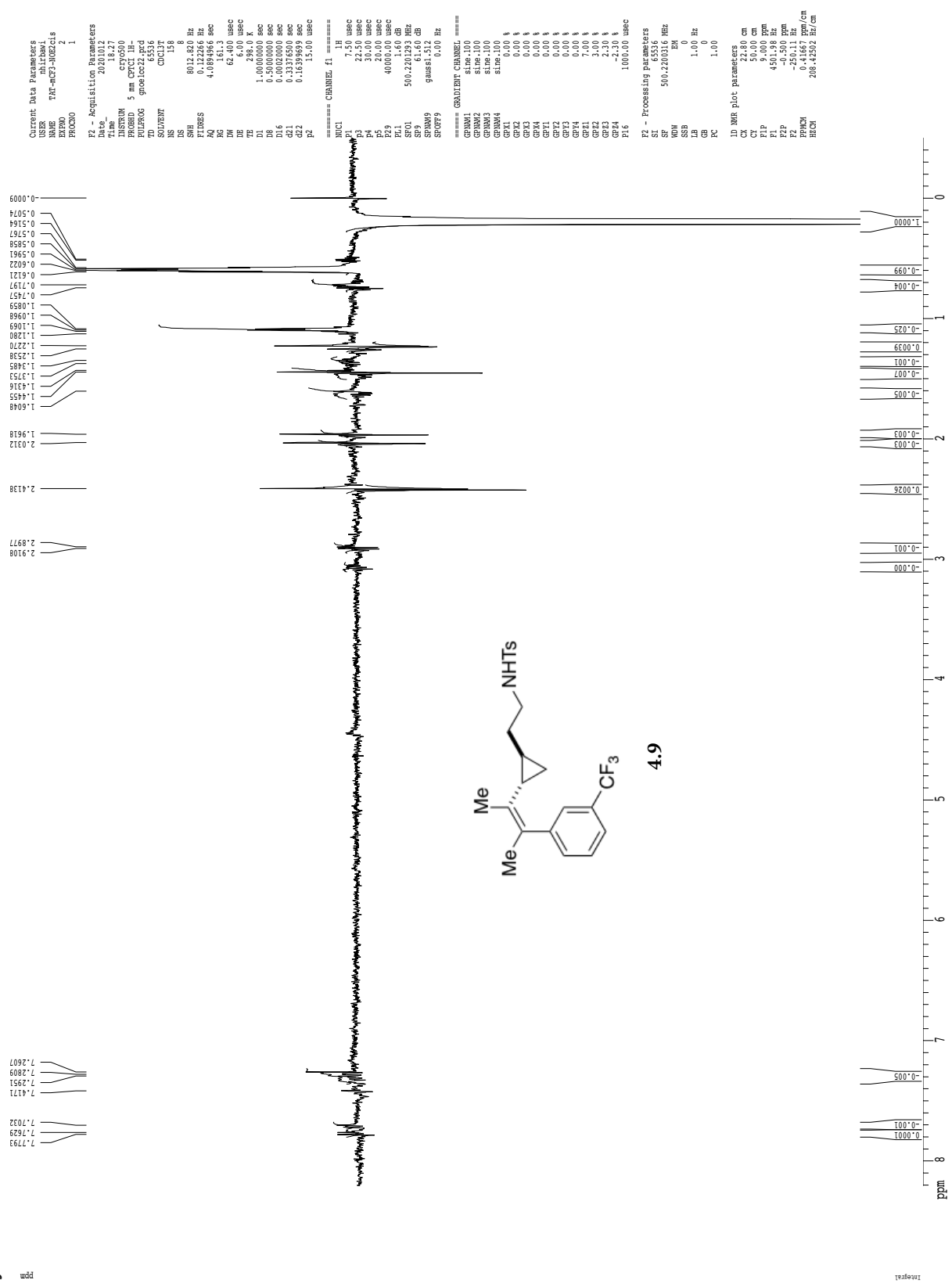
===== GRADIENT CHANNEL =====  
 GGRAD1 size:1.00  
 GGRAD2 size:1.00  
 GGRAD3 size:1.00  
 GGRAD4 size:1.00  
 GXY1 0.00 V  
 GXY2 0.00 V  
 GXY3 0.00 V  
 GXY4 0.00 V  
 GXY5 0.00 V  
 GXY6 0.00 V  
 GXY7 0.00 V  
 GXY8 0.00 V  
 GXY9 0.00 V  
 GXY10 0.00 V  
 GXY11 0.00 V  
 GXY12 0.00 V  
 GXY13 0.00 V  
 GXY14 0.00 V  
 GXY15 0.00 V  
 GXY16 0.00 V  
 P16 1000.00 usec  
 P17 -2.20 V  
 P18 1000.00 usec

F2 - Processing Parameters  
 SI 65536  
 SF 500.2200315 MHz  
 DS 8  
 SFO 0.00 Hz  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

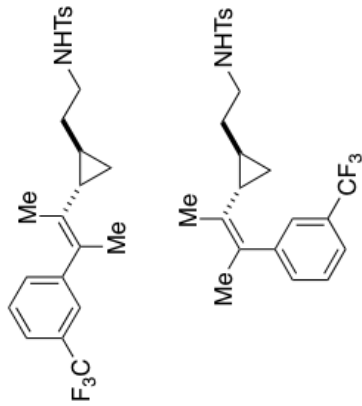
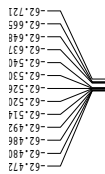
ID MR parameters  
 CX 22.88 cm  
 CY 50.00 cm  
 FIP 9.000 ppm  
 F2 4.900 ppm  
 FZ -250.11 Hz  
 FPMCH 0.41667 ppm/cm  
 RECN 200.42502 Hz/cm



gnoe



19F spectrum with inverse gated 1H decoupling



4.9

```

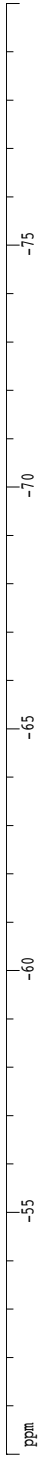
Current Data Parameters
USER          mhcbvnl
EXPNO        2
PROCNO       1

F2 - Acquisition Parameters
Date_         20110712
Time         17:36
INSTRUM      av600
PROBHD       5 mm CPBBO BB-
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           16
DS           4
AQ           118571.432 Hz
FIDRES       1.362492 Hz
RG           0.3670516 sec
AQ           0.275
RG           18.00 usec
DE           298.0 K
TE           298.0 K
D1           3.0000000 sec
D11          0.0300000 sec
D3           0.0002000 sec
TD0          1

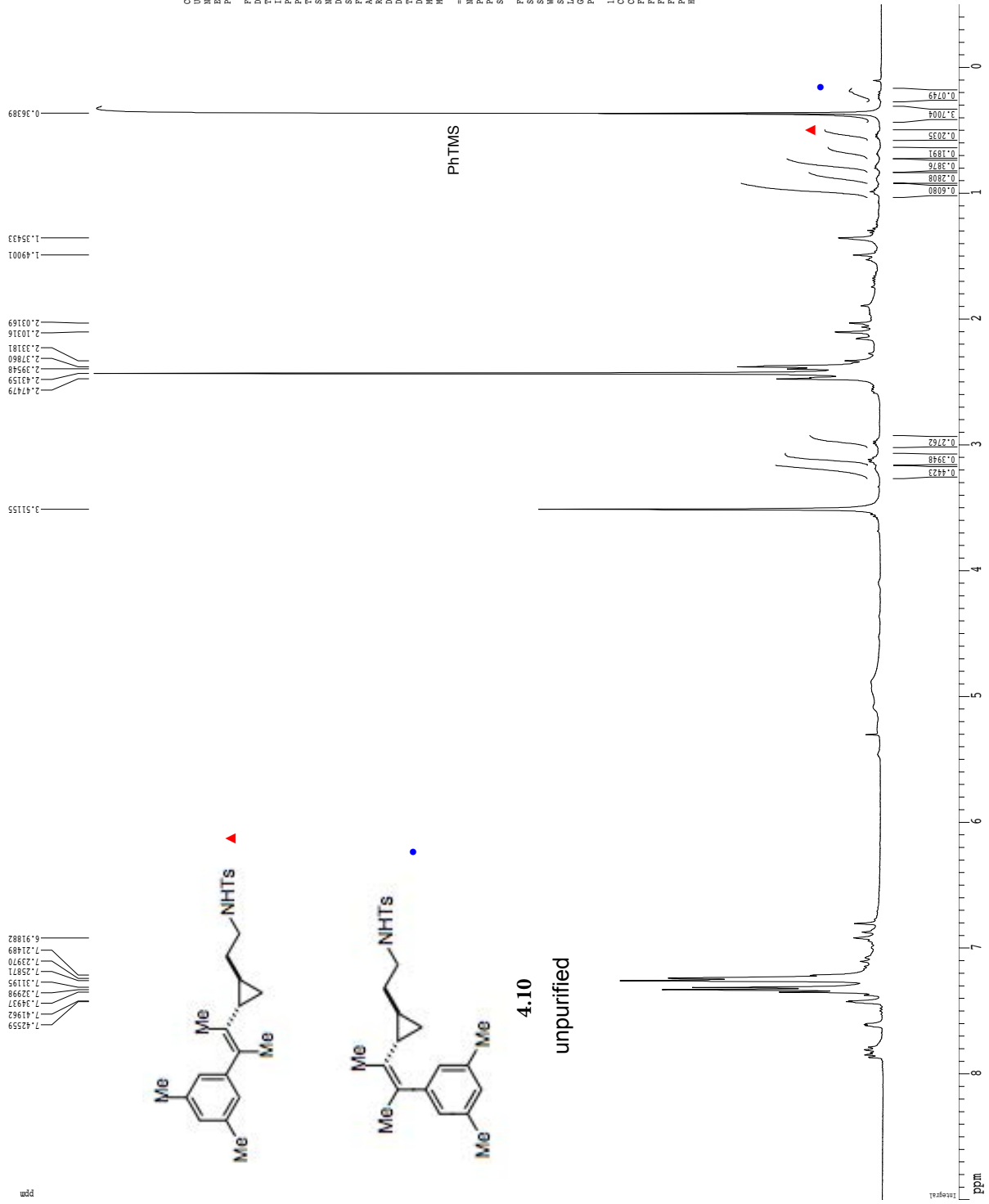
===== CHANNEL f1 =====
NUC1          19F
P1           18.25 usec

F2 - Processing parameters
SI           564.484408 MHz
SF           564.484408 MHz
WDW          no
SSB          0
LB           0.10 Hz
GB           0
PC           1.00

ID NAME plot parameters
CY          15.00 cm
CT          50.000 ppm
F1P         -50.4000 ppm
F1          -28234.32 Hz
F2          -45.0000 ppm
F3          -45.0000 ppm
PRGCM       1.31579 ppm/cm
HZCM        743.00842 Hz/cm
  
```



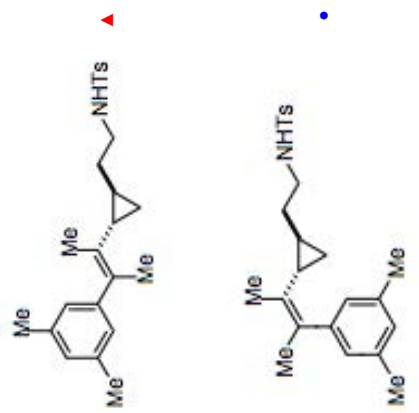
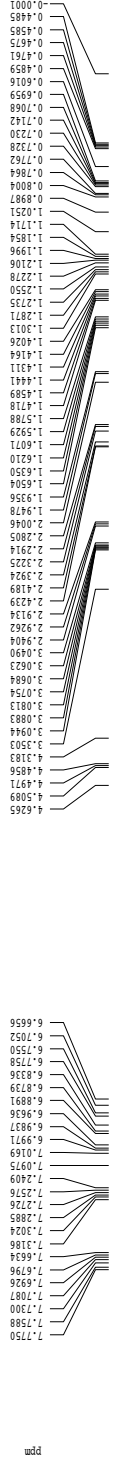
1H spectrum



Current Data Parameters  
 USER karewitt1  
 NAME K08-111-283-crude  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20200923  
 Time 11:06  
 PROBHD 5 mm QNP HIF/PP  
 PULPROG zg30  
 TD 38460  
 SFOV 10.000000  
 ACOUENT COCL19  
 NS 2  
 DS 2  
 SWH 6410.256 Hz  
 FIDRES 0.000000 Hz  
 AQ 2.599934 sec  
 RG 31  
 DW 78.400 usec  
 DE 4.50 usec  
 DI 21.50 usec  
 D1 0.1000000 sec  
 MCREST 0.0000000 sec  
 MCORR 0.0500000 sec  
 ===== CHANNEL f1 =====  
 NU1 1H  
 P1 12.00 usec  
 PL 0.00 dB  
 SFO1 400.132000 MHz  
 F2 - Processing parameters  
 SI 65516  
 SF 400.132000 MHz  
 RG 31  
 WFO 0  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 2.00  
 ID NMR plot parameters  
 CX 12.80 cm  
 CY 12.80 cm  
 FID 9.000 ppm  
 F1 3601.17 Hz  
 F2 -0.500 ppm  
 F3 0.000 ppm  
 FWHM 0.41663 ppm/cm  
 HCN 166.72084 Hz/cm



1H spectrum



4.10

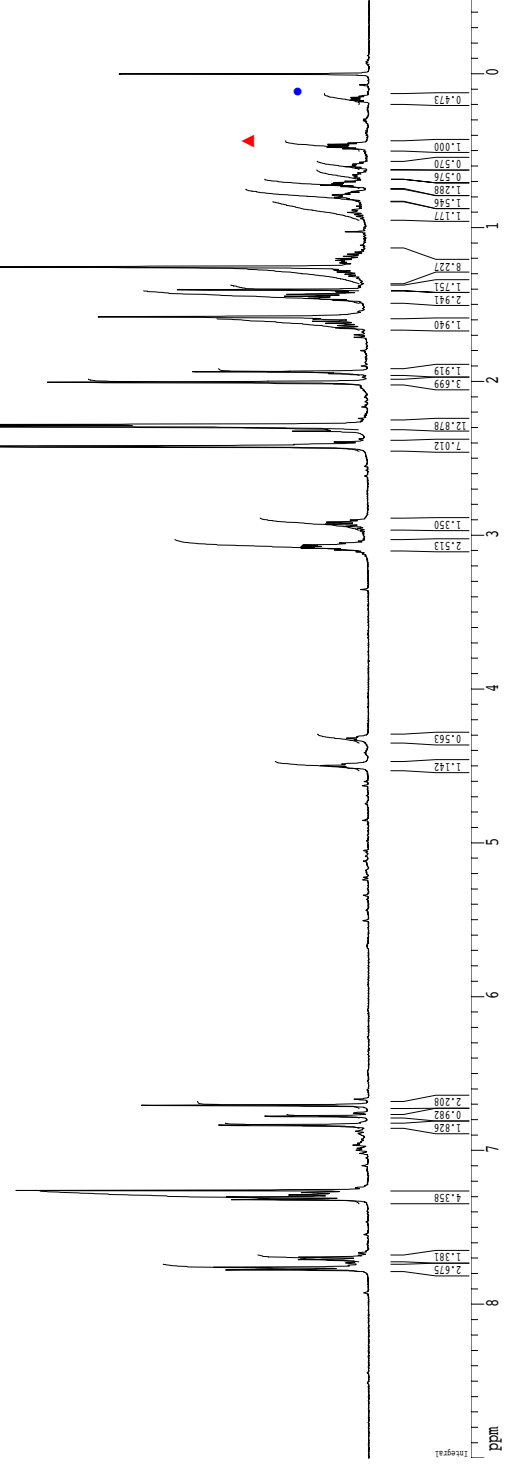
Current Data Parameters  
 USER: KRW  
 NAME: KRF-III-281-2  
 EXNO: 2  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20201010  
 Time: 12.48  
 INSTRUM: spect  
 PROBHD: 5 mm CPY131-4H  
 PULPROG: zgpg30  
 TD: 48074  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 8012.820 Hz  
 FIDRES: 0.166677 Hz  
 AQ: 2.3398677 sec  
 RG: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 0.10000000 sec  
 MCHRG1: 0.00000000 sec  
 MCHRG2: 0.00000000 sec  
 MCHRG3: 0.00000000 sec

===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 1.50 usec  
 PL1: 1.66 dB  
 SFO1: 500.2235015 MHz

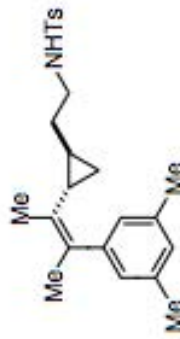
F2 - Processing Parameters  
 SI: 65536  
 SF: 500.2200332 MHz  
 WDW: no  
 SSB: 0  
 GB: 0  
 PC: 1.00

D0 NMR file parameters  
 CY: 22.80 cm  
 CX: 15.00 cm  
 FIP: 9.000 ppm  
 F1: 4501.500 Hz  
 F2: -250.11 Hz  
 PPRGM: 0.41667 ppm/cm  
 HZCM: 206.44502 Hz/cm

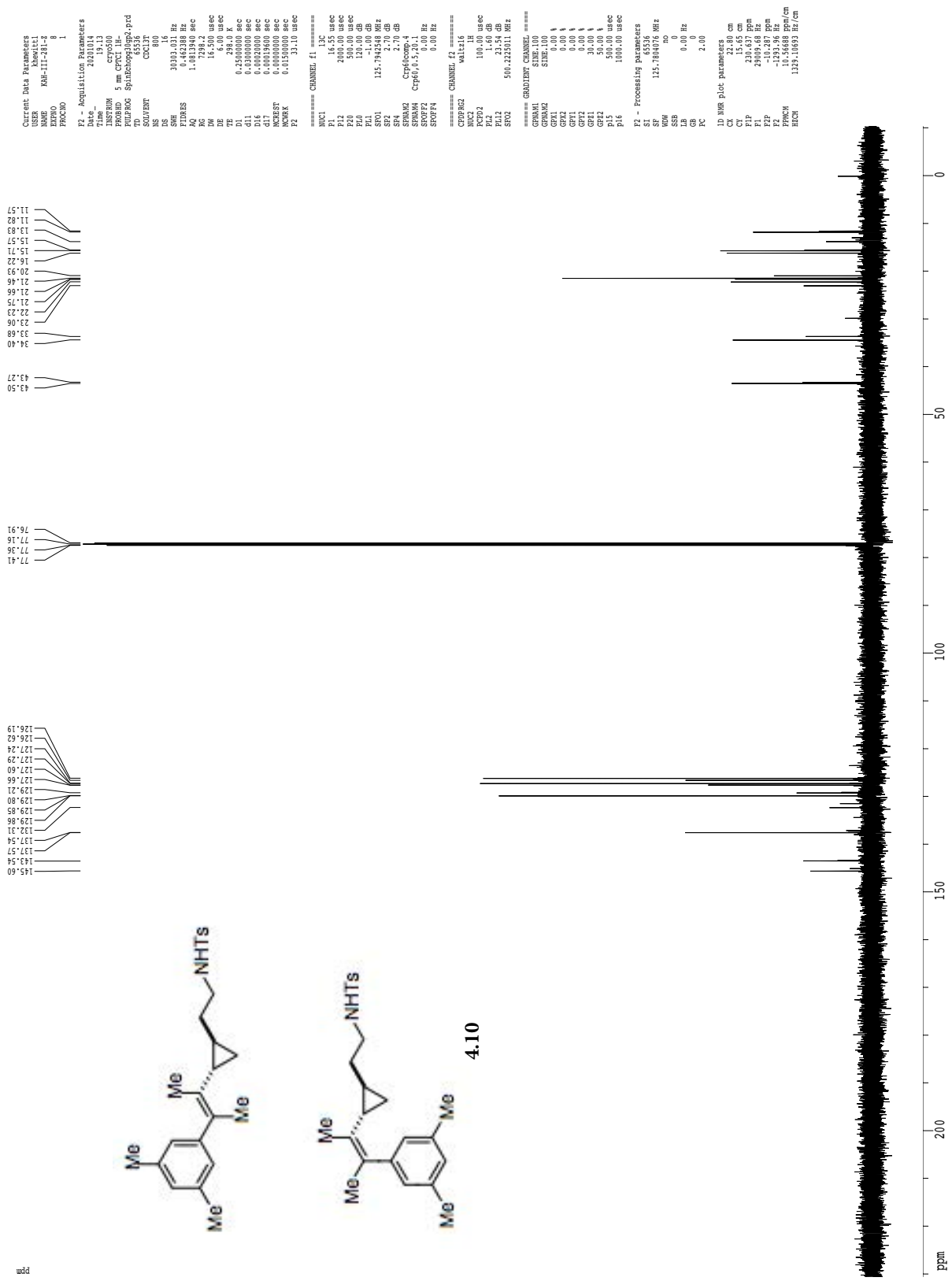


Z-restored spin-echo 13C spectrum with 1H decoupling

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4.10





gcosy60

4.10

```

Current Data Parameters
USR      20201010
Name     gcosy60
EXPNO   1
PROCNO  1

F2 - Acquisition Parameters
Date_    20201010
Time     15.00
INSTRUM  crys500
PROBHD   5 mm CPCL1 1H-
PULPROG  cosygp60.prd
TD       2048
AQ       0.1278452
RG       327.00
DE       6.00
TE       298.0 K
d0       0.0000300 sec
d1       1.0000000 sec
d11      0.0000000 sec
d16      0.0002000 sec
IN0      0.00012480 sec

===== CHANNEL f1 =====
NUC1     13C
P1       7.15 usec
PL1      1.40 dB
SFO1     500.2235015 MHz

===== CHANNEL f2 =====
CPDPRG2  gpg2
SOLVENT  CDCl3
GPR2     0.00 %
GP2      0.00 %
GP2      0.00 %
GP2      0.00 %
GP2      17.00 %
P16      1000.00 usec

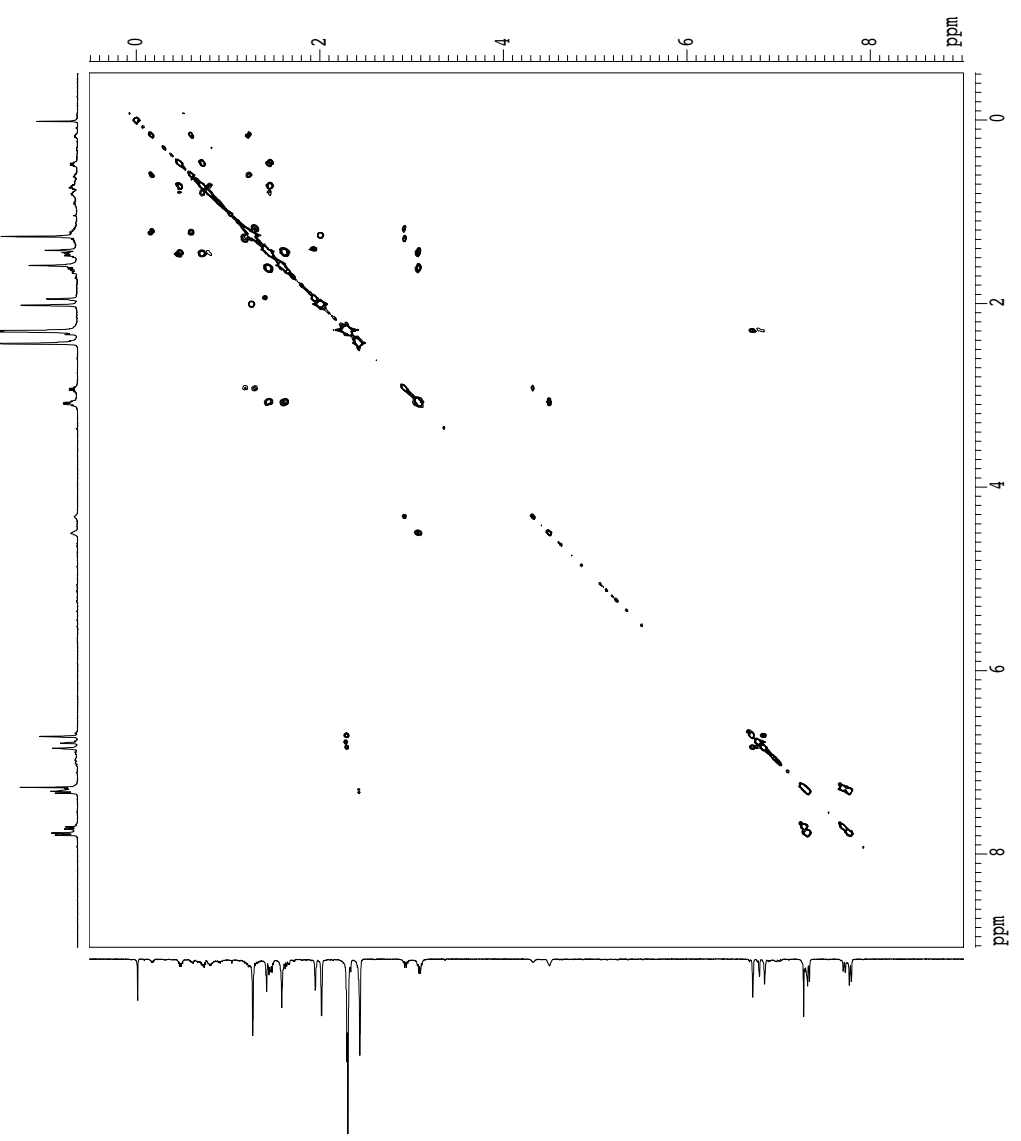
F1 - Acquisition Parameters
NUC1     13C
P1       7.15 usec
PL1      1.40 dB
SFO1     500.2235015 MHz

===== CHANNEL f2 =====
CPDPRG2  gpg2
SOLVENT  CDCl3
GPR2     0.00 %
GP2      0.00 %
GP2      0.00 %
GP2      0.00 %
GP2      17.00 %
P16      1000.00 usec

F2 - Processing Parameters
SI       1024
SF       500.2200332 MHz
WDW      SINE
SSB      0
GB       0
PC       1.00

F1 - Processing Parameters
SI       1024
SF       500.2200332 MHz
WDW      SINE
SSB      0
GB       0

2D NMR Plot Parameters
CX1      15.00 cm
FZP10    5.014 ppm
FZP11    45.014 ppm
FZP12    -10.513 ppm
FZP13    -256.39 Hz
F1P10    8.014 ppm
F1P11    45.094 ppm
F1P12    -10.513 ppm
F2P10CM  0.63511 ppm/cm
F2P11CM  317.69580 Hz/cm
F2P12CM  0.63511 ppm/cm
F2P13CM  317.69580 Hz/cm
  
```



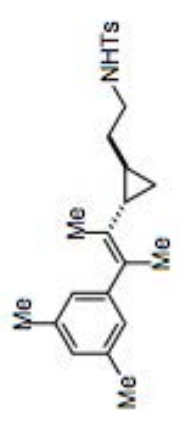
gnoe

ppm



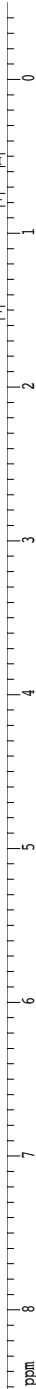
```

Current Data Parameters
NAME      khawatt1
EXPNO    5
PROCNO   1
=====
F2 - Acquisition Parameters
Date_    20201010
Time     17:36
PROBHD   5 mm CRYD1 H
PULPROG  zgpg30
PCPDPRG2  gnoe1cc22.prd
TD        65536
SOLVENT  CDCl3
NS        128
DS        4
SWH       8012.622 Hz
FIDRES    0.122246 Hz
AQ        4.089496 sec
RG         62.000
AQ        4.000000 sec
TE        298.0 K
DL        1.0000000 sec
DE        0.1800000 sec
DI        0.0000000 sec
DQ        0.33375000 sec
dZ1       0.33375000 sec
dZ2       0.1639669 sec
F2        15.00 usec
===== CHANNEL f1 =====
NUC1      1H
P1        7.50 usec
PL1       0.00 dB
PC1       22.50 usec
RG1       20.000
RF1       40000.00 usec
SFO1      500.220666 MHz
WDW        EM
SSB        0.00 dB
GB         0.00 Hz
SF        500.130
===== GRADIENT CHANNEL =====
GAMMA1    1H
P1         0.00 usec
PL1        0.00 dB
PC1        22.50 usec
RG1       20.000
RF1       40000.00 usec
SFO1      500.220666 MHz
WDW        EM
SSB        0.00 dB
GB         0.00 Hz
SF        500.130
=====
F2 - Processing parameters
SI        65536
SF        500.220666 MHz
WDW        EM
SSB        0
GB         0.00 Hz
PC        1.00
=====
ID NMR Plot parameters
CX        22.88 cm
CY        50.00 cm
PR        300.000000 ppm
PIP       650.000000 ppm
FZP       -0.500000 ppm
F2        -250.11 Hz
PPMCM     0.41667 ppm/cm
HZCM      208.42500 Hz/cm
  
```

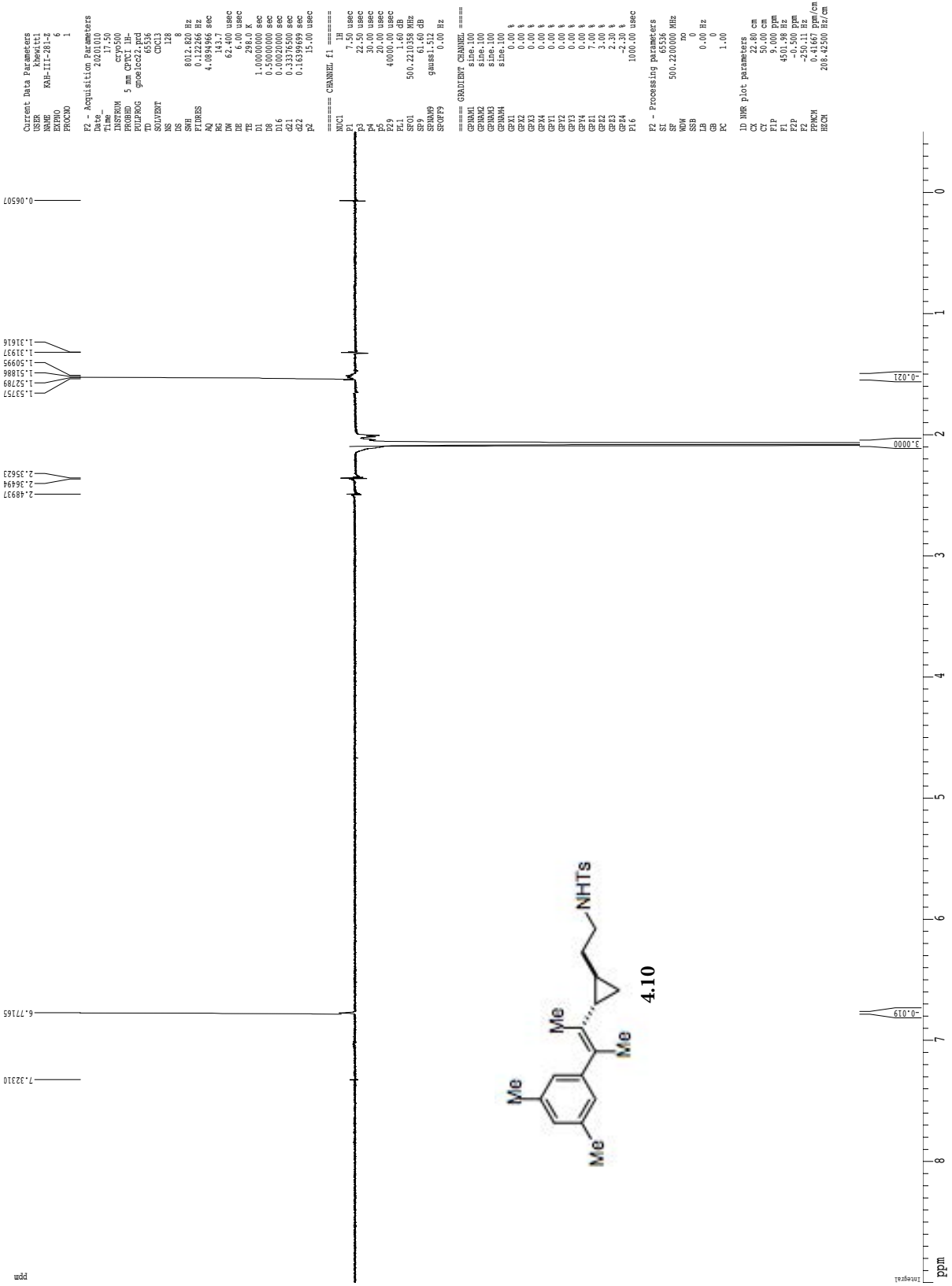


4.10

Integral

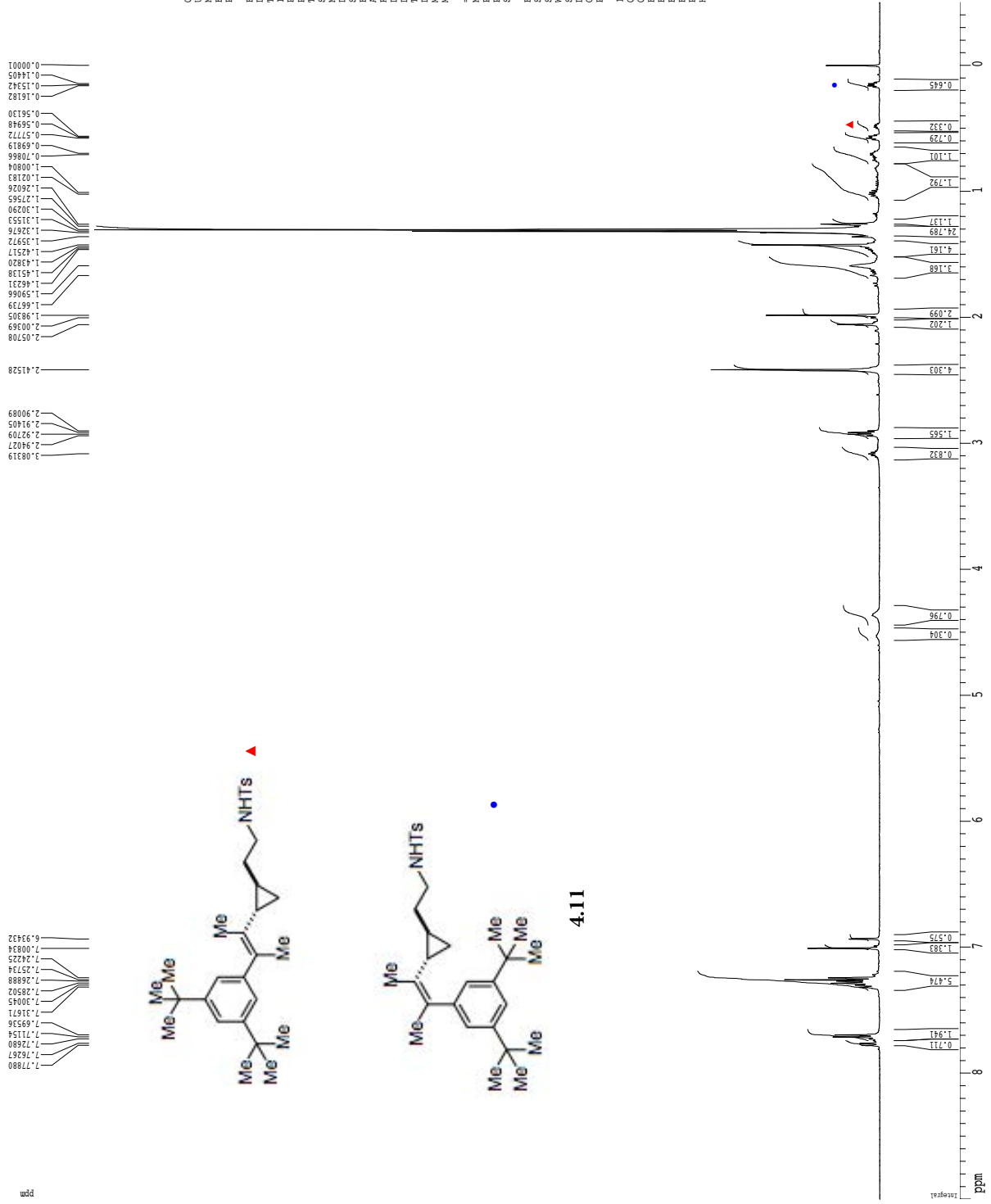


gnoe





1H spectrum



Current Data Parameters  
USER: Kshmitt  
NAME: KSH-III-190-2  
EXNO: 2  
PROCNO: 1

F2 - Acquisition Parameters  
Date: 20200911  
Time: 10:40  
INSTRUM: cryo  
PROBHD: 5 mm CRY1 1H  
PULPROG: zgpg30  
TD: 48074  
SOLVENT: CDCl3  
DS: 9  
SFO: 500.136261 MHz  
SHE: 8012.820 Hz  
FIDRES: 0.16667 Hz  
AQ: 2.399867 sec  
RG: 62.400 usec  
DE: 6.00 usec  
TE: 298.0 K  
T1: 0.100000 sec  
MCBRES: 0.000000 sec  
MORREK: 0.0050000 sec

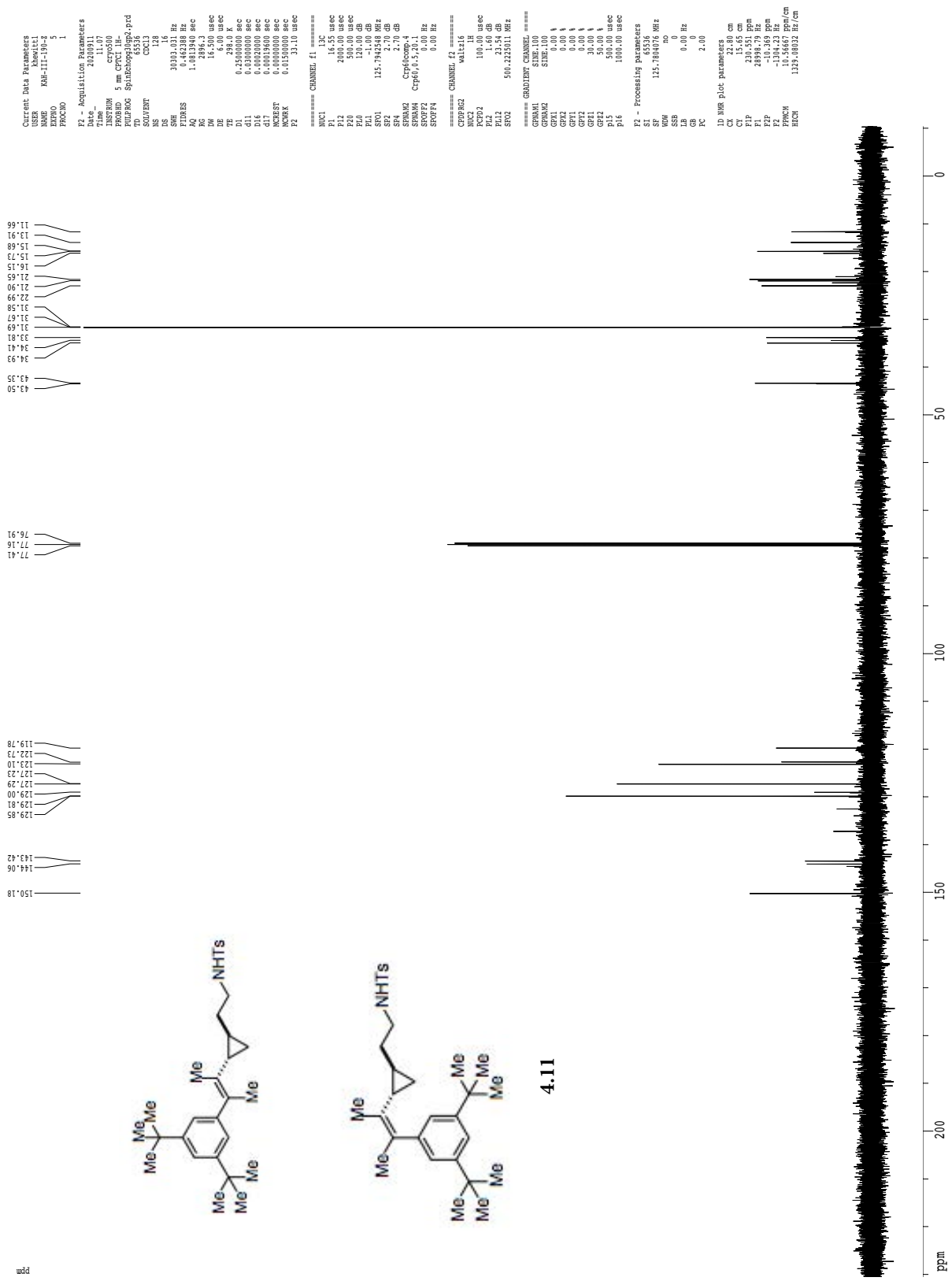
===== CHANNEL f1 =====  
NUC1: 1H  
P1: 1.50 usec  
PL1: 1.66 dB  
SFO1: 500.2235015 MHz

F2 - Processing Parameters  
SI: 65536  
SF: 500.220031 MHz  
WDW: no  
SSB: 0  
GB: 0  
PC: 1.00

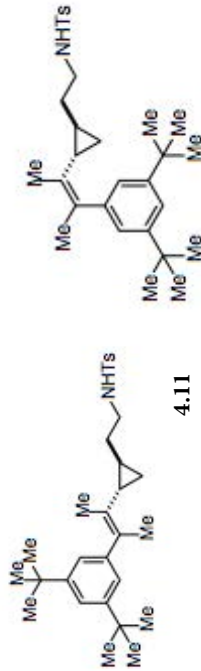
1D NMR P102 Parameters  
CY: 22.80 cm  
C1: 15.00 cm  
F1P: 9.000 ppm  
F1: 49019.88 Hz  
F2: 250.136 MHz  
PRGM: 0.44667 ppm/cm  
HCHN: 208.44502 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling

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Current Data Parameters  
 USER kbevitcl  
 NAME KRM-II-190-2  
 EXPNO 4  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20200911  
 Time 10:55  
 INSTRUM cryo000  
 PULPROG zgpg30  
 FIDRES 0.15  
 PULPROG cosypp60-nd  
 TD 2048  
 SOLVENT ccd13  
 NS 1  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 3.912510 Hz  
 AQ 0.1278452 sec  
 RG 181  
 DW 62.400 usec  
 DE 1.00 usec  
 TE 298.0 K  
 D0 0.40000300 sec  
 D1 1.40000000 sec  
 d13 0.00000300 sec  
 L16 0.00020000 sec  
 L18 0.00014000 sec

===== CHANNEL f1 =====

NUC1 1H  
 P1 7.50 usec  
 PL 0.00 dB  
 SFO1 500.2235015 MHz

===== GRADIENT CHANNEL =====

GRAN1 sine,100  
 GRAN2 sine,100  
 GRAMZ 0.00 %  
 GP1 0.00 %  
 GP2 0.00 %  
 GP3 17.00 %  
 GP4 17.00 %  
 P16 1000.00 usec

F1 - Acquisition parameters

ND0 1  
 SI 512  
 SF 500.2235 MHz  
 SFO1 500.2235 MHz  
 FIDRES 15.650040 Hz  
 SW 16.018 ppm  
 FWHM 0.6

F2 - Processing parameters

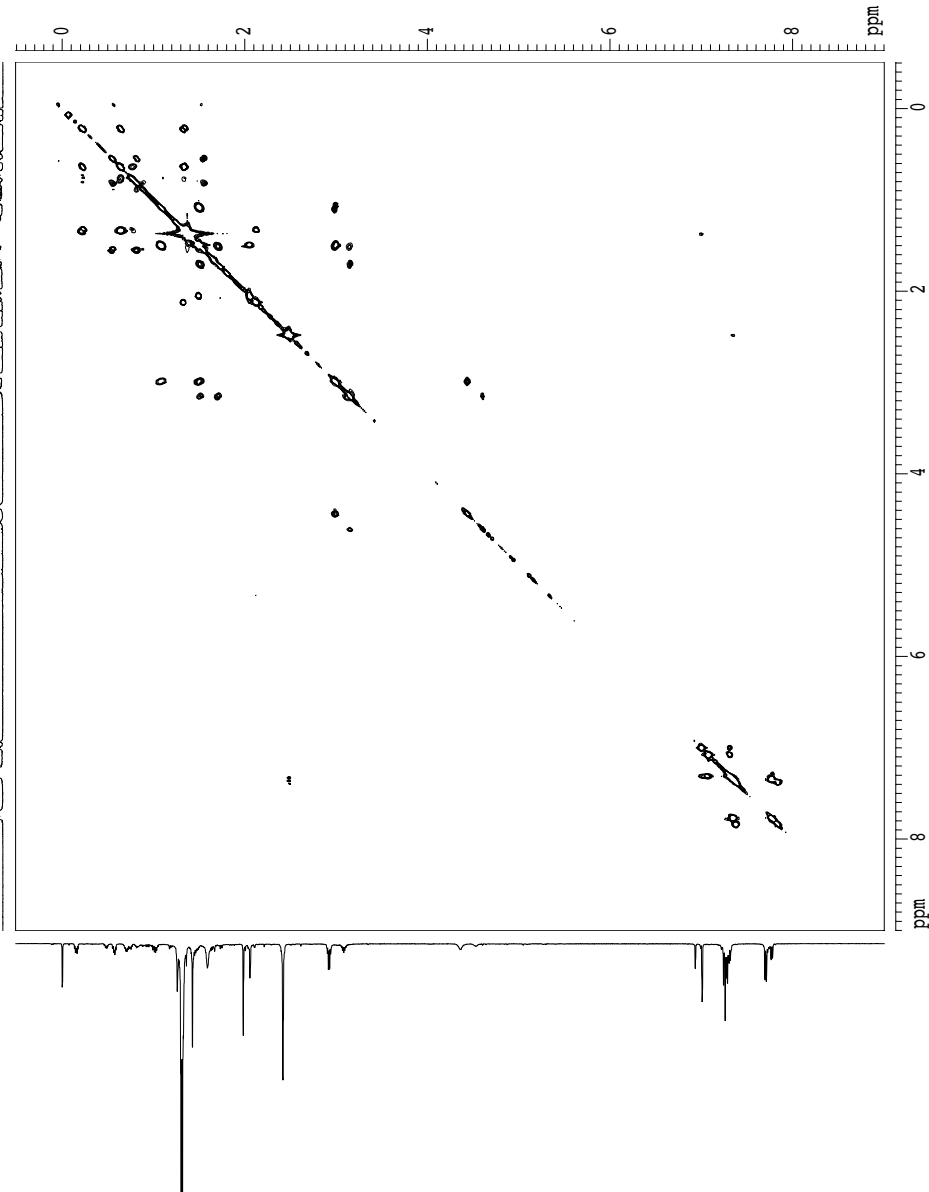
SI 1024  
 SF 500.2200000 MHz  
 SFB 0  
 SB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters

SI 1024  
 SF 500.2200000 MHz  
 SFB 0  
 SB 0.00 Hz  
 LB 0  
 GB 0

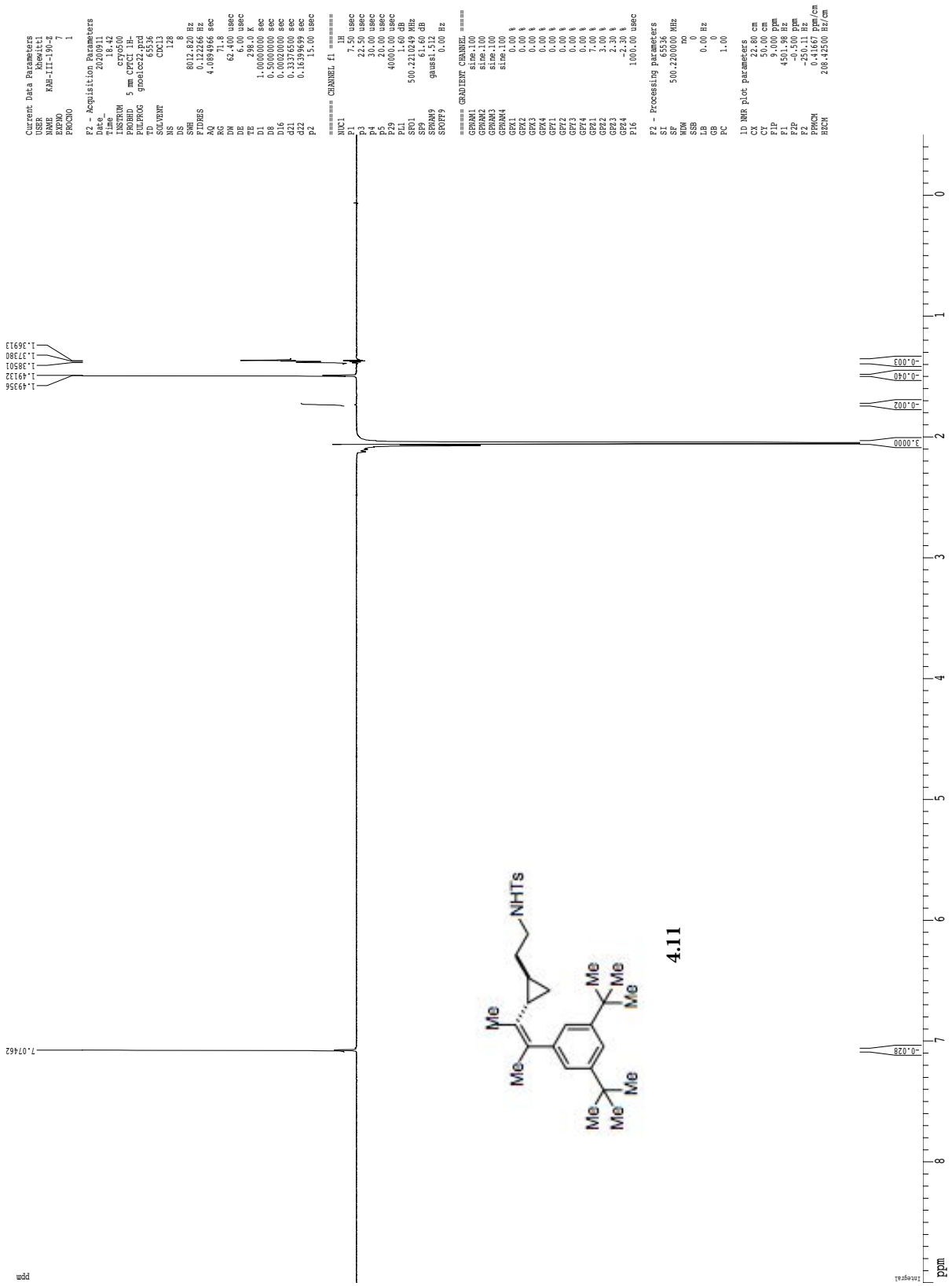
2D NMR plot parameters

CX1 15.00 cm  
 F2PL0 5.002 ppm  
 F2PL1 10.509 ppm  
 F2PH -254.47 Hz  
 F1PL0 9.002 ppm  
 F1PL1 4503.14 Hz  
 F1PH -21.509 ppm  
 F2PRCM 0.63407 ppm/cm  
 F1PRCM 317.17416 Hz/cm  
 F2BCH 0.63407 ppm/cm  
 F1BCH 317.17416 Hz/cm

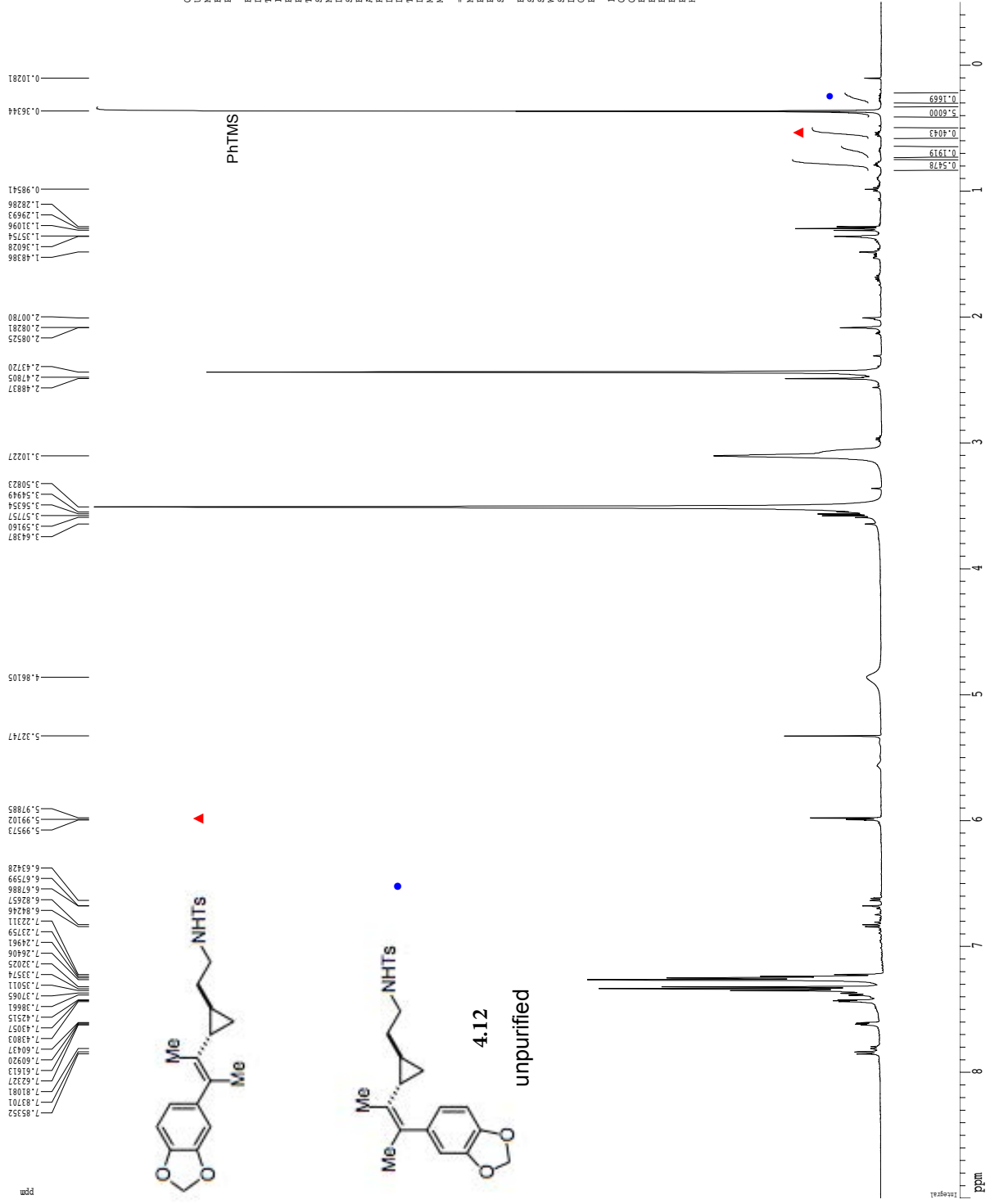




gnoe

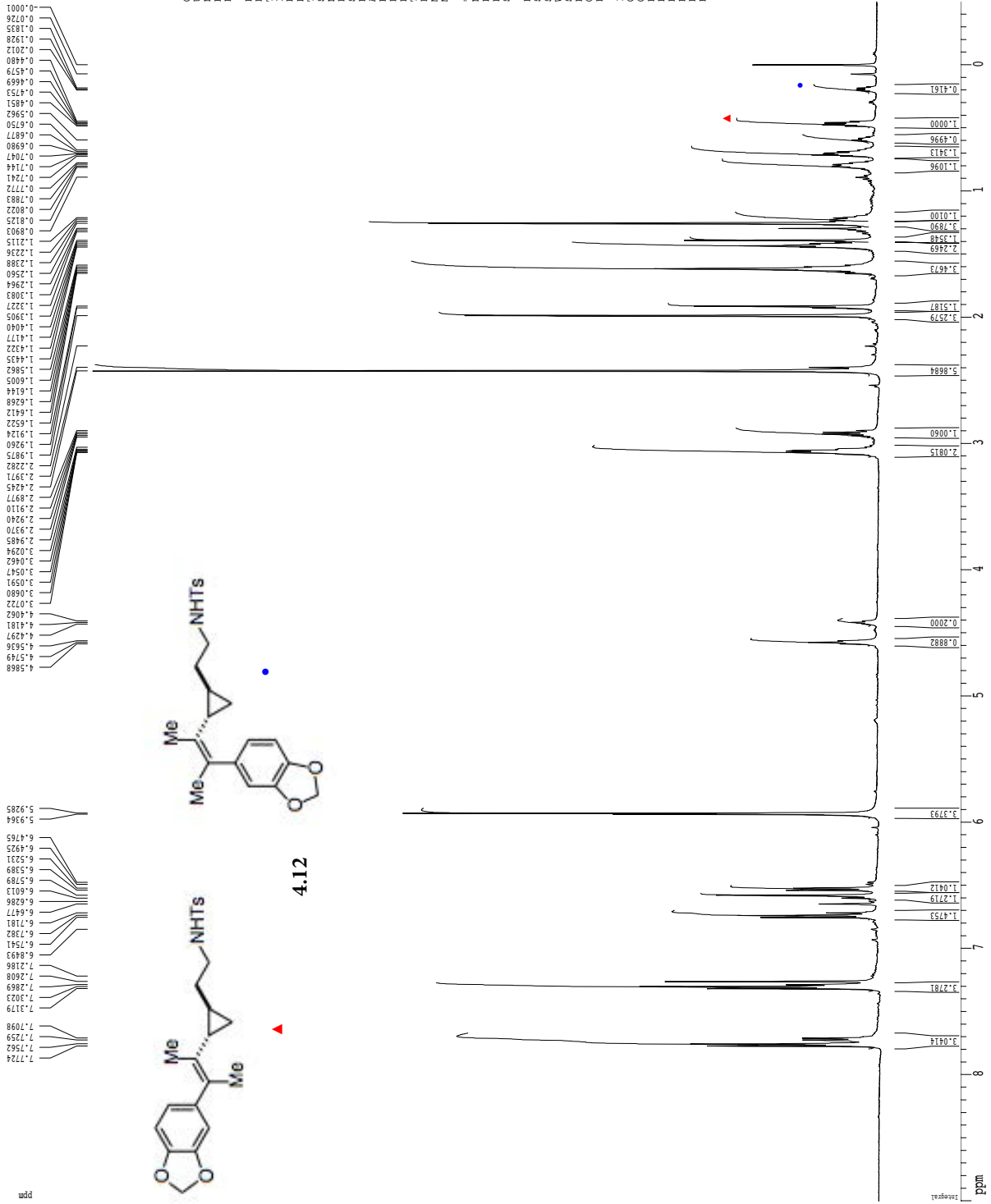


1H spectrum



Current Data Parameters  
 USER Kavit  
 NAME KAH-II-191-crude  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 2007016  
 Time 17:56  
 CHANNELNAME  
 PROBHD 5 mm CPCL H  
 PULPROG zg30  
 TD 48074  
 SFO 500.136000 MHz  
 NUC1 1H  
 NS 6  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.469677 Hz  
 AQ 2.593877 sec  
 RG 312  
 DW 62.400 usec  
 DE 6.00 usec  
 DI 0.1000000 sec  
 MCOREST 0.0000000 sec  
 MCHRG 0.0150000 sec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.50 usec  
 PL 0.00 dB  
 SFO1 500.235015 MHz  
 F2 - Processing parameters  
 SI 6554  
 SF 500.235000 MHz  
 NW 0  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00  
 ID NMR Plot parameters  
 CX 0  
 CY 22.80 cm  
 FIP 9.000 ppm  
 F1 4801.98 Hz  
 F2 -0.500 ppm  
 F3 0.000 ppm  
 FWHM 0.41667 ppm/cm  
 RESZM 208.42500 Hz/cm

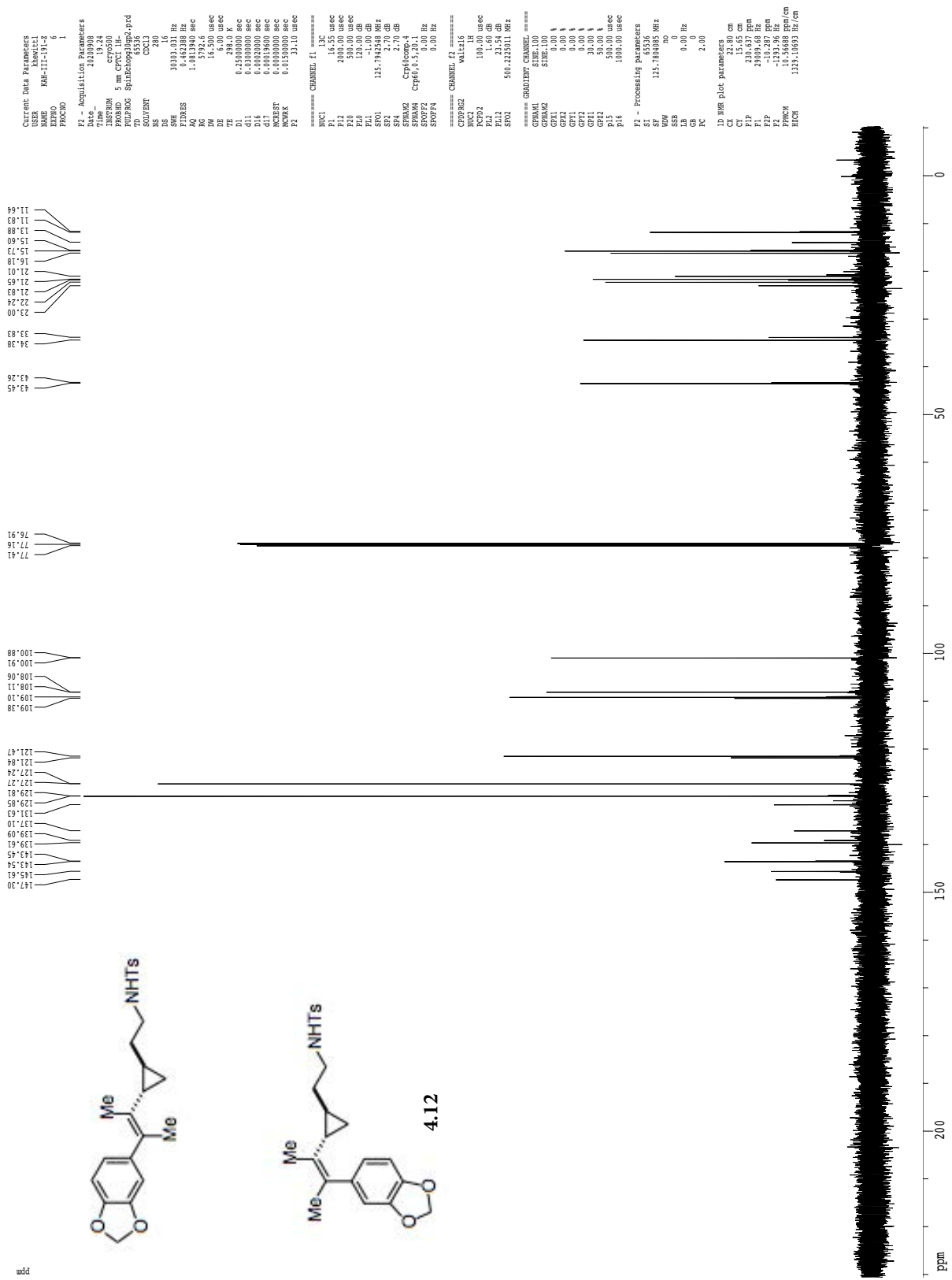
1H spectrum

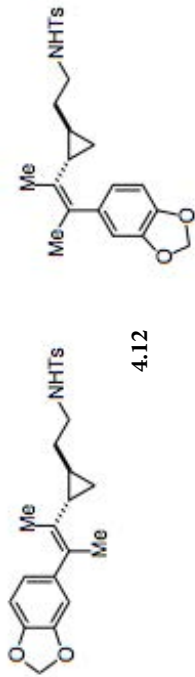


```

Current Data Parameters
=====
NAME      K01-III-191-2
EXPNO    2
PROCNO   1
F2 - Acquisition Parameters
=====
Date_      20200908
Time      18:40
INSTRUM   spect
PROBHD    5 mm CPY 13
PULPROG   zgpg
TD         48674
SOLVENT   CDCl3
AQ         0.2
RG         2
DS         2
SFR        8012.420 Hz
FIDRES    0.16647 Hz
AQ         2.999677 sec
DE         62.400 uSsec
TE         298.0 K
NUC1       13
NUC2       13
ACQRES    0.1000000 sec
SFO1       500.1300000 MHz
===== CHANNEL f1 =====
NUC1       13
P1         7.50 uSsec
PL1        1.60 dB
SFO1       500.2235015 MHz
F2 - Processing parameters
=====
SI         65536
SF         500.220013 MHz
WDW        no
SSB        0
GB         0
PC         1.00
ID         1.00
===== LD parameters =====
CX         2280 cm
CY         15.000 cm
FLP        9.000 ppm
FL         450.138 Hz
F2         250.069 Hz
F3         -250.11 Hz
PPHMCN    0.41667 ppm/cm
HZCN      208.42602 Hz/cm
    
```

Z-restored spin-echo 13C spectrum with 1H decoupling





gcosy60

4.12

```

Current Data Parameters
USER      Khevit11
NAME      KM-11-191-2
EXPNO     3
PROCNO    1

F2 - Acquisition Parameters
Date_     20200908
Time      18.42
INSTRUM   cryo500
PROBHD    5 mm cryo1H
PULPROG   zgpg30
TD        2048
SOLVENT   CDCl3
NS         1
DS         16
AQ         8013.85 Hz
FIDRES    3.912510 Hz
AQ         0.1278452 sec
RG         181
DM         62.400 usec
DE         298.0 usec
TE         298.0 usec
d0         0.0000300 sec
d1         1.0000000 sec
d13        0.0000300 sec
d16        0.0002000 sec
d10        0.0001400 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
PC         0.00 usec
SFO1       500.225015 MHz

===== GRADIENT CHANNEL =====
GPRM1     sine,100
GPRM2     sine,100
GPRM3     sine,100
GP2        0.00 %
GP1        0.00 %
GP2        0.00 %
GP1        17.00 %
GP2        17.00 %
P16        1000.00 usec

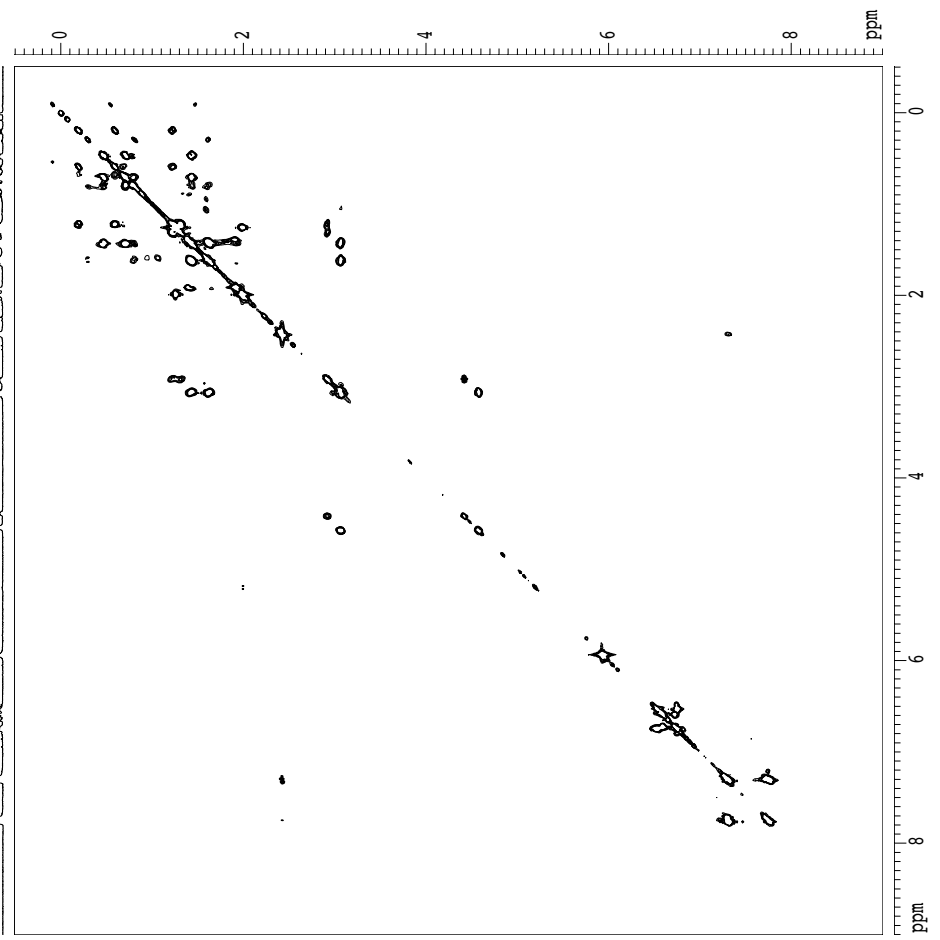
F1 - Acquisition parameters
NUC1       1H
P1         7.50 usec
PC         0.00 usec
SFO1       500.225015 MHz

===== GRADIENT CHANNEL =====
GPRM1     sine,100
GPRM2     sine,100
GPRM3     sine,100
GP2        0.00 %
GP1        0.00 %
GP2        0.00 %
GP1        17.00 %
GP2        17.00 %
P16        1000.00 usec

F2 - Processing parameters
SI         1024
SF         500.220313 MHz
WDW        0
SSB        0
GB         0.00 Hz
PC         1.00

F1 - Processing parameters
SI         1024
SF         500.220313 MHz
WDW        0
SSB        0
GB         0.00 Hz
PC         1.00

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2FLO      5.002 ppm
F2FID      450.509 Hz
F2F1F2     -50.509 Hz
F2H1       -254.44 Hz
F1FLO      5.002 ppm
F1FID      4503.17 Hz
F1F1F2     -0.509 ppm
F1F1F1     -2.000 ppm
F2PRGM     0.63407 ppm/cm
F2PRGMCM  317.17413 Hz/cm
F1PRGM     0.63407 ppm/cm
F1PRGMCM  317.17413 Hz/cm
  
```



gnoe

ppm

2.4396  
2.4290  
2.4258  
2.4207  
2.4181  
2.4012  
2.4013  
2.3955  
2.3912  
2.2742  
2.2728  
2.2744  
2.1829  
2.1793  
2.1759

6.0098  
6.0100  
6.0120

7.7710  
7.7553

Current Data Parameters  
 USER khewitt1  
 NAME KAH-LII-191-SM-Z  
 EXPNO 4  
 PROCNO 1

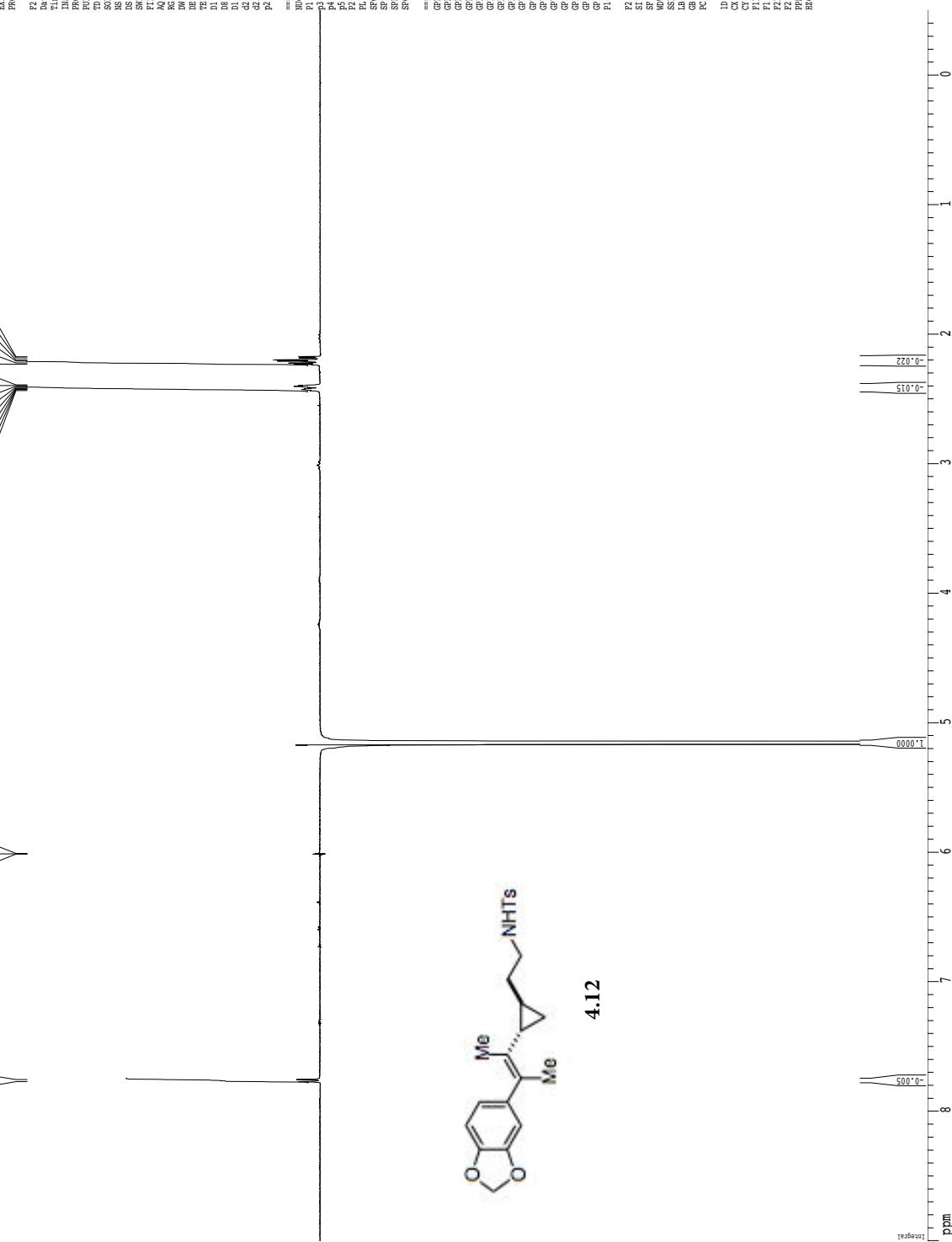
F2 - Acquisition Parameters  
 Date\_ 20200711  
 Time 17:19  
 INSTRUM spect  
 PROBHD 5 mm CPYCI 1H  
 PULPROG gnoe22.prd  
 TD 65536  
 SFO100 499.813  
 AQ 1.41  
 RG 327.5  
 NS 128  
 DS 8  
 SWH 8012.822 Hz  
 FIDRES 0.122266 Hz  
 AQ 4.10999 sec  
 RG 30.5  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 300.2 K  
 D1 1.00200000 sec  
 D6 0.50000000 sec  
 D16 0.00200000 sec  
 d21 0.33376500 sec  
 d22 0.18398900 sec  
 P2 15.00 usec

==== CHANNEL f1 =====  
 NUC1 1H  
 P1 1.20000000 usec  
 PL1 0.00 dB  
 P2 22.50 usec  
 PL2 0.00 dB  
 P3 30.00 usec  
 PL3 0.00 dB  
 P4 30.00 usec  
 PL4 0.00 dB  
 P5 20.00 usec  
 PL5 0.00 dB  
 P6 40000.00 usec  
 PL6 0.00 dB  
 SFO1 500.225288 MHz  
 SF99 61.46 dB  
 SP0A99 9  
 SP0P99 1512  
 SC0P99 0.00 Hz

==== GRADIENT CHANNEL =====  
 G0A0A1 size:100  
 G0B0A2 size:100  
 G0B0A3 size:100  
 G0B0A4 size:6.00  
 G0X1 0.00  
 G0X2 0.00  
 G0X3 0.00  
 G0Y1 0.00  
 G0Y2 0.00  
 G0Y3 0.00  
 G0Y4 0.00  
 G0Z1 0.00  
 G0Z2 2.30  
 G0Z3 2.30  
 G0Z4 -2.30  
 P16 1000.00 usec

F2 - Processing Parameters  
 SI 65536  
 SF 500.220000 MHz  
 INSTRUM spect  
 NS 128  
 DS 8  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

ID NMR file parameters  
 CX 22.88 cm  
 CY 56.00 cm  
 FIP 9.000 ppm  
 F2P 4.000 ppm  
 F2 -250.11 Hz  
 F2 0.4166 ppm/cm  
 RECIN 200.4250 Hz/cm



Integral

ppm



gnoe

ppm

3.03786  
3.03278  
3.0297  
3.00780  
2.98781  
2.98286  
2.43462  
2.42973  
2.42518  
2.40906  
2.39255  
2.39157  
2.30257  
2.29780  
2.29318  
2.27627  
2.27166  
2.26785  
2.22967  
2.22505  
2.20878  
2.19952  
2.18244  
2.17369  
2.05946  
2.02992  
2.01376  
2.00448  
1.98888  
1.97829

5.15254  
5.15980

6.01015  
6.01156  
6.01294  
6.01437  
6.01547  
6.38383  
6.38529  
6.59566

7.75354

Current Data Parameters  
USER khewitt1  
NAME KAH-111-191-SH-2  
EXPNO 5  
PROCNO 1

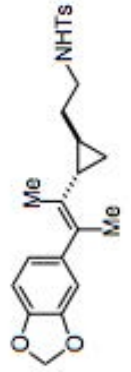
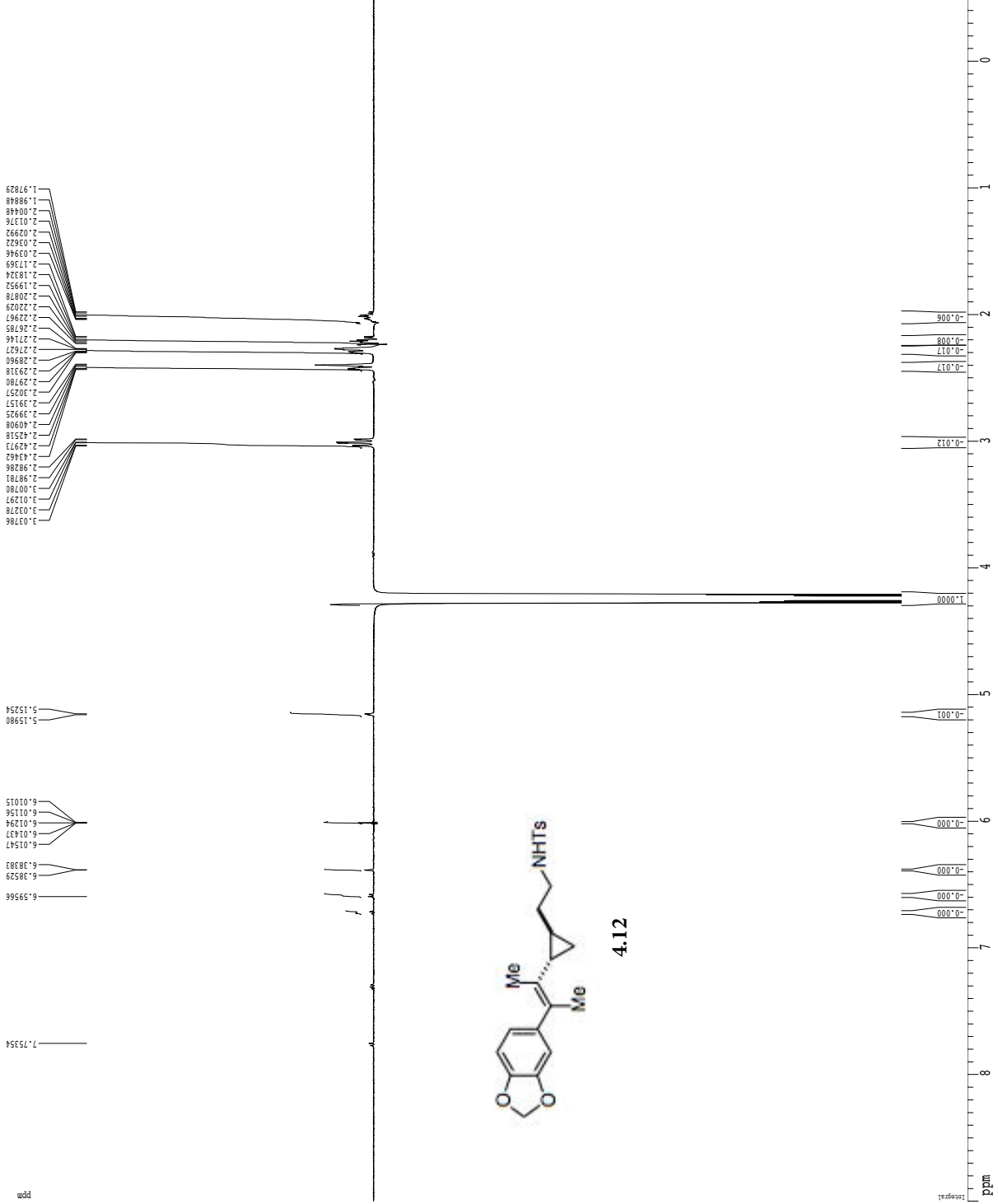
F2 - Acquisition Parameters  
Date\_ 20200711  
Time 17:53:13  
INSTRUM spect  
PROBHD 5 mm CPYCI 1H  
PULPROG gnoe.ces22.prd  
TD 65536  
AQ 0.02000000 sec  
RG 327.5  
NS 121  
DS 8  
SWH 8012.822 Hz  
FIDRES 0.122666 Hz  
AQ 4.10000000 sec  
RG 327.5  
WDW 62.400 usec  
DE 6.00 usec  
TE 300.2 K  
D1 1.00000000 sec  
D2 0.50000000 sec  
D16 0.00020000 sec  
dZ1 0.33376500 sec  
dZ2 0.18398600 sec  
PC 15.00 usec

===== CHANNEL f1 =====  
NUC1 1H  
P1 1.00 usec  
PL1 0.00 dB  
PC2 30.00 usec  
PC4 30.00 usec  
PC5 20.00 usec  
PC9 40000.00 usec  
PC10 40000.00 usec  
SFO1 500.2221214 MHz  
SFO9 61.66 dB  
SFO99 9.512  
SFO999 0.00 Hz

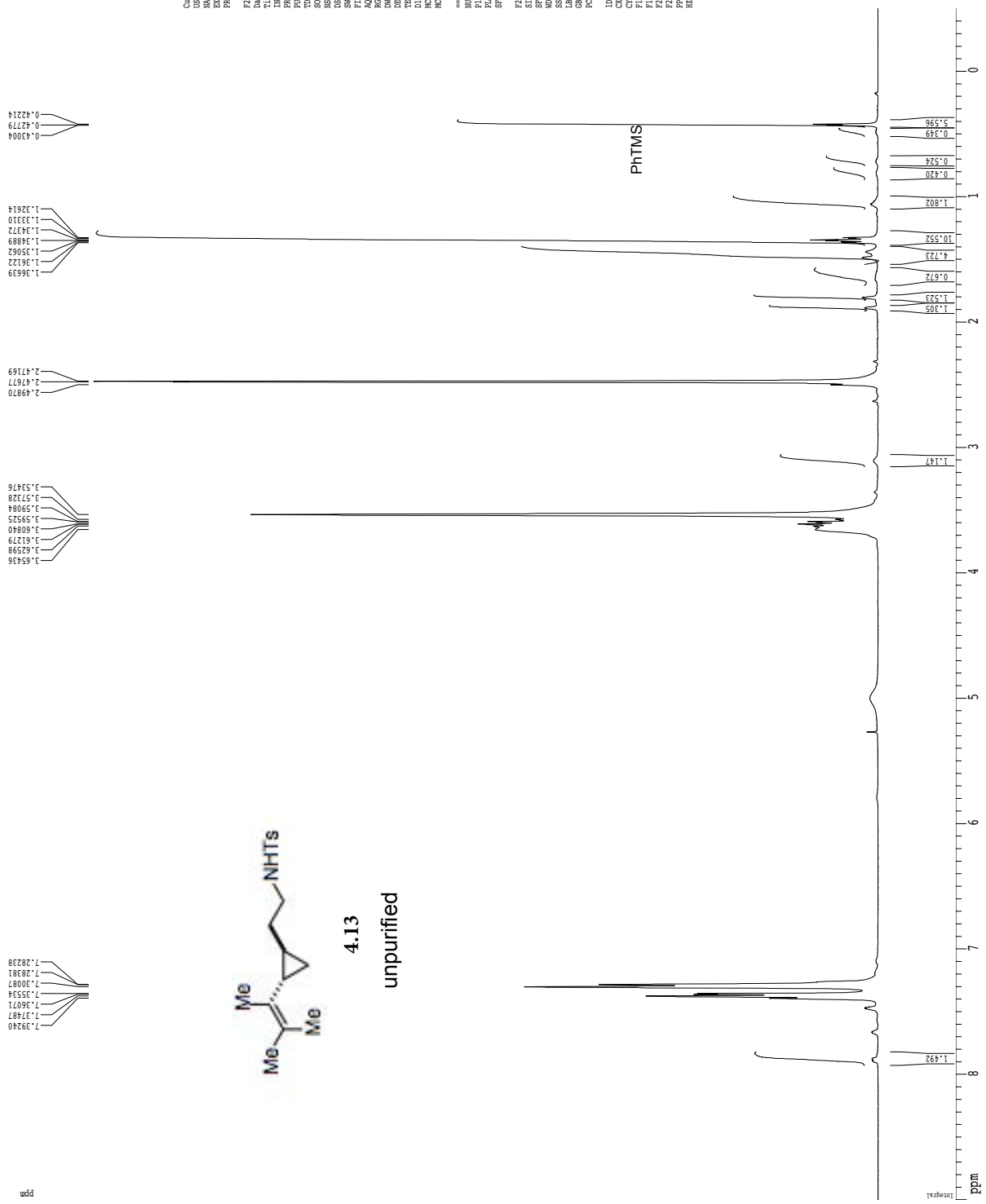
===== GRADIENT CHANNEL =====  
GRDM1 sine.100  
GRDM2 sine.100  
GRDM3 sine.100  
GRDM4 sine.100  
GX1 0.00 G  
GX2 0.00 G  
GX3 0.00 G  
GX4 0.00 G  
GX5 0.00 G  
GX6 0.00 G  
GX7 0.00 G  
GX8 0.00 G  
GX9 0.00 G  
GX10 0.00 G  
GX11 0.00 G  
GX12 0.00 G  
GX13 0.00 G  
GX14 0.00 G  
GX15 0.00 G  
GX16 0.00 G  
GX17 0.00 G  
GX18 0.00 G  
GX19 0.00 G  
GX20 0.00 G  
GX21 0.00 G  
GX22 2.38 G  
GX23 2.38 G  
GX24 -2.38 G  
P16 1000.00 usec

F2 - Processing parameters  
SI 65536  
SF 500.2200000 MHz  
WDW no  
SSB no  
LB 0.00 Hz  
GB 0  
PC 1.00

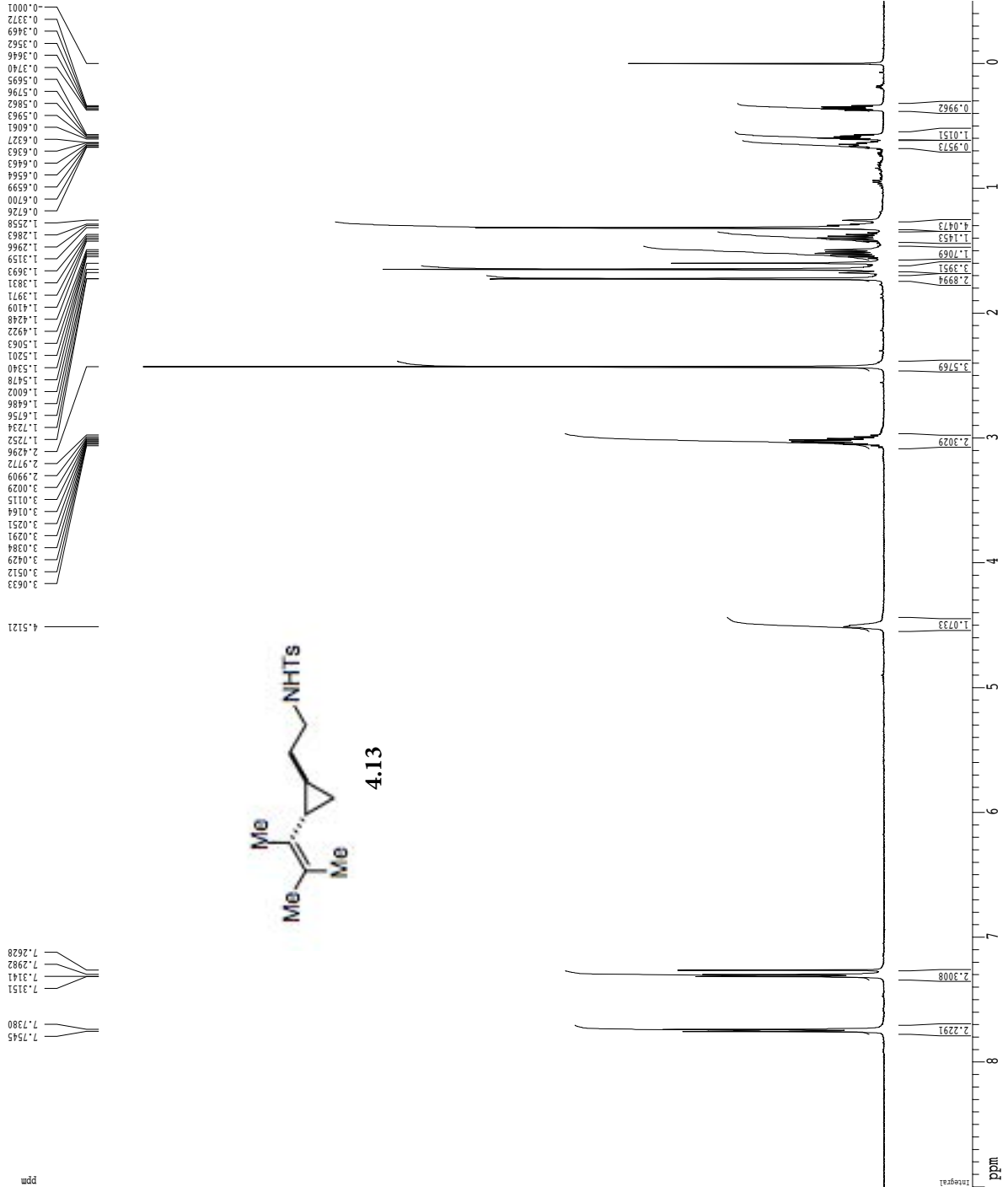
ID MR parameters  
CX 22.88 cm  
CY 50.00 cm  
FIP 9.000 ppm  
Z 4.000 ppm  
E2P -250.11 Hz  
F2 0.41667 ppm/cm  
REC1 208.42500 Hz/cm



1H spectrum



1H spectrum



Current Data Parameters

USER khe Wittl  
NAME KAH-III-239-COSY  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters

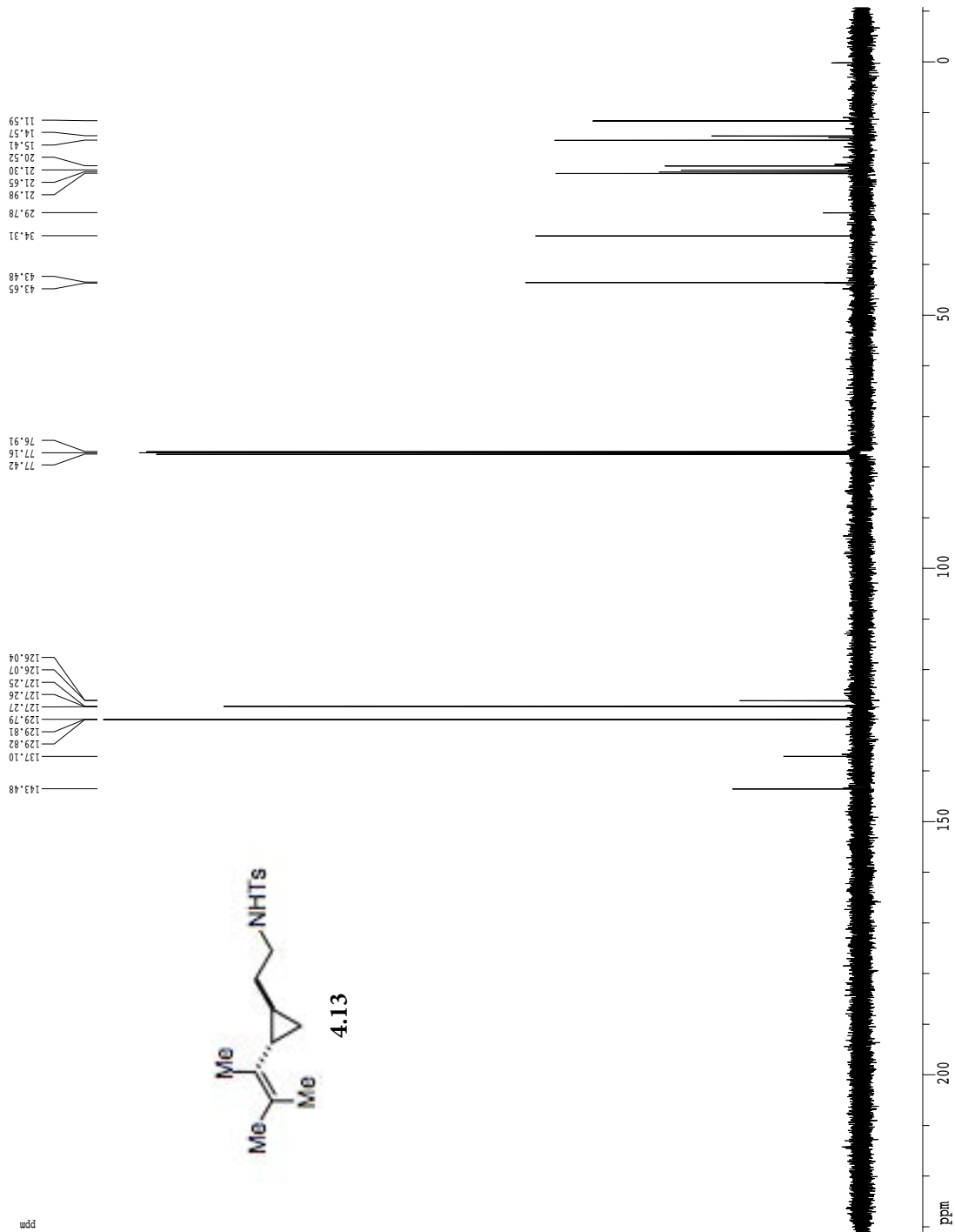
Date\_ 20200821  
Time 11.57  
INSTRUM gn500  
PROBHD 5 mm broadband  
PULPROG zg30  
TD 48074  
SOLVENT CDCl3T  
NS 8  
DS 2  
SWH 8012.820 Hz  
FIDRES 0.166677 Hz  
AQ 2.9998677 sec  
RG 812.7  
DW 62.400 usec  
DE 6.00 usec  
TE 298.0 K  
D1 0.1000000 sec  
MCREST 0.0000000 sec  
MCWRK 0.0150000 sec

==== CHANNEL f1 =====  
NUC1 1H  
P1 12.00 usec  
PL1 -6.00 dB  
SFO1 498.7534913 MHz

F2 - Processing parameters  
SI 65536  
SF 498.7500299 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
CY 12.50 cm  
FILP 9.000 ppm  
F1 4488.75 Hz  
F2 -0.500 ppm  
F2P -249.38 Hz  
PPMCM 0.47500 ppm/cm  
HZCM 236.90627 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters  
 USER Khewitt1  
 NAME KAH-III-239-13C  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20200819  
 Time\_ 11:41  
 INSTRUM gm500  
 PROBHD 5 mm broadband  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3T  
 NS 632  
 DS 4  
 SWH 30303.031 Hz  
 FIDRES 0.462388 Hz  
 AQ 1.0813940 sec  
 RG 5792.6  
 DW 16.500 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.25000000 sec  
 d11 0.03000000 sec  
 MCREST 0.00000000 sec  
 MCWRRK 0.01500000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 14.20 usec  
 PL1 -6.00 dB  
 SF01 125.4245824 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 -6.00 dB  
 PL12 12.30 dB  
 SF02 498.7524937 MHz

F2 - Processing parameters  
 SI 65536  
 SF 125.4107757 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 2.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 12.50 cm  
 F1P 230.907 ppm  
 F1 28958.26 Hz  
 F2P -10.723 ppm  
 F2 -1344.77 Hz  
 PPMCM 12.08151 ppm/cm  
 HZCM 1515.15149 Hz/cm



4.13

gc05y60

Current Data Parameters  
 USER: JG  
 NAME: RM-111-234-C05Y  
 EXPNO: 2  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20200421  
 Time: 15:00  
 INSTRUM: spect  
 PROBRD: 5 mm broadband  
 PULPROG: cosygp6-prd  
 SOLVENT: CDCl3  
 NS: 1  
 DS: 4  
 SWH: 8012.830 Hz  
 FIDRES: 3.912510 Hz  
 AQ: 0.1278452 sec  
 RG: 327.5  
 INCR: 1.0000000  
 DE: 6.00 usec  
 TE: 0.000298.0 K  
 D1: 1.00000000 sec  
 d11: 0.00003000 sec  
 d12: 0.00003000 sec  
 d13: 0.00003000 sec  
 d14: 0.00003000 sec  
 d15: 0.00003000 sec  
 d16: 0.00003000 sec  
 d17: 0.00003000 sec  
 d18: 0.00003000 sec  
 d19: 0.00003000 sec  
 d20: 0.00003000 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NU1: 12.00 usec  
 PL1: -6.00 dB  
 SFO1: 498.754513 MHz

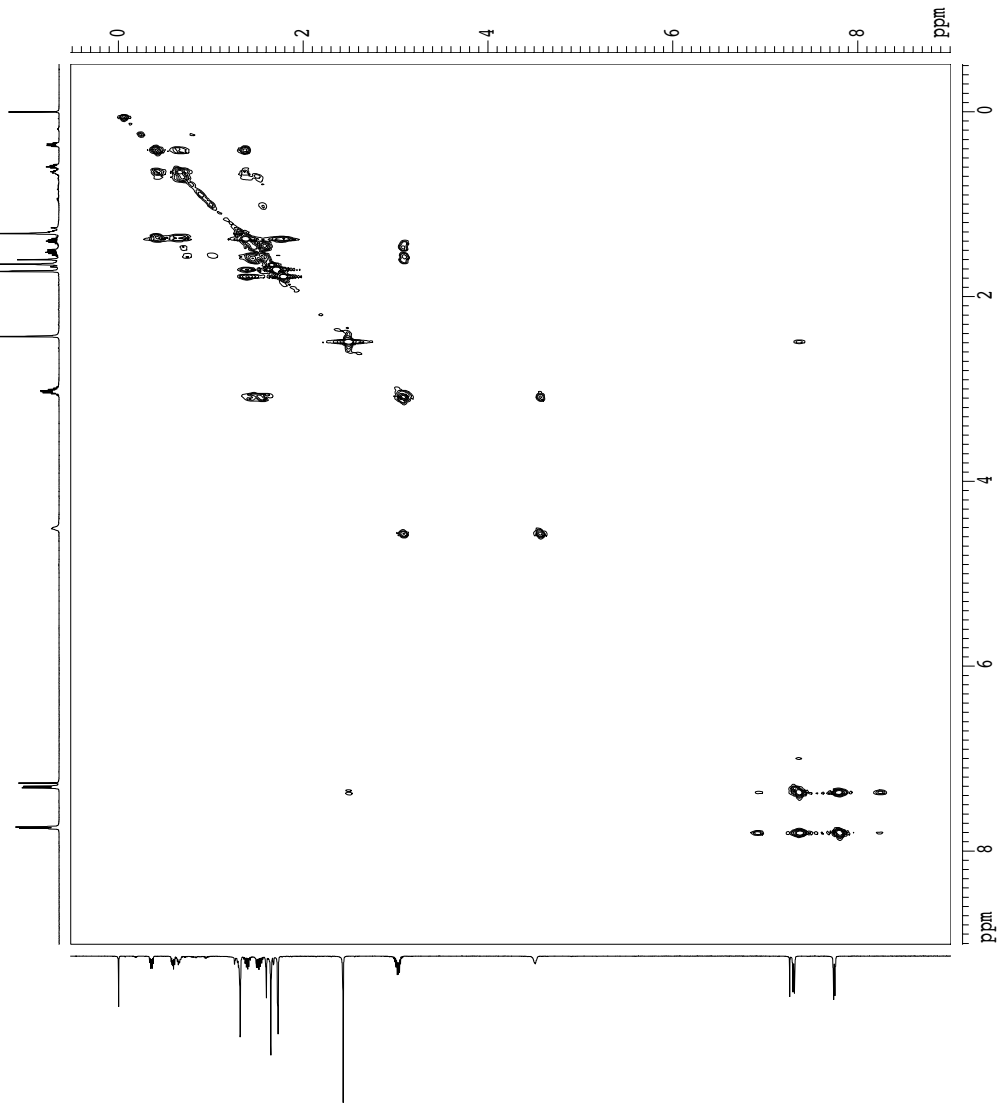
\*\*\*\*\* GRABUNT CHANNEL \*\*\*\*\*  
 GRABM1: SINE.100  
 GRABP1: 0.00 usec  
 GRABR1: 0.00 Hz  
 GRABF1: 0.00 Hz  
 GRABD1: 0.00 Hz  
 GRABE1: 17.00 Hz  
 GRABZ1: 0.00 Hz  
 GRABW1: 0.00 Hz  
 GRABX1: 0.00 Hz  
 GRABY1: 0.00 Hz  
 GRABV1: 0.00 Hz  
 GRABU1: 0.00 Hz  
 GRABT1: 0.00 Hz  
 GRABQ1: 0.00 Hz  
 GRABO1: 0.00 Hz  
 GRABN1: 0.00 Hz  
 GRABM1: 0.00 Hz  
 GRABL1: 0.00 Hz  
 GRABK1: 0.00 Hz  
 GRABJ1: 0.00 Hz  
 GRABI1: 0.00 Hz  
 GRABH1: 0.00 Hz  
 GRABG1: 0.00 Hz  
 GRABF1: 0.00 Hz  
 GRABE1: 0.00 Hz  
 GRABD1: 0.00 Hz  
 GRABC1: 0.00 Hz  
 GRABB1: 0.00 Hz  
 GRABA1: 0.00 Hz  
 GRAB91: 0.00 Hz  
 GRAB81: 0.00 Hz  
 GRAB71: 0.00 Hz  
 GRAB61: 0.00 Hz  
 GRAB51: 0.00 Hz  
 GRAB41: 0.00 Hz  
 GRAB31: 0.00 Hz  
 GRAB21: 0.00 Hz  
 GRAB11: 0.00 Hz

F1 - Acquisition parameters  
 INU: 256  
 SFO1: 498.7535 MHz  
 FIDRES: 31.380079 Hz  
 WDW: EM  
 SSB: 0  
 GB: 0  
 PC: 1.00

F2 - Processing parameters  
 SI: 498.750000 MHz  
 SF: 498.750000 MHz  
 WDW: EM  
 SSB: 0  
 GB: 0  
 PC: 1.00

F1 - Processing parameters  
 SI: 1024  
 SF: 498.750000 MHz  
 WDW: EM  
 SSB: 0  
 GB: 0

2D NMR plot parameters  
 CK1: 15.00 cm  
 CK2: 15.00 cm  
 FZLO: 8.000 ppm  
 FZHI: 15.515 ppm  
 FZBL: -256.44 Hz  
 FZBO: 0.000 ppm  
 FZHL: -46.515 ppm  
 FZHM: 256.44 Hz  
 FZLN: 316.6537 ppm/cm  
 FZLON: 0.63489 ppm/cm  
 FZLOR: 316.6537 Hz/cm



gnoe

PM

Current Data Parameters  
USER Khevitil  
NAME KAH-III-239-NOE  
EXPTNO 2  
PROCNO 1

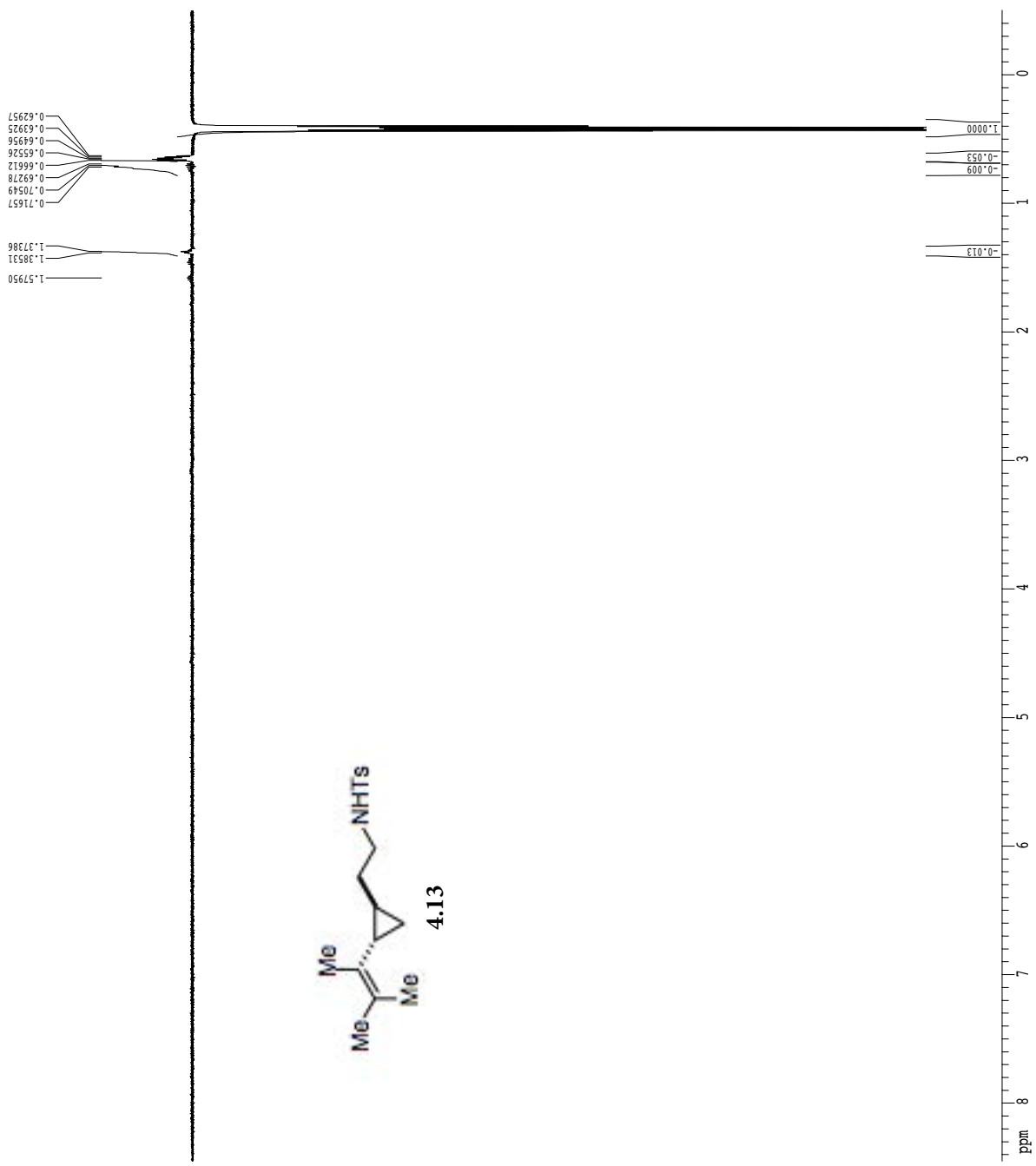
F2 - Acquisition Parameters  
Date\_ 20/01/21  
Time 14:46  
INSTRUM spect  
PROBHD 5 mm broadband  
PULPROG gnoelcs22.prd  
TD 65536  
SOLVENT CDCl3  
NS 128  
DS 8  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894966 sec  
RG 5792.6  
DM 62.400 usec  
DE 6.00 usec  
TE 298.0 K  
D1 1.00000000 sec  
D8 0.50000000 sec  
D16 0.00020000 sec  
d21 0.33374399 sec  
d22 0.16399699 sec  
p2 24.00 usec

==== CHANNEL f1 =====  
NUC1 <sup>1</sup>H  
P1 12.00 usec  
P3 36.00 usec  
P4 48.00 usec  
P5 32.00 usec  
F29 40000.00 usec  
PL1 -6.00 dB  
SFO1 498.7502076 MHz  
SFO2 99.7504153 MHz  
SFO99 gauss1.513  
SFOF99 0.00 Hz

==== GRADIENT CHANNEL =====  
GPM1 sine,100  
GPM2 sine,100  
GPM3 sine,100  
GPM4 sine,100  
GPX1 0.00 \$  
GPX2 0.00 \$  
GPX3 0.00 \$  
GPX4 0.00 \$  
GPI1 0.00 \$  
GPI2 0.00 \$  
GPI3 0.00 \$  
GPI4 0.00 \$  
GPI5 0.00 \$  
GPI6 0.00 \$  
GPI7 0.00 \$  
GPI8 0.00 \$  
GPI9 0.00 \$  
GPI10 0.00 \$  
GPI11 0.00 \$  
GPI12 0.00 \$  
GPI13 0.00 \$  
GPI14 0.00 \$  
GPI15 0.00 \$  
GPI16 0.00 \$  
P16 1000.00 usec

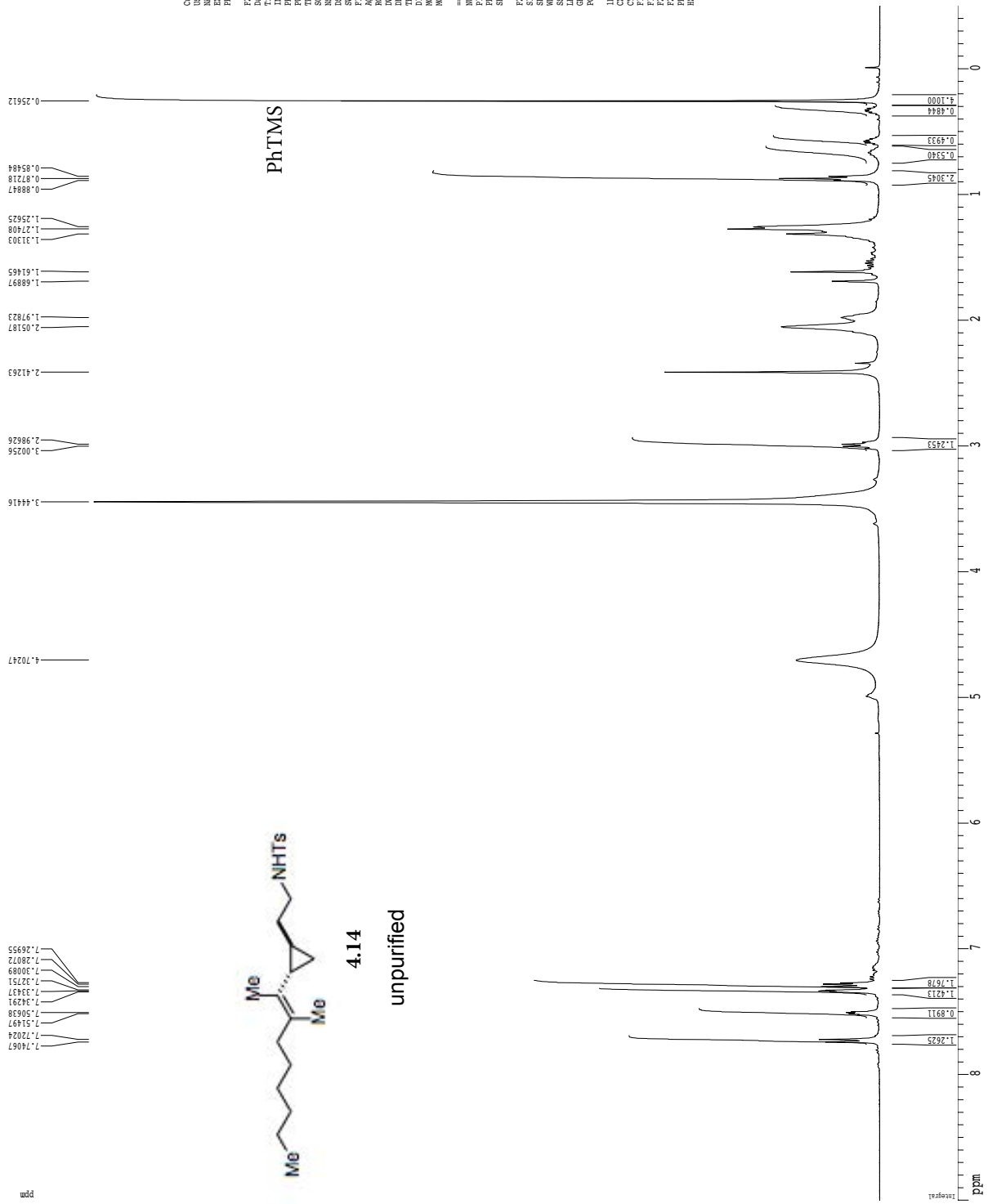
F2 - Processing parameters  
S1 65536  
SF 498.7500000 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

ID, NMR Plot parameters  
CX 20.00 cm  
CY 12.50 cm  
F1P 9.000 ppm  
F1 4488.75 Hz  
F2P -0.500 ppm  
F2 -249.38 Hz  
PPMCH 0.47500 ppm/cm  
HZCM 236.90625 Hz/cm



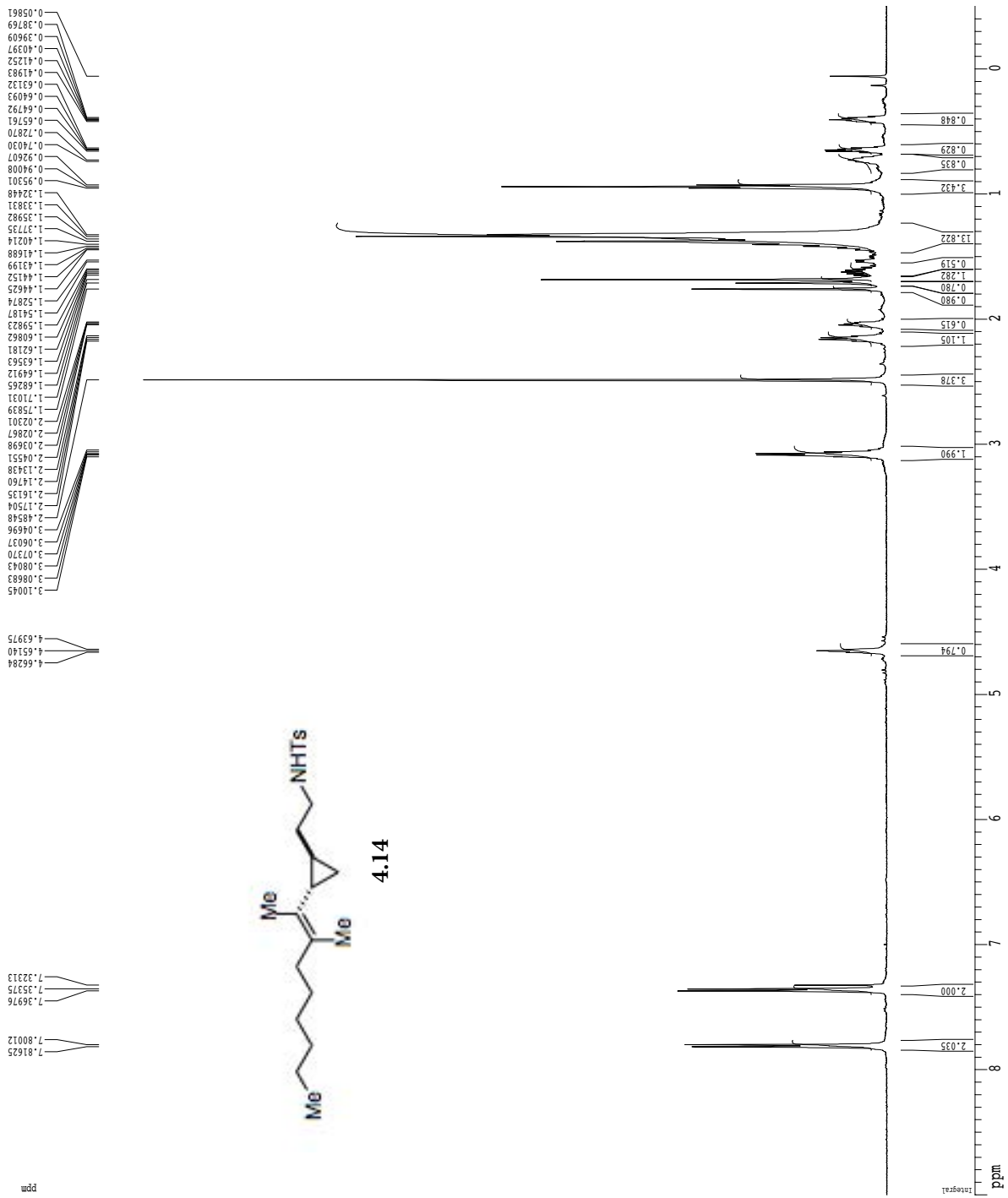
Integral

1H spectrum



Current Data Parameters  
 USER K. Bennett  
 NAME R01-III-216-crude  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20200724  
 Time 14:46  
 PROBN 5 nm QNP HXFP  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6410.256 Hz  
 FIDRES 0.464973 Hz  
 AQ 2.399977 sec  
 RG 64  
 DM 78.000 us/cyc  
 DE 4.50 us/cyc  
 DL 0.1000000 sec  
 MCHEST 0.0000000 sec  
 MCWRR 0.0150000 sec  
 ===== CHANNEL f1 =====  
 NUCL 1H  
 P1 12.00 us/cyc  
 PL1 4.10 dB  
 SFO1 400.1326009 MHz  
 F2 - Processing parameters  
 SI 65526  
 SF 400.1300170 MHz  
 WDW 0  
 SSB 0  
 LB 0.00 Hz  
 GB 2.00  
 PC 2.00  
 ID NMR plot Parameters  
 CX 12.00 cm  
 CY 12.00 cm  
 C1P 9.000 ppm  
 F1 360.117 Hz  
 F2P -0.500 ppm  
 F3P 0.000 ppm  
 FWHM 0.41667 ppm/cm  
 HZCM 166.72084 Hz/cm

1H spectrum



Current Data Parameters  
 USER khewitt1  
 NAME KAH-III-206-Z  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20200911  
 Time 13.40  
 INSTRUM gn500  
 PROBHD 5 mm broadband  
 PULPROG zg30  
 TD 48074  
 SOLVENT CDCl3T  
 NS 8  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.9998677 sec  
 RG 114  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.4 K  
 D1 0.10000000 sec  
 MCREST 0.00000000 sec  
 MCWRK 0.01500000 sec

==== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -6.00 dB  
 SF01 498.7534913 MHz

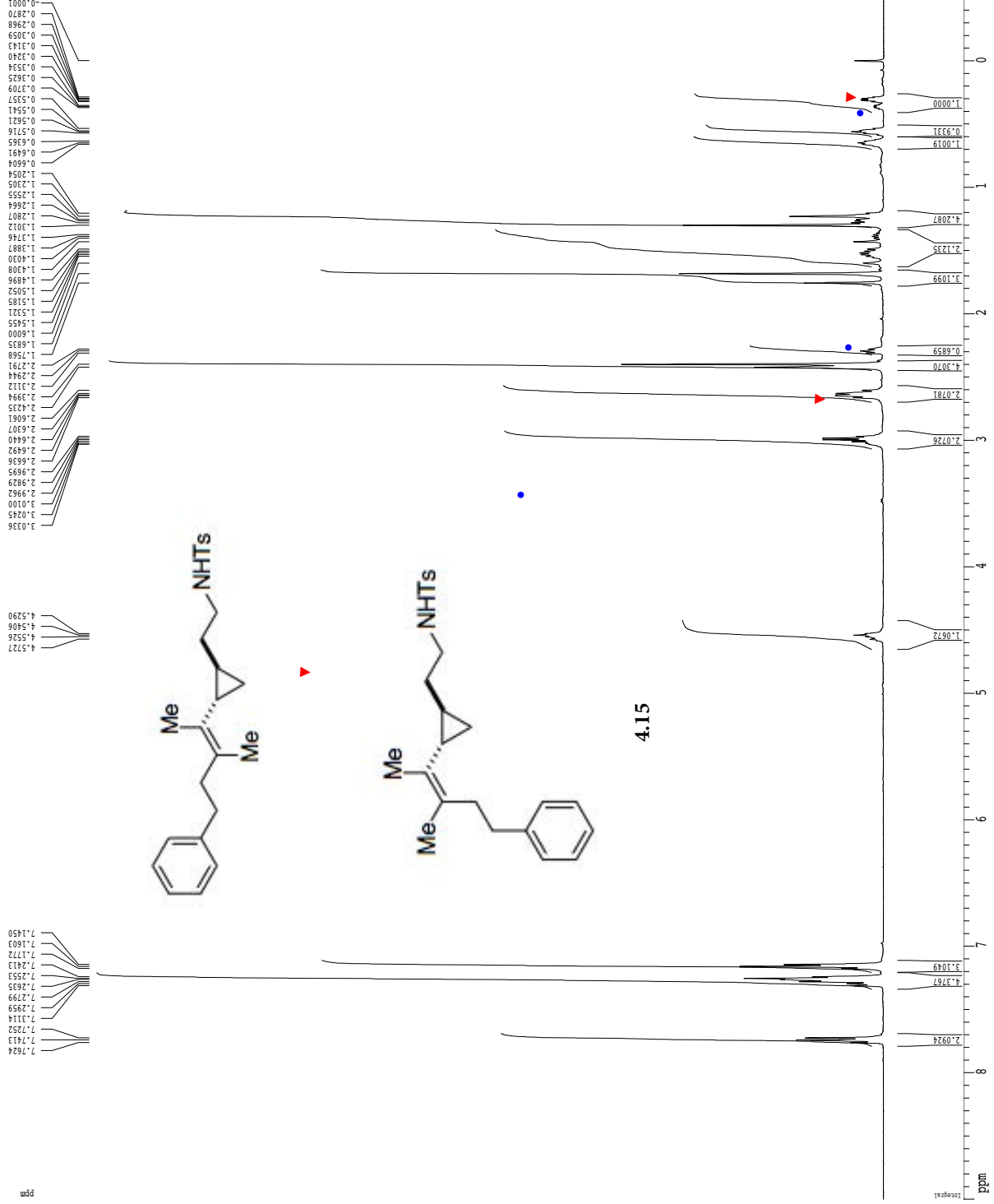
F2 - Processing parameters  
 SI 65536  
 SF 498.7500000 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

ID NMR plot parameters  
 CX 20.00 cm  
 CY 12.50 cm  
 F1P 9.000 ppm  
 F1 4488.75 Hz  
 F2P -0.500 ppm  
 F2 -249.38 Hz  
 PPMCM 0.47500 ppm/cm  
 HZCM 236.90625 Hz/cm





1H spectrum



Current Data Parameters  
 USER TWTW/20carbon  
 NAME TWTW/20carbon  
 EXPO 1  
 PROCNO 1

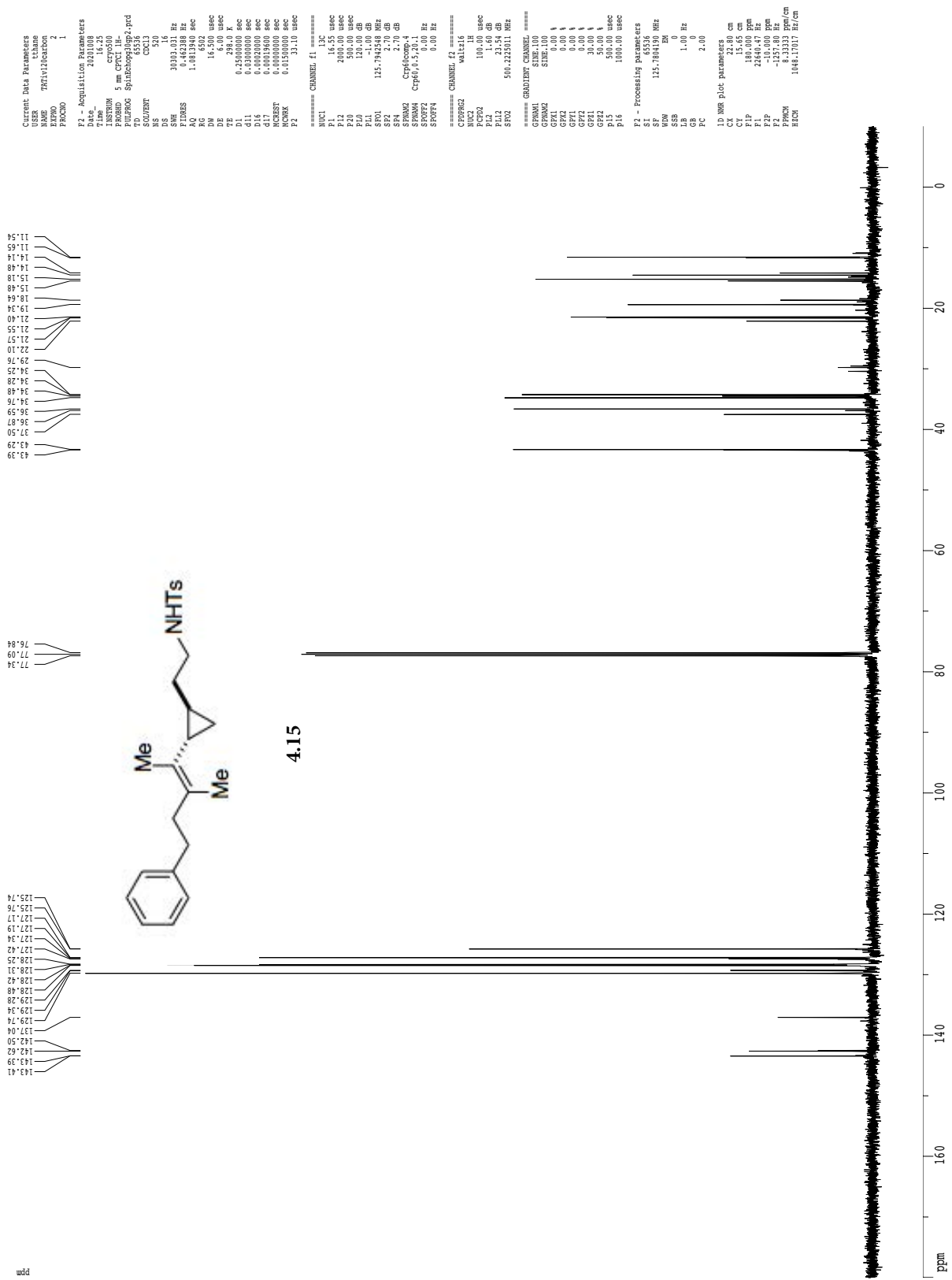
F2 - Acquisition Parameters  
 Date\_ 2020.008  
 Time 16.23  
 Operator  
 PULPROG zgpg30  
 PROCNO 5  
 TD 8178  
 SFO 400.1419999  
 SOLVENT CDCl3  
 DS 9  
 SFR 8012.820 Hz  
 FIDRES 0.098044 Hz  
 AQ 5.1998774 sec  
 RG 62.400 usec  
 DE 6.00 usec  
 TE 298.2 K  
 0.1000000 sec  
 MCHRES 0.0000000 sec  
 ACQRES 0.0150000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 1.50 usec  
 PL1 1.66 dB  
 SFO1 500.2235015 MHz

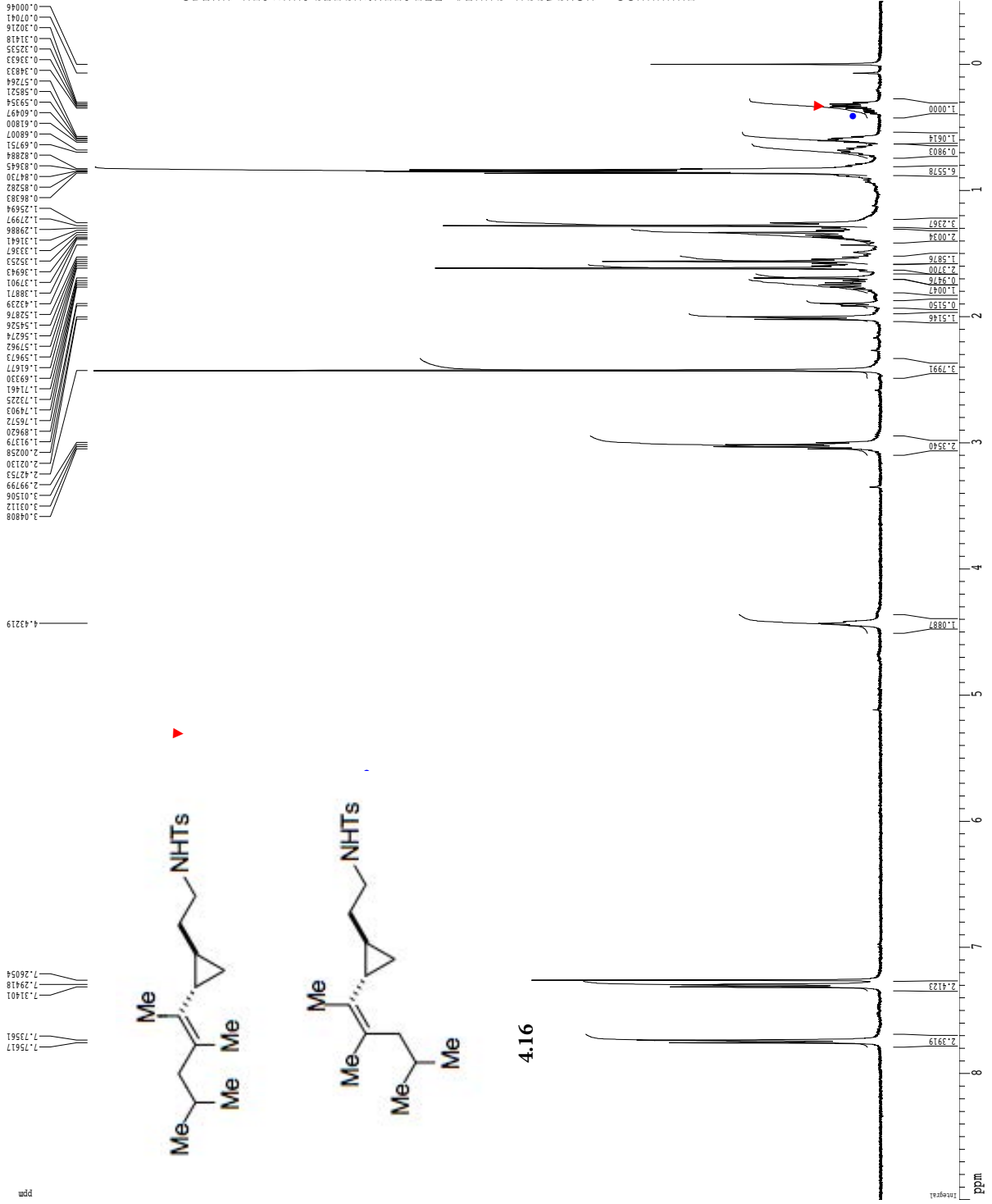
F2 - Processing Parameters  
 SI 65536  
 SF 500.220359 MHz  
 WDW no  
 GB 0  
 PC 1.00

LD NMR file parameters  
 CY 22.80 cm  
 C1 5.00 cm  
 F1P 9.000 ppm  
 F2 4501.98 Hz  
 F2 250.11 Hz  
 PPMCM 0.41667 ppm/cm  
 HZCM 206.44502 Hz/cm

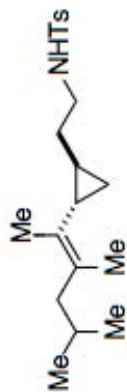
Z-restored spin-echo 13C spectrum with 1H decoupling



1H spectrum

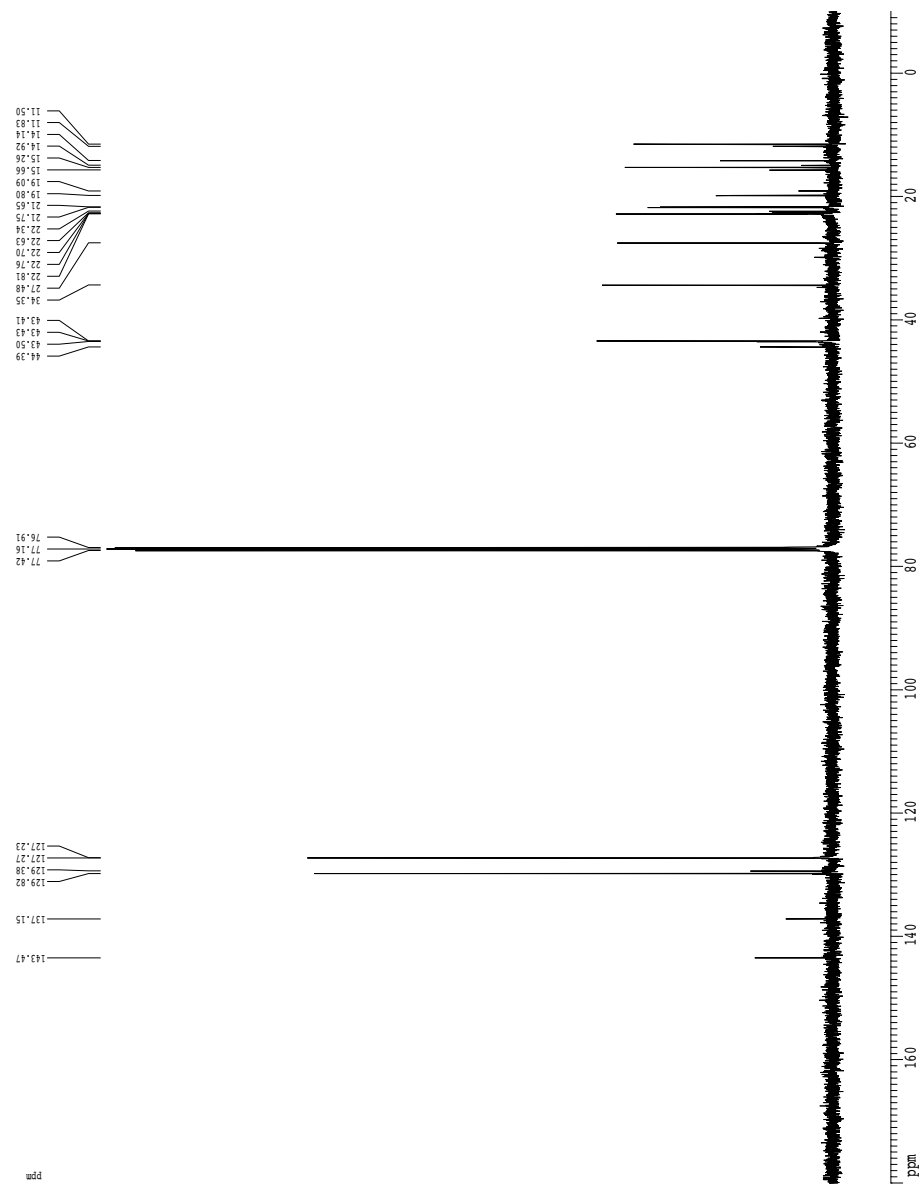


Current Data Parameters  
 USER TWY172-9-11-2020  
 NAME tname  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20200911  
 Time 11:13  
 PROBHD 5 mm QNP HIF/PP  
 PULPROG zg30  
 TD 65536  
 SFO 400.130034  
 ACOUENT COCL2  
 NS 2  
 DS 2  
 SWH 6410.256 Hz  
 FIDRES 0.1000000 Hz  
 AQ 5.111873 sec  
 RG 322.5  
 DW 78.000 usec  
 DE 4.50 usec  
 DI 24.00 usec  
 D1 0.1000000 sec  
 MCREST 0.0000000 sec  
 MCRBK 0.0150000 sec  
 ===== CHANNEL f1 =====  
 NUCL 1H  
 P1 12.00 usec  
 PL 0.00 dB  
 SFO1 400.130034 MHz  
 F2 - Processing parameters  
 SI 65536  
 SF 400.130034 MHz  
 NS 0  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 2.00  
 ID NMR plot parameters  
 CX 42.80 cm  
 CY 42.80 cm  
 FIP 9.000 ppm  
 F1 3601.17 Hz  
 F2 -0.500 ppm  
 FZP -0.500 ppm  
 FWHM 0.41667 ppm/cm  
 HCN 166.72086 Hz/cm



4.16

<sup>13</sup>C spectrum with <sup>1</sup>H decoupling



```

Current Data Parameters
USER          tthane
NAME         TMA117Zcarbon
EXPNO        2
PROCNO       1

F2 - Acquisition Parameters
Date_        20200718
Time         11.45
INSTRUM      gm500
PROBHD       5 mm broadband
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           704
DS           4
SWH          30303.031 Hz
FIDRES       0.462388 Hz
AQ           1.0813940 sec
RG           5792.6
DW           16.500 usec
DE           6.00 usec
TE           297.9 K
D1           0.25000000 sec
d11          0.03000000 sec
ACREST       0.00000000 sec
MCWRK       0.01500000 sec

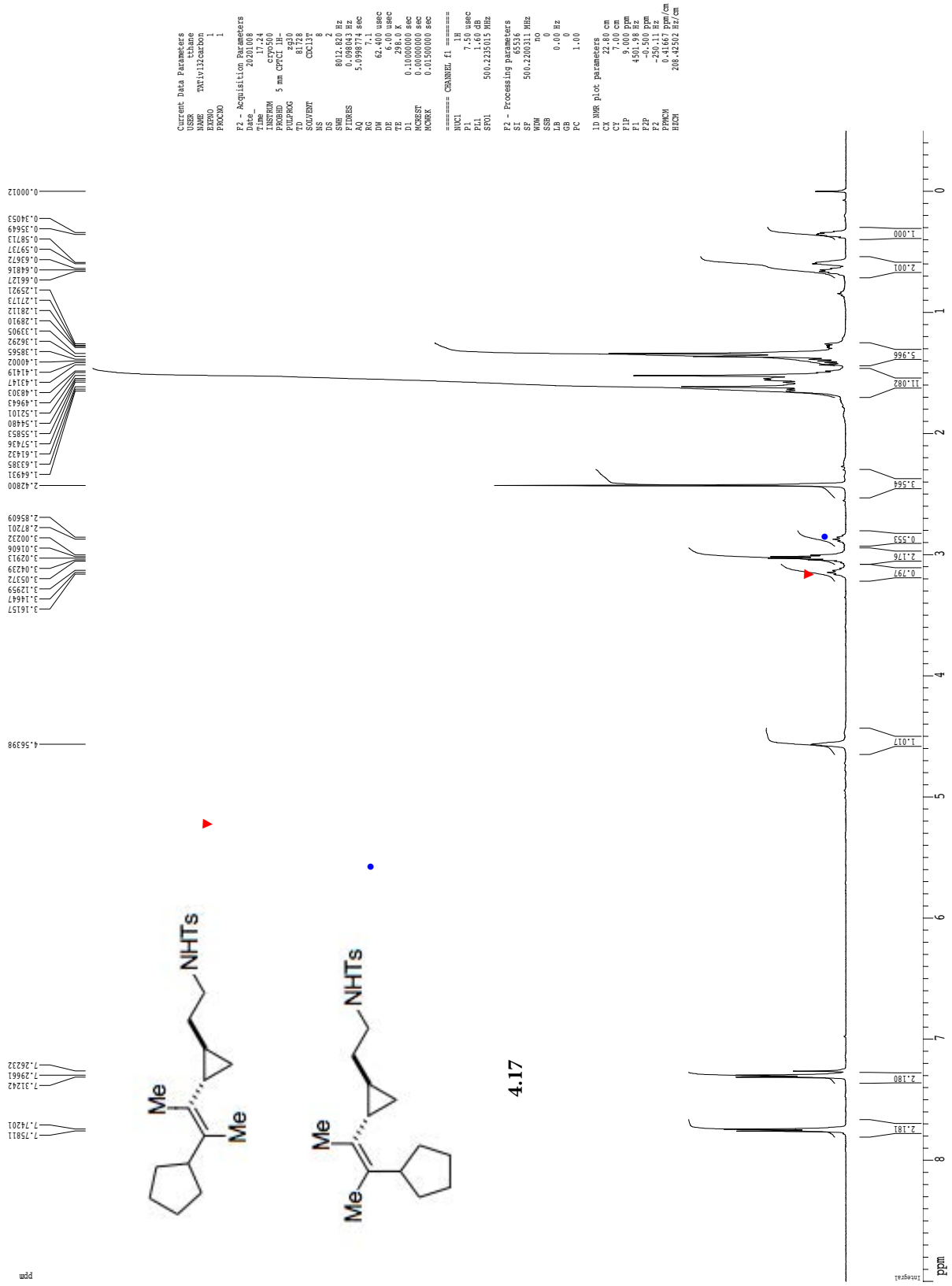
===== CHANNEL f1 =====
NUC1         13C
P1           14.20 usec
PL1          -6.00 dB
SFO1         125.4245824 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2        80.00 usec
PL2          -6.00 dB
PL12         12.30 dB
SFO2         498.7524937 MHz

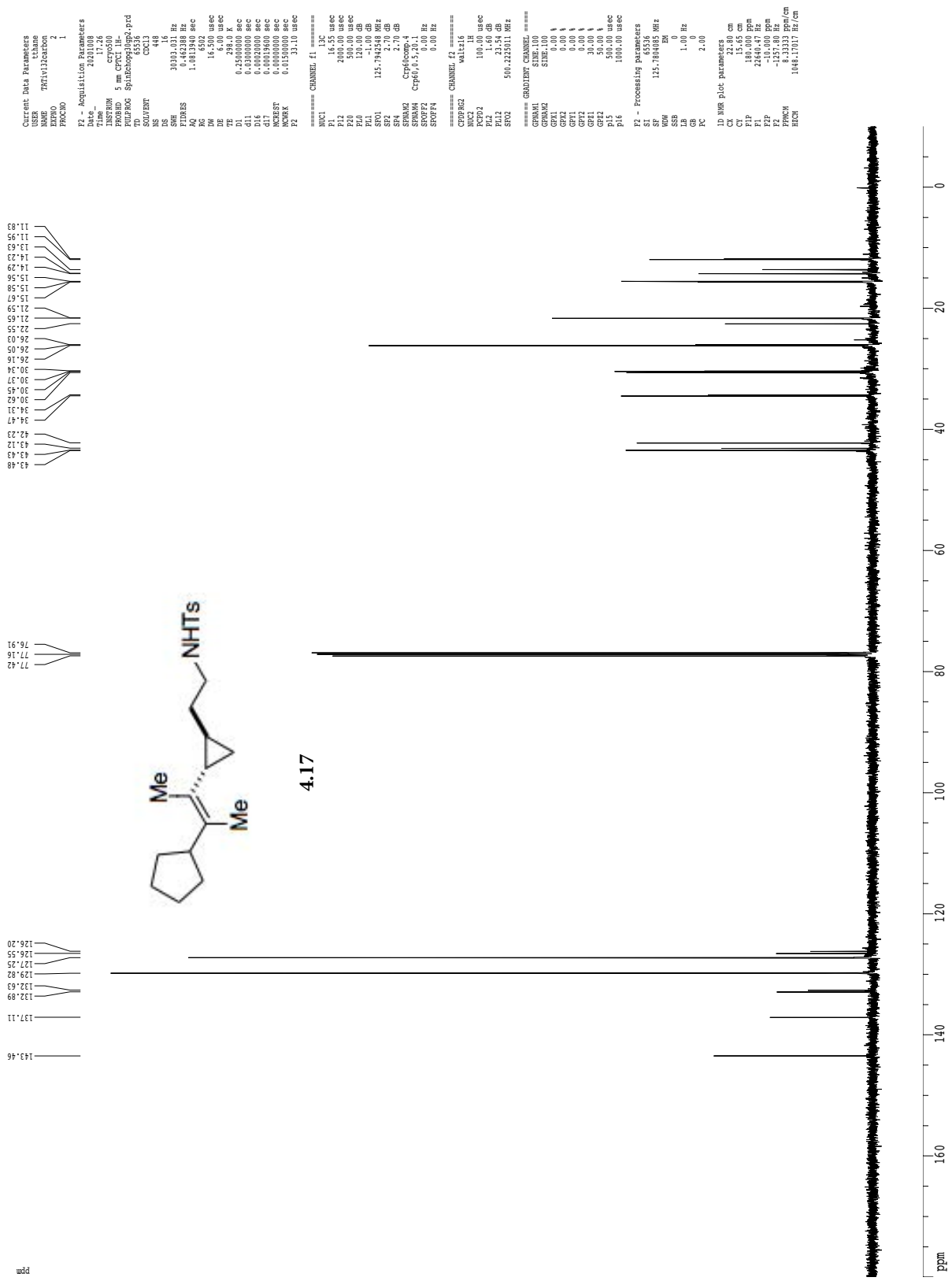
F2 - Processing parameters
SI           65536
SF           125.4107752 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00

1D NMR plot parameters
CX           20.00 cm
CY           12.50 cm
FLP          180.000 ppm
F1           22573.94 Hz
F2           -10.000 ppm
F3           -1254.11 Hz
PPMCM       9.50000 ppm/cm
HZCM        1191.40234 Hz/cm
  
```

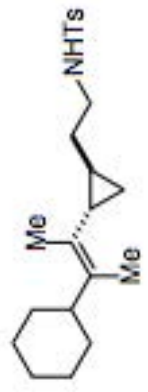
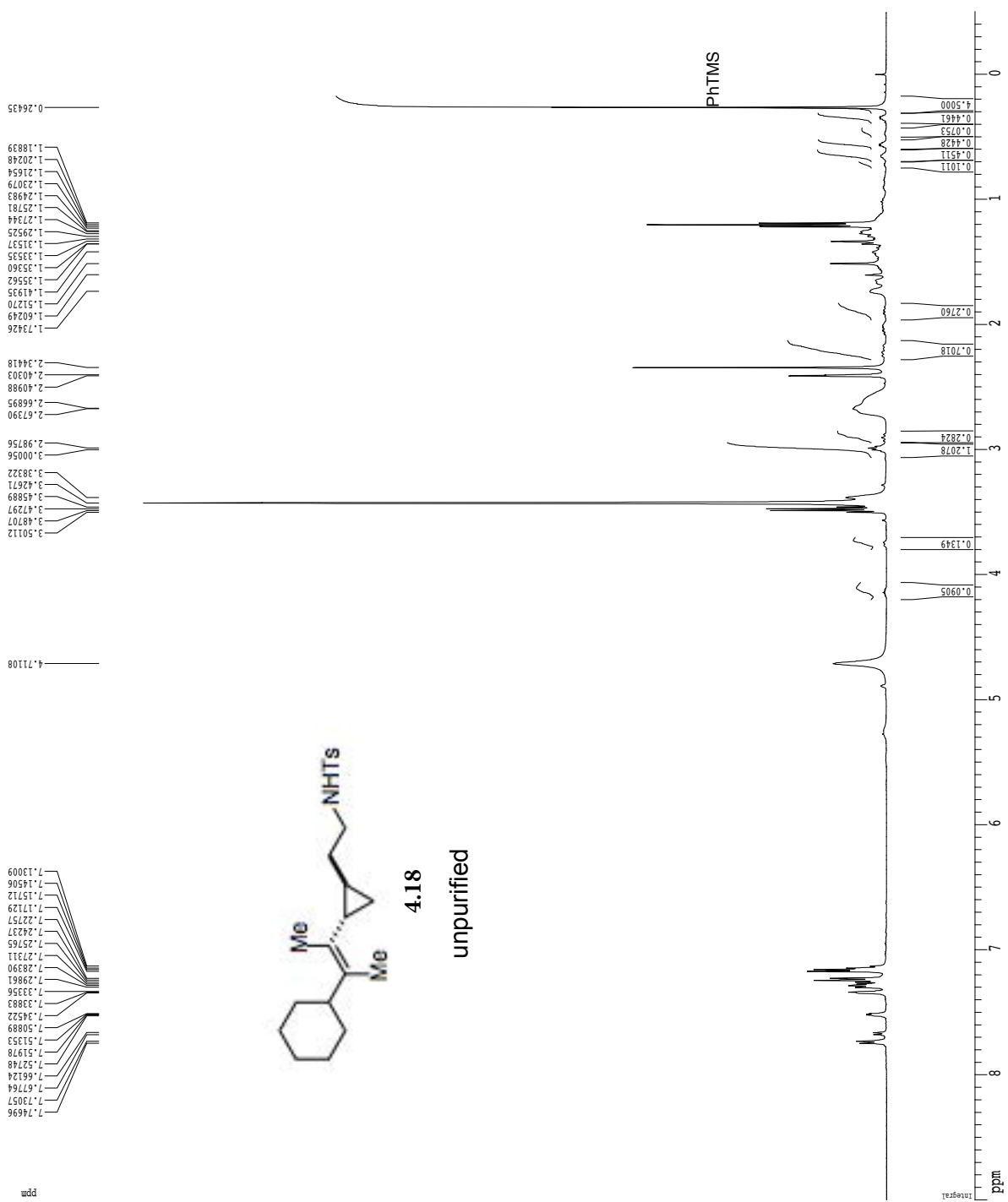
1H spectrum



Z-restored spin-echo 13C spectrum with 1H decoupling



1H spectrum



4.18  
unpurified

Current Data Parameters  
 USER khewitt1  
 NAME KAH-III-145-Crude  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20200617  
 Time\_ 18.55  
 INSTRUM gn500  
 PROBHD 5 mm broadband  
 PULPROG zg30  
 TD 81728  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.098043 Hz  
 AQ 5.0998774 sec  
 RG 40.3  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 MCREST 0.00000000 sec  
 MCWRK 0.01500000 sec

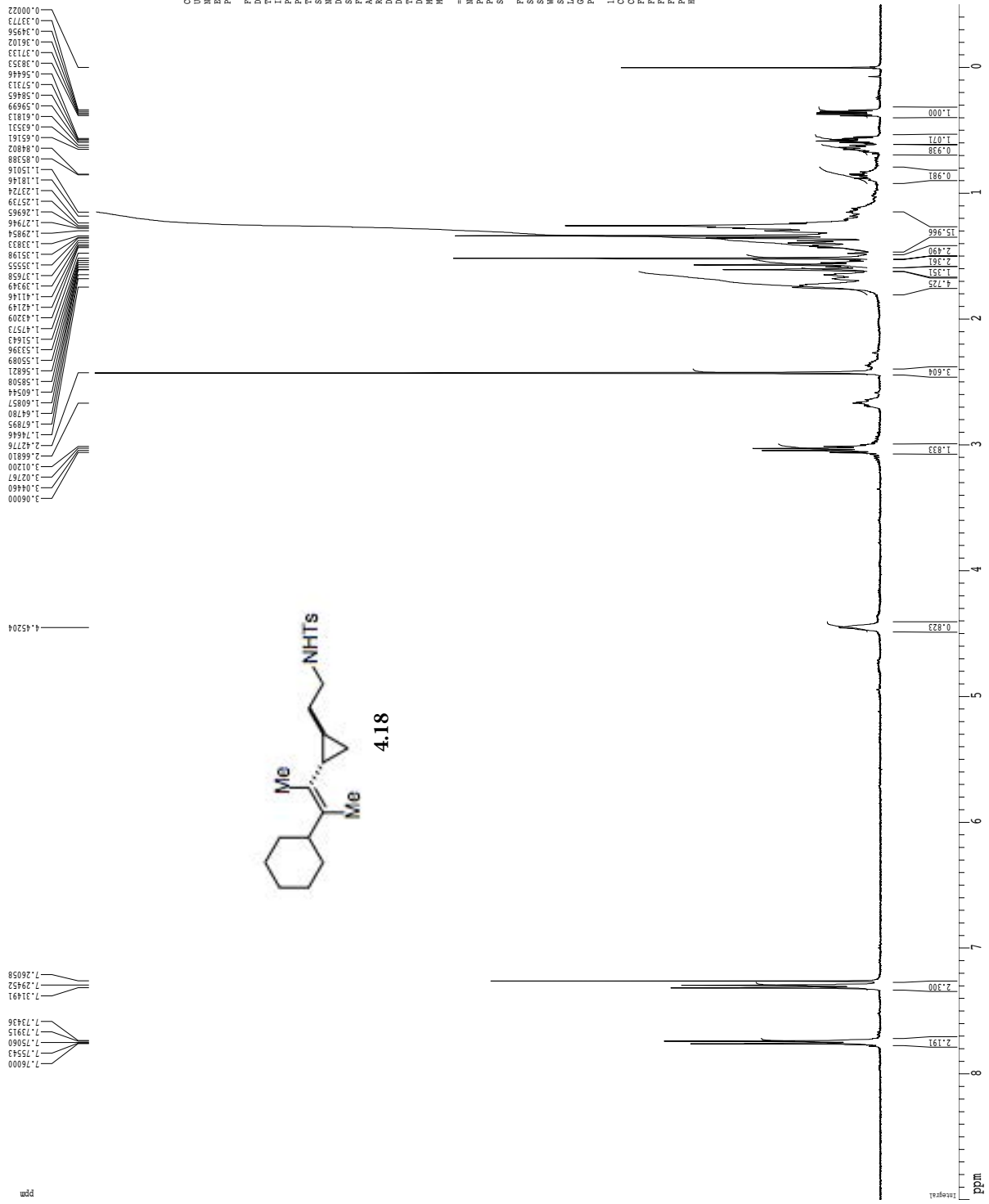
=====  
 CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -6.00 dB  
 SF01 498.7534913 MHz

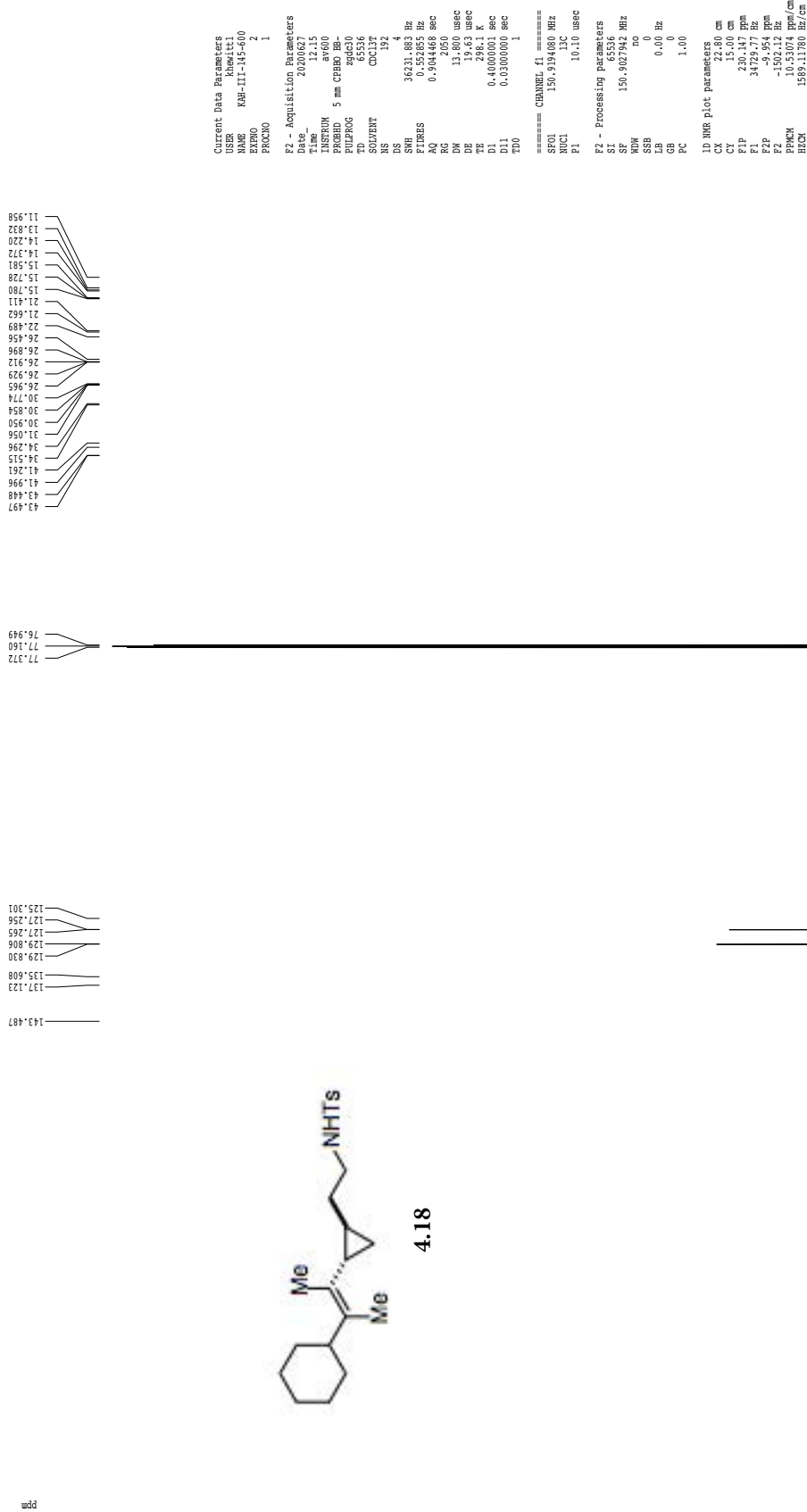
F2 - Processing parameters  
 SI 65536  
 SF 498.7500244 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 12.50 cm  
 FIP 9.000 ppm  
 F1 4488.75 Hz  
 F2p -0.500 ppm  
 F2 -249.38 Hz  
 PPMCM 0.47500 ppm/cm  
 HZCM 236.90627 Hz/cm

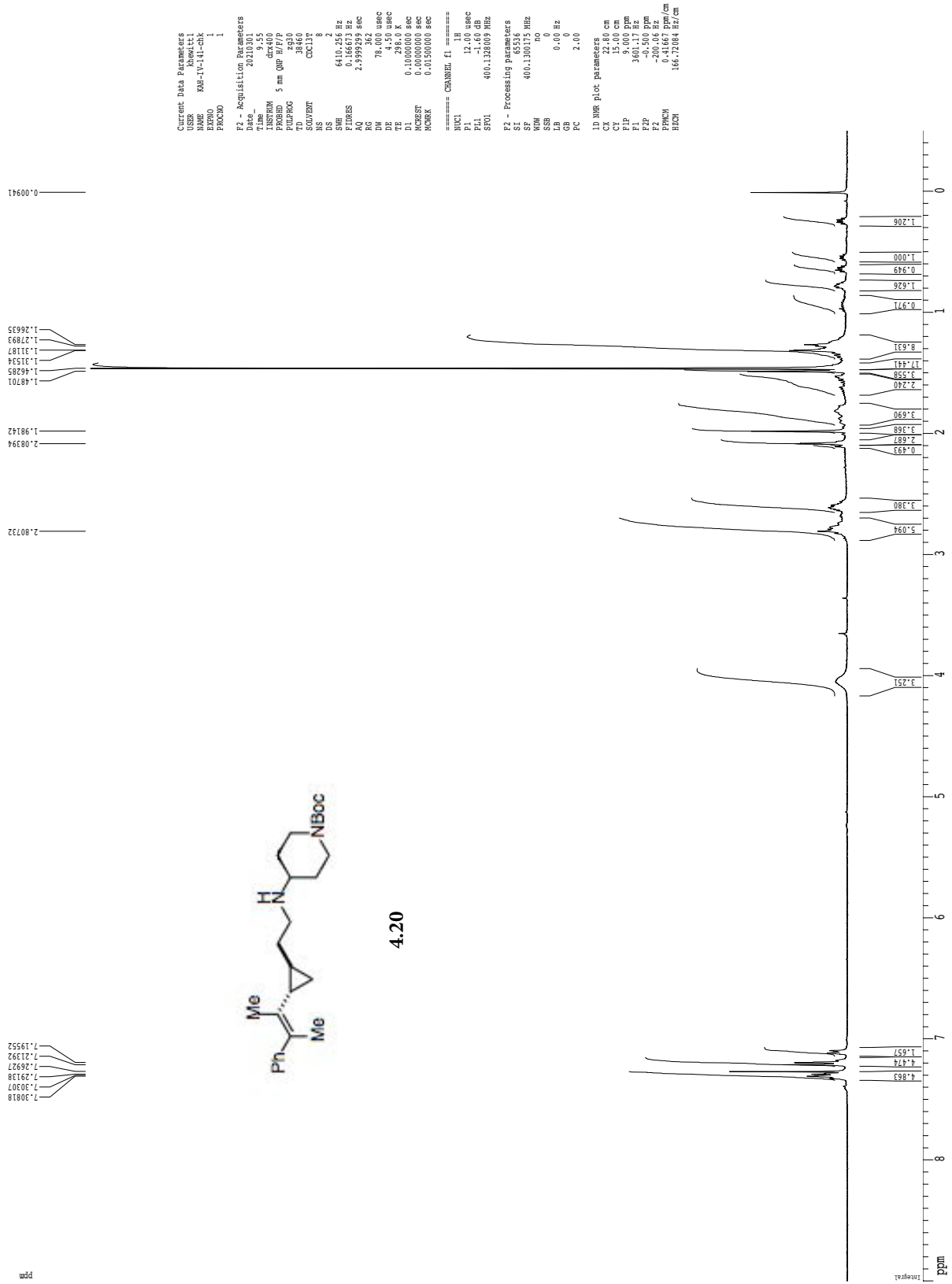


1H spectrum





1H spectrum



Current Data Parameters  
 USER: Komettt  
 NAME: KAU-TV-141-CHK  
 EXNO: 1  
 PROCNO: 1

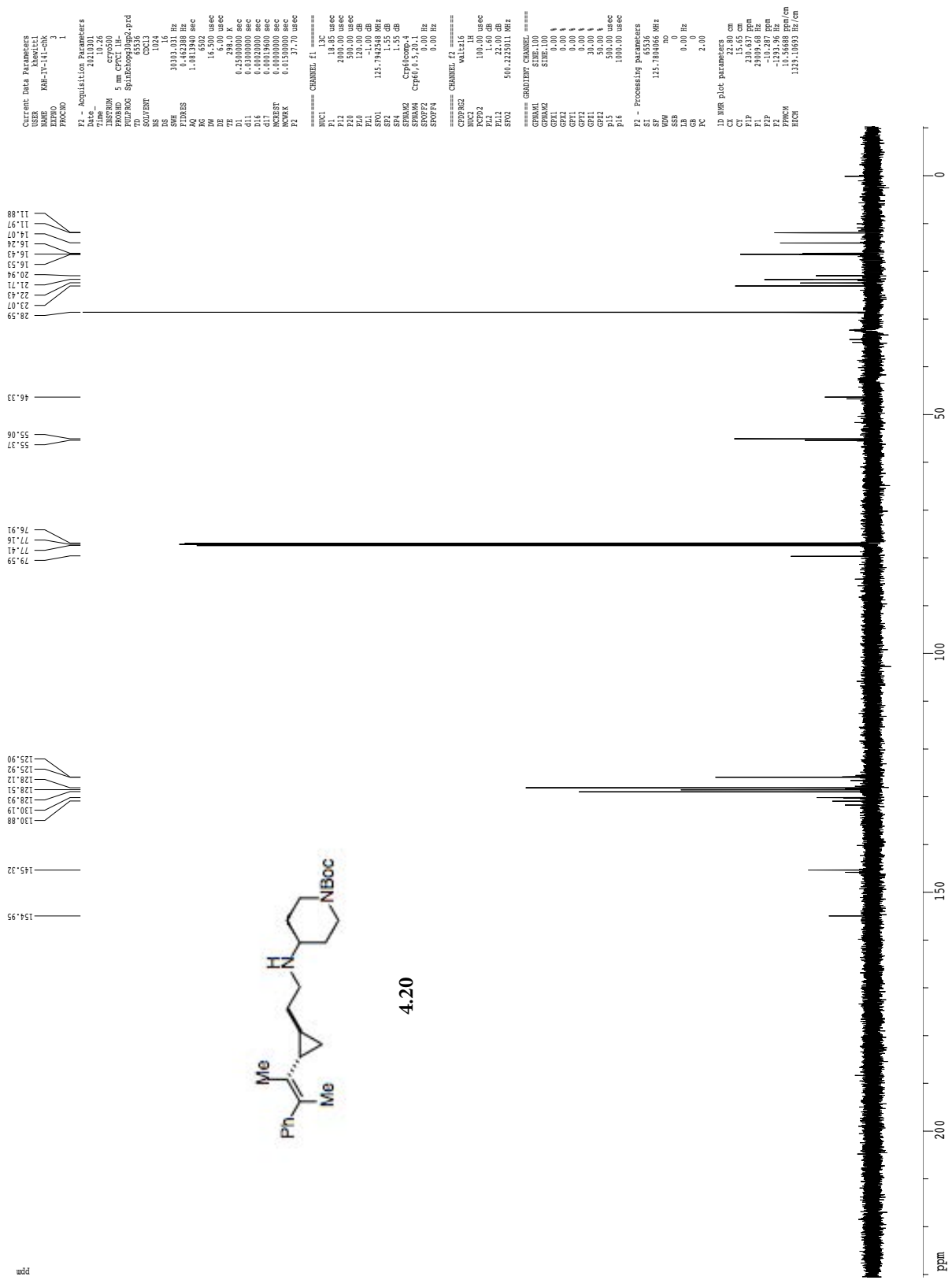
F2 - Acquisition Parameters  
 Date\_: 20210301  
 Time: 9.55  
 INSTRUM: spect  
 PROCNO: 1  
 PULPROG: zgpg30  
 TD: 32768  
 SOLVENT: CDCl3  
 DS: 4  
 SFO: 6410.258 Hz  
 FIDRES: 0.16667 Hz  
 AQ: 2.399729 sec  
 RG: 327.68  
 DW: 78.000 usec  
 DE: 4.50 usec  
 TE: 298.2 K  
 TRANSF: 0.100000 sec  
 MORGAN: 0.000000 sec  
 MCOREK: 0.0150000 sec

===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -1.68 dB  
 SFO1: 400.1328009 MHz

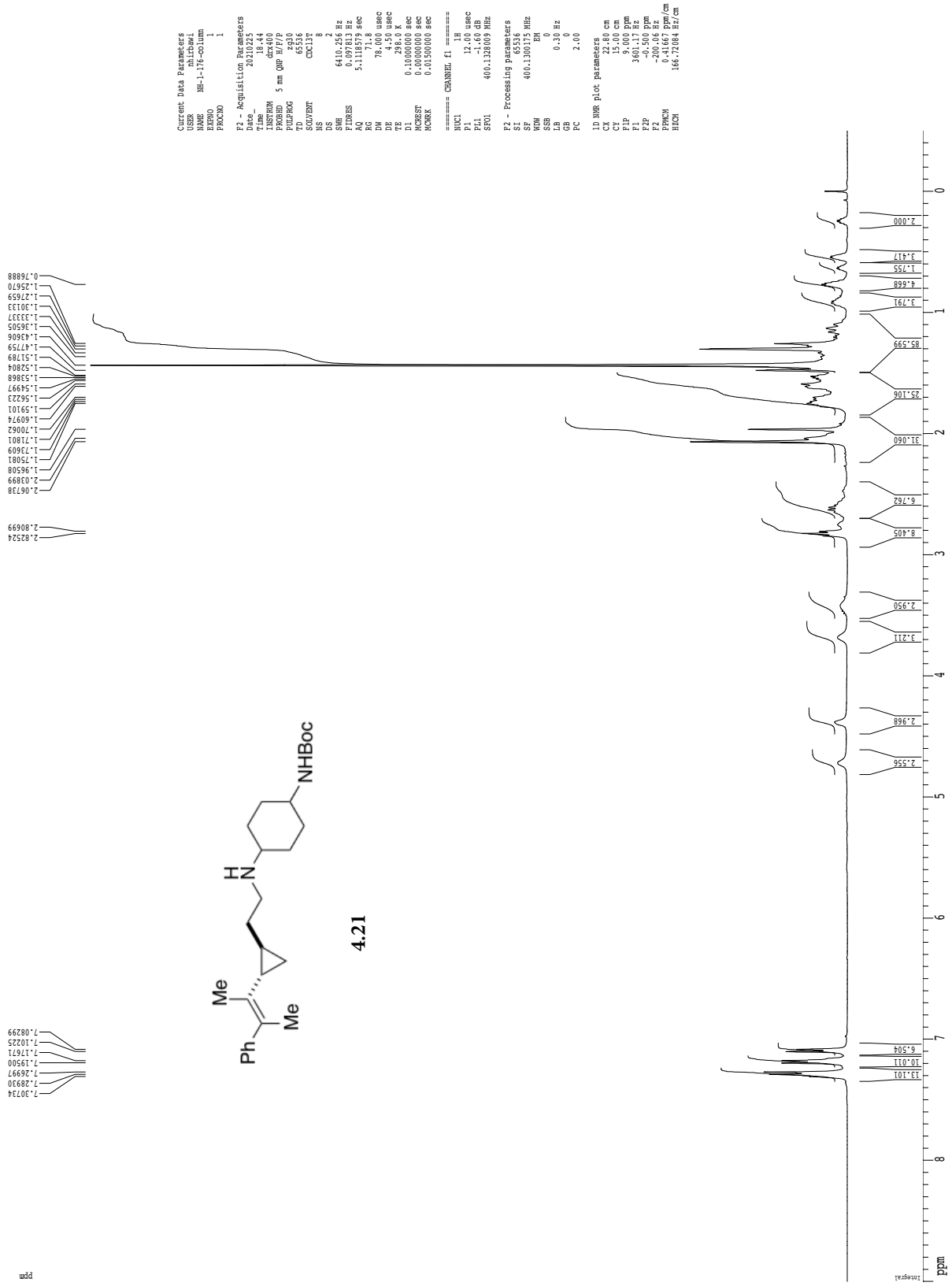
F2 - Processing Parameters  
 SI: 65536  
 SF: 400.1300175 MHz  
 WDW: no  
 SSB: 0  
 GB: 0  
 PC: 2.00

ID: NMR PLOT Parameters  
 CY: 22.80 cm  
 CX: 15.00 cm  
 FID: 9.000 ppm  
 FT: 3600.17 Hz  
 F2: 400.1300175 MHz  
 PPMX: -2000.00 Hz  
 PPMY: 0.41667 ppm/cm  
 HZCM: 166.72084 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



1H spectrum





Current Data Parameters  
 USER NH-1-155-HCARDGSSY  
 EXPRNO 2  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20210223  
 Time 17:33  
 INSTRUM spect  
 PROBRD 5 mm CPXI 1H-  
 PULPROG zgpg30-pz  
 TD 2048  
 SOLVENT CDCl3T  
 NS 2  
 DS 16  
 SFO1 496.461 Hz  
 SF 2195509 Hz  
 AQ 0.2277876 sec  
 RG 143.7  
 DM 111.200 usec  
 DE 6.00 usec  
 TE 298.0 K  
 DO 0.0003000 sec  
 D1 1.0000000 sec  
 D13 0.0003000 sec  
 D16 0.0020000 sec  
 INO 0.0022240 sec

===== CHANNEL f1 =====

NUC1 1H  
 P1 9.75 usec  
 PL 0.00 dB  
 SFO1 500.2220314 MHz

===== GRADIENT CHANNEL =====

GPRM1 SMSQ10.100  
 GPRM2 SMSQ10.100  
 GPX1 0.00 %  
 GPZ1 0.00 %  
 GPY1 0.00 %  
 GPZ2 0.00 %  
 GPY2 0.00 %  
 GPC1 17.00 %  
 GPC2 17.00 %  
 PL6 1000.00 usec

F1 - Acquisition Parameters

NUC0 25  
 P1 25.00 usec  
 PL 0.00 dB  
 SFO1 500.222 MHz  
 FIDRES 17.564074 Hz  
 SN 8.989 ppm  
 FMODE QF

F2 - Processing parameters

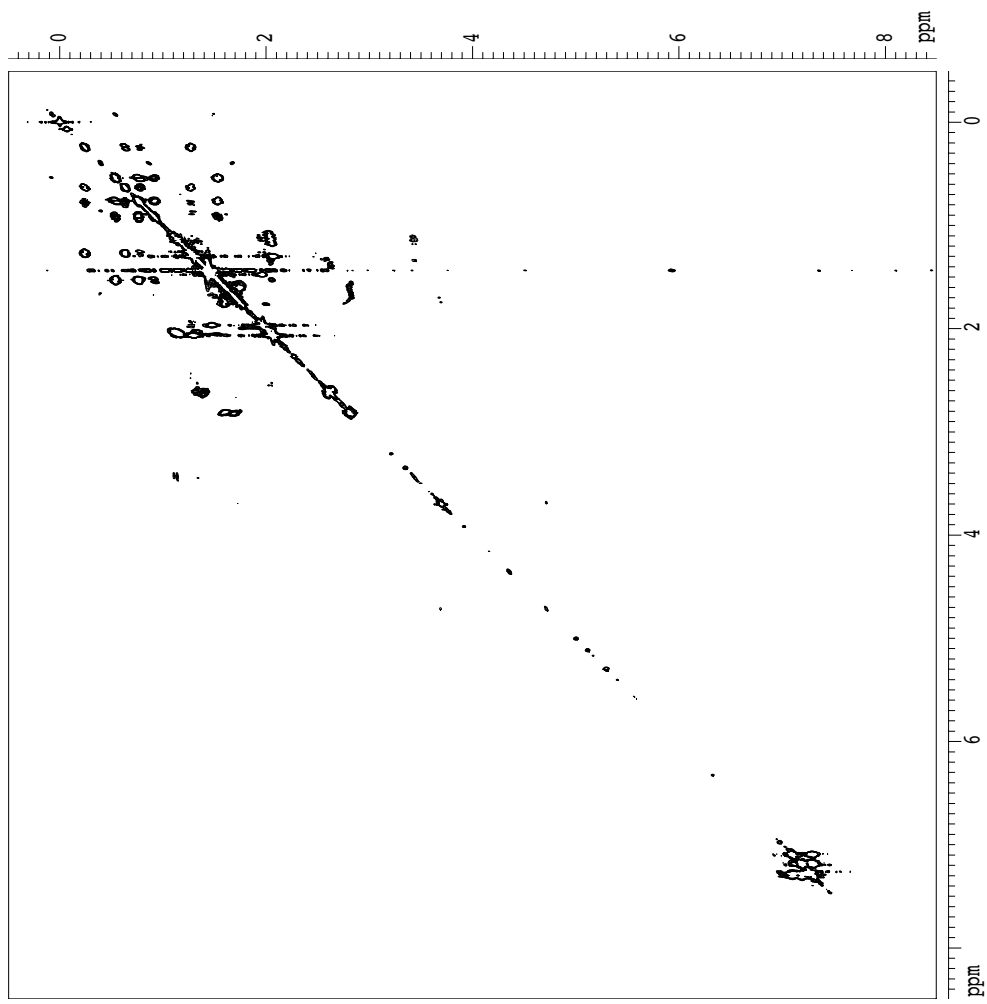
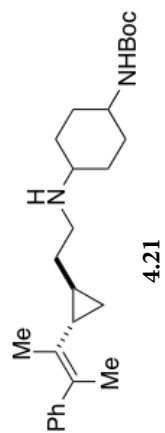
SI 1024  
 SF 500.2200309 MHz  
 SFO 500.2200309 MHz  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 EC 1.00

F1 - Processing parameters

SI 1024  
 SF 500.2200309 MHz  
 SFO 500.2200309 MHz  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters

CV2 15.60 cm  
 CX1 15.60 cm  
 F2FLO 8.494 ppm  
 FZLO 4249.08 Hz  
 FZHI -0.454 ppm  
 FZHI -247.32 Hz  
 F1FLO 8.494 ppm  
 F1FLO 4249.08 Hz  
 F1FHI -0.454 ppm  
 F1FHI -247.32 Hz  
 F2PMCK 0.59926 ppm/cm  
 FZPMCK 299.76016 Hz/cm  
 F1PMCK 0.59926 ppm/cm  
 F1ZPMCK 299.76016 Hz/cm

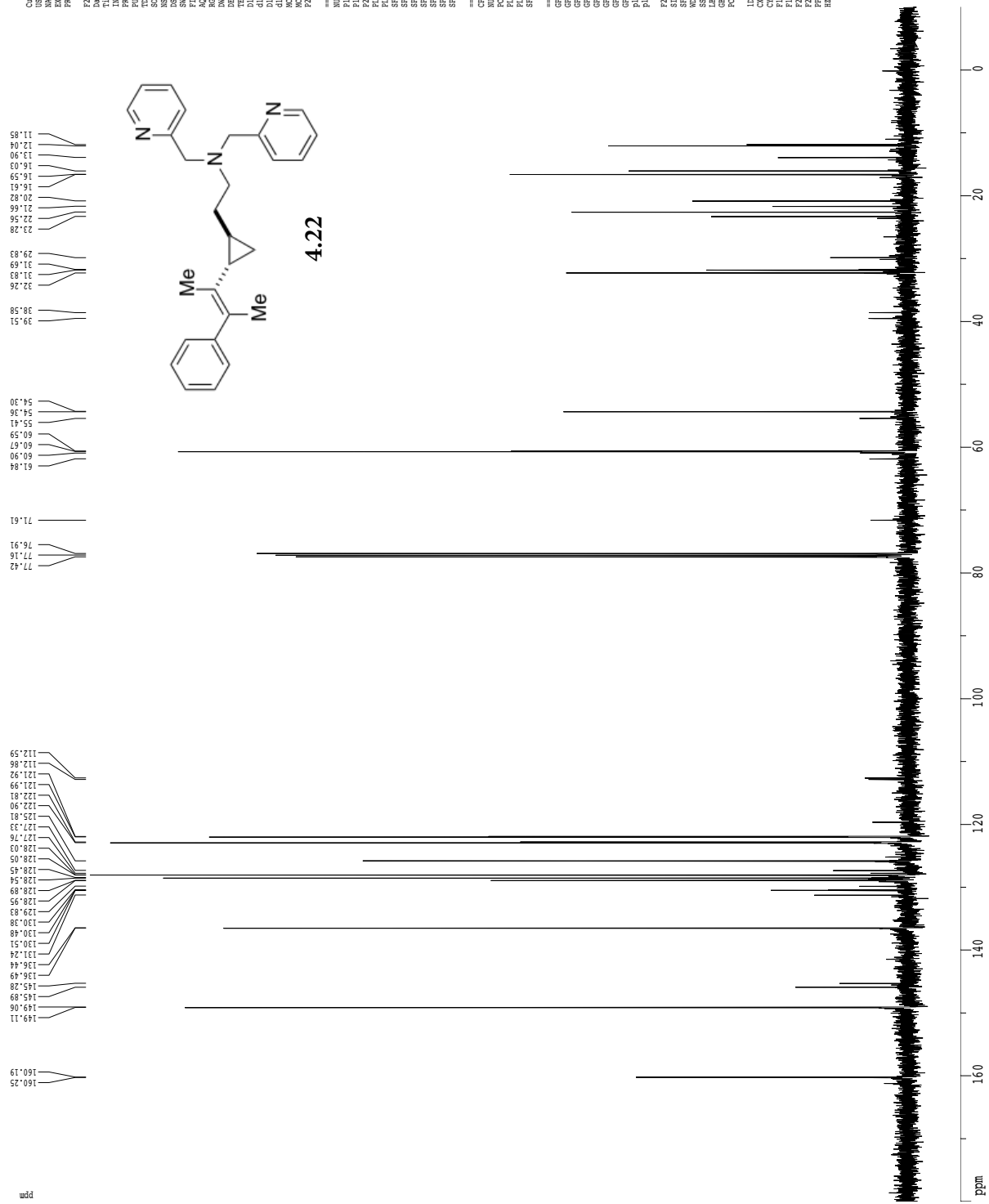


gcosy60





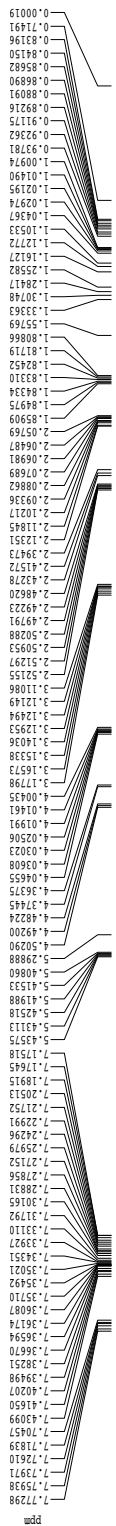
Z-restored spin-echo 13C spectrum with 1H decoupling



```

Current Data Parameters
USER      khwatt1
NAME      TMLV13Carbon
PROBHD    5mm QNP1HHR
PROCNO    1
=====
F2 - Acquisition Parameters
Time      21.14
Date_     11.24
INSTRUM   crysov00
PROBHD    5mm QNP1HHR
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         2
DS         4
SWH        30803.801 Hz
FIDRES     0.462388 Hz
AQ          1.14256 sec
RG          728.2
WDW         EM
SSB         0
LB          16.500 usec
GB          0.000000 sec
PC          2.00
D1          0.25000000 sec
d11         0.00000000 sec
d12         0.00000000 sec
d13         0.00000000 sec
d14         0.00000000 sec
d15         0.00000000 sec
d16         0.00000000 sec
d17         0.00000000 sec
d18         0.00000000 sec
d19         0.00000000 sec
d20         0.00000000 sec
d21         0.00000000 sec
d22         0.00000000 sec
d23         0.00000000 sec
d24         0.00000000 sec
d25         0.00000000 sec
d26         0.00000000 sec
d27         0.00000000 sec
d28         0.00000000 sec
d29         0.00000000 sec
d30         0.00000000 sec
d31         0.00000000 sec
d32         0.00000000 sec
d33         0.00000000 sec
d34         0.00000000 sec
d35         0.00000000 sec
d36         0.00000000 sec
d37         0.00000000 sec
d38         0.00000000 sec
d39         0.00000000 sec
d40         0.00000000 sec
d41         0.00000000 sec
d42         0.00000000 sec
d43         0.00000000 sec
d44         0.00000000 sec
d45         0.00000000 sec
d46         0.00000000 sec
d47         0.00000000 sec
d48         0.00000000 sec
d49         0.00000000 sec
d50         0.00000000 sec
===== CHANNEL f1 =====
NUC1       13C
P1          15.00 usec
PL1         2000.00 dB
P2          500.00 usec
PL2         120.00 dB
P3          120.00 dB
PL3         120.00 dB
SFO1       125.764548 MHz
SF2         1.55 dB
SFO2       125.764548 MHz
SFO3       125.764548 MHz
SFO4       1.55 dB
SFO5       1.55 dB
SFO6       1.55 dB
SFO7       1.55 dB
SFO8       1.55 dB
SFO9       1.55 dB
SFO10      1.55 dB
SFO11      1.55 dB
SFO12      1.55 dB
SFO13      1.55 dB
SFO14      1.55 dB
SFO15      1.55 dB
SFO16      1.55 dB
SFO17      1.55 dB
SFO18      1.55 dB
SFO19      1.55 dB
SFO20      1.55 dB
SFO21      1.55 dB
SFO22      1.55 dB
SFO23      1.55 dB
SFO24      1.55 dB
SFO25      1.55 dB
SFO26      1.55 dB
SFO27      1.55 dB
SFO28      1.55 dB
SFO29      1.55 dB
SFO30      1.55 dB
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
P2          100.00 usec
PL2         1.60 dB
PL12        22.00 dB
SFO1        500.1325011 MHz
===== GRADIENT CHANNEL =====
GPMAX1     5.00 G
SHE1100    5.00 G
SHE2100    5.00 G
GXA1        0.40 V
GXA2        0.40 V
GXA3        0.40 V
GXA4        0.40 V
GXA5        0.40 V
GXA6        0.40 V
GXA7        0.40 V
GXA8        0.40 V
GXA9        0.40 V
GXA10       0.40 V
GXA11       0.40 V
GXA12       0.40 V
GXA13       0.40 V
GXA14       0.40 V
GXA15       0.40 V
GXA16       0.40 V
GXA17       0.40 V
GXA18       0.40 V
GXA19       0.40 V
GXA20       0.40 V
===== Processing parameters =====
SI          32768
SF          125.764548 MHz
WDW         EM
SSB         0
GB          0
LB          1.60000000 Hz
PC          2.00
===== ID WDR p/cr parameters =====
CX          22.80 cm
CY          15.45 cm
CZ          15.45 cm
P1          23840.37 ppm
P2          23840.37 ppm
P3          23840.37 ppm
P4          23840.37 ppm
P5          23840.37 ppm
P6          23840.37 ppm
P7          23840.37 ppm
P8          23840.37 ppm
P9          23840.37 ppm
P10         23840.37 ppm
P11         23840.37 ppm
P12         23840.37 ppm
P13         23840.37 ppm
P14         23840.37 ppm
P15         23840.37 ppm
P16         23840.37 ppm
P17         23840.37 ppm
P18         23840.37 ppm
P19         23840.37 ppm
P20         23840.37 ppm
P21         23840.37 ppm
P22         23840.37 ppm
P23         23840.37 ppm
P24         23840.37 ppm
P25         23840.37 ppm
P26         23840.37 ppm
P27         23840.37 ppm
P28         23840.37 ppm
P29         23840.37 ppm
P30         23840.37 ppm
P31         23840.37 ppm
P32         23840.37 ppm
P33         23840.37 ppm
P34         23840.37 ppm
P35         23840.37 ppm
P36         23840.37 ppm
P37         23840.37 ppm
P38         23840.37 ppm
P39         23840.37 ppm
P40         23840.37 ppm
P41         23840.37 ppm
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P52         23840.37 ppm
P53         23840.37 ppm
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P55         23840.37 ppm
P56         23840.37 ppm
P57         23840.37 ppm
P58         23840.37 ppm
P59         23840.37 ppm
P60         23840.37 ppm
P61         23840.37 ppm
P62         23840.37 ppm
P63         23840.37 ppm
P64         23840.37 ppm
P65         23840.37 ppm
P66         23840.37 ppm
P67         23840.37 ppm
P68         23840.37 ppm
P69         23840.37 ppm
P70         23840.37 ppm
P71         23840.37 ppm
P72         23840.37 ppm
P73         23840.37 ppm
P74         23840.37 ppm
P75         23840.37 ppm
P76         23840.37 ppm
P77         23840.37 ppm
P78         23840.37 ppm
P79         23840.37 ppm
P80         23840.37 ppm
P81         23840.37 ppm
P82         23840.37 ppm
P83         23840.37 ppm
P84         23840.37 ppm
P85         23840.37 ppm
P86         23840.37 ppm
P87         23840.37 ppm
P88         23840.37 ppm
P89         23840.37 ppm
P90         23840.37 ppm
P91         23840.37 ppm
P92         23840.37 ppm
P93         23840.37 ppm
P94         23840.37 ppm
P95         23840.37 ppm
P96         23840.37 ppm
P97         23840.37 ppm
P98         23840.37 ppm
P99         23840.37 ppm
P100        23840.37 ppm
=====

```

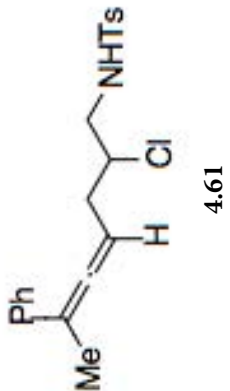


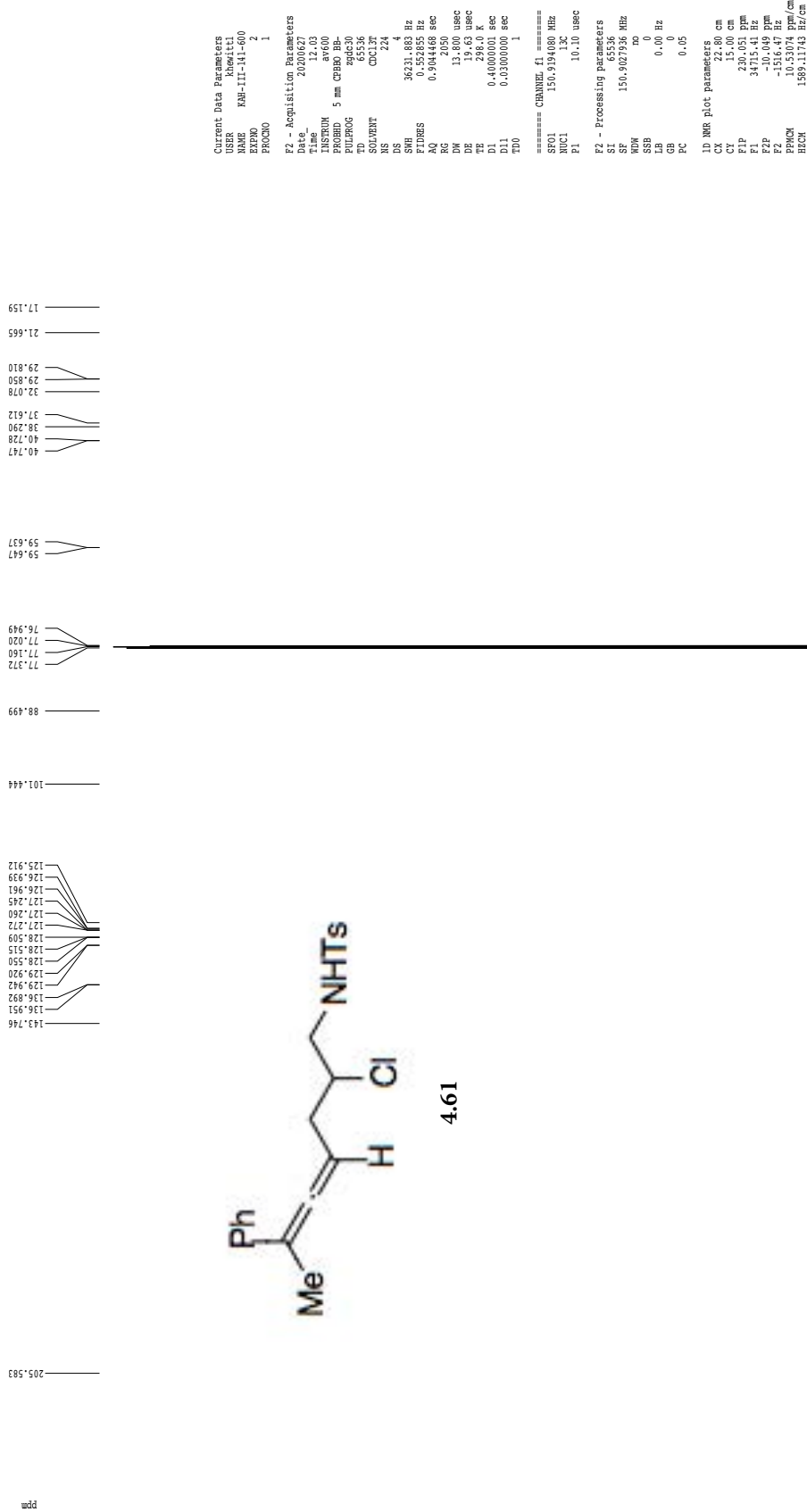
Current Data Parameters  
 USER Khe Wittl  
 NAME KM-11-141-000  
 PRACNO 1  
 F2 - Acquisition Parameters  
 Date\_ 2020627  
 Time 11:58  
 OPERATOR  
 PROBNM 5 mm CP1PRB00  
 PULPROG zg30  
 TD 98074  
 SOLVENT CDCl3  
 NS 8  
 DS 8  
 SWH 9615.385 Hz  
 F1RES 0.098042 Hz  
 AQ 5.0998979 sec  
 RG 10  
 DM 52.000 usec  
 DE 248.0 usec  
 DI 0.1000000 sec  
 TD0 1

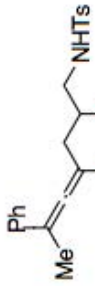
===== CHANNEL f1 =====  
 NU1 600.13418 MHz  
 NU2  
 FI 9.50 usec

F2 - Processing parameters  
 SI 65536  
 SF 600.1300351 MHz  
 NQ 0  
 SSB 0  
 GB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 22.80 cm  
 CY 15.00 cm  
 CZ 6.000 ppm  
 F1 540.117 Hz  
 F2 -300.000 ppm  
 F3 -300.000 ppm  
 PRACNM 0.41697 ppm/cm  
 HZCM 250.05418 Hz/cm







gcosy60

```

Current Data Parameters
=====
USBR      Kspdt11
KAM-IT-141-43
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
=====
Date_     20070727
Time      17:27
INSTRUM   cryo600
PROBHD    5 mm CPXI 1H-
PULPROG   cosypr60.prd
TD         2048
SOLVENT    CDCl3
NS         16
DS         4
SWH        8012.820 Hz
FIDRES     3.912510 Hz
AQ         0.1178952 sec
RG         655
DM         62.400 usec
DE         6.00 usec
TE         298.0 K
d0         0.0000300 sec
d1         1.0000000 sec
d13        0.0000000 sec
d16        0.0002000 sec
IN0        0.00012480 sec

===== CHANNEL f1 =====
NUC1       13
P1         1.50 usec
PL1        1.60 dB
SFO1       500.2235015 MHz

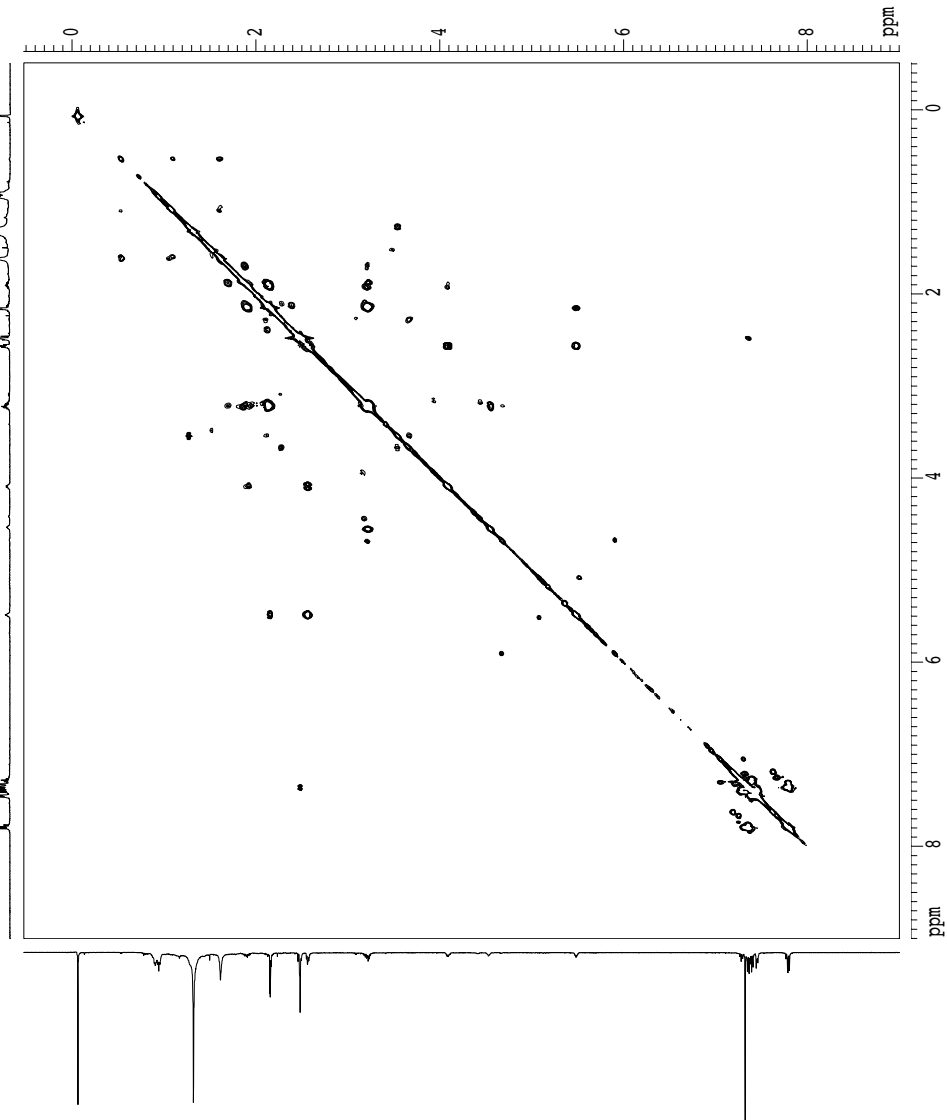
===== GRABBER CHANNEL =====
CONVMT     1
CPDPRG2    Sine-100
GPA1       0.00 %
GPA2       0.00 %
GPI1       0.00 %
GPI2       0.00 %
GPR2       17.00 %
GPP2       17.00 %
PL16       1000.00 usec

F1 - Acquisition parameters
=====
NUC0       13
P0         1.50 usec
PL0        1.60 dB
SFO0       500.2235 MHz
FIDRES     15.650040 Hz
SN         16.018 ppm
FMODE      QF

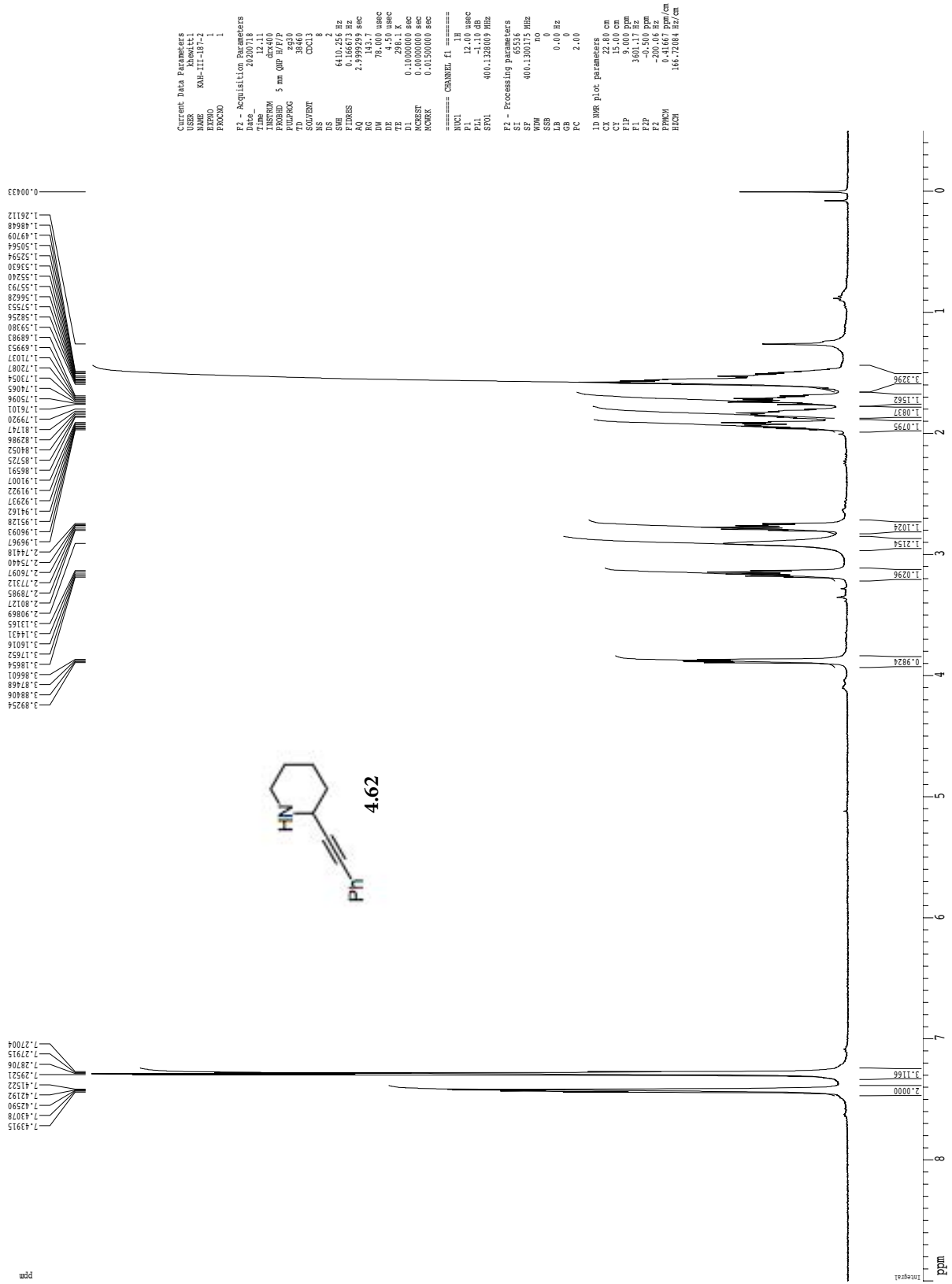
F2 - Processing parameters
=====
SI         1024
SF         500.2200000 MHz
WDW        SINE
SSB        0
GB         0
PC         1.00

F1 - Processing parameters
=====
SI         1024
SF         500.2200000 MHz
WDW        SINE
SSB        0
GB         0
PC         1.00

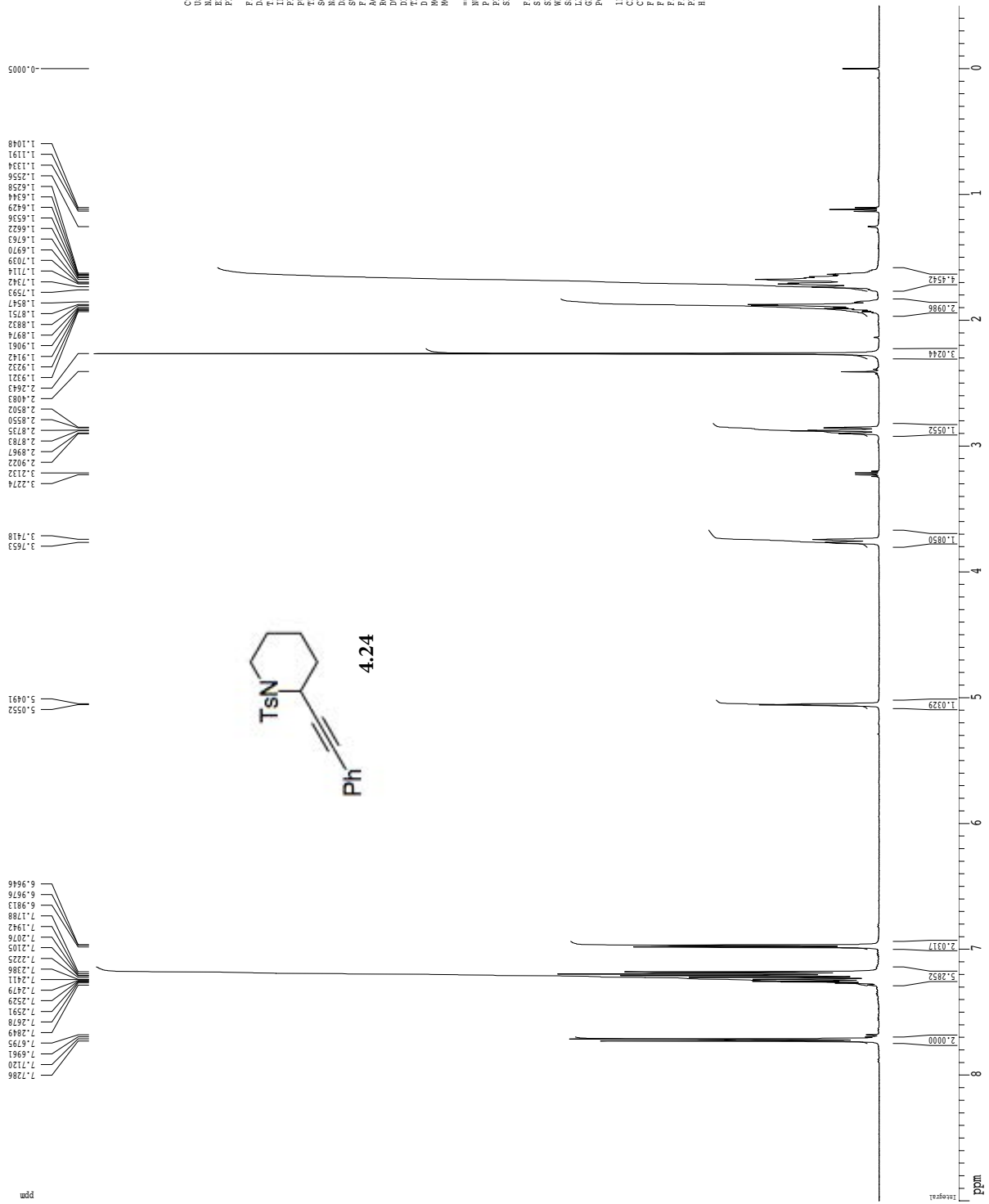
2D NMR plot parameters
=====
CX2        15.00 cm
CX1         9.002 ppm
F2FLO      48.500 ppm
F2FID      -20.508 ppm
F2H1       -25.447 Hz
F2FLO      9.002 ppm
F2FID      4503.14 Hz
F2H1       -20.524 ppm
F2FMCN     0.63407 ppm/cm
F2HCMC     317.1416 Hz/cm
F2FMCN     0.63511 ppm/cm
F2HCMC     317.69581 Hz/cm
  
```



1H spectrum



1H spectrum

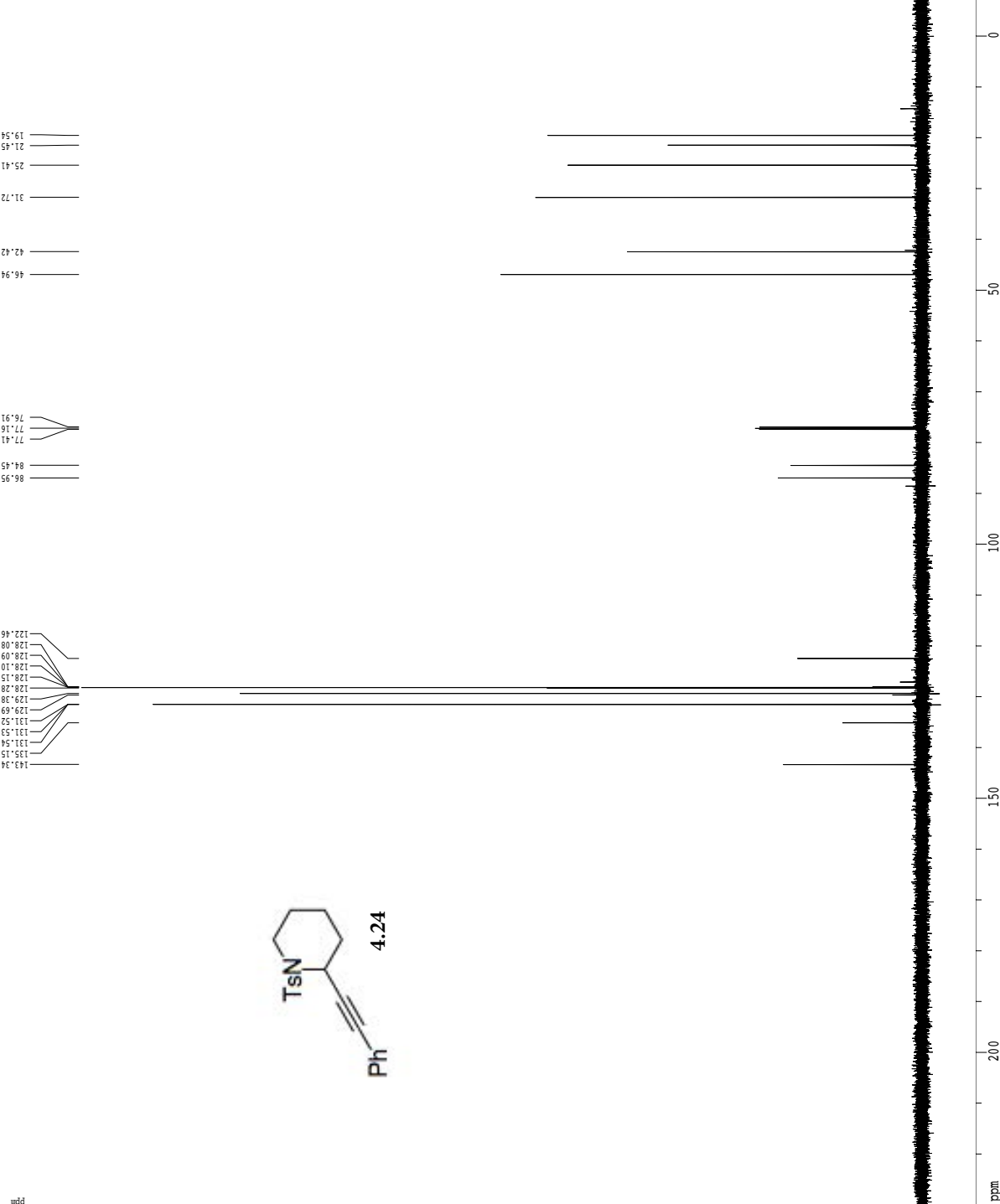
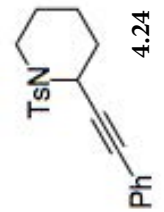
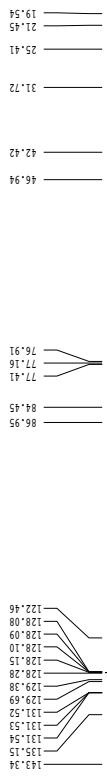


Current Data Parameters  
 USER: KIMWELT  
 NAME: R04-III-209-2  
 EXNO: 1  
 PROCNO: 1

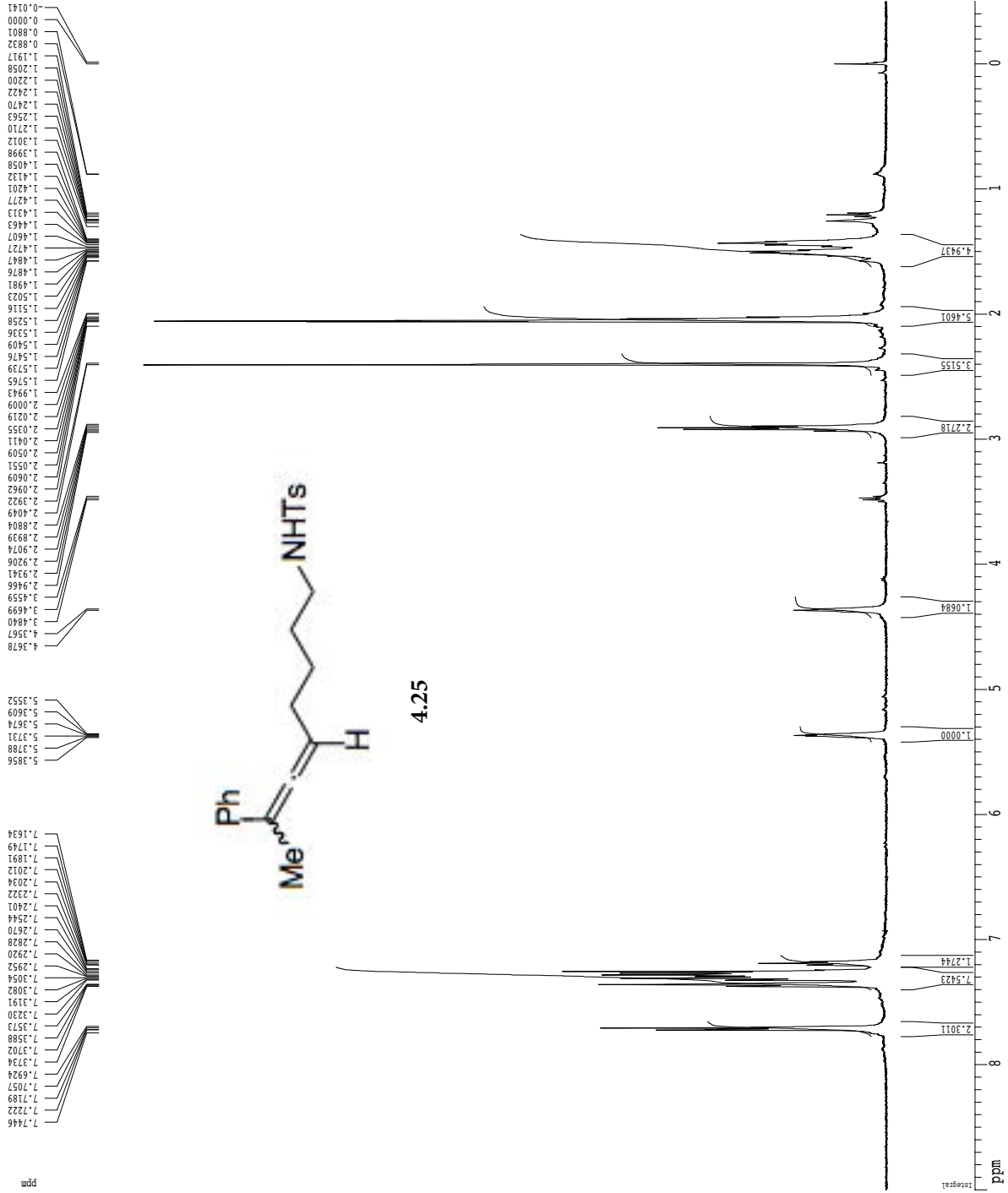
F2 - Acquisition Parameters  
 Date\_: 20201119  
 Time: 17:32  
 SYSTEM: cryo-1  
 PROBNM: 5 mm CRYO-1H  
 PULPROG: zg30  
 TD: 8178  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 500.136260 MHz  
 SFE: 8012.820 Hz  
 FIDRES: 0.096044 Hz  
 AQ: 5.1998774 sec  
 RG: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 0.1000000 sec  
 T2: 0.0000000 sec  
 T3: 0.0000000 sec  
 T4: 0.0000000 sec  
 T5: 0.0000000 sec  
 T6: 0.0000000 sec  
 T7: 0.0000000 sec  
 T8: 0.0000000 sec  
 T9: 0.0000000 sec  
 T10: 0.0000000 sec  
 T11: 0.0000000 sec  
 T12: 0.0000000 sec  
 T13: 0.0000000 sec  
 T14: 0.0000000 sec  
 T15: 0.0000000 sec  
 T16: 0.0000000 sec  
 T17: 0.0000000 sec  
 T18: 0.0000000 sec  
 T19: 0.0000000 sec  
 T20: 0.0000000 sec  
 T21: 0.0000000 sec  
 T22: 0.0000000 sec  
 T23: 0.0000000 sec  
 T24: 0.0000000 sec  
 T25: 0.0000000 sec  
 T26: 0.0000000 sec  
 T27: 0.0000000 sec  
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 T44: 0.0000000 sec  
 T45: 0.0000000 sec  
 T46: 0.0000000 sec  
 T47: 0.0000000 sec  
 T48: 0.0000000 sec  
 T49: 0.0000000 sec  
 T50: 0.0000000 sec  
 T51: 0.0000000 sec  
 T52: 0.0000000 sec  
 T53: 0.0000000 sec  
 T54: 0.0000000 sec  
 T55: 0.0000000 sec  
 T56: 0.0000000 sec  
 T57: 0.0000000 sec  
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 T60: 0.0000000 sec  
 T61: 0.0000000 sec  
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 T63: 0.0000000 sec  
 T64: 0.0000000 sec  
 T65: 0.0000000 sec  
 T66: 0.0000000 sec  
 T67: 0.0000000 sec  
 T68: 0.0000000 sec  
 T69: 0.0000000 sec  
 T70: 0.0000000 sec  
 T71: 0.0000000 sec  
 T72: 0.0000000 sec  
 T73: 0.0000000 sec  
 T74: 0.0000000 sec  
 T75: 0.0000000 sec  
 T76: 0.0000000 sec  
 T77: 0.0000000 sec  
 T78: 0.0000000 sec  
 T79: 0.0000000 sec  
 T80: 0.0000000 sec  
 T81: 0.0000000 sec  
 T82: 0.0000000 sec  
 T83: 0.0000000 sec  
 T84: 0.0000000 sec  
 T85: 0.0000000 sec  
 T86: 0.0000000 sec  
 T87: 0.0000000 sec  
 T88: 0.0000000 sec  
 T89: 0.0000000 sec  
 T90: 0.0000000 sec  
 T91: 0.0000000 sec  
 T92: 0.0000000 sec  
 T93: 0.0000000 sec  
 T94: 0.0000000 sec  
 T95: 0.0000000 sec  
 T96: 0.0000000 sec  
 T97: 0.0000000 sec  
 T98: 0.0000000 sec  
 T99: 0.0000000 sec  
 T100: 0.0000000 sec

# Z-restored spin-echo 13C spectrum with 1H decoupling

Current Data Parameters  
 USER Khowatt1  
 NAME RMH-111-709-1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Time 21.133  
 INSTRUM spect  
 PULPROG zgpg30  
 TO FIDPROC  
 SOLVENT CDCl3  
 NS 8  
 DS 4  
 SWH 30803.401 Hz  
 FIDRES 0.462308 Hz  
 AQ 1.1425654 sec  
 RG 10380.4 Hz  
 DW 16.500 usec  
 DE 6.00 usec  
 DI 0.25000000 sec  
 d11 0.00000000 sec  
 d12 0.00000000 sec  
 d17 0.00000000 sec  
 ACQRES1 0.00000000 sec  
 ACQRES2 0.00000000 sec  
 ACQRES3 0.00000000 sec  
 NUC1 13C  
 P1 14.00 usec  
 P12 2000.00 usec  
 P20 500.00 usec  
 P21 120.00 dB  
 SFO1 125.7942548 MHz  
 SFO2 1.55 GHz  
 SFO3 1.55 GHz  
 SF0M01 Cp66,0,5,20,1  
 SF0M02 Cp66,0,5,20,1  
 SF0F02 0.00 Hz  
 SF0F04 0.00 Hz  
 CPDPRG2 waltz16  
 RF2 100.00 usec  
 RF22 1.40 dB  
 PL12 500.212011 MHz  
 SFO2 500.212011 MHz  
 ===== GRADIENT CHANNEL =====  
 GPMW1 STEE100  
 GPC1 0.00 V  
 GPC2 0.00 V  
 GPC3 0.00 V  
 GPC4 0.00 V  
 GPC5 0.00 V  
 GPC6 0.00 V  
 GPC7 50.00 V  
 GPC8 50.00 usec  
 GPC9 100.00 usec  
 F2 - Processing parameters  
 SI 32768  
 SF 125.760122 MHz  
 WDW no  
 SSB 0  
 GB 0  
 PC 2.00  
 ID WDR p1/cr parameters  
 CX 22.80 cm  
 CY 15.45 cm  
 F1 2.00456 EPM  
 F2 -10.287 EPM  
 F2 -1293.96 Hz  
 F2 1293.96 Hz  
 EPCW 1324.10931 EPCW



1H spectrum



Current Data Parameters  
 USER khewitt1  
 NAME TATV181F2  
 EXPNO 2  
 PROCNO 1

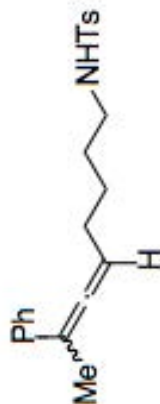
F2 - Acquisition Parameters  
 Date\_ 20201116  
 Time 10.32  
 INSTRUM gn500  
 PROBHD 5 mm broadband  
 PULPROG zg30  
 TD 81728  
 SOLVENT CDCl3T  
 NS 8  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.098043 Hz  
 AQ 5.0998774 sec  
 RG 812.7  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 MCREST 0.00000000 sec  
 MCWRK 0.01500000 sec

==== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -6.00 dB  
 SF01 498.7534913 MHz

F2 - Processing parameters  
 SI 65536  
 SF 498.7500352 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

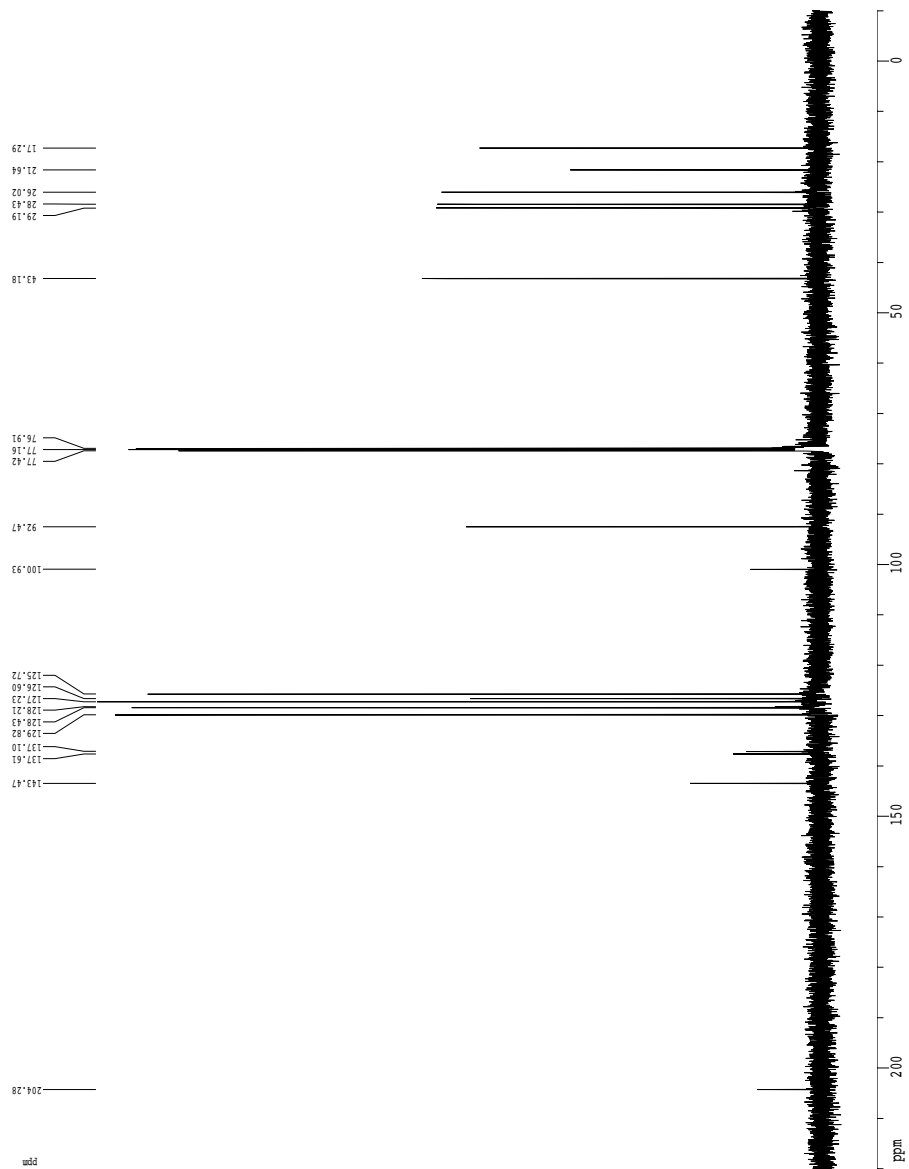
ID NMR plot parameters  
 CX 20.00 cm  
 CY 12.50 cm  
 FLP 9.000 ppm  
 F1 4488.75 Hz  
 F2P -0.500 ppm  
 F2 -249.38 Hz  
 PPMCM 0.47500 ppm/cm  
 HZCM 236.90627 Hz/cm





<sup>13</sup>C spectrum with <sup>1</sup>H decoupling

4.25



Current Data Parameters  
 USER Knewitt1  
 NAME TR1V187F2  
 EXPNO 3  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20201116  
 Time\_ 10.34  
 INSTRUM gn500  
 PROBHD 5 mm broadband  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 616  
 DS 4  
 SWH 30303.031 Hz  
 FIDRES 0.462388 Hz  
 AQC 1.0613940 sec  
 RG 4096  
 DW 16.500 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.25000000 sec  
 d11 0.03000000 sec  
 MCREST 0.00000000 sec  
 MCWRRK 0.01500000 sec

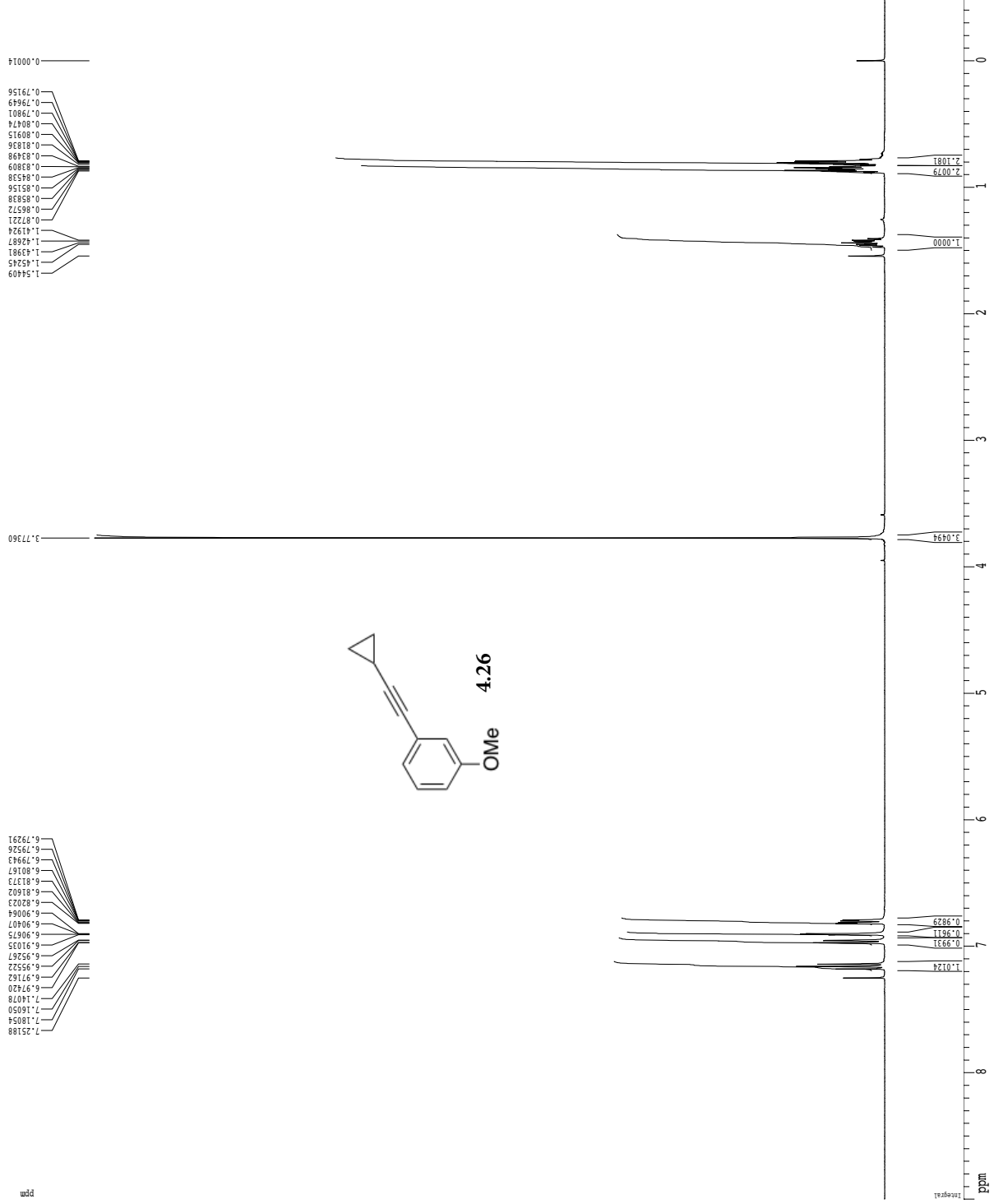
===== CHANNEL f1 =====  
 NUC1 <sup>13</sup>C  
 P1 14.20 usec  
 PL1 -6.00 dB  
 SFO1 125.4245824 MHz

===== CHANNEL f2 =====  
 CDPRG2 wait216  
 NUC2 <sup>1</sup>H  
 PCPD2 80.00 usec  
 PL2 -6.00 dB  
 PL12 12.30 dB  
 SFO2 498.7524937 MHz

F2 - Processing parameters  
 SI 65536  
 SF 125.4107762 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 2.00

ID NMR plot parameters  
 CX 20.00 cm  
 CY 12.50 cm  
 F1P 220.000 ppm  
 F1 27590.37 Hz  
 F2P -10.000 ppm  
 F2 -1254.11 Hz  
 PPMCM 11.50000 ppm/cm  
 HZCM 1442.22388 Hz/cm

1H spectrum



Current Data Parameters  
 USER: hsbane  
 NAME: 20210105check  
 EXNO: 1  
 PROCNO: 1

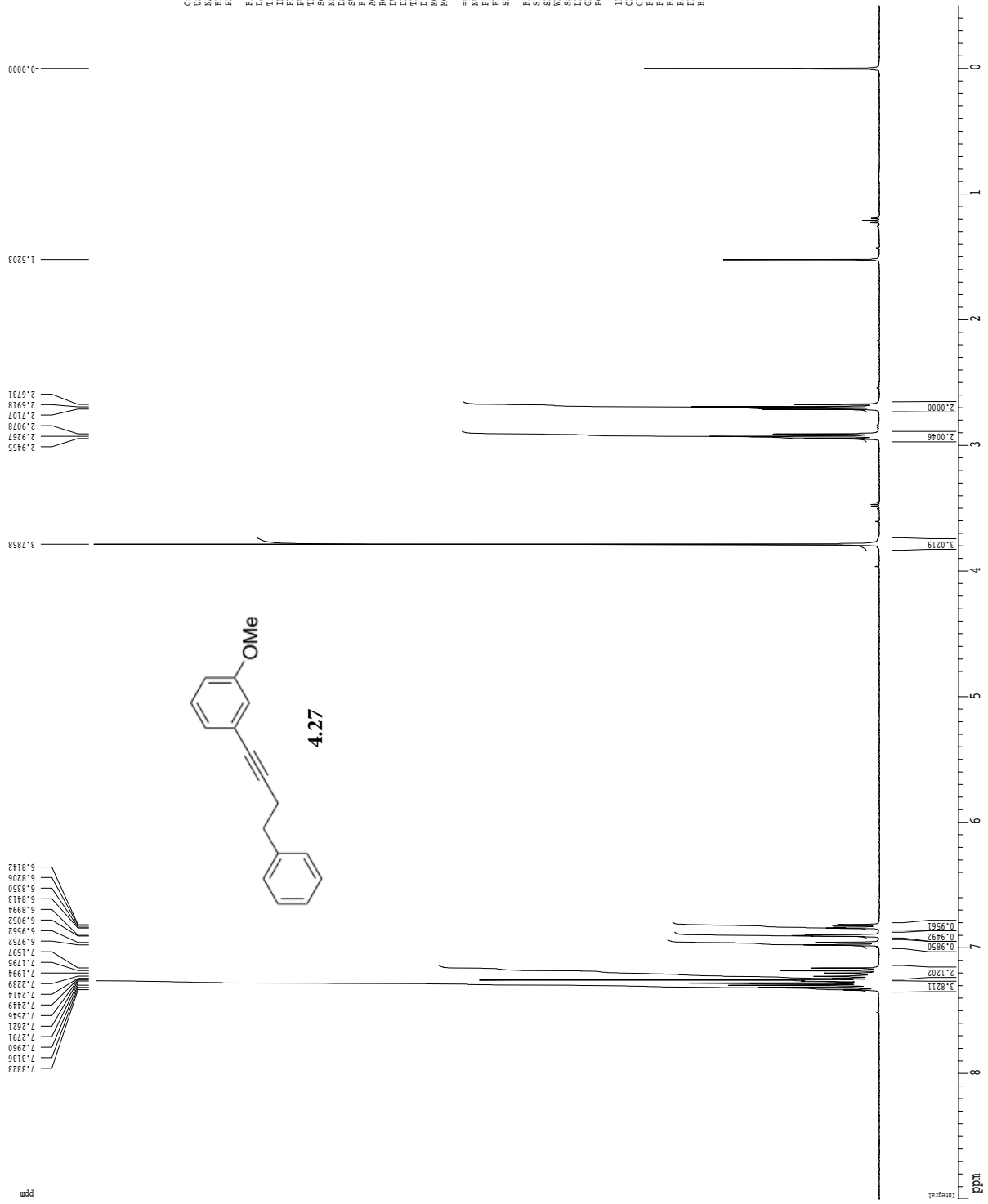
F2 - Acquisition Parameters  
 Date\_: 20200824  
 Time: 8:58  
 PROBNM: 20210105  
 PROCNO: 5  
 PULPROG: zgpg30  
 TD: 65536  
 SOLVENT: CDCl3  
 DS: 2  
 SWH: 6410.256 Hz  
 FIDRES: 0.09781 Hz  
 AQ: 5.11857 sec  
 RG: 327.8  
 DW: 78.000 usec  
 DE: 4.50 usec  
 TE: 297.2 K  
 T1: 0.100000 sec  
 T2: 0.000000 sec  
 T3: 0.000000 sec  
 MCHRES: 0.000000 Hz  
 MSRES: 0.000000 Hz  
 AXCORE: 0.000000 sec

===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -1.10 dB  
 SFO1: 400.1326009 MHz

F2 - Processing Parameters  
 SI: 65536  
 SF: 400.1300249 MHz  
 WDW: no  
 GB: 0.00 Hz  
 PC: 2.00

1D NMR File Parameters  
 CY: 22.80 cm  
 CX: 15.00 cm  
 FIP: 9.000 ppm  
 F1: 360.11 Hz  
 F2: -200.00 Hz  
 PPRM1: 0.41667 ppm/cm  
 HZCM: 166.72086 Hz/cm

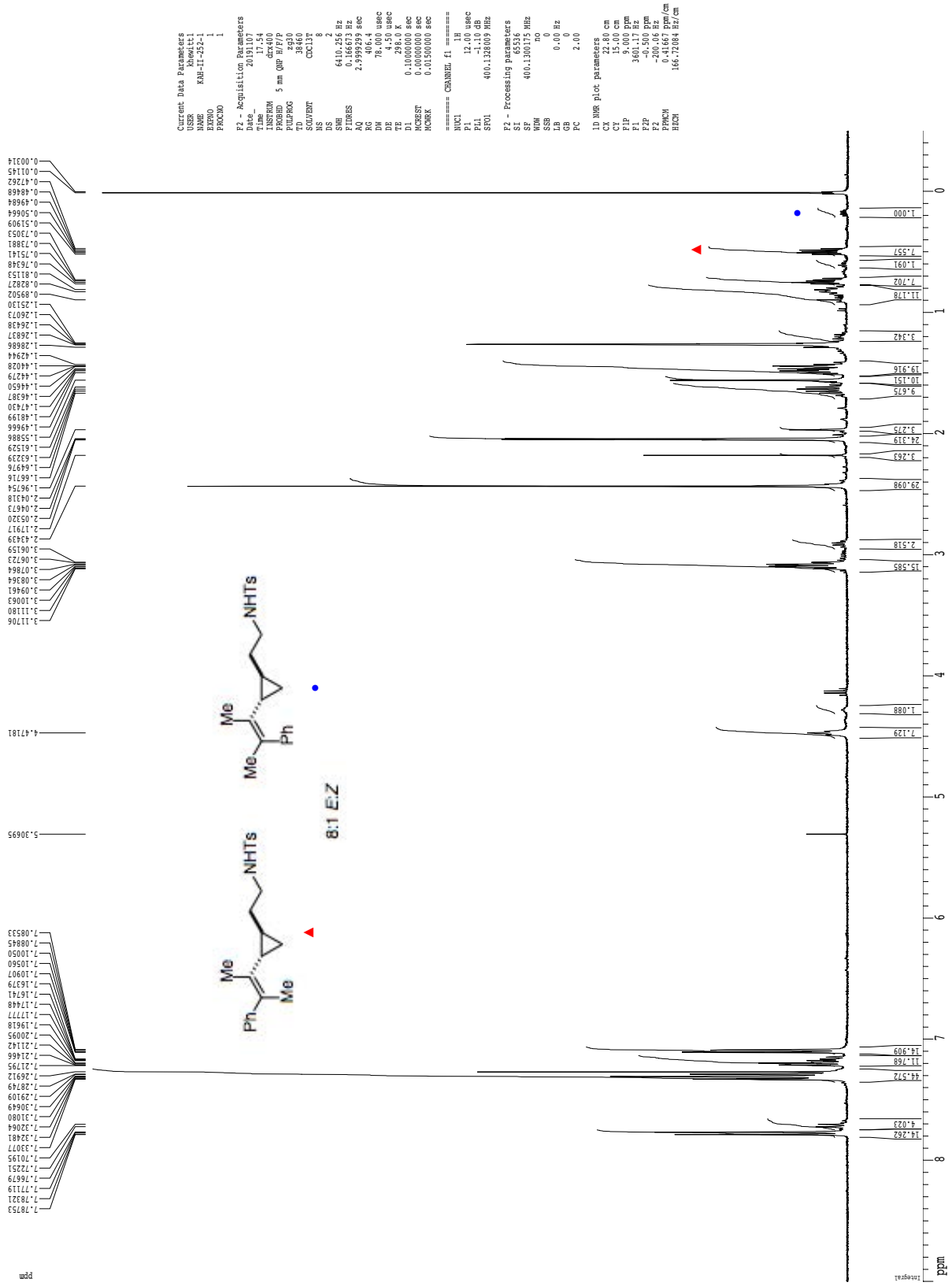
1H spectrum



Current Data Parameters  
 USER: mlr1000  
 NAME: NH-1-62-column  
 EXPO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20201003  
 Time: 14.26  
 INSTRUM: spect  
 PROBHD: 5 mm QNP HET  
 PULPROG: zgpg30  
 TD: 65536  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 400.130234 MHz  
 SHF: 6410.256 Hz  
 FIDRES: 0.09781 Hz  
 AQ: 5.11857 sec  
 RG: 327.5  
 DW: 78.000 usec  
 DE: 4.50 usec  
 TE: 298.2 K  
 T1: 0.100000 sec  
 T2: 0.000000 sec  
 T3: 0.000000 sec  
 MCHRES: 0.0050000 sec  
 AQCRES: 0.0050000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -1.10 dB  
 SFO1: 400.132809 MHz  
 F2 - Processing parameters  
 SI: 65536  
 SF: 400.130234 MHz  
 WHW: 9000 Hz  
 LB: 0.30 Hz  
 GB: 0  
 PC: 2.00  
 ID: NMR file parameters  
 CP: 22.80 cm  
 CY: 15.00 cm  
 FIP: 9.000 ppm  
 FL: 360.11 Hz  
 F2: 400.130234 MHz  
 F3: -2000.00 Hz  
 PPMCH: 0.41667 ppm/cm  
 HZCH: 166.72086 Hz/cm



1H spectrum



Current Data Parameters  
 USER KAWAII  
 NAME KAN-11-232-1  
 EXNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20191107  
 Time 17.54  
 PROBN 1  
 PROCNO 5 mm QNP 1H/1  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 DS 9  
 SFO 6410.258 Hz  
 FIDRES 0.166672 Hz  
 AQ 2.3398239 sec  
 RG 660  
 DW 78.000 usec  
 DE 4.50 usec  
 TE 298.2 K  
 T1 0.1000000 sec  
 T2 0.0000000 sec  
 T3 0.0000000 sec  
 MCRST 0.0150000 sec  
 MCRCK 0.0150000 sec

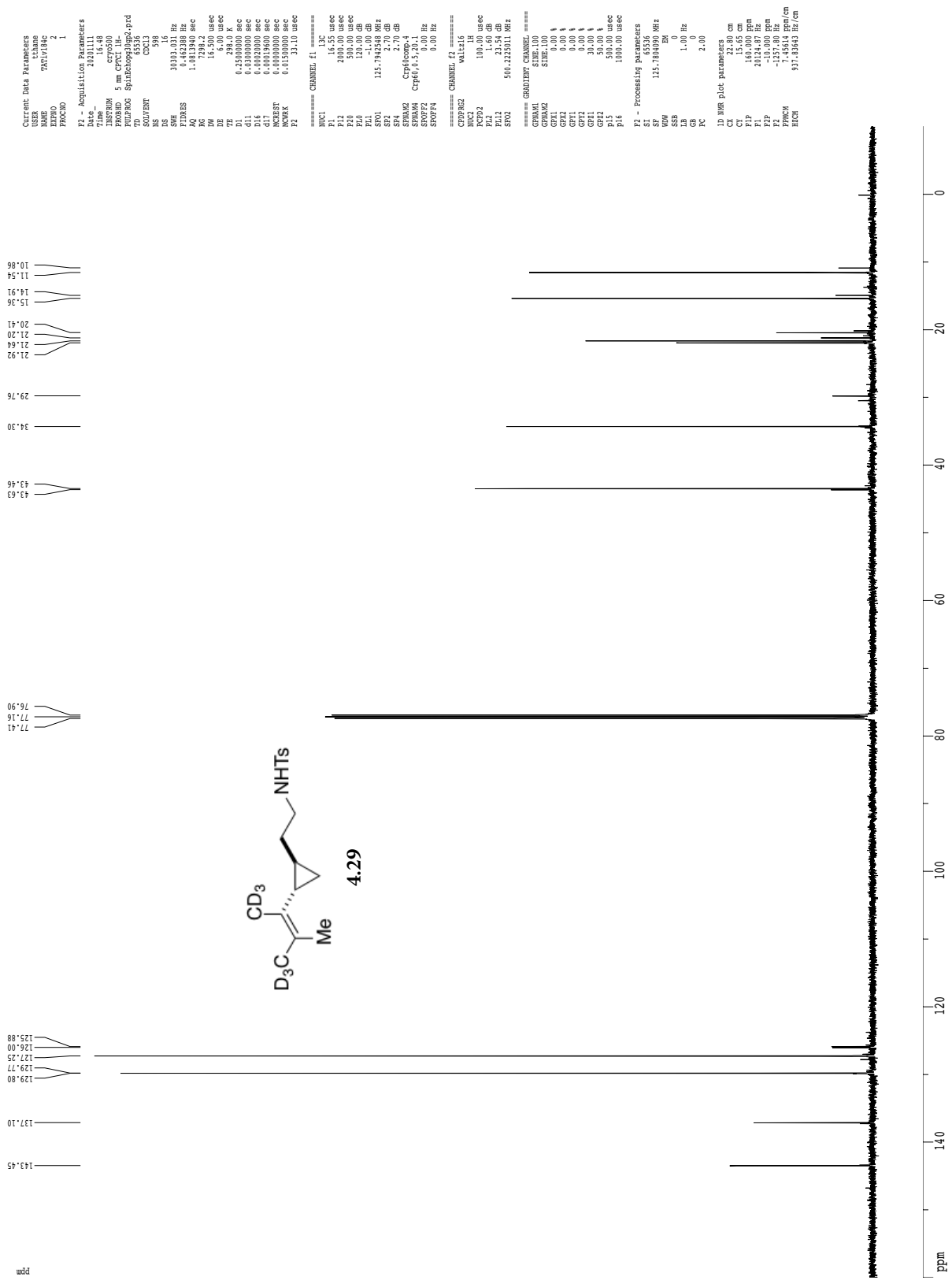
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.1328009 MHz

F2 - Processing Parameters  
 SI 65536  
 SF 400.1300175 MHz  
 NDM no  
 LB 0.00 Hz  
 GB 0  
 PC 2.00

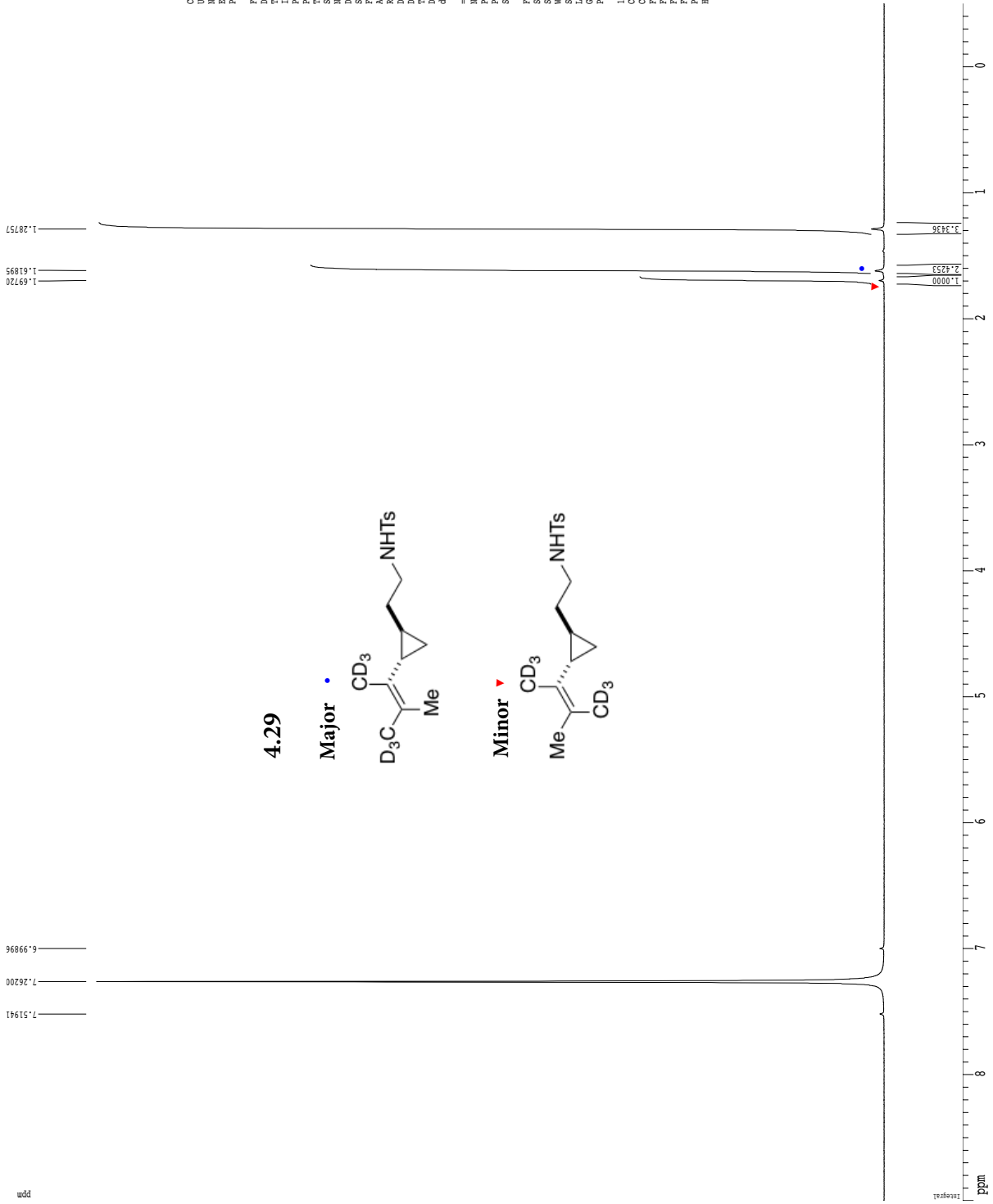
DJ MR Parameters  
 CY 22.80 cm  
 CV 15.00 cm  
 FIP 9.000 ppm  
 FL 3600.17 Hz  
 F2 200.000 MHz  
 F3 200.000 MHz  
 PPMCH 0.41667 ppm/cm  
 HZCH 166.72084 Hz/cm



Z-restored spin-echo 13C spectrum with 1H decoupling

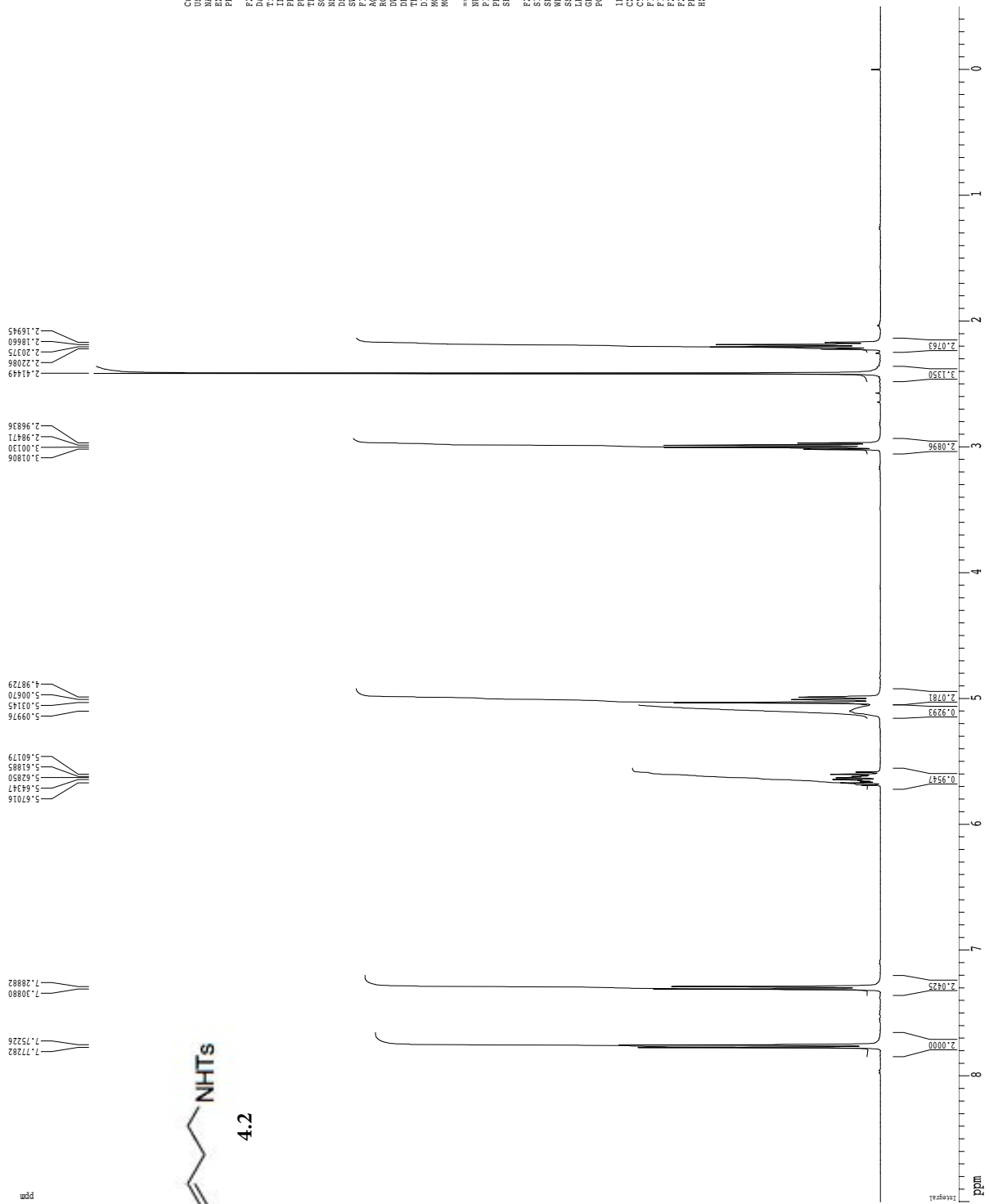


2H spectrum (measure via lock channel without changing any cables)





1H spectrum



Current Data Parameters  
 USER kshel11  
 NAME KNE-1-102E  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20180922  
 Time 12.03  
 INSTRUM spect  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 DS 2  
 SWH 6410.256 Hz  
 FIDRES 0.166673 Hz  
 AQ 2.999929 sec  
 RG 327.5  
 DW 78.000 ns  
 DE 4.50 ns  
 TE 297.2 K  
 T1 1.000000 sec  
 T2 0.000000 sec  
 T3 0.000000 sec  
 WPCORE 0.01500000 sec

===== CHANNEL f1 =====  
 NU1 12.00 MHz  
 PL1 -1.10 dB  
 SFO1 400.1328009 MHz

F2 - Processing parameters  
 SI 65536  
 SF 400.1300086 MHz  
 WDW no  
 SSB no  
 LB 0.00 Hz  
 GB 0  
 PC 2.00

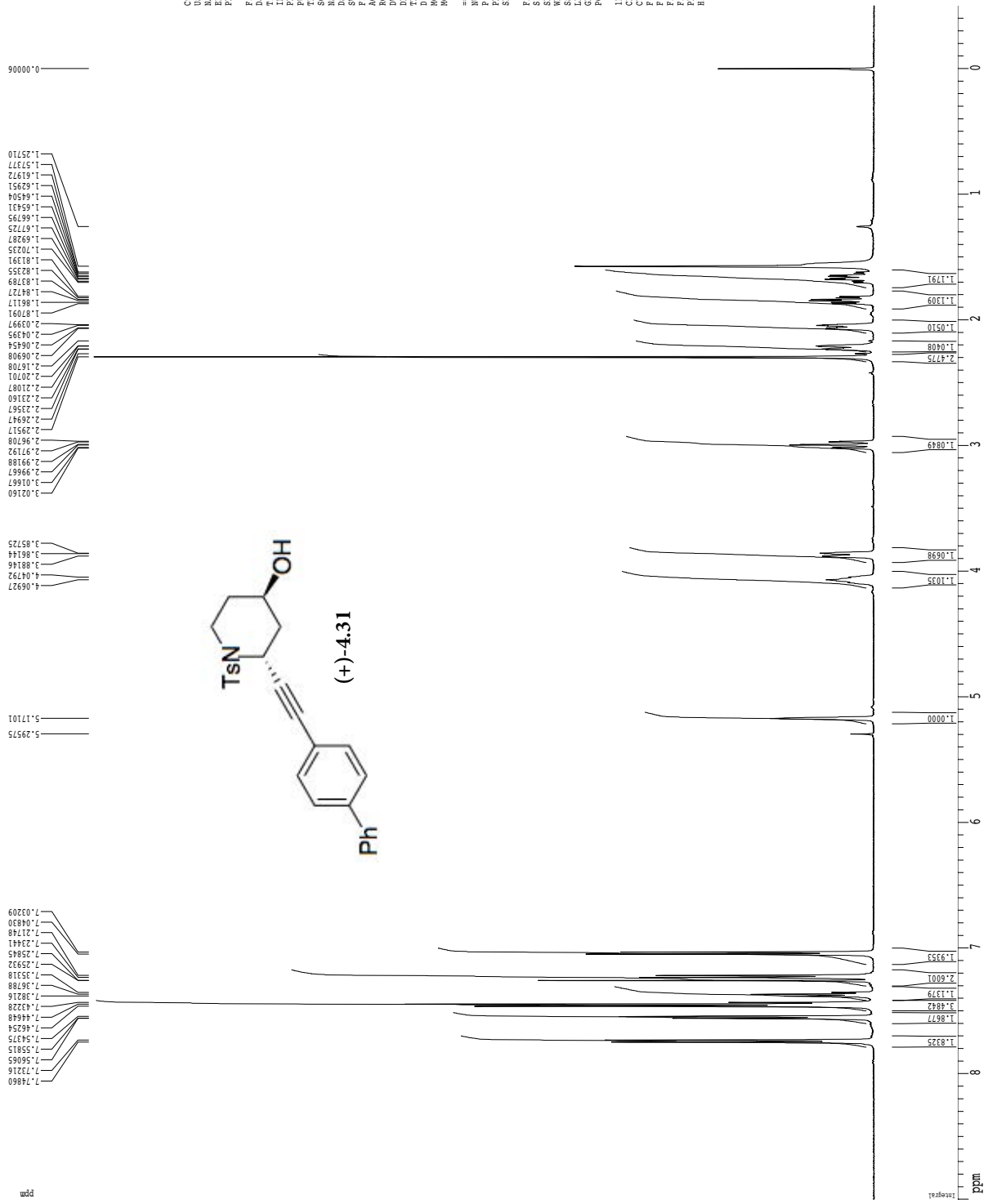
F3 - MMR plot parameters  
 CY 22.80 cm  
 CX 15.00 cm  
 F1P 9.000 ppm  
 F1 360.17 Hz  
 F2 49.00 Hz  
 F2 49.00 Hz  
 FWHM 0.41667 ppm/cm  
 HZCN 166.72084 Hz/cm

1H spectrum



Current Data Parameters  
USER: Komettt  
NAME: BLA-IV-112  
EXNO: 1  
PROCNO: 1  
P2 - Acquisition Parameters  
Date\_: 20190218  
Time: 10.51  
PROBHD: 5 mm QNP 1H/1  
PULPROG: zg30  
TD: 38460  
SOLVENT: CDCl3  
DS: 2  
SFO: 400.132809 MHz  
SHE: 6410.256 Hz  
FIDRES: 0.166672 Hz  
AQ: 2.399229 sec  
RG: 78.000  
DE: 4.50 usec  
TE: 298.2 K  
WALTZ16: 0.100000 sec  
NUC1: 13C  
NUC2: 1H  
PC: 2.00  
P1: 12.00 usec  
P12: -1.10 dB  
SFO1: 400.132809 MHz  
P2 - Processing Parameters  
SI: 65536  
SF: 400.130022 MHz  
WDW: no  
SSB: 0.00 Hz  
GB: 0  
PC: 2.00  
ID: NMR file parameters  
CY: 22.80 cm  
CY: 15.00 cm  
FIP: 11.000 ppm  
FL: 4400.48 Hz  
F2: 200.132809 MHz  
F2: -200.00 Hz  
P1P1CM: 0.50439 ppm/cm  
HZCM: 201.81998 Hz/cm

1H spectrum



Current Data Parameters  
 USER KAWAII  
 NAME KAN-TY-031-2  
 EXNO 1  
 PROCNO 1

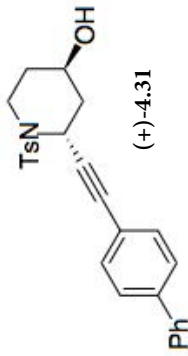
F2 - Acquisition Parameters  
 Date\_ 20201119  
 Time 17:37  
 PROBNM 07201119  
 PULPROG zgpg30  
 TD 48074  
 SOLVENT CDCl3  
 DS 9  
 SFO 8012.820 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.3998677 sec  
 RG 62.400 usec  
 DE 6.00 usec  
 TE 298.2 K  
 T1 1.000000 sec  
 T2 0.100000 sec  
 T3 0.100000 sec  
 TMRK 0.0150000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 9.75 usec  
 PL1 1.66 dB  
 SFO1 500.2235015 MHz

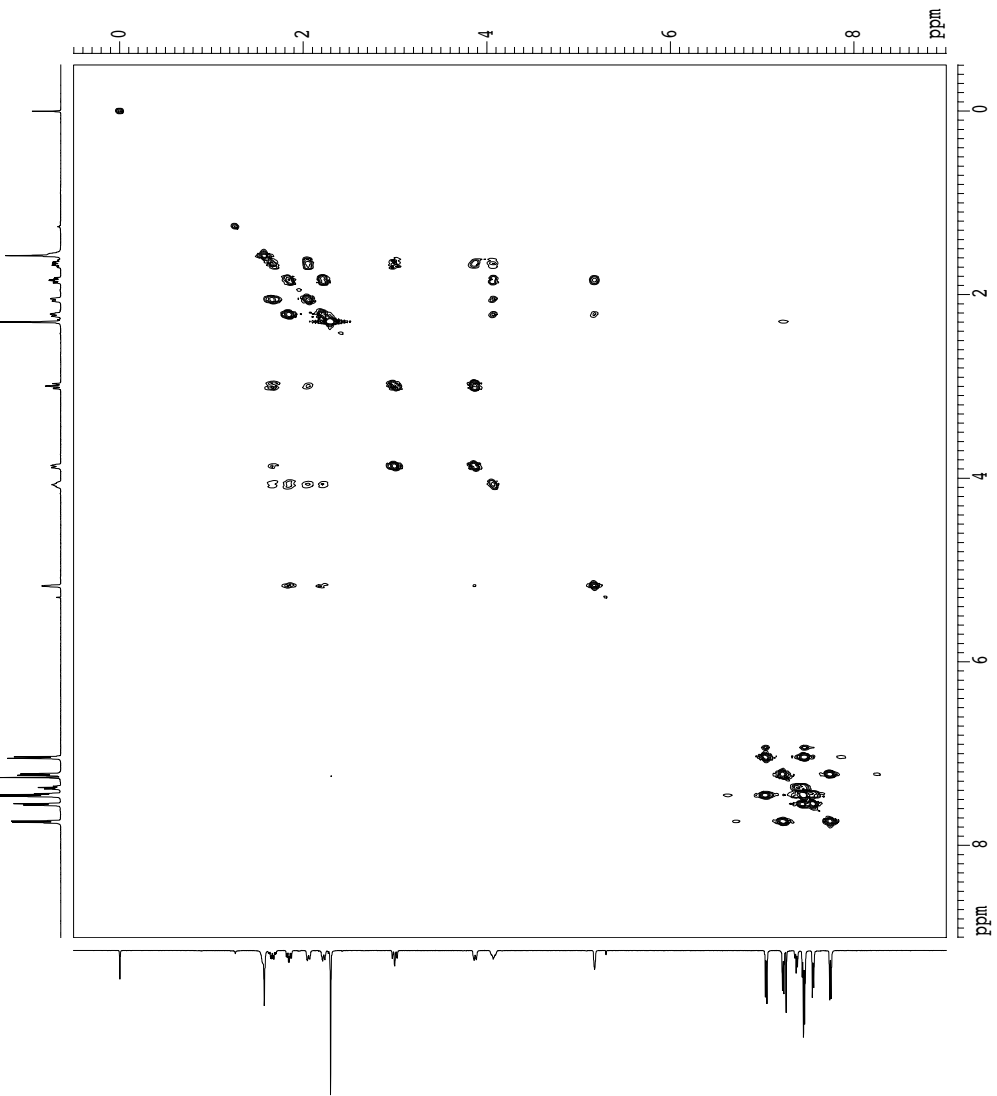
F2 - Processing Parameters  
 SI 65536  
 SF 500.2200319 MHz  
 WDW no  
 GB 0  
 PC 1.00

D0 NMR PLOT Parameters  
 CY 22.86 cm  
 CX 15.06 cm  
 FIP 9.000 ppm  
 F1 4500.938 Hz  
 F2 250.11 Hz  
 PPMX 0.41667 ppm/cm  
 HZCM 206.44502 Hz/cm





gcosy60



Current Data Parameters  
 USER khevatli  
 NAME KAN-19-131-2  
 PROCD 1

F2 - Acquisition Parameters

Date 2/10/10  
 Time 14:01  
 INSTRM spect  
 PULPROG zgpg30  
 TD 32768  
 SFO1 498.131313  
 SOLVENT CDCl3  
 NS 16  
 SH 812.820 Hz  
 F2 498.131313 MHz  
 AQ 0.1278452 sec  
 RG 5180.4  
 RW 62.00 usec  
 TE 298.0 K  
 DE 0.0000300 sec  
 d13 0.0000300 sec  
 d14 0.0000300 sec  
 DI 6  
 D16 0.0020000 sec  
 INU 0.0013480 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*

NUC1 1H  
 P1 12.00 usec  
 PL1 -6.00 dB  
 SFO1 498.131313 MHz

\*\*\*\*\* GRABED CHANNEL \*\*\*\*\*

CPDPRG1 SINE-100  
 GRABM 0.00 usec  
 GRABW 0.00 A  
 GPR2 0.00 A  
 GPR1 0.00 A  
 GPR2 0.00 A  
 GPR3 0.00 A  
 GPR4 17.00 A  
 P16 1000.00 usec

F1 - Acquisition Parameters

NUC1 13C  
 P1 12.00 usec  
 PL1 -6.00 dB  
 SFO1 498.131313 MHz  
 FIDPR 31.300075 Hz  
 SW 16.066 ppm  
 PHASE2 0°

F2 - Processing Parameters

SI 32768  
 SF 498.131313 MHz  
 WDW HANN  
 SSB 0  
 GB 0  
 PC 1.00

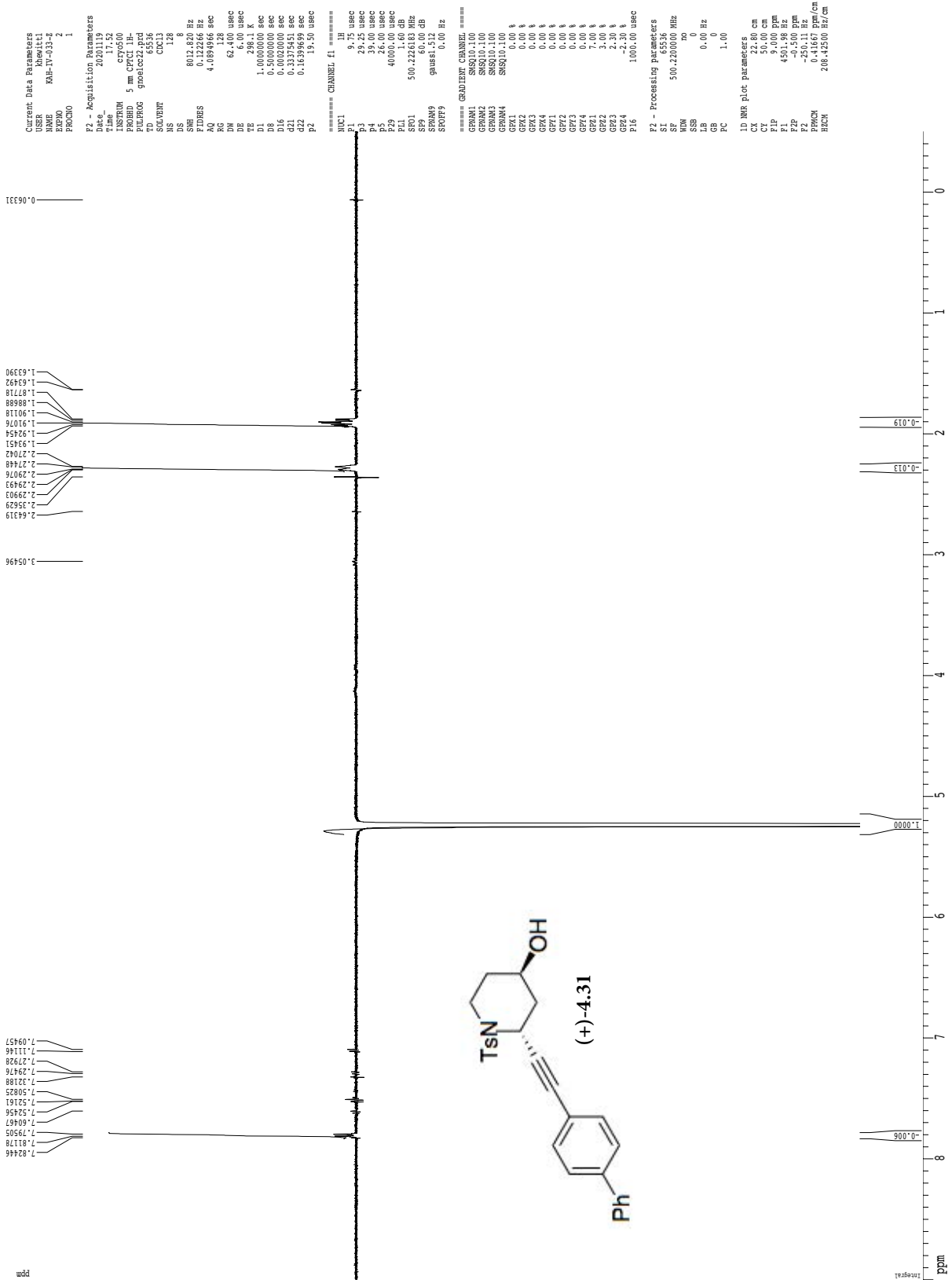
F1 - Processing Parameters

SI 1024  
 SF 498.131313 MHz  
 WDW HANN  
 SSB 0  
 GB 0  
 PC 1.00

2D NMR plot parameters

CT 15.00 cm  
 C1 15.00 cm  
 F2F1O 9.006 ppm  
 F2F1 49.935 Hz  
 F2F1O -254.24 Hz  
 F1F1O 9.006 ppm  
 F1F1 49.935 Hz  
 F1F1O -4.532 ppm  
 F1F1 -254.24 Hz  
 F2F1PCW 0.63385 ppm/cm  
 F1F1PCW 0.63385 ppm/cm  
 F1F1PCW 316.13806 Hz/cm

gnoe



gnoe

ppm

Current Data Parameters  
USER Khevitli  
NAME KAH-IV-03-2  
EXNO 7  
PROCNO 1

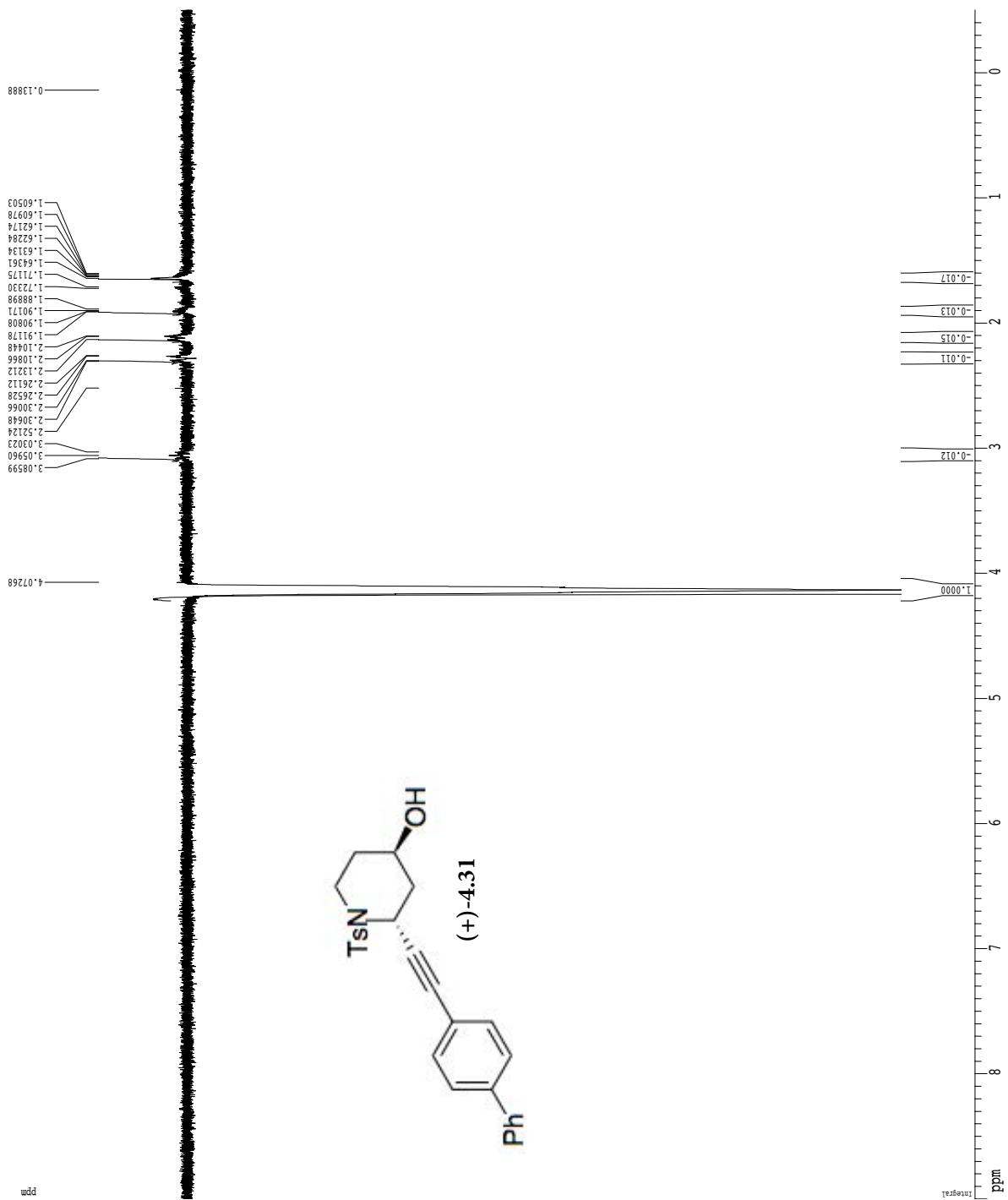
F2 - Acquisition Parameters  
Date\_ 20/01/20  
Time\_ 16:50  
INSTRUM spect  
PROBHD 5 mm broadband  
PULPROG zgpg30c22.prd  
TD 65536  
SOLVENT CDCl3  
NS 128  
DS 8  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894966 sec  
RG 5160.6  
DM 62.400 usec  
DE 6.00 usec  
TE 298.0 K  
D1 1.00000000 sec  
D2 5.00000000 sec  
D16 0.00020000 sec  
d21 0.33374399 sec  
d22 0.16399699 sec  
p2 24.00 usec

==== CHANNEL f1 =====  
NUC1 <sup>1</sup>H  
P1 12.00 usec  
P3 36.00 usec  
P4 48.00 usec  
P5 32.00 usec  
P29 40000.00 usec  
PL1 -6.00 dB  
SFO1 498.7520000 MHz  
SFO2 313.0000000 MHz  
SFO3 156.5000000 MHz  
SFO4 78.2500000 MHz  
SFO5 39.1250000 MHz  
SFO6 19.5625000 MHz  
SFO7 9.7812500 MHz  
SFO8 4.8906250 MHz  
SFO9 2.4453125 MHz  
SFO10 1.22265625 MHz

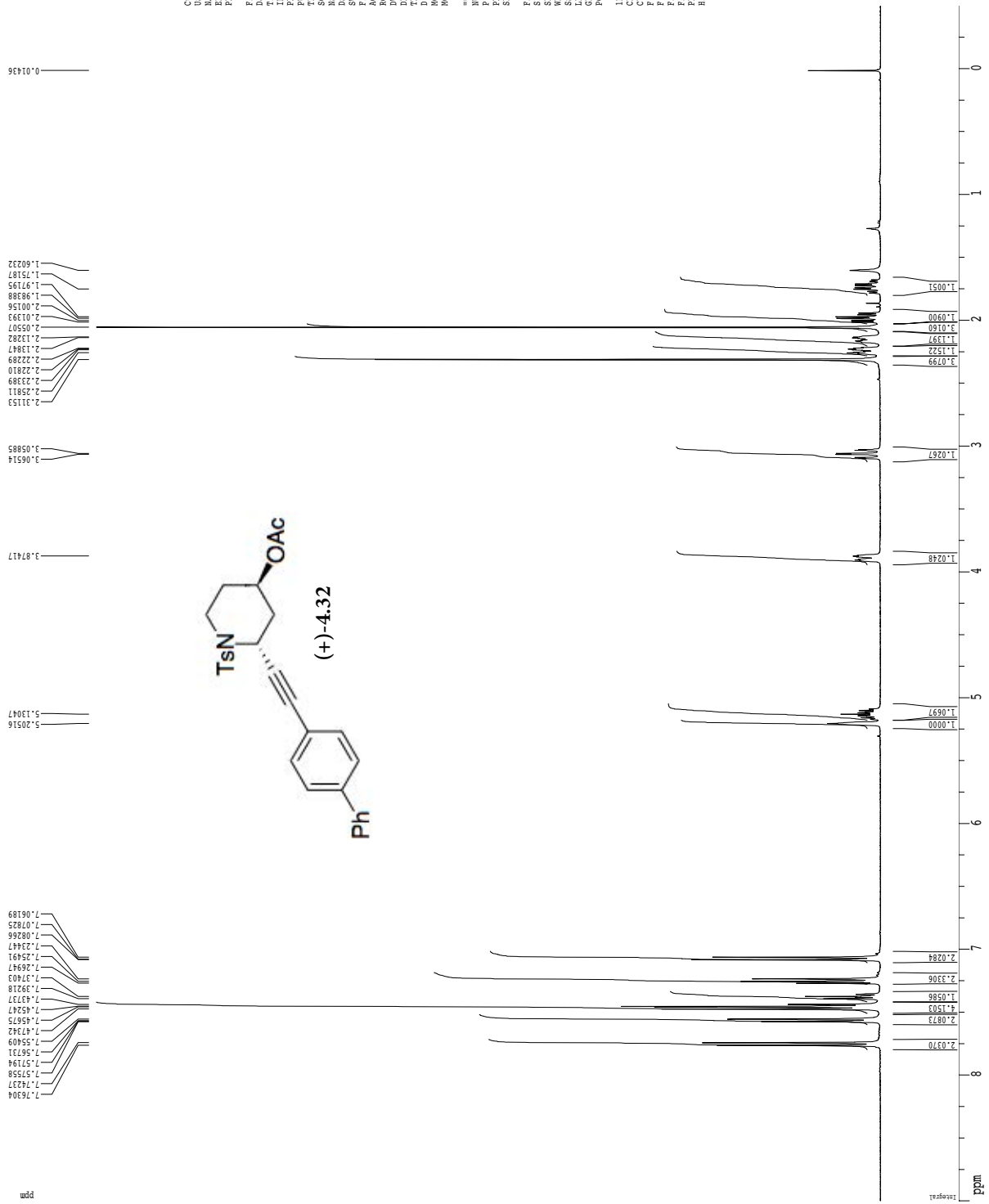
==== GRADIENT CHANNEL =====  
GPM1 sine,100  
GPM2 sine,100  
GPM3 sine,100  
GPM4 sine,100  
GPX1 0.00 \$  
GPX2 0.00 \$  
GPX3 0.00 \$  
GPX4 0.00 \$  
GPI1 0.00 \$  
GPI2 0.00 \$  
GPI3 0.00 \$  
GPI4 0.00 \$  
GPI5 0.00 \$  
GPI6 0.00 \$  
GPI7 0.00 \$  
GPI8 0.00 \$  
GPI9 0.00 \$  
GPI10 0.00 \$  
GPI11 0.00 \$  
GPI12 0.00 \$  
GPI13 0.00 \$  
GPI14 0.00 \$  
GPI15 0.00 \$  
GPI16 0.00 \$  
GPI17 0.00 \$  
GPI18 0.00 \$  
GPI19 0.00 \$  
GPI20 0.00 \$  
GPI21 0.00 \$  
GPI22 0.00 \$  
GPI23 0.00 \$  
GPI24 0.00 \$  
GPI25 0.00 \$  
GPI26 0.00 \$  
GPI27 0.00 \$  
GPI28 0.00 \$  
GPI29 0.00 \$  
GPI30 0.00 \$  
GPI31 0.00 \$  
GPI32 0.00 \$  
GPI33 0.00 \$  
GPI34 0.00 \$  
GPI35 0.00 \$  
GPI36 0.00 \$  
GPI37 0.00 \$  
GPI38 0.00 \$  
GPI39 0.00 \$  
GPI40 0.00 \$  
GPI41 0.00 \$  
GPI42 0.00 \$  
GPI43 0.00 \$  
GPI44 0.00 \$  
GPI45 0.00 \$  
GPI46 0.00 \$  
GPI47 0.00 \$  
GPI48 0.00 \$  
GPI49 0.00 \$  
GPI50 0.00 \$  
GPI51 0.00 \$  
GPI52 0.00 \$  
GPI53 0.00 \$  
GPI54 0.00 \$  
GPI55 0.00 \$  
GPI56 0.00 \$  
GPI57 0.00 \$  
GPI58 0.00 \$  
GPI59 0.00 \$  
GPI60 0.00 \$  
GPI61 0.00 \$  
GPI62 0.00 \$  
GPI63 0.00 \$  
GPI64 0.00 \$  
GPI65 0.00 \$  
GPI66 0.00 \$  
GPI67 0.00 \$  
GPI68 0.00 \$  
GPI69 0.00 \$  
GPI70 0.00 \$  
GPI71 0.00 \$  
GPI72 0.00 \$  
GPI73 0.00 \$  
GPI74 0.00 \$  
GPI75 0.00 \$  
GPI76 0.00 \$  
GPI77 0.00 \$  
GPI78 0.00 \$  
GPI79 0.00 \$  
GPI80 0.00 \$  
GPI81 0.00 \$  
GPI82 0.00 \$  
GPI83 0.00 \$  
GPI84 0.00 \$  
GPI85 0.00 \$  
GPI86 0.00 \$  
GPI87 0.00 \$  
GPI88 0.00 \$  
GPI89 0.00 \$  
GPI90 0.00 \$  
GPI91 0.00 \$  
GPI92 0.00 \$  
GPI93 0.00 \$  
GPI94 0.00 \$  
GPI95 0.00 \$  
GPI96 0.00 \$  
GPI97 0.00 \$  
GPI98 0.00 \$  
GPI99 0.00 \$  
GPI100 0.00 \$

F2 - Processing parameters  
SI 65536  
SF 498.7500000 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

ID\_NMR Plot parameters  
CX 20.00 cm  
CY 12.50 cm  
F1P 9.000 ppm  
F1 4488.75 Hz  
F2P -0.500 ppm  
F2 -249.38 Hz  
FPMCH 0.47500 ppm/cm  
HZCN 236.90625 Hz/cm



1H spectrum



Current Data Parameters  
 USER KAMELIT  
 NAME KAM-TY-035-2  
 EXNO 1  
 PROCD 1

F2 - Acquisition Parameters  
 Date\_ 20201121  
 Time 11:51  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/13  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 DS 9  
 SFO 6410.258 Hz  
 FIDRES 0.166673 Hz  
 AQ 2.339229 sec  
 RG 655.000  
 DW 78.000 usec  
 DE 4.50 usec  
 TE 298.2 K  
 T1 1.000000 sec  
 T2 0.000000 sec  
 T3 0.000000 sec  
 MCRBK 0.0150000 sec

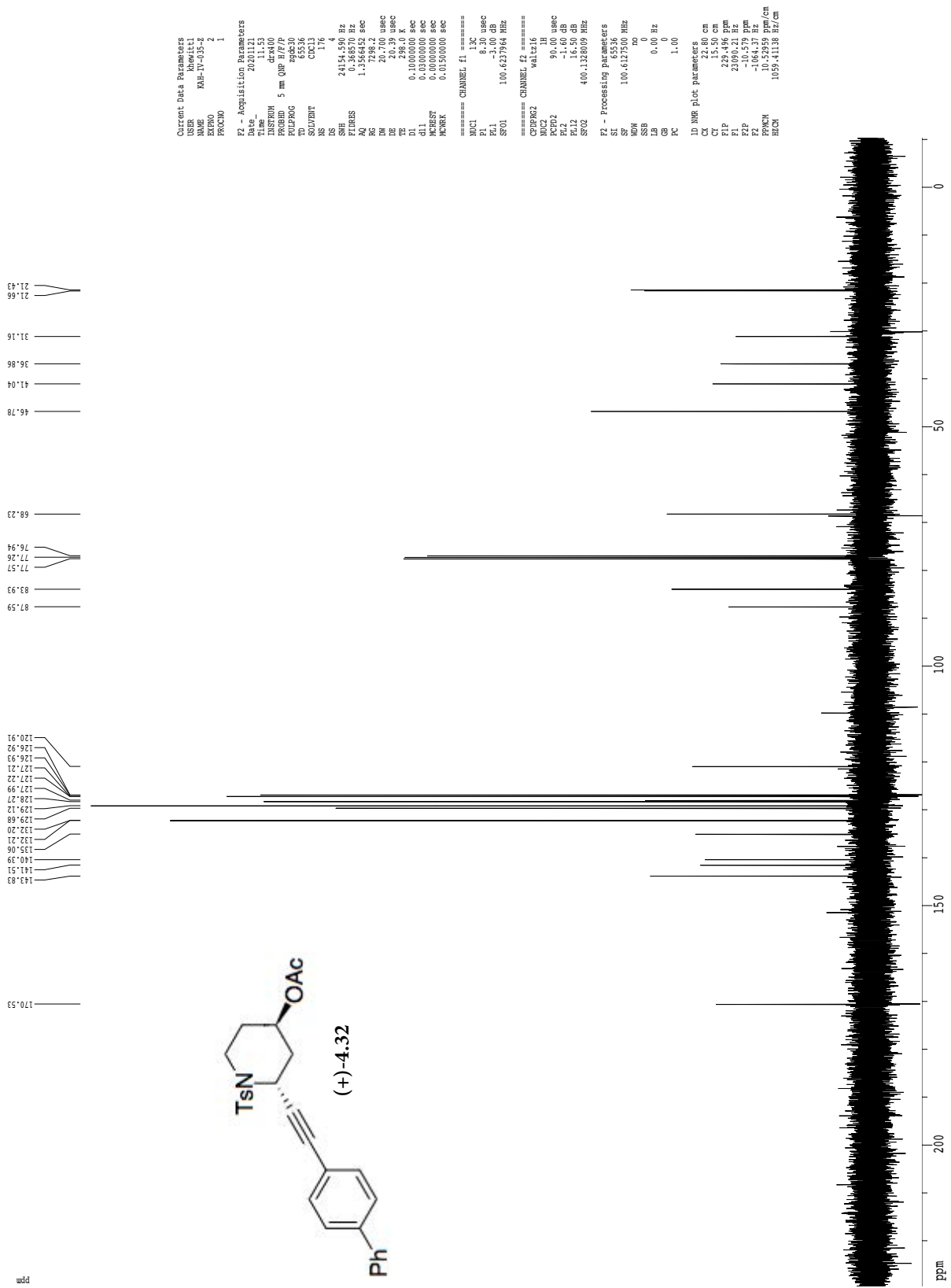
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 0.00 dB  
 SFO1 400.1328009 MHz

F2 - Processing Parameters  
 SI 65536  
 SF 400.1300175 MHz  
 WDW no  
 GB 0  
 PC 2.00

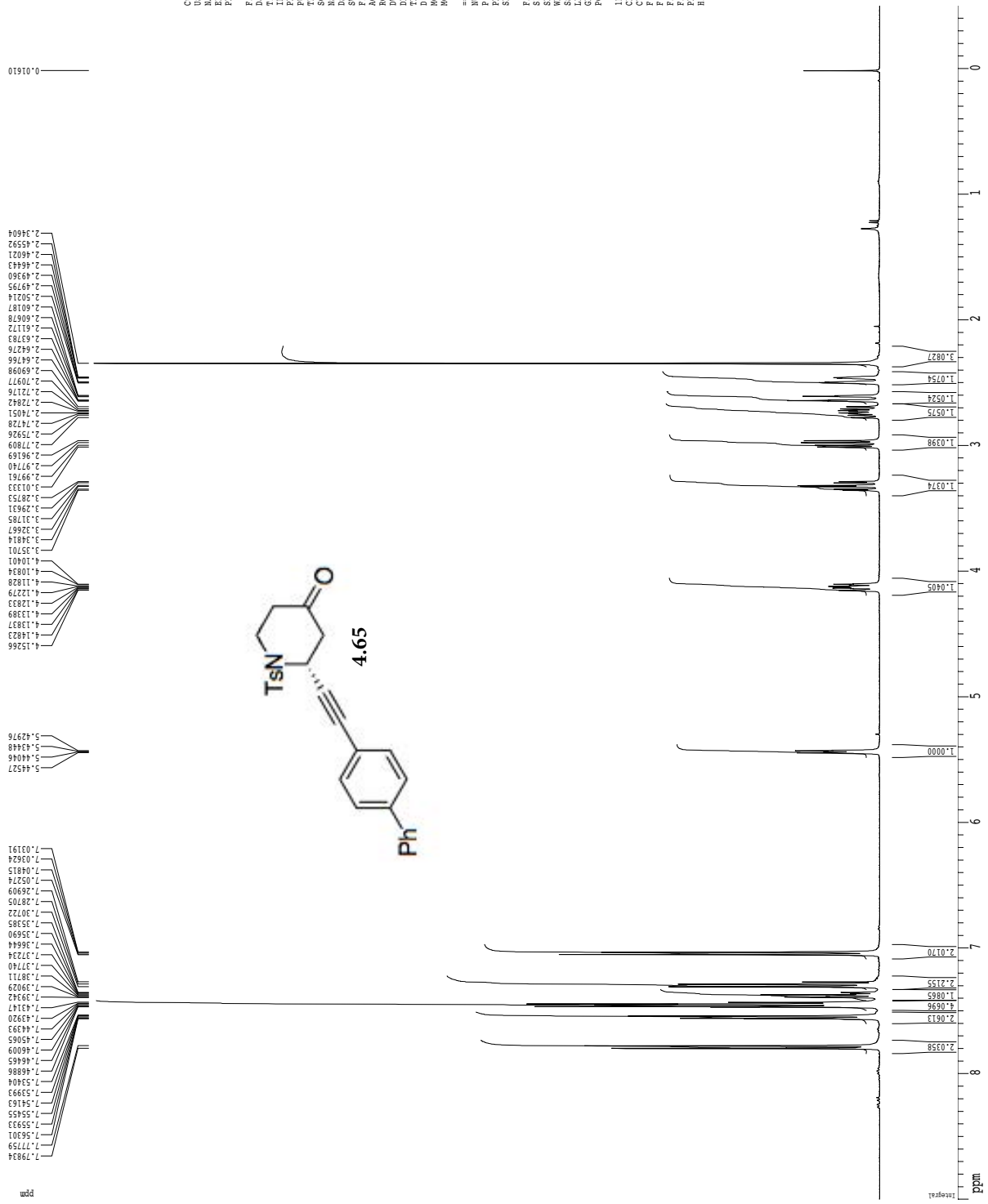
F3 NMR P1/QF Parameters  
 CY 22.80 cm  
 CV 15.00 cm  
 FIP 9.000 ppm  
 FL 3600.17 Hz  
 F2 -2000.00 Hz  
 F3 0.41667 ppm/cm  
 HZCM 166.72084 Hz/cm



13C spectrum with 1H decoupling



1H spectrum



Current Data Parameters  
 USER KRWETT  
 NAME KRN-TY-048-1  
 EXNO 1  
 PROCNO 1

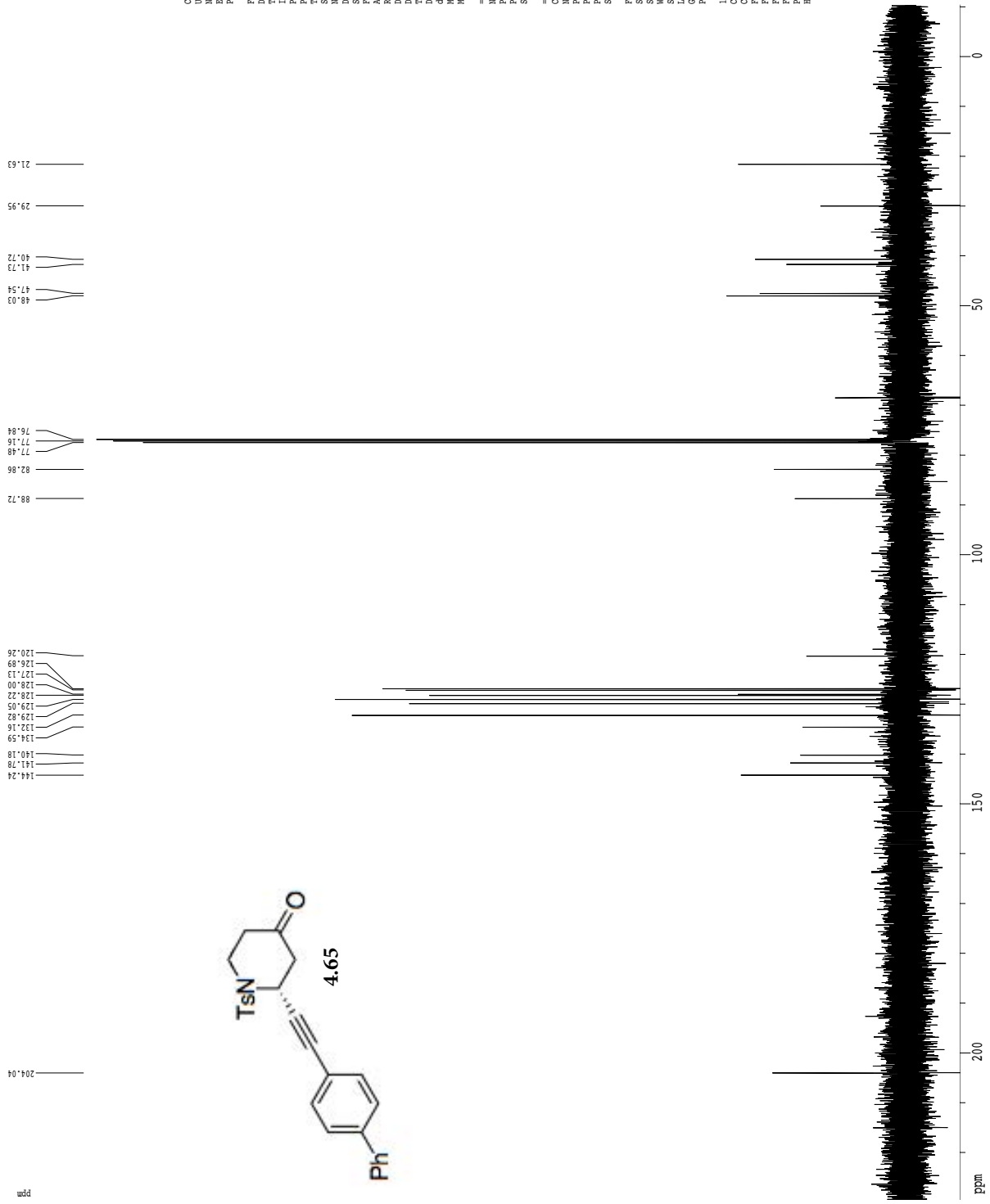
F2 - Acquisition Parameters  
 Date\_ 20201123  
 Time 14:08  
 PROBN 0010  
 PROCNO 5 mm QNP HET-7  
 PULPROG zg30  
 TD 38460  
 SOLVENT CDCl3  
 DS 9  
 SFO 5  
 SFR 6410.258 Hz  
 FIDRES 0.16667 Hz  
 AQ 2.399279 sec  
 RG 655  
 DD 78.000 usec  
 DE 4.50 usec  
 TE 298.2 K  
 T1 0.100000 sec  
 T2 0.000000 sec  
 T3 0.000000 sec  
 WDEXT 0.0150000 sec  
 WDSREK 0.0150000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -1.68 dB  
 SFO1 400.1328009 MHz

F2 - Processing Parameters  
 SI 65536  
 SF 400.1300175 MHz  
 WDM no  
 LB 0 Hz  
 GB 0 Hz  
 PC 2.00

D0 NMR P1.CE Parameters  
 C1 22.80 cm  
 C2 15.00 cm  
 F1P 9.000 ppm  
 F1 3600.17 Hz  
 F2 -2000.00 Hz  
 P1P1M 0.41667 ppm/cm  
 H1CM 166.72084 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters  
 USER: kmetz11  
 NAME: R01-IV-440-2  
 EXPNO: 2  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_ 20201116  
 Time 10.15  
 INSTRUM: cryo400  
 PULPROG: zgpg30  
 SFOREQ: 5 mm QNP 499.989 MHz  
 TD: 65536  
 SOLVENT: CDCl3  
 NS: 408  
 DS: 4  
 SWH: 24154.500 Hz  
 FIDRES: 0.368570 Hz  
 AQ: 1.3566452 sec  
 RG: 33004  
 DE: 2.71 usec  
 TE: 297.9 K  
 D1: 0.10000000 sec  
 d11: 0.03000000 sec  
 MCKEY: 0.00000000 sec  
 MCHP: 0.01000000 sec

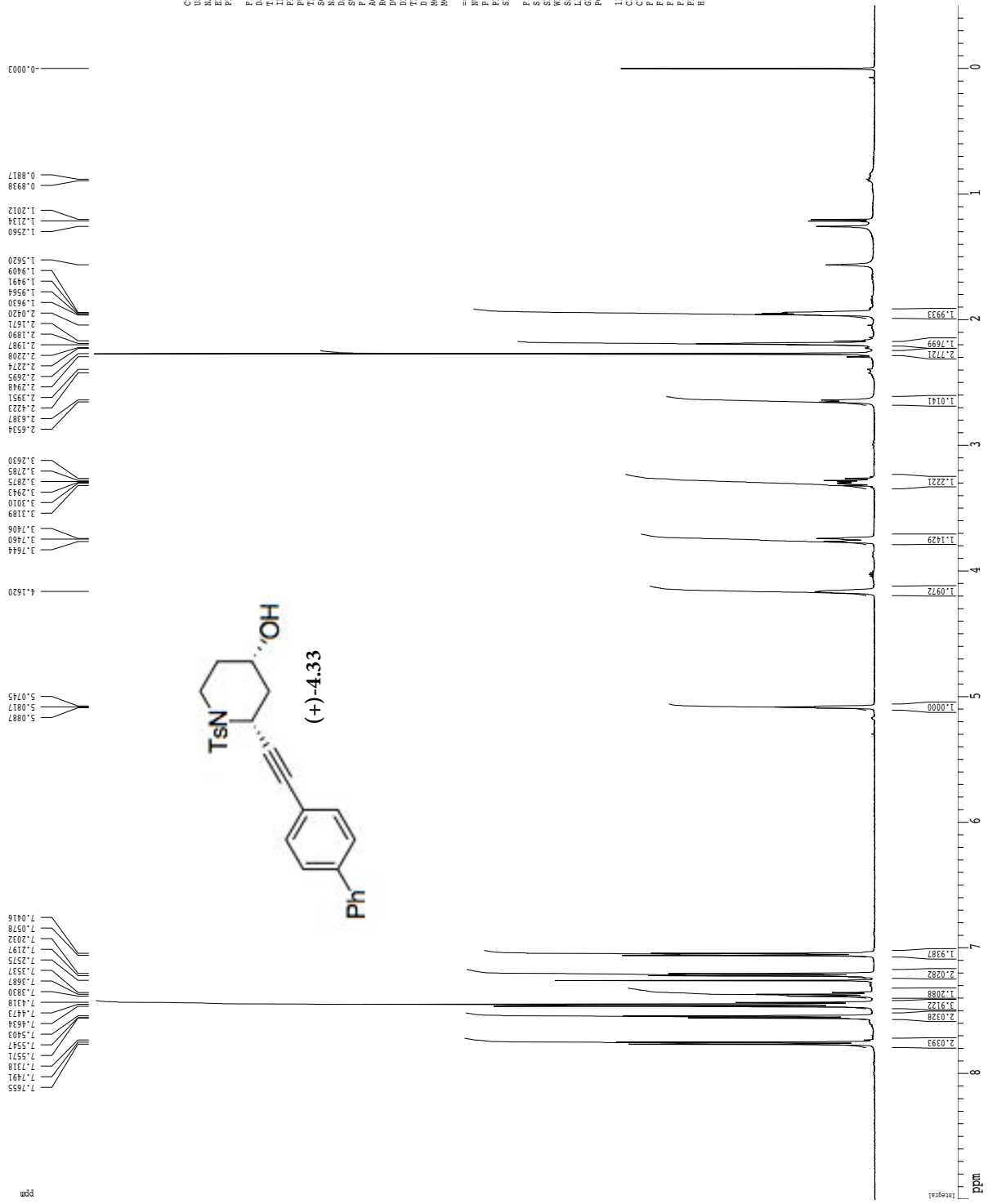
===== CHANNEL f1 =====  
 NUCL1: 13C  
 P1: 1.70 usec  
 PL1: -1.00 dB  
 SFO1: 100.6277664 MHz

===== CHANNEL f2 =====  
 NUCR2: 1H  
 PULPROG: waltz16  
 PCPD2: 90.00 usec  
 PL2: -1.10 dB  
 SFO2: 400.1526009 MHz

F2 - Processing parameters  
 SI: 65536  
 SF: 100.627580 MHz  
 SWH: 0  
 DS: 0  
 GB: 0.00 Hz  
 GB1: 0  
 GB2: 0  
 GB3: 0  
 PC: 1.00

1D NMR plot parameters  
 CX: 22.80 cm  
 CY: 15.50 cm  
 FIP: 22.496 ppm  
 FIDRES: 0.368570 Hz  
 F2: 400.1526009 MHz  
 F2F1: -1684.37 Hz  
 FWHM: 10.52959 ppm/cm  
 BR2CN: 1059.41150 Hz/cm

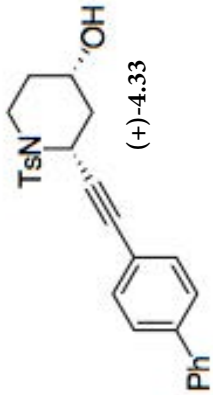
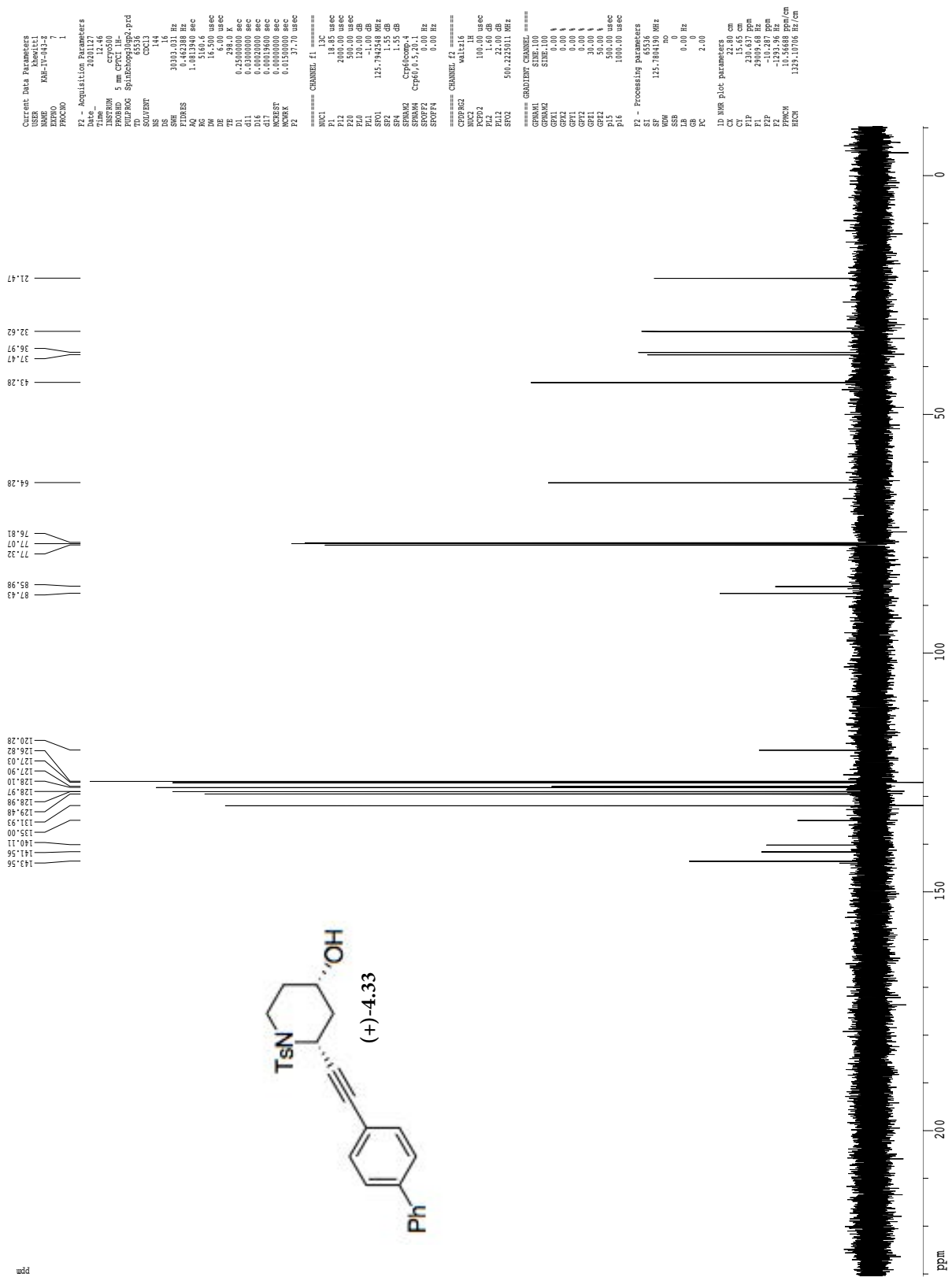
1H spectrum

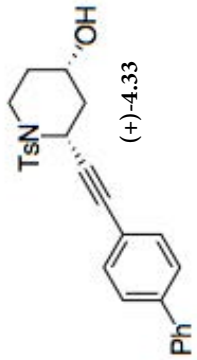


Current Data Parameters  
 USER KRM-TY-03-2  
 NAME KRM-TY-03-2  
 EKWO 3  
 PROCD 1

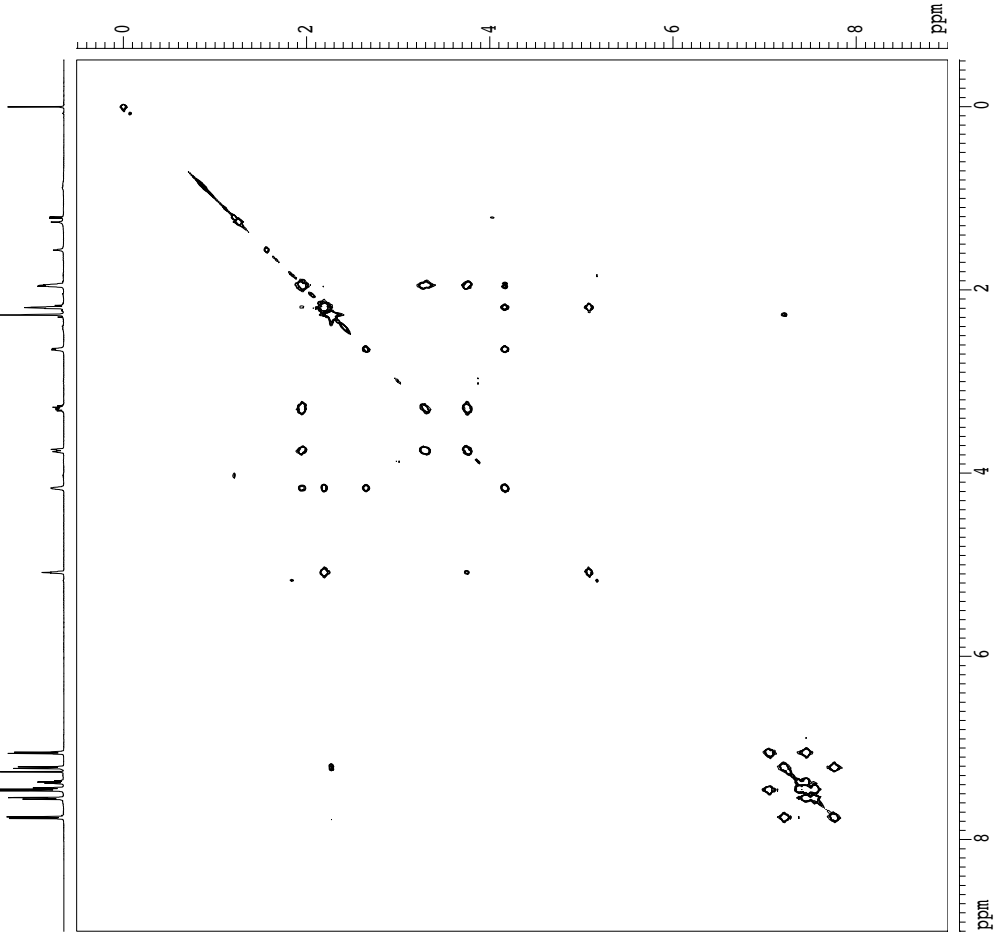
F2 - Acquisition Parameters  
 Date 20201127  
 Time 12.01  
 PROBN 679000  
 PULPROG zgpg30  
 PROCNO 5  
 TD 48074  
 SOLVENT CHCl3  
 DS 9  
 SFO 8012.820 Hz  
 FIDRES 0.16667 Hz  
 AQ 2.3998677 sec  
 RG 62.400 usec  
 DE 6.00 usec  
 TE 298.2 K  
 T1 0.16000000 sec  
 T2 0.00000000 sec  
 T3 0.00000000 sec  
 T4 0.00000000 sec  
 T5 0.00000000 sec  
 T6 0.00000000 sec  
 T7 0.00000000 sec  
 T8 0.00000000 sec  
 T9 0.00000000 sec  
 T10 0.00000000 sec  
 T11 0.00000000 sec  
 T12 0.00000000 sec  
 T13 0.00000000 sec  
 T14 0.00000000 sec  
 T15 0.00000000 sec  
 T16 0.00000000 sec  
 T17 0.00000000 sec  
 T18 0.00000000 sec  
 T19 0.00000000 sec  
 T20 0.00000000 sec  
 T21 0.00000000 sec  
 T22 0.00000000 sec  
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 T30 0.00000000 sec  
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 T34 0.00000000 sec  
 T35 0.00000000 sec  
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 T39 0.00000000 sec  
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 T49 0.00000000 sec  
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 T61 0.00000000 sec  
 T62 0.00000000 sec  
 T63 0.00000000 sec  
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 T66 0.00000000 sec  
 T67 0.00000000 sec  
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 T71 0.00000000 sec  
 T72 0.00000000 sec  
 T73 0.00000000 sec  
 T74 0.00000000 sec  
 T75 0.00000000 sec  
 T76 0.00000000 sec  
 T77 0.00000000 sec  
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 T89 0.00000000 sec  
 T90 0.00000000 sec  
 T91 0.00000000 sec  
 T92 0.00000000 sec  
 T93 0.00000000 sec  
 T94 0.00000000 sec  
 T95 0.00000000 sec  
 T96 0.00000000 sec  
 T97 0.00000000 sec  
 T98 0.00000000 sec  
 T99 0.00000000 sec  
 T100 0.00000000 sec

Z-restored spin-echo 13C spectrum with 1H decoupling





Current Data Parameters  
 USER khewitt1  
 NAME RM-1V-043-2  
 EXPNO 6  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20211127  
 Time 12:32  
 SYSTEM spect  
 PULPROG zgpg30  
 PROBRD 5 mm CPXI 1H-  
 PULPROG cosypp60.prd  
 TD 2048  
 SOLVENT cdc13  
 NS 14  
 DS 14  
 SWH 8012.820 Hz  
 FIDRES 3.912510 Hz  
 AQ 0.12788452 sec  
 RG 256  
 RE 62.00 usec  
 DE 15.00 usec  
 TE 298.0 K  
 TD 0.0000300 sec  
 d0 0.0000300 sec  
 d1 1.0000000 sec  
 d13 0.0000300 sec  
 d14 0.0000300 sec  
 d15 0.0000300 sec  
 TD0 0.0012480 sec  
 ===== CHANNEL f1 =====  
 NU0 1  
 NU1 1  
 F1 511.136 MHz  
 F2 500.136 MHz  
 SF01 500.2235015 MHz  
 ===== GRADIENT CHANNEL =====  
 GRAM1 SISO0.100  
 CHAN2 SISO0.100  
 GPX2 0.00 %  
 GPY2 0.00 %  
 GPZ2 0.00 %  
 GRX2 0.00 %  
 GRZ2 0.00 %  
 GY2 17.00 %  
 GZ2 17.00 %  
 PL16 1000.00 usec  
 F1 - Acquisition parameters  
 NU0 511.136 MHz  
 SF01 500.2235 MHz  
 FIDRES 15.655040 Hz  
 SW 16.018 ppm  
 PHASE2 QF  
 F2 - Processing parameters  
 SI 1024  
 SF 500.2200324 MHz  
 NDMW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 1.00  
 PC 1.00  
 F1 - Processing parameters  
 SI 1024  
 SF 500.2200324 MHz  
 NDMW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 1.00  
 2D NMR plot parameters  
 CX2 15.00 cm  
 CZ2 15.00 cm  
 FZPLO 9.000 ppm  
 FZPLD 9.000 Hz  
 FZPHI 0.000 Hz  
 FZPHL -255.54 Hz  
 FZPL0 9.000 ppm  
 FZPLD 9.000 Hz  
 FZPHI -0.511 ppm  
 FZPHL 0.511 ppm  
 FZPRCH 0.63407 ppm/cm  
 FZPRCH 317.17413 Hz/cm  
 FZPRCH 0.63407 ppm/cm  
 FZPRCH 317.17413 Hz/cm



gnoe

ppm

7.82995  
7.81866

2.22350  
2.22916  
2.23171  
2.23037  
2.23365  
2.26218

```

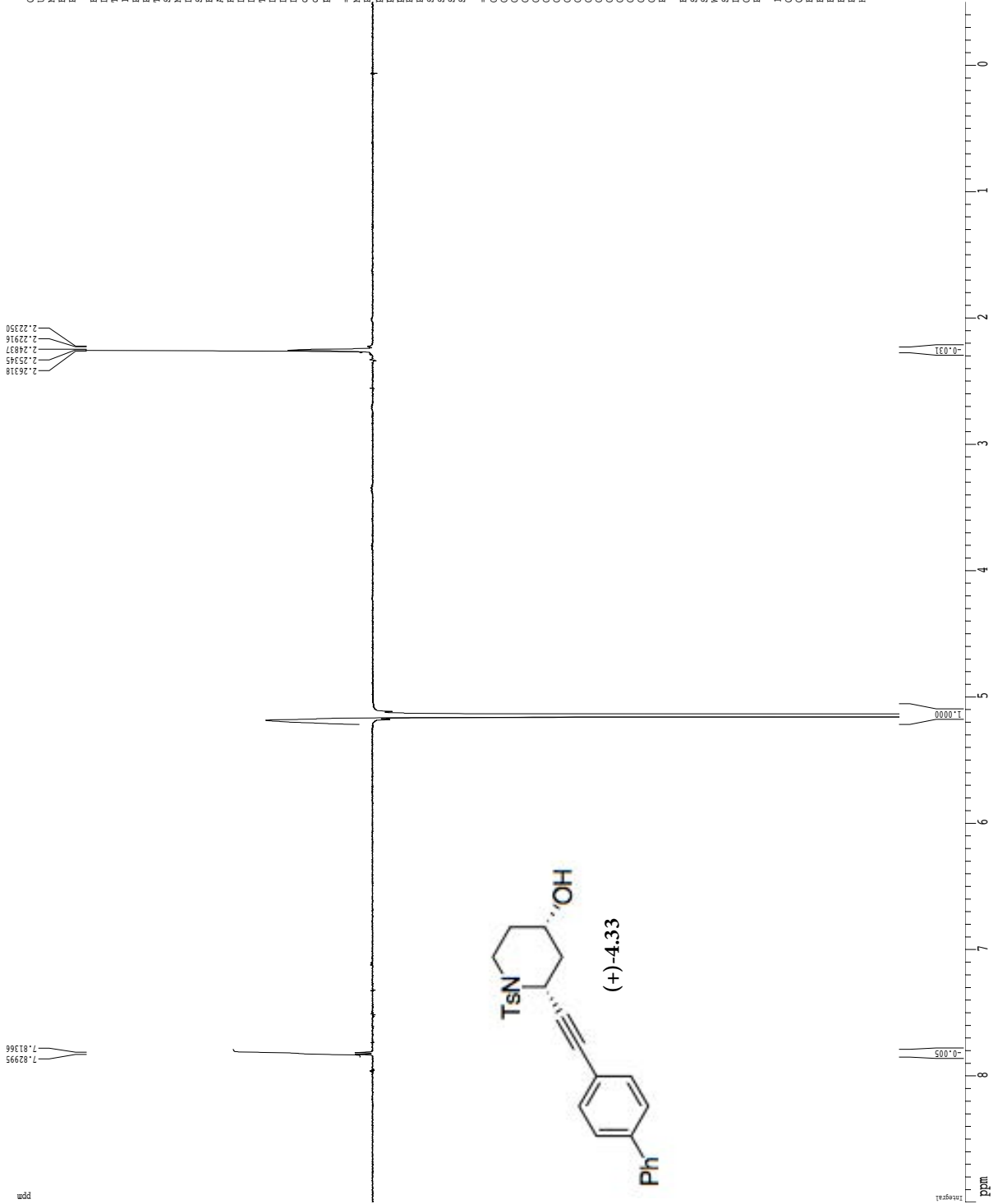
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NAME      KMF-TV-043-2
EXPNO     4
PROCNO    1

F2 - Acquisition Parameters
Date_     20201127
Time      12.17
Operator  gnoe
PULPROG   sm
PROBHD    5 mm CPCT 1H
PULPROG2  gmgdco22.prd
TD        65536
SOLVENT   CDCl3
NS        128
DS        8
SWH       8012.822 Hz
FIDRES    0.122266 Hz
AQ        4.1089496 sec
RG        62.400 usec
DE        6.00 usec
TE        298.2 K
D1        1.0000000 sec
d11       0.0100000 sec
d12       0.0020000 sec
d21       0.33375451 sec
d22       0.16398659 sec
P2        19.50 usec

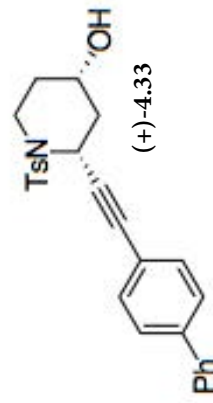
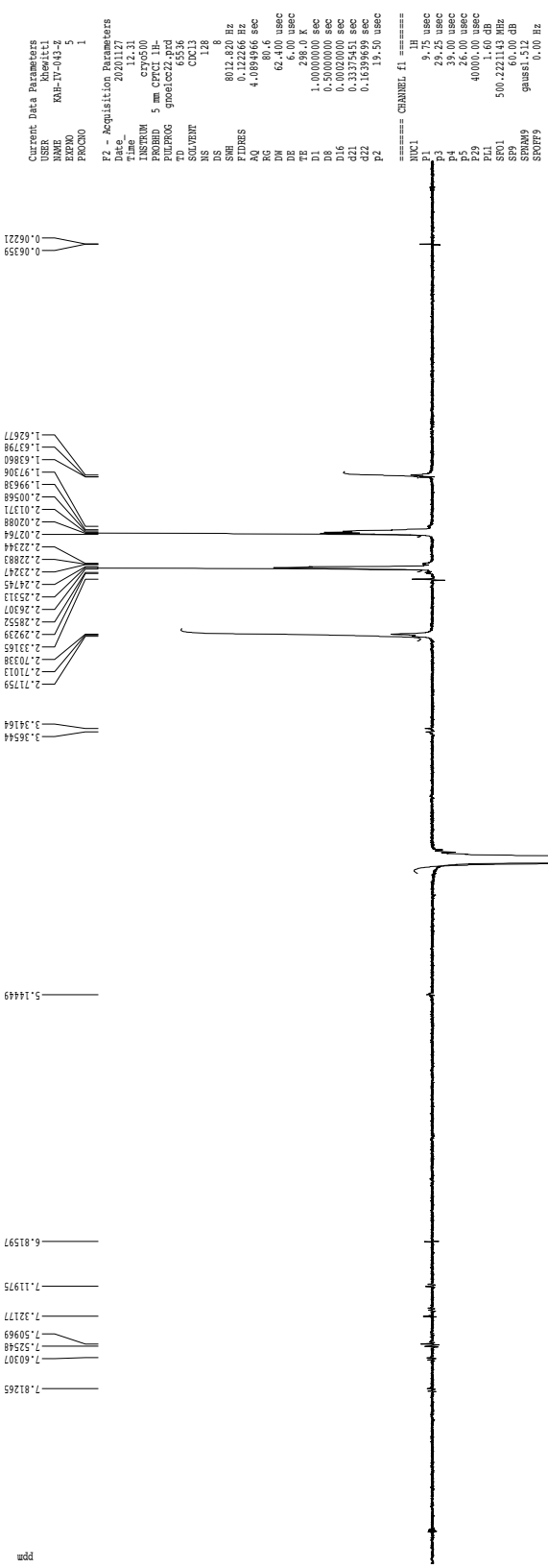
===== CHANNEL f1 =====
NUC1      1H
P1        9.75 usec
P2        29.25 usec
P3        29.25 usec
P4        29.25 usec
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P6        29.25 usec
P7        29.25 usec
P8        29.25 usec
P9        29.25 usec
P10       29.25 usec
P11       40000.00 usec
P12       40000.00 usec
P13       40000.00 usec
P14       40000.00 usec
P15       40000.00 usec
P16       40000.00 usec
P17       40000.00 usec
P18       40000.00 usec
P19       40000.00 usec
P20       40000.00 usec
P21       40000.00 usec
P22       40000.00 usec
P23       40000.00 usec
P24       40000.00 usec
P25       40000.00 usec
P26       40000.00 usec
P27       40000.00 usec
P28       40000.00 usec
P29       40000.00 usec
P30       40000.00 usec
P31       40000.00 usec
P32       40000.00 usec
P33       40000.00 usec
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P58       40000.00 usec
P59       40000.00 usec
P60       40000.00 usec
P61       40000.00 usec
P62       40000.00 usec
P63       40000.00 usec
P64       40000.00 usec
P65       40000.00 usec
P66       40000.00 usec
P67       40000.00 usec
P68       40000.00 usec
P69       40000.00 usec
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P71       40000.00 usec
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P73       40000.00 usec
P74       40000.00 usec
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P94       40000.00 usec
P95       40000.00 usec
P96       40000.00 usec
P97       40000.00 usec
P98       40000.00 usec
P99       40000.00 usec
P100      40000.00 usec
===== GRABF2 CHANNEL =====
GRABF2    SWSQ10.100
GRABF3    SWSQ10.100
GRABF4    SWSQ10.100
GRABF5    SWSQ10.100
GRABF6    SWSQ10.100
GRABF7    SWSQ10.100
GRABF8    SWSQ10.100
GRABF9    SWSQ10.100
GRABF10   SWSQ10.100
GRABF11   SWSQ10.100
GRABF12   SWSQ10.100
GRABF13   SWSQ10.100
GRABF14   SWSQ10.100
GRABF15   SWSQ10.100
GRABF16   SWSQ10.100
GRABF17   SWSQ10.100
GRABF18   SWSQ10.100
GRABF19   SWSQ10.100
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GRABF21   SWSQ10.100
GRABF22   SWSQ10.100
GRABF23   SWSQ10.100
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GRABF27   SWSQ10.100
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GRABF57   SWSQ10.100
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GRABF87   SWSQ10.100
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GRABF95   SWSQ10.100
GRABF96   SWSQ10.100
GRABF97   SWSQ10.100
GRABF98   SWSQ10.100
GRABF99   SWSQ10.100
GRABF100  SWSQ10.100
===== GRABF3 CHANNEL =====
GRABF3    SWSQ10.100
GRABF4    SWSQ10.100
GRABF5    SWSQ10.100
GRABF6    SWSQ10.100
GRABF7    SWSQ10.100
GRABF8    SWSQ10.100
GRABF9    SWSQ10.100
GRABF10   SWSQ10.100
GRABF11   SWSQ10.100
GRABF12   SWSQ10.100
GRABF13   SWSQ10.100
GRABF14   SWSQ10.100
GRABF15   SWSQ10.100
GRABF16   SWSQ10.100
GRABF17   SWSQ10.100
GRABF18   SWSQ10.100
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GRABF20   SWSQ10.100
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GRABF72   SWSQ10.100
GRABF73   SWSQ10.100
GRABF74   SWSQ10.100
GRABF75   SWSQ10.100
GRABF76   SWSQ10.100
GRABF77   SWSQ10.100
GRABF78   SWSQ10.100
GRABF79   SWSQ10.100
GRABF80   SWSQ10.100
GRABF81   SWSQ10.100
GRABF82   SWSQ10.100
GRABF83   SWSQ10.100
GRABF84   SWSQ10.100
GRABF85   SWSQ10.100
GRABF86   SWSQ10.100
GRABF87   SWSQ10.100
GRABF88   SWSQ10.100
GRABF89   SWSQ10.100
GRABF90   SWSQ10.100
GRABF91   SWSQ10.100
GRABF92   SWSQ10.100
GRABF93   SWSQ10.100
GRABF94   SWSQ10.100
GRABF95   SWSQ10.100
GRABF96   SWSQ10.100
GRABF97   SWSQ10.100
GRABF98   SWSQ10.100
GRABF99   SWSQ10.100
GRABF100  SWSQ10.100
===== GRABF4 CHANNEL =====
GRABF4    SWSQ10.100
GRABF5    SWSQ10.100
GRABF6    SWSQ10.100
GRABF7    SWSQ10.100
GRABF8    SWSQ10.100
GRABF9    SWSQ10.100
GRABF10   SWSQ10.100
GRABF11   SWSQ10.100
GRABF12   SWSQ10.100
GRABF13   SWSQ10.100
GRABF14   SWSQ10.100
GRABF15   SWSQ10.100
GRABF16   SWSQ10.100
GRABF17   SWSQ10.100
GRABF18   SWSQ10.100
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GRABF80   SWSQ10.100
GRABF81   SWSQ10.100
GRABF82   SWSQ10.100
GRABF83   SWSQ10.100
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GRABF97   SWSQ10.100
GRABF98   SWSQ10.100
GRABF99   SWSQ10.100
GRABF100  SWSQ10.100
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RG        62.400 usec
WDW       EM
SSB       0
GB        0
LB        0.00 Hz
PC        1.00

ID NMR plot parameters
CX        22.88 cm
CY        50.00 cm
CZ        50.00 cm
FL        480.00000 ppm
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F2P2      -250.11 Hz
F2P3      0.41667 ppm/cm
F2P4      200.42500 Hz/cm
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```



gnoe



Current Data Parameters  
USER khwittl  
NAME KMF-TV-043-2  
EXPNO 5  
PROCNO 1

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Time 17.31  
Operator gnoe  
PROBHD 5 mm CPDQ1 1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
DS 128  
SS 18

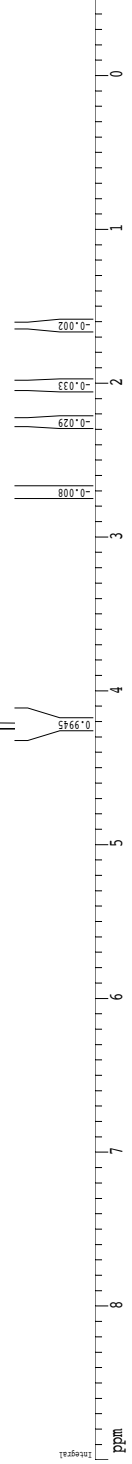
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P3 28.25 usec  
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P100 40000.00 usec

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GFRM99 SWSQ10.10  
GFRM100 SWSQ10.10

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GB 0  
LB 0.00 Hz  
GB 0  
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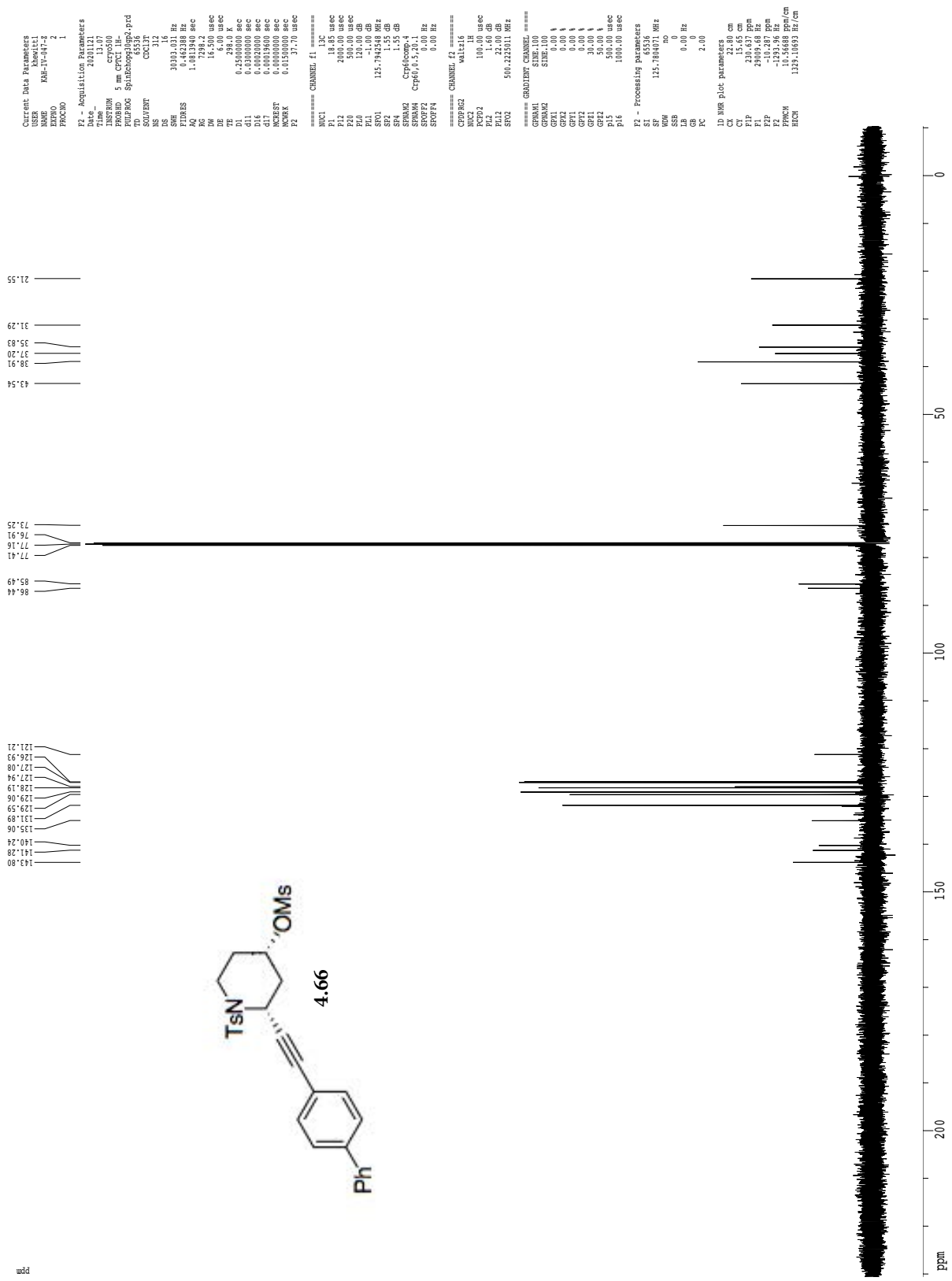
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CY 50.00 cm  
CZ 50.00 cm  
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FY 450.00 ppm  
FZ 0.5000 ppm  
F2 -250.11 Hz  
PRGK 0.41667 ppm/cm  
HCN 200.42500 Hz/cm



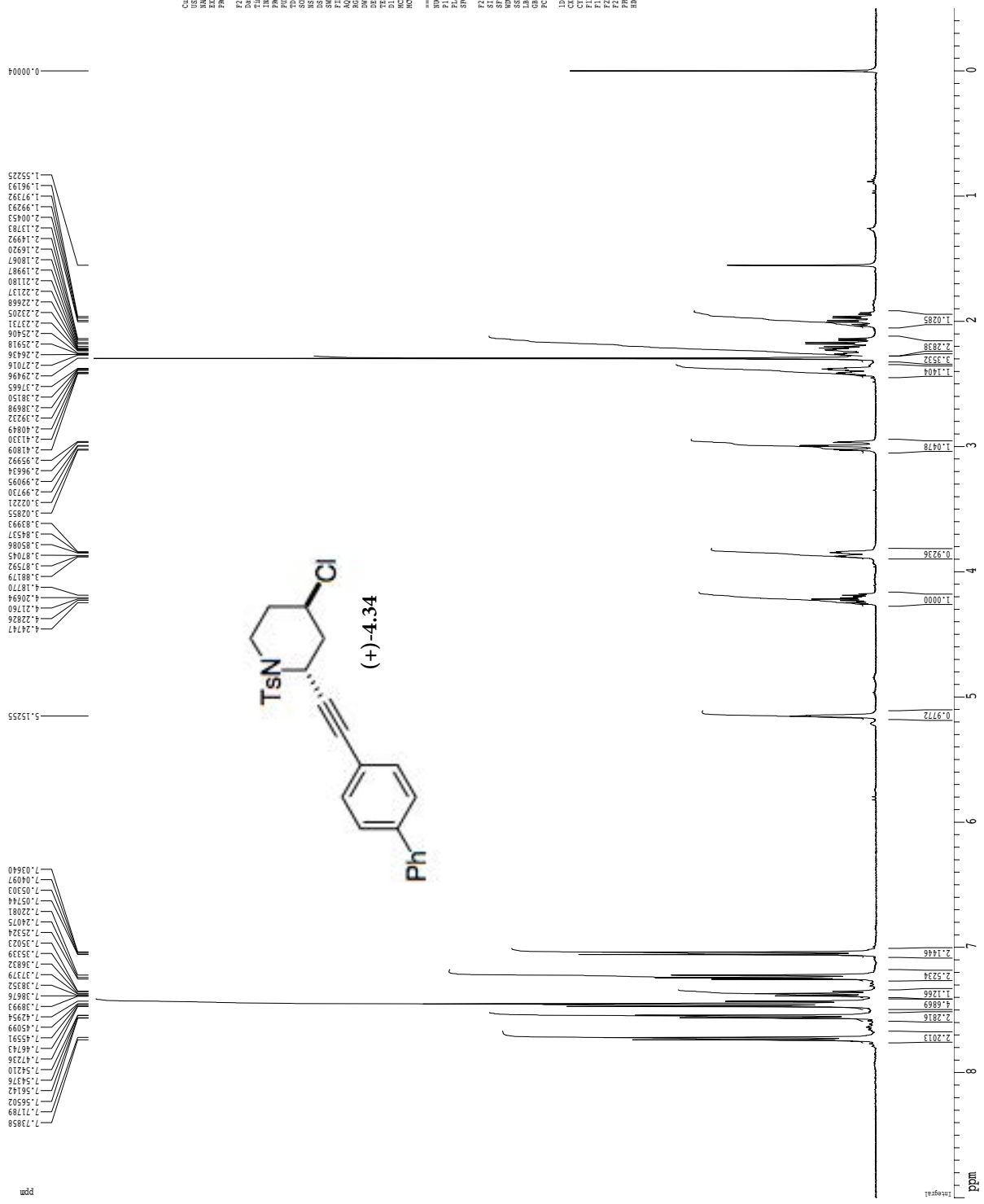




Z-restored spin-echo 13C spectrum with 1H decoupling



1H spectrum



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 NAME EL-11-123-shia-phrase  
 PROCNO 1

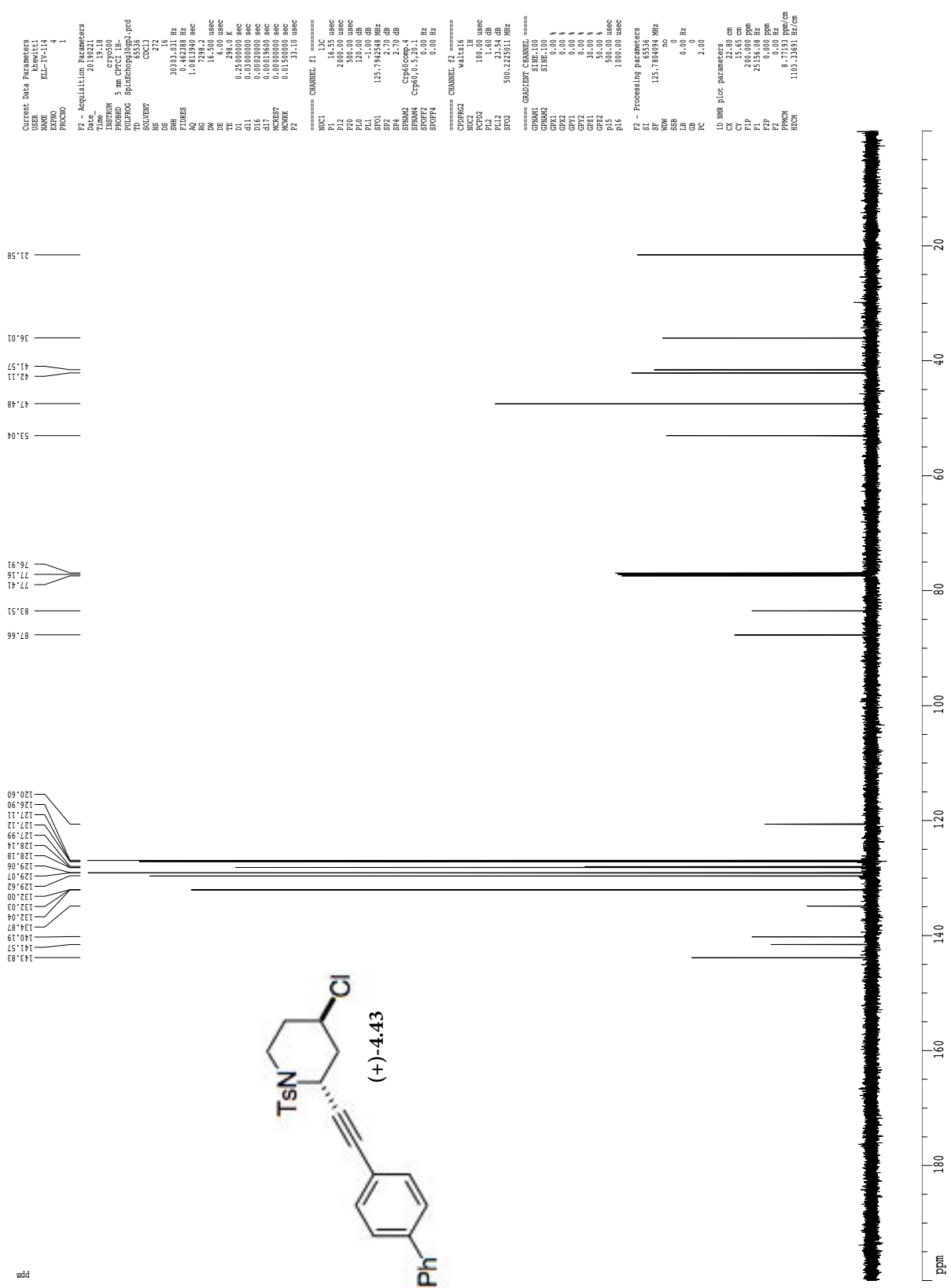
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 PULPROG zgpg30  
 TD 32640  
 CQ 1.00  
 COUPLERT  
 NS 8  
 DS 6410.42 Hz  
 FIDRES 0.166673 Hz  
 AQ 2.999989 sec  
 RG 78.800 us/c  
 SW 78.800 us/c  
 DE 4.50 us/c  
 TE 300.2 K  
 D1 0.1000000 sec  
 MCHYST 0.0000000 sec  
 MONK 0.0150000 sec

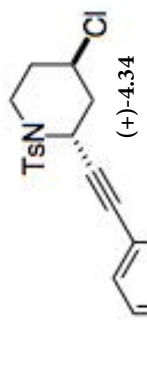
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 PL1 -1.10 dB  
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F2 - Processing parameters  
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 SF 400.1330000 MHz  
 WM 0  
 WOP 0  
 CB 0  
 GB 0  
 PC 2.00

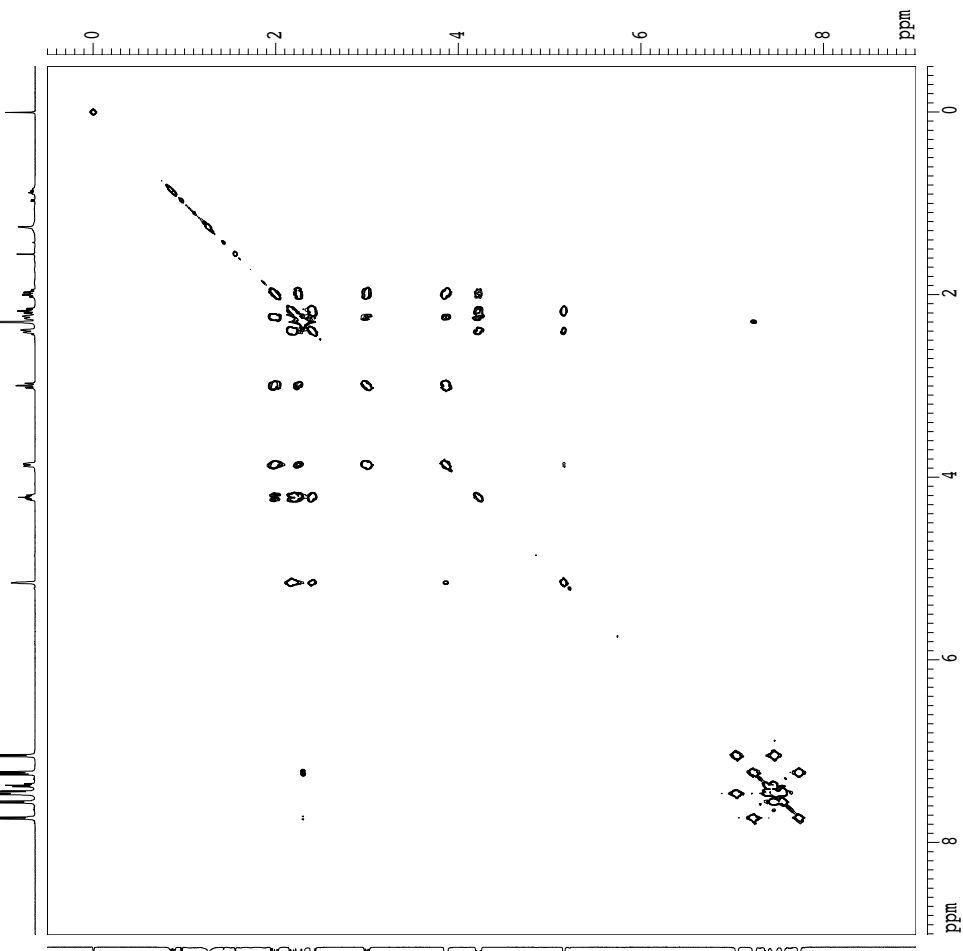
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 CT 15.00 cm  
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 F2 -0.200 ppm  
 F3 0.000 ppm  
 FWHM 0.41867 ppm/cm  
 BUCK 166.72886 Hz/cm

### Z-restored spin-echo 13C spectrum with 1H decoupling





gcosy60



```

Current Data Parameters
USER      Khevit11
NAME      ELL-11-14
EXPNO     3
PROCNO    1

F2 - Acquisition Parameters
Date_     20190221
Time      19.04
INSTRUM   cryo500
PROBHD    5 mm cryoProbe
PULPROG   zgpg30
TD         2048
SOLVENT   CDCl3
NS         1
DS         16
AQ         8013.85 Hz
FIDRES    3.912510 Hz
RG         0.1278452 sec
RG         143.7
DM         62.400 usec
DE         298.0 usec
TE         298.0
d0         0.00003300 sec
d1         1.00000000 sec
d13       0.00003300 sec
d16       0.00020000 sec
d10       0.00014000 sec

===== CHANNEL f1 =====
NUC1      1H
P1        7.50 usec
PC        0.00 usec
SFO1      500.225015 MHz

===== GRADIENT CHANNEL =====
GPRM1     sine,100
GPRM2     sine,100
GPRM3     0.00 %
GPRM4     0.00 %
GPRM5     0.00 %
GPRM6     0.00 %
GPRM7     17.00 %
GPRM8     17.00 %
PRF       1000.00 usec

F1 - Acquisition parameters
NUC1      1H
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PC        0.00 usec
SFO1      500.225015 MHz
SFO2      15.650040 Hz
SN        16.018 ppm
PRF       1000.00 usec

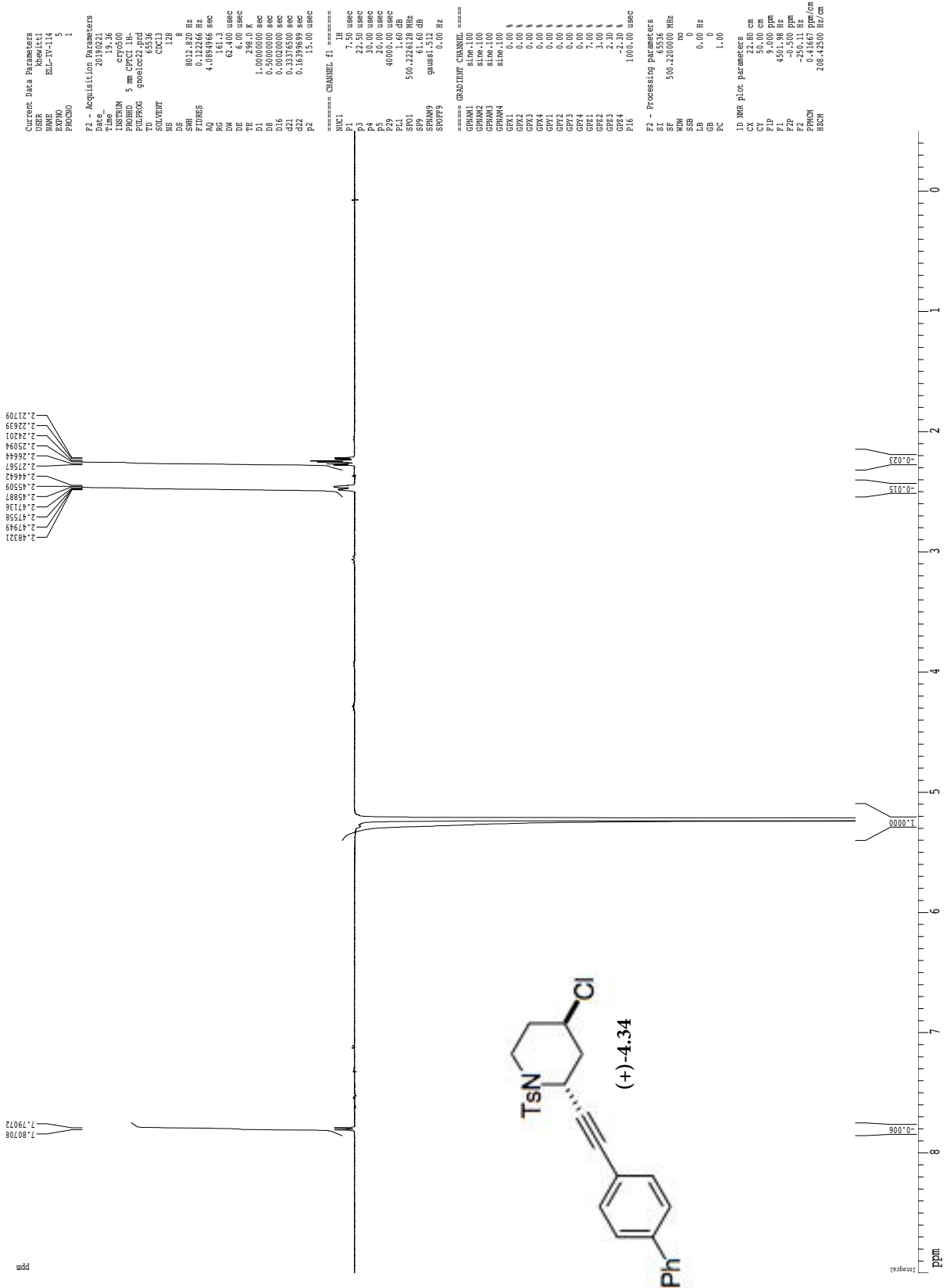
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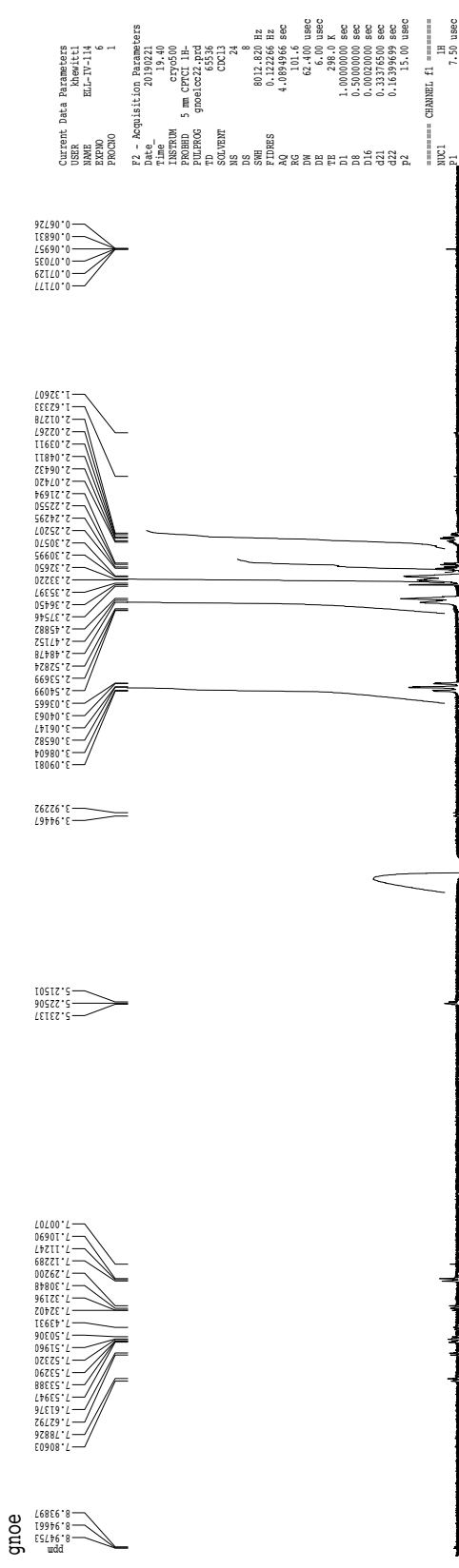
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SF         500.2203349 MHz
WDW        SINE
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GB         0.00 Hz

2D NMR plot parameters
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CX1        15.00 cm
F2PLO     5.011 ppm
F2PO      450.500 Hz
F2PHI     0.50000000
F2HI      -250.28 Hz
F1PLO     9.011 ppm
F1PO      4507.33 Hz
F1PHI     0.50000000
F1HI      -250.28 Hz
F2PRCM    0.63407 ppm/cm
F2PHCM    317.17413 Hz/cm
F1PRCM    0.63407 ppm/cm
F1PHCM    317.17413 Hz/cm
  
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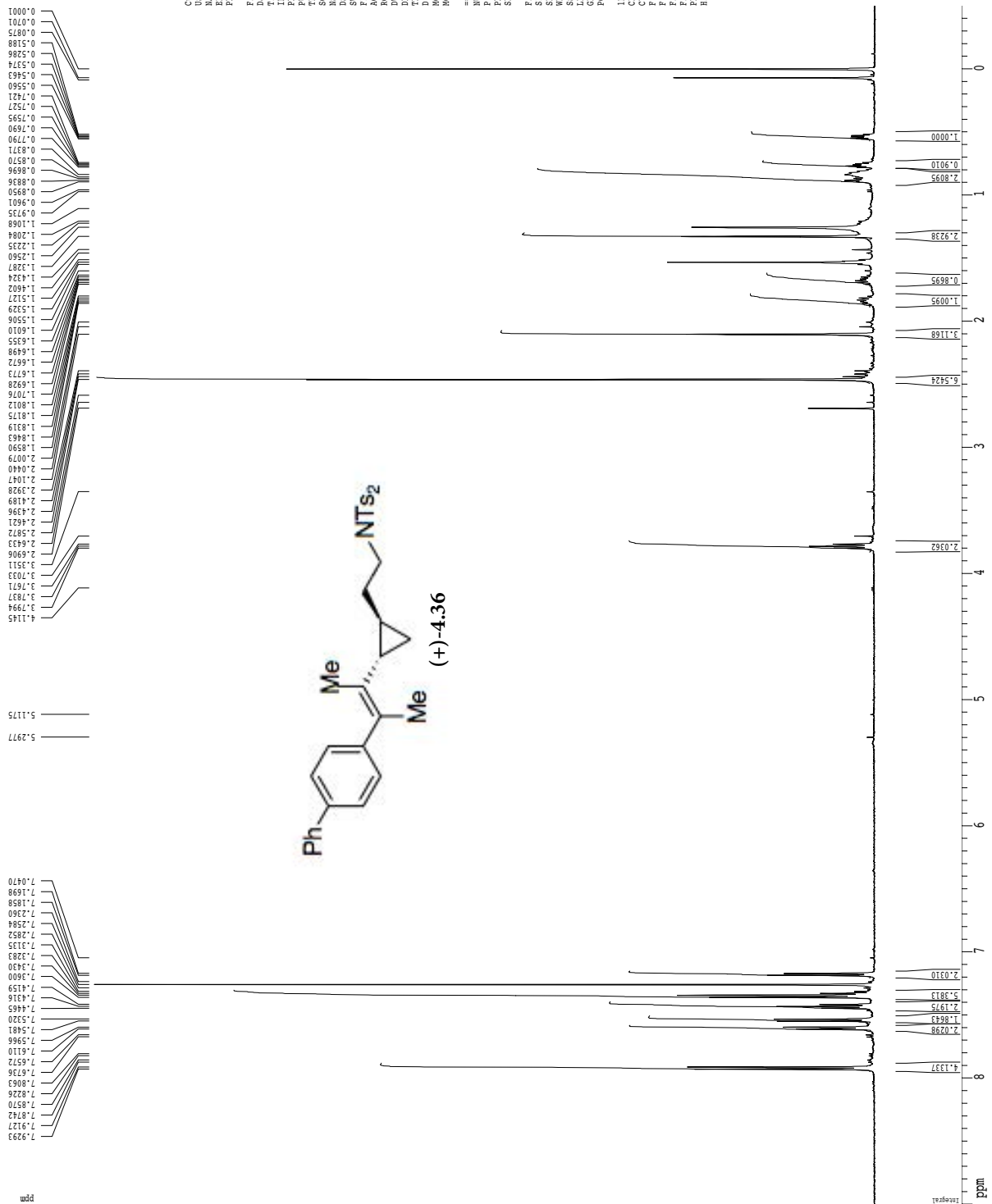
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ppm





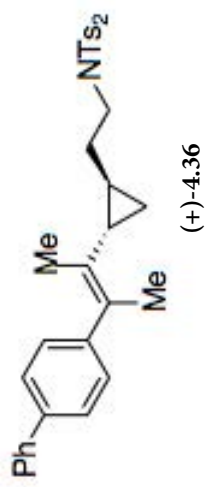
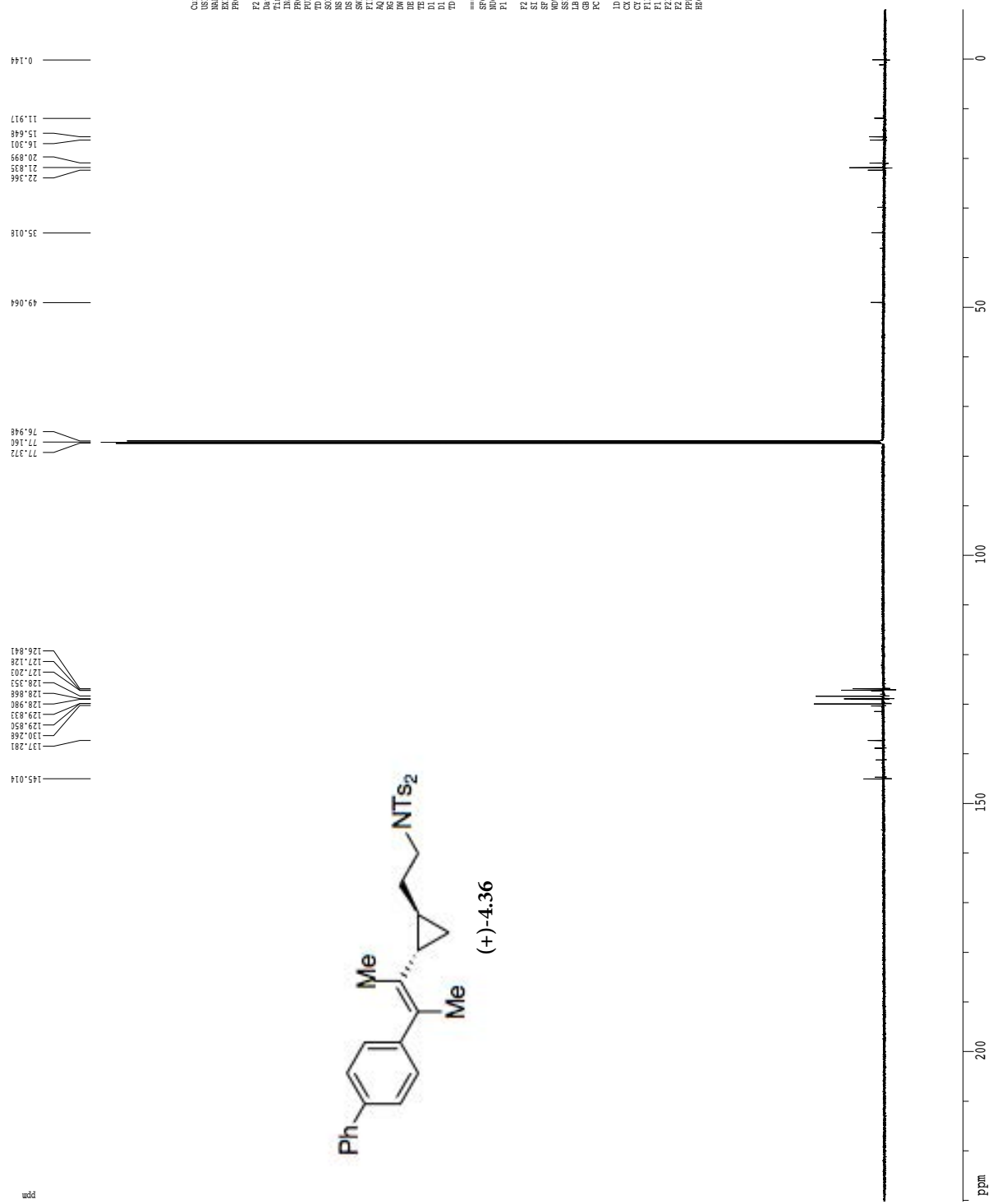
1H spectrum



Current Data Parameters  
USER KMH-1V-153-enant  
NAME KMH-1V-153-enant  
EXPNO 1  
PROCNO 1  
P2 - Acquisition Parameters  
Date\_ 20101023  
Time 14:43  
PROBHD 5 mm CPY 131  
PULPROG zgpg30  
TD 48074  
SOLVENT CDCl3  
DS 9  
SHE 8012.820 Hz  
FIDRES 0.16667 Hz  
AQ 2.3998677 sec  
RG 62.400 usec  
DE 6.00 usec  
TE 298.2 K  
AQ 0.16667 sec  
RG 62.400 usec  
MCORE 0.0150000 sec  
MCORR 0.0150000 sec  
===== CHANNEL f1 =====  
NUC1 13C  
P1 9.75 usec  
PL1 1.68 dB  
SFO1 500.2235015 MHz  
P2 - Processing parameters  
SI 65536  
SF 500.220320 MHz  
WDW no  
SSB 0.00 Hz  
GB 0  
PC 1.00  
ID NR file parameters  
CY 22.80 cm  
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FIP 9.000 ppm  
FL 4501.98 Hz  
F2 -250.11 Hz  
PRCM 0.41667 ppm/cm  
HZCM 206.44502 Hz/cm

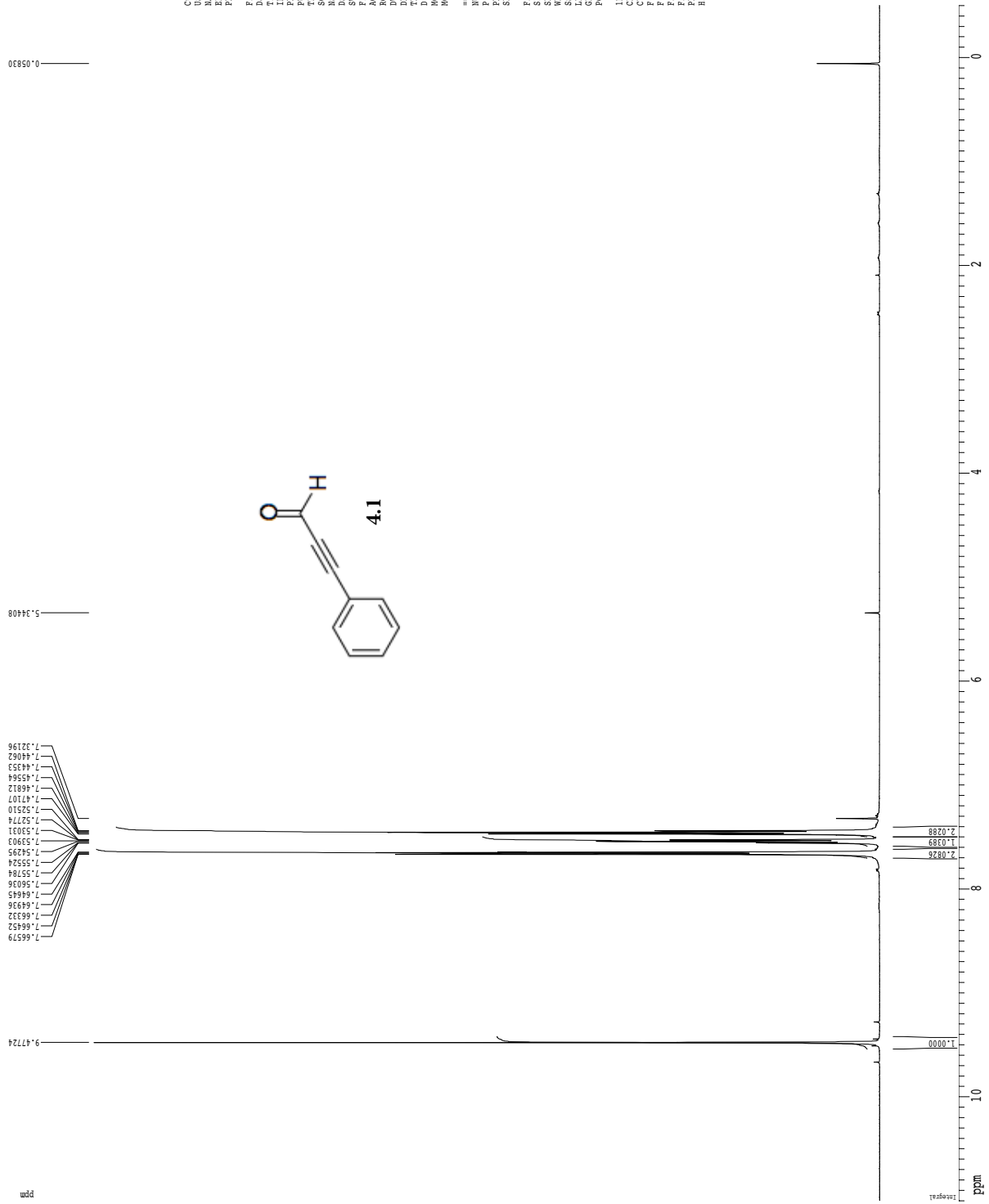


1H spectrum



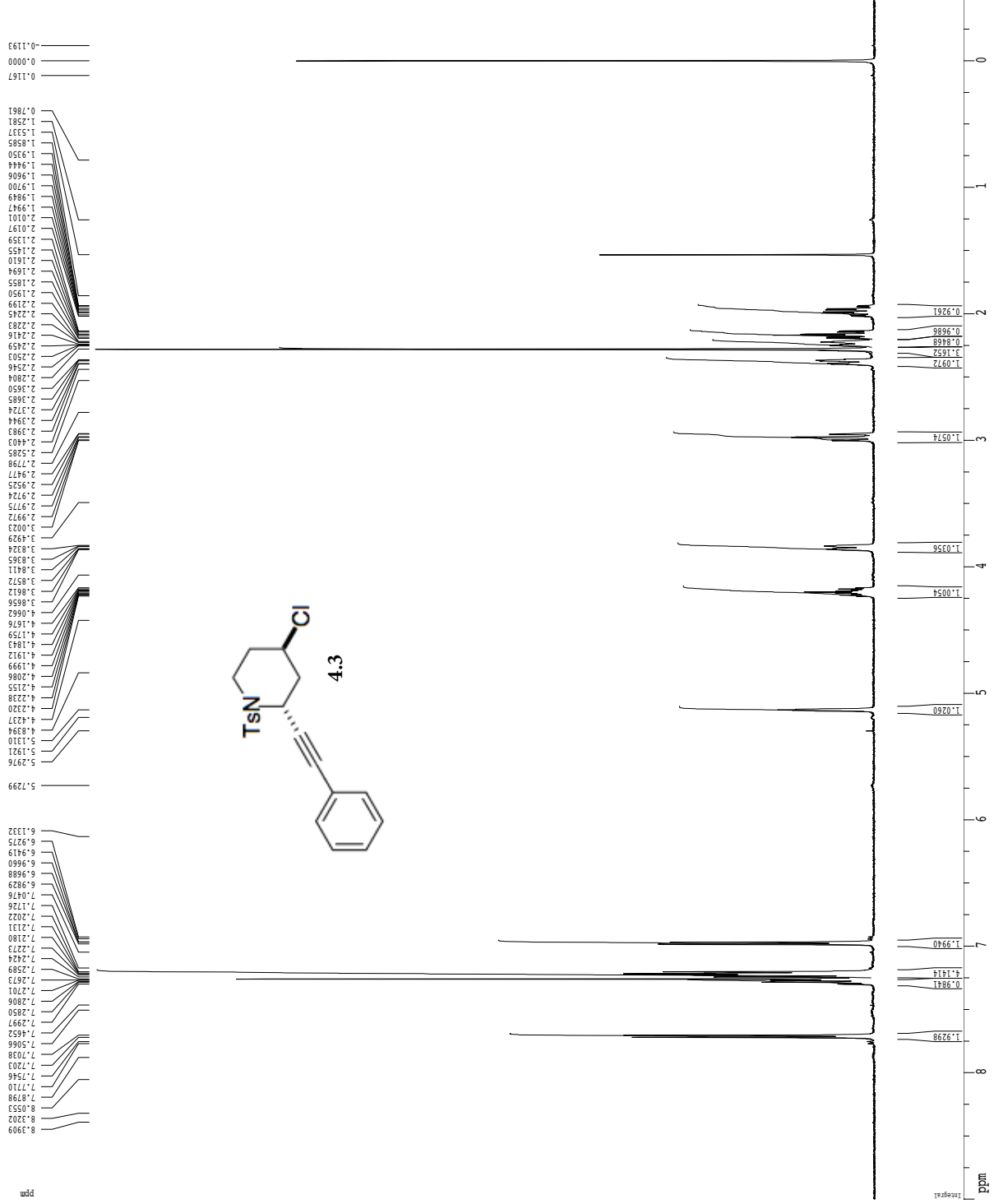
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 EXPNO KM-1V-153-unt  
 PROCNO 1  
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 Time 15:46  
 INSTRUM av600  
 PROBRD 5 mm CPBBO BB-  
 PULPROG zgpg30  
 SFO1 500.1360536  
 SOLVENT CDCl3  
 NS 640  
 DS 4  
 AS 3631.08 Hz  
 FTRES 0.5524655 Hz  
 AQ 0.9944468 sec  
 RG 2050  
 DW 11.800 usec  
 DE 1.000 usec  
 TE 298.0 K  
 D1 0.4000001 sec  
 D1.1 0.0300000 sec  
 TDD 1  
 ===== CHANNEL f1 =====  
 SFO1 150.9194880 MHz  
 NUCL1 13C  
 P1 10.10 usec  
 F2 - Processing parameters  
 SI 65536  
 SF 150.927931 MHz  
 DS 4  
 AS 0  
 SSB 0  
 LB 0.400 Hz  
 GB 0  
 PC 1.40  
 ID NMR plot parameters  
 CX 22.80 cm  
 CT 15.00 cm  
 CI 23.00 cm  
 F1 34730.87 Hz  
 F2 9.847 ppm  
 F3 -1501.01 Hz  
 FWHM 0.1818877 Hz/cm  
 HPCW 1589.11468 Hz/cm

1H spectrum



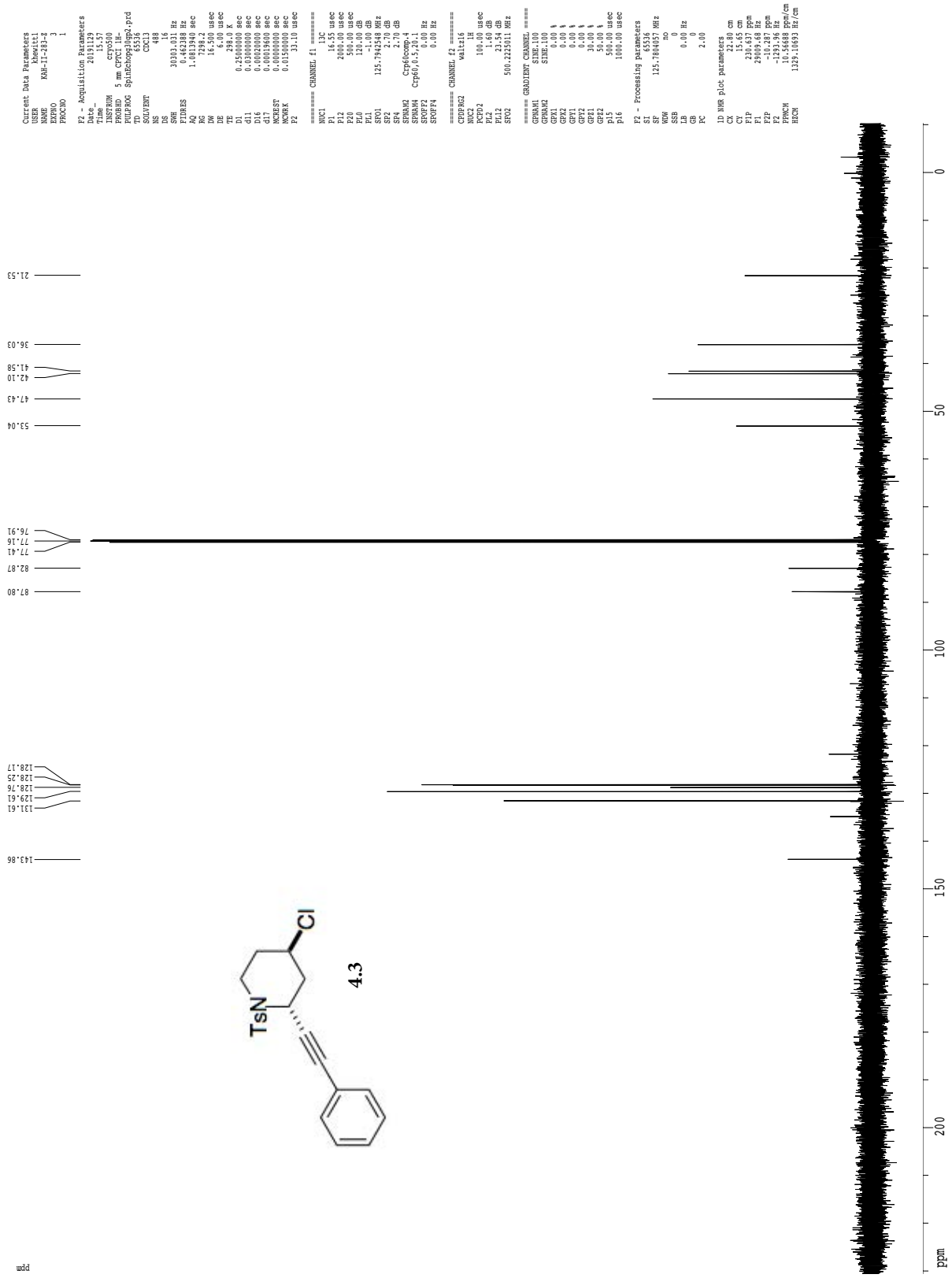
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PROBHD 5 mm CPY-1H  
PULPROG zgpg30  
TD 48074  
SOLVENT CDCl3  
DS 2  
SHE 8012.820 Hz  
FIDRES 0.166677 Hz  
AQ 2.399877 sec  
RG 62.400 usec  
DE 6.00 usec  
TE 298.2 K  
SFO1 500.223015 MHz  
MORSE 0.100000 sec  
MORSE 0.0150000 sec  
===== CHANNEL f1 =====  
NUC1 13C  
PUL1 1.50 usec  
PL1 1.66 dB  
SFO1 500.223015 MHz  
F2 - Processing Parameters  
SI 65536  
SF 500.220000 MHz  
WDW no  
SS 0.00 Hz  
GB 0  
PC 1.00  
ID Wdr File Parameters  
CY 22.80 cm  
CY 15.00 cm  
FIP 11.000 ppm  
FL 590.48 Hz  
F2 -250.11 Hz  
PRGCM 0.50439 ppm/cm  
HZCM 251.30396 Hz/cm

1H spectrum



Current Data Parameters  
 USER KRW  
 NAME KRM-11-283-2  
 EXNO 1  
 PROCNO 1  
 P2 - Acquisition Parameters  
 Date\_ 20191129  
 Time 15:38  
 INSTRON 6700  
 PROBHD 5 mm CPT-1H  
 PULPROG zgpg30  
 TD 48074  
 SOLVENT CDCl3  
 DS 9  
 SFO 8012.820 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.3398677 sec  
 RG 62.400 usec  
 DE 6.00 usec  
 TE 298.2 K  
 D0 1.000000 sec  
 MCHRES 0.000000 sec  
 MSRES 0.0150000 sec  
 ===== CHANNEL f1 =====  
 NUC1 13C  
 P1 1.50 usec  
 PL1 1.66 dB  
 SFO1 500.223015 MHz  
 P2 - Processing Parameters  
 SI 65536  
 SF 500.2200316 MHz  
 WDW no  
 GB 0.00 Hz  
 PC 1.00  
 ID WDR file parameters  
 CY 22.80 cm  
 CV 15.00 cm  
 FIP 9.000 ppm  
 FL 4301.98 Hz  
 F2 -250.11 Hz  
 PPMCM 0.41667 ppm/cm  
 HZCM 206.44502 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



gc0sy60

```

Current Data Parameters
USER      khevitt1
NAME      KAH-II-283-Z
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20121219
Time      15:15:00
INSTRUM   cryo500
PROBHD    5 mm CPXI 1H-
PULPROG   cosyg60.prd
TD        2048
SOLVENT   CDCl3
NS         1
DS         16
SFO1       500.131310 MHz
F2RES      3.972510 Hz
AQ         0.1278452 sec
RG         912.3
DW         62.400 usec
DE         6.00 usec
TE         298.0 K
d0         0.00000300 sec
d1         1.00000000 sec
d11        0.00000000 sec
d12        0.00000000 sec
d16        0.00020000 sec
IN0        0.00012489 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
PL1        1.60 dB
SFO1       500.2235015 MHz

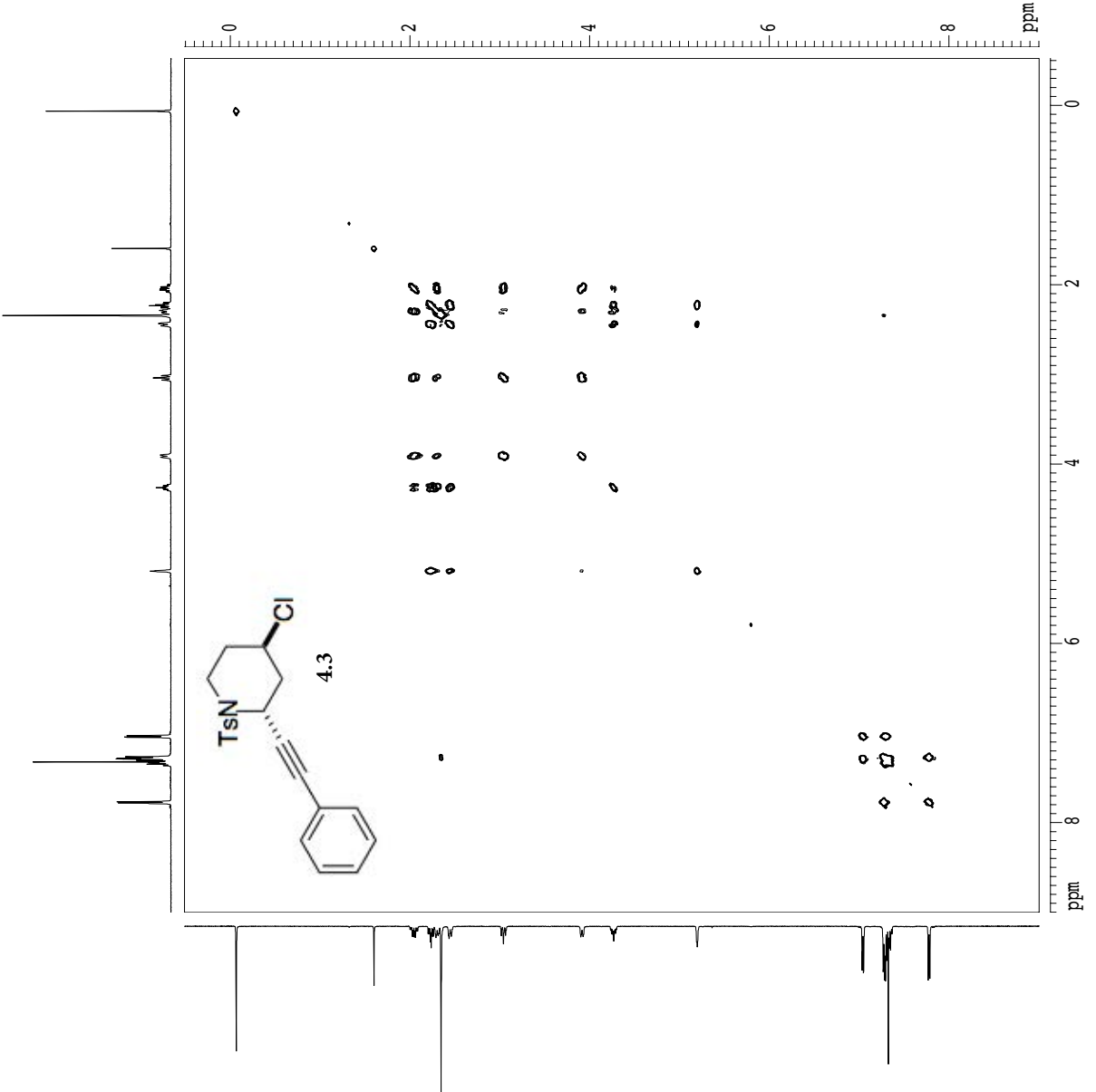
===== GRADIENT CHANNEL =====
GPRAM1     sine.100
GPRAM2     sine.100
GFX1       0.00 %
GFX2       0.00 %
GFI1       0.00 %
GFI2       0.00 %
GZ1        17.00 %
GZ2        17.00 %
P16        1000.00 usec

F1 - Acquisition parameters
ND0         1
TD          512
SFO1        500.2235 MHz
FIDRES      15.650040 Hz
SFO2        16.018 ppm
P1RO2       QF

F2 - Processing parameters
SI          1024
SF          500.2200000 MHz
WDW         SINE
SSB         0
LB          0.00 Hz
GB          0
PC          1.00

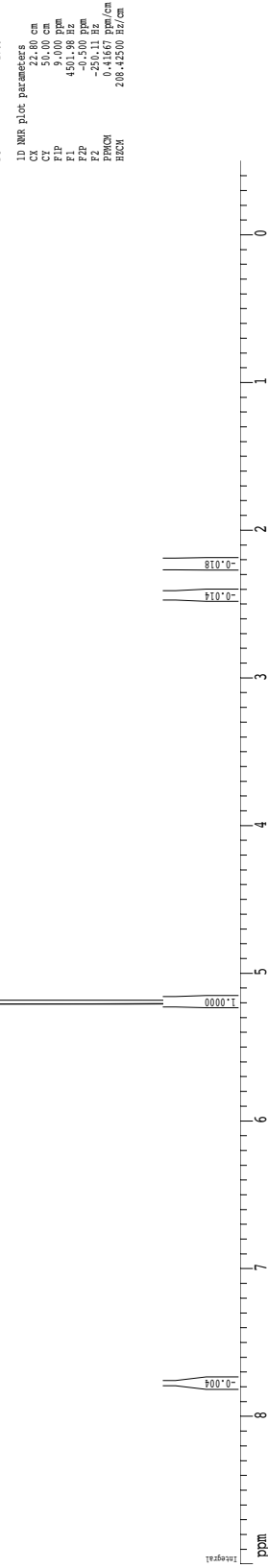
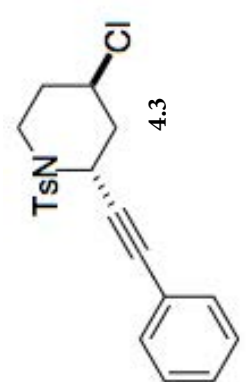
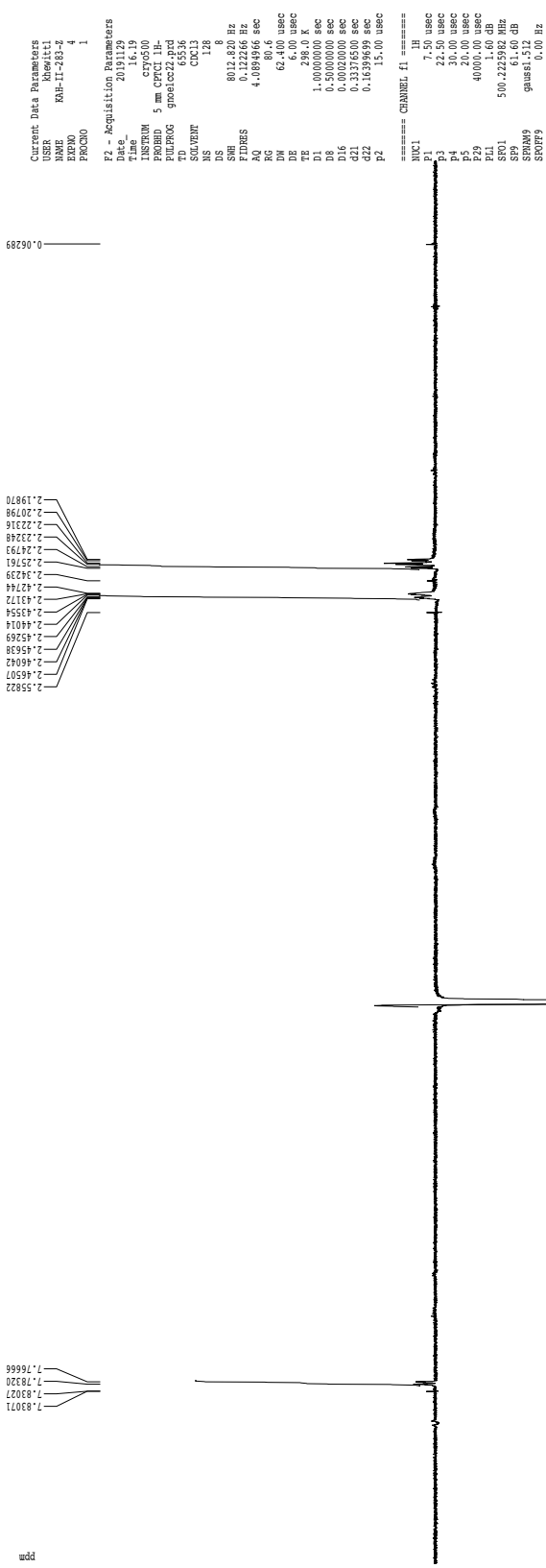
F1 - Processing parameters
SI          1024
MC2         QF
SF          500.2200000 MHz
WDW         SINE
SSB         0
LB          0.00 Hz
GB          0

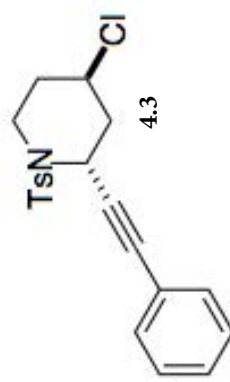
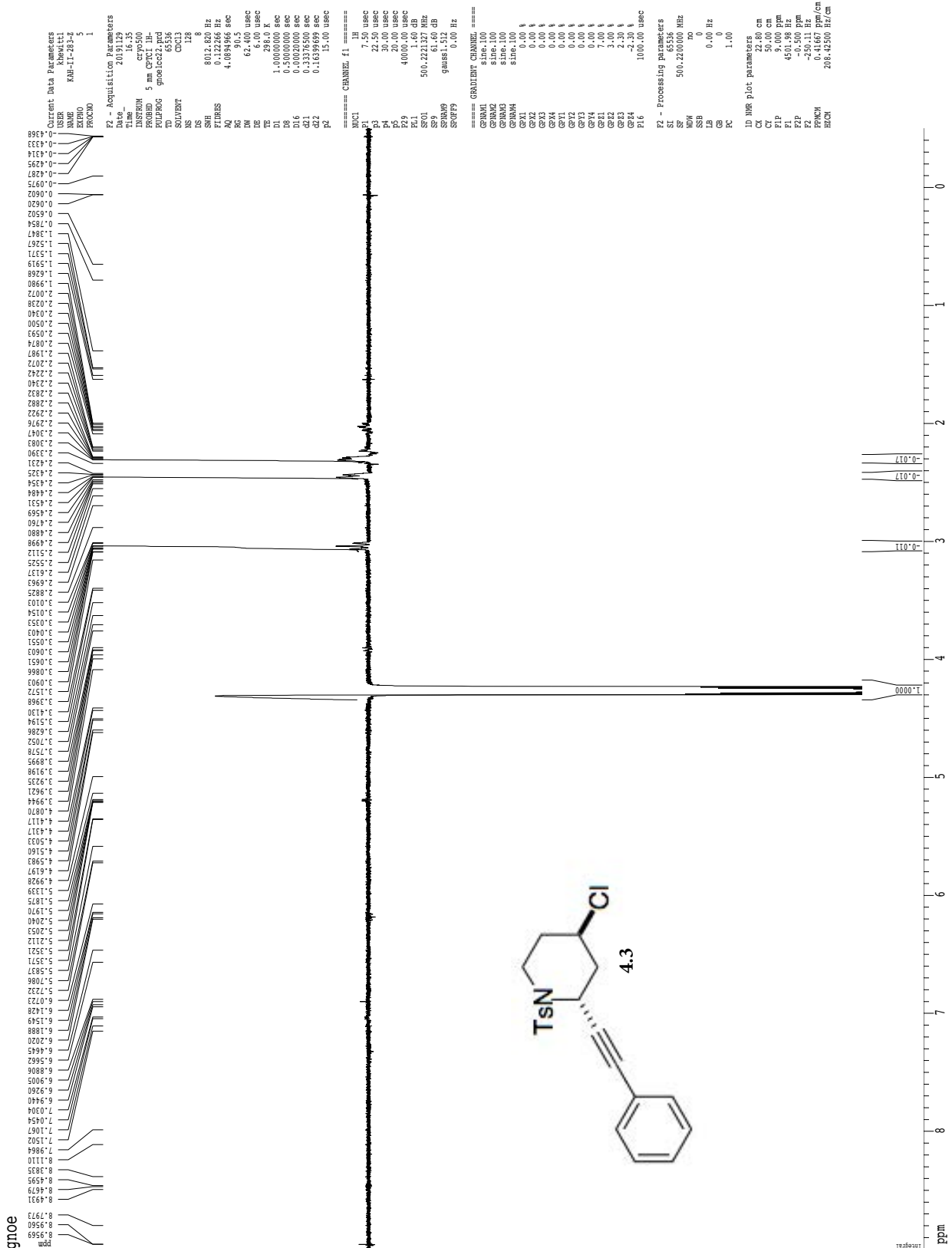
2D NMR plot parameters
CX2         15.00 cm
CX1         15.00 cm
F2PLO       9.002 ppm
F2LO        4502.14 Hz
F2PHI       -0.524 ppm
F2H1        -267.29 Hz
F2H2        4502.14 ppm
F1LO        4502.14 ppm
F1PHI       -0.509 ppm
F1H1        -254.47 Hz
F2PPMCM    0.65311 ppm/cm
F2HCM      317.65983 Hz/cm
F1PPMCM    0.65407 ppm/cm
F1HCM      317.17416 Hz/cm
  
```



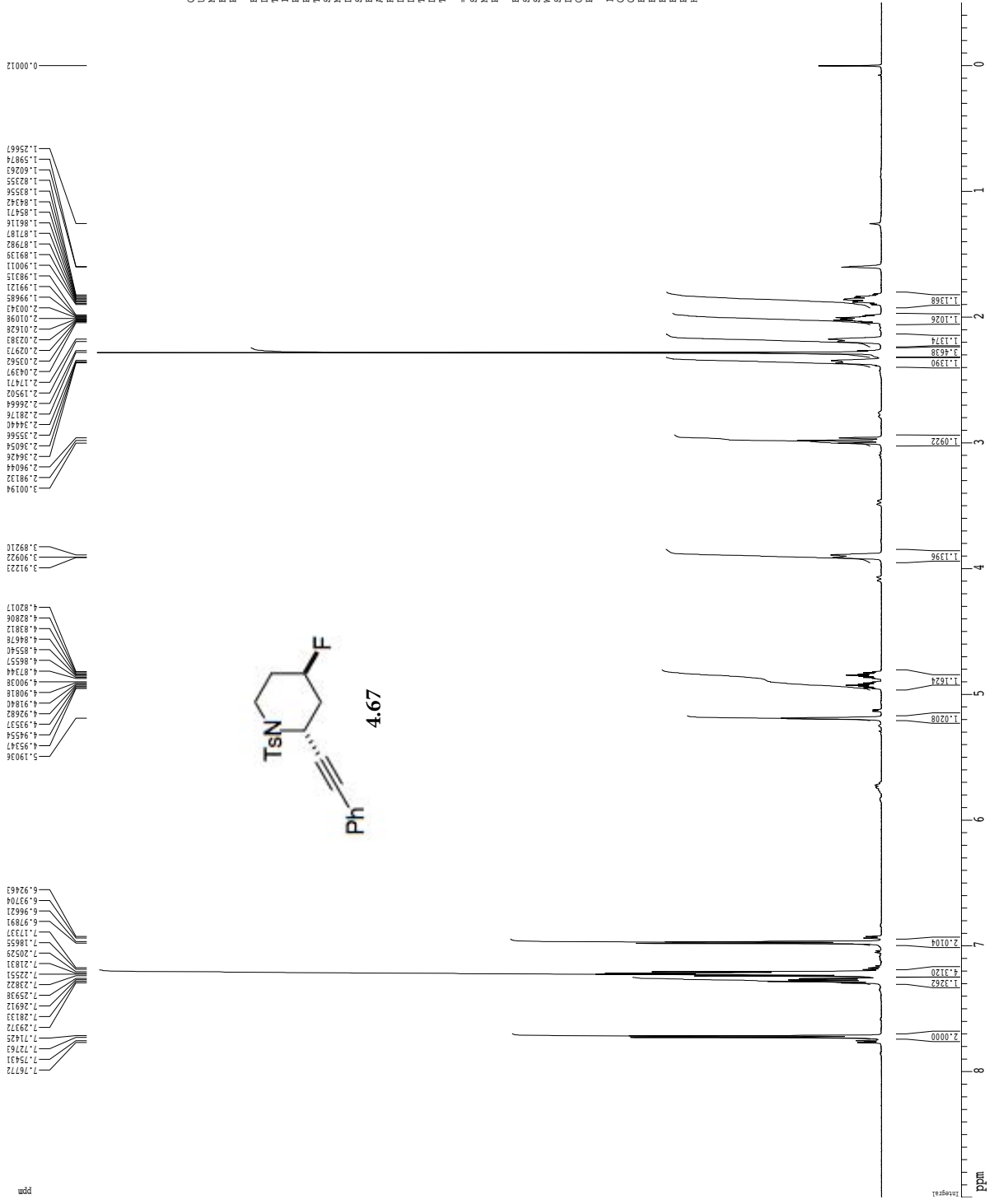
gnoe

ppm



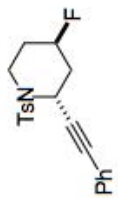


h1.c









9cosy60

4.67

```

Current Data Parameters
USER      RMR
EXPNO    4
PROCNO   1

F2 - Acquisition Parameters
Date_     200007
Time      10.03
INSTRUM   cryo500
PROBHD    5 mm CPCL IH-
PULPROG   zgpg30
SOLVENT   CDCl3
NS         16
DS         16
SWH        8012.820 Hz
FIDRES     3.392510 Hz
AQ         0.127674 sec
RG          512
DM          62.400 usec
DE          6.00 usec
TE         298.0 K
GQ         0.0000000 sec
H1         1.0000000 sec
d13        0.0000000 sec
d16        0.0002000 sec
IN0        0.00012480 sec

===== CHANNEL f1 =====
NUC1       13
P1         7.50 usec
PL1        1.60 dB
SFO1       500.2235015 MHz

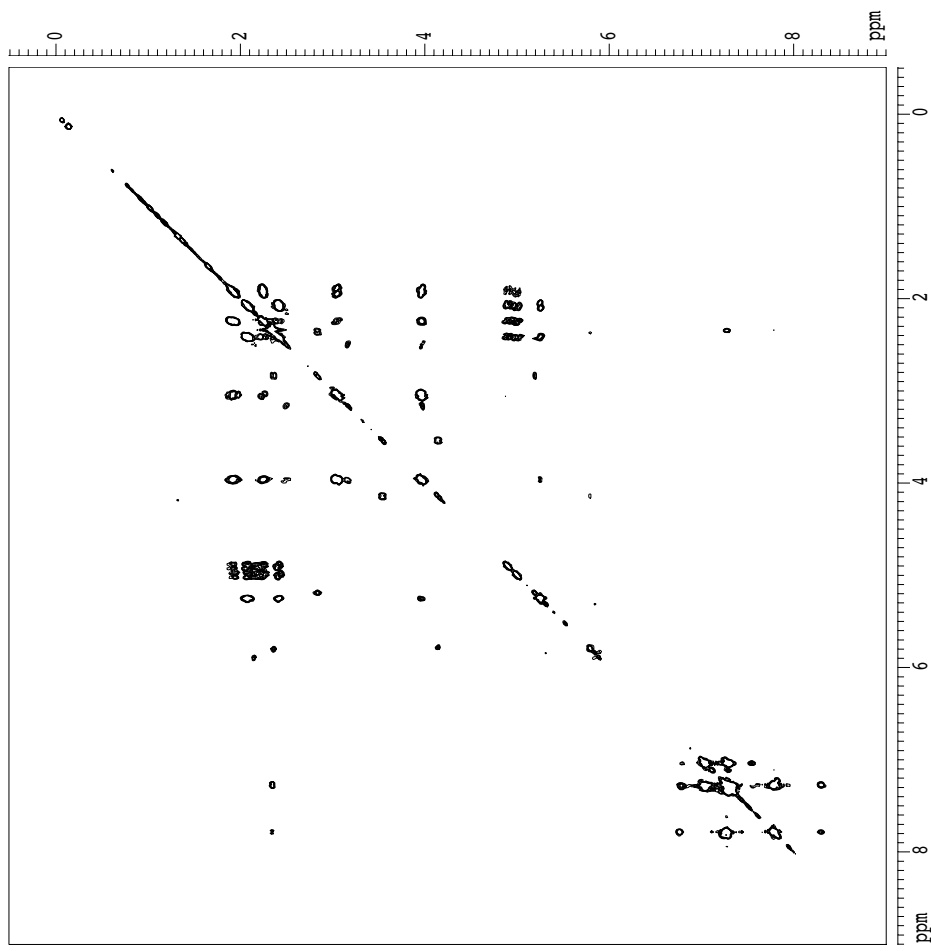
===== GRABBER CHANNEL =====
GRAMZ      size:100
GPX1       0.00 %
GPX2       0.00 %
GPT1       0.00 %
GPT2       0.00 %
GPT3       17.00 %
GPT4       17.00 %
P16        1000.00 usec

F1 - Acquisition parameters
NUC1       13
TD         512
SFO1       500.2235 MHz
FIDRES     15.650040 Hz
SN         16.018 ppm
FWD0E      QF

F2 - Processing parameters
SI         1024
SF         500.2200000 MHz
WDW        SINE
SSB         0
GB          0
PC          1.00

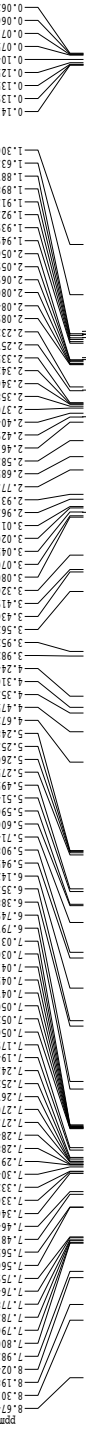
F1 - Processing parameters
SI         16
SF         500.2200000 MHz
WDW        SINE
SSB         0
GB          0

2D NMR plot parameters
CXZ        15.00 cm
FZPLO      15.00 cm
FZP0E      482.2 ppm
FZP1E      -0.509 ppm
FZHI       -254.47 Hz
FZPLO      9.002 ppm
FZP1E      450.114 Hz
FZHI       -254.47 Hz
FZP0E      0.63407 ppm/cm
FZHCN      317.17416 Hz/cm
FZP0E      0.63407 ppm/cm
FZHCN      317.17416 Hz/cm
  
```



gnoe

8.67413 8.0189 8.19871 8.02666 7.98988 7.80061 7.79682 7.78342 7.78171 7.76435 7.75993 7.56991 7.56151 7.54811 7.46416 7.34687 7.33664 7.33253 7.30873 7.29135 7.28187 7.28022 7.27335 7.27058 7.26788 7.25272 7.24300 7.19434 7.18962 7.18522 7.15209 7.05266 7.04792 7.04306 7.03818 7.03619 7.03188 6.79766 6.74902 6.58994 6.53535 6.42409 5.94558 5.90688 5.86911 5.59535 5.51466 5.45271 3.95222 3.92824 3.90284 3.56271 3.45937 3.43997 3.32629 3.08070 3.07039 3.02006 3.01150 2.96133 2.93133 2.87224 2.85826 2.85266 2.61800 2.49252 2.48464 2.37066 2.35809 2.34868 2.34208 2.32726 2.08928 2.08479 2.05996 2.05059 1.94927 1.93921 1.91388 1.89814 1.88718 1.63878 1.30659



Current Data Parameters  
USER khewll1  
NAME KMH-111-09H-P1-2D  
PROCNO 1

Date\_ 20200221  
INSTRUM cryo500  
PROBHD 5 mm CPYCI 1H-  
PULPROG gpcoc2d.prd  
SOLVENT CDCl3  
NS 128  
DS 8  
AQ 4.0954966 sec  
RG 1.28  
DQ 62.400 usec  
TE 298.15 K  
TD 258.4 K  
SFO 500.136160 MHz  
AQ 4.0954966 sec  
RG 1.28  
DQ 62.400 usec  
TE 298.15 K  
TD 258.4 K

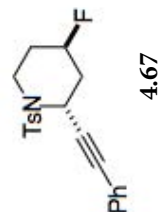
==== CHANNEL f1 =====  
NUC1 1H  
P1 7.50 usec  
PL1 0.00 dB  
PL2 22.50 usec  
PL3 22.50 usec  
PL4 22.50 usec  
PL5 22.50 usec  
PL6 22.50 usec  
PZ1 40000.00 usec  
PZ2 500.372160 dB  
SFO1 500.136160 MHz  
SFO2 61.450 MHz  
SFO3 61.450 MHz  
SFO4 61.450 MHz  
SFO5 61.450 MHz  
SFO6 61.450 MHz  
SFO7 61.450 MHz  
SFO8 61.450 MHz  
SFO9 61.450 MHz  
SFO10 61.450 MHz  
SFO11 61.450 MHz  
SFO12 61.450 MHz  
SFO13 61.450 MHz  
SFO14 61.450 MHz  
SFO15 61.450 MHz  
SFO16 61.450 MHz  
SFO17 61.450 MHz  
SFO18 61.450 MHz  
SFO19 61.450 MHz  
SFO20 61.450 MHz  
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SFO37 61.450 MHz  
SFO38 61.450 MHz  
SFO39 61.450 MHz  
SFO40 61.450 MHz  
SFO41 61.450 MHz  
SFO42 61.450 MHz  
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SFO90 61.450 MHz  
SFO91 61.450 MHz  
SFO92 61.450 MHz  
SFO93 61.450 MHz  
SFO94 61.450 MHz  
SFO95 61.450 MHz  
SFO96 61.450 MHz  
SFO97 61.450 MHz  
SFO98 61.450 MHz  
SFO99 61.450 MHz  
SFO100 61.450 MHz

==== CHANNEL f2 =====  
NUC2 13C  
P2 15.00 usec  
PL2 0.00 dB  
PL3 15.00 usec  
PL4 15.00 usec  
PL5 15.00 usec  
PL6 15.00 usec  
PL7 15.00 usec  
PL8 15.00 usec  
PL9 15.00 usec  
PL10 15.00 usec  
PL11 15.00 usec  
PL12 15.00 usec  
PL13 15.00 usec  
PL14 15.00 usec  
PL15 15.00 usec  
PL16 15.00 usec  
PL17 15.00 usec  
PL18 15.00 usec  
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PL21 15.00 usec  
PL22 15.00 usec  
PL23 15.00 usec  
PL24 15.00 usec  
PL25 15.00 usec  
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PL27 15.00 usec  
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PL39 15.00 usec  
PL40 15.00 usec  
PL41 15.00 usec  
PL42 15.00 usec  
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PL92 15.00 usec  
PL93 15.00 usec  
PL94 15.00 usec  
PL95 15.00 usec  
PL96 15.00 usec  
PL97 15.00 usec  
PL98 15.00 usec  
PL99 15.00 usec  
PL100 15.00 usec

==== GRADIENT CHANNELS =====  
GGRAD1 pulse:100  
GGRAD2 pulse:100  
GGRAD3 pulse:100  
GGRAD4 pulse:100  
GGRAD5 pulse:100  
GGRAD6 pulse:100  
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GGRAD98 pulse:100  
GGRAD99 pulse:100  
GGRAD100 pulse:100

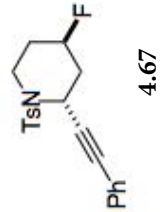
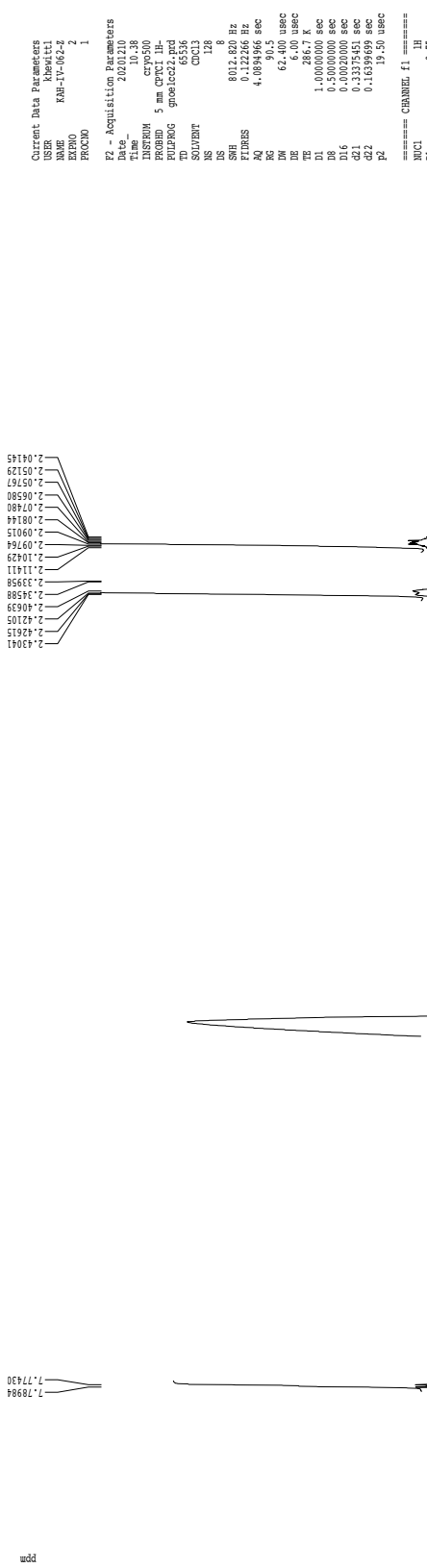
P2 - Processing parameters  
SI 65536  
SF 500.220000 MHz  
RG 1.28  
WDW EM  
SSB 0  
GB 0  
PC 1.00

ID NMR plot parameters  
CX 22.80 cm  
CY 5.00 cm  
CZ 9.0000 mm  
F1 4501.98 Hz  
F2 -4500.00 ppm  
F3 4501.98 Hz  
F4 4501.98 Hz  
F5 4501.98 Hz  
F6 4501.98 Hz  
F7 4501.98 Hz  
F8 4501.98 Hz  
F9 4501.98 Hz  
F10 4501.98 Hz  
F11 4501.98 Hz  
F12 4501.98 Hz  
F13 4501.98 Hz  
F14 4501.98 Hz  
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F16 4501.98 Hz  
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F18 4501.98 Hz  
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F29 4501.98 Hz  
F30 4501.98 Hz  
F31 4501.98 Hz  
F32 4501.98 Hz  
F33 4501.98 Hz  
F34 4501.98 Hz  
F35 4501.98 Hz  
F36 4501.98 Hz  
F37 4501.98 Hz  
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F88 4501.98 Hz  
F89 4501.98 Hz  
F90 4501.98 Hz  
F91 4501.98 Hz  
F92 4501.98 Hz  
F93 4501.98 Hz  
F94 4501.98 Hz  
F95 4501.98 Hz  
F96 4501.98 Hz  
F97 4501.98 Hz  
F98 4501.98 Hz  
F99 4501.98 Hz  
F100 4501.98 Hz



gnoe

ppm



Current Data Parameters  
USER khewitt1  
NAME KM-1V-062-2  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20201210  
Time 10:38  
PROBHD 5 mm CPCT 1H  
PULPROG zgpg30  
POLARIZ gnoe1cc22.prd  
TD 65536  
SOLVENT CDCl3  
NS 128  
DS 8  
SWH 8012.620 Hz  
FIDRES 0.122266 Hz  
AQ 4.089496 sec  
RG 62.00  
DE 6.00 usec  
TE 286.7 K  
D1 1.0000000 sec  
O1 0.0000000 sec  
D16 0.0000000 sec  
d21 0.3377451 sec  
d22 0.1639869 sec  
P2 19.50 usec

==== CHANNEL f1 ====  
NUC1 1H  
P1 9.75 usec  
P2 29.25 usec  
P3 29.00 usec  
P4 29.00 usec  
E29 40000.00 usec  
PL1 1.66 dB  
SFO1 500.228258 MHz  
GPROB 60.00 dB  
SFOB9 9000.00 Hz  
SFOFF9 0.00 Hz

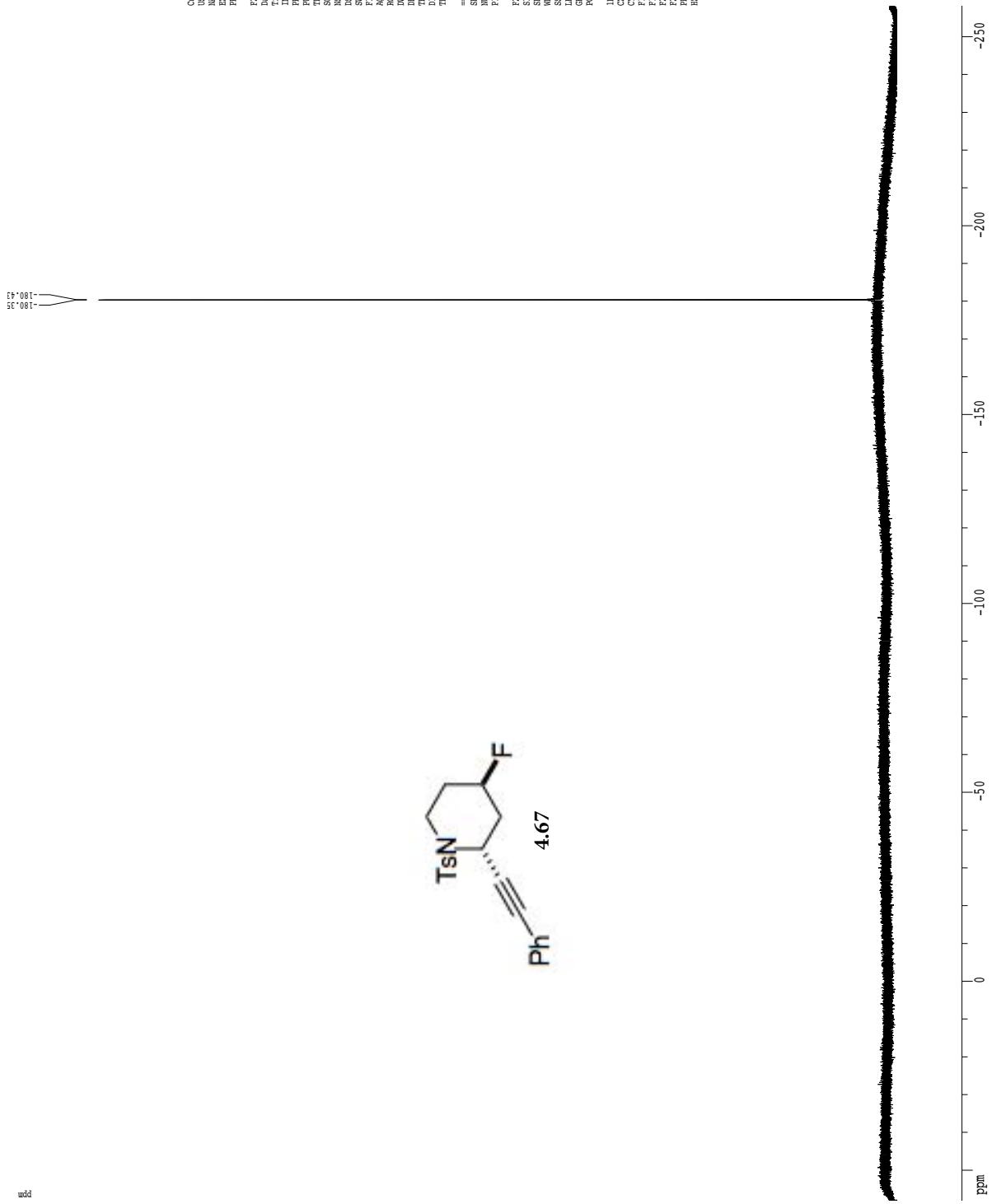
==== CHANNEL CHANNEL2 ====  
SFO10 500.000000 MHz  
SFOB10 9000.00 Hz  
SFOB2 9000.00 Hz  
SFOB3 9000.00 Hz  
SFOB4 9000.00 Hz  
SFOB5 9000.00 Hz  
SFOB6 9000.00 Hz  
SFOB7 9000.00 Hz  
SFOB8 9000.00 Hz  
SFOB9 9000.00 Hz  
SFOB10 9000.00 Hz  
SFOB11 9000.00 Hz  
SFOB12 9000.00 Hz  
SFOB13 9000.00 Hz  
SFOB14 9000.00 Hz  
SFOB15 9000.00 Hz  
SFOB16 9000.00 Hz  
SFOB17 9000.00 Hz  
SFOB18 9000.00 Hz  
SFOB19 9000.00 Hz  
SFOB20 9000.00 Hz  
SFOB21 9000.00 Hz  
SFOB22 9000.00 Hz  
SFOB23 9000.00 Hz  
SFOB24 9000.00 Hz  
SFOB25 9000.00 Hz  
SFOB26 9000.00 Hz  
SFOB27 9000.00 Hz  
SFOB28 9000.00 Hz  
SFOB29 9000.00 Hz  
SFOB30 9000.00 Hz  
SFOB31 9000.00 Hz  
SFOB32 9000.00 Hz  
SFOB33 9000.00 Hz  
SFOB34 9000.00 Hz  
SFOB35 9000.00 Hz  
SFOB36 9000.00 Hz  
SFOB37 9000.00 Hz  
SFOB38 9000.00 Hz  
SFOB39 9000.00 Hz  
SFOB40 9000.00 Hz  
SFOB41 9000.00 Hz  
SFOB42 9000.00 Hz  
SFOB43 9000.00 Hz  
SFOB44 9000.00 Hz  
SFOB45 9000.00 Hz  
SFOB46 9000.00 Hz  
SFOB47 9000.00 Hz  
SFOB48 9000.00 Hz  
SFOB49 9000.00 Hz  
SFOB50 9000.00 Hz  
SFOB51 9000.00 Hz  
SFOB52 9000.00 Hz  
SFOB53 9000.00 Hz  
SFOB54 9000.00 Hz  
SFOB55 9000.00 Hz  
SFOB56 9000.00 Hz  
SFOB57 9000.00 Hz  
SFOB58 9000.00 Hz  
SFOB59 9000.00 Hz  
SFOB60 9000.00 Hz  
SFOB61 9000.00 Hz  
SFOB62 9000.00 Hz  
SFOB63 9000.00 Hz  
SFOB64 9000.00 Hz  
SFOB65 9000.00 Hz  
SFOB66 9000.00 Hz  
SFOB67 9000.00 Hz  
SFOB68 9000.00 Hz  
SFOB69 9000.00 Hz  
SFOB70 9000.00 Hz  
SFOB71 9000.00 Hz  
SFOB72 9000.00 Hz  
SFOB73 9000.00 Hz  
SFOB74 9000.00 Hz  
SFOB75 9000.00 Hz  
SFOB76 9000.00 Hz  
SFOB77 9000.00 Hz  
SFOB78 9000.00 Hz  
SFOB79 9000.00 Hz  
SFOB80 9000.00 Hz  
SFOB81 9000.00 Hz  
SFOB82 9000.00 Hz  
SFOB83 9000.00 Hz  
SFOB84 9000.00 Hz  
SFOB85 9000.00 Hz  
SFOB86 9000.00 Hz  
SFOB87 9000.00 Hz  
SFOB88 9000.00 Hz  
SFOB89 9000.00 Hz  
SFOB90 9000.00 Hz  
SFOB91 9000.00 Hz  
SFOB92 9000.00 Hz  
SFOB93 9000.00 Hz  
SFOB94 9000.00 Hz  
SFOB95 9000.00 Hz  
SFOB96 9000.00 Hz  
SFOB97 9000.00 Hz  
SFOB98 9000.00 Hz  
SFOB99 9000.00 Hz  
SFOB100 9000.00 Hz

F2 - Processing parameters  
SI 65536  
SF 500.228258 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00  
ID NMR plot parameters  
CX 22.88 cm  
CY 50.00 cm  
FID 1.0000000000000000  
F1P 450.0000000000000000  
F2P -0.5000000000000000  
F2 -250.11111111111111 Hz  
PPHMM 0.4166666666666667  
HSCN 208.42500000000000 Hz/cm



f19.c

8



Current Data Parameters  
USER khovatt1  
NAME RMH-IV-662\_F  
EXPNO 2  
PROCNO 1

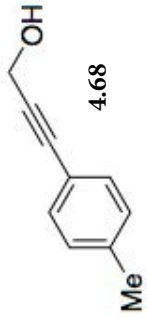
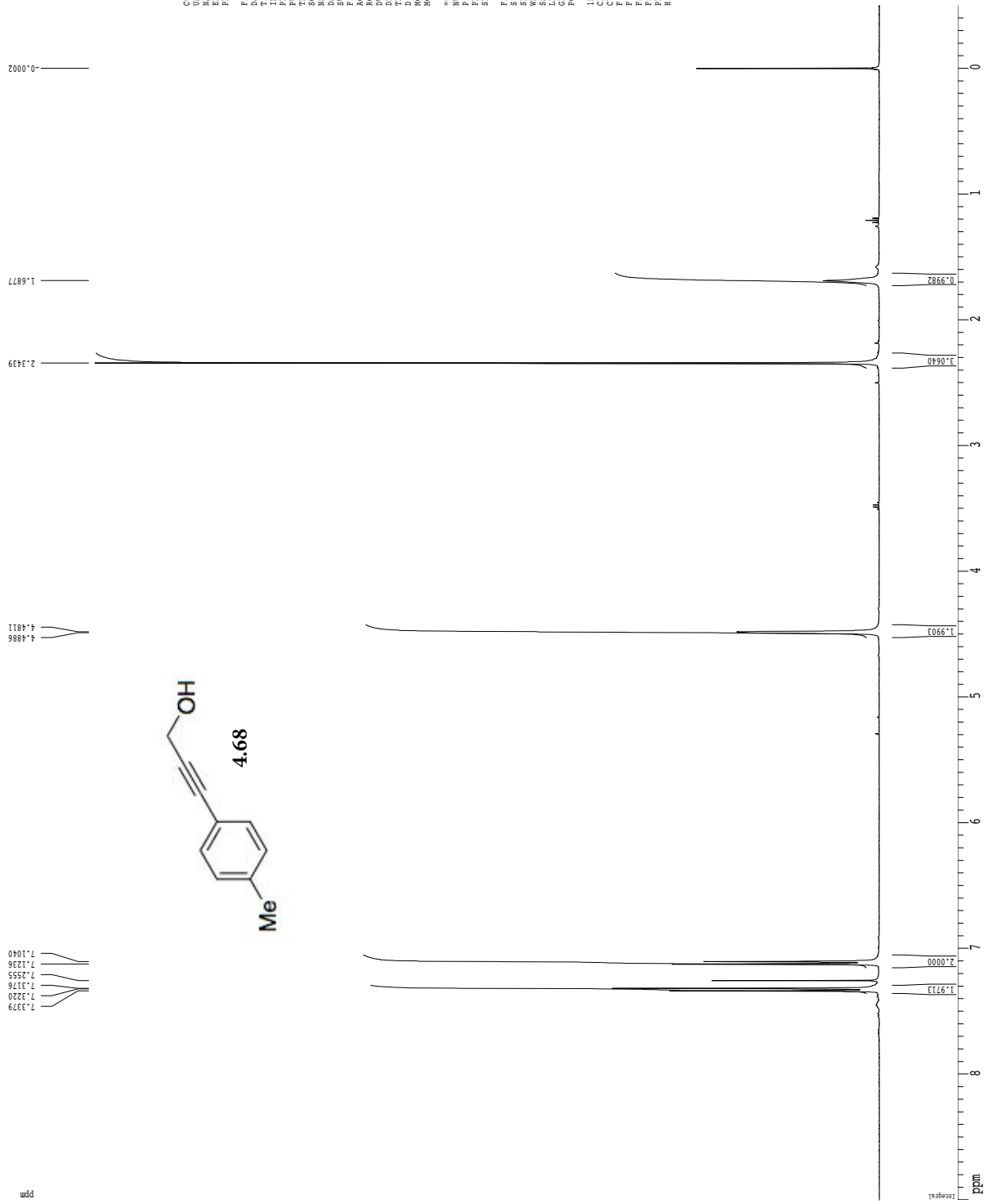
F2 - Acquisition Parameters  
Date\_ 20160908  
Time 15:12  
INSTRUM av600  
PROBHD 5 mm CPBBO BB-  
PULPROG zgpg30  
AQ 1.112  
SOLVENT CDCl3  
NS 16  
DS 2  
SHF 175.71422 MHz  
NUC1 13C  
MAG 0.327031686 G  
RG 515  
DM 2.800 usec  
DE 18.000 usec  
TE 300.2 K  
D1 3.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO 500.136195 MHz  
NUC1 13C  
P1 18.25 usec

F2 - Processing parameters  
SI 32768  
SF 564.636412 MHz  
WDW no  
SSB 0  
GB 0  
PC 1.00

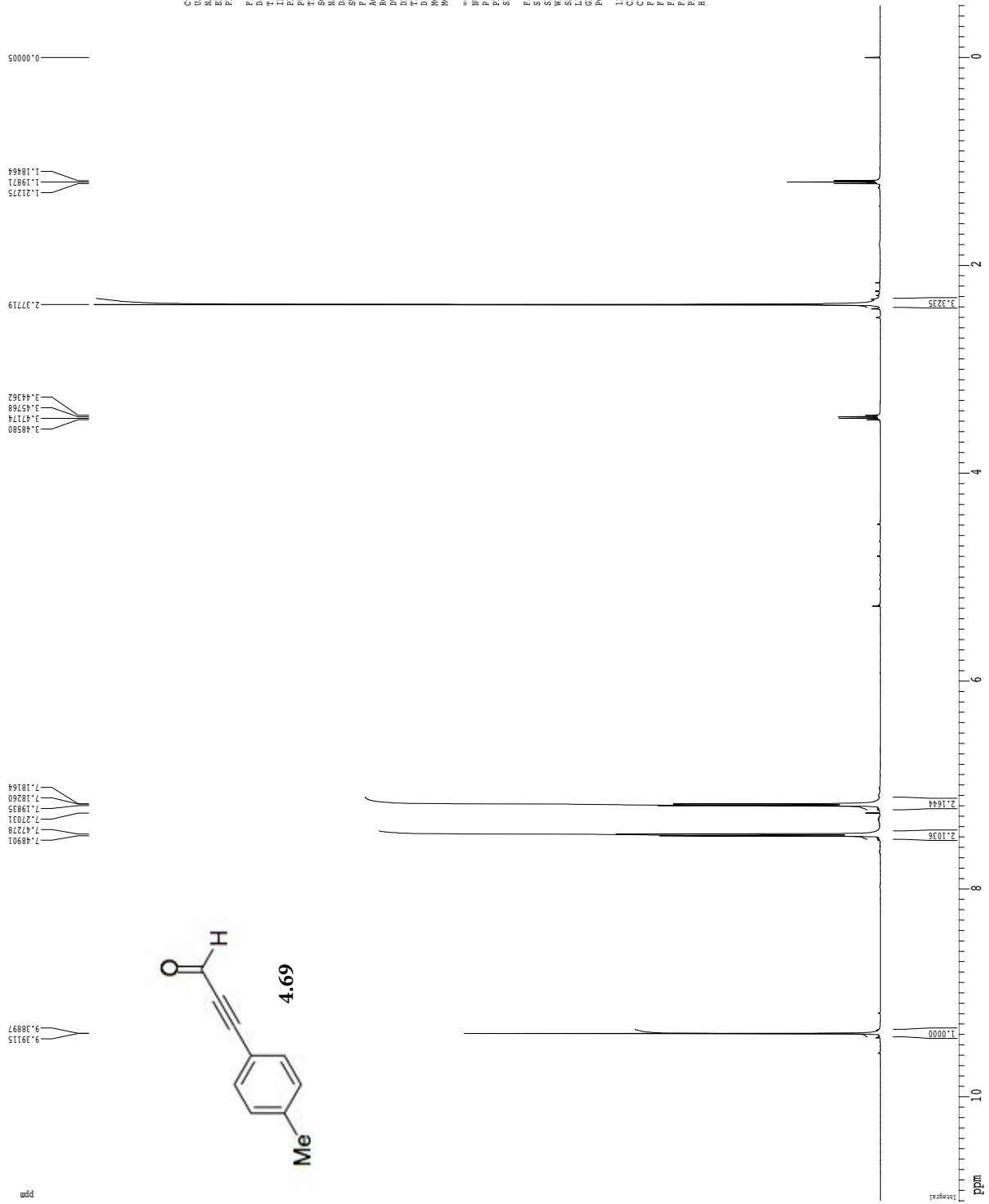
ID MR plot parameters  
CT 4.00 cm  
CX 15.00  
FLP 58.057 ppm  
F1 32784.16 Hz  
F2P -258.174 ppm  
F3P 13.86459 Hz  
FRACM 13.86459 Hz/cm  
HZCM 7832.08057 Hz/cm

1H spectrum



Current Data Parameters  
 USER kowatt1  
 NAME ACH-4-132-136-check  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20100116  
 Time 11:02:16  
 INSTRUM spect  
 PROBHD 5 mm QNP H/F/PP  
 PULPROG zgpg30  
 TD 65536  
 SFO 400.130333  
 SOLVENT CDCl3  
 NS 8  
 DS 8  
 SWH 6410.256 Hz  
 FIDRES 0.097813 Hz  
 AQ 5.1118579 sec  
 SFO 400.130333 MHz  
 DQ 78.000 uSAC  
 DE 4.50 uSAC  
 TE 300.2 K  
 D1 0.10000000 sec  
 D11 0.02000000 sec  
 D12 0.02000000 sec  
 D13 0.02000000 sec  
 D14 0.02000000 sec  
 D15 0.02000000 sec  
 D16 0.02000000 sec  
 D17 0.02000000 sec  
 D18 0.02000000 sec  
 D19 0.02000000 sec  
 D20 0.02000000 sec  
 D21 0.02000000 sec  
 D22 0.02000000 sec  
 D23 0.02000000 sec  
 D24 0.02000000 sec  
 D25 0.02000000 sec  
 D26 0.02000000 sec  
 D27 0.02000000 sec  
 D28 0.02000000 sec  
 D29 0.02000000 sec  
 D30 0.02000000 sec  
 D31 0.02000000 sec  
 D32 0.02000000 sec  
 D33 0.02000000 sec  
 D34 0.02000000 sec  
 D35 0.02000000 sec  
 D36 0.02000000 sec  
 D37 0.02000000 sec  
 D38 0.02000000 sec  
 D39 0.02000000 sec  
 D40 0.02000000 sec  
 D41 0.02000000 sec  
 D42 0.02000000 sec  
 D43 0.02000000 sec  
 D44 0.02000000 sec  
 D45 0.02000000 sec  
 D46 0.02000000 sec  
 D47 0.02000000 sec  
 D48 0.02000000 sec  
 D49 0.02000000 sec  
 D50 0.02000000 sec  
 D51 0.02000000 sec  
 D52 0.02000000 sec  
 D53 0.02000000 sec  
 D54 0.02000000 sec  
 D55 0.02000000 sec  
 D56 0.02000000 sec  
 D57 0.02000000 sec  
 D58 0.02000000 sec  
 D59 0.02000000 sec  
 D60 0.02000000 sec  
 D61 0.02000000 sec  
 D62 0.02000000 sec  
 D63 0.02000000 sec  
 D64 0.02000000 sec  
 D65 0.02000000 sec  
 D66 0.02000000 sec  
 D67 0.02000000 sec  
 D68 0.02000000 sec  
 D69 0.02000000 sec  
 D70 0.02000000 sec  
 D71 0.02000000 sec  
 D72 0.02000000 sec  
 D73 0.02000000 sec  
 D74 0.02000000 sec  
 D75 0.02000000 sec  
 D76 0.02000000 sec  
 D77 0.02000000 sec  
 D78 0.02000000 sec  
 D79 0.02000000 sec  
 D80 0.02000000 sec  
 D81 0.02000000 sec  
 D82 0.02000000 sec  
 D83 0.02000000 sec  
 D84 0.02000000 sec  
 D85 0.02000000 sec  
 D86 0.02000000 sec  
 D87 0.02000000 sec  
 D88 0.02000000 sec  
 D89 0.02000000 sec  
 D90 0.02000000 sec  
 D91 0.02000000 sec  
 D92 0.02000000 sec  
 D93 0.02000000 sec  
 D94 0.02000000 sec  
 D95 0.02000000 sec  
 D96 0.02000000 sec  
 D97 0.02000000 sec  
 D98 0.02000000 sec  
 D99 0.02000000 sec  
 D100 0.02000000 sec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 uSAC  
 PL1 0.00 dB  
 SFO1 400.130333 MHz  
 F2 - Processing parameters  
 SI 32768  
 SF 400.130333 MHz  
 DS 8  
 WDW EM  
 SSB 0  
 GB 0  
 PC 2.00  
 LD MR p/ct parameters  
 CX 22.80 cm  
 CY 15.00 cm  
 F1 3001.17 Hz  
 F2 -0.500 ppm  
 F3 -200.06 Hz  
 F4 164.72886 Hz/cm  
 F5 164.72886 Hz/cm

1H spectrum

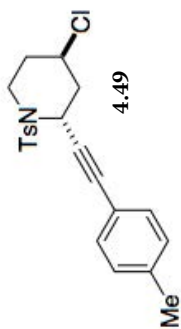


Current Data Parameters  
 USER: khmett1  
 NAME: ACM-158c1  
 EXPNO: 1  
 PROCNO: 1  
 P2 - Acquisition Parameters  
 Date\_: 20200214  
 Time: 15:59  
 PROBNM: gms00  
 PROCNO: 5  
 PULPROG: zg30  
 TD: 8178  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 500  
 SFE: 8012.820 Hz  
 FIDRES: 0.098044 Hz  
 AQ: 5.1998774 sec  
 RG: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 0.10000000 sec  
 T2: 0.00000000 sec  
 MCHRES: 0.00000000 sec  
 MCORE: 0.00500000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -5.00 dB  
 SFO1: 498.654919 MHz  
 P2 - Processing Parameters  
 SI: 65536  
 SF: 498.6500217 MHz  
 WDM: EM  
 LB: 0 Hz  
 GB: 0 Hz  
 PC: 1.00  
 ID: NMR P1 of Parameters  
 CY: 22.80 cm  
 CV: 15.00 cm  
 PIP: 11.000 ppm  
 F1: 5487.35 Hz  
 F2: 2443.67 Hz  
 PPMCH: 0.50439 ppm/cm  
 HZCH: 251.61296 Hz/cm









```

Current Data Parameters
USER      Kheshti1
NAME      KAH-III-192-SH-2
EXPNO     6
PROCNO    1

P1 - Acquisition Parameters
Date_     20200716
Time      17.34
INSTRUM   cryo500
PROBHD    5 mm CRYO-
PULPROG   zgpg30
TD        2048
SOLVENT   CDCl3
NS         1
DS         16
SWH        8013.81 Hz
FIDRES     3.912510 Hz
AQ         0.1278452 sec
RG         181
DM         62.400 usec
DE         298.0 K
TE         298.0 K
d0         0.40000300 sec
d1         1.40000000 sec
d13        0.00000300 sec
d16        0.00020000 sec
d10        0.00014000 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
PC         0.00 usec
SFO1       500.225015 MHz

===== GRADIENT CHANNEL =====
GPRAM1    sine,1.00
GP12      0.00 %
GP22      0.00 %
GP32      0.00 %
GP42      0.00 %
GP52      17.00 %
GP62      100.00 usec

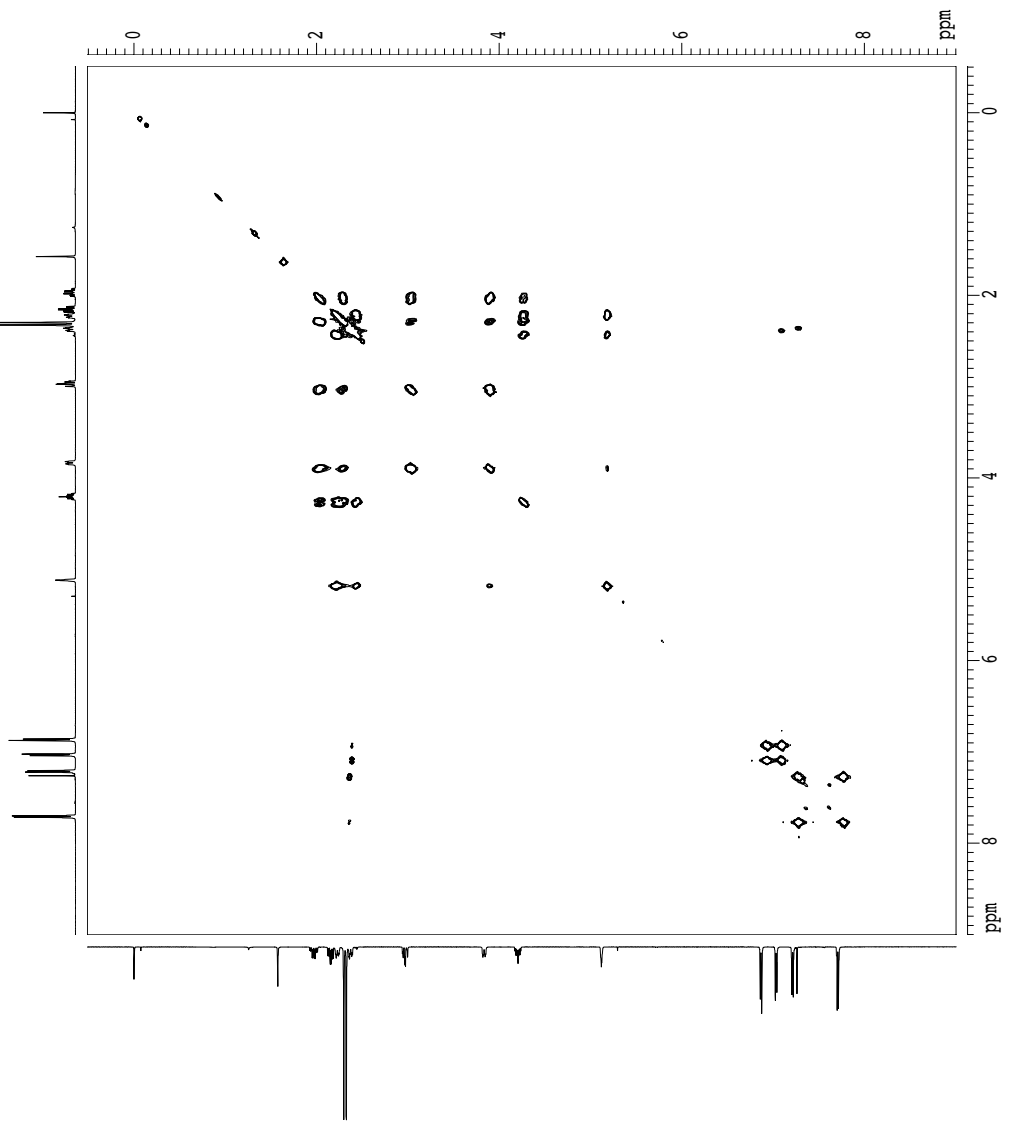
P2 - Acquisition parameters
NUC2       13C
P2         5.12 usec
PC         0.00 usec
SFO2       125.760320 MHz
FIDRES     15.650140 Hz
SN         16.018 ppm
PMD00B     0

P2 - Processing parameters
SI         1
SF         500.2200000 MHz
WDW        EM
SSB        0
GB         0.00 Hz
PC         1.00

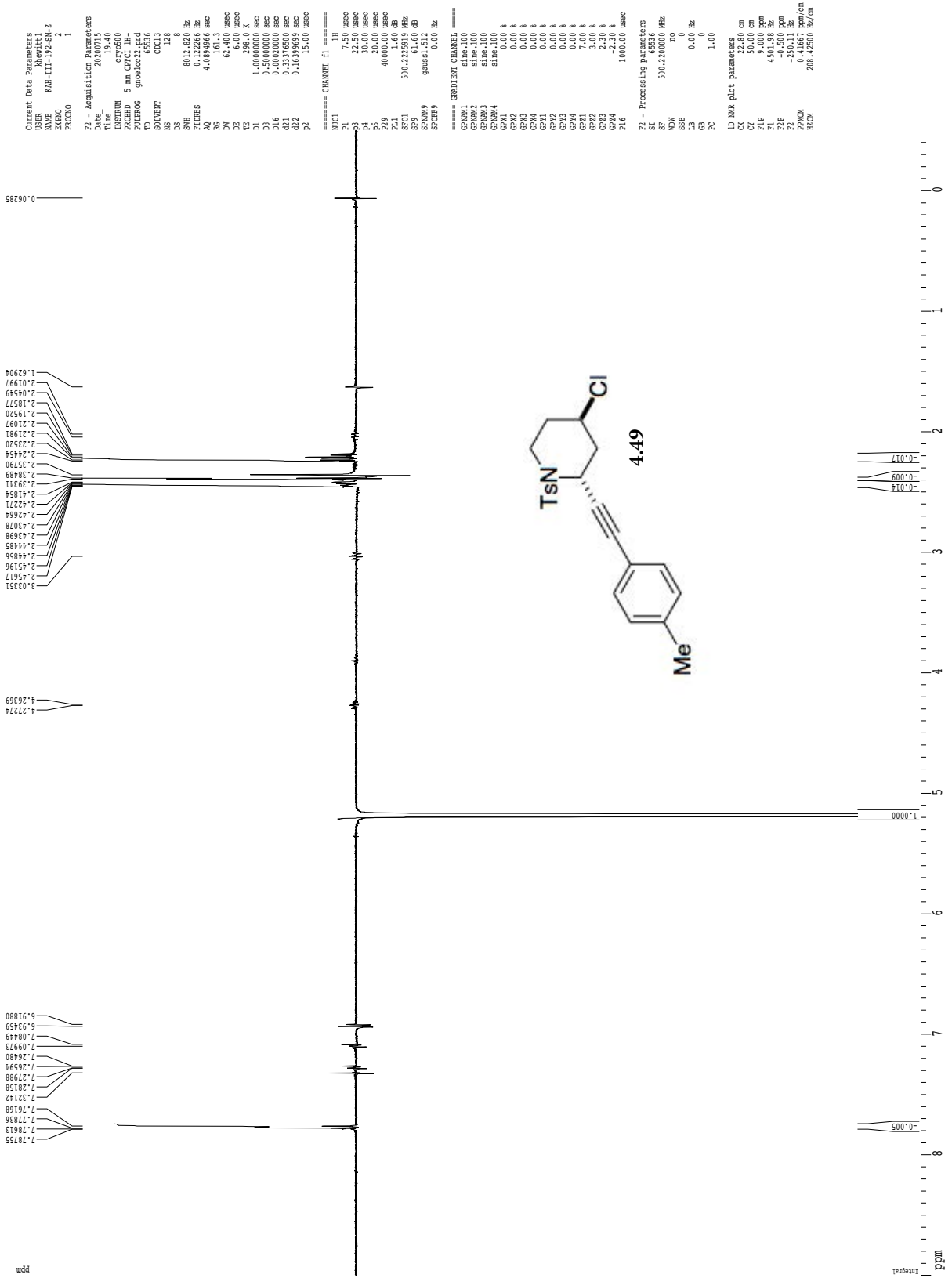
P1 - Processing parameters
SI         1
SF         500.2200000 MHz
WDW        EM
SSB        0
GB         0.00 Hz

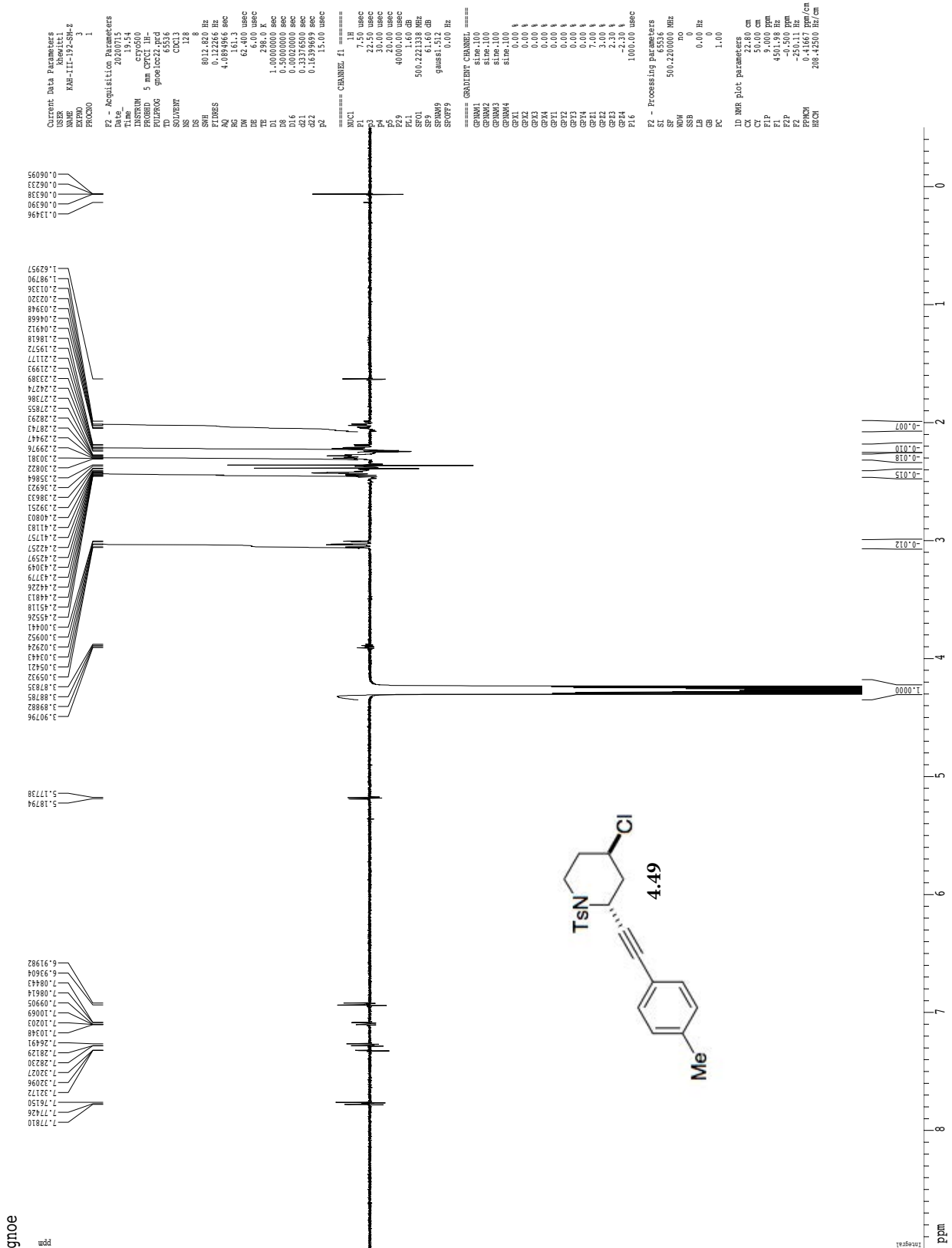
2D NMR Plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2P1Q0    5.002 ppm
F2P1Q1    45.056 ppm
F2P1R0    0.5169 ppm
F2P1R1    -254.47 Hz
F1P1Q0    5.002 ppm
F1P1Q1    4503.14 Hz
F1P1R0    0.509 ppm
F1P1R1    -254.47 Hz
F2P1QM0   0.63407 ppm/cm
F2P1QM1   317.17416 Hz/cm
F1P1RM0   0.63407 ppm/cm
F1P1RM1   317.17416 Hz/cm

```

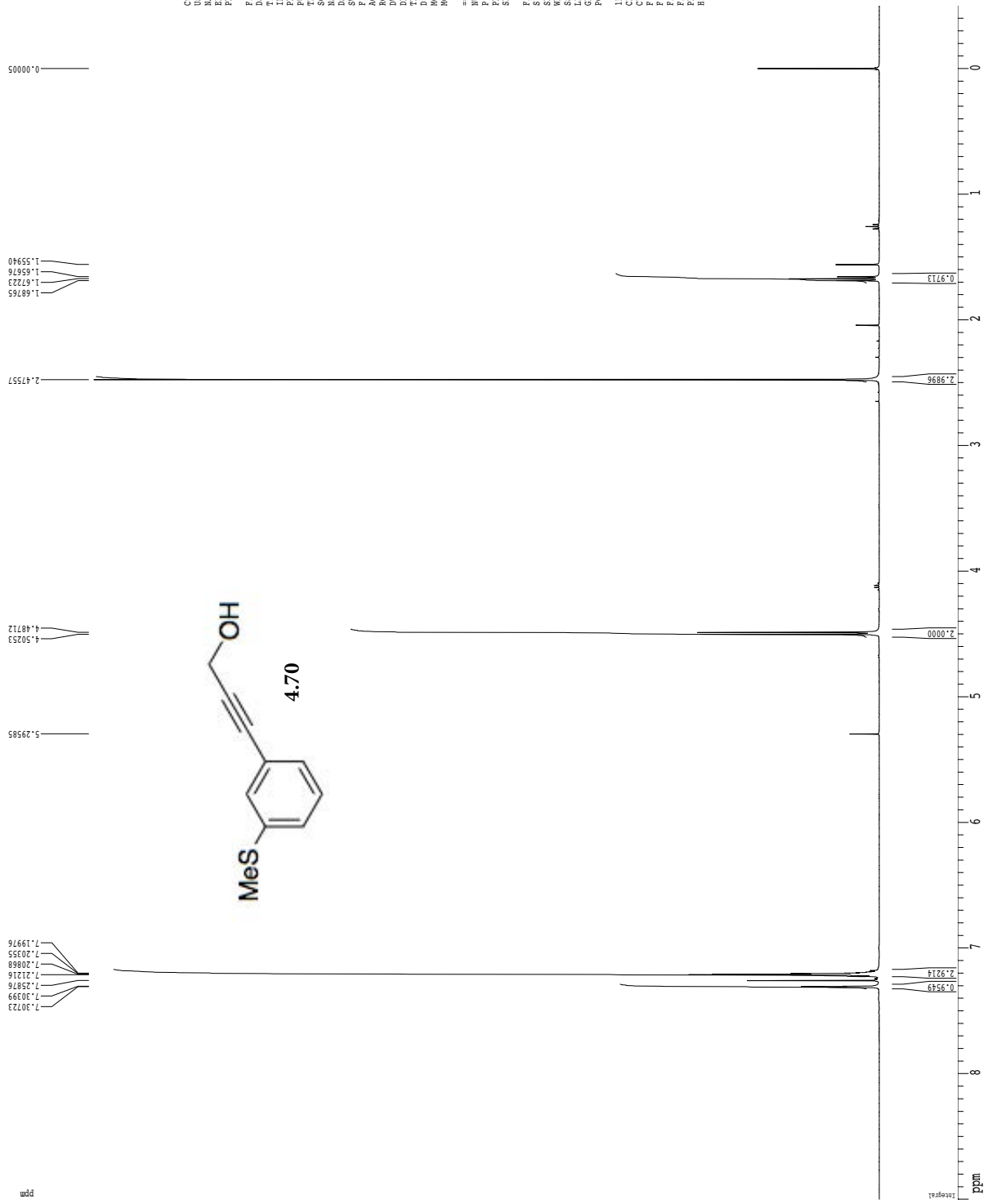


gnoe





1H spectrum



Current Data Parameters  
 USER: Komettt  
 NAME: ACH-164-b1g  
 EXNO: 1  
 PROCD: 1

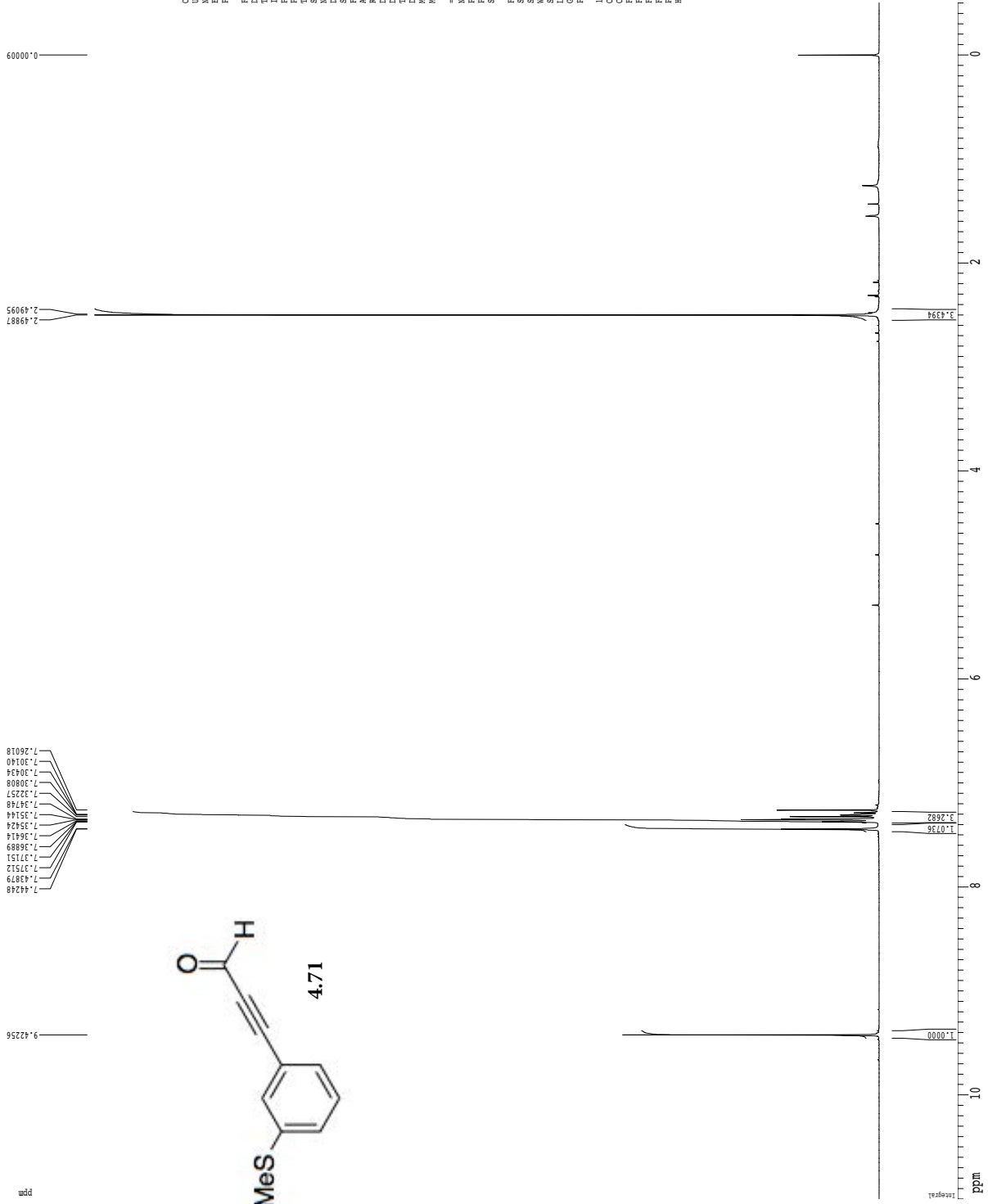
F2 - Acquisition Parameters  
 Date\_: 20200306  
 Time: 9:36  
 INSTR: spect  
 PROCNO: 5  
 PULPROG: zgpg30  
 TD: 65536  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 400.1300218 MHz  
 SHF: 6410.256 Hz  
 FIDRES: 0.097813 Hz  
 AQ: 5.118517 sec  
 RG: 384.000  
 DW: 78.000 usec  
 DE: 4.50 usec  
 TE: 298.1 K  
 T1: 0.100000 sec  
 T2: 0.000000 sec  
 T3: 0.000000 sec  
 MCHRG: 0.000000 sec  
 MCHRG: 0.000000 sec

===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -1.10 dB  
 SFO1: 400.1326009 MHz

F2 - Processing Parameters  
 SI: 65536  
 SF: 400.1300218 MHz  
 WHW: EM  
 LB: 0.30 Hz  
 GB: 0  
 PC: 2.00

LD NMR P1/Q2 Parameters  
 CY: 22.80 cm  
 CV: 15.00 cm  
 FIP: 9.000 ppm  
 FL: 3600.10 Hz  
 F2: 400.1300218 MHz  
 F3: -2000.00 Hz  
 PPRM1: 0.41667 ppm/cm  
 HZCM: 166.72086 Hz/cm

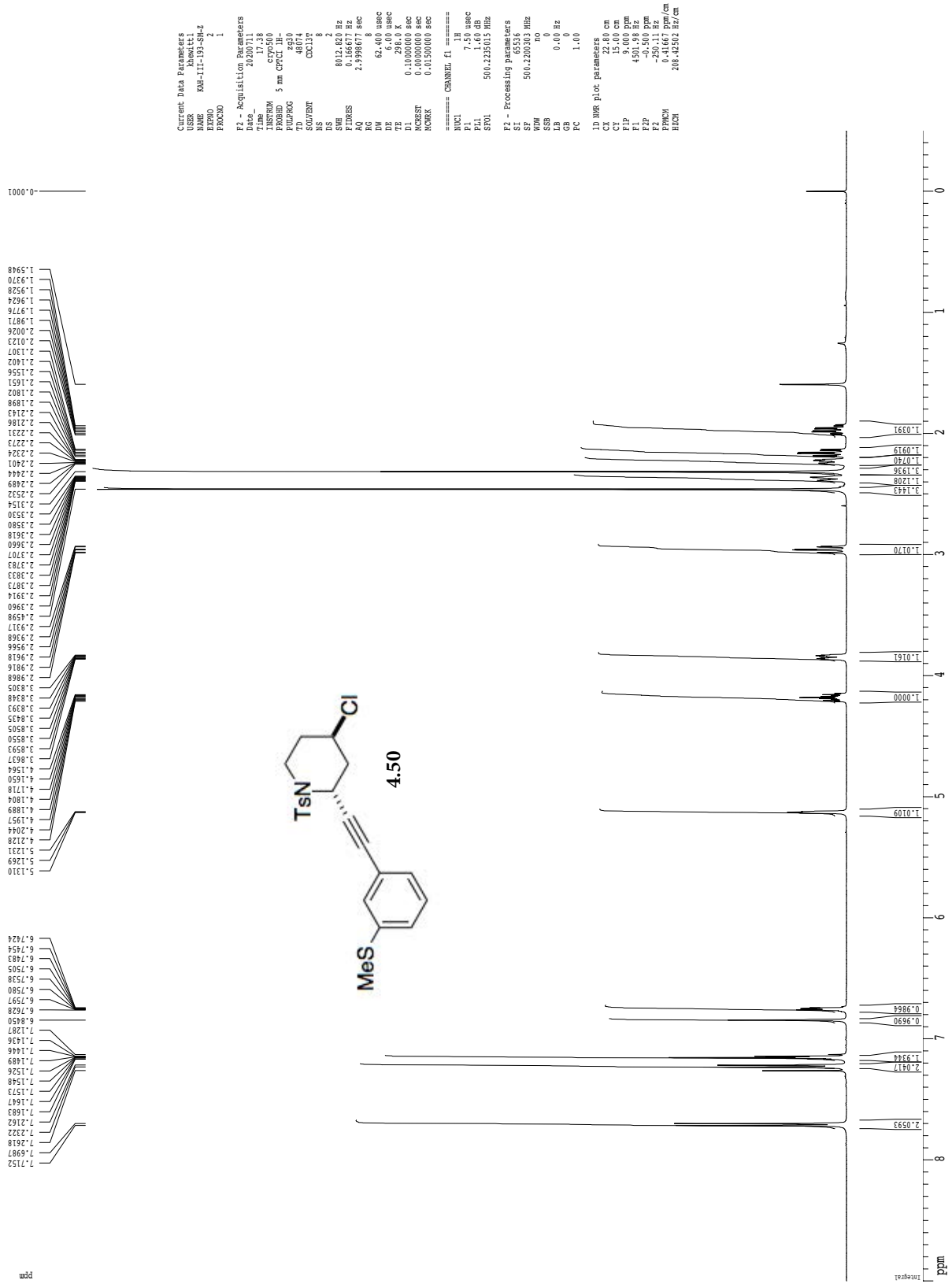
1H spectrum



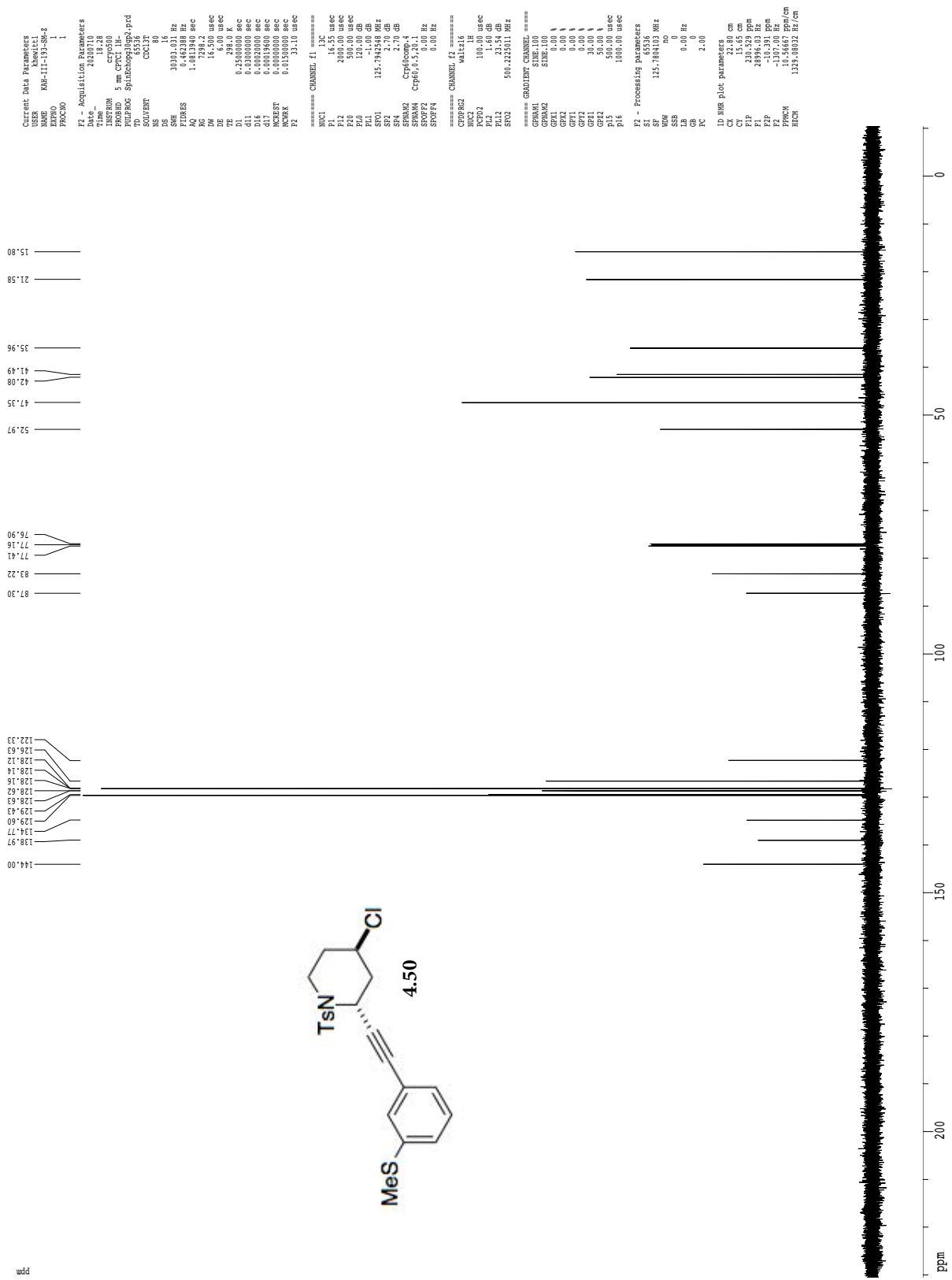
```

Current Data Parameters
=====
USER      khawit11
NAME      ACP-1-159-checking
EXPNO     1
PROCNO    1
Date_     20100116
Time      15
INSTRUM   dx400
PROBHD    5 mm QNP 1H/1
PULPROG   zgpg30
CQ1       13.625
SOLVENT   CDCl3
NS         8
DS         2
AQ         6310.2 Hz
F1RES     0.497113 Hz
F2RES     5.1118579 Hz
RG         374.7
AQ1       7.0000000 usec
DE         4.50 usec
TE         298.0 K
DL        0.1000000 sec
PC        0.1000000 sec
=====
===== CHANNEL f1 =====
NUC1       13C
P1         12.00 usec
PL1        -1.10 dB
SFO1      400.1326009 MHz
=====
F2 - Processing parameters
SI         400.1300214 MHz
SF         400.1300214 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         2.00
=====
ID NAME plot parameters
CA         22.80 cm
CT         11.40 cm
FLP        11.000 cm
F1         4401.43 Hz
F2P        -0.500 ppm
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         201.813498 Hz/cm
    
```

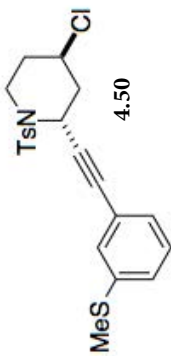
1H spectrum



Z-restored spin-echo 13C spectrum with 1H decoupling







gcosy

```

Current Data Parameters
USER      Khevit11
NAME      KAH-III-193-SH-2
EXPNO    6
PROCNO    1

P1 - Acquisition Parameters
Date_     20200713
Time      18:43
INSTRUM   cryso00
PROBHD    5 mm cryoProbe
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1
DS         16
SWH        8013.85 Hz
FIDRES    0.1278452 Hz
AQ         0.1278452 sec
RG         228.1
DM         62.400 usec
DE         228.0 usec
TE         298.0 K
d0         0.0000300 sec
d1         1.0000000 sec
d13        0.0000300 sec
d16        0.0002000 sec
d10        0.0001400 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
PC         0.00 usec
SFO1       500.225015 MHz

===== GRADIENT CHANNEL =====
GPRAM1    sine,1.00
GPRMZ     0.00 usec
GPRZ      0.00 usec
GPR1      0.00 usec
GPR2      0.00 usec
GPR3      17.00 usec
GPR4      1000.00 usec

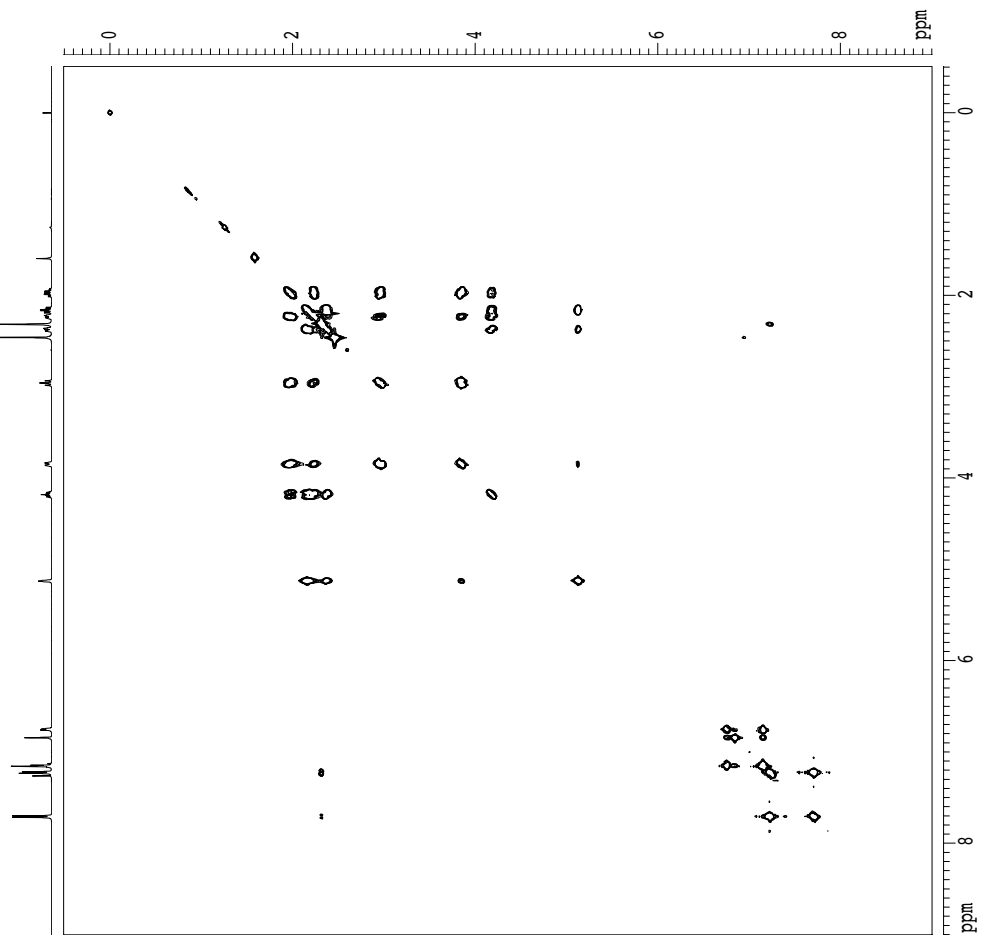
P2 - Acquisition parameters
NUC2       13C
P2         7.50 usec
PC         0.00 usec
SFO2       125.760350 MHz

===== CHANNEL f2 =====
GPRAM2    sine,1.00
GPRMZ     0.00 usec
GPRZ      0.00 usec
GPR1      0.00 usec
GPR2      0.00 usec
GPR3      17.00 usec
GPR4      1000.00 usec

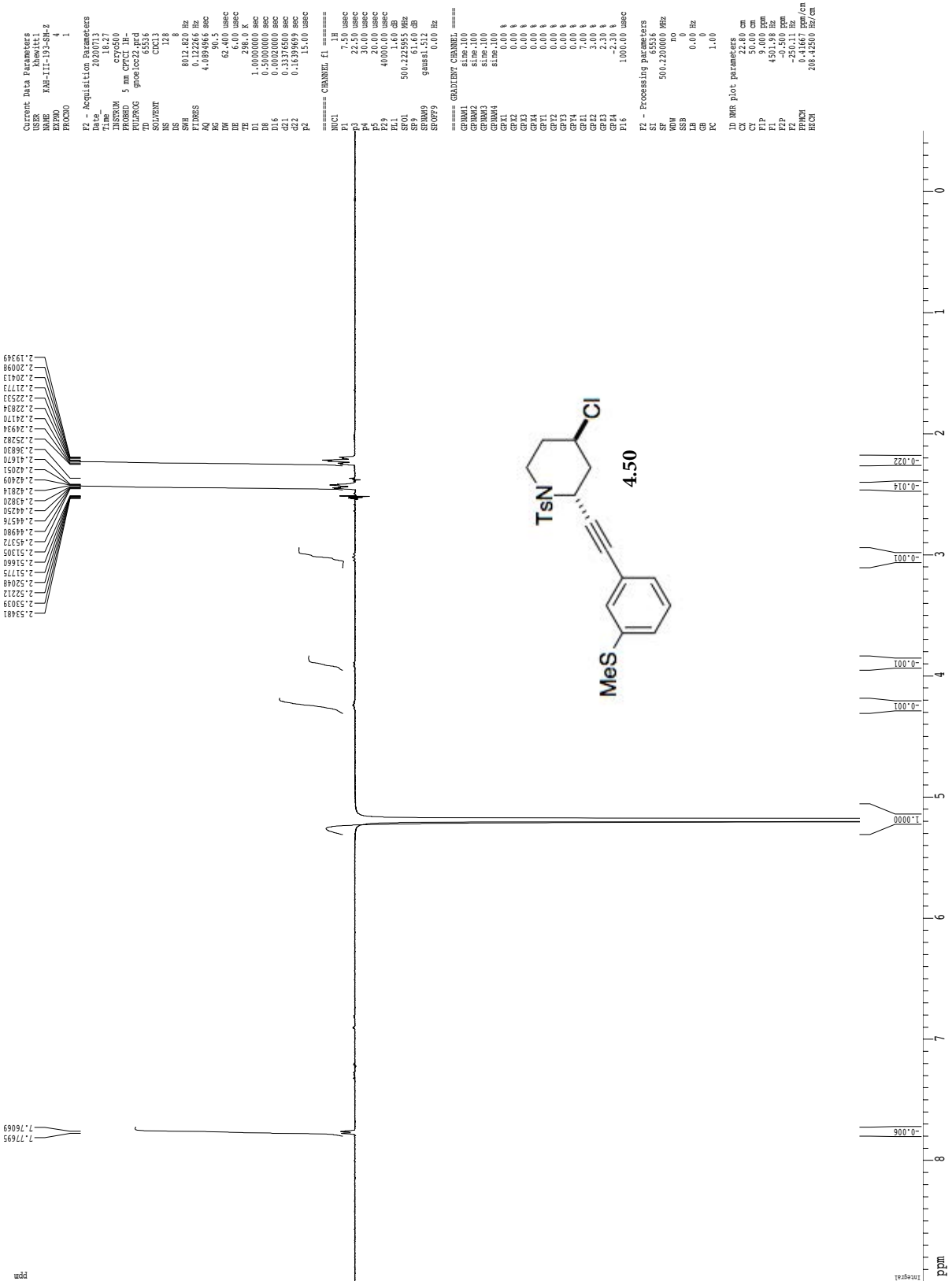
P1 - Processing parameters
SI         1024
SF         500.2203309 MHz
WDW        EM
SSB        0
GB         0.00 Hz
PC         1.00

P2 - Processing parameters
SI         1024
SF         500.2203309 MHz
WDW        EM
SSB        0
GB         0.00 Hz
PC         1.00

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO     5.003 ppm
F2FID     450.508 Hz
F2NUC     13C
F2H1      -254.07 Hz
F1PLO     5.003 ppm
F1LO      4503.54 Hz
F1PH1     0.508 ppm
F1PH2     -2.000 ppm
F2PRGM    0.63407 ppm/cm
F2PRCM    317.17413 Hz/cm
F1PRGM    0.63407 ppm/cm
F1PRCM    317.17413 Hz/cm
  
```

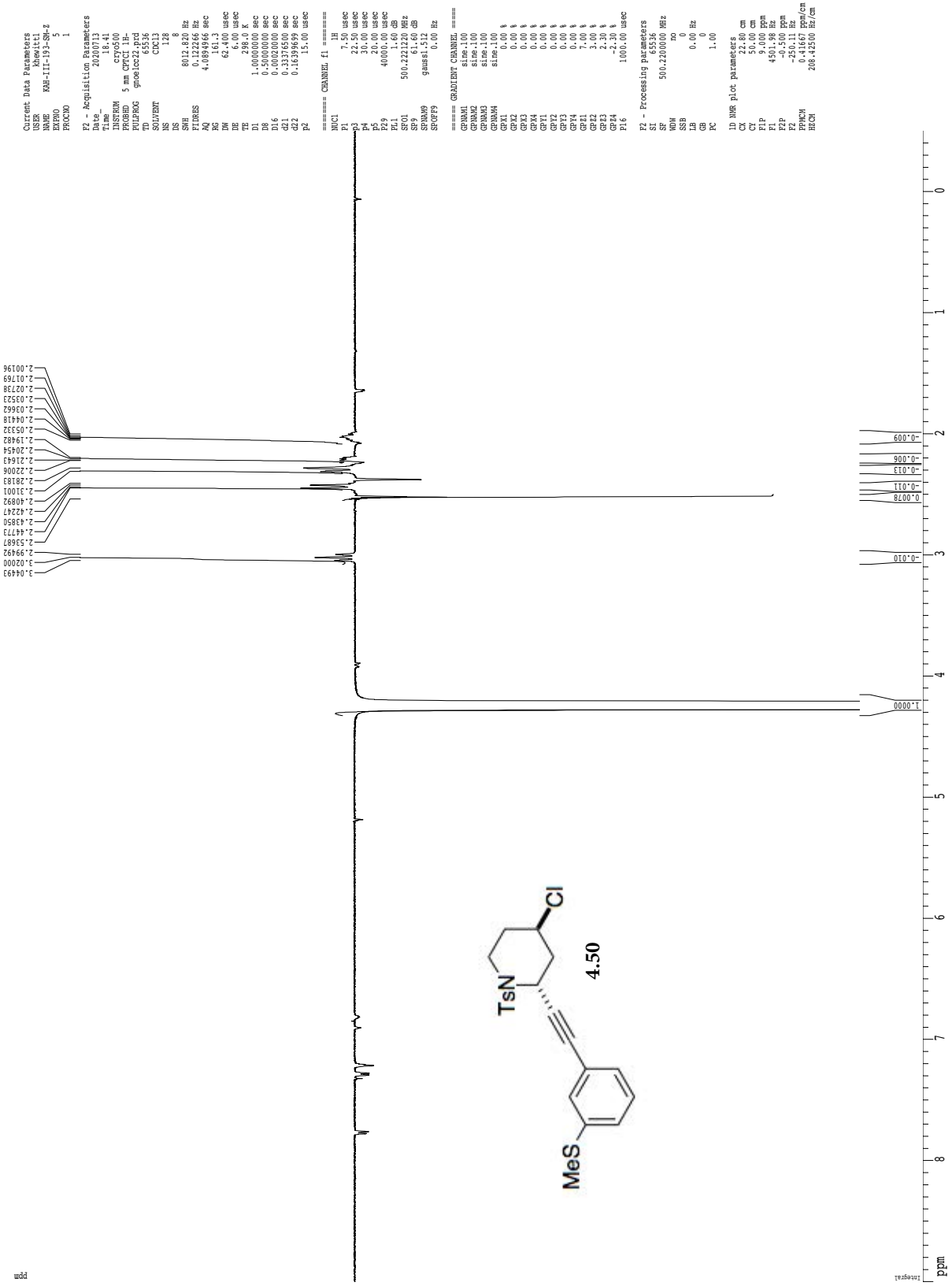


gnoe

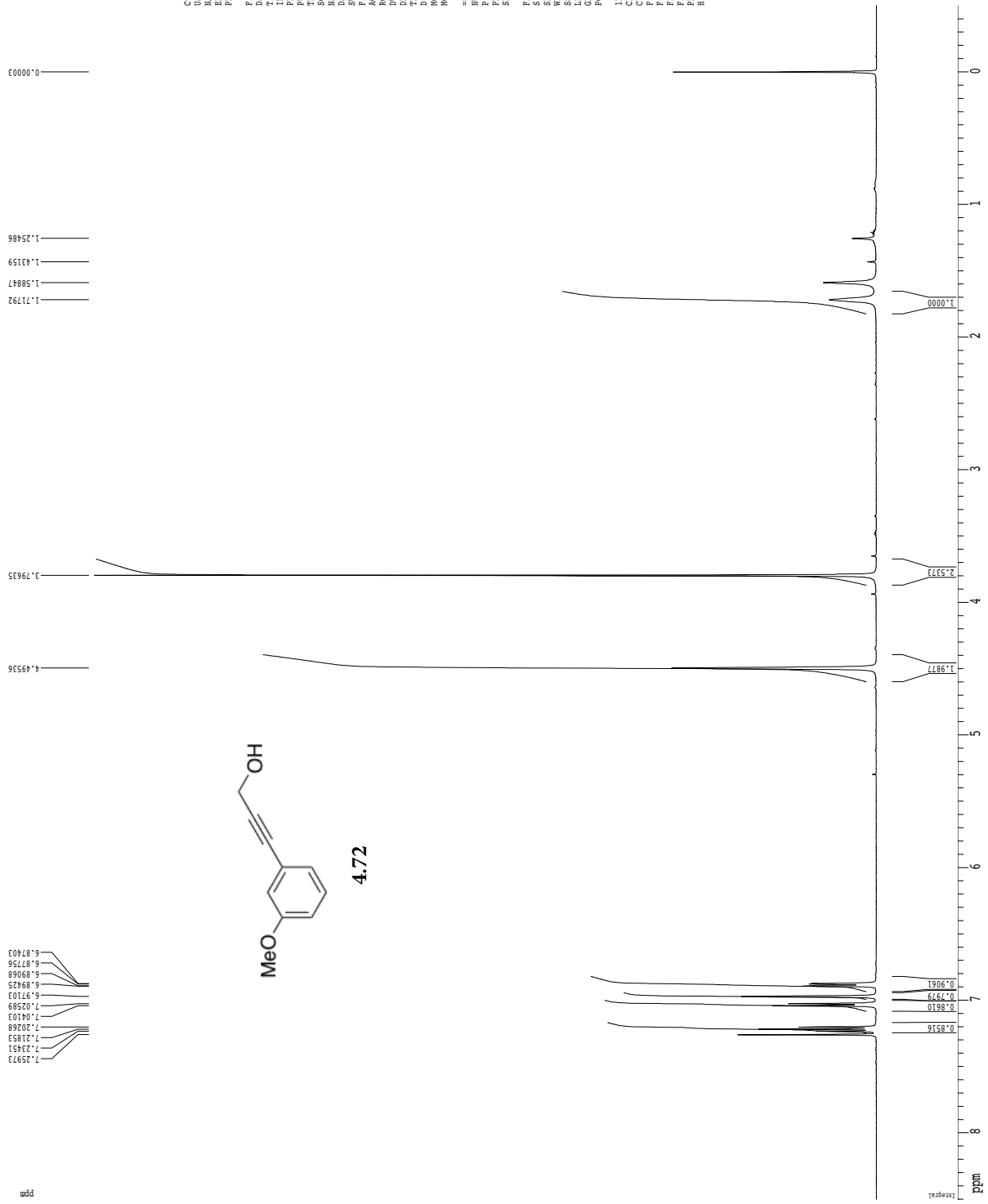


gnoe

100



1H spectrum



Current Data Parameters  
 USER: matus  
 NAME: AOM-110-0-b-2  
 EXNO: 1  
 PROCNO: 1

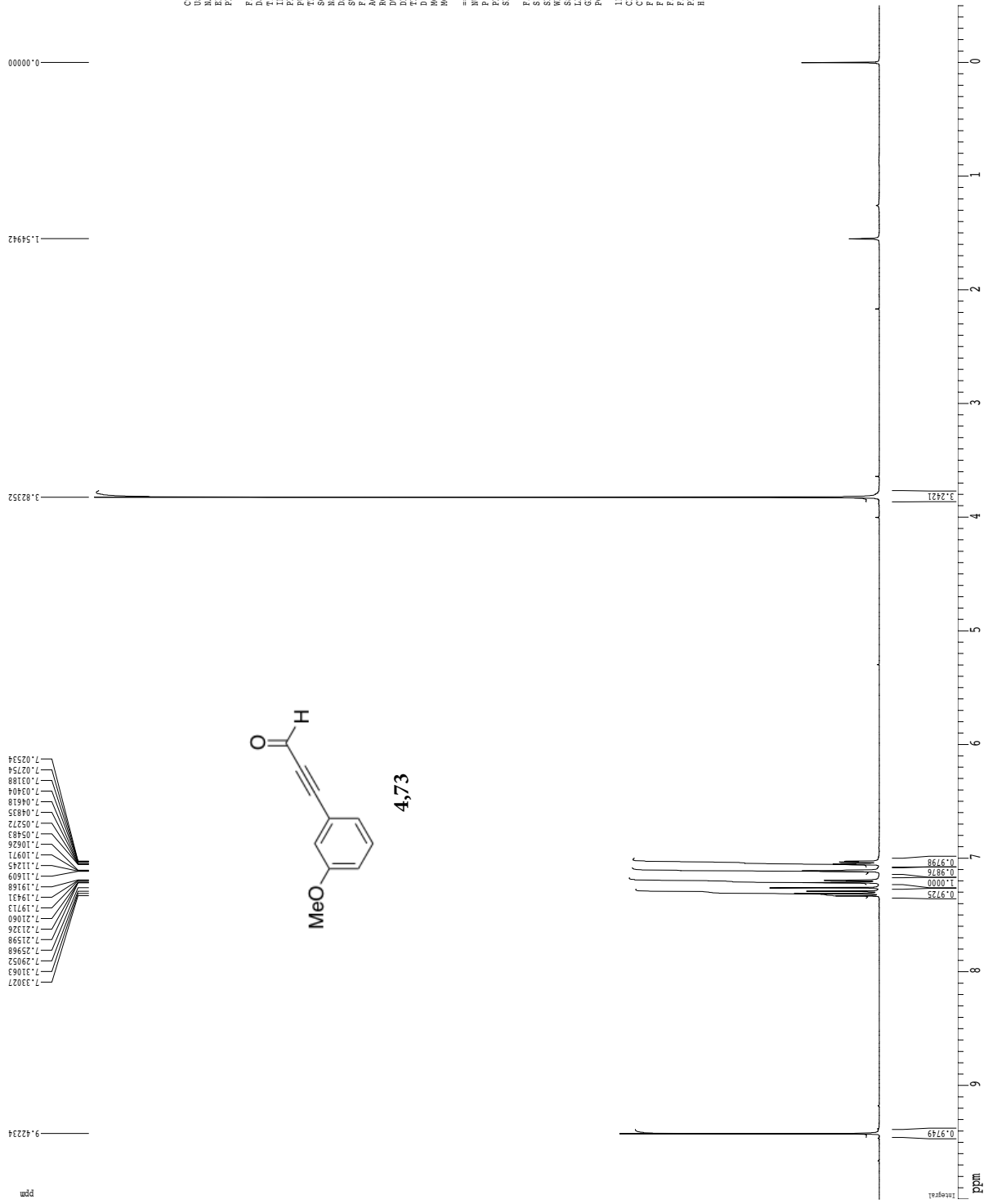
F2 - Acquisition Parameters  
 Date\_: 20191121  
 Time: 17.00  
 INSTR: spect  
 PROBNM: 5 mm CPY-1H  
 PULPROG: zg30  
 TD: 8178  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 500.136261 MHz  
 SFE: 8012.820 Hz  
 FIDRES: 0.096044 Hz  
 AQ: 5.1998774 sec  
 RG: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 0.100000 sec  
 T2: 0.000000 sec  
 T3: 0.000000 sec  
 MCHRG: 0.0150000 sec  
 MCHRG: 0.0150000 sec

===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 1.50 usec  
 PL1: 1.66 dB  
 SFO1: 500.223015 MHz

F2 - Processing Parameters  
 SI: 65536  
 SF: 500.220018 MHz  
 WDM: EM  
 LB: 0.30 Hz  
 GB: 0  
 PC: 1.00

1D NMR P1/Q2 Parameters  
 CY: 22.80 cm  
 C1: 15.00 cm  
 P1P: 8.500 ppm  
 F1: 425.18 Hz  
 F2: -250.1 Hz  
 P1Q1M: 0.39474 ppm/cm  
 HZCM: 197.45528 Hz/cm

1H spectrum



Current Data Parameters  
 USER: mlr100  
 NAME: NH-1-4t-column  
 EXPO: 1  
 PROC: 1

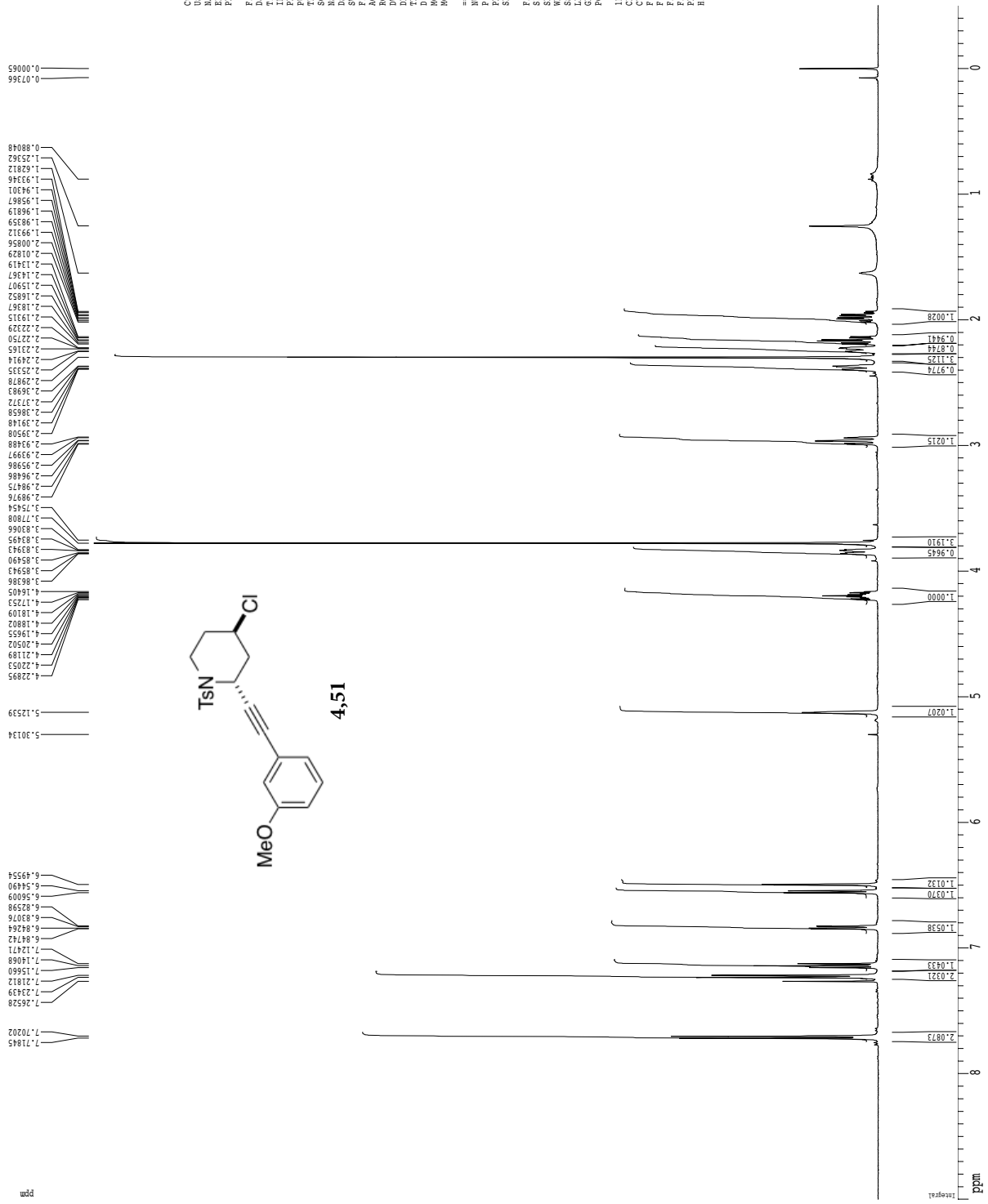
F2 - Acquisition Parameters  
 Date\_: 20200821  
 Time: 15:55  
 PROB: zgpg30  
 PULPROG: zgpg30  
 TD: 65536  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 400.1328009 MHz  
 SHF: 6410.256 Hz  
 FIDRES: 0.09781 Hz  
 AQ: 5.111857 sec  
 RG: 327.5  
 DD: 78.000 usec  
 DE: 4.50 usec  
 TE: 298.2 K  
 T1: 0.100000 sec  
 T2: 0.000000 sec  
 T3: 0.000000 sec  
 MCRST: 0.000000 sec  
 MCRBK: 0.0050000 sec

===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -1.10 dB  
 SFO1: 400.1328009 MHz

F2 - Processing Parameters  
 SI: 65536  
 SF: 400.1300216 MHz  
 WDM: EM  
 LB: 0.30 Hz  
 GB: 0  
 PC: 2.00

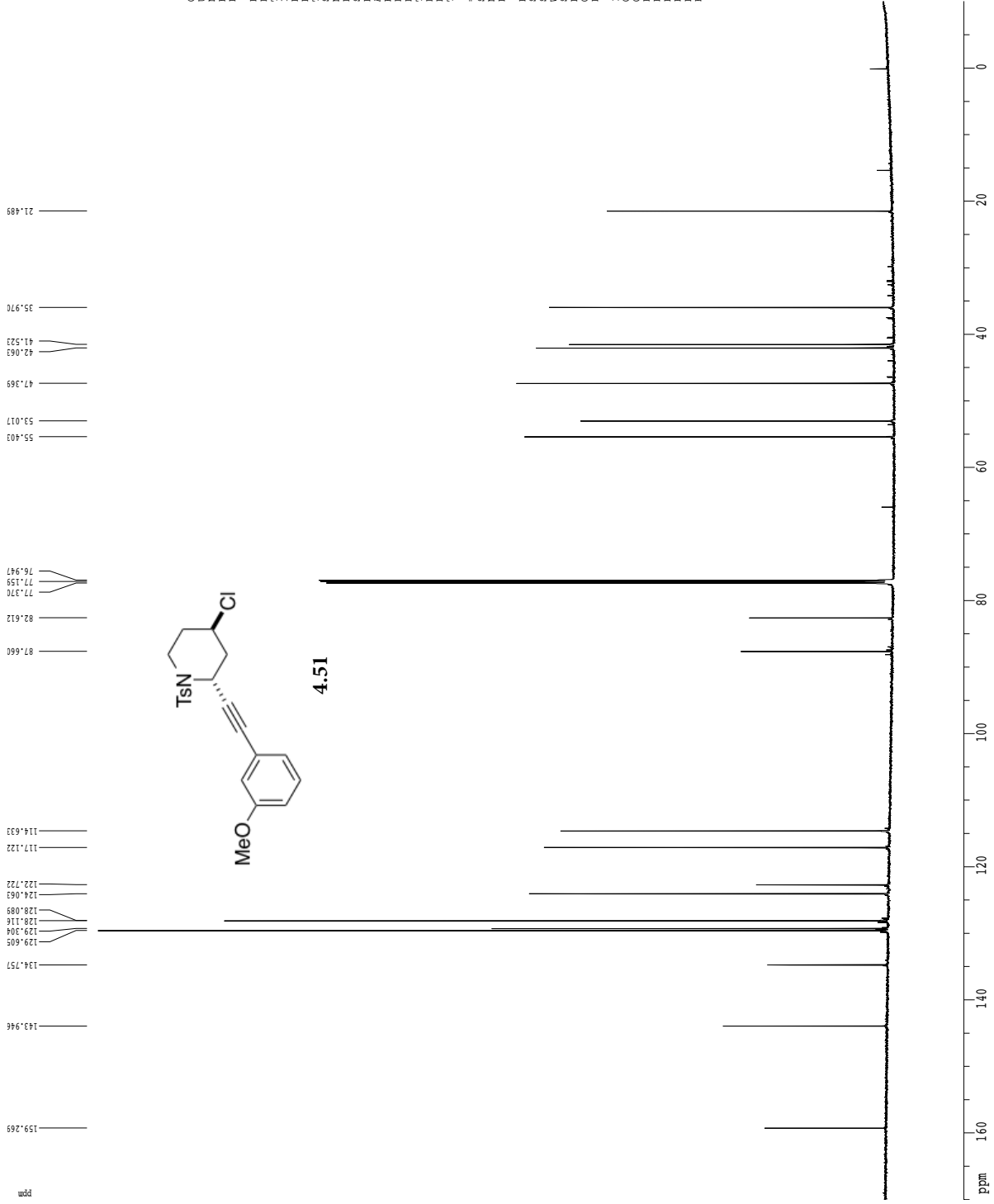
LD NMR P1.CF Parameters  
 CY: 22.80 cm  
 CV: 15.00 cm  
 FIP: 10.000 ppm  
 FL: 400.130 Hz  
 F2: -200.0 Hz  
 F3: -200.0 Hz  
 PPMCM: 0.46053 ppm/cm  
 HZCM: 184.27042 Hz/cm

1H spectrum



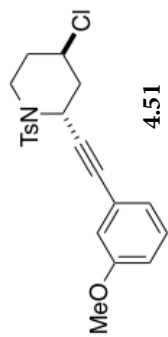
Current Data Parameters  
 USER: tsbans  
 NAME: ACM-1-114  
 EXNO: 1  
 PROCNO: 1  
 P2 - Acquisition Parameters  
 Date\_: 20200718  
 Time: 9:03  
 PROBNM: 627110  
 PROCNO: 1  
 PULPROG: zgpg30  
 TD: 81728  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 8012.820 Hz  
 FIDRES: 0.098044 Hz  
 AQ: 5.1998774 sec  
 RG: 62.400 usec  
 DE: 6.00 usec  
 TE: 560.2 K  
 D0: 0.1000000 sec  
 MCOREST: 0.0000000 sec  
 MCOREK: 0.01500000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 1.50 usec  
 PL1: 1.66 dB  
 SFO1: 500.2235015 MHz  
 P2 - Processing Parameters  
 SI: 65536  
 SF: 500.220289 MHz  
 WDW: no  
 SSB: 0.00 Hz  
 GB: 0  
 PC: 1.00  
 ID: NMR file parameters  
 CY: 22.80 cm  
 C1: 15.00 cm  
 P1P: 9.000 ppm  
 F1: 4501.500 Hz  
 F2: -250.1 Hz  
 PPRM1: 0.41667 ppm/cm  
 HZCM: 206.44502 Hz/cm

carbon



Current Data Parameters  
USER natasa  
PROCNO 1  
F2 - Acquisition Parameters  
Time 20.000000  
Date\_ 18.18  
INSTRUM av600  
PROBHD 5 mm CPBBO BB-  
PULPROG zgpg30  
SOLVENT CDCl3  
NS 504  
DS 4  
AQ 3.671000 Hz  
FIDRES 0.5524655 Hz  
AQRES 0.9944468 sec  
RG 2.050  
DM 11.800 usec  
DE 19.000 usec  
TE 298.0 K  
D1 0.4000001 sec  
D1.1 0.0300000 sec  
D10 1  
===== CHANNEL f1 =====  
SF01 150.919400 MHz  
NUC1 13C  
P1 10.10 usec  
F2 - Processing parameters  
SI 65536  
SF 150.927981 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.00  
ID NMR plot parameters  
CX 22.80 cm  
CT 15.00 cm  
FL 17.00 cm  
F1 25653.48 Hz  
F2P -10.000 ppm  
F2 -19.09.03 Hz  
FREQ 1191.33168 MHz/cm  
RECN 1191.33168 MHz/cm

COSY



Current Data Parameters  
USER matusa  
NAME ACH-I-114-Charac  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20191107  
Time 19.03  
INSTRUM av600  
PROBHD 5 mm CPBBO BB-  
PULPROG cosygprf  
TD 2048  
SOLVENT CDCl3  
NS 1  
DS 16  
SR 5411.255 Hz  
FIDRES 2.642215 Hz  
AQ 0.1892852 sec  
RG 114  
DM 92.400 usec  
DE 10.00 usec  
TE 298.0 K  
DO 0.00000300 sec  
D1 1.48689198 sec  
D13 0.00000400 sec  
D16 0.00020000 sec  
IN0 0.00018480 sec

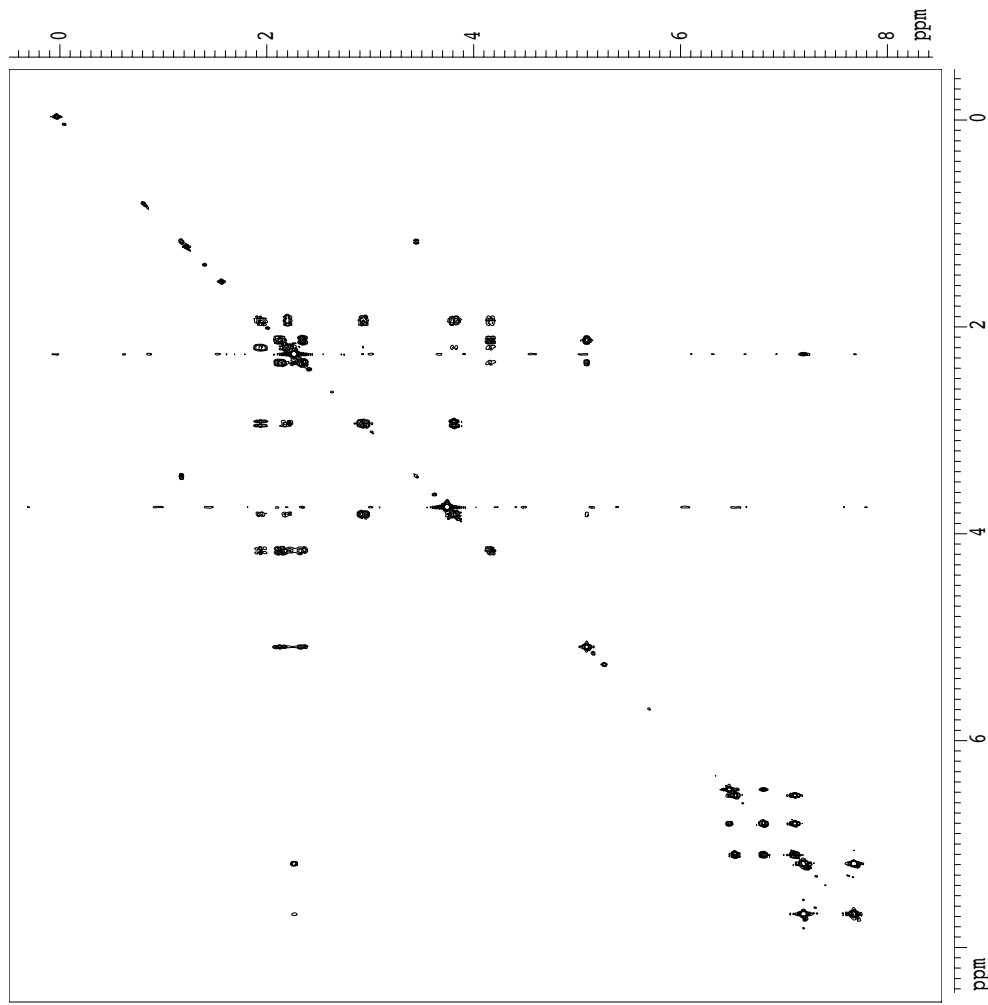
==== CHANNEL f1 =====  
SFO1 600.1324113 MHz  
NUC1 1H  
P0 12.00 usec  
P1 12.00 usec

F1 - Acquisition Parameters  
ND0 1  
TD 256  
SFO1 600.1324 MHz  
FIDRES 21.137716 Hz  
SN 9.017 ppm  
FNAME QF

F2 - Processing Parameters  
SI 1024  
SF 600.1300549 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.40

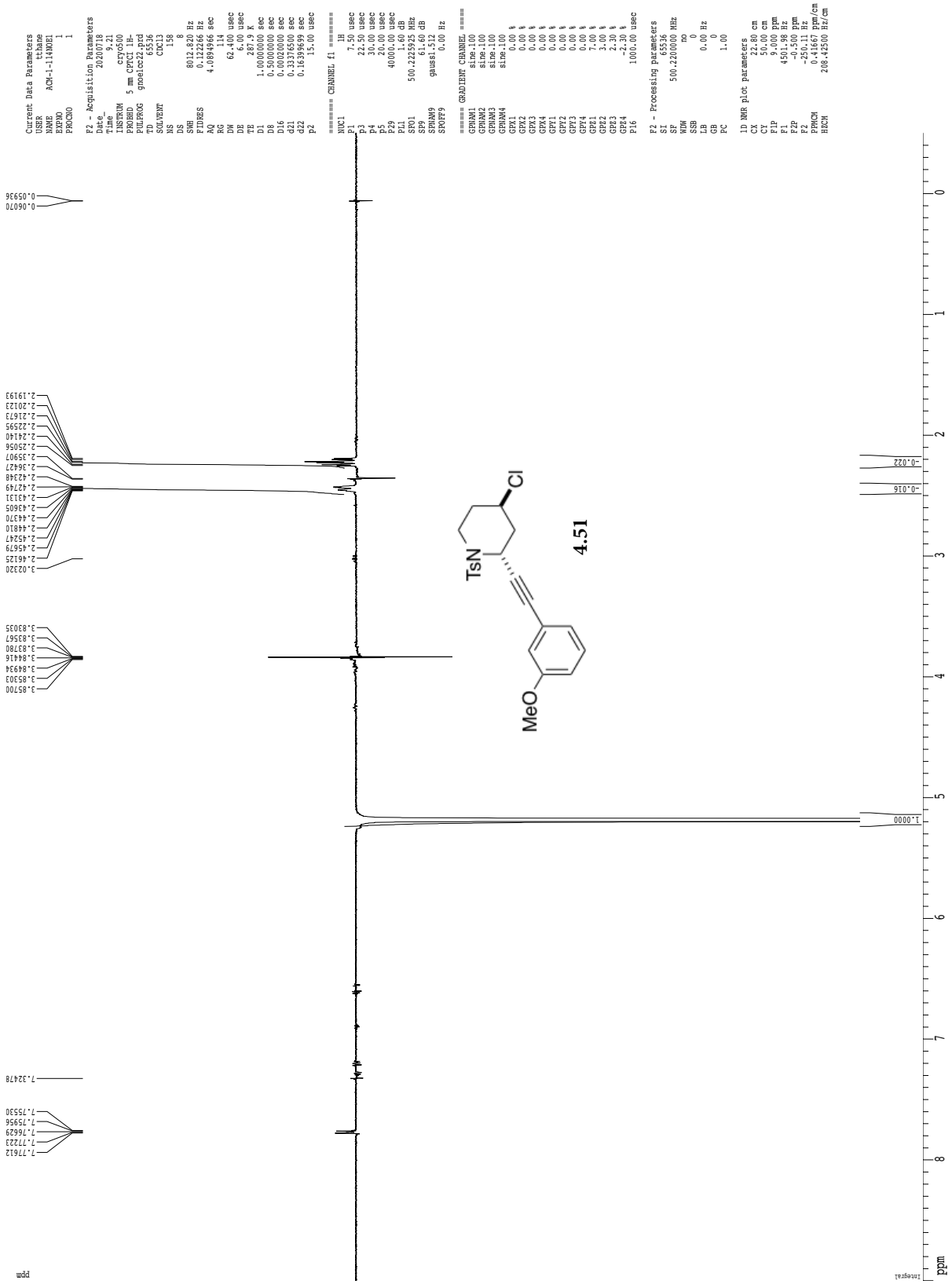
F1 - Processing Parameters  
SI 1024  
MC2 QF  
SF 600.1300549 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0

2D NMR plot parameters  
CX2 15.00 cm  
CK1 15.00 cm  
F2P/0 8.526 ppm  
F2L/0 5116.35 Hz  
F2PH1 -0.490 ppm  
F2HI -294.31 Hz  
F1P/0 8.526 ppm  
F1L/0 5116.35 Hz  
F1PH1 -0.490 ppm  
F1HI -294.31 Hz  
F2PPMCH 0.60112 ppm/cm  
F2HZCH 360.75040 Hz/cm  
F1PPMCH 0.60112 ppm/cm  
F1HZCH 360.75040 Hz/cm

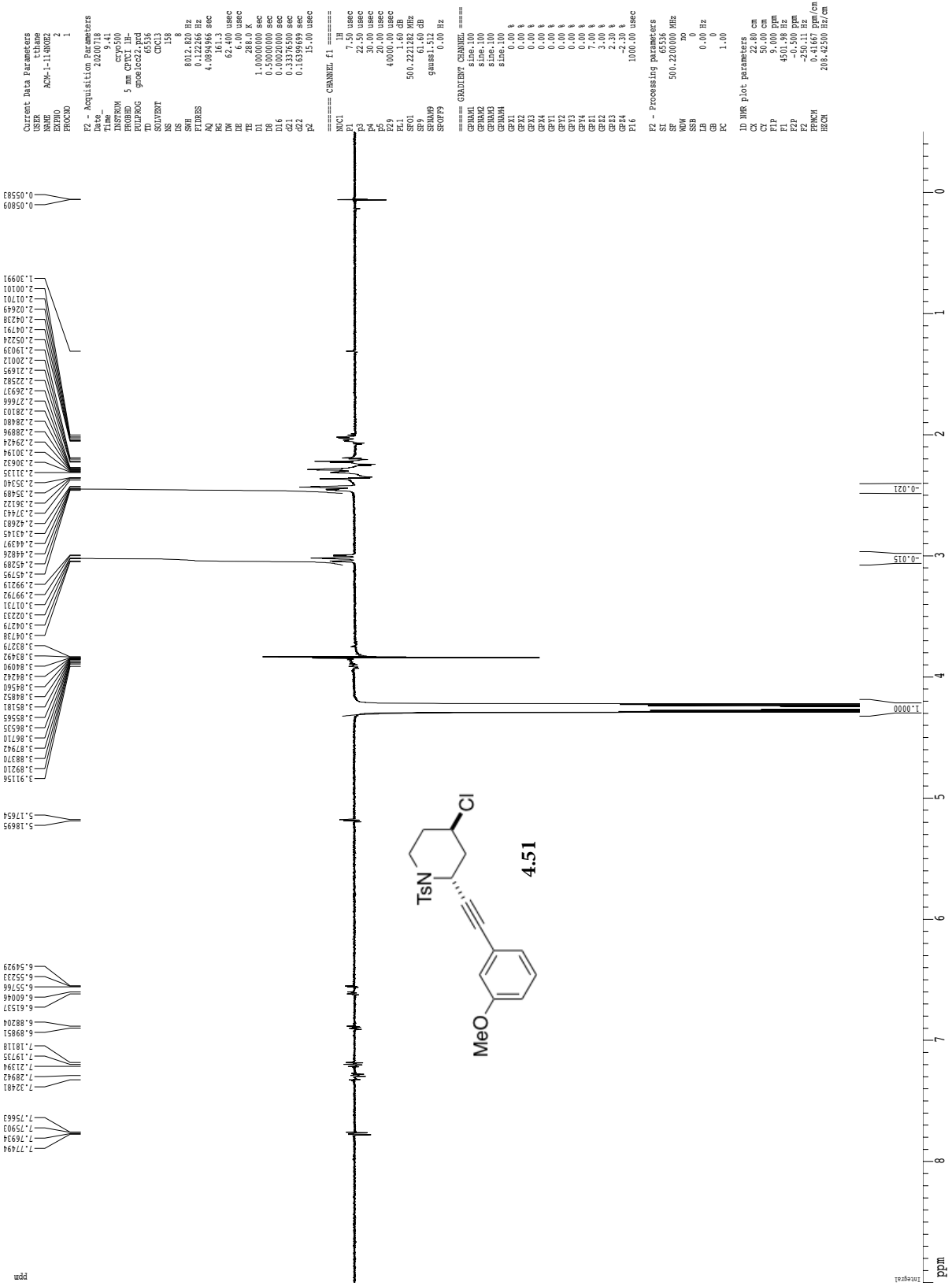




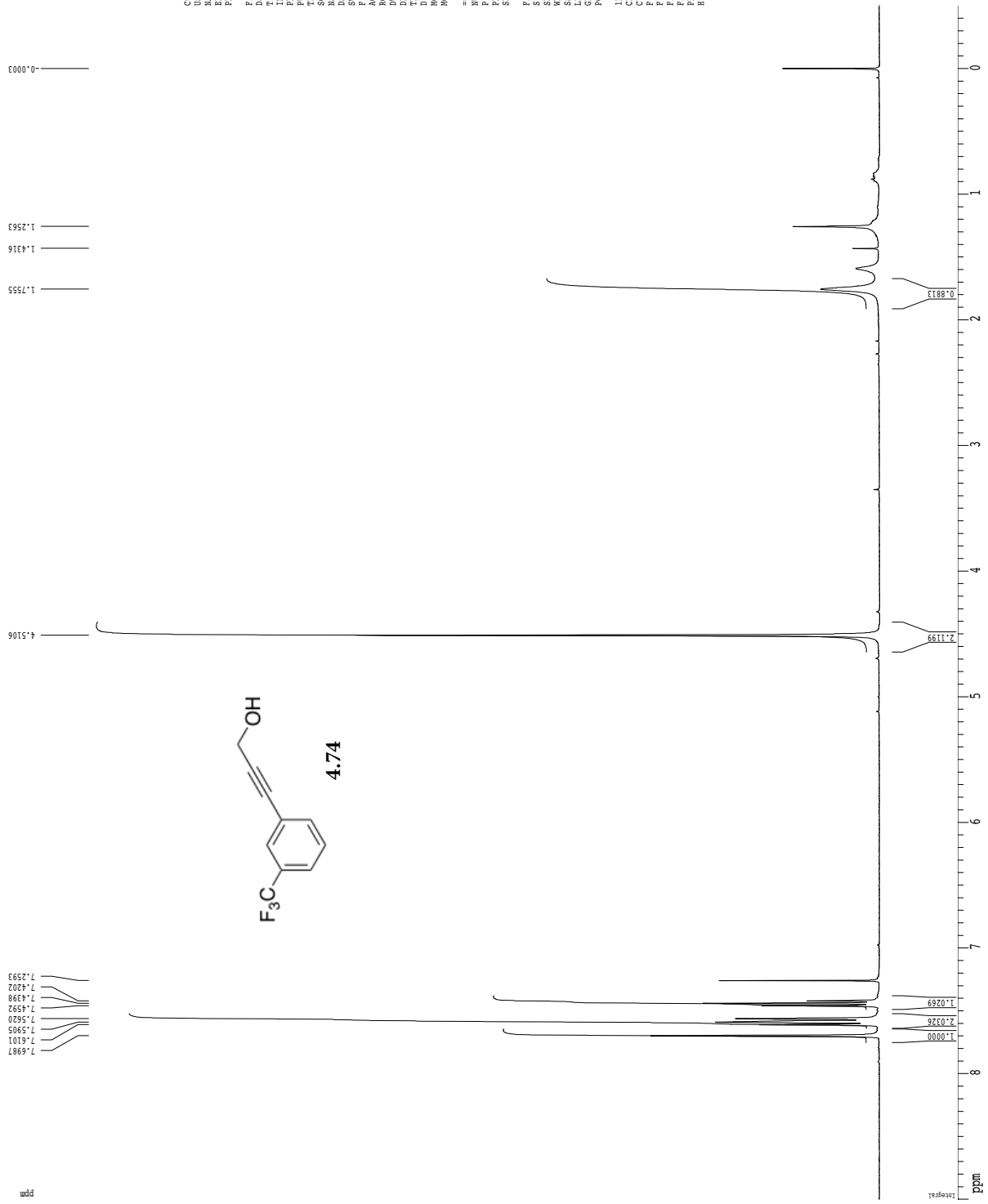
gnoe



gnoe



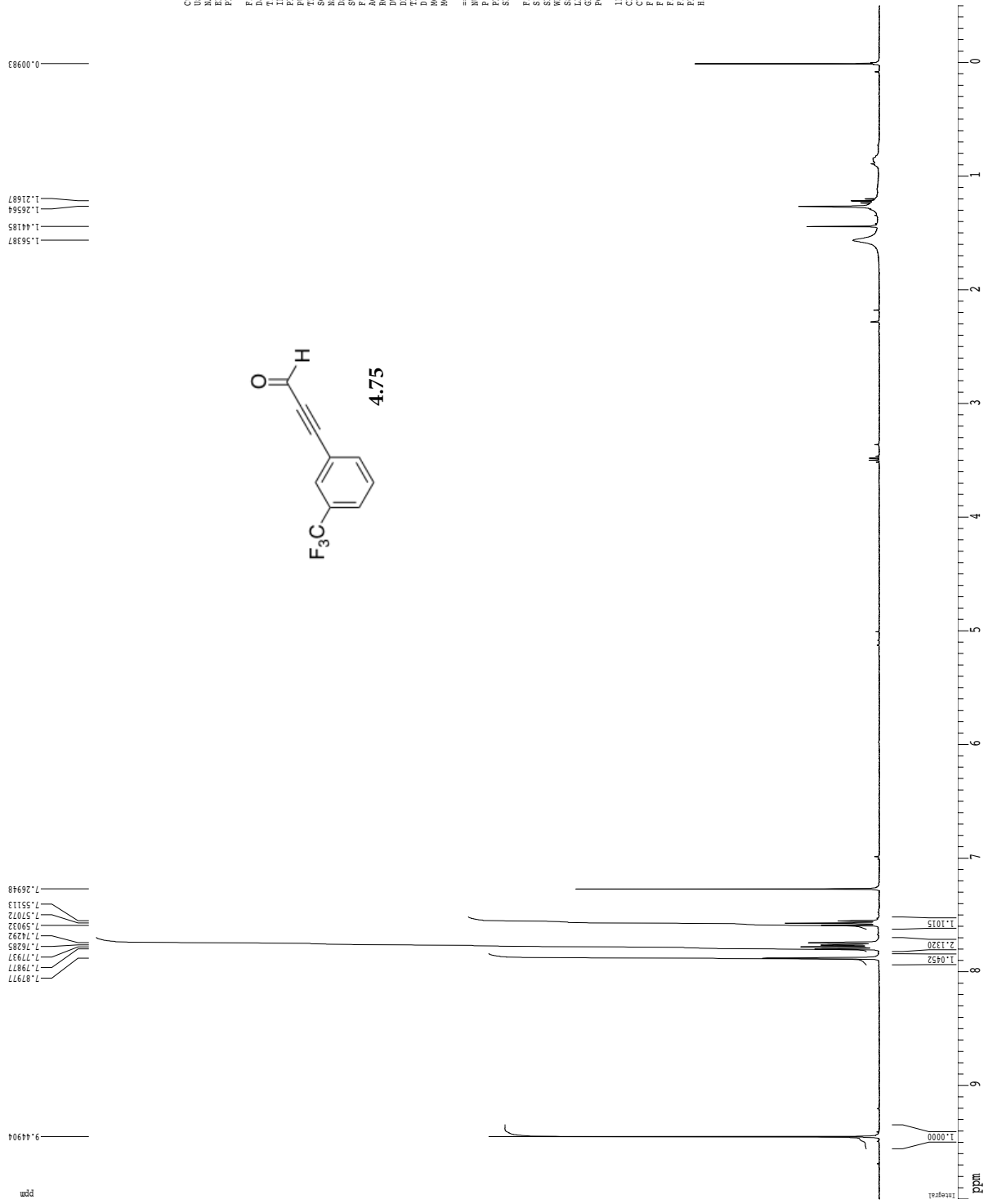
1H spectrum



Current Data Parameters  
 USER: mlrjames  
 NAME: NH-1-26-dty-11023  
 EXNO: 1  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 2020.02.3  
 Time: 11.34  
 INSTR: spect  
 PROCNO: 5 mm QNP HET  
 PULPROG: zg30  
 TD: 65536  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 500.1350900 MHz  
 SFE: 6410.256 Hz  
 FIDRES: 0.097813 Hz  
 AQ: 5.1118579 sec  
 RG: 327.5  
 DD: 78.000 usec  
 DE: 4.50 usec  
 TE: 298.2 K  
 T1: 0.10000000 sec  
 T2: 0.00000000 sec  
 T3: 0.00000000 sec  
 T4: 0.00000000 sec  
 T5: 0.00000000 sec  
 T6: 0.00000000 sec  
 T7: 0.00000000 sec  
 T8: 0.00000000 sec  
 T9: 0.00000000 sec  
 T10: 0.00000000 sec  
 T11: 0.00000000 sec  
 T12: 0.00000000 sec  
 T13: 0.00000000 sec  
 T14: 0.00000000 sec  
 T15: 0.00000000 sec  
 T16: 0.00000000 sec  
 T17: 0.00000000 sec  
 T18: 0.00000000 sec  
 T19: 0.00000000 sec  
 T20: 0.00000000 sec  
 T21: 0.00000000 sec  
 T22: 0.00000000 sec  
 T23: 0.00000000 sec  
 T24: 0.00000000 sec  
 T25: 0.00000000 sec  
 T26: 0.00000000 sec  
 T27: 0.00000000 sec  
 T28: 0.00000000 sec  
 T29: 0.00000000 sec  
 T30: 0.00000000 sec  
 T31: 0.00000000 sec  
 T32: 0.00000000 sec  
 T33: 0.00000000 sec  
 T34: 0.00000000 sec  
 T35: 0.00000000 sec  
 T36: 0.00000000 sec  
 T37: 0.00000000 sec  
 T38: 0.00000000 sec  
 T39: 0.00000000 sec  
 T40: 0.00000000 sec  
 T41: 0.00000000 sec  
 T42: 0.00000000 sec  
 T43: 0.00000000 sec  
 T44: 0.00000000 sec  
 T45: 0.00000000 sec  
 T46: 0.00000000 sec  
 T47: 0.00000000 sec  
 T48: 0.00000000 sec  
 T49: 0.00000000 sec  
 T50: 0.00000000 sec  
 T51: 0.00000000 sec  
 T52: 0.00000000 sec  
 T53: 0.00000000 sec  
 T54: 0.00000000 sec  
 T55: 0.00000000 sec  
 T56: 0.00000000 sec  
 T57: 0.00000000 sec  
 T58: 0.00000000 sec  
 T59: 0.00000000 sec  
 T60: 0.00000000 sec  
 T61: 0.00000000 sec  
 T62: 0.00000000 sec  
 T63: 0.00000000 sec  
 T64: 0.00000000 sec  
 T65: 0.00000000 sec  
 T66: 0.00000000 sec  
 T67: 0.00000000 sec  
 T68: 0.00000000 sec  
 T69: 0.00000000 sec  
 T70: 0.00000000 sec  
 T71: 0.00000000 sec  
 T72: 0.00000000 sec  
 T73: 0.00000000 sec  
 T74: 0.00000000 sec  
 T75: 0.00000000 sec  
 T76: 0.00000000 sec  
 T77: 0.00000000 sec  
 T78: 0.00000000 sec  
 T79: 0.00000000 sec  
 T80: 0.00000000 sec  
 T81: 0.00000000 sec  
 T82: 0.00000000 sec  
 T83: 0.00000000 sec  
 T84: 0.00000000 sec  
 T85: 0.00000000 sec  
 T86: 0.00000000 sec  
 T87: 0.00000000 sec  
 T88: 0.00000000 sec  
 T89: 0.00000000 sec  
 T90: 0.00000000 sec  
 T91: 0.00000000 sec  
 T92: 0.00000000 sec  
 T93: 0.00000000 sec  
 T94: 0.00000000 sec  
 T95: 0.00000000 sec  
 T96: 0.00000000 sec  
 T97: 0.00000000 sec  
 T98: 0.00000000 sec  
 T99: 0.00000000 sec  
 T100: 0.00000000 sec

1H spectrum



Current Data Parameters  
 USER: mlr1000  
 NAME: MH-1-25-dry  
 EXNO: 1  
 PROCNO: 1

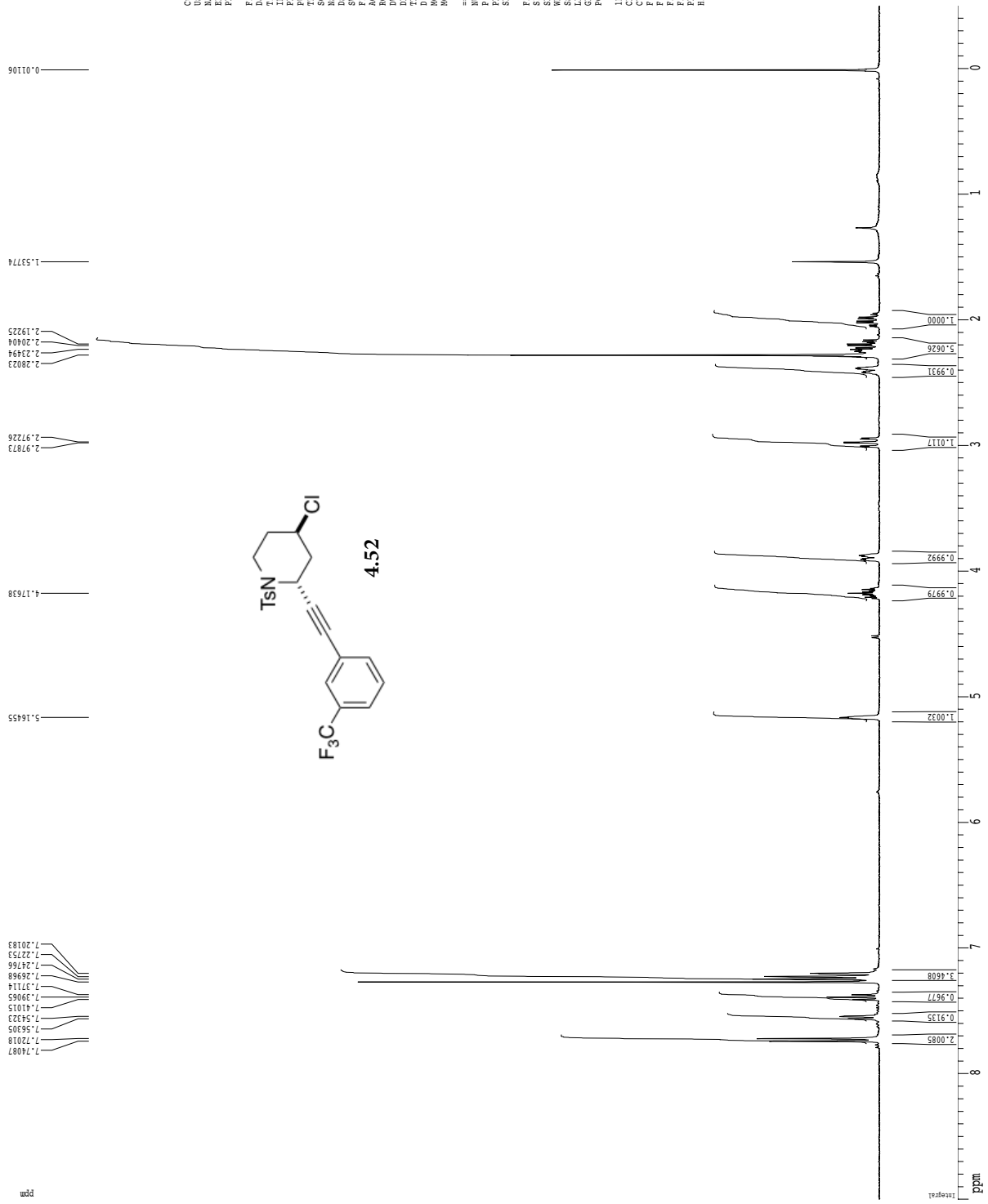
F2 - Acquisition Parameters  
 Date\_: 20201022  
 Time: 16:29  
 PROGM: zgpg30  
 PULPROG: zgpg30  
 TD: 65536  
 SOLVENT: CDCl3  
 DS: 4  
 SFO: 500.136051  
 SWH: 6410.256 Hz  
 FIDRES: 0.09781 Hz  
 AQ: 5.11857 sec  
 RG: 327.68  
 DD: 78.400 usec  
 DE: 4.50 usec  
 TE: 298.2 K  
 T1: 0.100000 sec  
 T2: 0.000000 sec  
 T3: 0.000000 sec  
 MCHRG: 0.000000 sec  
 MCORE: 0.000000 sec

===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -1.10 dB  
 SFO1: 400.132809 MHz

F2 - Processing Parameters  
 SI: 65536  
 SF: 400.1300175 MHz  
 WDM: EN  
 LB: 0.30 Hz  
 GB: 0  
 PC: 2.00

LD NMR P1: Parameters  
 CY: 22.80 cm  
 C1: 7.50 cm  
 P1P: 10.000 ppm  
 F1: 400.130 Hz  
 F2: -200.0 Hz  
 P1PC1: 0.46053 ppm/cm  
 HZCN: 184.27040 Hz/cm

1H spectrum



Current Data Parameters  
 USER: mlrjpbw  
 NAME: NH-1-3-2011-19  
 EXPO: 1  
 PROC: 1

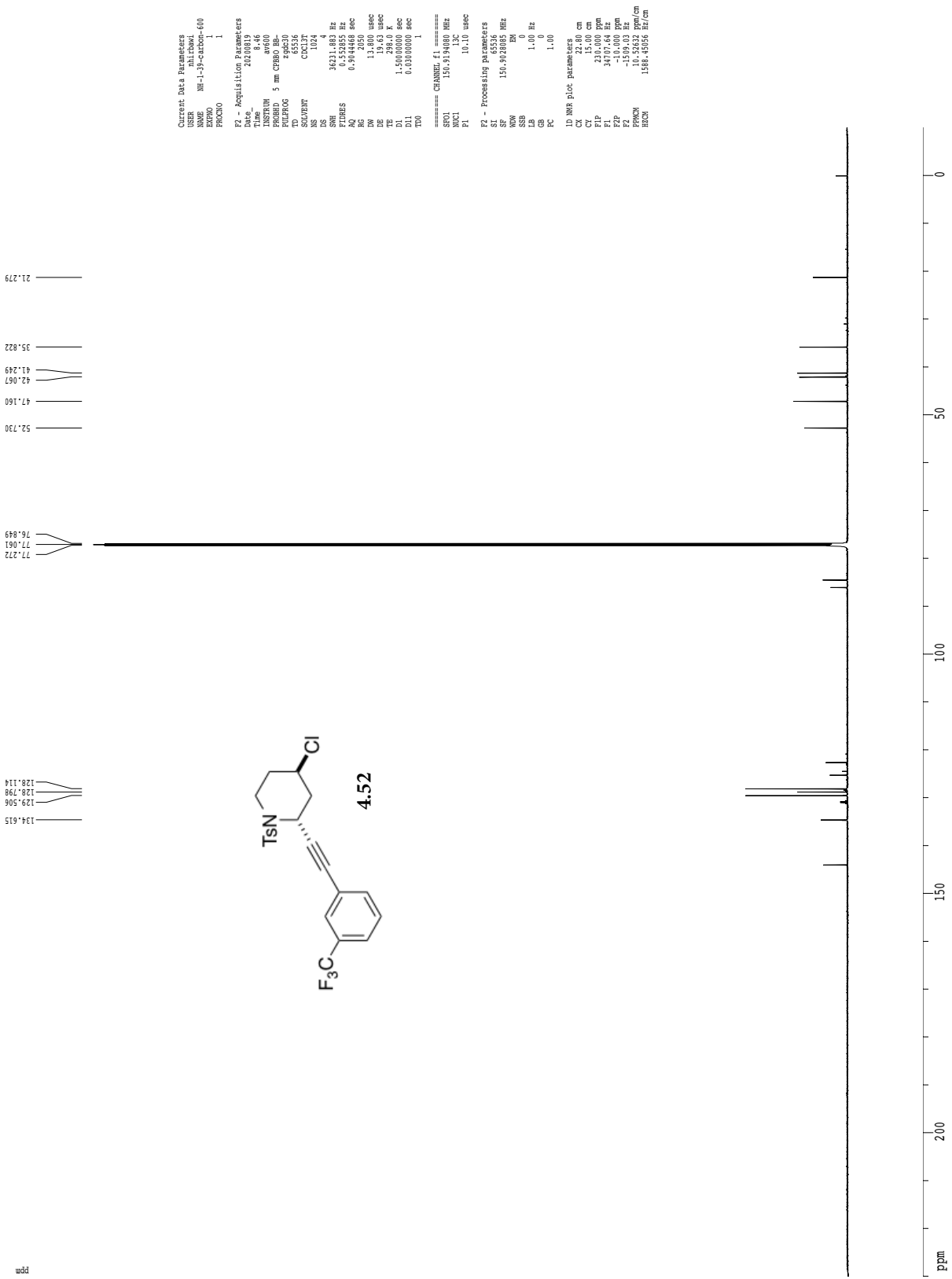
F2 - Acquisition Parameters  
 Date\_: 20201119  
 Time: 16.33  
 PROB: zgpg30  
 PULPROG: zgpg30  
 TD: 65536  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 6410.256 Hz  
 FIDRES: 0.097813 Hz  
 AQ: 5.1118579 sec  
 RG: 384  
 DD: 78.000 usec  
 DE: 4.50 usec  
 TE: 298.2 K  
 T1: 0.10000000 sec  
 T2: 0.00000000 sec  
 T3: 0.00000000 sec  
 MCHRG: 0.00000000 sec  
 MCORE: 0.00000000 sec

===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -1.68 dB  
 SFO1: 400.1328009 MHz

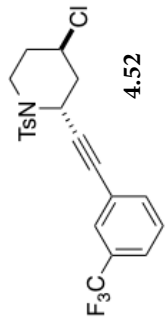
F2 - Processing Parameters  
 SI: 65536  
 SF: 400.1300175 MHz  
 EQ: EM  
 LB: 0.30 Hz  
 GB: 0  
 PC: 2.00

DJ VWR PLOT Parameters  
 CY: 22.80 cm  
 CX: 10.00 cm  
 FIP: 9.000 ppm  
 FL: 3600.17 Hz  
 F2: 400.1300175 MHz  
 F3: -200.00 Hz  
 PPMCM: 0.41667 ppm/cm  
 HZCM: 166.72084 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters  
 USER: mh1804  
 NAME: NH-1,3-c-acbor-640  
 EXPNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20200319  
 Time: 14.06  
 INSTRUM: av600  
 PROBHD: 5 mm CPBBO BB-  
 PULPROG: zgpg30  
 SFO: 125.760  
 SOLVENT: CDCl3  
 NS: 1024  
 DS: 4  
 SWH: 36231.86 Hz  
 FIDRES: 0.552855 Hz  
 AQ: 0.3044468 sec  
 RG: 2050  
 W: 13.200 usec  
 DE: 18.43 usec  
 TE: 298.4 K  
 D1: 1.5000000 sec  
 D11: 0.0300000 sec  
 D12: 0.0300000 sec  
 D13: 0.0300000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 13C  
 NU1: 13C  
 P1: 10.10 usec  
 F2 - Processing parameters  
 SF: 125.760000 MHz  
 CF: 62.880000 MHz  
 WDW: EM  
 SSB: 0  
 LB: 1.00 Hz  
 GB: 0  
 CB: 0  
 FC: 1.00  
 ID: WALTZ16 parameters  
 CX: 22.80 cm  
 CY: 15.00 cm  
 CZ: 15.00 cm  
 F1: 3.000000 MHz  
 F2: 3.000000 MHz  
 F3: 3.000000 MHz  
 F4: 3.000000 MHz  
 F5: 3.000000 MHz  
 F6: 3.000000 MHz  
 F7: 3.000000 MHz  
 F8: 3.000000 MHz  
 F9: 3.000000 MHz  
 F10: 3.000000 MHz  
 F11: 3.000000 MHz  
 F12: 3.000000 MHz  
 F13: 3.000000 MHz  
 F14: 3.000000 MHz  
 F15: 3.000000 MHz  
 F16: 3.000000 MHz  
 F17: 3.000000 MHz  
 F18: 3.000000 MHz  
 F19: 3.000000 MHz  
 F20: 3.000000 MHz  
 F21: 3.000000 MHz  
 F22: 3.000000 MHz  
 F23: 3.000000 MHz  
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 F31: 3.000000 MHz  
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 F34: 3.000000 MHz  
 F35: 3.000000 MHz  
 F36: 3.000000 MHz  
 F37: 3.000000 MHz  
 F38: 3.000000 MHz  
 F39: 3.000000 MHz  
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 F45: 3.000000 MHz  
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 F80: 3.000000 MHz  
 F81: 3.000000 MHz  
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 F83: 3.000000 MHz  
 F84: 3.000000 MHz  
 F85: 3.000000 MHz  
 F86: 3.000000 MHz  
 F87: 3.000000 MHz  
 F88: 3.000000 MHz  
 F89: 3.000000 MHz  
 F90: 3.000000 MHz  
 F91: 3.000000 MHz  
 F92: 3.000000 MHz  
 F93: 3.000000 MHz  
 F94: 3.000000 MHz  
 F95: 3.000000 MHz  
 F96: 3.000000 MHz  
 F97: 3.000000 MHz  
 F98: 3.000000 MHz  
 F99: 3.000000 MHz  
 F100: 3.000000 MHz



Current Data Parameters  
 USER nhitbavi  
 NAME NH-1-19-on11-19-COSY  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20201119  
 Time 17:09  
 INSTRUM crys500  
 PROBHD 5 mm CPTCI LH-  
 PULPROG cosy90.prd  
 TD 2048  
 CONVNT CCL12  
 NS 2  
 DS 16  
 SFO 5000.000 Hz  
 FIDRES 2.441406 Hz  
 AQ 0.2048500 sec  
 RG 1448.2  
 DW 100.000 usec  
 DE 6.000 usec  
 TE 298.0 K  
 d0 0.00000300 sec  
 d1 1.00000000 sec  
 d13 0.00000300 sec  
 d16 0.00020000 sec  
 INO 0.00020000 sec

==== CHANNEL f1 =====

NUC1 1H  
 P1 9.75 usec  
 PL1 1.60 dB  
 SFO1 500.222827 MHz

==== CHANNEL f2 =====

GENM1 SWSQ10.100  
 GENM2 SWSQ10.100  
 GE1 0.00 %  
 GE2 0.00 %  
 GE3 0.00 %  
 GE4 0.00 %  
 GE5 17.00 %  
 GE6 17.00 %  
 P16 1000.00 usec

F1 - Acquisition Parameters

NUO 1  
 TD 256  
 SFO1 500.2223 MHz  
 FIDRES 19.331800 Hz  
 SFO2 9.499 Ppm  
 FWHM0 QP

F2 - Processing parameters

SI 1024  
 SF 500.2200318 MHz  
 WM SINE  
 SFO 0.00 Hz  
 LB 0  
 GB 0  
 PC 1.00

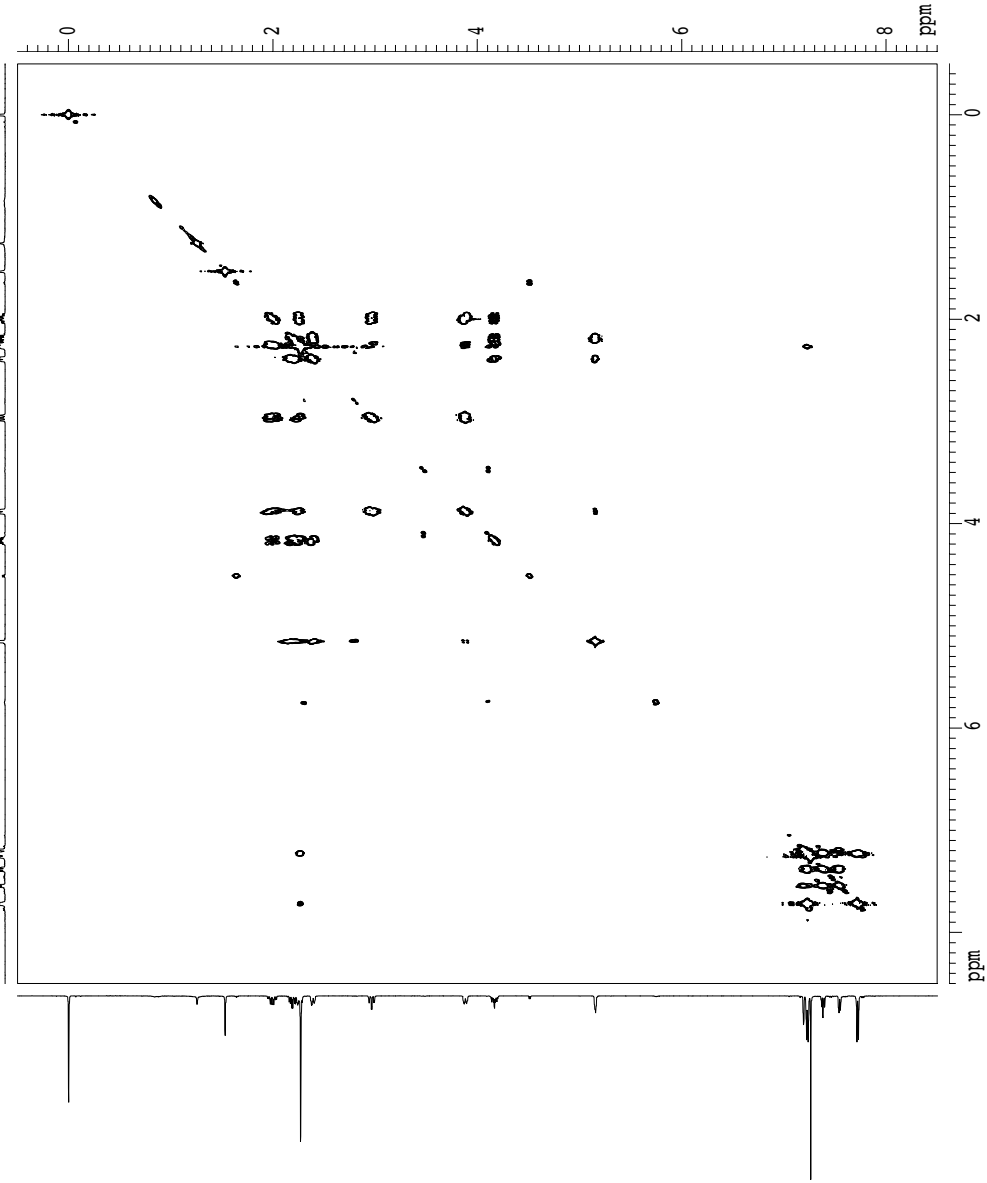
F1 - Processing parameters

SI 1024  
 SF 500.2200318 MHz  
 WM SINE  
 SFO 0  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters

SI 512  
 CX1 15.00 cm  
 F2PLO 8.500 ppm  
 FZLO 4251.87 Hz  
 FZPHI -0.498 ppm  
 FZHI -249.01 Hz  
 F1PLO 8.500 ppm  
 F1PHI 4251.87 Hz  
 F1HI -249.01 Hz  
 F2PFCM 0.55985 ppm/cm  
 FZFCM 300.05869 Hz/cm  
 F1PFCM 0.55985 ppm/cm  
 FZFCM 300.05869 Hz/cm

gc05y60



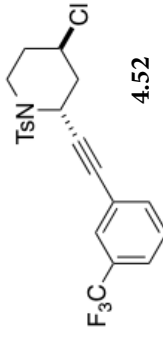
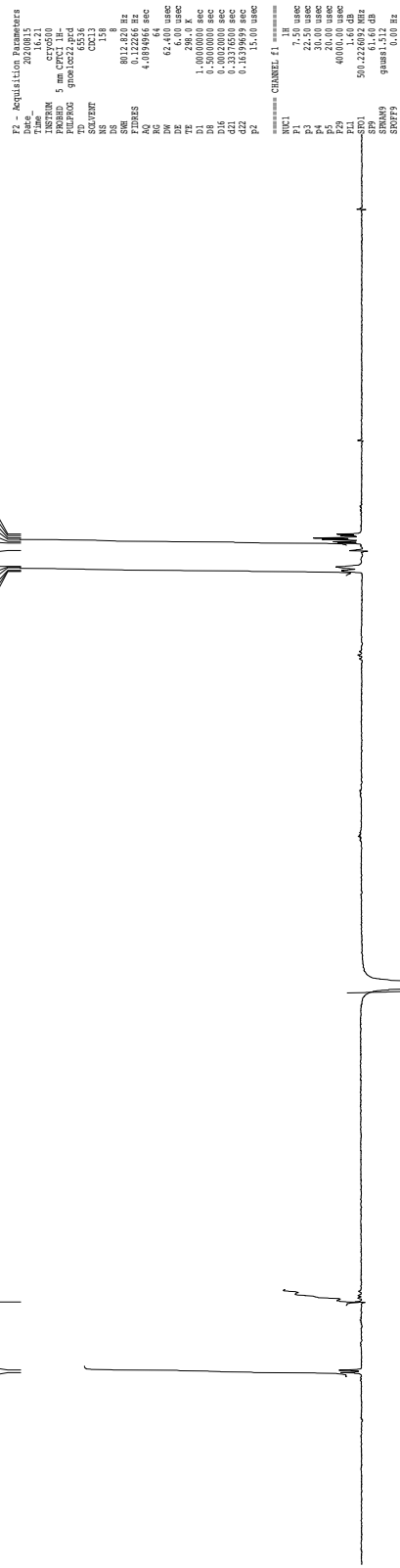
gnoe

ppm

2.40433  
2.39649  
2.37893  
2.37489  
2.37128  
2.29512  
2.20282  
2.19015  
2.18061  
2.16330  
2.15576

7.25649  
7.70886  
7.72330

Current Data Parameters  
USER mh1bwa  
NAME NH-133-mercapain  
PROCNO 2  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20200815  
Time 15:42:11  
PROBHD 5 mm CPTCI H-  
PULPROG zgpg30  
SOLVENT CDCl3  
NS 158  
DS 8012.800 Hz  
FIDRES 0.122266 Hz  
AQ 4.10595000 sec  
RG 64  
ZM 62.400 uSAC  
SFO 500.136260 MHz  
TE 296.2 K  
D1 1.00000000 sec  
D2 0.50000000 sec  
d21 0.33316500 sec  
d22 0.16339899 sec  
F2 - Processing Parameters  
SI 500.272632 MHz  
SF 500.272632 MHz  
WDW EM  
SS 0  
LB 1.00 Hz  
GB 0  
PC 1.00  
ID MR Parameters  
CI 22.80 cm  
PI 9.400 ppm  
F1 450.136 Hz  
F2 -255.11 Hz  
PRCN 0.41667 ppm/cm  
RICK 208.42512 Hz/cm

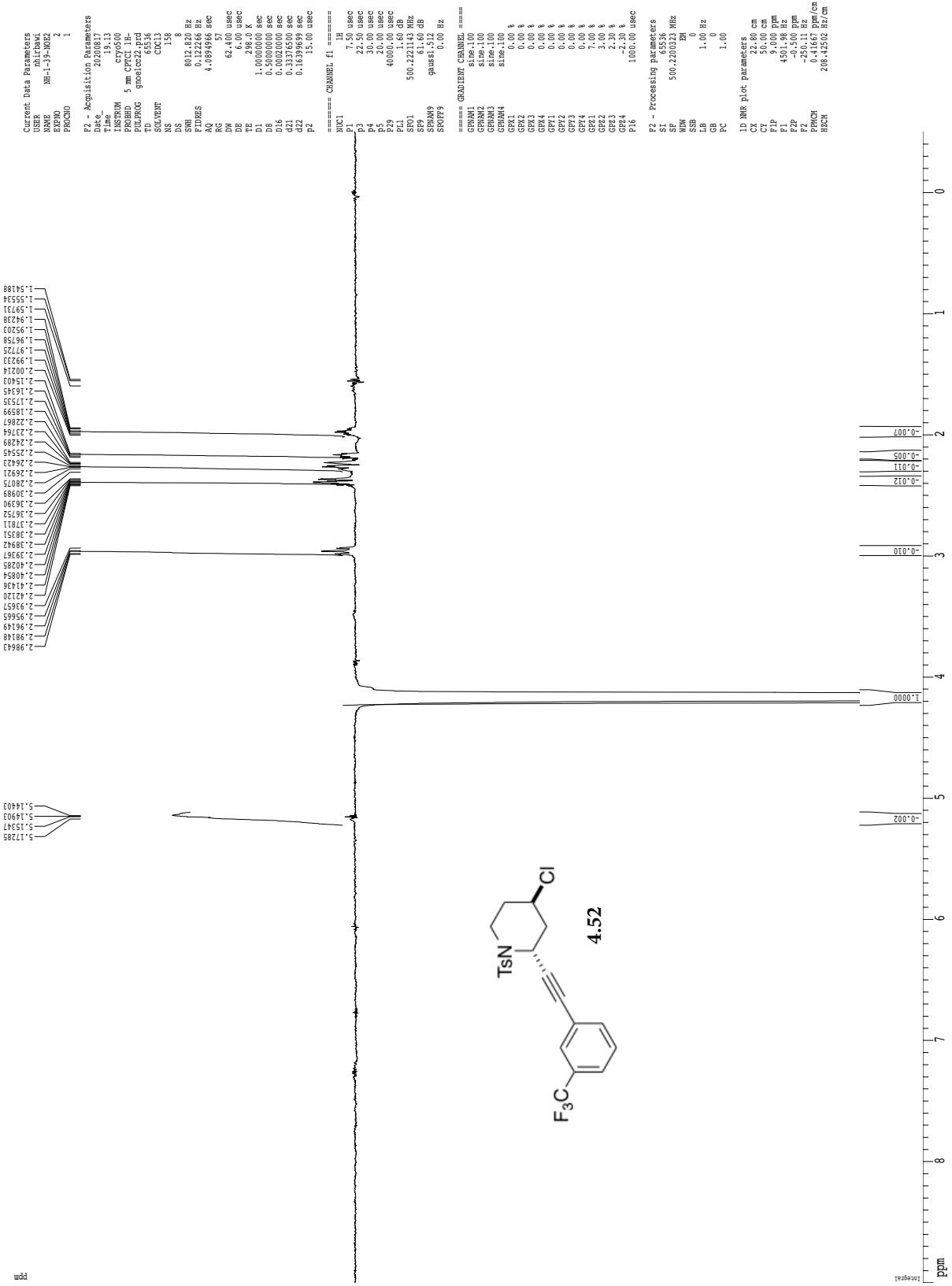


Integral



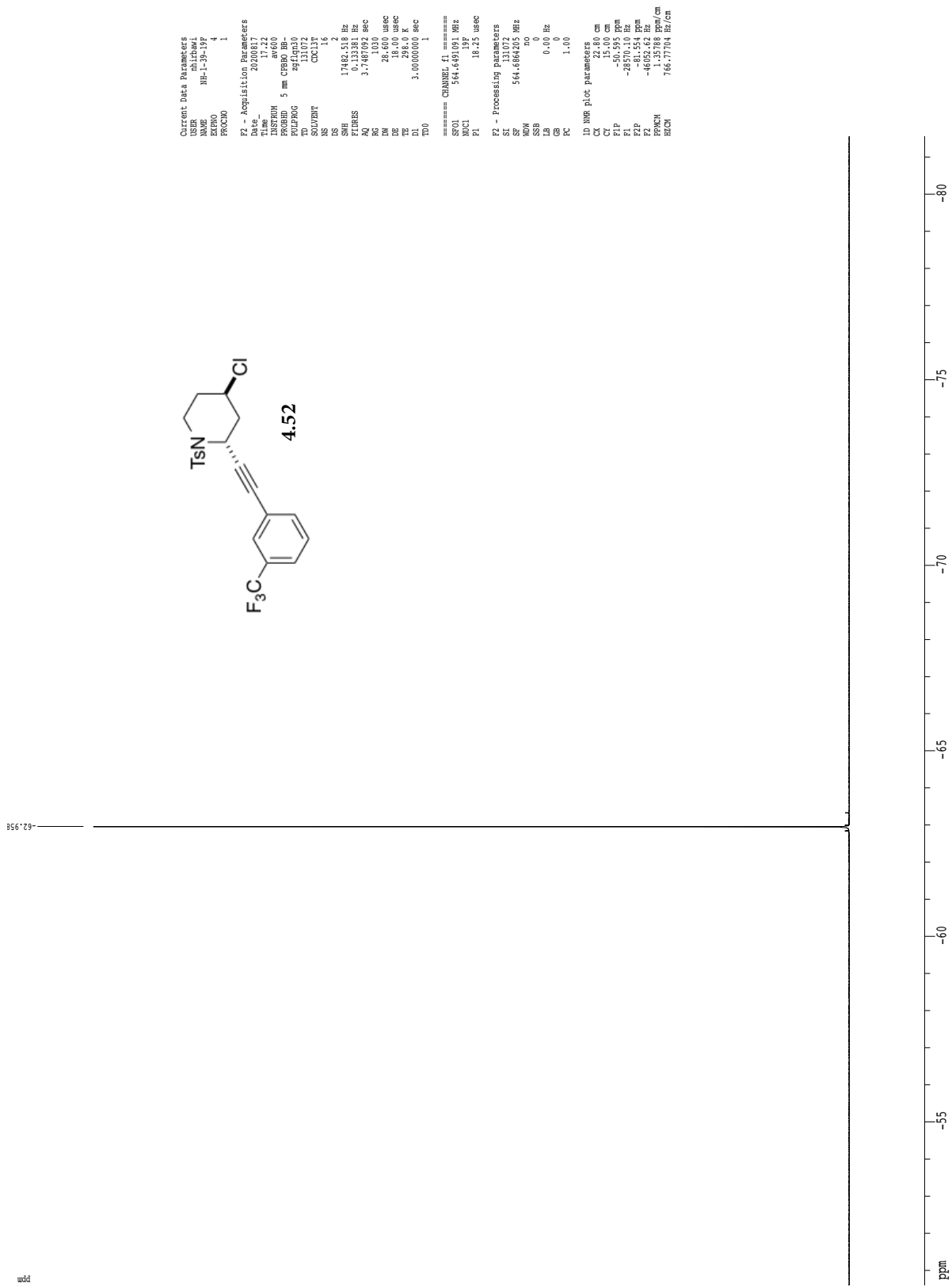


gnoe



19F spectrum

8



Current Data Parameters  
USER nhilchwa  
NAME JHE-1-35-19F  
PROCNO 4  
PROCNO 1

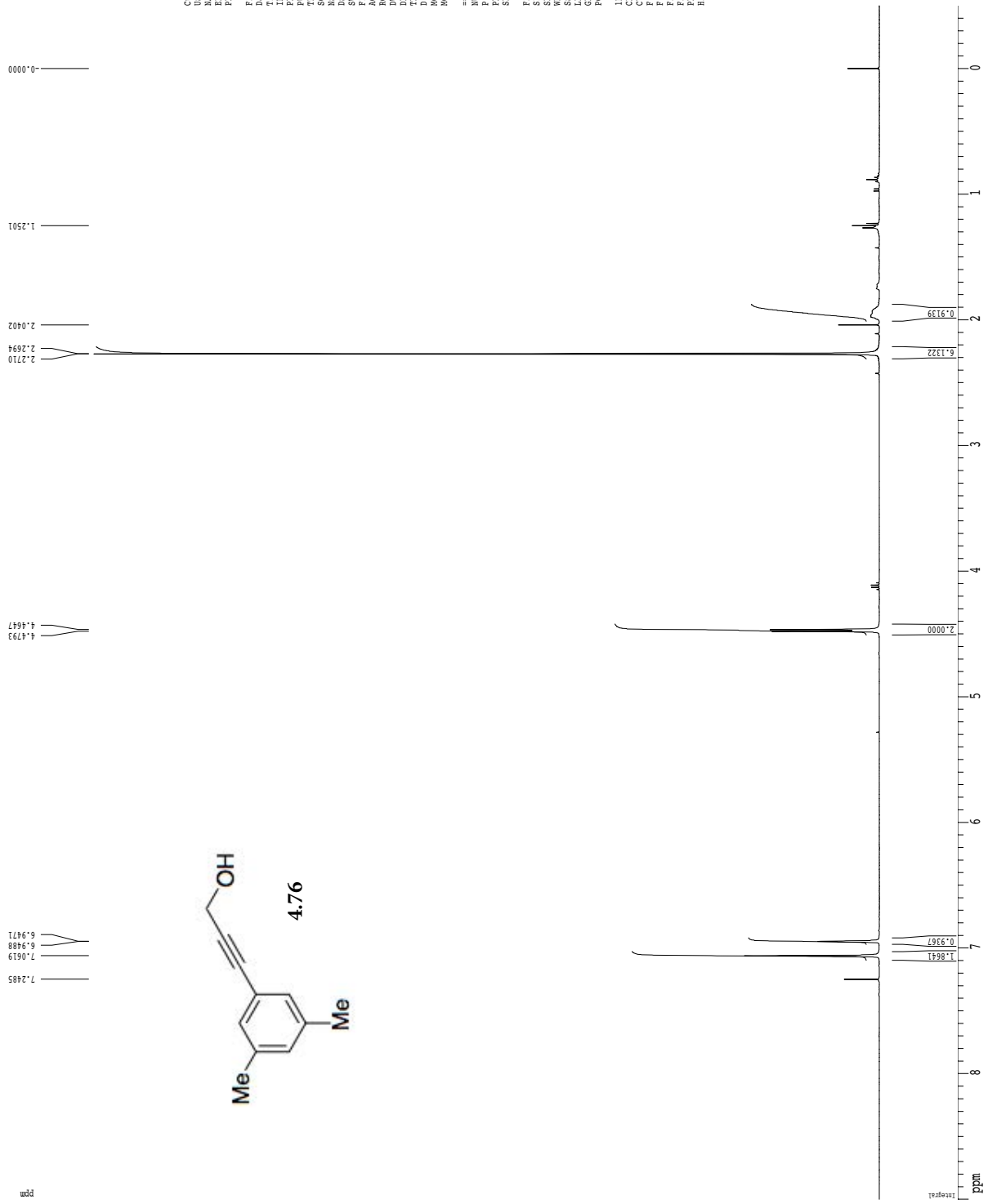
F2 - Acquisition Parameters  
Date\_ 20190717  
Time\_ 11:27  
INSTRUM av600  
PROBHD 5 mm CPBBO BB-  
PULPROG zgpg30  
AQ 1.12  
SOLVENT CDCl3  
NS 16  
DS 2  
SHH 17482.318 Hz  
NUC1 19F  
NUC2 13C  
MA 3.7487892 sec  
RG 1.030  
DM 28.600 usec  
DE 18.000 usec  
TE 300.2 K  
D1 3.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO 500.136193 MHz  
NUC1 19F  
PL 18.25 usec

F2 - Processing parameters  
SI 564.686405 MHz  
WDW no  
SSB 0  
GB 0  
PC 1.00

IDMR plot parameters  
CX 0.00 cm  
CY 15.00 cm  
FLP -50.595 ppm  
F1 -28370.10 Hz  
F2P -81.554 ppm  
F3P -105.222 ppm  
FRACM 1.35182  
HZCM 766.77048 Hz/cm

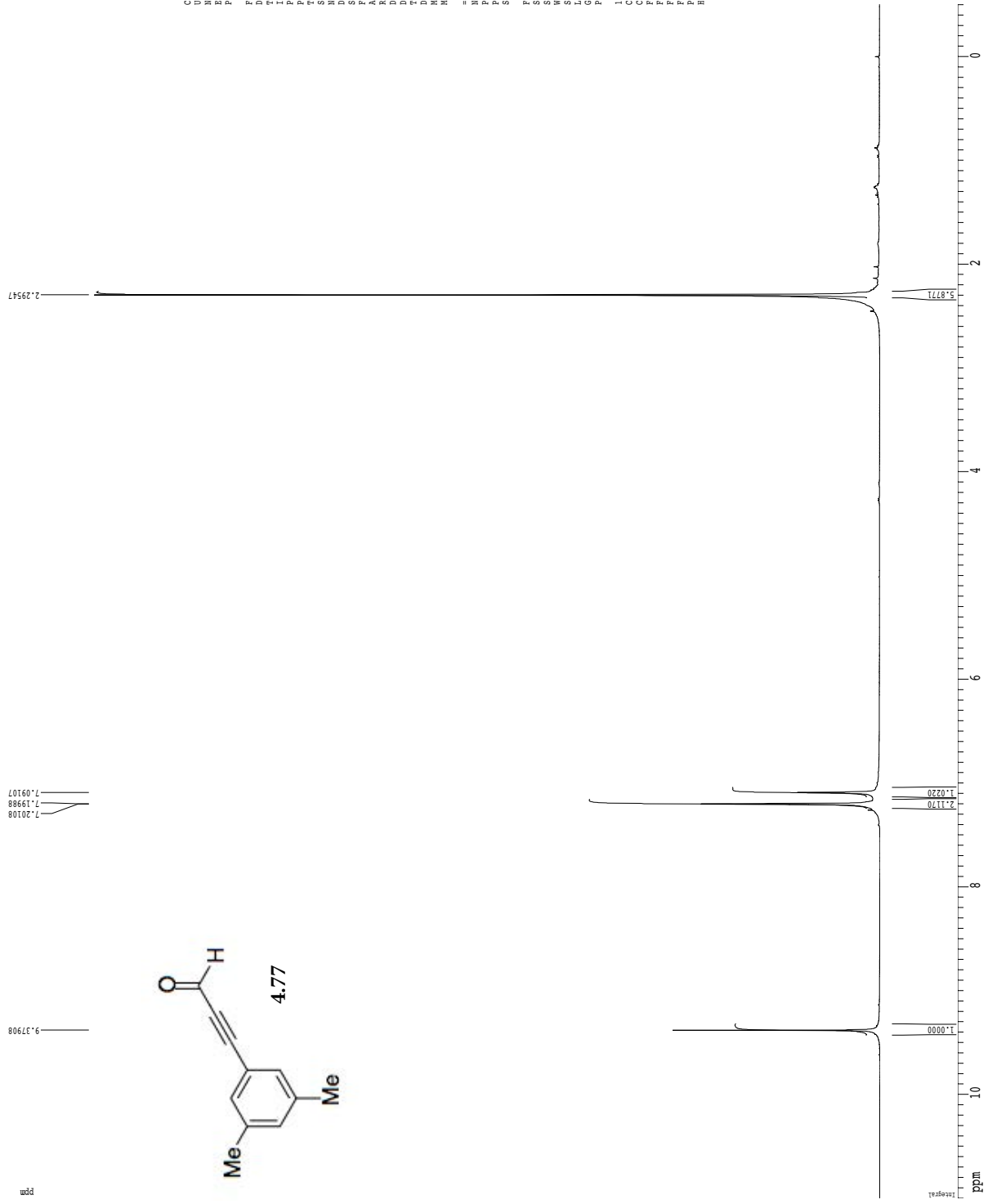
1H spectrum



Current Data Parameters  
 USER Kmett  
 NAME R04-III-254-1  
 EXNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20200829  
 Time 17:40  
 INSTRUM spect  
 PROBNM 5 mm QNP HET  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 DS 9  
 SFO 6410.258 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.399929 sec  
 RG 327.5  
 DE 78.000 usec  
 TE 298.2 K  
 T1 0.100000 sec  
 T2 0.000000 sec  
 T3 0.000000 sec  
 T4 0.000000 sec  
 T5 0.000000 sec  
 T6 0.000000 sec  
 T7 0.000000 sec  
 T8 0.000000 sec  
 T9 0.000000 sec  
 T10 0.000000 sec  
 T11 0.000000 sec  
 T12 0.000000 sec  
 T13 0.000000 sec  
 T14 0.000000 sec  
 T15 0.000000 sec  
 T16 0.000000 sec  
 T17 0.000000 sec  
 T18 0.000000 sec  
 T19 0.000000 sec  
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 T91 0.000000 sec  
 T92 0.000000 sec  
 T93 0.000000 sec  
 T94 0.000000 sec  
 T95 0.000000 sec  
 T96 0.000000 sec  
 T97 0.000000 sec  
 T98 0.000000 sec  
 T99 0.000000 sec  
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1H spectrum



Current Data Parameters  
 USER KIMSEIHT  
 NAME KMF-III-259-1  
 EXNO 1  
 PROCNO 1

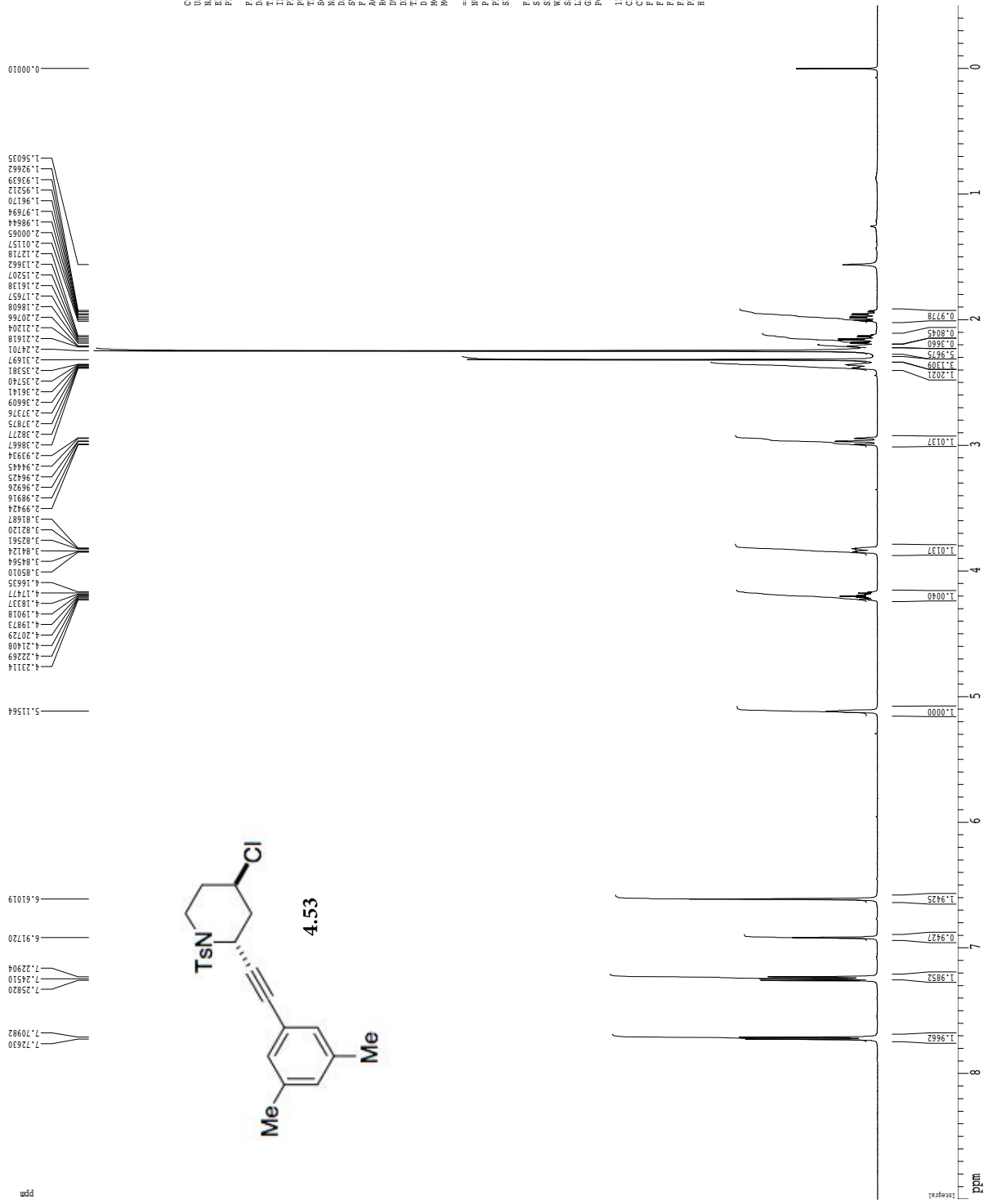
F2 - Acquisition Parameters  
 Date\_ 20200902  
 Time 16:53  
 INSTRUM spect  
 PROBRD 5 mm QNP 7F1  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 DS 2  
 SWH 6410.256 Hz  
 FIDRES 0.166673 Hz  
 AQ 2.999929 sec  
 RG 327.5  
 DW 78.000 usec  
 DE 4.50 usec  
 TE 298.0 K  
 PRGNAME 0100000 sec  
 MCBST 0100000 sec  
 MCBREK 0.0150000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.1328009 MHz

F2 - Processing Parameters  
 SI 65536  
 SF 400.1300204 MHz  
 WDW no  
 SS 0  
 GB 0  
 PC 2.00

F3 NMR P1 of Parameters  
 CY 72.80 cm  
 CV 15.00 cm  
 FIP 11.000 ppm  
 FL 440.143 Hz  
 F2 -200.07 Hz  
 PPMCM 0.50439 ppm/cm  
 HZCM 201.81998 Hz/cm

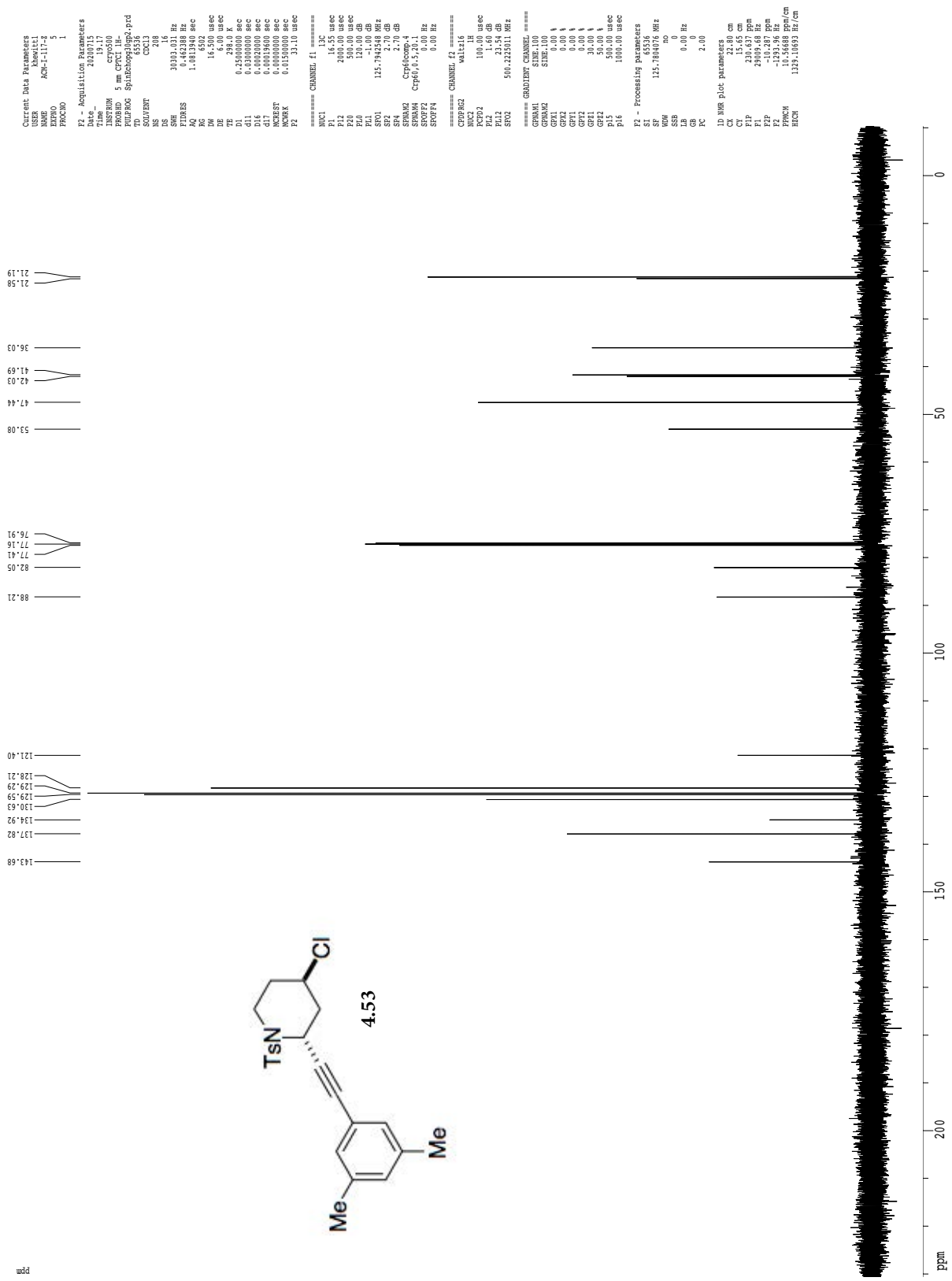
1H spectrum

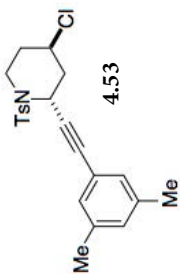


Current Data Parameters  
 USER: khawatt  
 NAME: ACM-1-17-2  
 EXNO: 1  
 PROCU: 1

F2 - Acquisition Parameters  
 Date\_: 20200115  
 Time: 16:34  
 INSTR: 670  
 PROBHD: 5 mm CPYCH-1H  
 PULPROG: zgpg30  
 TD: 48074  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 8012.820 Hz  
 FIDRES: 0.16667 Hz  
 AQ: 2.3998677 sec  
 RG: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 0.10000000 sec  
 T2: 0.00000000 sec  
 T3: 0.00000000 sec  
 T4: 0.00000000 sec  
 T5: 0.00000000 sec  
 T6: 0.00000000 sec  
 T7: 0.00000000 sec  
 T8: 0.00000000 sec  
 T9: 0.00000000 sec  
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 T11: 0.00000000 sec  
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 T98: 0.00000000 sec  
 T99: 0.00000000 sec  
 T100: 0.00000000 sec

Z-restored spin-echo 13C spectrum with 1H decoupling





gcosy60

```

Current Data Parameters
USER      Khevit11
NAME      ACH-1-117-2
EXPNO     4
PROCNO    1

P1 - Acquisition Parameters
Date_     20200715
Time      19.05
INSTRUM   cryo500
PROBHD    5 mm cryo1H-1
PULPROG   zgpg30
TD         2048
SOLVENT   CDCl3
NS         1
DS         16
AQ         8013.85 Hz
FIDRES    3.912510 Hz
AQ         0.1278452 sec
RG         228.1
DM         62.400 usec
DE         228.0 usec
TE         298.0
d0         0.00003300 sec
d1         1.00000000 sec
d13        0.00003300 sec
d16        0.00020000 sec
d10        0.00014900 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
SFO1       500.225015 MHz

===== GRADIENT CHANNEL =====
GPRM1     sine.100
GPRM2     sine.100
GPRM3     0.00 %
GPRM4     0.00 %
GPRM5     0.00 %
GPRM6     0.00 %
GPRM7     17.00 %
GPRM8     0.00 %
PR6       1000.00 usec

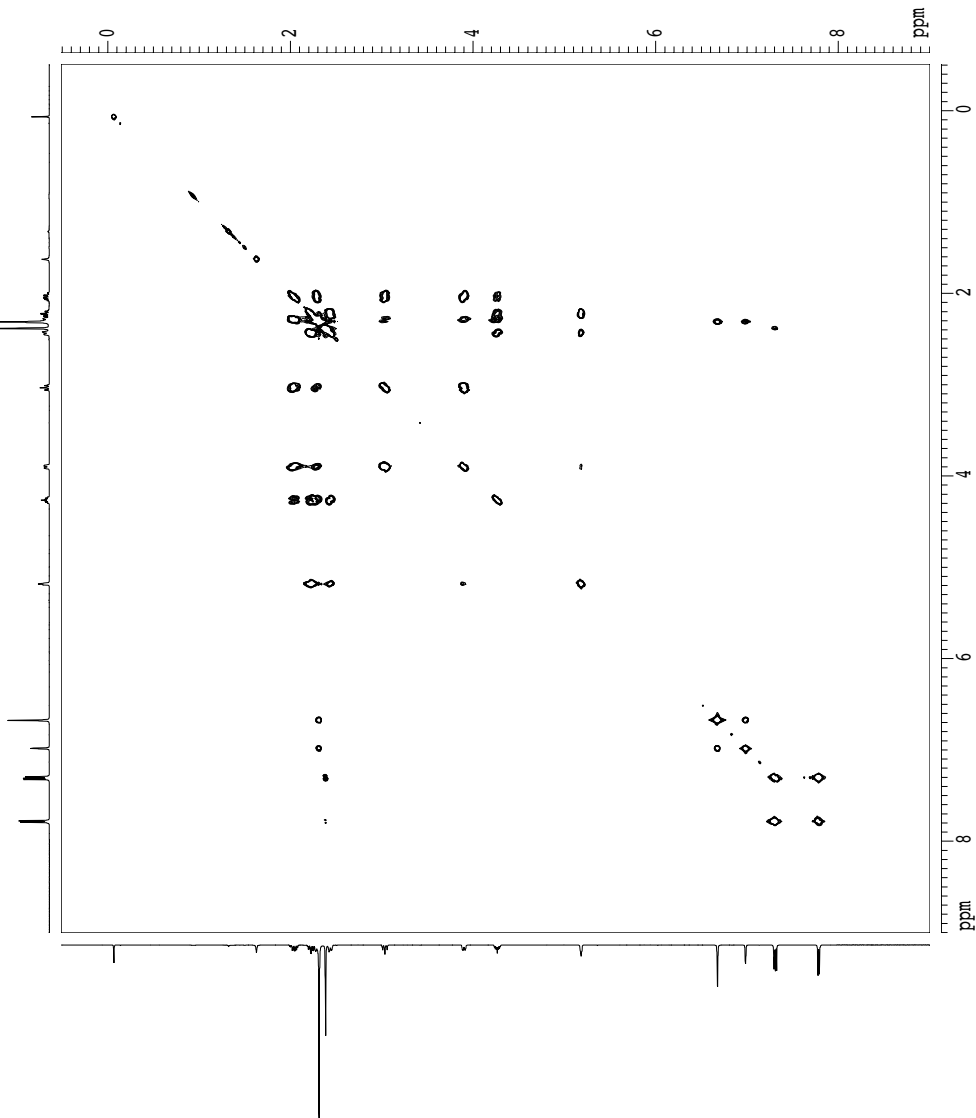
P2 - Acquisition parameters
NUC2       13C
P2         10.00 usec
SFO2       125.761150 MHz
SF02       500.225015 MHz

===== GRADIENT CHANNEL =====
GPRM1     sine.100
GPRM2     sine.100
GPRM3     0.00 %
GPRM4     0.00 %
GPRM5     0.00 %
GPRM6     0.00 %
GPRM7     17.00 %
GPRM8     0.00 %
PR6       1000.00 usec

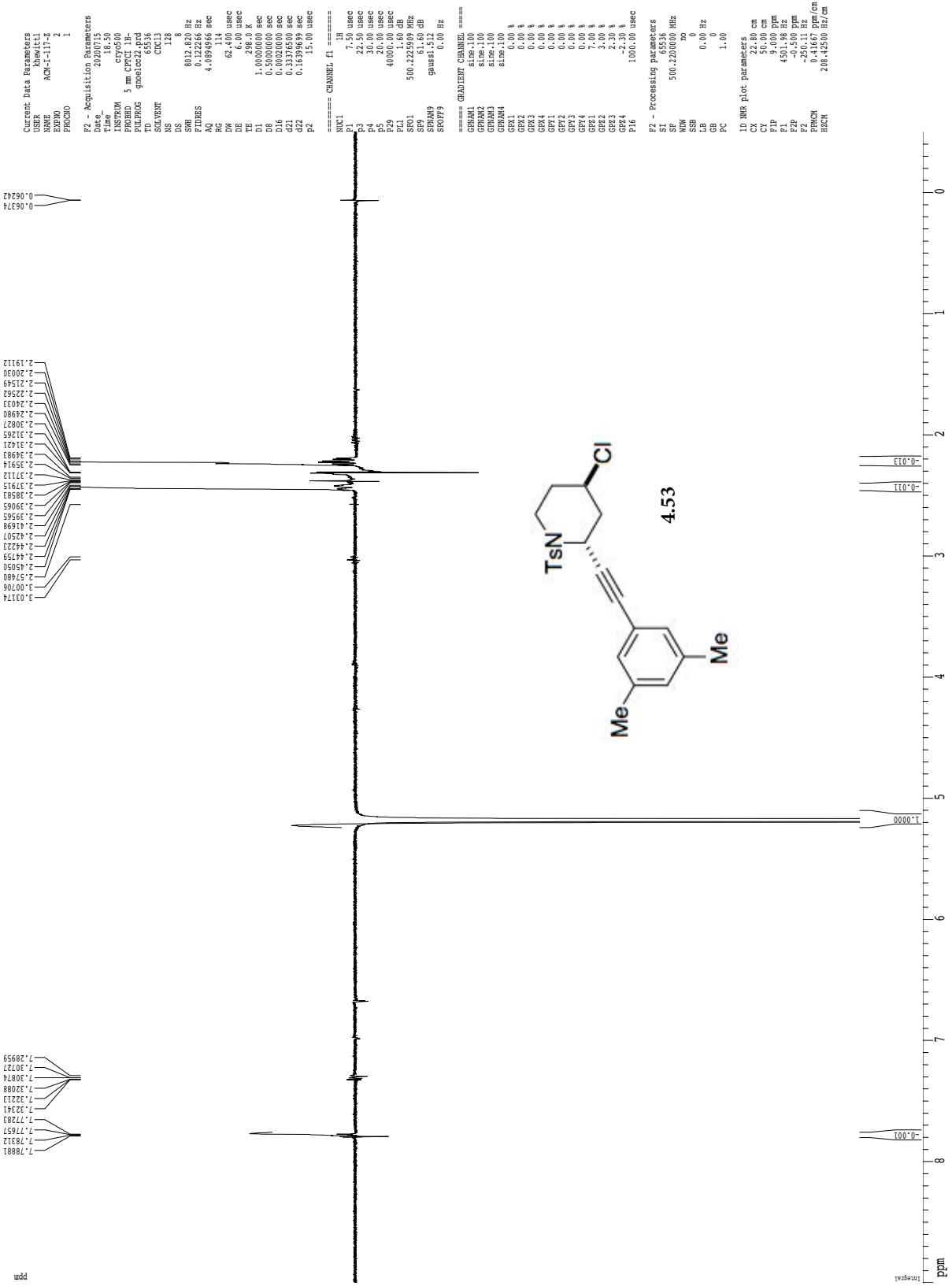
P3 - Processing parameters
SI         32768
SF         500.2200000 MHz
WDW        EM
SSB        0
GB         0.00 Hz
PC         1.00

P4 - Processing parameters
SI         1024
SF         500.2200000 MHz
WDW        EM
SSB        0
GB         0.00 Hz

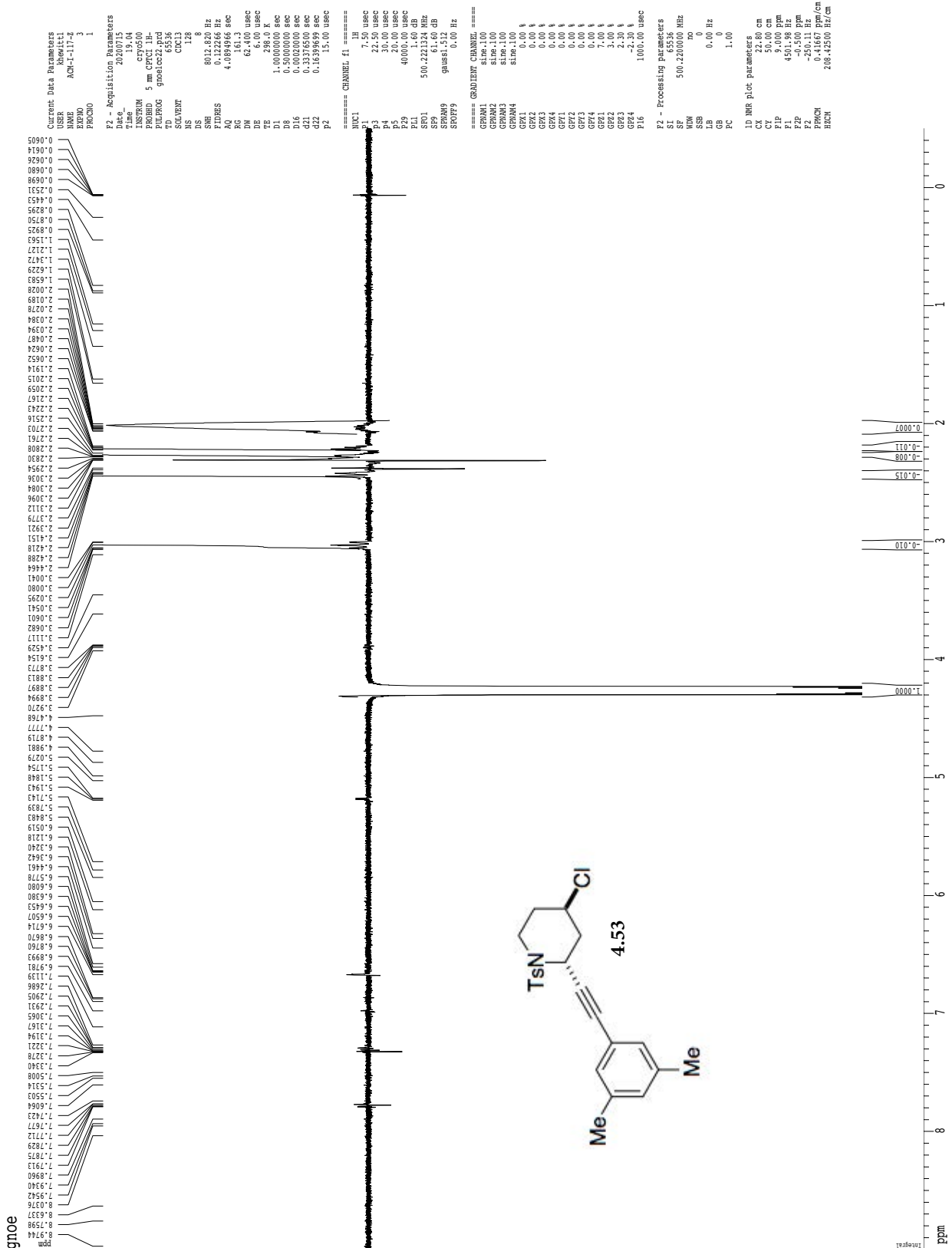
2D NMR plot parameters
CX2        15.00 cm
F2P1O     15.00 cm
F2P1R     5.002 ppm
F2P1I     45.0569 Hz
F2P1M     -254.47 Hz
F1P1O     5.002 ppm
F1P1R     4503.14 Hz
F1P1I     0.509 ppm
F1P1M     -2.00 Hz
F2PRCM    0.63407 ppm/cm
F1PRCM    317.17416 Hz/cm
F1BPCM    0.63407 ppm/cm
F1BPCM    317.17416 Hz/cm
  
```



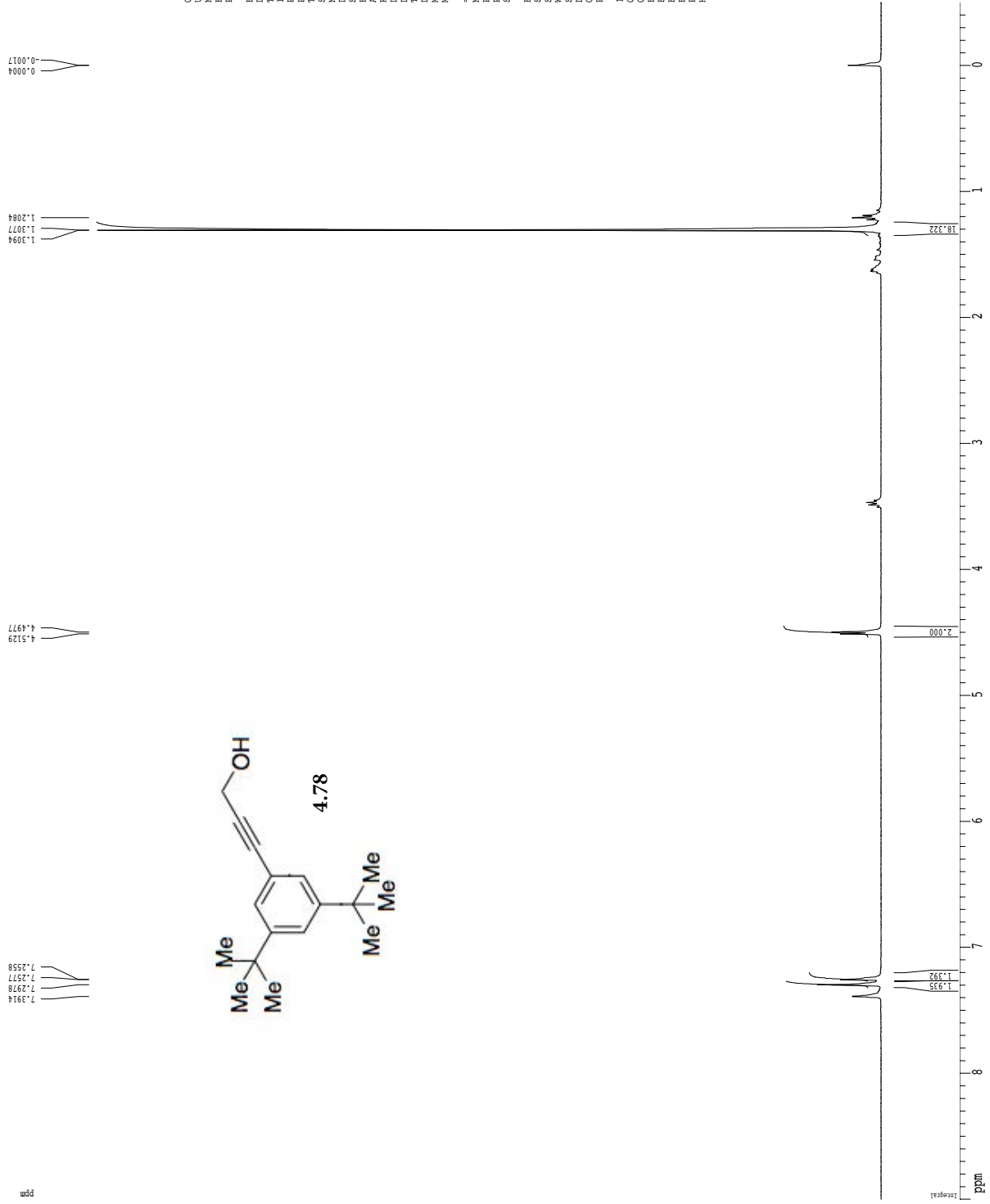
gnoe





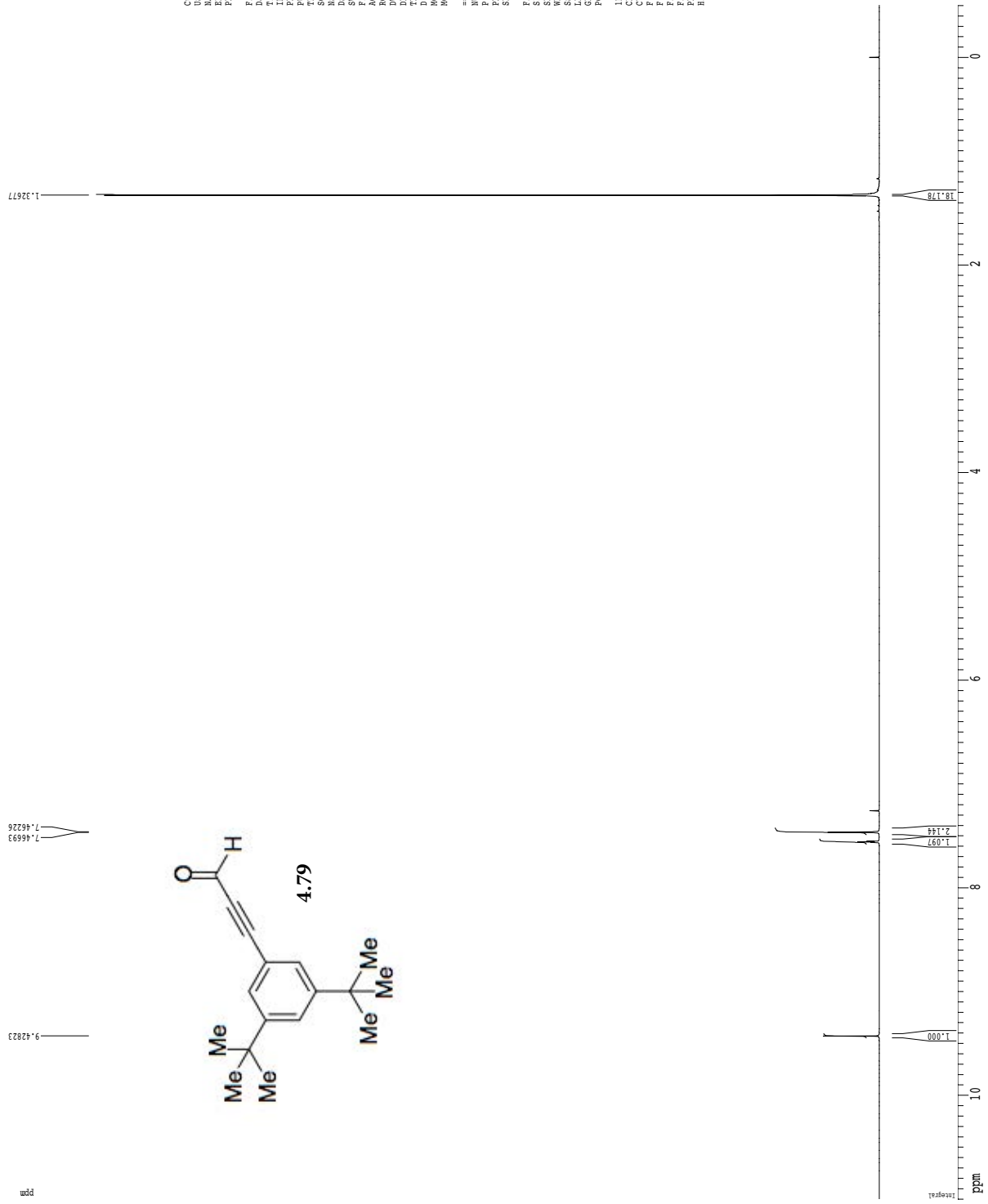


1H spectrum



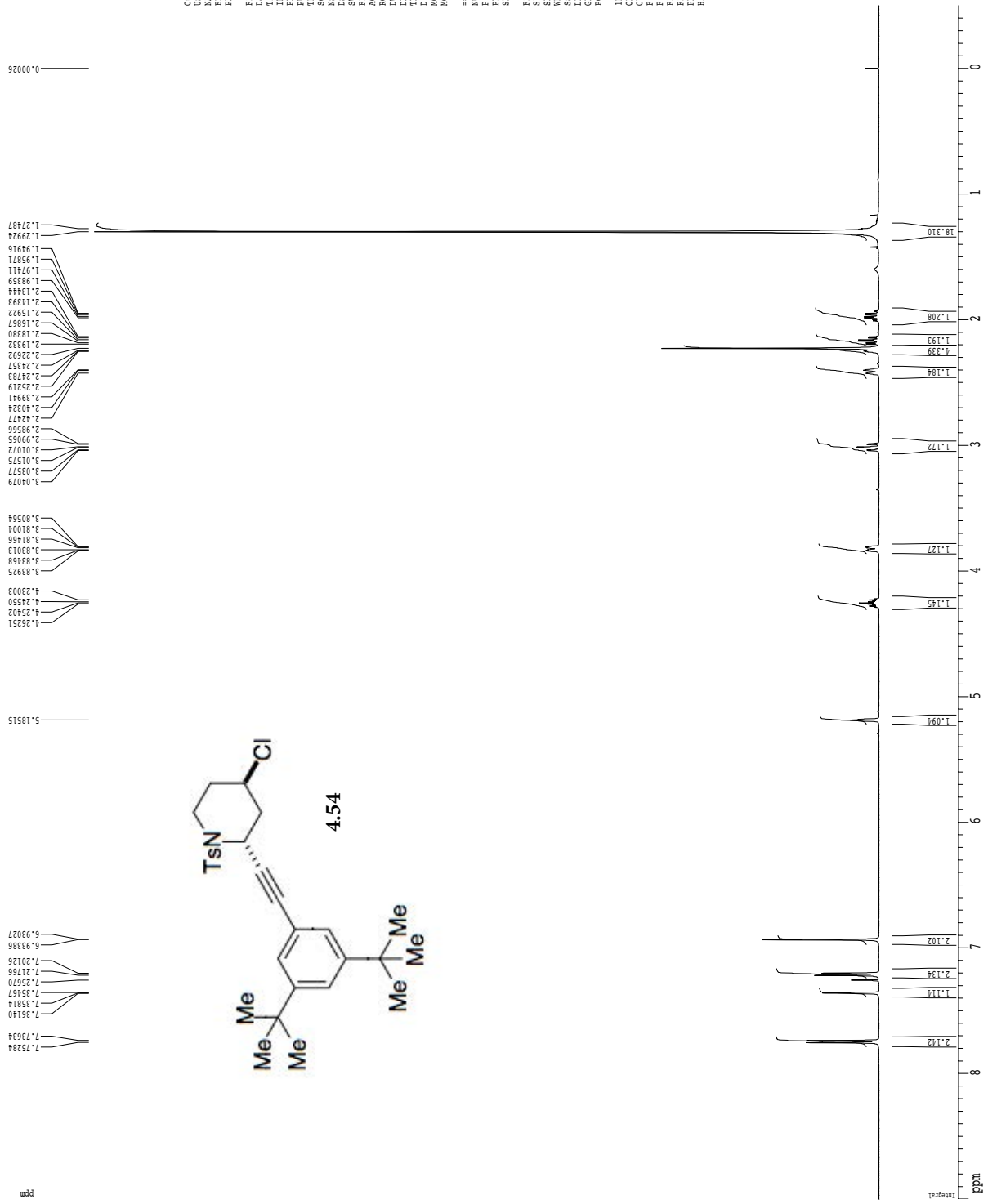
Current Data Parameters  
USER: Kowett  
NAME: ACH-1-150-pdt-all  
EXPNO: 1  
PROCNO: 1  
F2 - Acquisition Parameters  
Date\_: 20200121  
Time: 9:59  
PROBHD: 5 mm QNP 1H/1  
PULPROG: zg30  
TD: 65536  
SOLVENT: CDCl3  
DS: 9  
SHE: 6410.258 Hz  
FIDRES: 0.097813 Hz  
AQ: 5.118577 sec  
RG: 384  
DE: 78.000 usec  
TE: 298.2 K  
T1: 0.100000 sec  
T1RHO: 0.000000 sec  
T1DELTA: 0.000000 sec  
T1REPEAT: 0.000000 sec  
T1ROFFS: 0.000000 sec  
T1SCAN: 0.000000 sec  
T1SLOPE: 0.000000 sec  
T1SPIN: 0.000000 sec  
T1TILT: 0.000000 deg  
T1XFLIP: 0.000000 sec  
T1YFLIP: 0.000000 sec  
T1ZFLIP: 0.000000 sec  
T2: 12.00 usec  
P1: 0.00 usec  
P11: -1.10 dB  
SFO1: 400.1328009 MHz  
F2 - Processing Parameters  
SI: 65536  
SF: 400.1300218 MHz  
WDW: EM  
SSB: 0  
LB: 0.30 Hz  
GB: 0  
PC: 2.00  
ID: NMR file parameters  
CY: 22.80 cm  
C1: 15.00 cm  
F1P: 9.000 ppm  
F1: 360.11 Hz  
F2: 360.11 Hz  
F2: 360.11 Hz  
F3: 360.11 Hz  
PRFCH: 0.414967 ppm/cm  
HCHN: 166.72086 Hz/cm

1H spectrum



Current Data Parameters  
 USER Kowalcz  
 NAME ACH-151-pch-1b-1  
 EXNO 1  
 PROCNO 1  
 P2 - Acquisition Parameters  
 Date\_ 20200124  
 Time 10.14  
 INSTRUM spect  
 PROBHD 5 mm QNP HET  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 DS 2  
 SC 2  
 SFO 6410.256 Hz  
 FIDRES 0.097813 Hz  
 AQ 5.1118579 sec  
 RG 655.36  
 DD 78.000 usec  
 DE 4.50 usec  
 TE 298.2 K  
 D1 1.000000 sec  
 MCHRG 0.000000 sec  
 ACQREK 0.00500000 sec  
 ===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.1328009 MHz  
 P2 - Processing Parameters  
 SI 65536  
 SF 400.130024 MHz  
 WDM EM  
 LB 0.30 Hz  
 GB 0  
 PC 2.00  
 ID NMR File Parameters  
 CY 22.80 cm  
 CV 15.00 cm  
 FIP 11.000 ppm  
 FL 4400.48 Hz  
 F2 200.00 MHz  
 P1 2000.00 Hz  
 PPMCM 0.50439 ppm/cm  
 HZCM 201.81998 Hz/cm

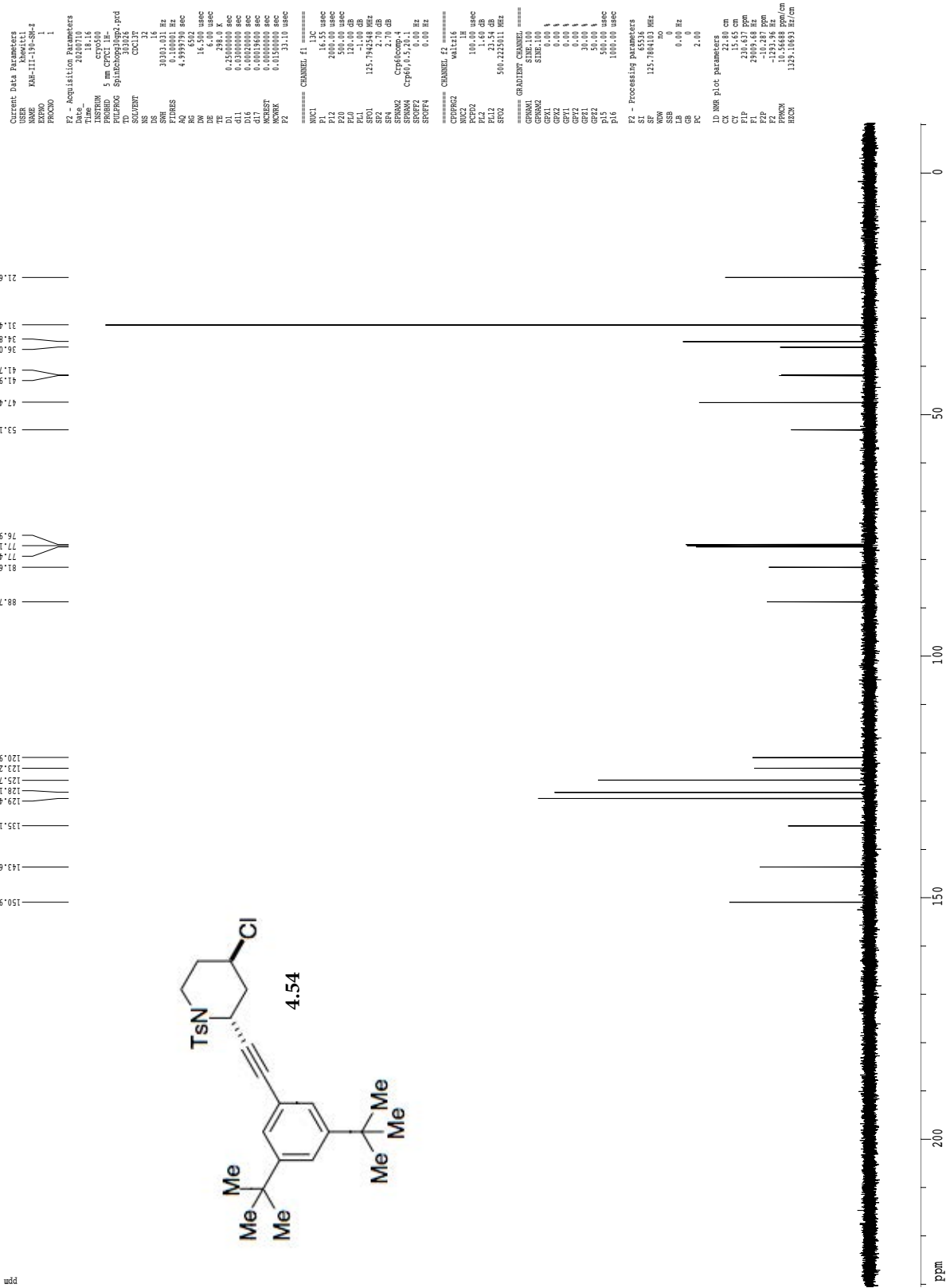
1H spectrum

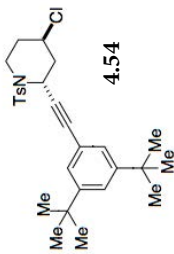


Current Data Parameters  
 USER: Kowalski  
 NAME: KAP-III-194-SM-2  
 EXNO: 3  
 PROCNO: 1

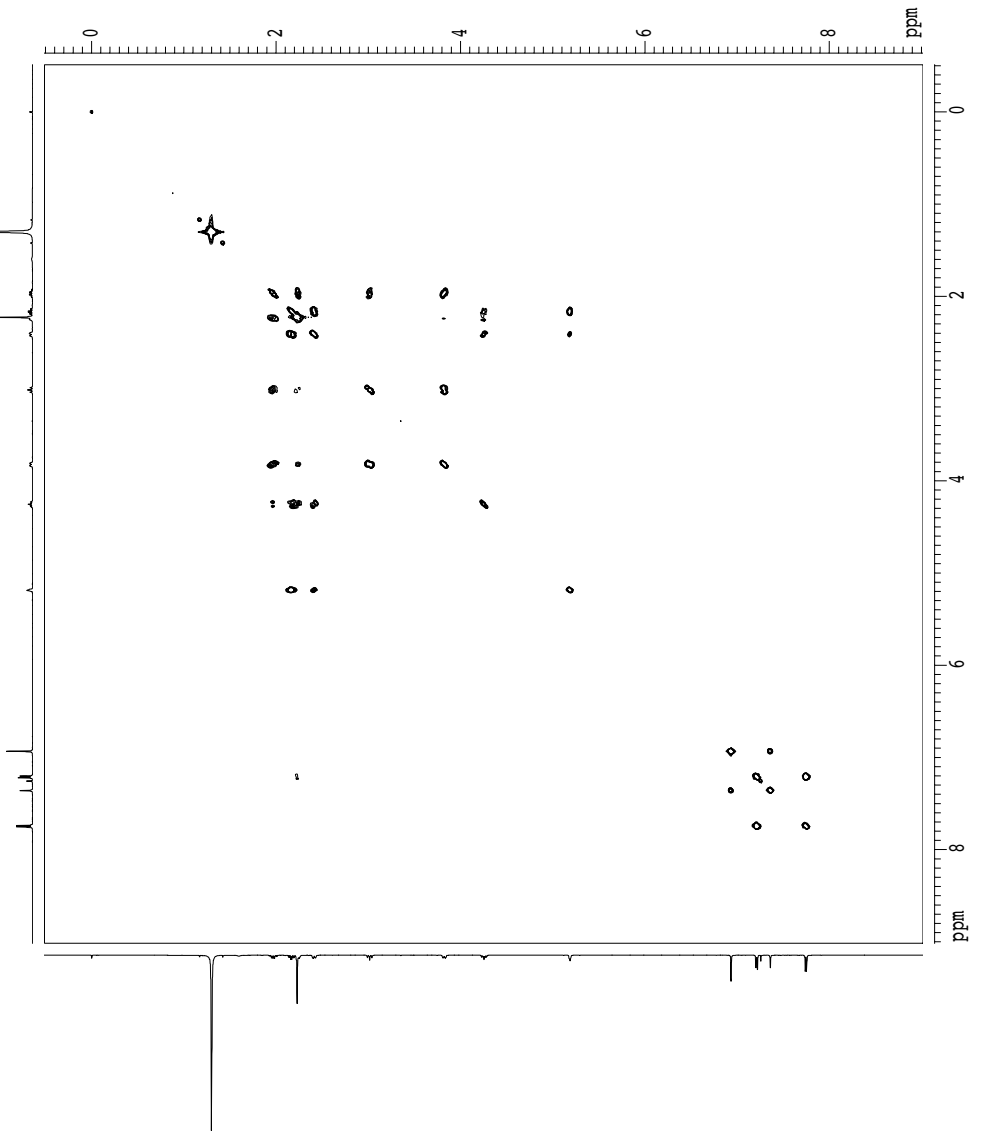
F2 - Acquisition Parameters  
 Date\_: 20200111  
 Time: 10.10  
 PROBNM: 07910  
 PROCNO: 3  
 PULPROG: zgpg30  
 TD: 48074  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 500.136260 MHz  
 SHF: 8012.820 Hz  
 FIDRES: 0.16667 Hz  
 AQ: 2.3398677 sec  
 RG: 327.500  
 DD: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 0.100000 sec  
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 T98: 0.100000 sec  
 T99: 0.100000 sec  
 T100: 0.100000 sec

Z-restored spin-echo <sup>13</sup>C spectrum with <sup>1</sup>H decoupling





gc05y60



Current Data Parameters  
 USER NAME KAF-III-190-SK-Z  
 EXPERNO 7  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20200711  
 Time\_ 10.45  
 INSTRUM cryo500  
 PROBHD 5 mm CPCTC IH-  
 PULPROG zgpg30  
 SOLVENT CDCl3  
 NS 1  
 DS 16  
 SWH 8012.820 Hz  
 FIDRES 3.27653 Hz  
 RG 64  
 AQ 6.4  
 DM 62.400 usec  
 DE 6.00 usec  
 TE 298.2 K  
 D1 0.000000 sec  
 D11 1.000000 sec  
 d13 0.000000 sec  
 D16 0.0002000 sec  
 INO 0.0001280 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.50 usec  
 PL1 1.60 dB  
 SFO1 500.2235015 MHz

===== GRANITUM CHANNEL =====  
 GPRM1 size:100  
 GPRM2 size:100  
 GPR1 0.00 %  
 GPR2 0.00 %  
 GPR3 0.00 %  
 GPR4 0.00 %  
 GPR5 17.00 %  
 GPR6 17.00 %  
 P16 1000.00 usec

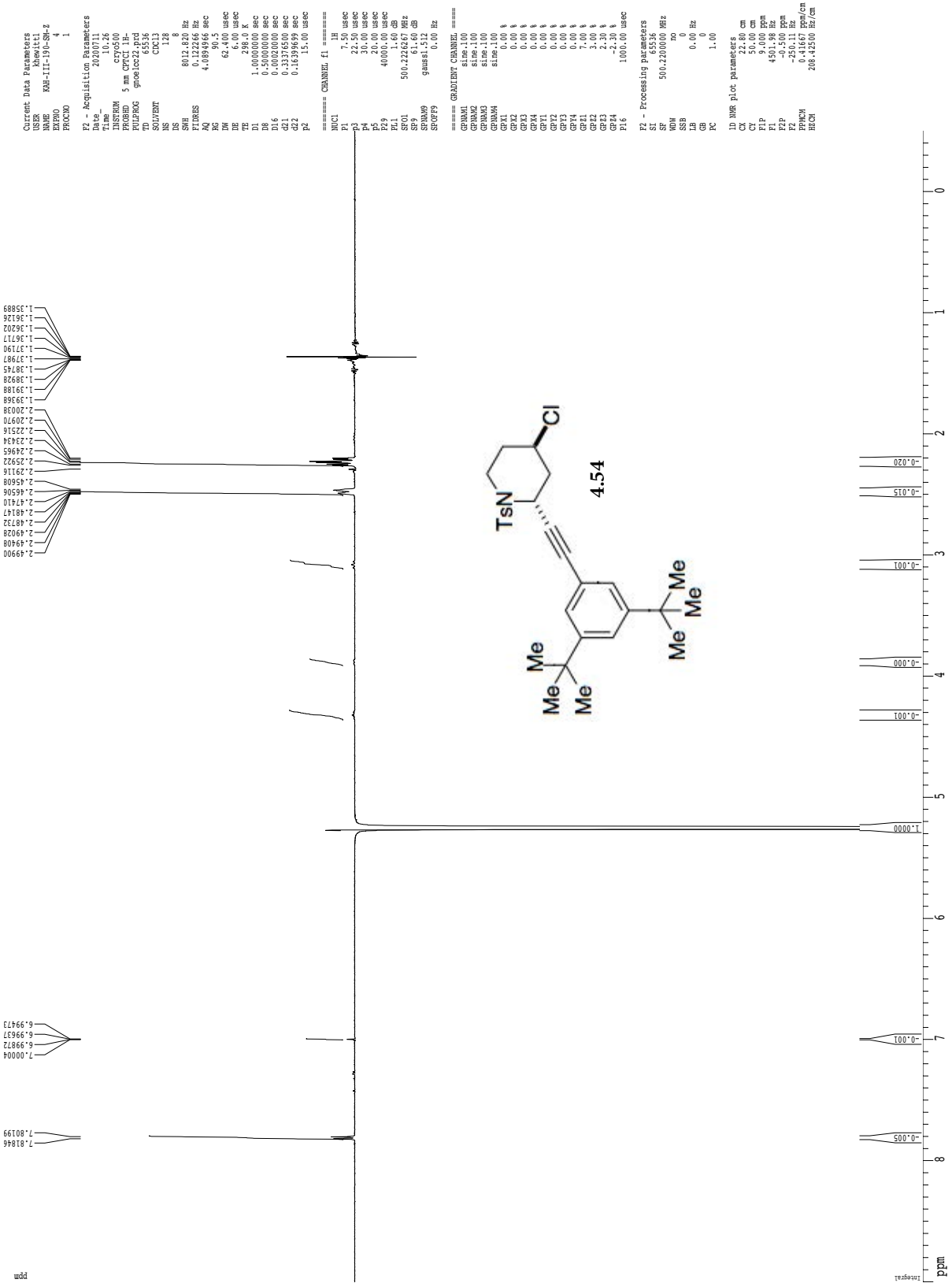
F1 - Acquisition parameters  
 NU0 512  
 TD 512  
 SFO1 500.2235 MHz  
 FIDRES 15.650040 Hz  
 SN 16.018 ppm  
 FWHM 0.0

F2 - Processing parameters  
 SI 1024  
 SF 500.2200330 MHz  
 SFO 500.2200330 MHz  
 DS 0  
 EN 0  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 16384  
 SF 500.2200330 MHz  
 SFO 500.2200330 MHz  
 DS 0  
 EN 0  
 GB 0  
 PC 1.00

2D NMR plot parameters  
 CZ 15.00 cm  
 CX 15.00 cm  
 FZLO 4596.73 ppm  
 FZHI -0.512 ppm  
 FZFI -256.15 Hz  
 FZFO 9.015 ppm  
 FZLO 4596.29 Hz  
 FZHI -256.15 ppm  
 FZFO 9.015 ppm/cm  
 FZPCHN 0.65511 ppm/cm  
 FZPCMN 317.65986 Hz/cm  
 FZPCMN 317.65986 Hz/cm

gnoe



gnoe

ppm

1.10559  
3.11013  
3.08142  
3.07677  
3.05644  
3.05203  
2.99373  
2.90098  
2.89009  
2.87412  
2.86988  
2.86512  
2.85887  
2.85069  
2.83339  
2.80939  
2.80216  
2.79315  
2.78838  
2.78294  
2.77348  
2.76666  
2.72018  
2.05063  
2.04125  
2.02772  
2.01515  
1.99828  
1.98859  
1.98163  
1.97808  
1.97088  
1.96498  
1.95778  
1.95077  
1.93976

5.24992  
5.25678

6.99344  
6.99199  
7.00090

7.80293

Current Data Parameters  
USER khewitt1  
NAME KAH-111-19A-SH-2  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20200711  
Time 14:14  
INSTRUM spect  
PROBHD 5 mm CPYCI 1H  
PULPROG gnoe.ces22.prd  
TD 65536  
AQ 0.113  
RG 121  
NS 8  
DS 8  
SWH 8012.822 Hz  
FIDRES 0.112266 Hz  
AQ 4.10979 sec  
RG 40.5  
DM 62.400 usec  
DE 6.00 usec  
TE 300.2 K  
D1 1.00000000 sec  
D2 0.50000000 sec  
D16 0.00020000 sec  
d21 0.33376500 sec  
d22 0.18396600 sec  
d2 1.5100 usec

===== CHANNEL f1 =====  
NUC1 1H  
P1 1.00 usec  
PL1 22.50 dB  
PC 30.00 usec  
P2 20.00 usec  
PL2 40000.00 usec  
PC2 30.00 usec  
SFO1 500.2221610 MHz  
SFO2 500.2221610 MHz  
SFO3 61.66 dB  
SFO4 61.66 dB  
SFO5 0.00 Hz  
SFO6 0.00 Hz  
SFO7 0.00 Hz  
SFO8 0.00 Hz  
SFO9 0.00 Hz  
SFO10 0.00 Hz  
SFO11 0.00 Hz  
SFO12 0.00 Hz  
SFO13 0.00 Hz  
SFO14 0.00 Hz  
SFO15 0.00 Hz  
SFO16 0.00 Hz  
SFO17 2.30 Hz  
SFO18 -2.30 Hz  
SFO19 1000.00 usec  
SFO20 1000.00 usec

===== GRADIENT CHANNEL =====  
GRDM1 size:100  
GRDM2 size:100  
GRDM3 size:100  
GRDM4 size:100  
GX1 size:0.00 Hz  
GX2 size:0.00 Hz  
GX3 size:0.00 Hz  
GX4 size:0.00 Hz  
GX5 size:0.00 Hz  
GX6 size:0.00 Hz  
GX7 size:0.00 Hz  
GX8 size:0.00 Hz  
GX9 size:0.00 Hz  
GX10 size:0.00 Hz  
GX11 size:0.00 Hz  
GX12 size:0.00 Hz  
GX13 size:0.00 Hz  
GX14 size:0.00 Hz  
GX15 size:0.00 Hz  
GX16 size:0.00 Hz  
GX17 size:0.00 Hz  
GX18 size:0.00 Hz  
GX19 size:0.00 Hz  
GX20 size:0.00 Hz

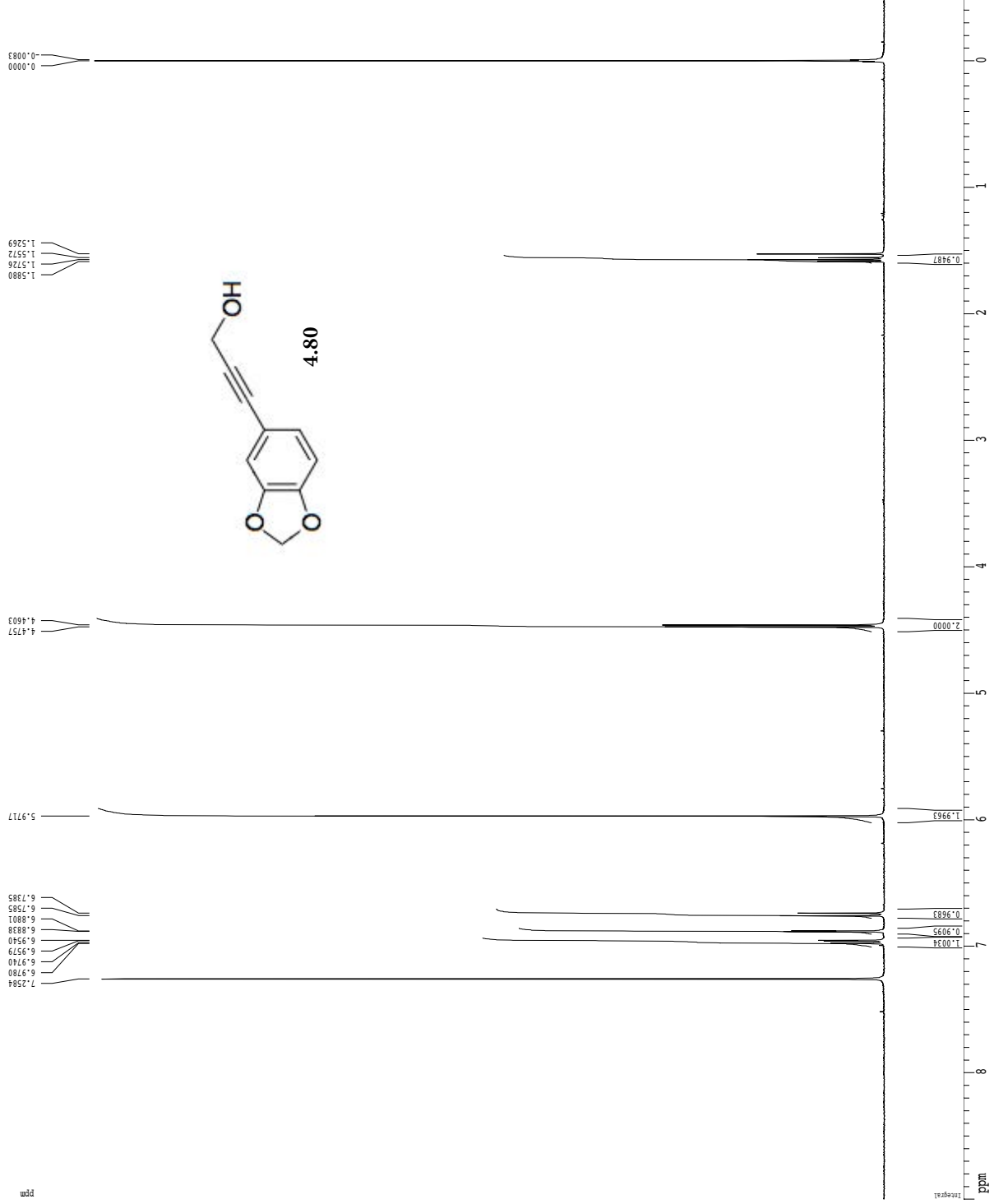
F2 - Processing parameters  
SI 65536  
SF 500.220000 MHz  
WDW no  
SSB no  
LB 0.00 Hz  
GB 0  
PC 1.00

ID MR plot parameters  
CX 22.88 cm  
CY 50.00 cm  
FID 9.000 ppm  
F1P 4.000 ppm  
F2P -0.500 ppm  
F2 -250.11 Hz  
FPMCN 0.41667 ppm/cm  
HECN 206.42500 Hz/cm



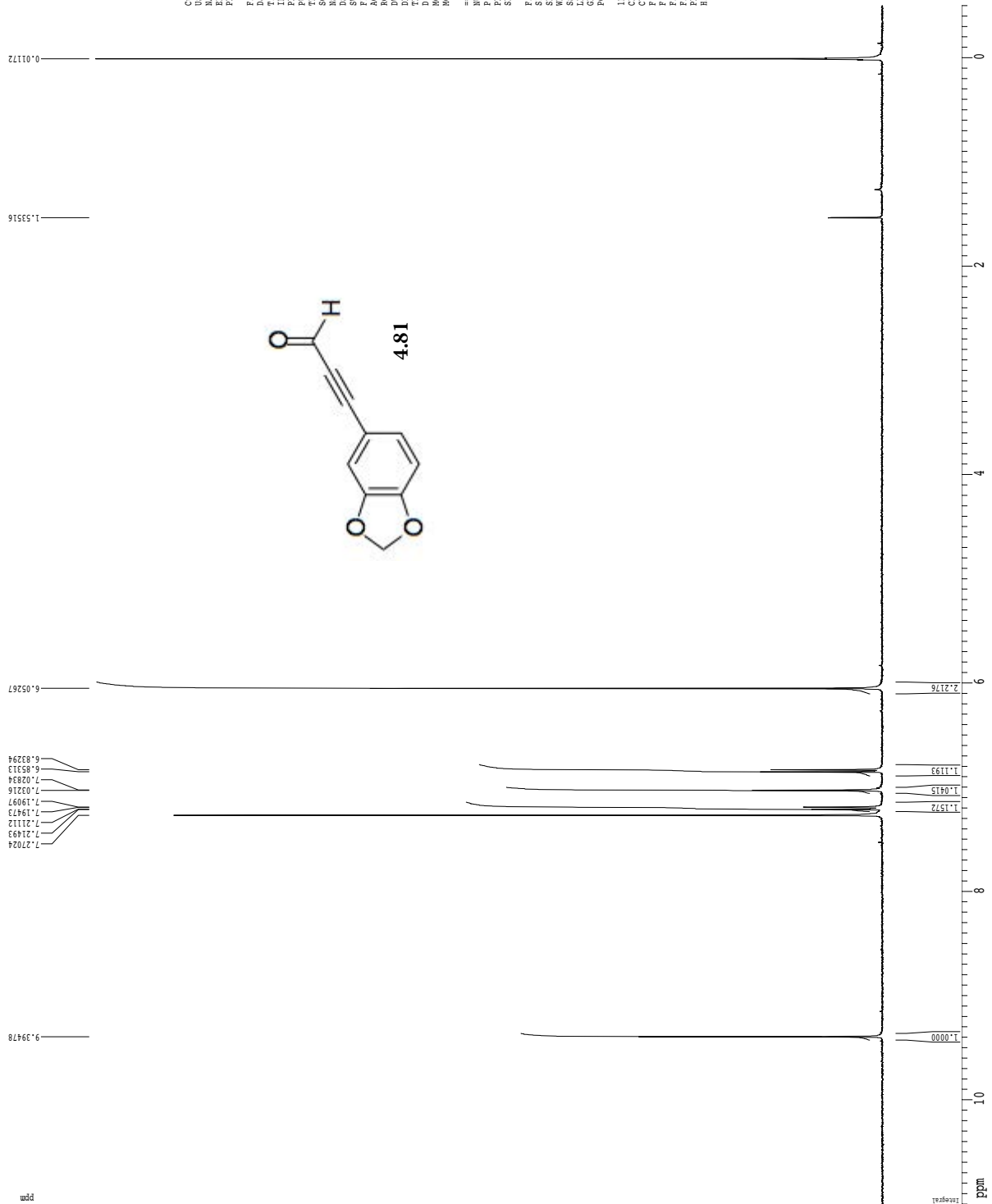


1H spectrum



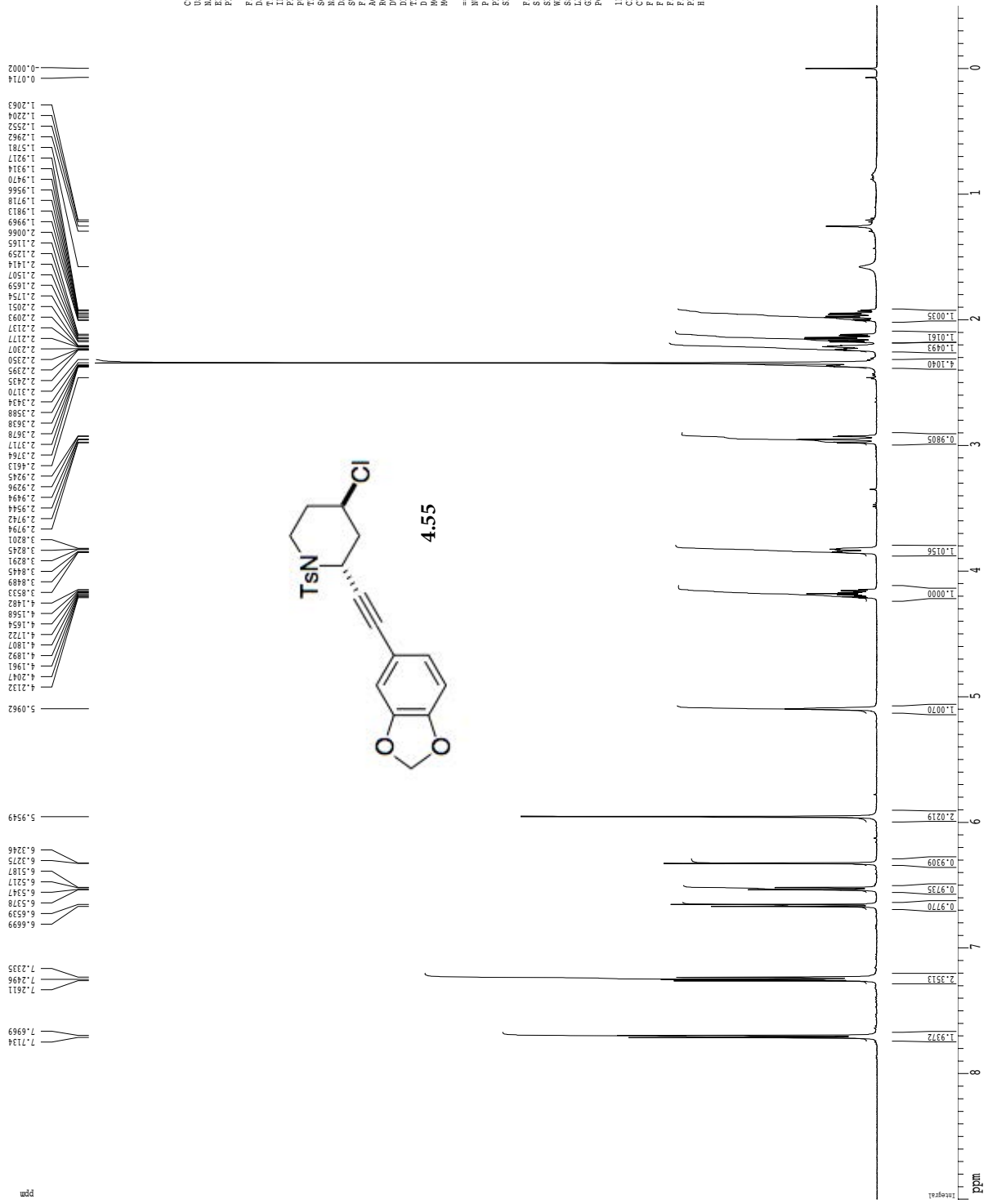
Current Data Parameters  
 USER Kowalcz  
 NAME ACH-154-b1-g  
 EXNO 1  
 PROCSO 1  
 P2 - Acquisition Parameters  
 Date\_ 20200130  
 Time 9:43  
 INSTRON HPLC  
 PROBHD 5 mm QNP HPLC  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 DS 9  
 SFO 6410.258 Hz  
 FIDRES 0.097813 Hz  
 AQ 5.118579 sec  
 RG 384.00  
 DW 28.000 usec  
 DE 4.50 usec  
 TE 298.2 K  
 T1 0.100000 sec  
 T2 0.000000 sec  
 MCHRES 0.000000 Hz  
 MSRES 0.0050000 sec  
 ===== CHANNEL f1 =====  
 NUC1 12.00 usec  
 P1 1.10 dB  
 SFO1 400.132809 MHz  
 P2 - Processing Parameters  
 SI 65536  
 SF 400.130021 MHz  
 WDM EN  
 LB 0.30 Hz  
 GB 0  
 PC 2.00  
 ID WDR P1 of Parameters  
 CY 22.80 cm  
 CV 15.00 cm  
 FIP 9.000 ppm  
 FL 3600.17 Hz  
 F2 200.000 MHz  
 PPMCM 0.41667 ppm/cm  
 HZCM 166.72086 Hz/cm

1H spectrum



Current Data Parameters  
USER: Kowalett1  
NAME: AOM-1155-4-1-ppt  
EXPNO: 1  
PROCNO: 1  
F2 - Acquisition Parameters  
Date\_: 20200203  
Time: 16:48  
PROBHD: 5 mm QNP 1H/13  
PULPROG: zg30  
TD: 65536  
SOLVENT: CDCl3  
DS: 2  
SHE: 6410.258 Hz  
FIDRES: 0.097811 Hz  
AQ: 5.118577 sec  
RG: 384  
DSO: 28.000 usec  
DE: 4.50 usec  
TE: 297.2 K  
MAGRES: 0.1000000 sec  
MORPH: 0.0000000 sec  
MORPH: 0.0000000 sec  
MORPH: 0.0000000 sec  
===== CHANNEL f1 =====  
NUC1: 13C  
P1: 12.00 usec  
PL1: -1.10 dB  
SFO1: 400.1328009 MHz  
F2 - Processing Parameters  
SI: 65536  
SF: 400.1300175 MHz  
WDW: EM  
SSB: 0  
GB: 0  
PC: 2.00  
ID: NMR PLOT Parameters  
CY: 22.80 cm  
CY: 15.00 cm  
FIP: 11.000 ppm  
FL: 4400.48 Hz  
FZ: 0.0000000 ppm  
PZ: -2000.00 Hz  
PRGCM: 0.50439 ppm/cm  
HZCM: 201.81996 Hz/cm

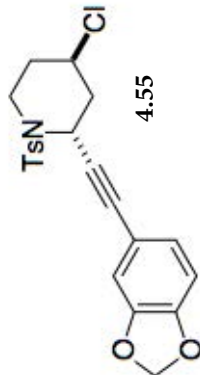
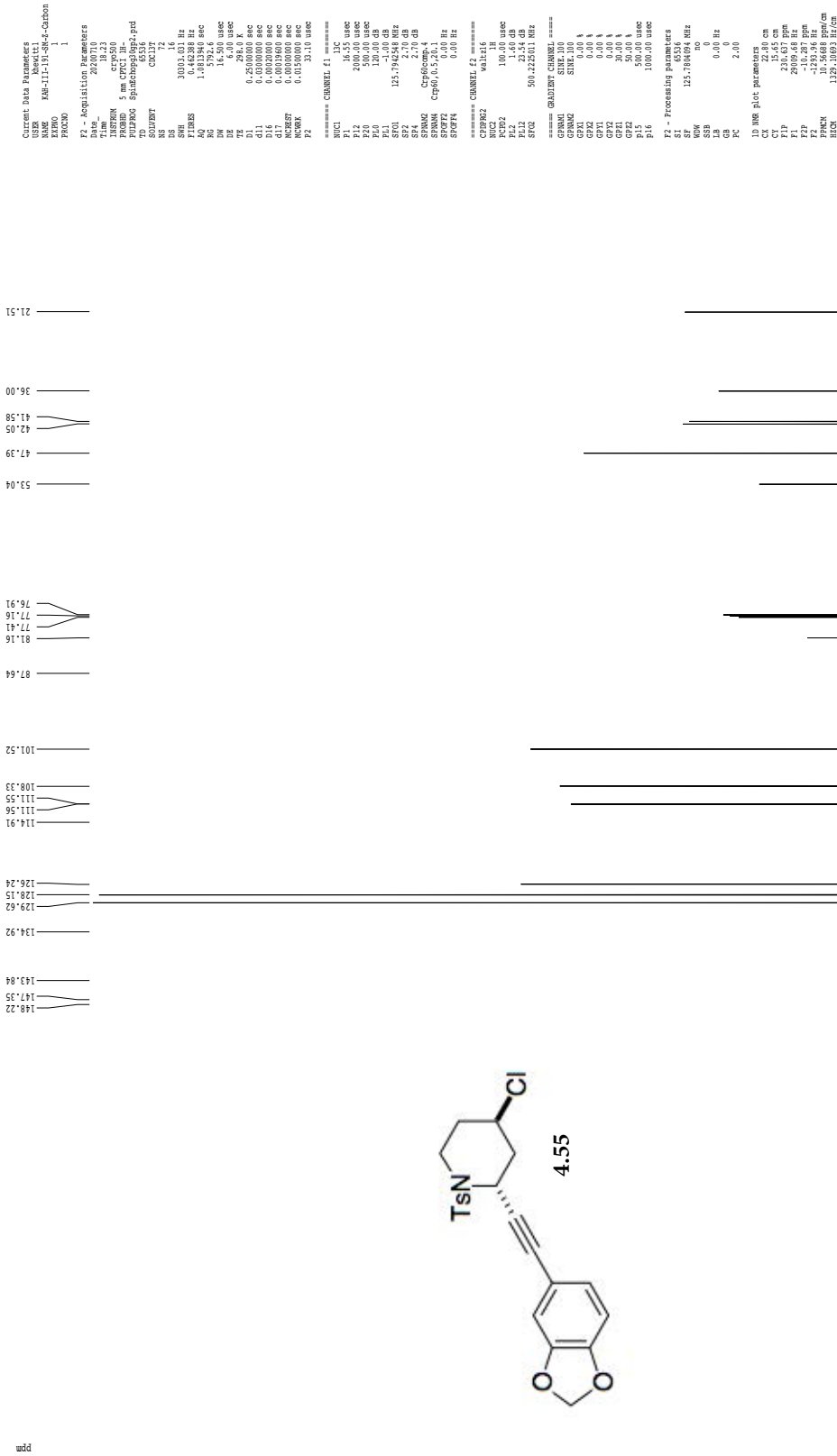
1H spectrum



Current Data Parameters  
 USER: khawatt  
 NAME: KHU-III-191-SH-2  
 EXNO: 7  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20200714  
 Time: 10:41  
 Operator: khawatt  
 PULPROG: zgpg30  
 PROCNO: 5  
 TD: 48074  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 8012.820 Hz  
 FIDRES: 0.166677 Hz  
 AQ: 2.3998677 sec  
 RG: 62.400  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 0.100000 sec  
 T2: 0.000000 sec  
 T3: 0.000000 sec  
 T4: 0.000000 sec  
 T5: 0.000000 sec  
 T6: 0.000000 sec  
 T7: 0.000000 sec  
 T8: 0.000000 sec  
 T9: 0.000000 sec  
 T10: 0.000000 sec  
 T11: 0.000000 sec  
 T12: 0.000000 sec  
 T13: 0.000000 sec  
 T14: 0.000000 sec  
 T15: 0.000000 sec  
 T16: 0.000000 sec  
 T17: 0.000000 sec  
 T18: 0.000000 sec  
 T19: 0.000000 sec  
 T20: 0.000000 sec  
 T21: 0.000000 sec  
 T22: 0.000000 sec  
 T23: 0.000000 sec  
 T24: 0.000000 sec  
 T25: 0.000000 sec  
 T26: 0.000000 sec  
 T27: 0.000000 sec  
 T28: 0.000000 sec  
 T29: 0.000000 sec  
 T30: 0.000000 sec  
 T31: 0.000000 sec  
 T32: 0.000000 sec  
 T33: 0.000000 sec  
 T34: 0.000000 sec  
 T35: 0.000000 sec  
 T36: 0.000000 sec  
 T37: 0.000000 sec  
 T38: 0.000000 sec  
 T39: 0.000000 sec  
 T40: 0.000000 sec  
 T41: 0.000000 sec  
 T42: 0.000000 sec  
 T43: 0.000000 sec  
 T44: 0.000000 sec  
 T45: 0.000000 sec  
 T46: 0.000000 sec  
 T47: 0.000000 sec  
 T48: 0.000000 sec  
 T49: 0.000000 sec  
 T50: 0.000000 sec  
 T51: 0.000000 sec  
 T52: 0.000000 sec  
 T53: 0.000000 sec  
 T54: 0.000000 sec  
 T55: 0.000000 sec  
 T56: 0.000000 sec  
 T57: 0.000000 sec  
 T58: 0.000000 sec  
 T59: 0.000000 sec  
 T60: 0.000000 sec  
 T61: 0.000000 sec  
 T62: 0.000000 sec  
 T63: 0.000000 sec  
 T64: 0.000000 sec  
 T65: 0.000000 sec  
 T66: 0.000000 sec  
 T67: 0.000000 sec  
 T68: 0.000000 sec  
 T69: 0.000000 sec  
 T70: 0.000000 sec  
 T71: 0.000000 sec  
 T72: 0.000000 sec  
 T73: 0.000000 sec  
 T74: 0.000000 sec  
 T75: 0.000000 sec  
 T76: 0.000000 sec  
 T77: 0.000000 sec  
 T78: 0.000000 sec  
 T79: 0.000000 sec  
 T80: 0.000000 sec  
 T81: 0.000000 sec  
 T82: 0.000000 sec  
 T83: 0.000000 sec  
 T84: 0.000000 sec  
 T85: 0.000000 sec  
 T86: 0.000000 sec  
 T87: 0.000000 sec  
 T88: 0.000000 sec  
 T89: 0.000000 sec  
 T90: 0.000000 sec  
 T91: 0.000000 sec  
 T92: 0.000000 sec  
 T93: 0.000000 sec  
 T94: 0.000000 sec  
 T95: 0.000000 sec  
 T96: 0.000000 sec  
 T97: 0.000000 sec  
 T98: 0.000000 sec  
 T99: 0.000000 sec  
 T100: 0.000000 sec

Z-restored spin-echo 13C spectrum with 1H decoupling



```

Current Data Parameters
USR   Khowtel
XPRO  XH-11-131-13C-Carb
PROC  1

F2 - Acquisition Parameters
Date_  2010110
Time_  07:50:21
PROBHD 5 mm CPTCI H-
PULPROG zgpg30
TD      65536
SOLVENT CDCl3
NS      16
DS      4
SWH     31083.031 Hz
AQ      1.9034840 Hz
RG      1792.4
WDW     EM
SS      16.500 usec
SL      16.500 usec
TE      298.0 K
D1      0.35000000 sec
d11     0.00100000 sec
D16     0.00200000 sec
d17     0.00100000 sec
AQ17    0.00200000 sec
PCP17   125.794348 MHz
RG17    2.70 dB
SFO17   500.136199 MHz
SFO18   125.761330 MHz
SFO19   101.626182 MHz
SFO20   101.626182 MHz
SFO21   101.626182 MHz
SFO22   101.626182 MHz
SFO23   101.626182 MHz
SFO24   101.626182 MHz
SFO25   101.626182 MHz
SFO26   101.626182 MHz

===== CHANNEL f1 =====
NUC1    13C
P1      15.00 usec
PL1     -1.00 dB
PL2     -1.00 dB
PL3     -1.00 dB
PL4     -1.00 dB
PL5     -1.00 dB
PL6     -1.00 dB
PL7     -1.00 dB
PL8     -1.00 dB
PL9     -1.00 dB
PL10    -1.00 dB
PL11    -1.00 dB
PL12    -1.00 dB
PL13    -1.00 dB
PL14    -1.00 dB
PL15    -1.00 dB
PL16    -1.00 dB
PL17    -1.00 dB
PL18    -1.00 dB
PL19    -1.00 dB
PL20    -1.00 dB
PL21    -1.00 dB
PL22    -1.00 dB
PL23    -1.00 dB
PL24    -1.00 dB
PL25    -1.00 dB
PL26    -1.00 dB
PL27    -1.00 dB
PL28    -1.00 dB
PL29    -1.00 dB
PL30    -1.00 dB

===== CHANNEL f2 =====
CPDPRG2 waltz16
P2      100.00 usec
PL2     1.50 dB
PL3     1.50 dB
PL4     1.50 dB
PL5     1.50 dB
PL6     1.50 dB
PL7     1.50 dB
PL8     1.50 dB
PL9     1.50 dB
PL10    1.50 dB
PL11    1.50 dB
PL12    1.50 dB
PL13    1.50 dB
PL14    1.50 dB
PL15    1.50 dB
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f3 =====
CPDPRG3 waltz16
P3      100.00 usec
PL3     1.50 dB
PL4     1.50 dB
PL5     1.50 dB
PL6     1.50 dB
PL7     1.50 dB
PL8     1.50 dB
PL9     1.50 dB
PL10    1.50 dB
PL11    1.50 dB
PL12    1.50 dB
PL13    1.50 dB
PL14    1.50 dB
PL15    1.50 dB
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f4 =====
CPDPRG4 waltz16
P4      100.00 usec
PL4     1.50 dB
PL5     1.50 dB
PL6     1.50 dB
PL7     1.50 dB
PL8     1.50 dB
PL9     1.50 dB
PL10    1.50 dB
PL11    1.50 dB
PL12    1.50 dB
PL13    1.50 dB
PL14    1.50 dB
PL15    1.50 dB
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f5 =====
CPDPRG5 waltz16
P5      100.00 usec
PL5     1.50 dB
PL6     1.50 dB
PL7     1.50 dB
PL8     1.50 dB
PL9     1.50 dB
PL10    1.50 dB
PL11    1.50 dB
PL12    1.50 dB
PL13    1.50 dB
PL14    1.50 dB
PL15    1.50 dB
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f6 =====
CPDPRG6 waltz16
P6      100.00 usec
PL6     1.50 dB
PL7     1.50 dB
PL8     1.50 dB
PL9     1.50 dB
PL10    1.50 dB
PL11    1.50 dB
PL12    1.50 dB
PL13    1.50 dB
PL14    1.50 dB
PL15    1.50 dB
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f7 =====
CPDPRG7 waltz16
P7      100.00 usec
PL7     1.50 dB
PL8     1.50 dB
PL9     1.50 dB
PL10    1.50 dB
PL11    1.50 dB
PL12    1.50 dB
PL13    1.50 dB
PL14    1.50 dB
PL15    1.50 dB
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f8 =====
CPDPRG8 waltz16
P8      100.00 usec
PL8     1.50 dB
PL9     1.50 dB
PL10    1.50 dB
PL11    1.50 dB
PL12    1.50 dB
PL13    1.50 dB
PL14    1.50 dB
PL15    1.50 dB
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f9 =====
CPDPRG9 waltz16
P9      100.00 usec
PL9     1.50 dB
PL10    1.50 dB
PL11    1.50 dB
PL12    1.50 dB
PL13    1.50 dB
PL14    1.50 dB
PL15    1.50 dB
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f10 =====
CPDPRG10 waltz16
P10     100.00 usec
PL10    1.50 dB
PL11    1.50 dB
PL12    1.50 dB
PL13    1.50 dB
PL14    1.50 dB
PL15    1.50 dB
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f11 =====
CPDPRG11 waltz16
P11     100.00 usec
PL11    1.50 dB
PL12    1.50 dB
PL13    1.50 dB
PL14    1.50 dB
PL15    1.50 dB
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f12 =====
CPDPRG12 waltz16
P12     100.00 usec
PL12    1.50 dB
PL13    1.50 dB
PL14    1.50 dB
PL15    1.50 dB
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f13 =====
CPDPRG13 waltz16
P13     100.00 usec
PL13    1.50 dB
PL14    1.50 dB
PL15    1.50 dB
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f14 =====
CPDPRG14 waltz16
P14     100.00 usec
PL14    1.50 dB
PL15    1.50 dB
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f15 =====
CPDPRG15 waltz16
P15     100.00 usec
PL15    1.50 dB
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f16 =====
CPDPRG16 waltz16
P16     100.00 usec
PL16    1.50 dB
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f17 =====
CPDPRG17 waltz16
P17     100.00 usec
PL17    1.50 dB
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f18 =====
CPDPRG18 waltz16
P18     100.00 usec
PL18    1.50 dB
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f19 =====
CPDPRG19 waltz16
P19     100.00 usec
PL19    1.50 dB
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f20 =====
CPDPRG20 waltz16
P20     100.00 usec
PL20    1.50 dB
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f21 =====
CPDPRG21 waltz16
P21     100.00 usec
PL21    1.50 dB
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f22 =====
CPDPRG22 waltz16
P22     100.00 usec
PL22    1.50 dB
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f23 =====
CPDPRG23 waltz16
P23     100.00 usec
PL23    1.50 dB
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f24 =====
CPDPRG24 waltz16
P24     100.00 usec
PL24    1.50 dB
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f25 =====
CPDPRG25 waltz16
P25     100.00 usec
PL25    1.50 dB
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f26 =====
CPDPRG26 waltz16
P26     100.00 usec
PL26    1.50 dB
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

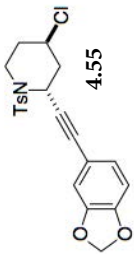
===== CHANNEL f27 =====
CPDPRG27 waltz16
P27     100.00 usec
PL27    1.50 dB
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f28 =====
CPDPRG28 waltz16
P28     100.00 usec
PL28    1.50 dB
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f29 =====
CPDPRG29 waltz16
P29     100.00 usec
PL29    1.50 dB
PL30    1.50 dB

===== CHANNEL f30 =====
CPDPRG30 waltz16
P30     100.00 usec
PL30    1.50 dB

===== CHANNEL f31 =====
CPDPRG31 waltz16
P31     100.00 usec
PL31    1.50 dB
=====
    
```



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```

Current Data Parameters
USER      Khevit11
NAME      KKH-III-191-SH-2
EXPNO     8
PROCNO    1

F2 - Acquisition Parameters
Date_     20200714
Time      10.42
INSTRUM   cryso00
PROBHD    5 mm CQNP131
PULPROG   zgpg30
TD        2048
SOLVENT   CDCl3
NS         1
DS         16
AQ         8013.85 Hz
FIDRES    3.912510 Hz
RG         0.1278452 sec
DM         62.400 usec
DE         298.0 K
TE         298.0 K
d0         0.00003300 sec
d1         1.00000000 sec
d13       0.00003300 sec
d16       0.00020000 sec
d10       0.00012480 sec

===== CHANNEL f1 =====
NUC1      1H
P1         7.50 usec
PC         0.00 usec
SFO1      500.225015 MHz

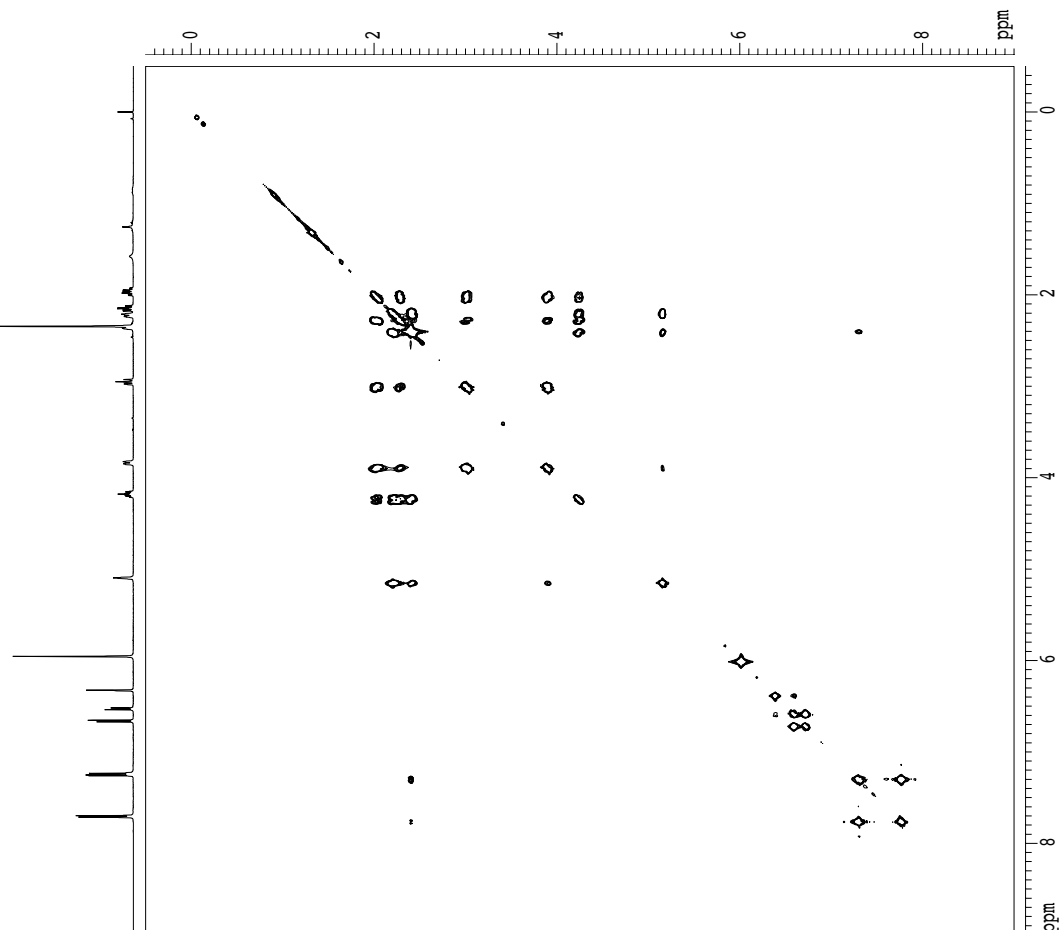
===== GRADIENT CHANNEL =====
GPRM1     sine,1.00
GPRM2     sine,1.00
GPRM3     sine,1.00
GPZ1      0.00 %
GPZ2      0.00 %
GPZ3      0.00 %
GPZ4      0.00 %
GPZ5      17.00 %
GPZ6      17.00 %
P16       1000.00 usec

F1 - Acquisition parameters
NUC1      1H
P1         5.12 usec
PC         0.00 usec
SFO1      500.225015 MHz
FIDRES    15.650040 Hz
SN        16.018 ppm
PnMODE    GP

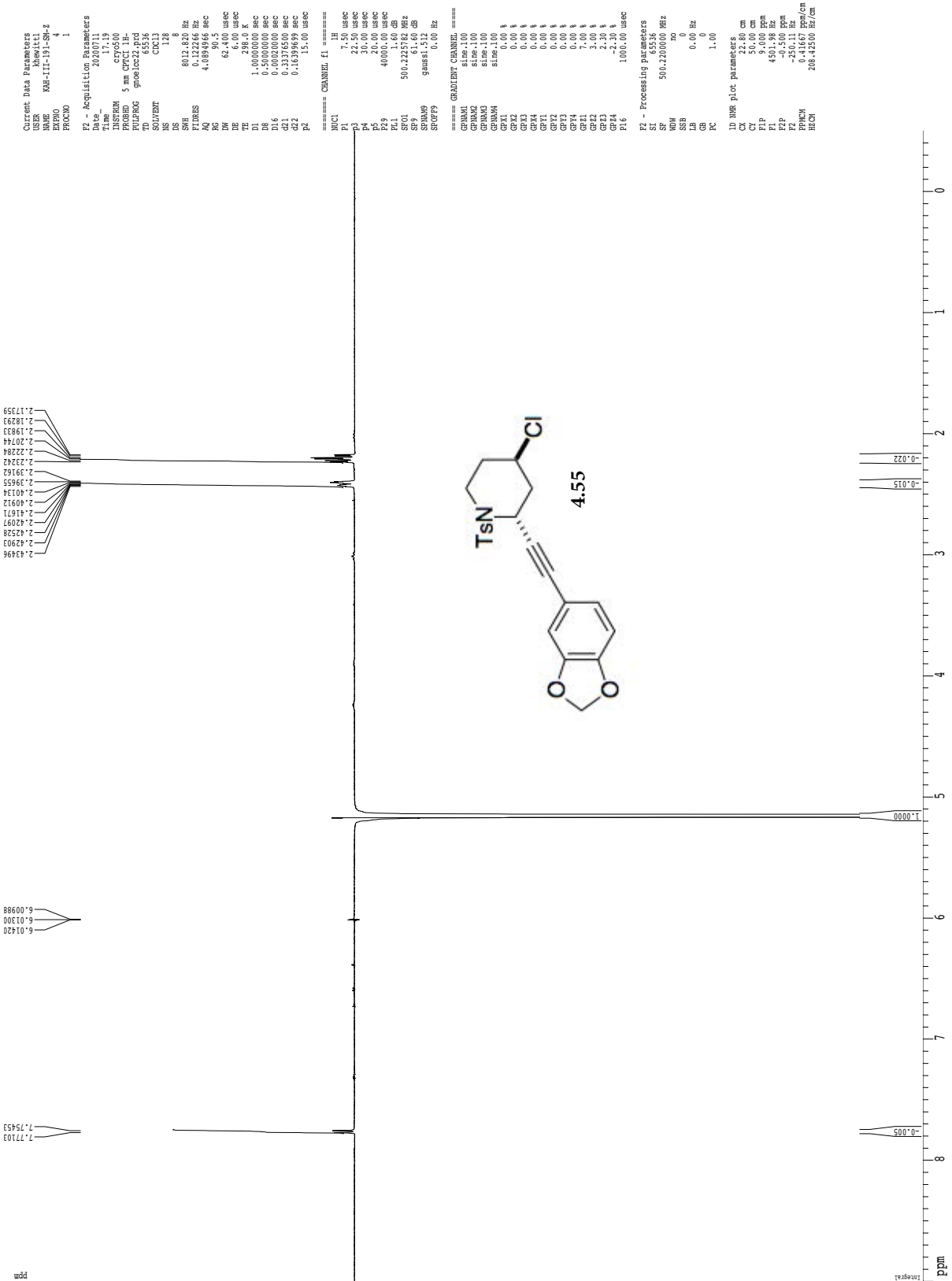
F2 - Processing parameters
SI         1024
SF         500.2200000 MHz
WDW        EM
SSB        0
GB         0.00 Hz
PC         1.00

F1 - Processing parameters
SI         1024
SF         500.2200000 MHz
WDW        EM
SSB        0
GB         0.00 Hz
PC         1.00

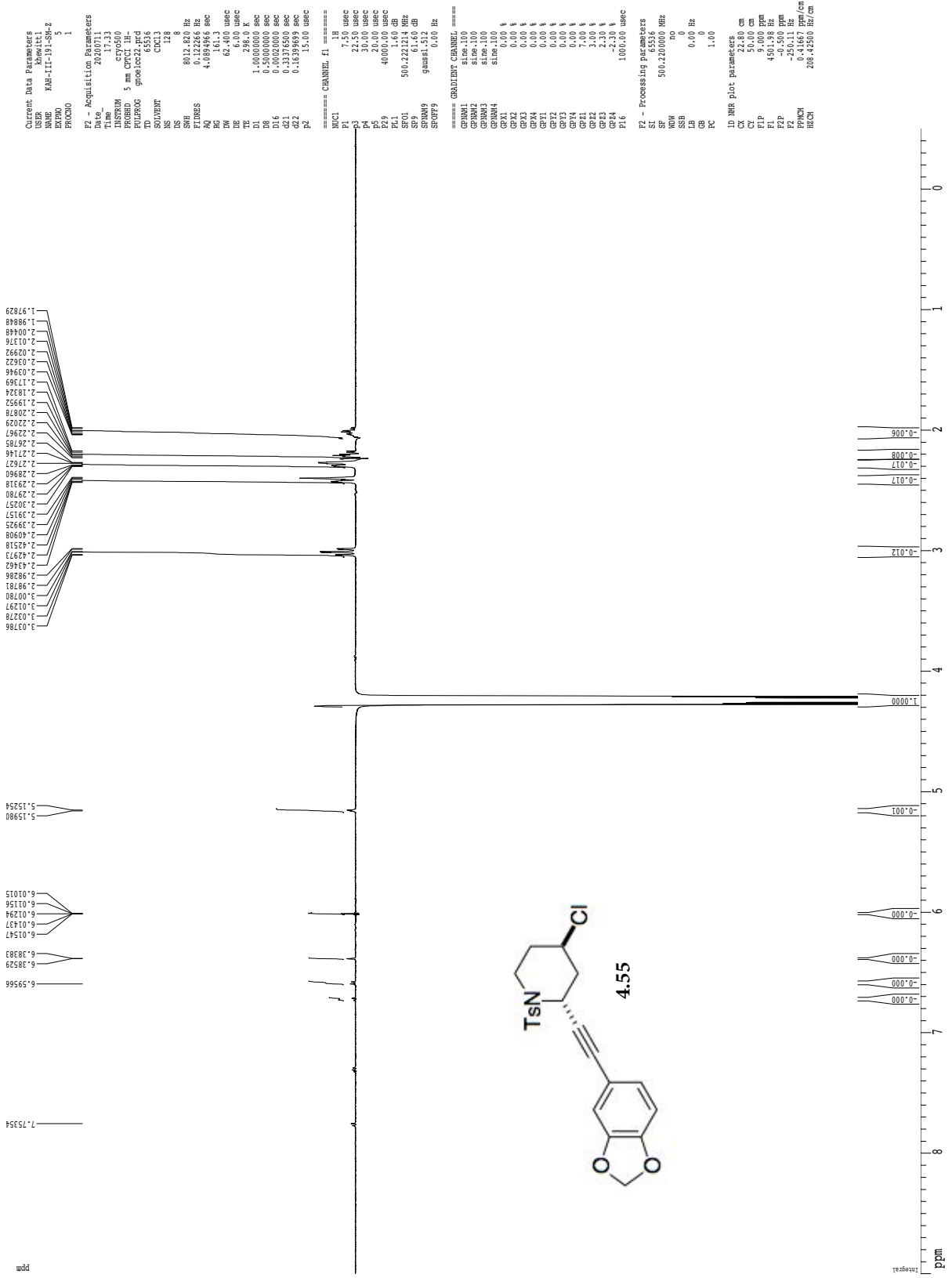
2D NMR plot parameters
CX1        15.00 cm
F2PL0     15.00 cm
F2PL1     45.00 ppm
F2PL2     45.00 Hz
F2PL3     0.5500 ppm
F2H1      -250.11 Hz
F1PL0     5.000 ppm
F1PL1     4501.98 Hz
F1PL2     0.500 ppm
F1PL3     -250.11 Hz
F1PR0CM   0.63333 ppm/cm
F1PR1CM   316.80600 Hz/cm
F1PR2CM   0.63333 ppm/cm
F1PR3CM   316.80600 Hz/cm
  
```



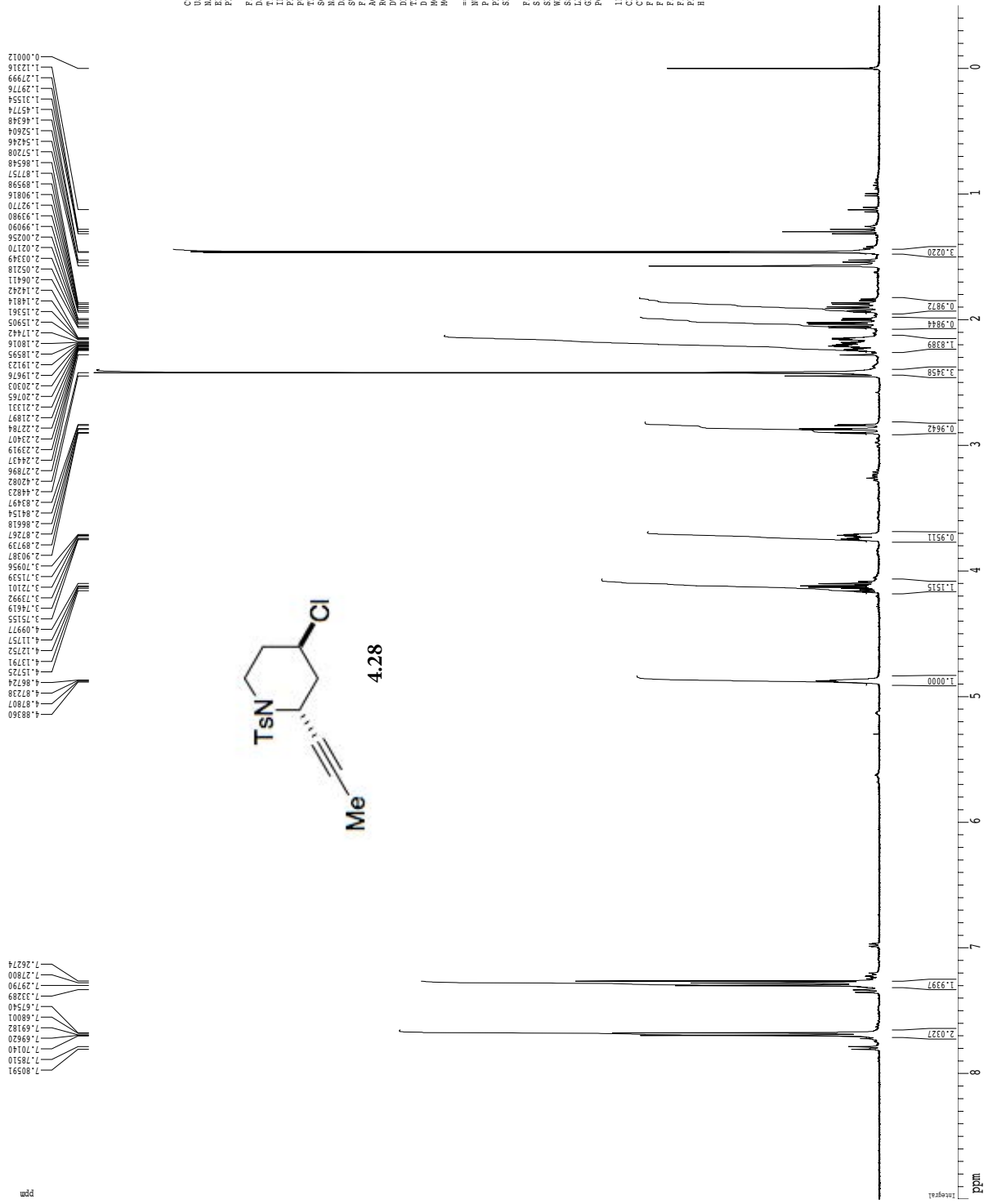
gnoe



gnoe



1H spectrum



Current Data Parameters  
 USER: Kowalski  
 NAME: KSM-17-03-2  
 EXPNO: 1  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20201104  
 Time: 16:07  
 PROBHD: 5 mm QNP 1H/1  
 PULPROG: zgpg30  
 TD: 65536  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 6410.258 Hz  
 FIDRES: 0.09781 Hz  
 AQ: 5.1118579 sec  
 RG: 409.6  
 DW: 78.000 usec  
 DE: 4.50 usec  
 TE: 298.2 K  
 T1: 0.16000000 sec  
 T2: 0.00000000 sec  
 T3: 0.00000000 sec  
 MCHRG1: 0.01500000 sec  
 MCHRG2: 0.01500000 sec

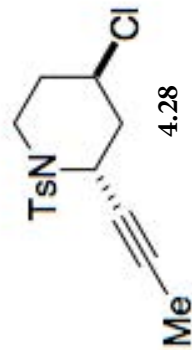
===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -1.10 dB  
 SFO1: 400.1328009 MHz

F2 - Processing Parameters  
 SI: 65536  
 SF: 400.1300204 MHz  
 WDM: no  
 LB: 0.00 Hz  
 GB: 0  
 PC: 2.00

1D NMR File Parameters  
 CY: 22.80 cm  
 C1: 15.00 cm  
 F1P: 9.000 ppm  
 F1: 360.110 Hz  
 F2: -200.000 Hz  
 PPRM1: 0.41667 ppm/cm  
 HZCM: 166.72086 Hz/cm



13C spectrum with 1H decoupling



Current Data Parameters  
 USER Kheiwittl  
 NAME KAH-IV-030-Z  
 EXPNO 8  
 PROCNO 1

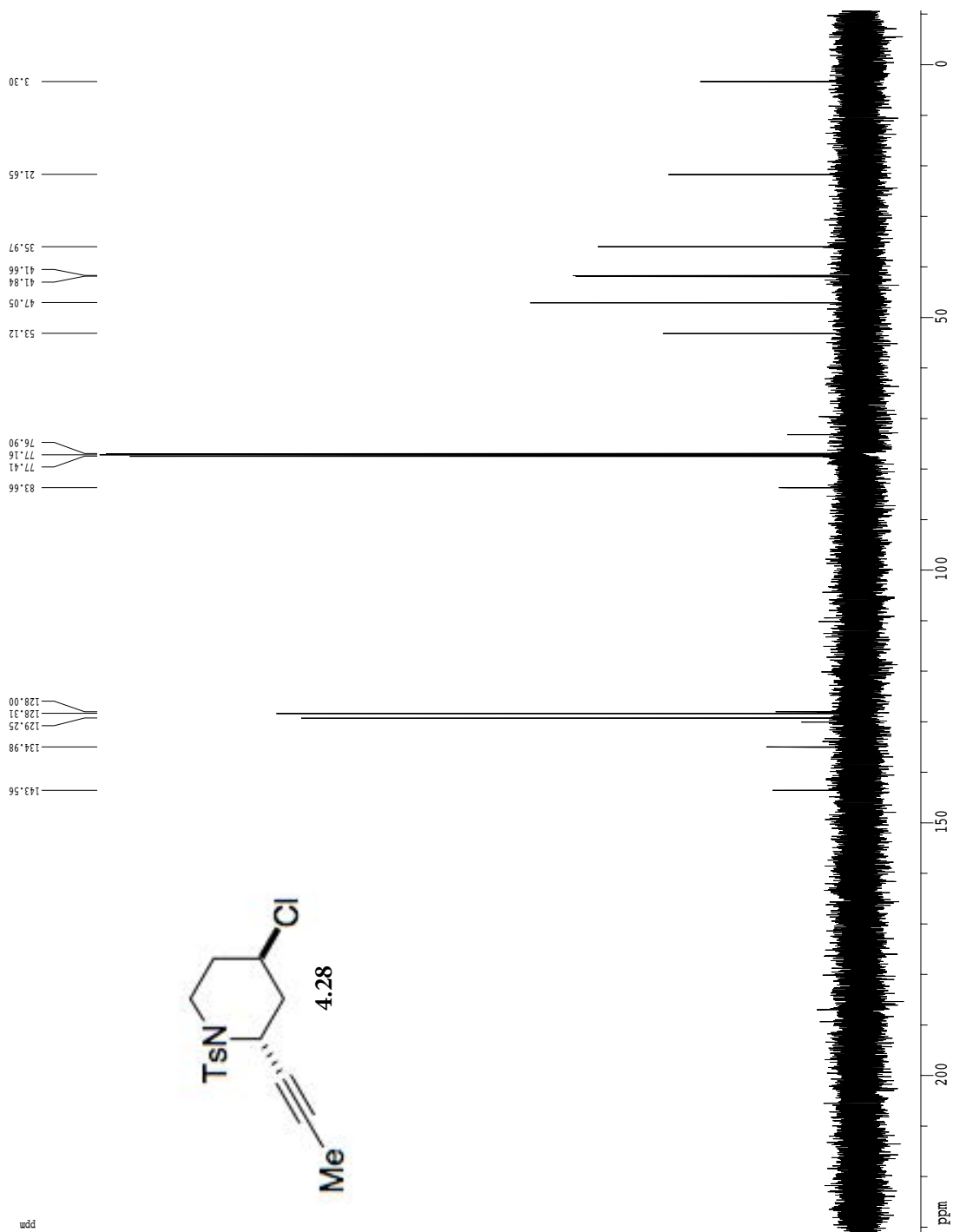
F2 - Acquisition Parameters  
 Date\_ 20201104  
 Time\_ 19.16  
 INSTRUM gms00  
 PROBD 5 mm broadband  
 PULPROG zgdc30  
 TD 65536  
 SOLVENT CDC13  
 NS 224  
 DS 4  
 SWH 30303.031 Hz  
 FIDRES 0.462388 Hz  
 AQ 1.0813940 sec  
 RG 3649.1  
 DW 16.500 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.25000000 sec  
 d11 0.03000000 sec  
 MCREST 0.00000000 sec  
 MCWRK 0.01500000 sec

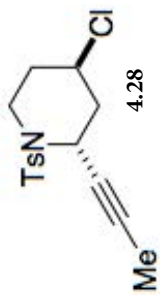
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 14.20 usec  
 PL1 -6.00 dB  
 SF01 125.4245824 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 -6.00 dB  
 PL12 12.30 dB  
 SF02 498.7524937 MHz

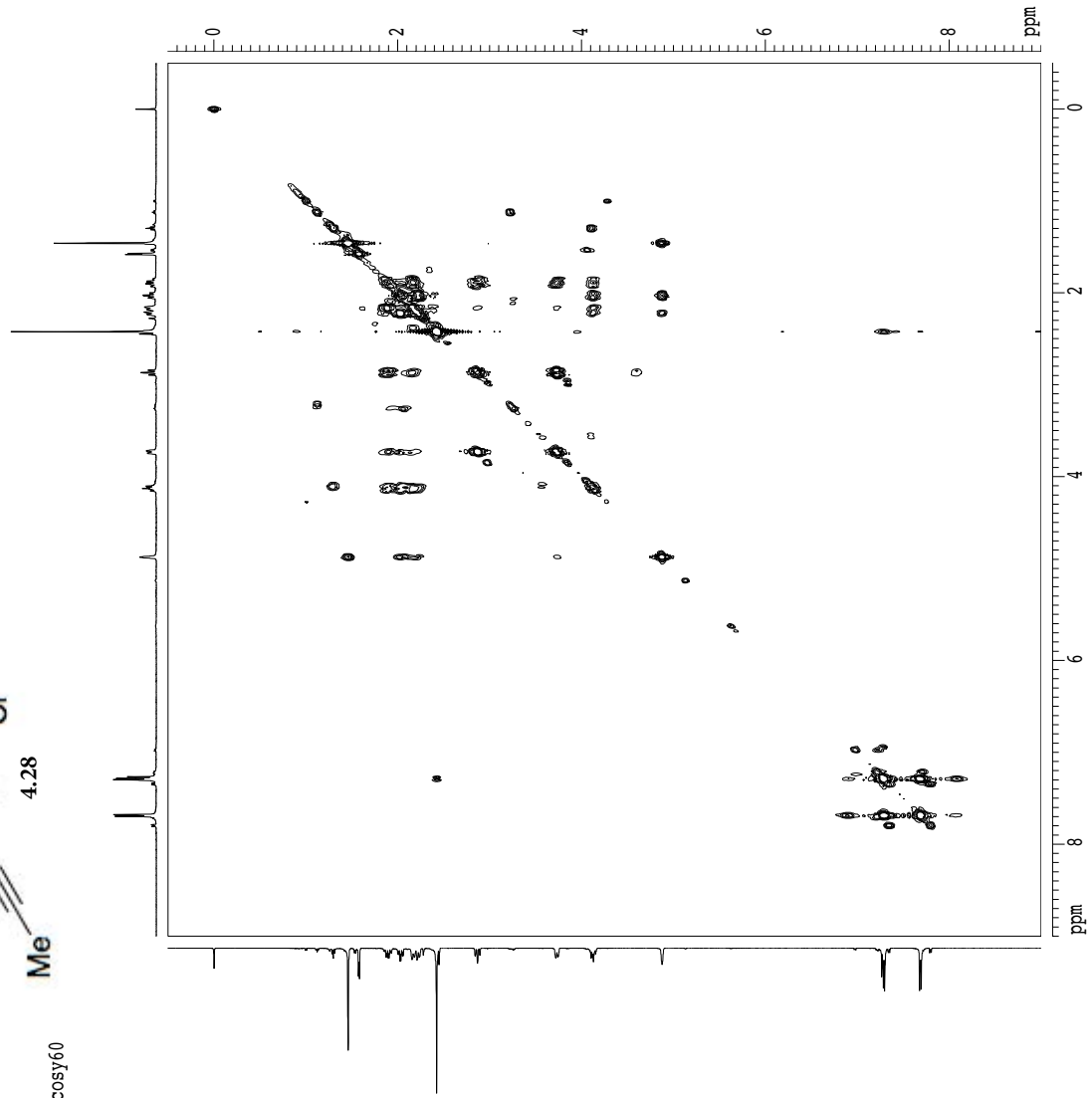
F2 - Processing parameters  
 SI 65536  
 SF 125.4107757 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 2.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 12.50 cm  
 FIP 230.907 ppm  
 F1 28958.25 Hz  
 F2P -10.723 ppm  
 F2 -1344.78 Hz  
 PPMCM 12.08151 ppm/cm  
 HZCM 1515.15149 Hz/cm





gcosy60



```

Current Data Parameters
USER          Newitt1
EXPNO        1
PROCNO       1
=====
F2 - Acquisition Parameters
Date_        20201104
Time         19.05
PROBHD       5 mm broadband
PULPROG      zgpg30
SOLVENT      CDCl3
NS           2048
DS           16
SWH          6032.16 Hz
FIDRES       0.127845 Hz
AQ           0.127845 sec
RG           5792.6
DE           6.00 usec
TE           300.2 K
=====
D1           1.0000000 sec
d13          0.4000000 sec
D16          0.1002000 sec
TD           0.10012490 sec
=====
===== CHANNEL f1 =====
NUC1         13C
P1           12.00 usec
PL1          -6.00 dB
SFO1         498.751913 MHz
===== GRADIENT CHANNEL =====
GPM01        SINE.100
GPM02        SINE.100
GPR2         0.00 V
GPT1         0.00 V
GPT2         17.00 V
GPT3         17.00 V
GPT4         17.00 V
GPT5         17.00 V
GPT6         17.00 V
P16          1000.00 usec
=====
===== F1 - Acquisition parameters =====
RG0          1
RG1          357
SFO1         498.751913 MHz
FIDRES       31.300079 Hz
SI           16.000
WDW          EM
SSB          0
GB           0
PC           1.00
=====
F2 - Processing parameters
SI           1024
WDW          EM
SSB          0
GB           0
PC           1.00
=====
F1 - Processing parameters
SI           1024
WDW          EM
SSB          0
GB           0
PC           1.00
=====
2D NMR Plot Parameters
CX1          15.00 cm
CX2          15.00 cm
FZ1Z0        9.000 ppm
FZ1Z1        9.000 ppm
FZ1Z2        -4.500 ppm
FZ1Z3        -4.500 ppm
FZ1Z4        -4.500 ppm
FZ1Z5        -4.500 ppm
FZ1Z6        -4.500 ppm
FZ1Z7        -4.500 ppm
FZ1Z8        -4.500 ppm
FZ1Z9        -4.500 ppm
FZ1Z10       -4.500 ppm
FZ1Z11       -4.500 ppm
FZ1Z12       -4.500 ppm
FZ1Z13       -4.500 ppm
FZ1Z14       -4.500 ppm
FZ1Z15       -4.500 ppm
FZ1Z16       -4.500 ppm
FZ1Z17       -4.500 ppm
FZ1Z18       -4.500 ppm
FZ1Z19       -4.500 ppm
FZ1Z20       -4.500 ppm
FZ1Z21       -4.500 ppm
FZ1Z22       -4.500 ppm
FZ1Z23       -4.500 ppm
FZ1Z24       -4.500 ppm
FZ1Z25       -4.500 ppm
FZ1Z26       -4.500 ppm
FZ1Z27       -4.500 ppm
FZ1Z28       -4.500 ppm
FZ1Z29       -4.500 ppm
FZ1Z30       -4.500 ppm
FZ1Z31       -4.500 ppm
FZ1Z32       -4.500 ppm
FZ1Z33       -4.500 ppm
FZ1Z34       -4.500 ppm
FZ1Z35       -4.500 ppm
FZ1Z36       -4.500 ppm
FZ1Z37       -4.500 ppm
FZ1Z38       -4.500 ppm
FZ1Z39       -4.500 ppm
FZ1Z40       -4.500 ppm
FZ1Z41       -4.500 ppm
FZ1Z42       -4.500 ppm
FZ1Z43       -4.500 ppm
FZ1Z44       -4.500 ppm
FZ1Z45       -4.500 ppm
FZ1Z46       -4.500 ppm
FZ1Z47       -4.500 ppm
FZ1Z48       -4.500 ppm
FZ1Z49       -4.500 ppm
FZ1Z50       -4.500 ppm
FZ1Z51       -4.500 ppm
FZ1Z52       -4.500 ppm
FZ1Z53       -4.500 ppm
FZ1Z54       -4.500 ppm
FZ1Z55       -4.500 ppm
FZ1Z56       -4.500 ppm
FZ1Z57       -4.500 ppm
FZ1Z58       -4.500 ppm
FZ1Z59       -4.500 ppm
FZ1Z60       -4.500 ppm
FZ1Z61       -4.500 ppm
FZ1Z62       -4.500 ppm
FZ1Z63       -4.500 ppm
FZ1Z64       -4.500 ppm
FZ1Z65       -4.500 ppm
FZ1Z66       -4.500 ppm
FZ1Z67       -4.500 ppm
FZ1Z68       -4.500 ppm
FZ1Z69       -4.500 ppm
FZ1Z70       -4.500 ppm
FZ1Z71       -4.500 ppm
FZ1Z72       -4.500 ppm
FZ1Z73       -4.500 ppm
FZ1Z74       -4.500 ppm
FZ1Z75       -4.500 ppm
FZ1Z76       -4.500 ppm
FZ1Z77       -4.500 ppm
FZ1Z78       -4.500 ppm
FZ1Z79       -4.500 ppm
FZ1Z80       -4.500 ppm
FZ1Z81       -4.500 ppm
FZ1Z82       -4.500 ppm
FZ1Z83       -4.500 ppm
FZ1Z84       -4.500 ppm
FZ1Z85       -4.500 ppm
FZ1Z86       -4.500 ppm
FZ1Z87       -4.500 ppm
FZ1Z88       -4.500 ppm
FZ1Z89       -4.500 ppm
FZ1Z90       -4.500 ppm
FZ1Z91       -4.500 ppm
FZ1Z92       -4.500 ppm
FZ1Z93       -4.500 ppm
FZ1Z94       -4.500 ppm
FZ1Z95       -4.500 ppm
FZ1Z96       -4.500 ppm
FZ1Z97       -4.500 ppm
FZ1Z98       -4.500 ppm
FZ1Z99       -4.500 ppm
FZ1Z100      -4.500 ppm
=====

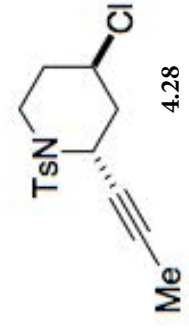
```

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ppm

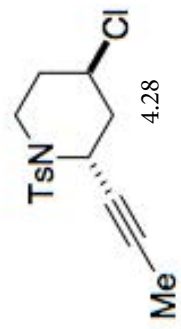
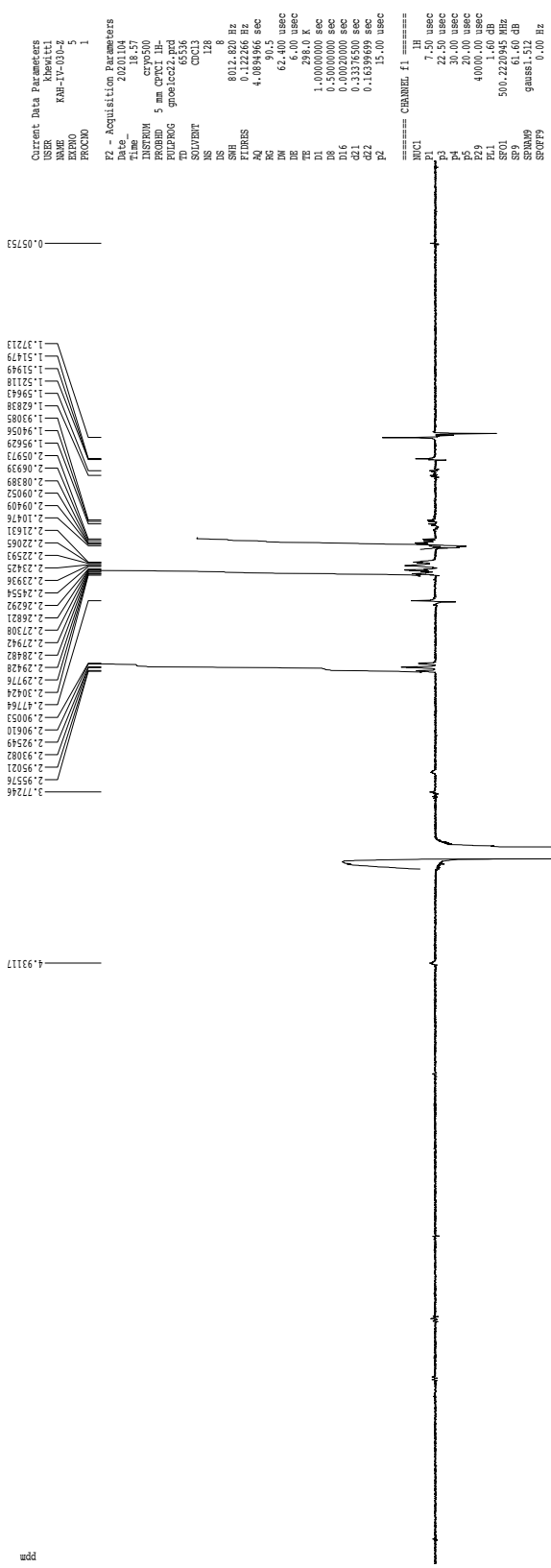
7.75409  
7.73757  
2.48474  
2.29889  
2.29508  
2.29136  
2.28649  
2.27995  
2.27451  
2.27053  
2.26888  
2.11765  
2.10841  
2.09304  
2.08381  
2.06893  
2.05876  
1.52882  
1.51880

```
Current Data Parameters
NAME          khawit1
EXPNO         4
PROCNO        1
F2 - Acquisition Parameters
Date_         20201104
Time          18.42
PROBHD0       5 mm CPCT 1H
PULPROG       zgpg30
TD             65536
SOLVENT       CDCl3
NS            128
DS            4
SWH           8012.820 Hz
FIDRES       0.122246 Hz
AQ           4.089496 sec
RG           62.000
DE           6.000 usec
TE           298.2 K
D1           1.0000000 sec
d11          0.1000000 sec
d12          0.1000000 sec
d13          0.3337500 sec
d14          0.3337500 sec
d2           0.1639699 sec
d22          15.00 usec
===== CHANNEL f1 =====
NUC1          1H
P1           7.50 usec
P2           22.50 usec
P3           21.00 usec
P4           21.00 usec
PR           40000.00 usec
PZ1          1.66 dB
PZ2          1.66 dB
SFO1         500.222688 MHz
SF           500.136400 MHz
WDW          gauss
SSB          0.00 Hz
STUFF9
===== GRABEDIT CHANNEL =====
GRAB10       sine.100
GRAB11       sine.100
GRAB12       sine.100
GRAB13       sine.100
GRAB14       sine.100
GRAB15       sine.100
GRAB16       sine.100
GRAB17       sine.100
GRAB18       sine.100
GRAB19       sine.100
GRAB20       sine.100
GRAB21       sine.100
GRAB22       sine.100
GRAB23       sine.100
GRAB24       sine.100
GRAB25       sine.100
GRAB26       sine.100
GRAB27       sine.100
GRAB28       sine.100
GRAB29       sine.100
GRAB30       sine.100
GRAB31       sine.100
GRAB32       sine.100
GRAB33       sine.100
GRAB34       sine.100
GRAB35       sine.100
GRAB36       sine.100
GRAB37       sine.100
GRAB38       sine.100
GRAB39       sine.100
GRAB40       sine.100
GRAB41       sine.100
GRAB42       sine.100
GRAB43       sine.100
GRAB44       sine.100
GRAB45       sine.100
GRAB46       sine.100
GRAB47       sine.100
GRAB48       sine.100
GRAB49       sine.100
GRAB50       sine.100
===== Processing parameters =====
SI           65536
SF           500.222688 MHz
WDW          0
SSB          0
LB           0.00 Hz
GB           0
PC           1.00
ID NMR Plot parameters
CX           22.88 cm
CY           50.00 cm
CZ           40.00 cm
F1          460.000 ppm
F2          -0.500 ppm
F3          -250.11 Hz
F4          0.41667 ppm/cm
F5          208.42500 Hz/cm
```

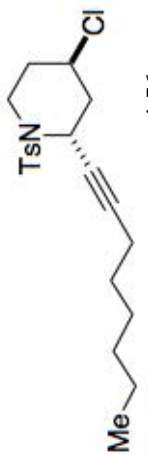
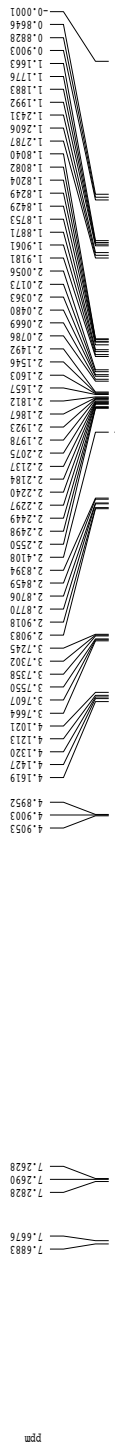


gnoe

ppm



# <sup>1</sup>H spectrum

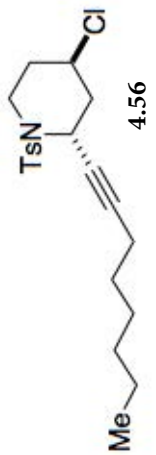
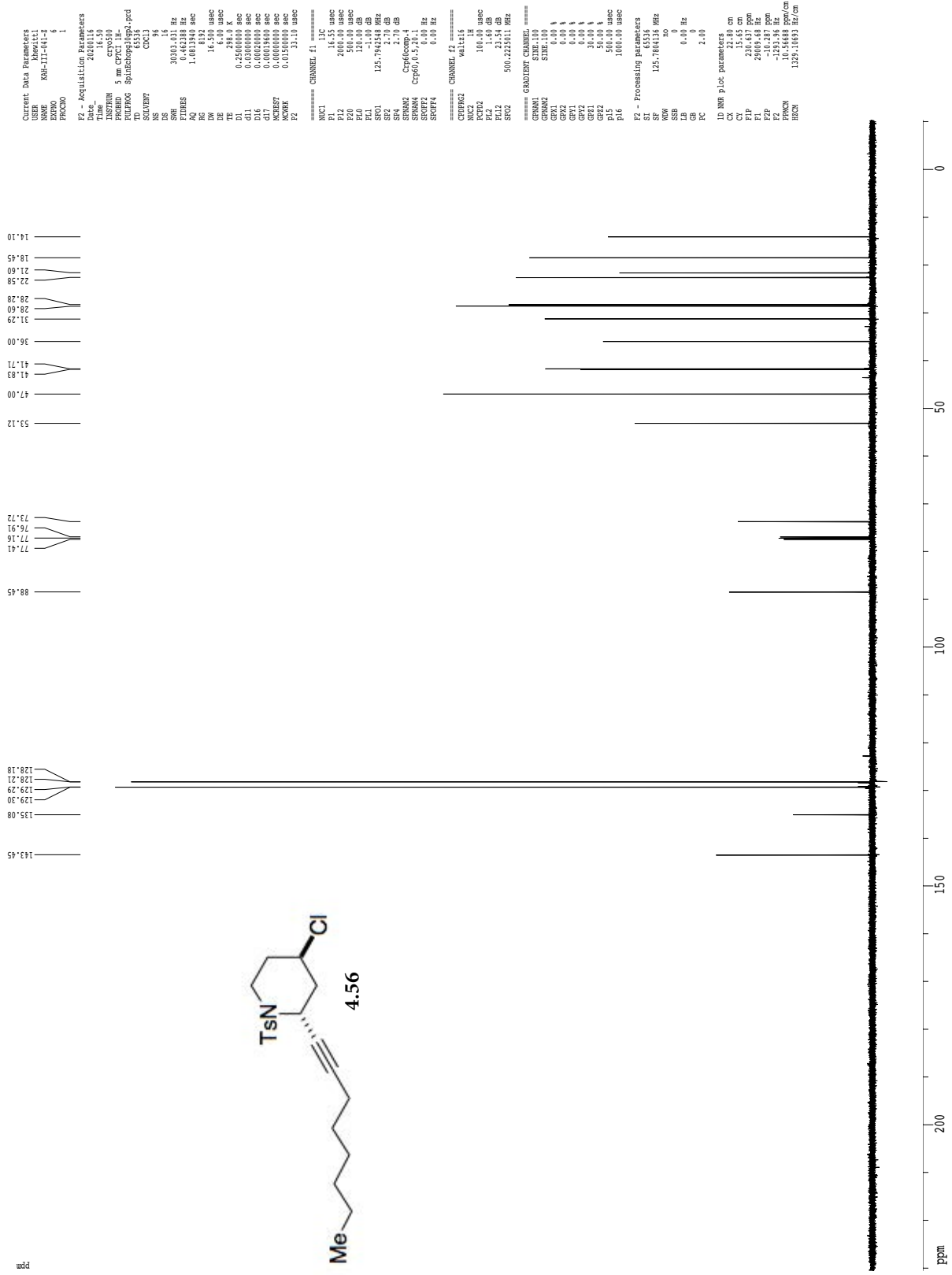


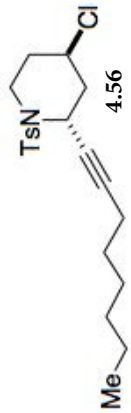
```

Current Data Parameters
=====
NAME           KMH-III-041-check
EXPNO          1
PROCNO        1
F2 - Acquisition Parameters
=====
Date_          20200115
Time           11:23
INSTRUM       spect
PROBHD        5 mm QNP H/P
PULPROG       zgpg30
TD            32768
SFO1          400.130
NUC1          13
ACQNT        4.00000000
DS            2
SWH           6410.256 Hz
FIDRES       0.10000000 Hz
AQ           2.5999776 sec
RG            71.8
AQ           78.000 usec
DE           4.50 usec
DI            0.10000000 sec
MCREST       0.00000000 sec
MCWPRG       0.01500000 sec
===== CHANNEL f1 =====
NUC1          1H
P1           12.00 usec
PL           0.00 dB
SFO1         400.130000 MHz
=====
F2 - Processing parameters
=====
SI           32768
SF           400.130000 MHz
WDW          EM
SSB          0
LB           0.00 Hz
GB           0
PC           2.00

ID MR plot parameters
=====
CX           12.00 cm
CY           12.00 cm
FIDP         9.000 ppm
F1           3601.17 Hz
F2           -0.500 ppm
F3           -0.500 ppm
PCMCN       0.41666 ppm/cm
PCMCX       166.72084 Hz/cm
  
```

Z-restored spin-echo 13C spectrum with 1H decoupling

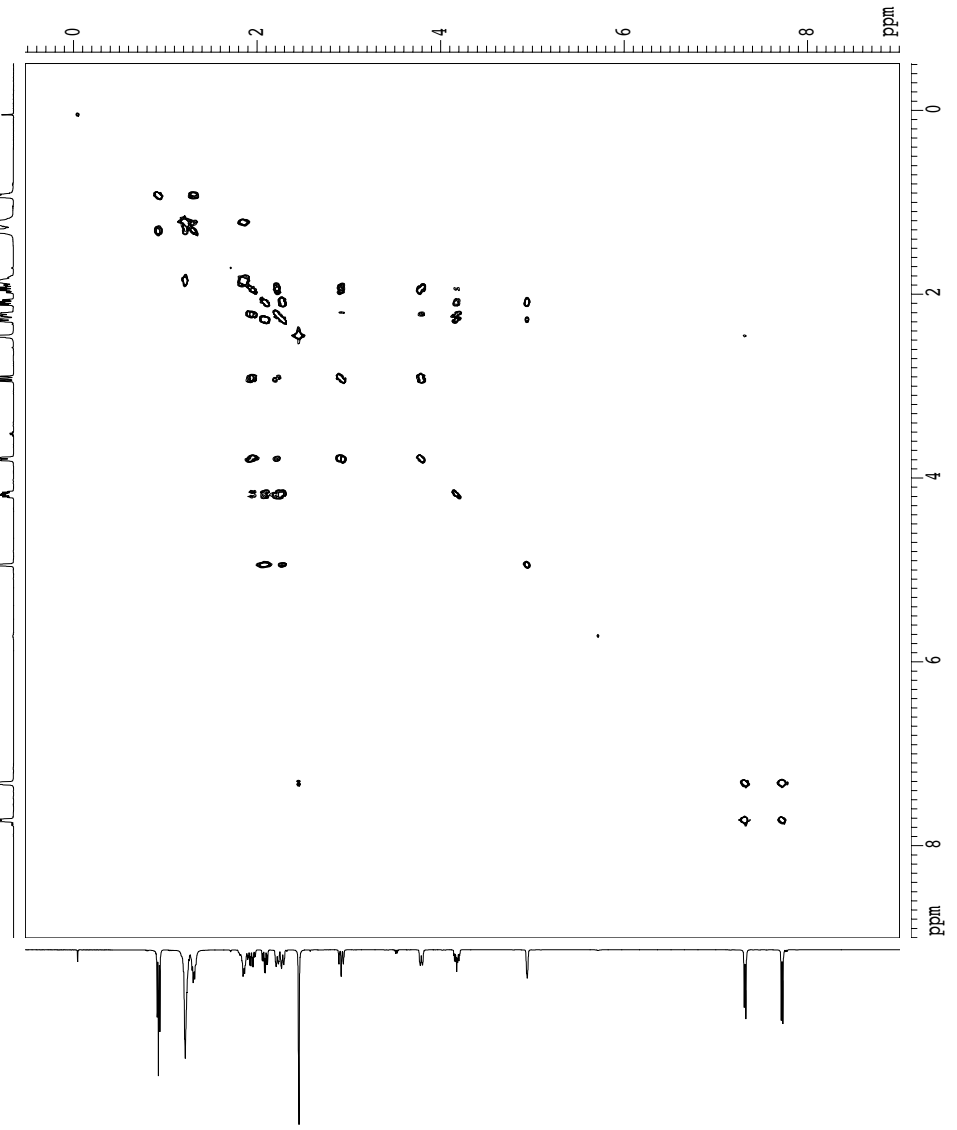




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```

Current Data Parameters
USR      Khevit1
EXPNO    2
PROCNO   1
=====
F2 - Acquisition Parameters
Date_     2000115
Time      16:06
INSTRUM   spect
PROBHD    5 mm CPTCI 1H-
PULPROG   cosygp60.prd
TD         2048
SOLVENT   CDCl3
NS         6
DS         2
SWH        8012.820 Hz
FIDRES     3.432510 Hz
AQ         0.1278432 sec
RG         62.400
DE         6.00 usec
TE         298.0 K
DO        0.0000300 sec
D1         1.0000000 sec
d11        0.0000000 sec
d12        0.0000000 sec
d16        0.0002000 sec
TD0        0.00012480 sec
===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
PL1        1.60 dB
SFO1       500.2235015 MHz
===== GRABBER CHANNEL =====
GPNMZ1     SINE 10.00
GPNMZ2     SINE 10.00
GPNMZ3     SINE 10.00
GPNMZ4     SINE 10.00
GPNMZ5     SINE 10.00
GPNMZ6     SINE 10.00
GPNMZ7     SINE 10.00
GPNMZ8     SINE 10.00
GPNMZ9     SINE 10.00
GPNMZ10    SINE 10.00
GPNMZ11    SINE 10.00
GPNMZ12    SINE 10.00
GPNMZ13    SINE 10.00
GPNMZ14    SINE 10.00
GPNMZ15    SINE 10.00
GPNMZ16    SINE 10.00
=====
F1 - Acquisition Parameters
NUC0       13C
P0         8.72 usec
PL0        1.90 dB
SFO0       101.253181 MHz
=====
F2 - Processing Parameters
SI         1024
SF         500.2200000 MHz
WDW        SINE
SSB        0
LB         0.0 Hz
GB         0.0
PC         1.00
=====
F1 - Processing Parameters
SI         1024
SF         500.2200000 MHz
WDW        SINE
SSB        0
LB         0.0 Hz
GB         0.0
=====
2D NMR plot parameters
CX2        15.00 cm
F2FLO      9.002 ppm
F2FID      9.002 ppm
F2F0F1     45.508 ppm
F2F0F2     50.508 ppm
F2F0F3     -254.47 Hz
F1FLO      9.002 ppm
F1FID      4503.14 Hz
F1F0F1     -10.224 ppm
F1F0F2     -10.224 ppm
F1F0F3     317.17146 Hz/cm
F1F0F4     0.63407 ppm/cm
F1F0F5     0.63311 ppm/cm
F1F0F6     317.69583 Hz/cm
  
```



gnoe

ppm

Current Data Parameters  
USER: KheWitt1  
NAME: KKH-11-041-2  
EXPNO: 3  
PROCNO: 1

F2 - Acquisition Parameters  
Date\_: 20200115  
Time: 17.58  
INSTRUM: zgpg30  
PROBHD: 5 mm CPCLP1H  
PULPROG: gnoeC22-pzd  
TD: 65536  
SOLVENT: CDCl3  
DS: 128  
SS: 8  
SFO: 8012.820 Hz  
FIDRES: 0.122266 Hz  
AQ: 4.089496 sec  
RG: 62.400 usec  
DE: 6.00 usec  
TE: 298.0 K  
D1: 1.0000000 sec  
D2: 0.0000000 sec  
D3: 0.0000000 sec  
d41: 0.33376500 sec  
d42: 0.16389699 sec  
P2: 15.00 usec

==== CHANNEL f1 =====  
NUC1: 1H  
P1: 7.50 usec  
P3: 22.50 usec  
P4: 20.00 usec  
P5: 20.00 usec  
P7: 40000.00 usec  
P11: 1.60 dB  
SFO1: 500.222477 MHz  
SFO2: 62.500000 MHz  
SFO3: 9.000000 MHz  
SFO4: 0.000000 MHz  
SFO5: 0.000000 MHz  
SFO6: 0.000000 MHz  
SFO7: 0.000000 MHz  
SFO8: 0.000000 MHz  
SFO9: 0.000000 MHz  
SFO10: 0.000000 MHz  
SFO11: 0.000000 MHz  
SFO12: 0.000000 MHz  
SFO13: 0.000000 MHz  
SFO14: 0.000000 MHz  
SFO15: 0.000000 MHz  
SFO16: 1000.00 usec

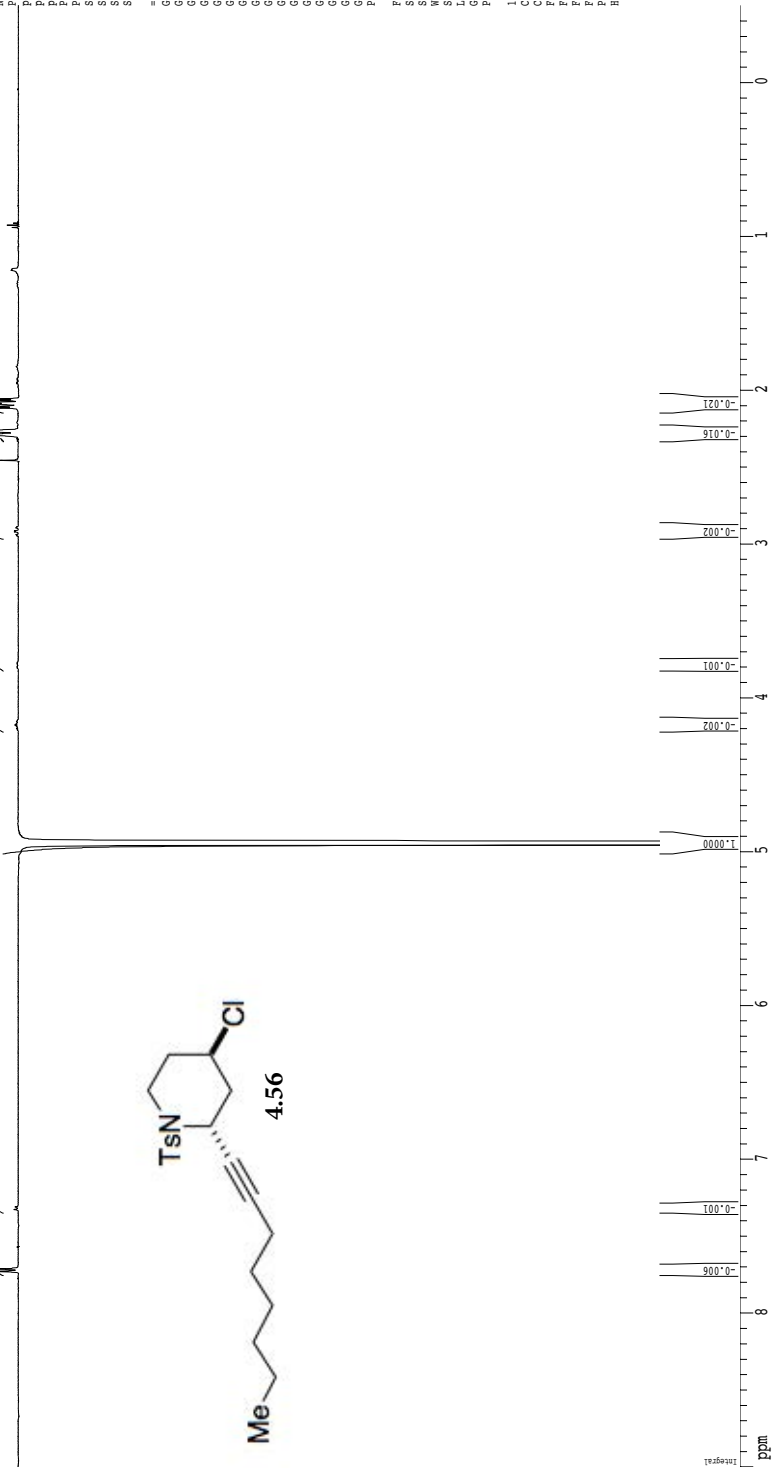
==== GRABBIT CHANNEL =====  
SFO1: 500.136000 MHz  
SFO2: 500.136000 MHz  
SFO3: 500.136000 MHz  
SFO4: 500.136000 MHz  
SFO5: 500.136000 MHz  
SFO6: 500.136000 MHz  
SFO7: 500.136000 MHz  
SFO8: 500.136000 MHz  
SFO9: 500.136000 MHz  
SFO10: 500.136000 MHz  
SFO11: 500.136000 MHz  
SFO12: 500.136000 MHz  
SFO13: 500.136000 MHz  
SFO14: 500.136000 MHz  
SFO15: 500.136000 MHz  
SFO16: 500.136000 MHz

F2 - Processing parameters  
SI: 65536  
SF: 500.2200000 MHz  
WDW: EM  
SSB: 0  
LB: 0.00 Hz  
GB: 0  
PC: 1.00

1D NMR plot parameters  
CX: 22.80 cm  
CY: 50.00 cm  
CZ: 50.00 cm  
IP: 1.00 mm  
F1: 450.00 MHz  
F2: -0.500 ppm  
F3: -250.11 Hz  
PROCN: 0+1667 hz/cm  
HZW: 208.42500 Hz/cm

0.9181  
0.9263  
0.9407  
1.2152  
1.2155  
1.2159  
2.0557  
2.0678  
2.0802  
2.0838  
2.1055  
2.1144  
2.2651  
2.2651  
2.2651  
2.2699  
2.2813  
2.2929  
2.4512

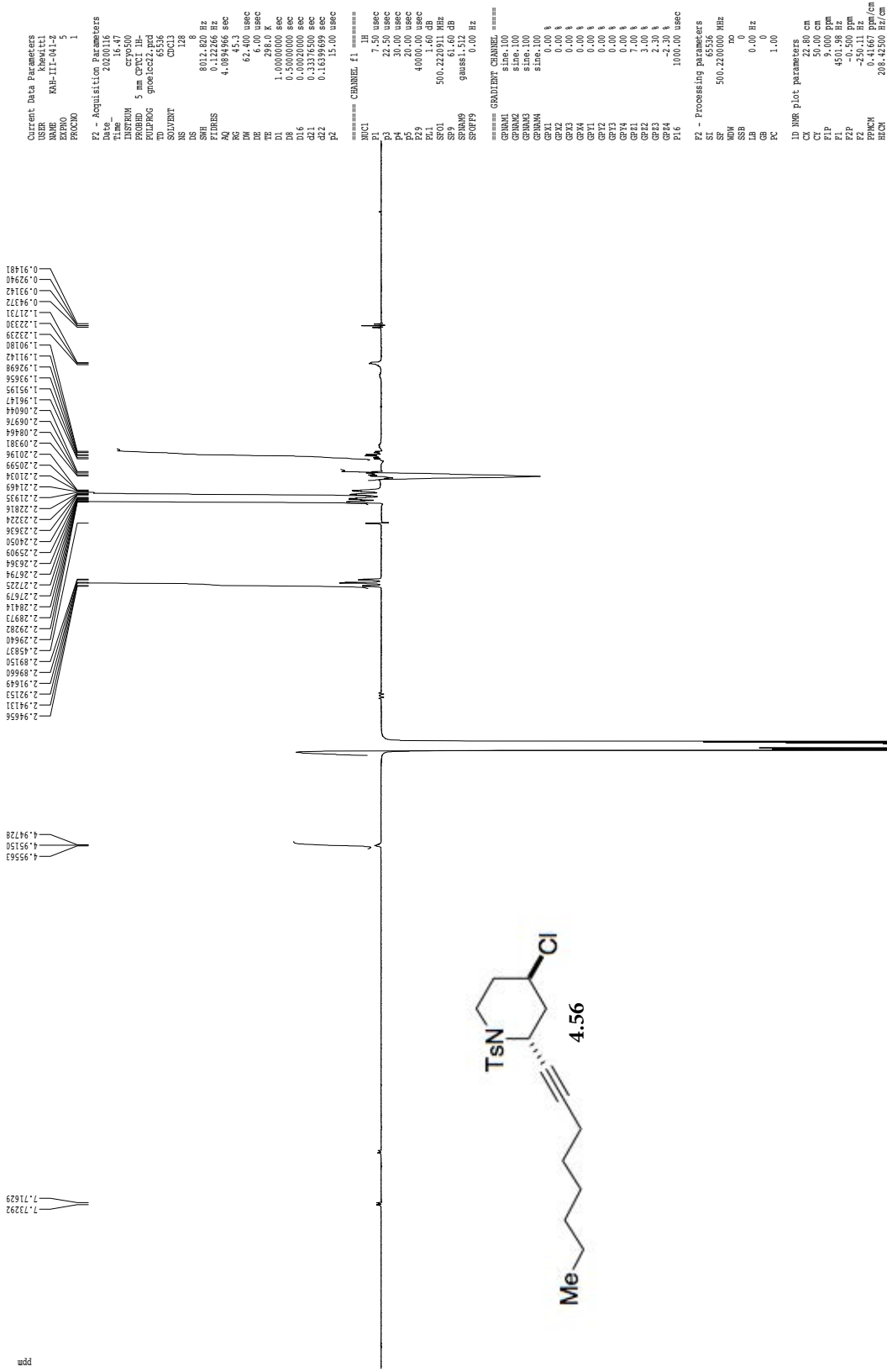
7.7258  
7.7125



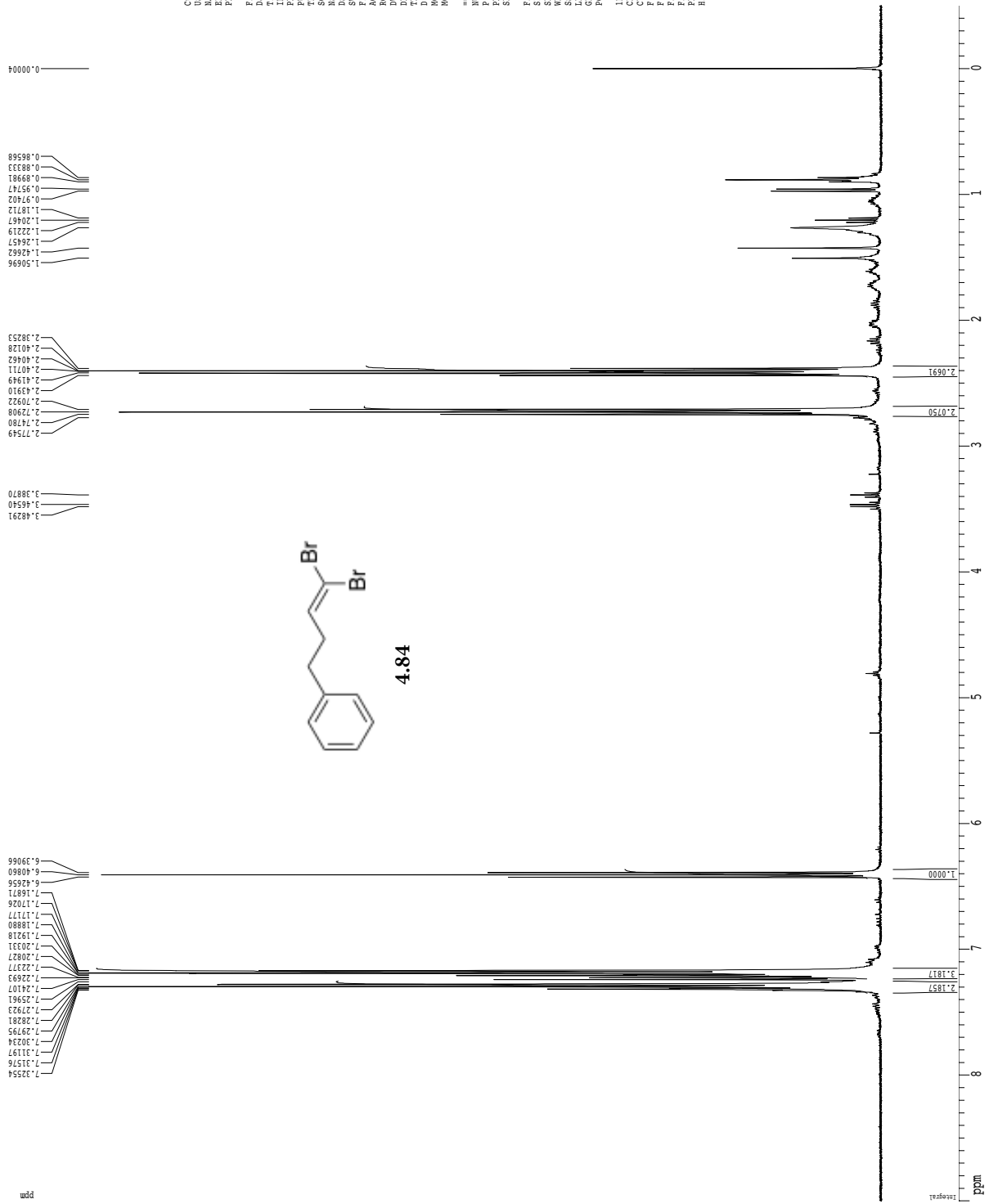


gnoe

ppm

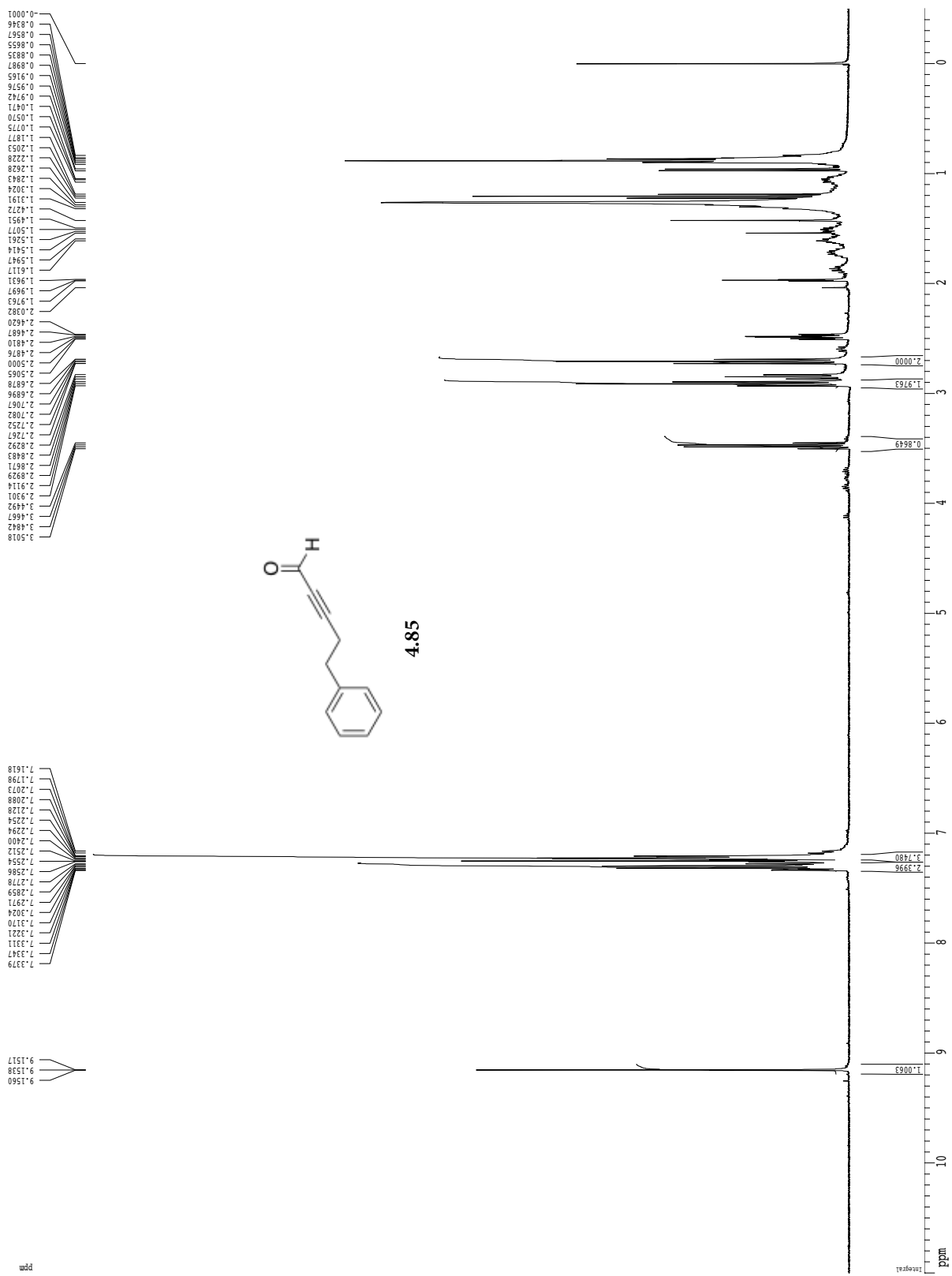


1H spectrum

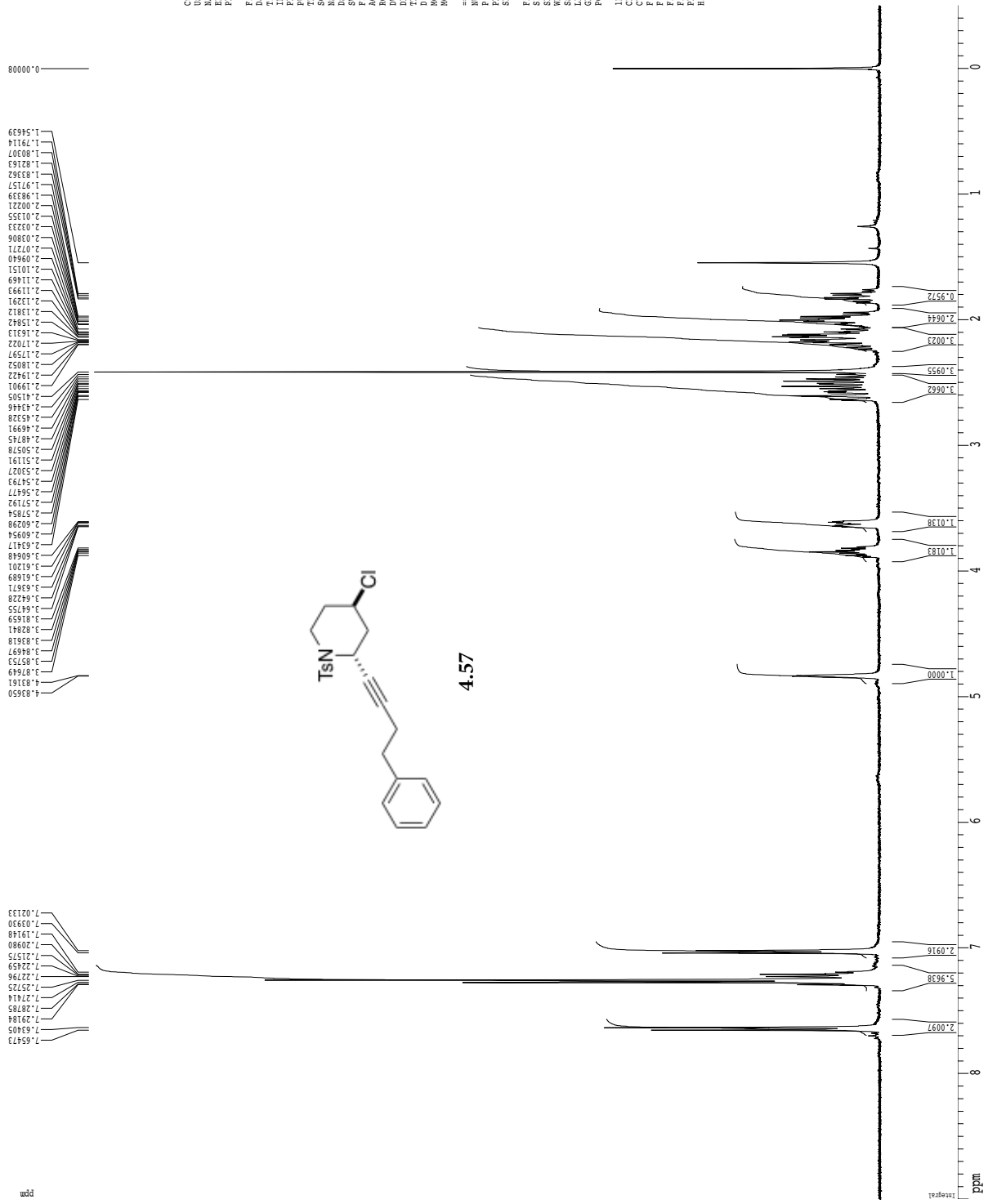


Current Data Parameters  
 USER TATSI100  
 NAME TATSI100  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20200805  
 Time 9:32  
 PROBNM 1000000  
 PULPROG 5 mm QNP H2/0  
 TD 65536  
 SOLVENT CDCl3  
 DS 9  
 SFO 6410.256 Hz  
 FIDRES 0.097813 Hz  
 AQ 5.118577 sec  
 RG 327.5  
 DQ 78.000 usec  
 DE 4.50 usec  
 TE 298.2 K  
 T1 0.1000000 sec  
 T1RHO 0.0000000 sec  
 T2 0.0000000 sec  
 T2RHO 0.0000000 sec  
 MORG 0.01500000 sec  
 ===== CHANNEL f1 =====  
 NUC1 13  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.1328009 MHz  
 F2 - Processing Parameters  
 SI 65536  
 SF 400.1300293 MHz  
 WDM no  
 LB 0.00 Hz  
 GB 0  
 PC 2.00  
 ID NMR P1.CF Parameters  
 C1 22.80 cm  
 C2 19.04 cm  
 F1P 9.000 ppm  
 F2 3600.17 Hz  
 F3 4000.800 Hz  
 F4 -2000.00 Hz  
 P1PCMH 0.41667 ppm/cm  
 P2PCMH 166.72086 Hz/cm

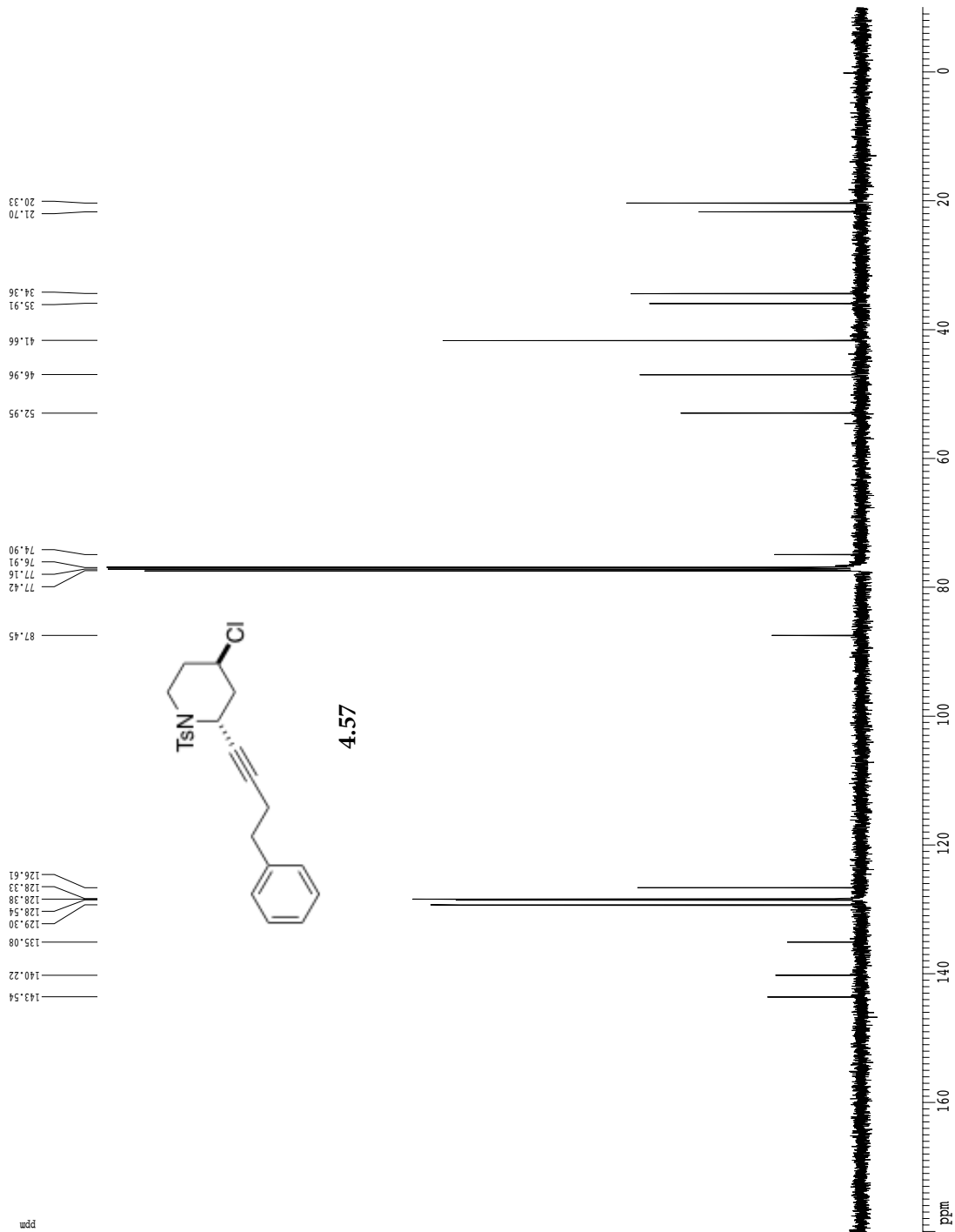
1H spectrum



1H spectrum



13C spectrum with 1H decoupling



Current Data Parameters  
 USER tthane  
 NAME TATV16carbon  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20200825  
 Time\_ 10.06  
 INSTRUM gm500  
 PROBDW 5 mm broadband  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3T  
 NS 568  
 DS 4  
 SWH 30303.031 Hz  
 FIDRES 0.462388 Hz  
 AQ 1.0813940 sec  
 RG 8192  
 DW 16.500 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.25000000 sec  
 d11 0.03000000 sec  
 MCREST 0.00000000 sec  
 MCWRRK 0.01500000 sec

==== CHANNEL f1 =====

NUC1 13C  
 P1 14.20 usec  
 PL1 -6.00 dB  
 SF01 125.4245824 MHz

==== CHANNEL f2 =====

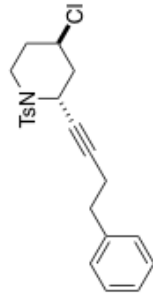
CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 -6.00 dB  
 PL12 12.30 dB  
 SF02 498.7524937 MHz

F2 - Processing parameters

SI 65536  
 SF 125.4107757 MHz  
 NDMW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 2.00

1D NMR plot parameters

CX 20.00 cm  
 CY 12.50 cm  
 FLP 180.000 ppm  
 F1 22573.94 Hz  
 F2P -10.000 ppm  
 F2 -1254.11 Hz  
 PPMCM 9.50000 ppm/cm  
 HZCM 1191.40234 Hz/cm



gcosy60

```

Current Data Parameters
=====
NAME      tthane
EXPNO1    1
PROCNO    1

F2 - Acquisition Parameters
=====
Date_     2001012
Time      17.0
INSTRUM   cpd450
PROBHD    5 mm CPCLP 1H-
PULPROG   cosyprg0.prd
TD        2848
SOLVENT   CDCl3
DS        2
SFO1      500.136260 MHz
SF        500.136260 MHz
AQ        0.1278452 sec
RG        640.000
DE        6.000 usec
TE        298.0 K
d0        0.0000000 sec
d1        1.0000000 sec
d11       0.0000000 sec
d12       0.0000000 sec
d16       0.0002000 sec
d18       0.0002000 sec
d19       0.0002000 sec
d30       0.0002480 sec

===== CHANNEL f1 =====
NUC1      13C
P1        1.50 usec
PL1       1.60 dB
SFO1      500.2235015 MHz

===== GRABBER CHANNEL =====
GNUM1     1
GPRG1     SINE
GPRZ1     180.000
GPA1      0.00 usec
GPA2      0.00 usec
GPT1      0.00 usec
GPT2      0.00 usec
GPT3      0.00 usec
GPT4      0.00 usec
GPT5      0.00 usec
GPT6      0.00 usec
GPT7      0.00 usec
GPT8      0.00 usec
GPT9      0.00 usec
GPT10     0.00 usec
GPT11     0.00 usec
GPT12     0.00 usec
GPT13     0.00 usec
GPT14     0.00 usec
GPT15     0.00 usec
GPT16     0.00 usec

===== CHANNEL f2 =====
NUC2      13C
P2        1.50 usec
PL2       1.60 dB
SFO2      500.2235015 MHz

===== GRABBER CHANNEL =====
GNUM2     1
GPRG2     SINE
GPRZ2     180.000
GPA2      0.00 usec
GPA3      0.00 usec
GPT2      0.00 usec
GPT3      0.00 usec
GPT4      0.00 usec
GPT5      0.00 usec
GPT6      0.00 usec
GPT7      0.00 usec
GPT8      0.00 usec
GPT9      0.00 usec
GPT10     0.00 usec
GPT11     0.00 usec
GPT12     0.00 usec
GPT13     0.00 usec
GPT14     0.00 usec
GPT15     0.00 usec
GPT16     0.00 usec

F1 - Acquisition Parameters
=====
NUC1      13C
P1        1.50 usec
PL1       1.60 dB
SFO1      500.2235 MHz
SF        500.2235 MHz
AQ        0.1278452 sec
RG        640.000
DE        6.000 usec
TE        298.0 K
d0        0.0000000 sec
d1        1.0000000 sec
d11       0.0000000 sec
d12       0.0000000 sec
d16       0.0002000 sec
d18       0.0002000 sec
d19       0.0002000 sec
d30       0.0002480 sec

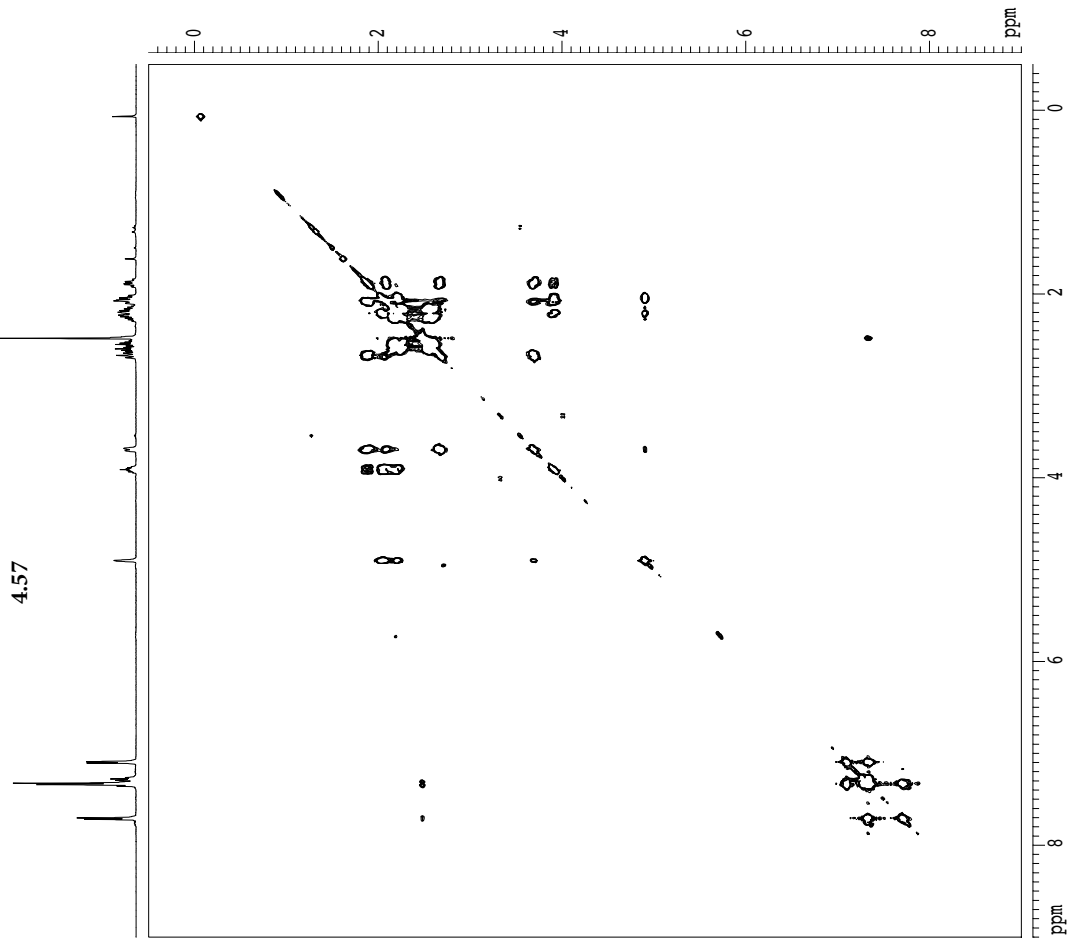
===== CHANNEL f1 =====
NUC1      1H
P1        1.50 usec
PL1       1.60 dB
SFO1      500.2235015 MHz

===== GRABBER CHANNEL =====
GNUM1     1
GPRG1     SINE
GPRZ1     180.000
GPA1      0.00 usec
GPA2      0.00 usec
GPT1      0.00 usec
GPT2      0.00 usec
GPT3      0.00 usec
GPT4      0.00 usec
GPT5      0.00 usec
GPT6      0.00 usec
GPT7      0.00 usec
GPT8      0.00 usec
GPT9      0.00 usec
GPT10     0.00 usec
GPT11     0.00 usec
GPT12     0.00 usec
GPT13     0.00 usec
GPT14     0.00 usec
GPT15     0.00 usec
GPT16     0.00 usec

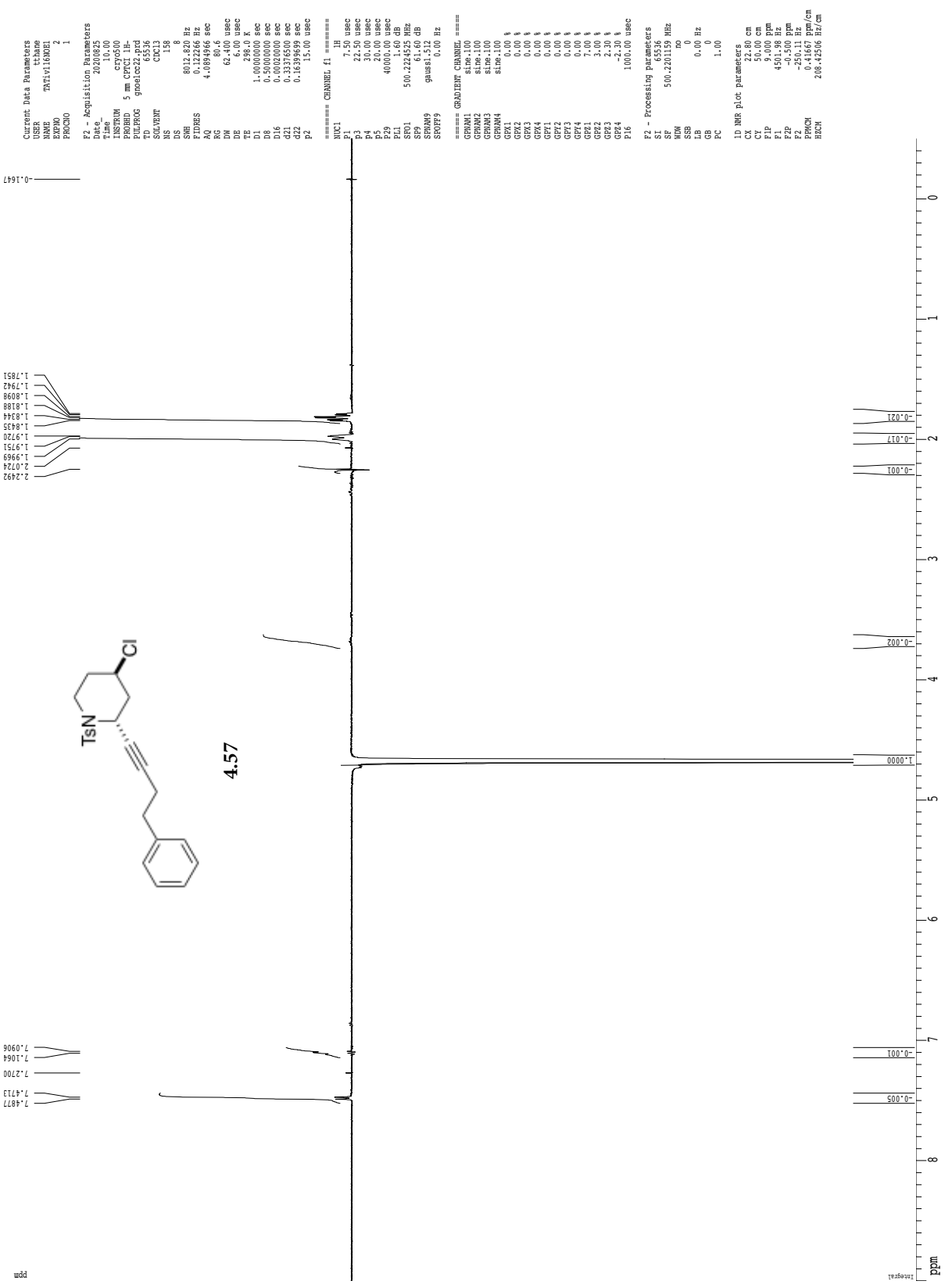
F2 - Processing Parameters
=====
SI        1024
SF        500.2200000 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0
PC        1.00

F1 - Processing Parameters
=====
SI        1024
SF        500.2200000 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0
PC        1.00

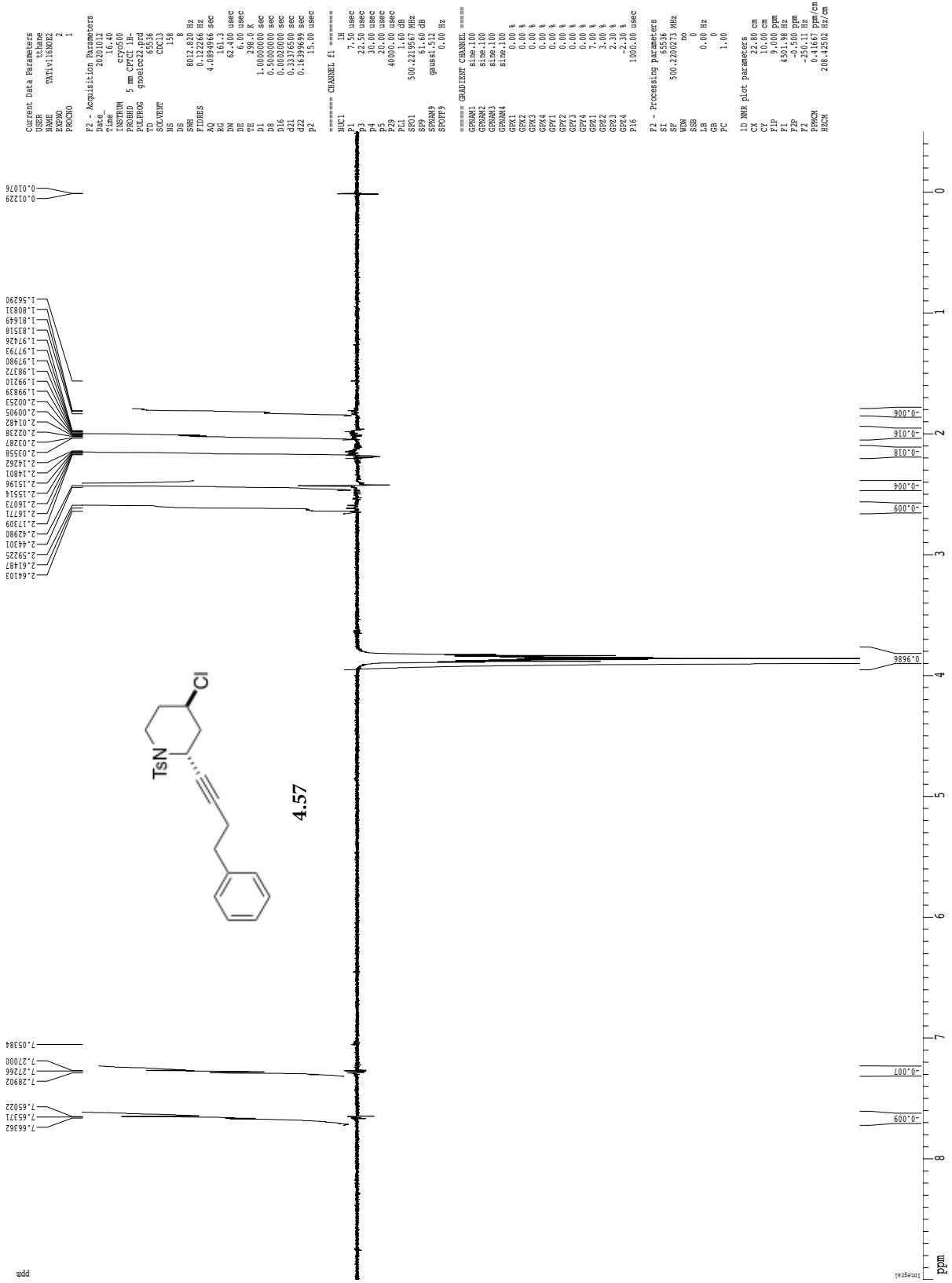
2D NMR plot parameters
=====
CX1       15.00 cm
CX2       15.00 cm
FZPC1     9.1000 ppm
FZPC2     9.1000 ppm
FZPC3     -0.5000 ppm
FZPC4     -250.11 Hz
F1PC1     9.4000 ppm
F1PC2     4501.98 Hz
F1PC3     -10.5000 ppm
F1PC4     -10.5000 ppm
F2PC1MCH 0.63333 ppm/cm
F2PC2MCH 316.80600 Hz/cm
F1PC1MCH 0.63333 ppm/cm
F1PC2MCH 316.80600 Hz/cm
  
```



gnoe

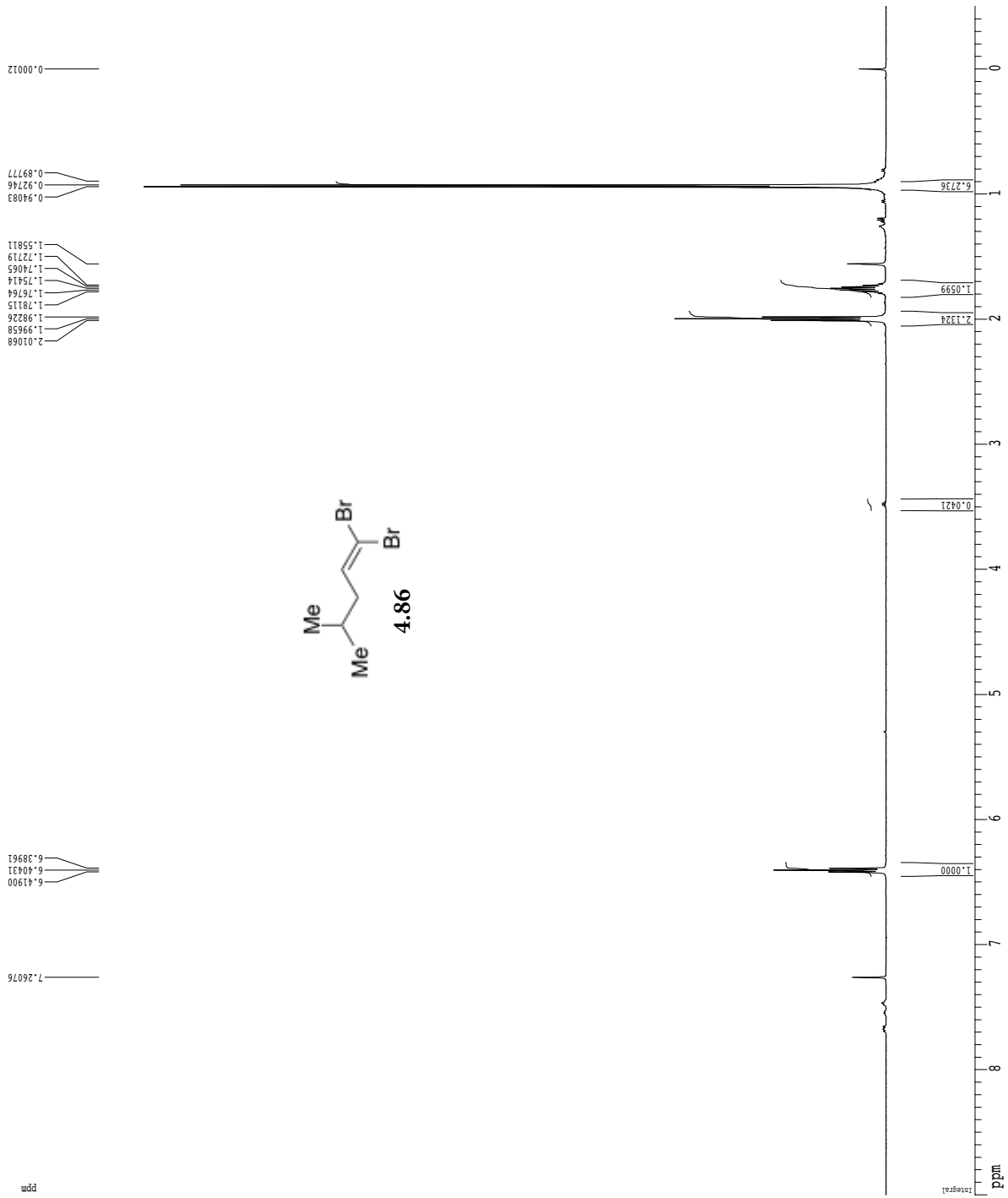


gnoe





1H spectrum



Current Data Parameters  
 USER tthane  
 NAME TATiv66c  
 EXPNO 1  
 PROCNO 1

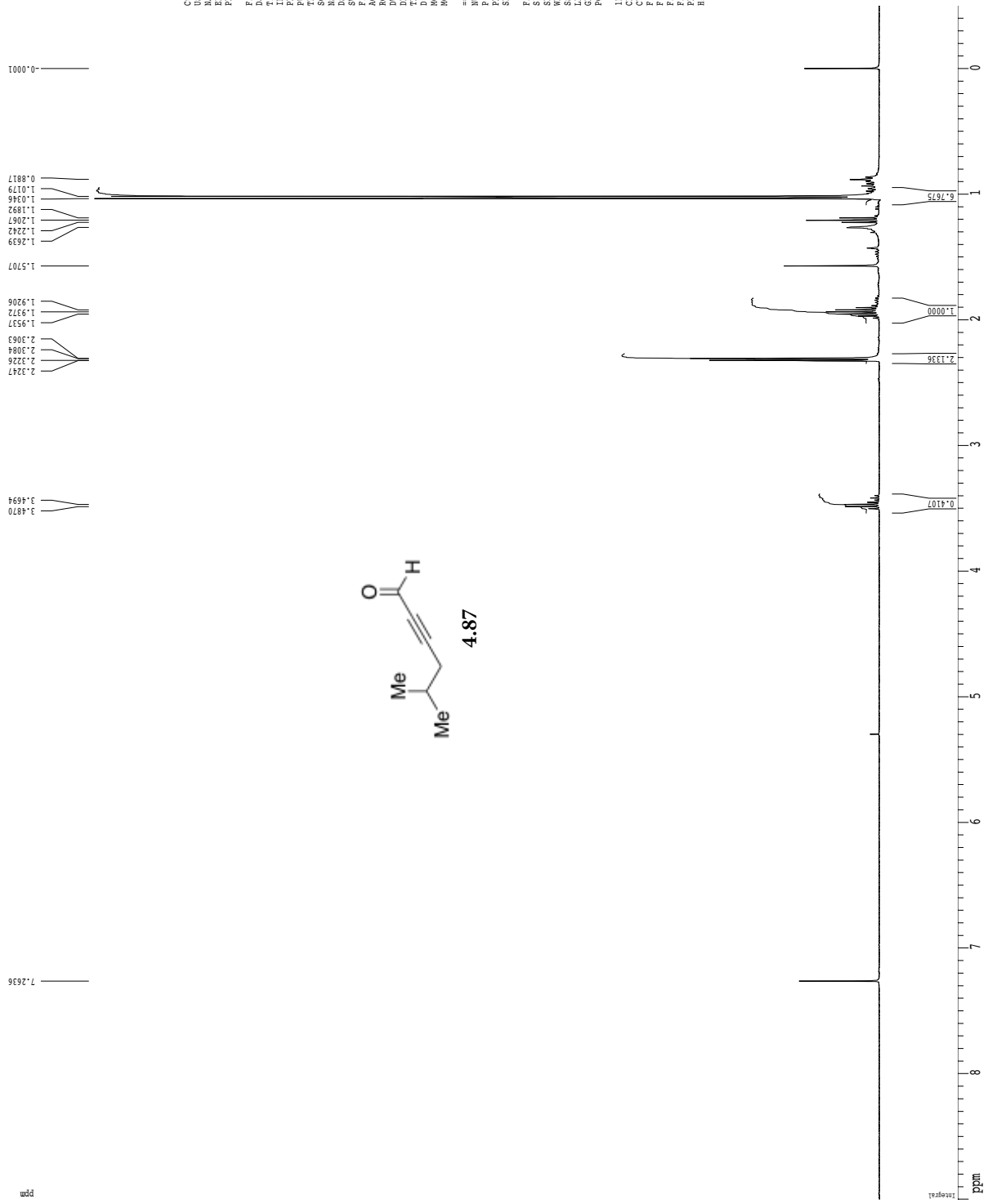
F2 - Acquisition Parameters  
 Date\_ 20200715  
 Time 11.43  
 INSTRUM gn500  
 PROBHD 5 mm broadband  
 PULPROG zg30  
 TD 81728  
 SOLVENT CDCl3T  
 NS 8  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.098043 Hz  
 AQ 5.0998774 sec  
 RG 512  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 MCREST 0.00000000 sec  
 MCWRK 0.01500000 sec

==== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -6.00 dB  
 SF01 498.7534913 MHz

F2 - Processing parameters  
 SI 65536  
 SF 498.7500309 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

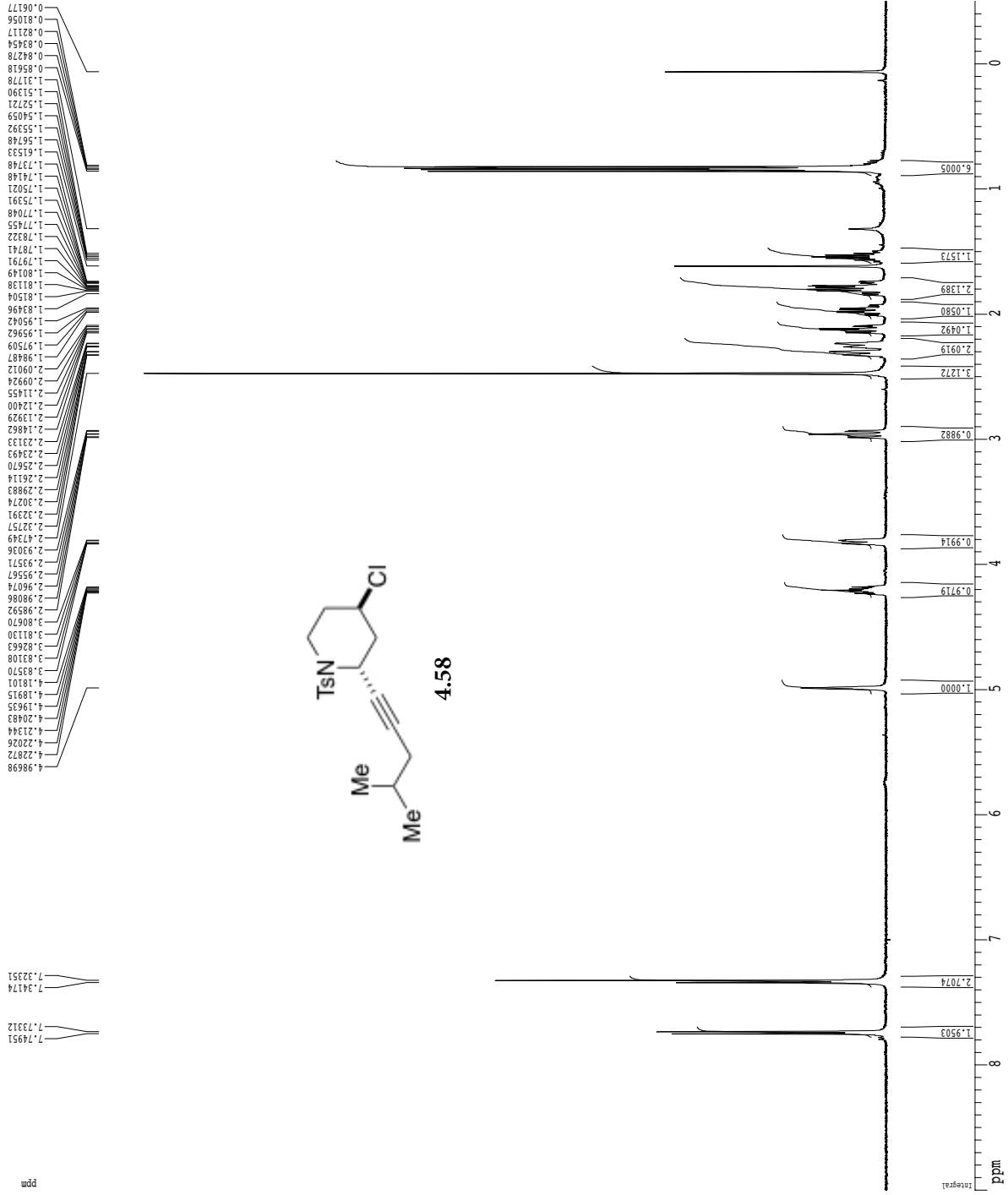
ID NMR plot parameters  
 CX 20.00 cm  
 CY 12.50 cm  
 FLP 9.000 ppm  
 F1 4488.75 Hz  
 F2P -0.500 ppm  
 F2 -249.38 Hz  
 PPMCM 0.47500 ppm/cm  
 HZCM 236.90627 Hz/cm

1H spectrum



Current Data Parameters  
 USER: etabane  
 NAME: TMTM99ure  
 EXNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20200711  
 Time: 15:40  
 PROBNM: 1000000000  
 PROCNO: 5 nm QNP H270  
 PULPROG: zg30  
 TD: 65536  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 6410.258 Hz  
 FIDRES: 0.09781 Hz  
 AQ: 5.11857 sec  
 RG: 384  
 DW: 78.000 usec  
 DE: 4.50 usec  
 TE: 298.1 K  
 T1: 0.100000 sec  
 T1RHO: 0.000000 sec  
 T2: 0.000000 sec  
 T2RHO: 0.000000 sec  
 MCHRG: 0.000000 sec  
 MCHRG: 0.000000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -1.10 dB  
 SFO1: 400.132809 MHz  
 F2 - Processing Parameters  
 SI: 65536  
 SF: 400.1300203 MHz  
 WDM: no  
 LB: 0.00 Hz  
 GB: 0  
 PC: 2.00  
 ID: NMR PLOT Parameters  
 CY: 22.80 cm  
 CX: 15.00 cm  
 FIP: 9.000 ppm  
 FT: 3600.17 Hz  
 F2: 2000.8 Hz  
 F2: -2000.8 Hz  
 PPMCM: 0.41667 ppm/cm  
 HZCM: 166.72086 Hz/cm

1H spectrum



Current Data Parameters  
 USER tthane  
 NAME FAT1V69C  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20200715  
 Time 11.46  
 INSTRUM gn500  
 PROBHD 5 mm broadband  
 PULPROG zg30  
 TD 81728  
 SOLVENT CDCl3T  
 NS 8  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.098043 Hz  
 AQ 5.0998774 sec  
 RG 912.3  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 MCREST 0.00000000 sec  
 MCWRK 0.01500000 sec

==== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -6.00 dB  
 SF01 498.7534913 MHz

F2 - Processing parameters

SI 6536  
 SF 498.7500000 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00  
 ID NMR plot parameters  
 CX 20.00 cm  
 CY 12.50 cm  
 FLP 9.000 ppm  
 F1 4488.75 Hz  
 F2P -0.500 ppm  
 F2 -249.38 Hz  
 PPMCM 0.47500 ppm/cm  
 HZCM 236.90625 Hz/cm



gc0sy60

```

Current Data Parameters
USER          tthane
NAME         TX1V16COSY
EXPNO        2
PROCNO       1

F2 - Acquisition Parameters
Date_        200805
Time         15:15
INSTRUM      cryo500
PROBHD       5 mm CPTCI 1H-
PULPROG      cosygp60.prd
TD           2048
SOLVENT      CDCl3
NS           2
DS           16
SFO1         8012.820 Hz
FIDRES       3.972510 Hz
AQ           0.1278452 sec
RG           456.1
DW           62.400 usec
DE           6.00 usec
TE           298.0 K
d0           0.0000000 sec
d1           1.0000000 sec
d11          0.0000000 sec
d16          0.0002000 sec
d18          0.0002000 sec
IN0          0.00012489 sec

===== CHANNEL f1 =====
NUC1         1H
P1           7.50 usec
PL1          1.60 dB
SFO1         500.2235015 MHz

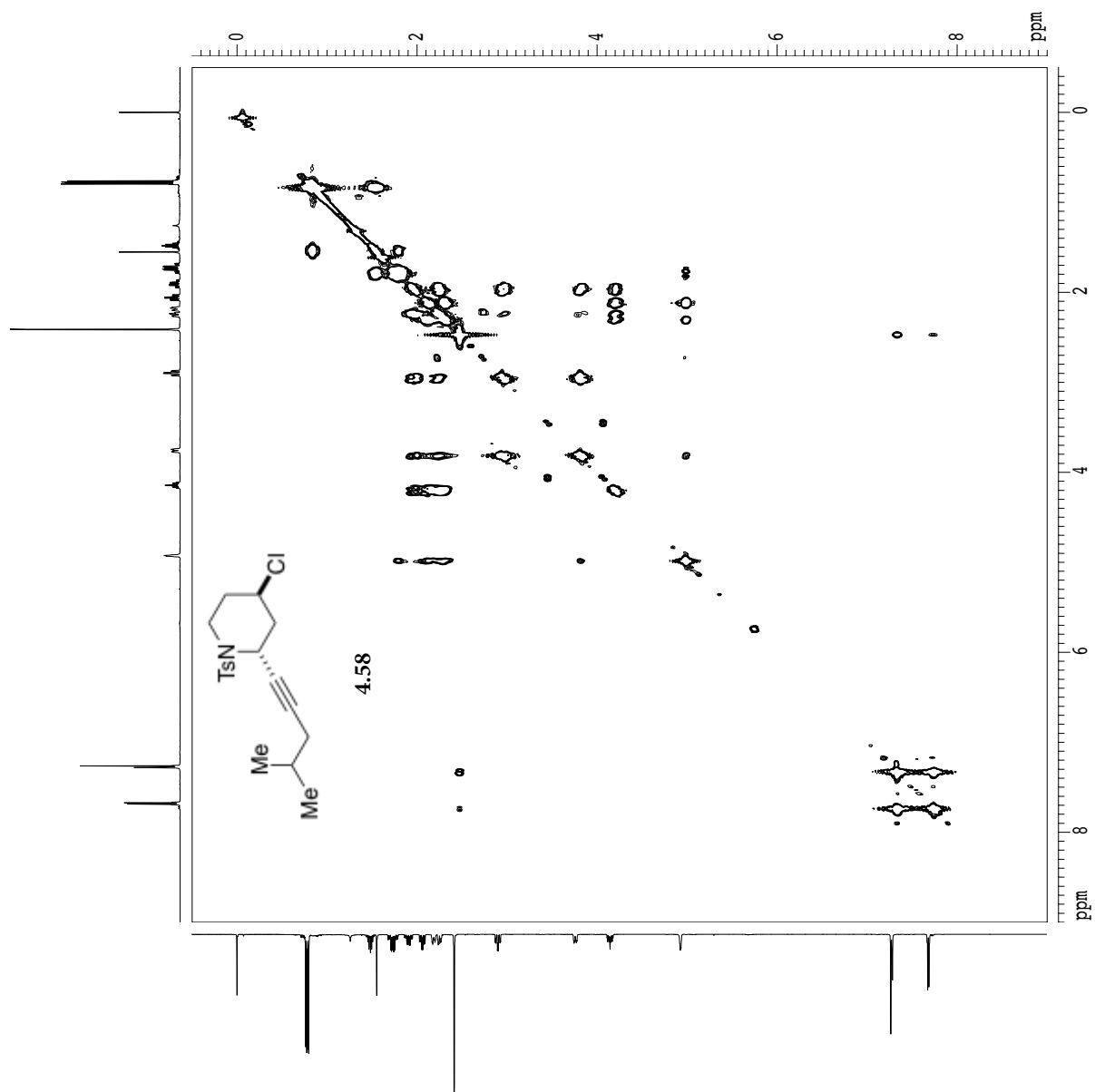
===== GRADIENT CHANNEL =====
GPRAM1      sine.100
GPRAM2      sine.100
GFX1        0.00 %
GFX2        0.00 %
GFY1        0.00 %
GFY2        0.00 %
GZ1         17.00 %
GZ2         17.00 %
P16         1000.00 usec

F1 - Acquisition parameters
ND0          1
TD           274
SFO1         500.2235 MHz
FIDRES       29.243870 Hz
SFO2         16.018 ppm
P16000      QF

F2 - Processing parameters
SI           1024
SF           500.2200000 MHz
WDW          SINE
SSB          0
LB           0.00 Hz
GB           0
PC           1.00

F1 - Processing parameters
SI           1024
MC2          QF
SF           500.2200000 MHz
WDW          SINE
SSB          0
LB           0.00 Hz
GB           0

2D NMR plot parameters
CX2          15.00 cm
CX1          15.00 cm
F2PLO        9.000 ppm
F2FLO        4501.98 Hz
F2PHI        -0.500 ppm
F2H1         -250.11 Hz
F2H2         4501.98 ppm
F2LO         4501.98 Hz
F2HI         -0.500 ppm
F2H1H        -250.11 Hz
F2PH1CH      0.65333 ppm/cm
F2PH2CH      316.80600 Hz/cm
F2PH1CM      0.65333 ppm/cm
F2PH2CM      316.80600 Hz/cm
  
```

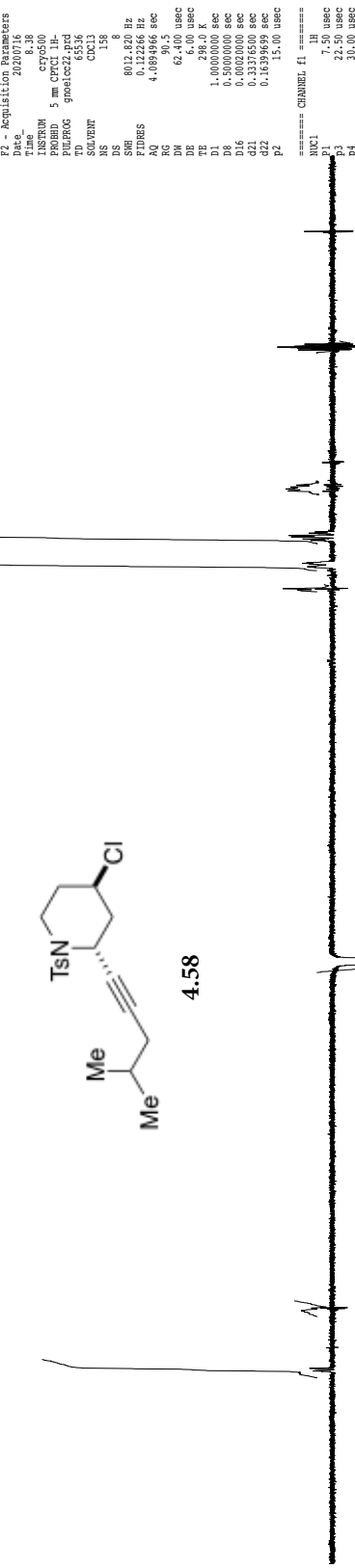


gnoe

```

ppm
0.002820
0.001591
0.010609
0.014164
0.771400
0.773700
0.781595
0.785922
0.792223
0.795011
0.797122
0.802221
0.805554
0.808404
1.567077
1.569222
1.719860
1.720888
1.723535
1.726877
1.735553
1.749111
1.750101
1.753085
1.761888
2.036277
2.045211
2.054145
2.060801
2.070303
2.084499
2.088452
2.246055
2.248055
2.249296
2.250511
2.264933
2.269188
2.272944
2.297311
2.320805
2.331333
2.420977
2.903767
2.910677

```

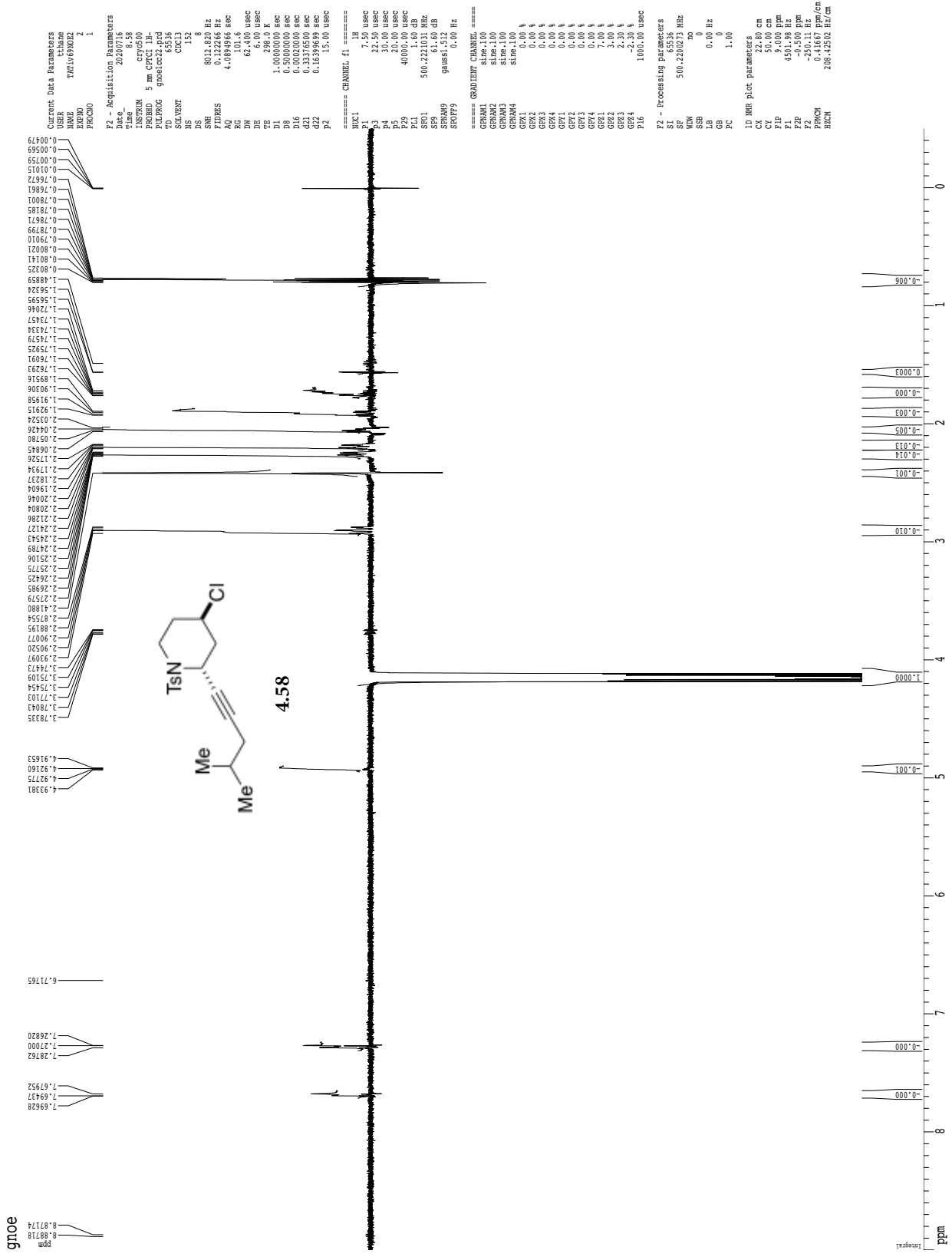


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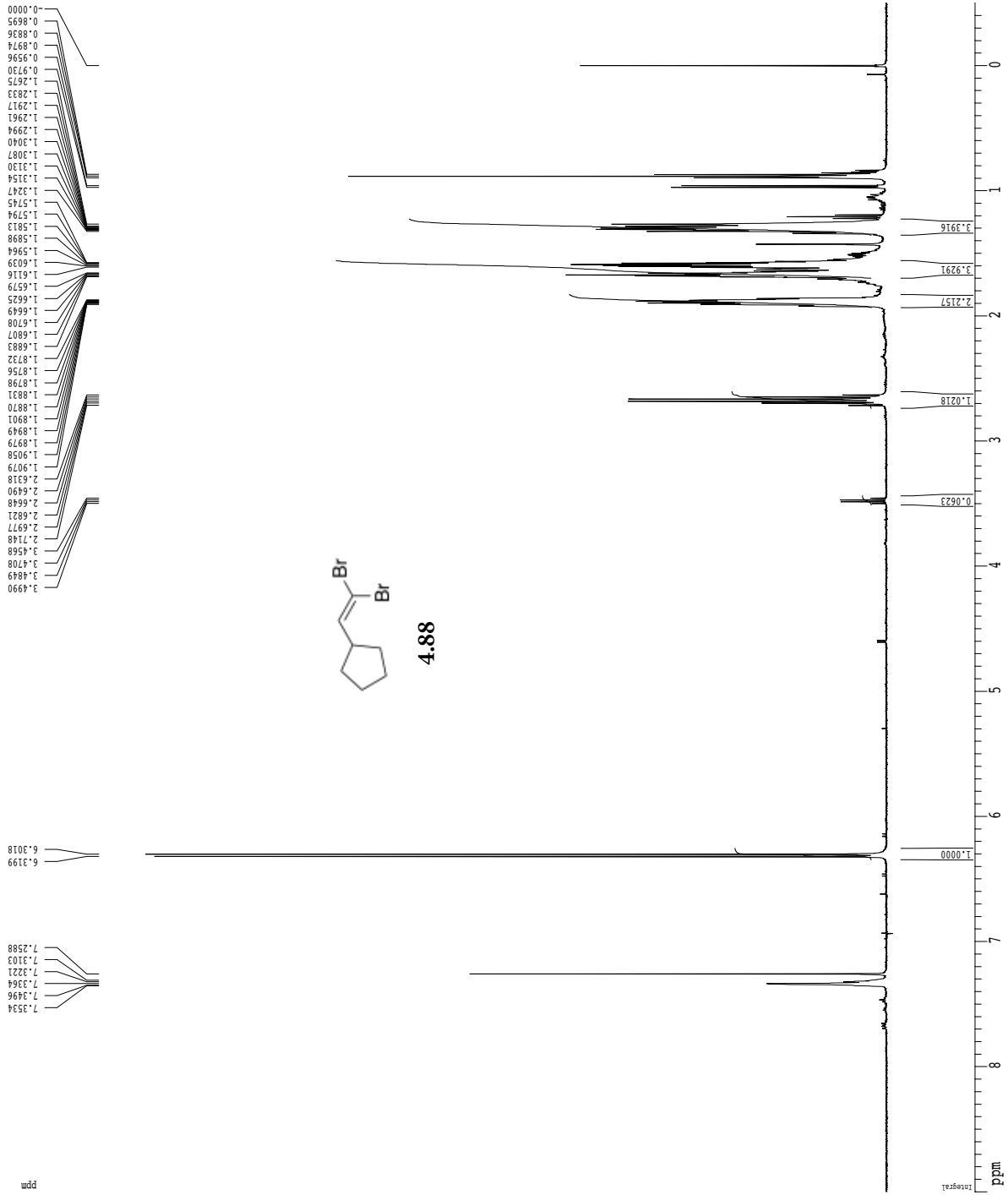
==== CHANNEL f1 =====
NUC1 1H
P1 7.50 usec
P2 22.50 usec
P3 20.00 usec
P4 20.00 usec
P5 20.00 usec
P29 40000.00 usec
P31 1.66 dB
RF1 500.2224946 MHz
SFO9 9045.157 dB
SFO99 0.00 Hz
SFOFF9
===== GRADIENT CHANNEL =====
GR1 0.00 Hz
GR2 0.00 Hz
GR3 0.00 Hz
GR4 0.00 Hz
GR5 0.00 Hz
GR6 0.00 Hz
GR7 0.00 Hz
GR8 0.00 Hz
GR9 0.00 Hz
GR10 0.00 Hz
GR11 0.00 Hz
GR12 0.00 Hz
GR13 0.00 Hz
GR14 0.00 Hz
GR15 0.00 Hz
GR16 1000.00 usec
P2 - Processing parameters
SI 655.36
FIDRES 500.2200255 MHz
NUC1 1H
NUC2 13C
SSB 0
GB 0.00 Hz
PC 1.00
ID NMR PLOT parameters
CX 22.88 cm
CY 50.00 cm
CZ 40.00 cm
FX 450.00 ppm
FY 450.00 ppm
FZ 0.5000 ppm
P2 -250.11 Hz
PRICM 0.41667 ppm/cm
HCHN 200.42502 Hz/cm

```





1H spectrum



Current Data Parameters  
 USER tthane  
 NAME TAT1118-1  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20200826  
 Time 15.29  
 INSTRUM gn500  
 PROBHD 5 mm broadband  
 PULPROG zg30  
 TD 81728  
 SOLVENT CDCl3T  
 NS 8  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.098043 Hz  
 AQ 5.0998774 sec  
 RG 181  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 MCREST 0.00000000 sec  
 MCWRK 0.01500000 sec

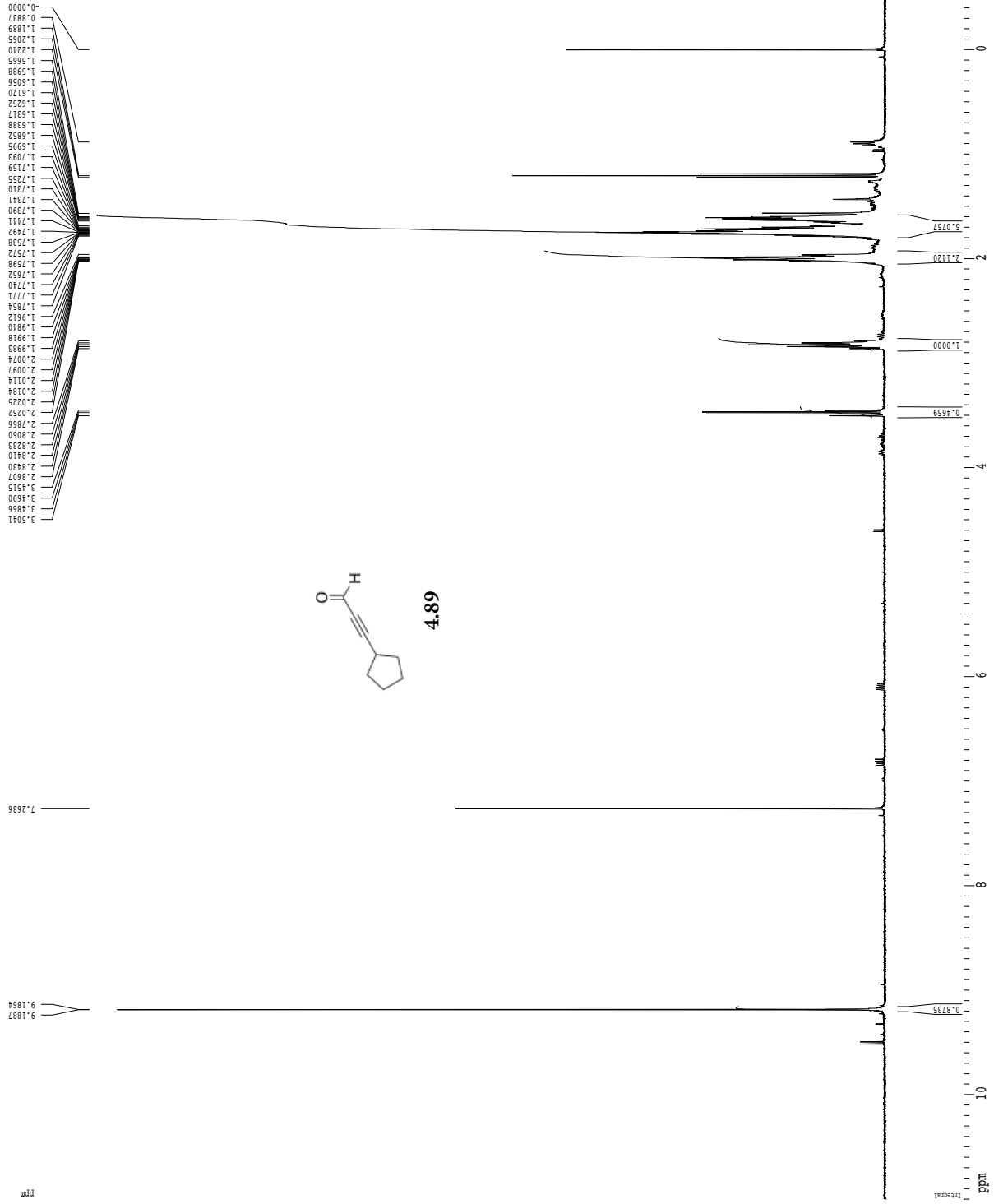
==== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -6.00 dB  
 SF01 498.7534913 MHz

F2 - Processing parameters  
 SI 65536  
 SF 498.7500319 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 12.50 cm  
 FL1 9.000 ppm  
 F1 4488.75 Hz  
 F2P -0.500 ppm  
 F2 -249.38 Hz  
 PPMCM 0.47500 ppm/cm  
 HZCM 236.90627 Hz/cm



1H spectrum



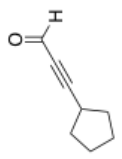
Current Data Parameters  
 USER: TUNUY12@ure  
 NAME: TUNUY12@ure  
 EKWO: 1  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20200827  
 Time: 17.04  
 PROBNM: 17040827  
 PROCNO: 5  
 PULPROG: zgpg30  
 TD: 65536  
 SOLVENT: CDCl3  
 DS: 2  
 SFO: 400.130203 MHz  
 SF: 400.130203 MHz  
 FIDRES: 0.097813 Hz  
 AQ: 5.118577 sec  
 RG: 327.5  
 DE: 4.50 usec  
 TE: 298.1 K  
 ACQST: 0.100000 sec  
 MCOREST: 0.100000 sec  
 MCOREK: 0.0150000 sec

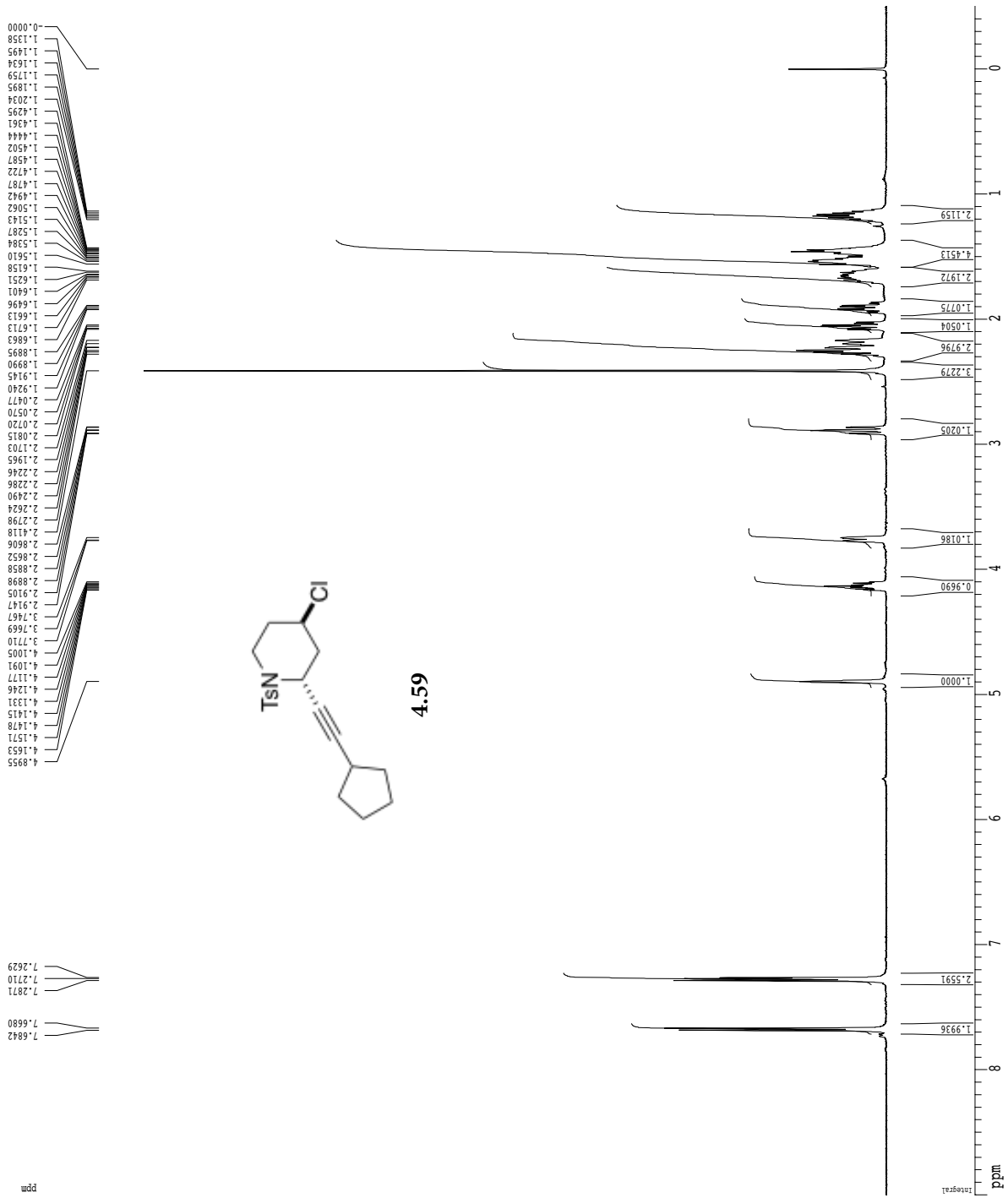
===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 12.00 usec  
 PL1: -1.10 dB  
 SFO1: 400.1326009 MHz

F2 - Processing Parameters  
 SI: 65536  
 SF: 400.130203 MHz  
 NDM: no  
 LB: 0.00 Hz  
 GB: 0  
 PC: 2.00

DJ MRB P1.C: Parameters  
 CY: 22.80 cm  
 CV: 15.00 cm  
 FIP: 11.000 ppm  
 F1: 4400.40 Hz  
 F2: 2000.20 Hz  
 PPMCM: 0.50439 ppm/cm  
 HZCM: 201.81998 Hz/cm



1H spectrum



Current Data Parameters  
 USER tthane  
 NAME TATIv129carbon  
 EXPNO 1  
 PROCNO 1

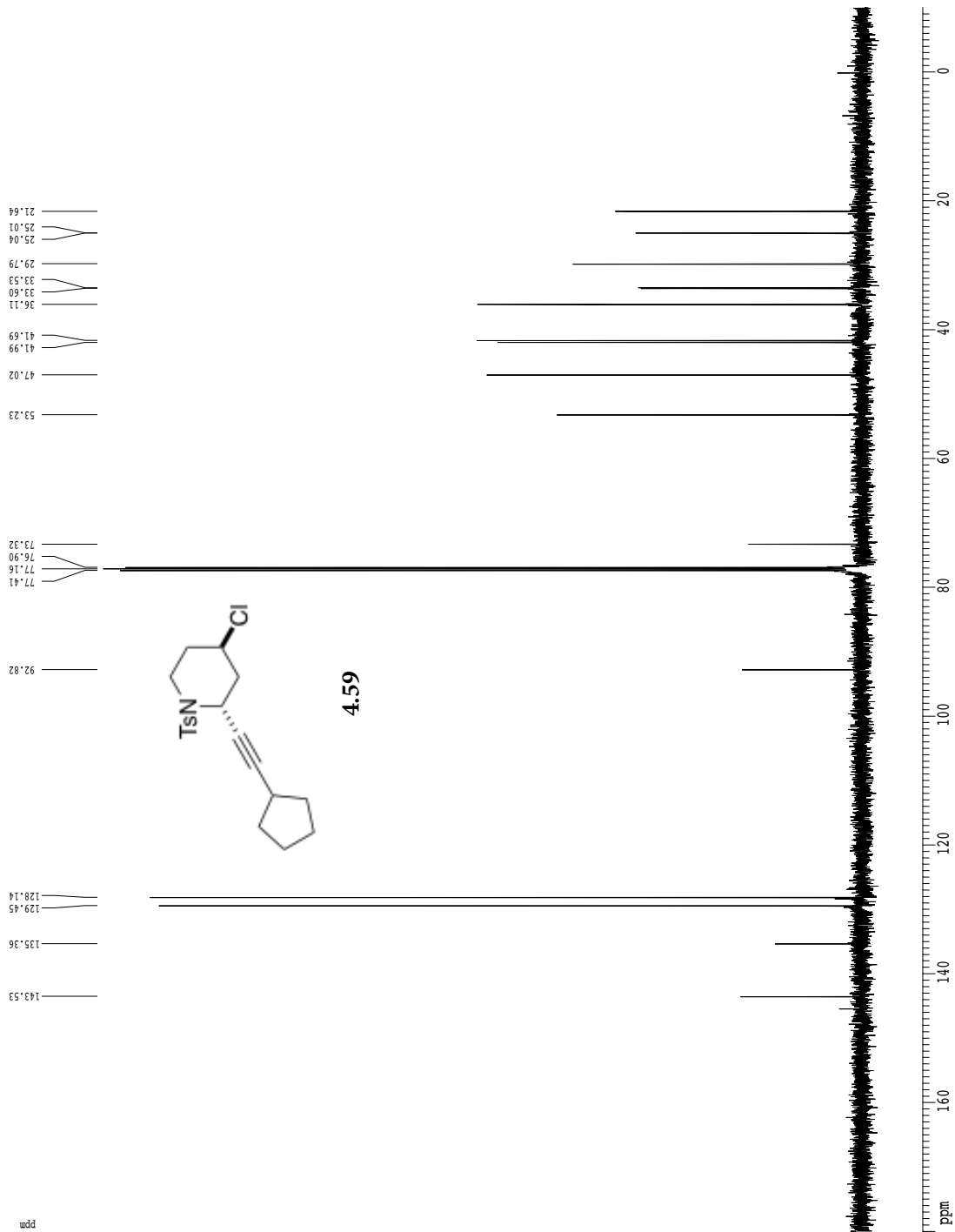
F2 - Acquisition Parameters  
 Date\_ 20201009  
 Time 15.43  
 INSTRUM gn500  
 PROBHD 5 mm broadband  
 PULPROG zg30  
 TD 81728  
 SOLVENT CDCl3T  
 NS 8  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.098043 Hz  
 AQ 5.0998774 sec  
 RG 724.1  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 MCREST 0.00000000 sec  
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -6.00 dB  
 SF01 498.7534913 MHz

F2 - Processing parameters  
 SI 65536  
 SF 498.7500301 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

ID NMR plot parameters  
 CX 20.00 cm  
 CY 12.50 cm  
 F1P 9.000 ppm  
 F1 4488.75 Hz  
 F2P -0.500 ppm  
 F2 -249.38 Hz  
 PPMCM 0.47500 ppm/cm  
 HZCM 236.90627 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters  
 USER tthane  
 NAME TATIv129carbon  
 EXPNO 2  
 PROCNO 1

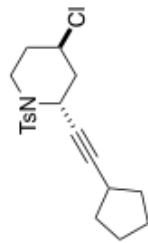
F2 - Acquisition Parameters  
 Date\_ 20201009  
 Time\_ 15.45  
 INSTRUM gm500  
 PROBDH 5 mm broadband  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 512  
 DS 4  
 SWH 30303.031 Hz  
 FIDRES 0.462388 Hz  
 AQ 1.0813940 sec  
 RG 46341  
 DW 16.500 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.25000000 sec  
 d11 0.03000000 sec  
 MCREST 0.00000000 sec  
 MCWRRK 0.01500000 sec

==== CHANNEL f1 =====  
 NUC1 13C  
 P1 14.20 usec  
 PL1 -6.00 dB  
 SF01 125.4245824 MHz

==== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 -6.00 dB  
 PL12 12.30 dB  
 SF02 498.7524937 MHz

F2 - Processing parameters  
 SI 65536  
 SF 125.4107762 MHz  
 NDMW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 2.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 12.50 cm  
 FL 180.000 ppm  
 F1 22573.94 Hz  
 F2 -10.000 ppm  
 F2 -1254.11 Hz  
 PPMCM 9.50000 ppm/cm  
 HZCM 1191.40234 Hz/cm



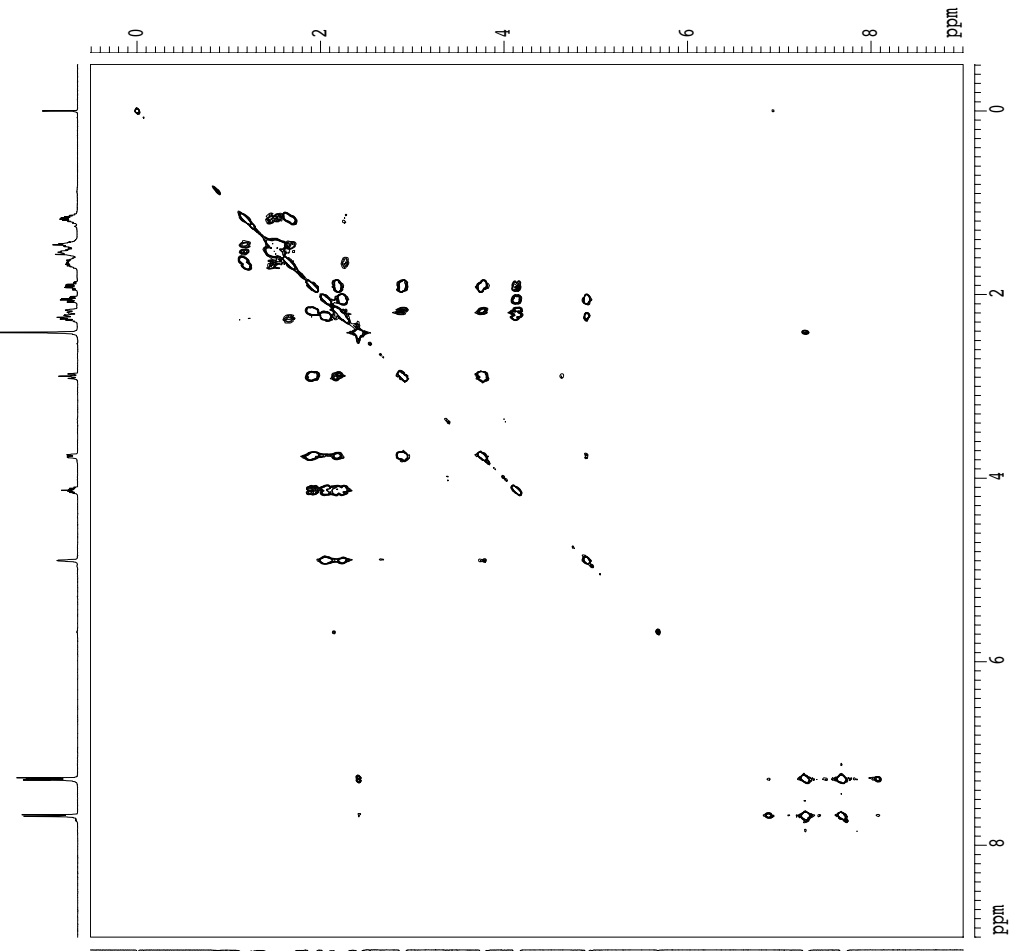
4.59

gcosy60

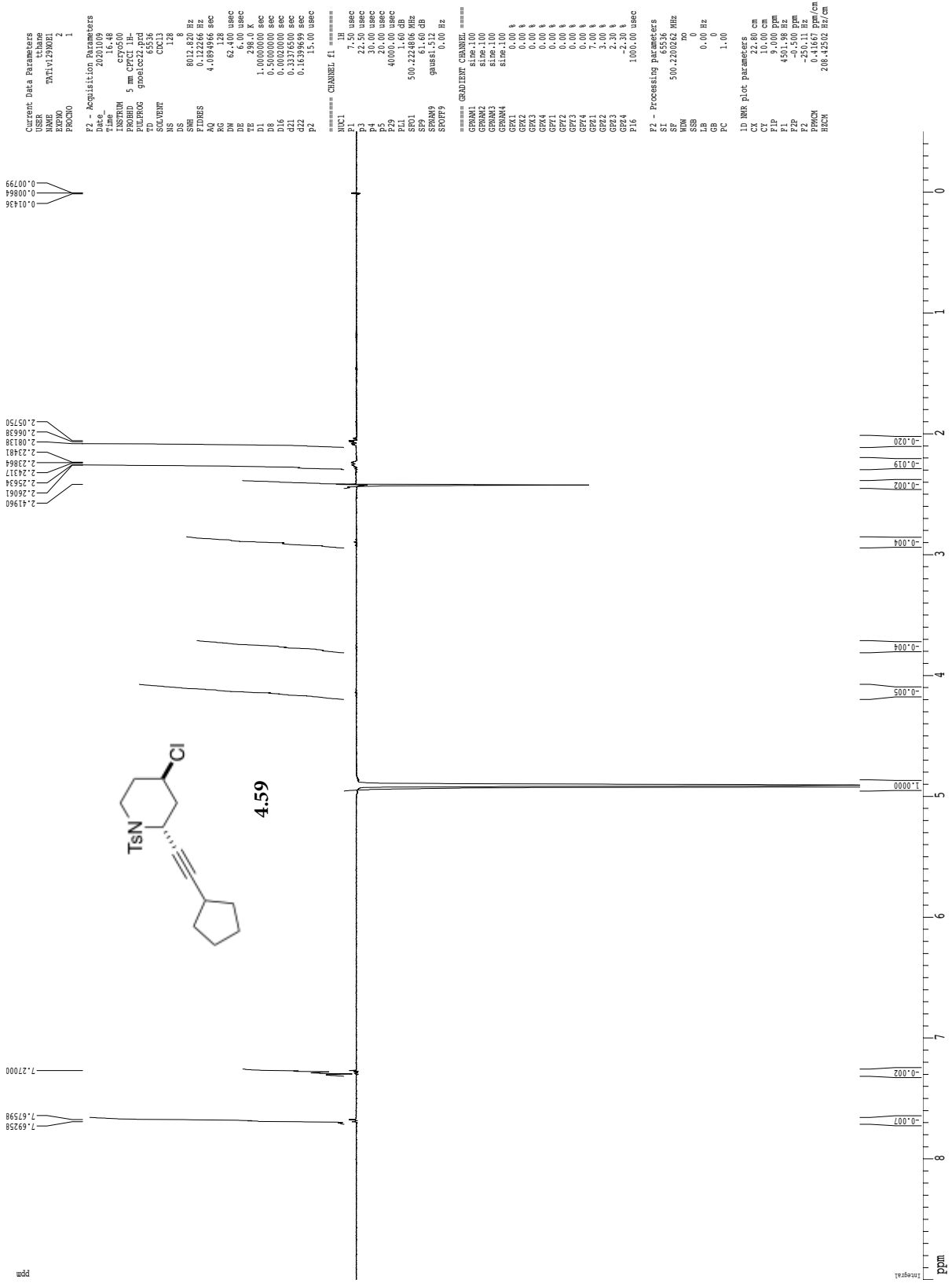
```

Current Data Parameters
=====
NAME      tthane
PROCNO    1
=====
Date_     2001010
Time      16.06
INSTRUM   crys500
PROBHD    5 mm CPXI 1H-
PULPROG   cosypr60.prd
TD        2848
SOLVENT   CDCl3
DS        2
AQ        8012.820 Hz
FIDRES    3.432510 Hz
RG         326.1
DE        6.00 USEC
TE        298.0 K
d0         0.0000000 sec
d1         1.0000000 sec
d11        0.0000000 sec
d12        0.0000000 sec
d16        0.0002000 sec
d18        0.0002480 sec
===== CHANNEL f1 =====
NUC1      1H
P1        7.00 USEC
PL1       1.60 dB
SFO1      500.2235015 MHz
===== GRADIENT CHANNEL =====
GMM1      GMR-100
GMM2      GMR-100
GMAX1     0.00 %
GMAX2     0.00 %
GPX2      0.00 %
GPT1      0.00 %
GPT2      0.00 %
GPR1      17.00 %
GPR2      17.00 %
PL16      1000.00 USEC
=====
F1 - Acquisition Parameters
=====
NUC1      1H
P1        7.00 USEC
PL1       1.60 dB
SFO1      500.2235015 MHz
===== GRADIENT CHANNEL =====
GMM1      GMR-100
GMM2      GMR-100
GMAX1     0.00 %
GMAX2     0.00 %
GPX2      0.00 %
GPT1      0.00 %
GPT2      0.00 %
GPR1      17.00 %
GPR2      17.00 %
PL16      1000.00 USEC
=====
F2 - Acquisition Parameters
=====
NUC1      13C
P1        12.00 USEC
PL1       1.90 dB
SFO1      100.6281500 MHz
===== GRADIENT CHANNEL =====
GMM1      GMR-100
GMM2      GMR-100
GMAX1     0.00 %
GMAX2     0.00 %
GPX2      0.00 %
GPT1      0.00 %
GPT2      0.00 %
GPR1      17.00 %
GPR2      17.00 %
PL16      1000.00 USEC
=====
F1 - Processing parameters
=====
SI         1024
SF         500.2200309 MHz
WDW        SINE
SSB        0
GB         0
PC         1.00
=====
F2 - Processing parameters
=====
SI         1024
SF         500.2200309 MHz
WDW        SINE
SSB        0
GB         0
PC         1.00
=====
2D NMR plot parameters
=====
CX1        15.00 cm
F2PQ0      5.000 ppm
F2PQ1      45.000 ppm
F2PH1      -0.508 ppm
F2PH0      -254.07 Hz
F1PQ0      9.403 ppm
F1PQ1      4503.54 Hz
F1PH1      -154.08 ppm
F1PH0      -754.07 Hz
F2PQM0M   0.63386 ppm/cm
F2PQM1M   317.07007 Hz/cm
F1PQM0M   0.63407 ppm/cm
F1PQM1M   317.17413 Hz/cm

```

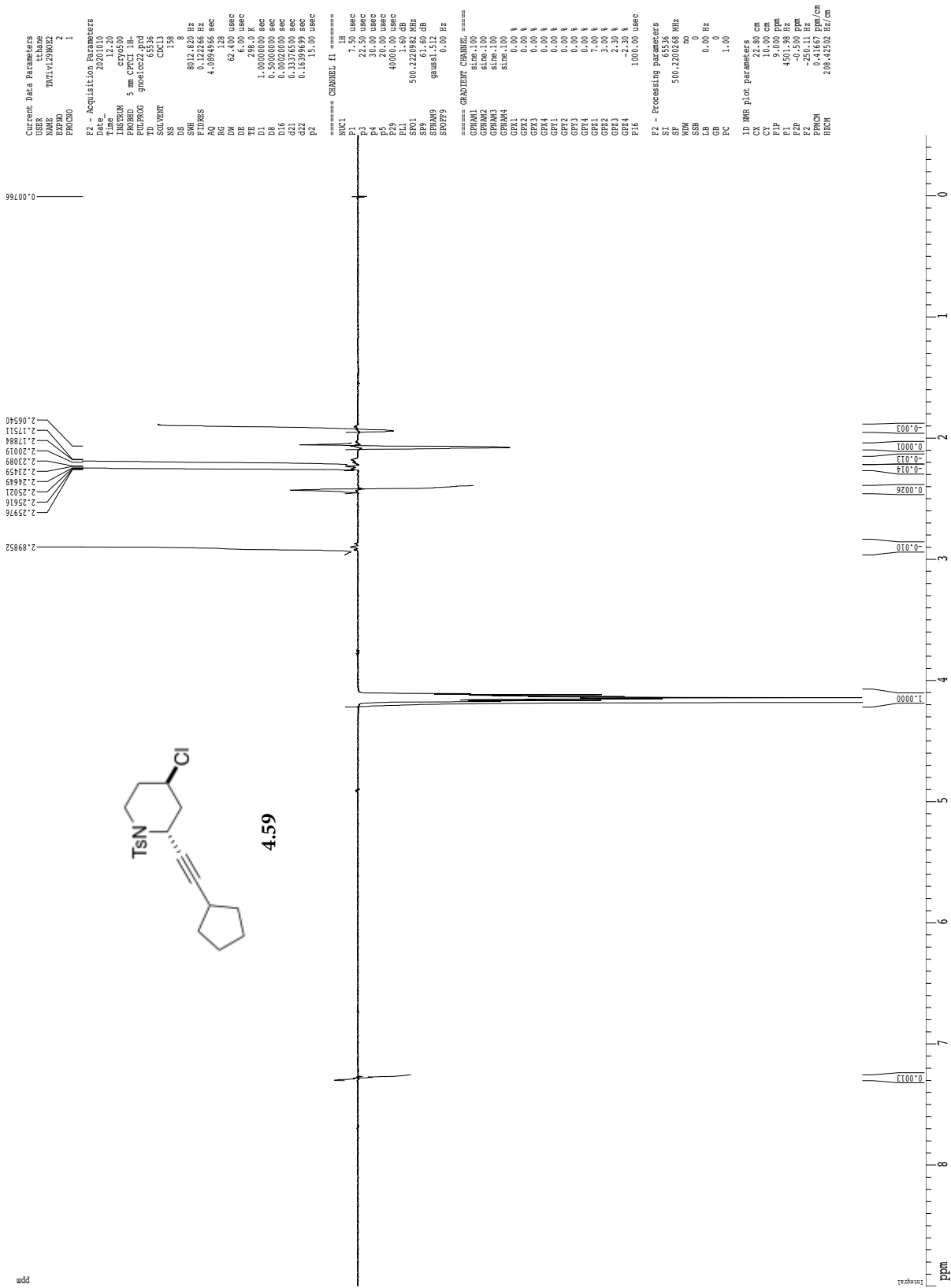


gnoe



gnoe

ppm



Current Data Parameters  
USER TVALV1290E2  
NAME TVALV1290E2  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20201010  
Time 12.20  
Operator gnoe  
PROBHD 5 mm CPDQ1 1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCL3  
DS 16  
SS 8  
SMH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894966 sec  
RG 62.400  
DE 6.00 usec  
TE 298.2 K  
D1 1.0000000 sec  
d11 0.0020000 sec  
d16 0.0020000 sec  
d21 0.33376500 sec  
d22 0.16398659 sec  
P2 15.00 usec

==== CHANNEL f1 =====  
NUC1 1H  
P1 7.50 usec  
P2 22.50 usec  
P3 21.00 usec  
P4 21.00 usec  
P5 21.00 usec  
PZ9 40004.00 usec  
PL1 1.66 dB  
SFO1 500.221098 MHz  
SFO2 500.136151 MHz  
SFO9 9999.151 MHz  
SFOFF9 0.00 Hz

==== GRABDET CHANNEL =====  
GRAB1 1000 Hz  
SFR1 1.0000000 sec  
GRAB2 1000 Hz  
SFR2 1.0000000 sec  
GRAB3 1000 Hz  
SFR3 1.0000000 sec  
GRAB4 1000 Hz  
SFR4 1.0000000 sec  
GRAB5 1000 Hz  
SFR5 1.0000000 sec  
GRAB6 1000 Hz  
SFR6 1.0000000 sec  
GRAB7 1000 Hz  
SFR7 1.0000000 sec  
GRAB8 1000 Hz  
SFR8 1.0000000 sec  
GRAB9 1000 Hz  
SFR9 1.0000000 sec  
GRAB10 1000 Hz  
SFR10 1.0000000 sec

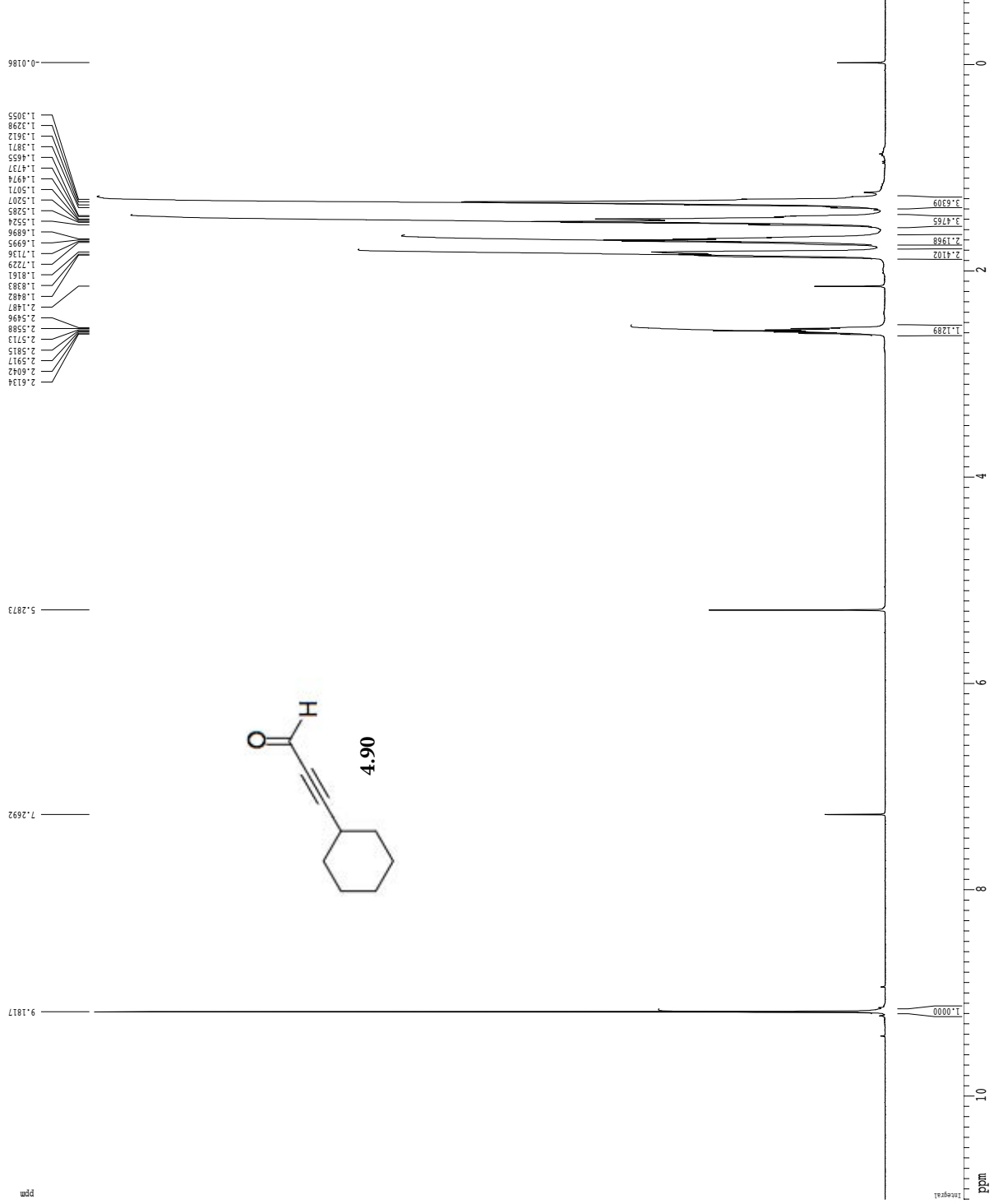
F2 - Processing parameters  
SI 65536  
SF 500.221098 MHz  
NUC1 1H  
NUC2 0  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

ID NMR plot parameters  
CX 22.88 cm  
CY 10.00 cm  
FID 1.0000000 cm  
LP 480.0000000 ppm  
FZP -0.5000000 ppm  
FZ -250.11 Hz  
PRGX 0.41666666 ppm/cm  
HCN 200.4250000 Hz/cm

Integral

ppm

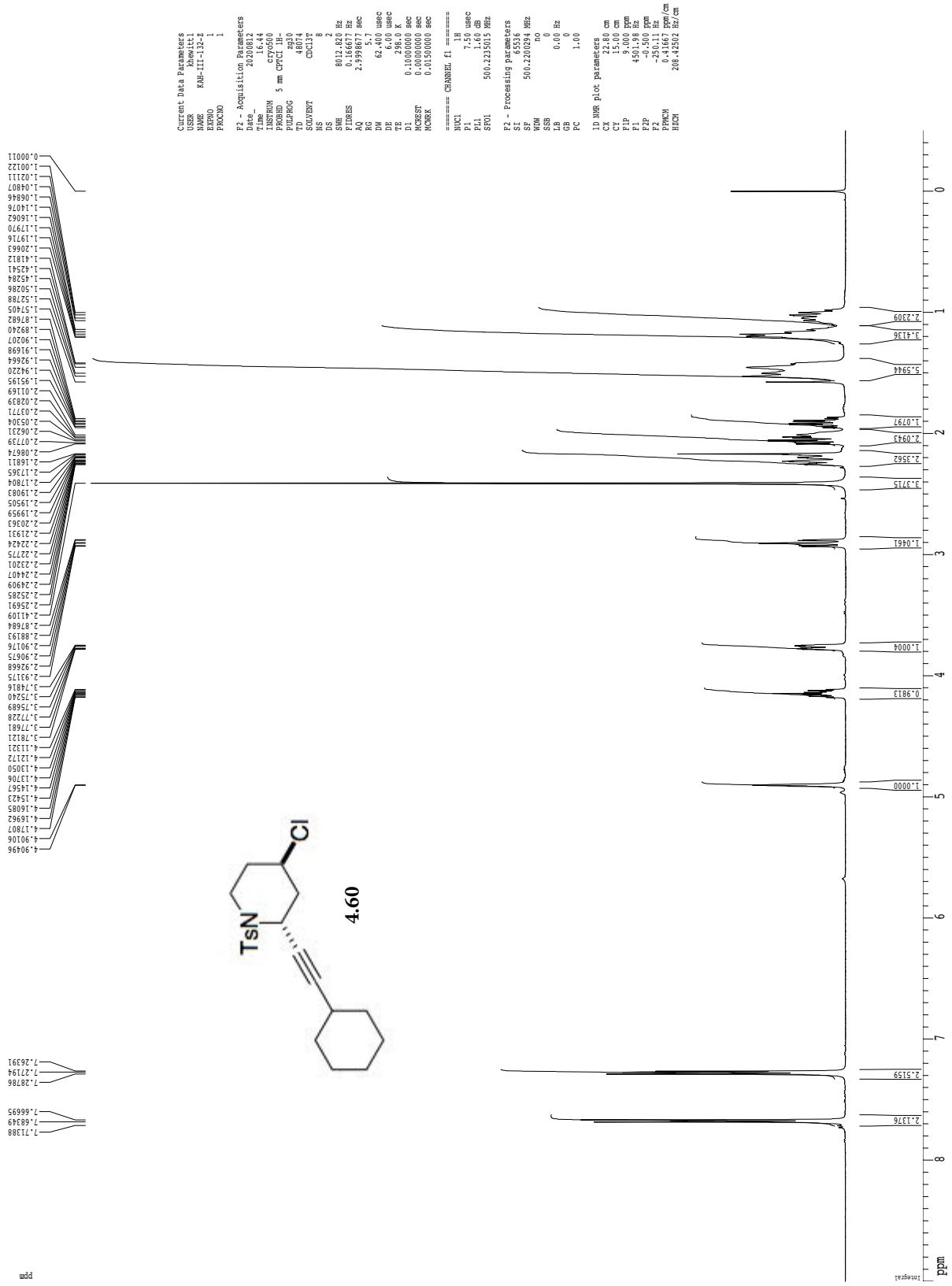
1H spectrum



Current Data Parameters  
 USER: Komettt  
 NAME: R04-III-135-1  
 EXNO: 1  
 PROCU: 1

F2 - Acquisition Parameters  
 Date\_: 20200612  
 Time: 13.07  
 PROGN: 1070  
 PROCNO: 5  
 PULPROG: zg30  
 TD: 38460  
 SOLVENT: CDCl3  
 DS: 2  
 SFO: 5  
 SHF: 6410.258 Hz  
 FIDRES: 0.166677 Hz  
 AQ: 2.399279 sec  
 RG: 327.5  
 DD: 78.000 usec  
 DE: 4.50 usec  
 TE: 298.2 K  
 T1: 0.100000 sec  
 T2: 0.000000 sec  
 T3: 0.000000 sec  
 T4: 0.000000 sec  
 T5: 0.000000 sec  
 T6: 0.000000 sec  
 T7: 0.000000 sec  
 T8: 0.000000 sec  
 T9: 0.000000 sec  
 T10: 0.000000 sec  
 T11: 0.000000 sec  
 T12: 0.000000 sec  
 T13: 0.000000 sec  
 T14: 0.000000 sec  
 T15: 0.000000 sec  
 T16: 0.000000 sec  
 T17: 0.000000 sec  
 T18: 0.000000 sec  
 T19: 0.000000 sec  
 T20: 0.000000 sec  
 T21: 0.000000 sec  
 T22: 0.000000 sec  
 T23: 0.000000 sec  
 T24: 0.000000 sec  
 T25: 0.000000 sec  
 T26: 0.000000 sec  
 T27: 0.000000 sec  
 T28: 0.000000 sec  
 T29: 0.000000 sec  
 T30: 0.000000 sec  
 T31: 0.000000 sec  
 T32: 0.000000 sec  
 T33: 0.000000 sec  
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 T53: 0.000000 sec  
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 T82: 0.000000 sec  
 T83: 0.000000 sec  
 T84: 0.000000 sec  
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 T87: 0.000000 sec  
 T88: 0.000000 sec  
 T89: 0.000000 sec  
 T90: 0.000000 sec  
 T91: 0.000000 sec  
 T92: 0.000000 sec  
 T93: 0.000000 sec  
 T94: 0.000000 sec  
 T95: 0.000000 sec  
 T96: 0.000000 sec  
 T97: 0.000000 sec  
 T98: 0.000000 sec  
 T99: 0.000000 sec  
 T100: 0.000000 sec

1H spectrum



Current Data Parameters  
 USER KSH-III-132-2  
 EXPO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20200812  
 Time 16:44  
 INSTRUM c750  
 PROBHD 5 mm CP131-4H  
 PULPROG zgpg30  
 TD 48074  
 SOLVENT CHCl3  
 DS 9  
 SFO 8012.820 Hz  
 FIDRES 0.16667 Hz  
 AQ 2.3398677 sec  
 RG 62.400 usec  
 DE 6.00 usec  
 TE 298.2 K  
 0.16000000 sec  
 0.00000000 sec  
 0.01500000 sec

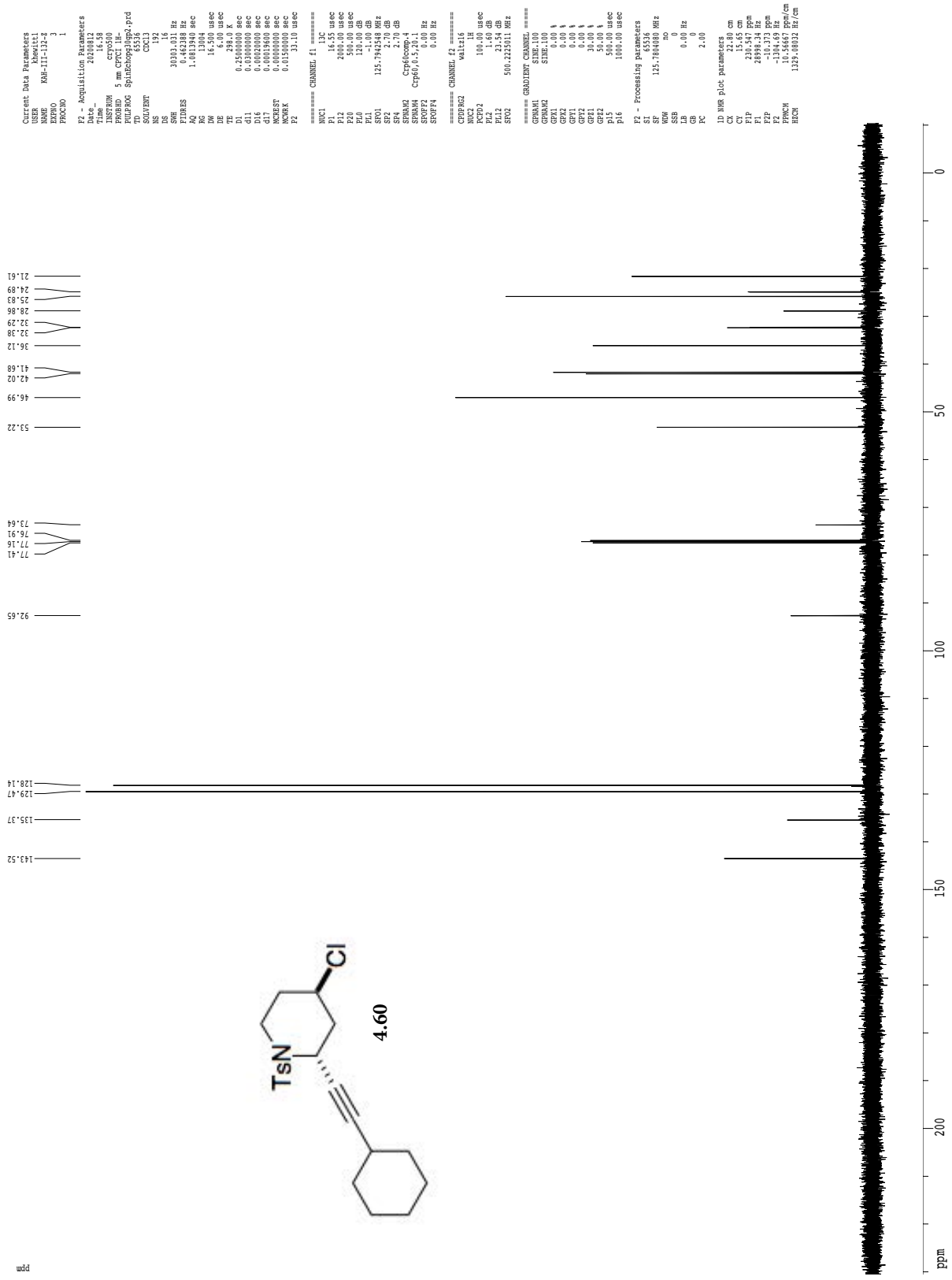
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 1.50 usec  
 PL1 1.66 dB  
 SFO1 500.2235015 MHz

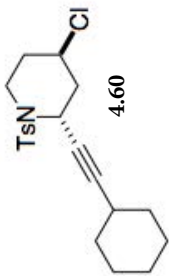
F2 - Processing Parameters  
 SI 65536  
 SF 500.2200294 MHz  
 WDW no  
 GB 0.00 Hz  
 CB 0  
 PC 1.00

D0 NMR P1.c Parameters  
 CY 22.80 cm  
 CV 15.00 cm  
 FIP 9.000 ppm  
 F1 4501.500 Hz  
 F2 -250.11 Hz  
 PPM0 0.44667 ppm/cm  
 HZ0 208.44502 Hz/cm



Z-restored spin-echo 13C spectrum with 1H decoupling





gcosy60

Current Data Parameters  
 USER Khevit11  
 NAME KM-11-132-2  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20200812  
 Time 16.45  
 INSTRUM cryo500  
 PULPROG zgpg30  
 PROCNO 1  
 TD 2048  
 SOLVENT CDCl3  
 NS 1  
 DS 16  
 SWH 8013.81 Hz  
 FIDRES 3.912510 Hz  
 AQ 0.1278452 sec  
 RG 114  
 DW 62.400 usec  
 DE 298.0 usec  
 TE 298.0 usec  
 d0 0.40000300 sec  
 d1 1.40000000 sec  
 d13 0.40000300 sec  
 d16 0.40000000 sec  
 d10 0.40000480 sec

===== CHANNEL f1 =====  
 NUC1 <sup>1</sup>H  
 P1 7.50 usec  
 PL 0.00 dB  
 SFO1 500.225015 MHz

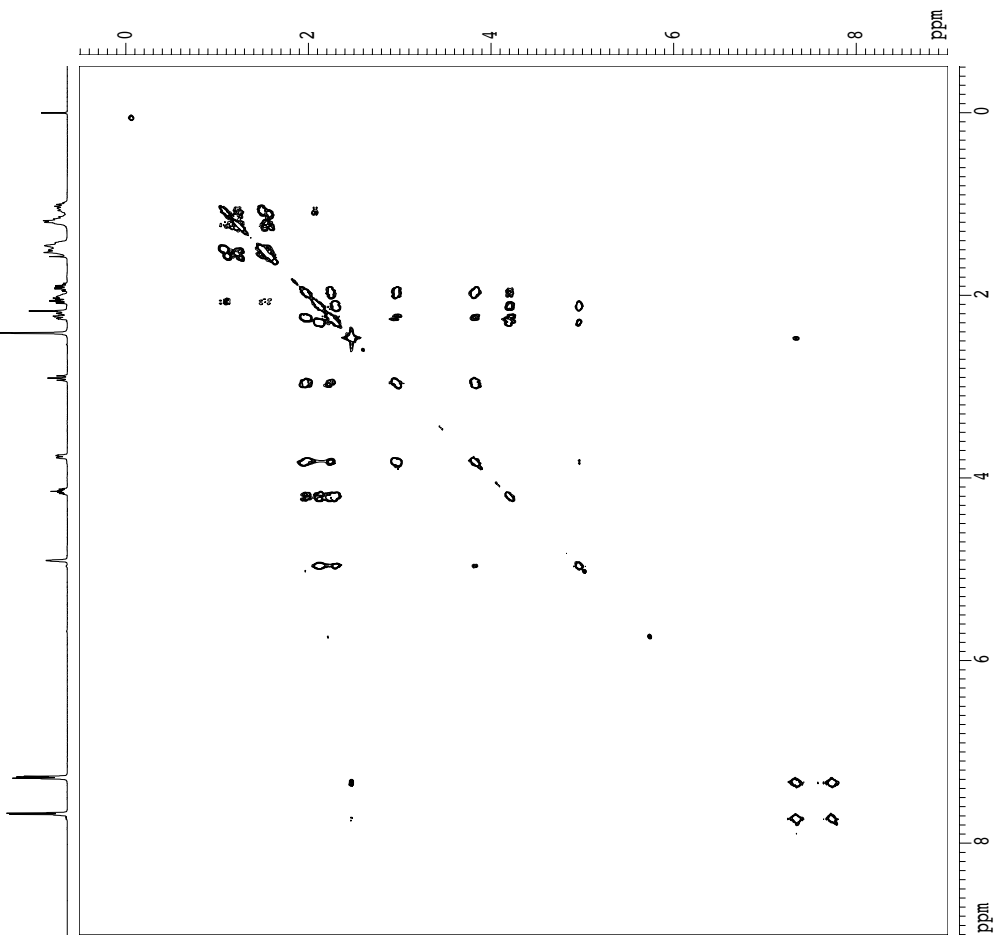
===== GRADIENT CHANNEL =====  
 GPRM1 sine,1.00  
 GPRM2 sine,1.00  
 GPCZ 0.00 %  
 GPV1 0.00 %  
 GPV2 0.00 %  
 GPV3 17.00 %  
 GPCX 0.00 %  
 GPCY 100.00 usec

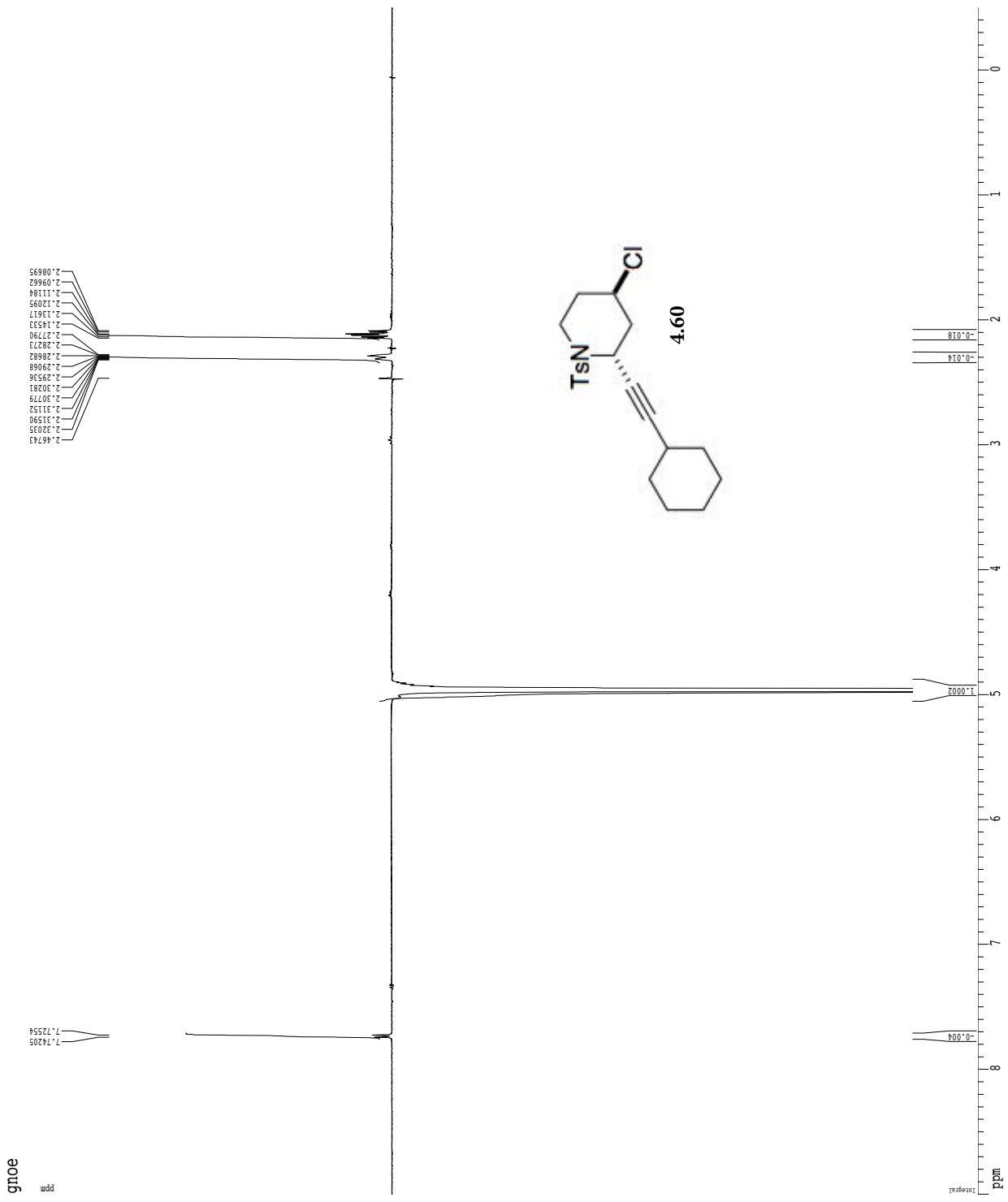
F1 - Acquisition parameters  
 NUC1 <sup>1</sup>H  
 P1 7.50 usec  
 PL 0.00 dB  
 SFO1 500.225015 MHz  
 FIDRES 3.912510 Hz  
 AQ 0.1278452 sec  
 RG 114  
 DW 62.400 usec  
 DE 298.0 usec  
 TE 298.0 usec  
 d0 0.40000300 sec  
 d1 1.40000000 sec  
 d13 0.40000300 sec  
 d16 0.40000000 sec  
 d10 0.40000480 sec

F2 - Processing parameters  
 SI 1024  
 SF 500.220000 MHz  
 WDM 1.00  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 1024  
 SF 500.220000 MHz  
 WDM 1.00  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 5.002 ppm  
 F2PO 5.002 ppm  
 F2PHI 45.569  
 F2PHI -254.47 Hz  
 F1PLO 9.002 ppm  
 F1PO 9.002 ppm  
 F1PHI 4503.14 Hz  
 F1PHI 0.509 ppm  
 F2PCOM 0.63407 ppm/cm  
 F2PCOM 317.17416 Hz/cm  
 F1PCOM 0.63407 ppm/cm  
 F1PCOM 317.17416 Hz/cm





gnoe

ppm

1.58811  
1.60165  
1.62479  
1.63512  
1.65965  
1.66207  
1.67120  
1.67604  
1.68566  
1.68823  
2.09533  
2.11132  
2.12065  
2.22276  
2.22385  
2.22481  
2.22550  
2.22595  
2.22616  
2.23003  
2.23155  
2.23564  
2.24222  
2.25393  
2.25834  
2.26155  
2.27709  
2.28262  
2.29006  
2.29482  
2.30165  
2.30779  
2.31164  
2.33616  
2.34373  
2.35084  
2.47076  
2.57964  
2.93944  
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2.96006  
2.96480  
2.98487  
2.98980  
3.80125  
3.81581  
3.81906  
3.82519  
3.84378  
4.95282  
4.96236

Current Data Parameters  
USER khewitt1  
NAME RMR-II-132-2  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20200813  
Time 10.45  
PROBHD 5 mm CPDCT 1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 128

SWH 8012.622 Hz  
FIDRES 0.122246 Hz  
AQ 4.089496 sec  
RG 312.50  
DM 62.400 usec  
DE 4.00 usec  
TE 288.0 K  
D1 1.0000000 sec  
d11 0.0000000 sec  
d16 0.0000000 sec  
d21 0.33375000 sec  
d22 0.16396699 sec  
F2 15.00 usec

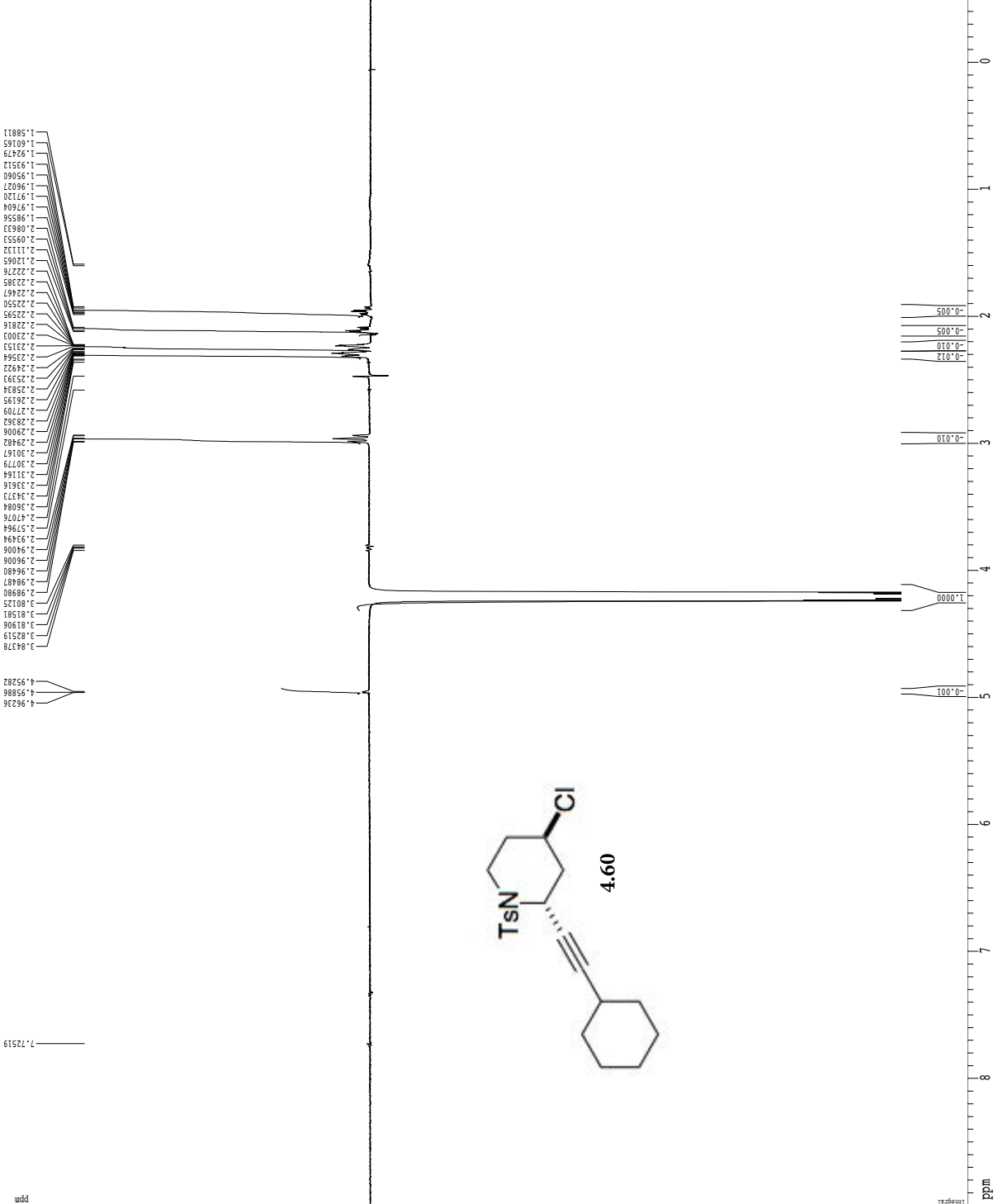
==== CHANNEL f1 =====  
NUC1 1H  
P1 7.50 usec  
P2 22.50 usec  
P3 20.00 usec  
P4 20.00 usec  
E29 40000.00 usec  
PL1 1.66 dB  
SFO1 500.222027 MHz  
GB19 6.00 dB  
SFO2 500.136157 MHz  
SFO3 500.136157 MHz  
SFO4 500.136157 MHz  
SFO5 500.136157 MHz  
SFO6 500.136157 MHz  
SFO7 500.136157 MHz  
SFO8 500.136157 MHz  
SFO9 500.136157 MHz  
SFO10 500.136157 MHz  
SFO11 500.136157 MHz  
SFO12 500.136157 MHz  
SFO13 500.136157 MHz  
SFO14 500.136157 MHz  
SFO15 500.136157 MHz  
SFO16 500.136157 MHz  
SFO17 500.136157 MHz  
SFO18 500.136157 MHz  
SFO19 500.136157 MHz  
SFO20 500.136157 MHz  
SFO21 500.136157 MHz  
SFO22 500.136157 MHz  
SFO23 500.136157 MHz  
SFO24 500.136157 MHz  
SFO25 500.136157 MHz  
SFO26 500.136157 MHz  
SFO27 500.136157 MHz  
SFO28 500.136157 MHz  
SFO29 500.136157 MHz  
SFO30 500.136157 MHz  
SFO31 500.136157 MHz  
SFO32 500.136157 MHz  
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SFO37 500.136157 MHz  
SFO38 500.136157 MHz  
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SFO43 500.136157 MHz  
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SFO45 500.136157 MHz  
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SFO81 500.136157 MHz  
SFO82 500.136157 MHz  
SFO83 500.136157 MHz  
SFO84 500.136157 MHz  
SFO85 500.136157 MHz  
SFO86 500.136157 MHz  
SFO87 500.136157 MHz  
SFO88 500.136157 MHz  
SFO89 500.136157 MHz  
SFO90 500.136157 MHz  
SFO91 500.136157 MHz  
SFO92 500.136157 MHz  
SFO93 500.136157 MHz  
SFO94 500.136157 MHz  
SFO95 500.136157 MHz  
SFO96 500.136157 MHz  
SFO97 500.136157 MHz  
SFO98 500.136157 MHz  
SFO99 500.136157 MHz  
SFO100 500.136157 MHz

==== GRADIENT CHANNEL =====  
G1 size 100  
G2 size 100  
G3 size 100  
G4 size 100  
G5 size 100  
G6 size 100  
G7 size 100  
G8 size 100  
G9 size 100  
G10 size 100  
G11 size 100  
G12 size 100  
G13 size 100  
G14 size 100  
G15 size 100  
G16 size 100  
G17 size 100  
G18 size 100  
G19 size 100  
G20 size 100  
G21 size 100  
G22 size 100  
G23 size 100  
G24 size 100  
G25 size 100  
G26 size 100  
G27 size 100  
G28 size 100  
G29 size 100  
G30 size 100  
G31 size 100  
G32 size 100  
G33 size 100  
G34 size 100  
G35 size 100  
G36 size 100  
G37 size 100  
G38 size 100  
G39 size 100  
G40 size 100  
G41 size 100  
G42 size 100  
G43 size 100  
G44 size 100  
G45 size 100  
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G49 size 100  
G50 size 100  
G51 size 100  
G52 size 100  
G53 size 100  
G54 size 100  
G55 size 100  
G56 size 100  
G57 size 100  
G58 size 100  
G59 size 100  
G60 size 100  
G61 size 100  
G62 size 100  
G63 size 100  
G64 size 100  
G65 size 100  
G66 size 100  
G67 size 100  
G68 size 100  
G69 size 100  
G70 size 100  
G71 size 100  
G72 size 100  
G73 size 100  
G74 size 100  
G75 size 100  
G76 size 100  
G77 size 100  
G78 size 100  
G79 size 100  
G80 size 100  
G81 size 100  
G82 size 100  
G83 size 100  
G84 size 100  
G85 size 100  
G86 size 100  
G87 size 100  
G88 size 100  
G89 size 100  
G90 size 100  
G91 size 100  
G92 size 100  
G93 size 100  
G94 size 100  
G95 size 100  
G96 size 100  
G97 size 100  
G98 size 100  
G99 size 100  
G100 size 100

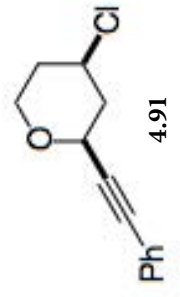
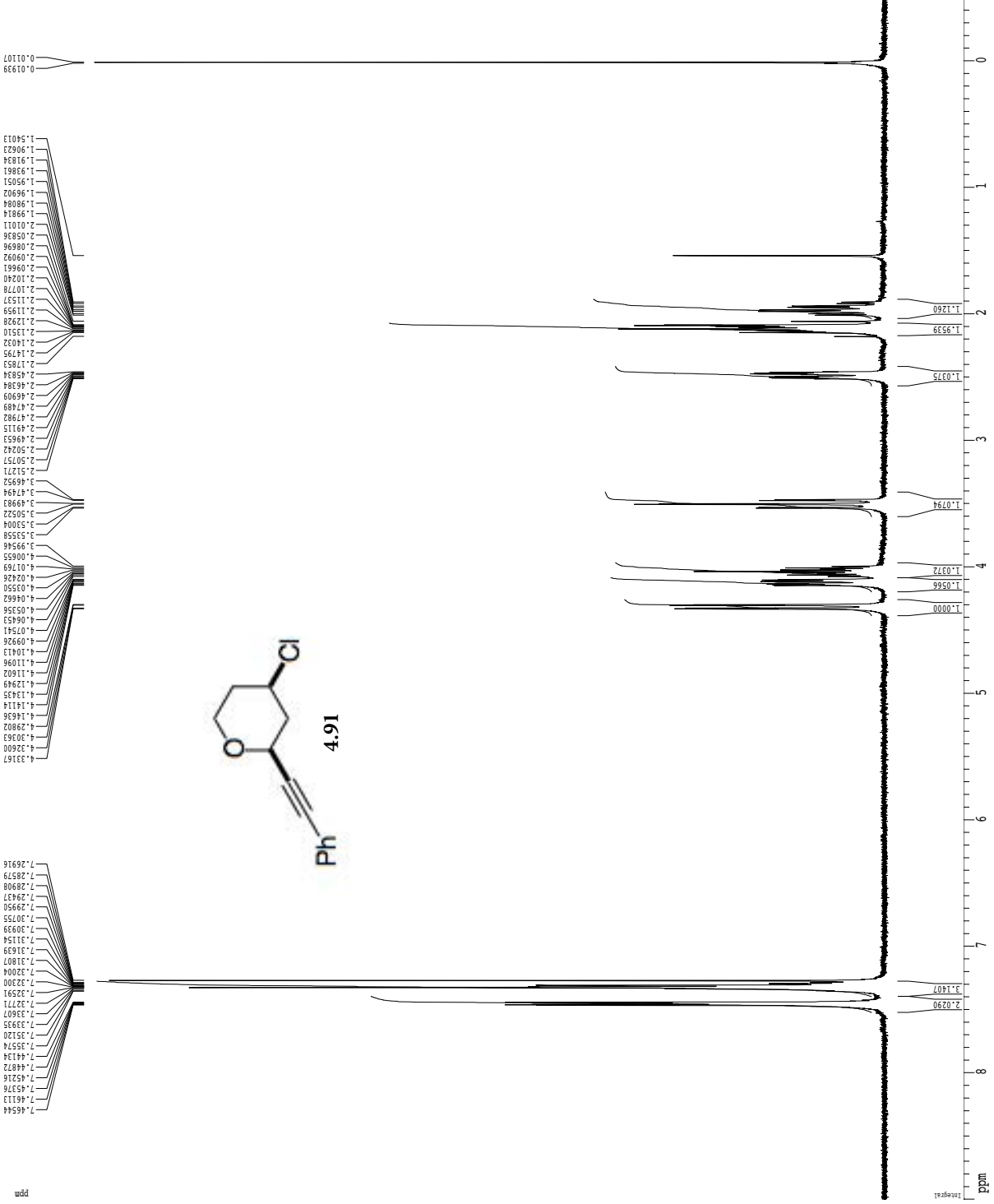
F2 - Processing parameters  
SI 65536  
SF 500.2220000 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

ID NMR plot parameters  
CX 22.88 cm  
CT 50.00 cm  
SI 65536  
SF 500.2220000 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

==== CHANNEL f1 =====  
NUC1 1H  
P1 7.50 usec  
P2 22.50 usec  
P3 20.00 usec  
P4 20.00 usec  
E29 40000.00 usec  
PL1 1.66 dB  
SFO1 500.222027 MHz  
GB19 6.00 dB  
SFO2 500.136157 MHz  
SFO3 500.136157 MHz  
SFO4 500.136157 MHz  
SFO5 500.136157 MHz  
SFO6 500.136157 MHz  
SFO7 500.136157 MHz  
SFO8 500.136157 MHz  
SFO9 500.136157 MHz  
SFO10 500.136157 MHz  
SFO11 500.136157 MHz  
SFO12 500.136157 MHz  
SFO13 500.136157 MHz  
SFO14 500.136157 MHz  
SFO15 500.136157 MHz  
SFO16 500.136157 MHz  
SFO17 500.136157 MHz  
SFO18 500.136157 MHz  
SFO19 500.136157 MHz  
SFO20 500.136157 MHz  
SFO21 500.136157 MHz  
SFO22 500.136157 MHz  
SFO23 500.136157 MHz  
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SFO25 500.136157 MHz  
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SFO62 500.136157 MHz  
SFO63 500.136157 MHz  
SFO64 500.136157 MHz  
SFO65 500.136157 MHz  
SFO66 500.136157 MHz  
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SFO69 500.136157 MHz  
SFO70 500.136157 MHz  
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SFO80 500.136157 MHz  
SFO81 500.136157 MHz  
SFO82 500.136157 MHz  
SFO83 500.136157 MHz  
SFO84 500.136157 MHz  
SFO85 500.136157 MHz  
SFO86 500.136157 MHz  
SFO87 500.136157 MHz  
SFO88 500.136157 MHz  
SFO89 500.136157 MHz  
SFO90 500.136157 MHz  
SFO91 500.136157 MHz  
SFO92 500.136157 MHz  
SFO93 500.136157 MHz  
SFO94 500.136157 MHz  
SFO95 500.136157 MHz  
SFO96 500.136157 MHz  
SFO97 500.136157 MHz  
SFO98 500.136157 MHz  
SFO99 500.136157 MHz  
SFO100 500.136157 MHz



1H spectrum



Current Data Parameters  
 USER KMH-11-284-check  
 NAME KMH-11-284-check  
 EXNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20191129  
 Time 15:22  
 PROBN 400000  
 PROCNO 5  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 DS 9  
 SFO 6410.256 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.399279 sec  
 RG 78.000  
 DE 4.50 usec  
 TE 297.2 K  
 D0 0.100000 sec  
 MCHRES 0.000000 sec  
 MSRES 0.000000 sec  
 ACQRES 0.0050000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 0.00 dB  
 SFO1 400.1328009 MHz

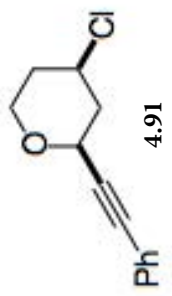
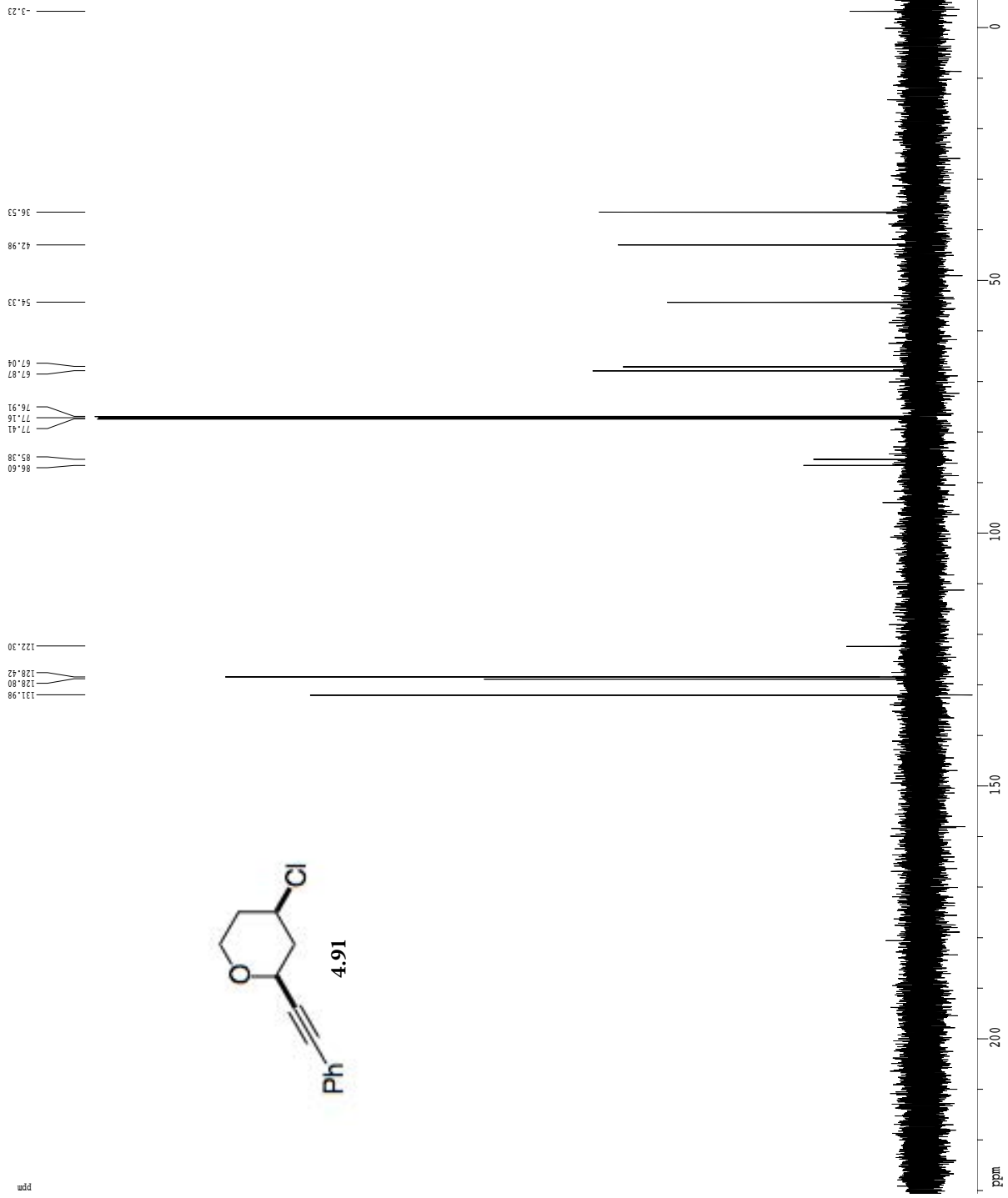
F2 - Processing Parameters  
 SI 65536  
 SF 400.1300175 MHz  
 WDW no  
 GB 0.00 Hz  
 PC 2.00

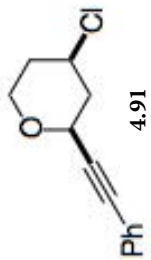
D0 NMR P1/QF Parameters  
 CY 22.80 cm  
 CV 15.00 cm  
 FIP 9.000 ppm  
 FL 3600.17 Hz  
 F2 2000.00 Hz  
 F2 2000.00 Hz  
 PPMCH 0.41667 ppm/cm  
 HZCH 166.72084 Hz/cm

# Z-restored spin-echo 13C spectrum with 1H decoupling

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```
Current Data Parameters
=====
USER      Kkwatt1
NAME      KM-11-28-1
PROCNO    1
PROCDS    1
=====
F2 - Acquisition Parameters
=====
Date_     20130926
Time      11:40
INSTRUM   spect
PROBHD    5 mm cryo500
PULPROG   zgpg30
F2 - Processing parameters
=====
SOLVENT   CDCl3
TD         65536
SI         16
SF         125.760462 MHz
WDW        no
SSB        0
GB         0
PC         2.00
=====
ID: NMR pulse parameters
=====
CX      22.80 cm
CY      15.65 cm
FI      2.000000 ppm
F2P     -10.487 ppm
F2      -1292.96 Hz
=====
===== CHANNEL f1 =====
NUC1      13C
P1        16.00 usec
PL1       0.00 dB
P2        2000.00 usec
PL2       1.60 dB
P3        500.00 usec
PL3       120.00 dB
SFO1      125.760462 MHz
SF2       2.70 dB
SFO2      125.760462 MHz
SFO3      125.760462 MHz
SFO4      125.760462 MHz
SFO5      125.760462 MHz
SFO6      125.760462 MHz
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       13C
P4        100.00 usec
PL4       1.60 dB
P5        500.00 usec
PL5       120.00 dB
SFO7      500.222011 MHz
===== GRADIENT CHANNEL =====
GPMAX1    3.00 G
SFO8      125.760462 MHz
SFO9      125.760462 MHz
SFO10     125.760462 MHz
SFO11     125.760462 MHz
SFO12     125.760462 MHz
SFO13     125.760462 MHz
SFO14     125.760462 MHz
=====
===== CHANNEL f3 =====
CPDPRG3   waltz16
NUC3       13C
P6        100.00 usec
PL6       1.60 dB
P7        500.00 usec
PL7       120.00 dB
SFO15     500.222011 MHz
===== GRADIENT CHANNEL =====
GPMAX1    3.00 G
SFO16     125.760462 MHz
SFO17     125.760462 MHz
SFO18     125.760462 MHz
SFO19     125.760462 MHz
SFO20     125.760462 MHz
SFO21     125.760462 MHz
SFO22     125.760462 MHz
SFO23     125.760462 MHz
SFO24     125.760462 MHz
SFO25     125.760462 MHz
SFO26     125.760462 MHz
SFO27     125.760462 MHz
SFO28     125.760462 MHz
SFO29     125.760462 MHz
SFO30     125.760462 MHz
SFO31     125.760462 MHz
SFO32     125.760462 MHz
SFO33     125.760462 MHz
SFO34     125.760462 MHz
SFO35     125.760462 MHz
SFO36     125.760462 MHz
SFO37     125.760462 MHz
SFO38     125.760462 MHz
SFO39     125.760462 MHz
SFO40     125.760462 MHz
SFO41     125.760462 MHz
SFO42     125.760462 MHz
SFO43     125.760462 MHz
SFO44     125.760462 MHz
SFO45     125.760462 MHz
SFO46     125.760462 MHz
SFO47     125.760462 MHz
SFO48     125.760462 MHz
SFO49     125.760462 MHz
SFO50     125.760462 MHz
SFO51     125.760462 MHz
SFO52     125.760462 MHz
SFO53     125.760462 MHz
SFO54     125.760462 MHz
SFO55     125.760462 MHz
SFO56     125.760462 MHz
SFO57     125.760462 MHz
SFO58     125.760462 MHz
SFO59     125.760462 MHz
SFO60     125.760462 MHz
SFO61     125.760462 MHz
SFO62     125.760462 MHz
SFO63     125.760462 MHz
SFO64     125.760462 MHz
SFO65     125.760462 MHz
SFO66     125.760462 MHz
SFO67     125.760462 MHz
SFO68     125.760462 MHz
SFO69     125.760462 MHz
SFO70     125.760462 MHz
SFO71     125.760462 MHz
SFO72     125.760462 MHz
SFO73     125.760462 MHz
SFO74     125.760462 MHz
SFO75     125.760462 MHz
SFO76     125.760462 MHz
SFO77     125.760462 MHz
SFO78     125.760462 MHz
SFO79     125.760462 MHz
SFO80     125.760462 MHz
SFO81     125.760462 MHz
SFO82     125.760462 MHz
SFO83     125.760462 MHz
SFO84     125.760462 MHz
SFO85     125.760462 MHz
SFO86     125.760462 MHz
SFO87     125.760462 MHz
SFO88     125.760462 MHz
SFO89     125.760462 MHz
SFO90     125.760462 MHz
SFO91     125.760462 MHz
SFO92     125.760462 MHz
SFO93     125.760462 MHz
SFO94     125.760462 MHz
SFO95     125.760462 MHz
SFO96     125.760462 MHz
SFO97     125.760462 MHz
SFO98     125.760462 MHz
SFO99     125.760462 MHz
SFO100    125.760462 MHz
=====
```





gcosy60

```

Current Data Parameters
USER      Kheshti1
NAME      KAH-II-194-COSY
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20190930
Time      17.55
INSTRUM   spect
PROBHD     5 mm hmc
PULPROG   zgpg30
TD         2048
SOLVENT   CDCl3
NS         1
DS         16
AQ         8013.85 Hz
FIDRES    0.1278452 Hz
RG         5160.6
DM         62.400 usec
DE         298.0 usec
TE         298.0 usec
d0         0.0000300 sec
d1         1.0000000 sec
d13        0.0000300 sec
d16        0.0002000 sec
d10        0.0001480 sec

===== CHANNEL f1 =====
NUC1       1H
P1         12.00 usec
PL         0.00 dB
SFO1       498.8534919 MHz

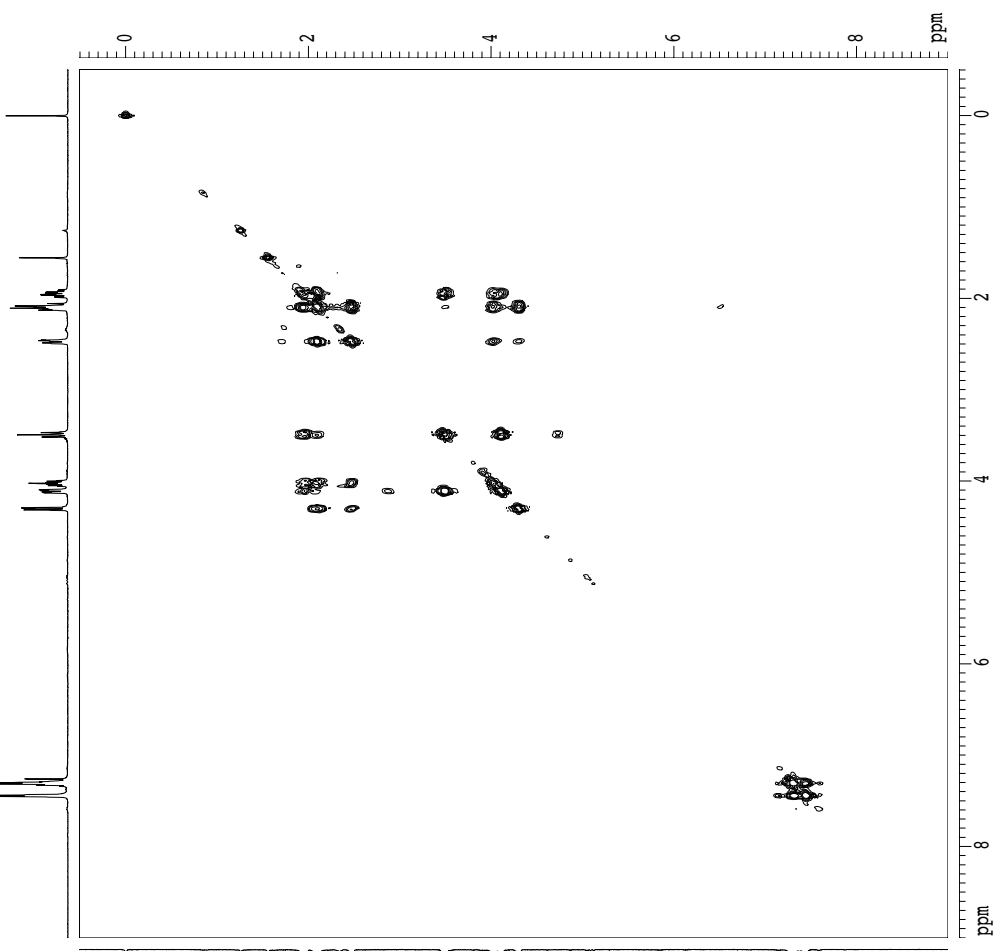
===== GRADIENT CHANNEL =====
GPRM1     SINE.100
GPRM2     SINE.100
GPZ1      0.00 %
GPZ2      0.00 %
GPT1      0.00 %
GPT2      0.00 %
GPT3      17.00 %
GPT4      17.00 %
PT6        1000.00 usec

F1 - Acquisition parameters
NUC2       13C
P2         12.00 usec
PL         0.00 dB
SFO2       101.254 MHz
SFO1SFO2   498.8535 MHz
FIDRES    31.300079 Hz
SF         16.062 ppm
PnMODE     GP

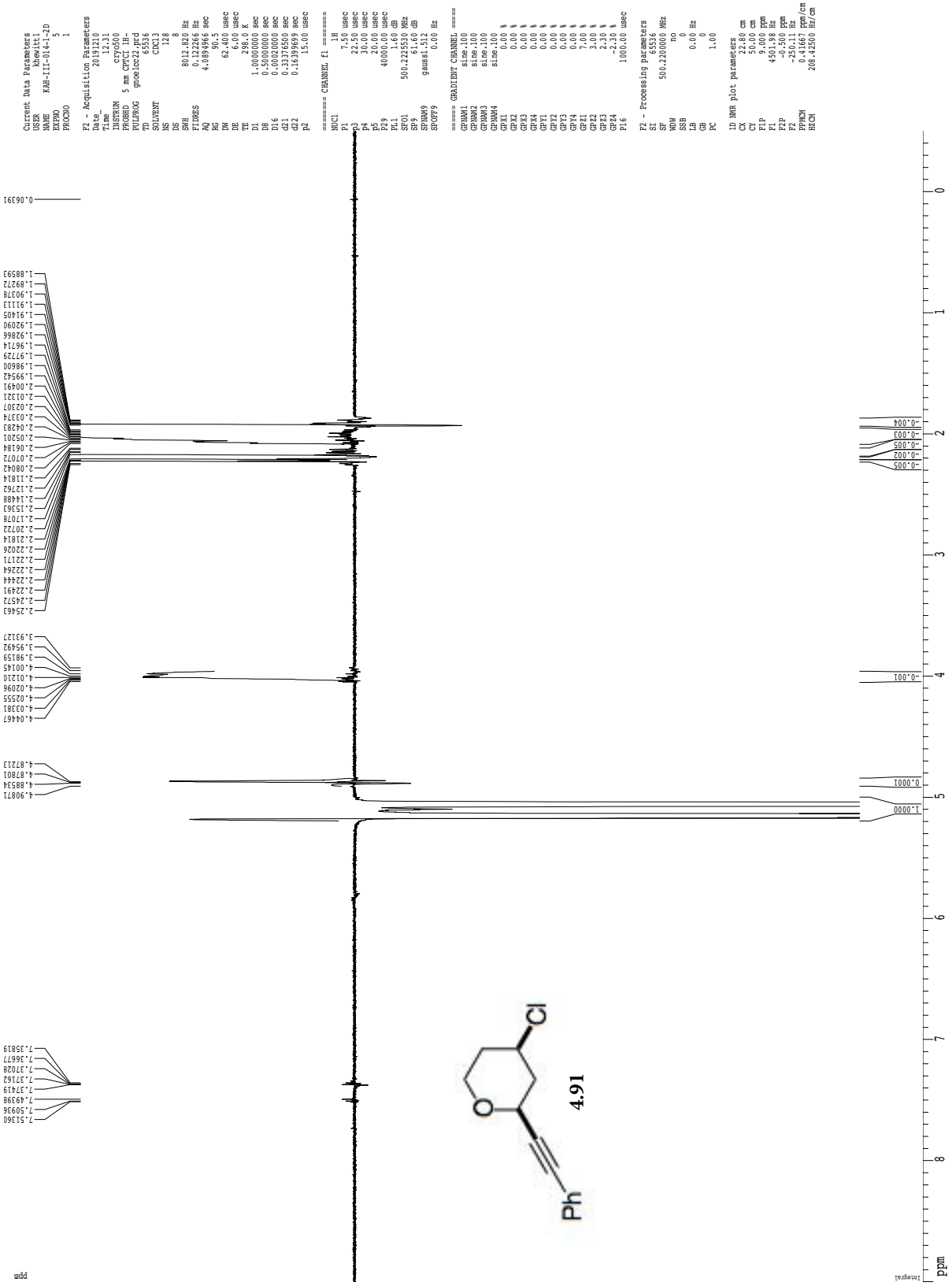
F2 - Processing parameters
SI         1
SF         498.850268 MHz
WDW        SINE
SSB        0
GB         0.00 Hz
PC         1.00

F1 - Processing parameters
SI         1024
SF         498.850268 MHz
WDW        SINE
SSB        0
GB         0.00 Hz
PC         1.00

2D NMR plot parameters
CX2        15.00 cm
F2P1O     5.001 ppm
F2P1C     44.0515 Hz
F2P1R     -251.74 Hz
F1P1O     5.001 ppm
F1P1C     4490.22 Hz
F1P1R     -251.74 Hz
F2P1MCM   0.63372 ppm/cm
F2P1RCH   316.13080 Hz/cm
F1P1MCM   0.63372 ppm/cm
F1P1RCH   316.13080 Hz/cm
  
```



gnoe





gnoe

ppm

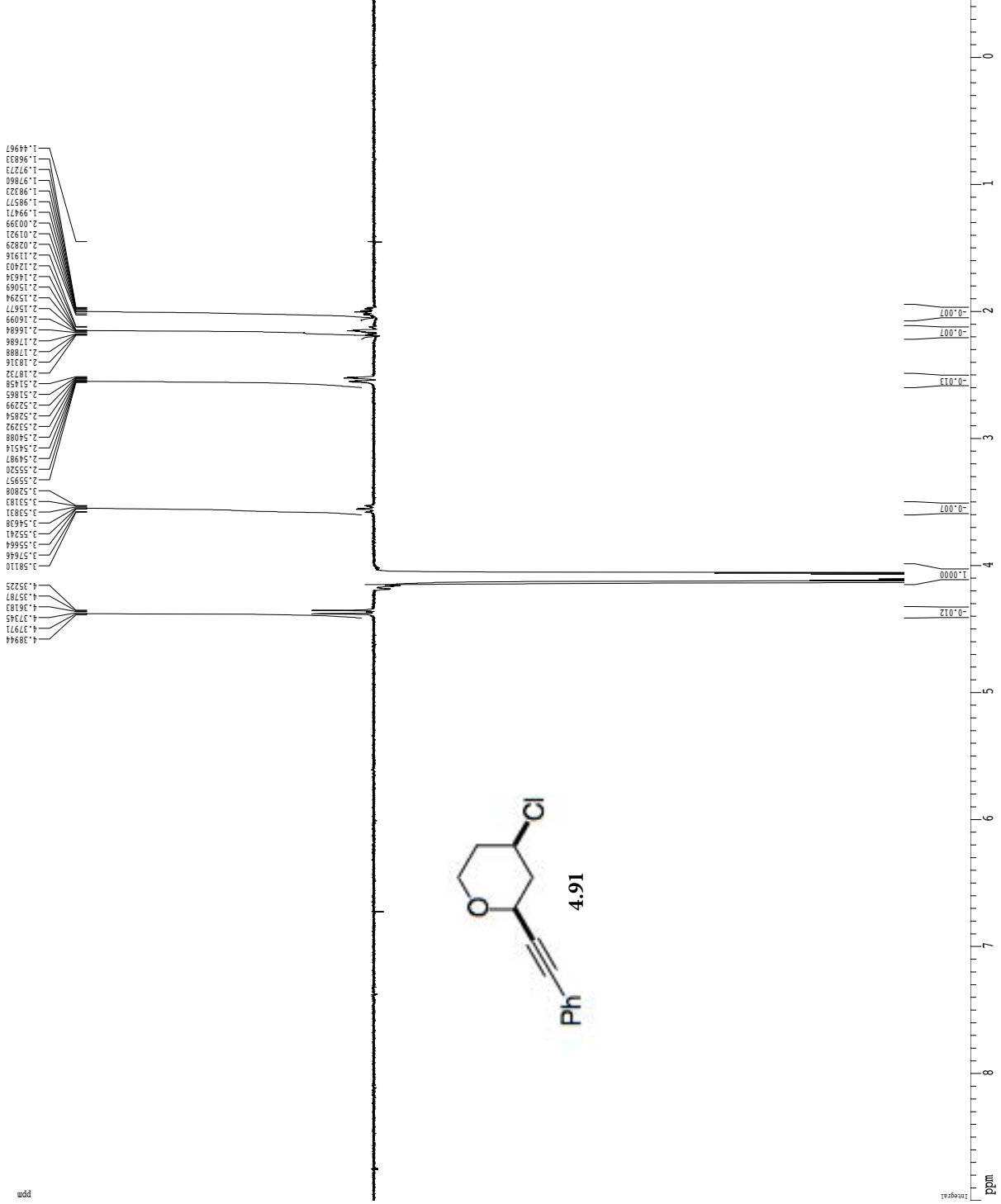
4.38944  
4.37971  
4.37493  
4.36283  
4.35787  
4.35225  
3.58110  
3.57646  
3.55664  
3.55241  
3.54638  
3.53931  
3.53808  
2.55957  
2.55520  
2.54987  
2.54524  
2.54088  
2.53292  
2.52584  
2.51985  
2.51598  
2.51458  
2.18732  
2.18316  
2.17888  
2.16864  
2.16099  
2.15677  
2.15294  
2.14969  
2.14639  
2.14003  
2.11916  
2.02829  
2.02192  
1.99595  
1.99471  
1.98577  
1.98233  
1.97860  
1.97611  
1.96833  
1.44496

Current Data Parameters  
USER khewitt1  
NAME KM-II-284-2  
EXPNO 4  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20191129  
Time 17:37  
PROBHD 5 mm CPDQ 1H  
PULPROG zgpg30  
PROGNAME gnoe.cp22.prd  
TD 65536  
SOLVENT CDCl3  
DS 128  
SS 8  
SMH 8012.822 Hz  
FIDRES 0.122266 Hz  
AQ 4.1089496 sec  
RG 62.400  
DE 6.00 usec  
TE 288.0 K  
D1 1.0000000 sec  
SFO1 500.2210449 MHz  
D16 0.0020000 sec  
dZ1 0.33376500 sec  
dZ2 0.16398659 sec  
PZ 15.00 usec

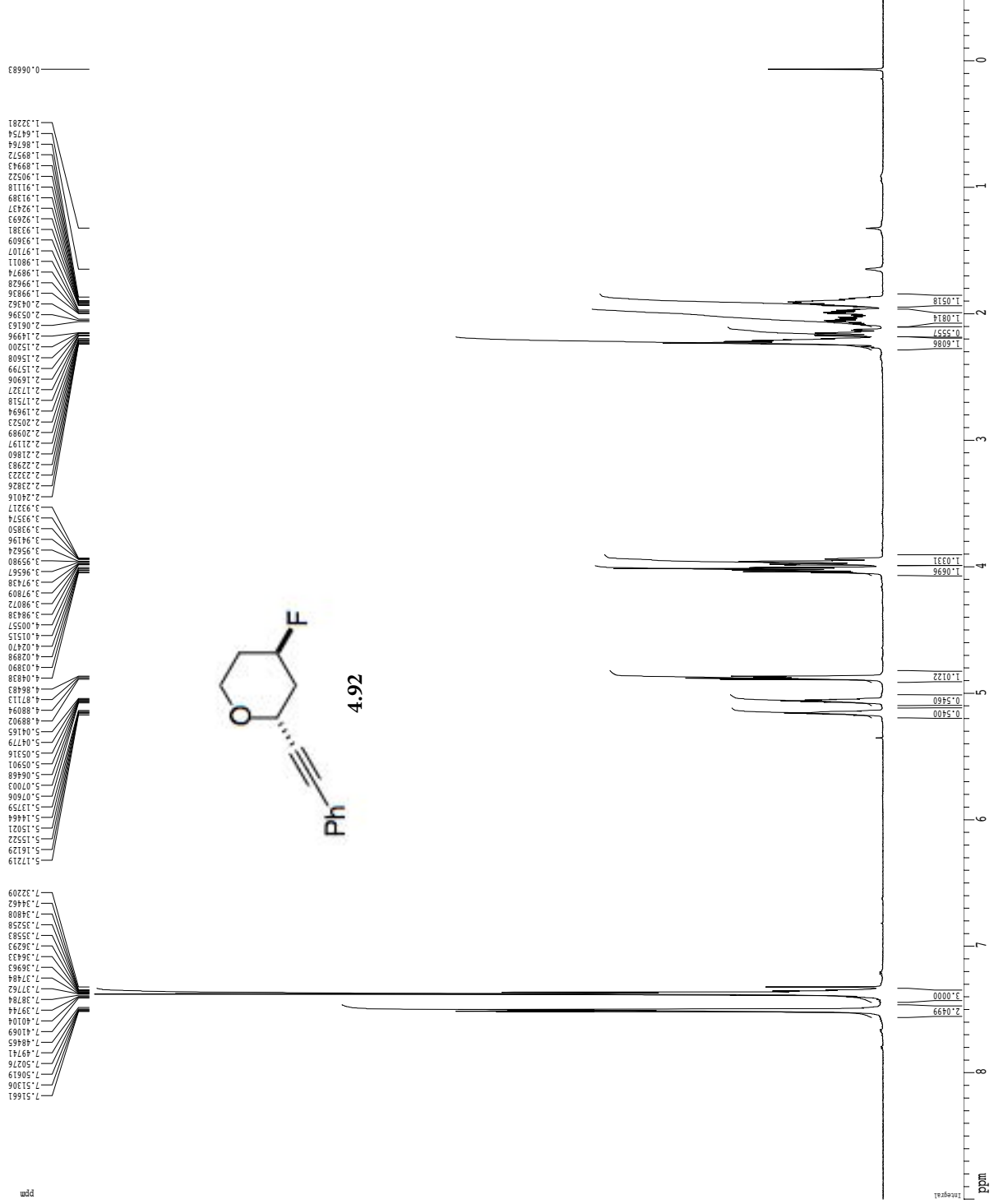
==== CHANNEL f1 ====  
NUC1 1H  
P1 7.50 usec  
P2 22.50 usec  
P3 21.00 usec  
P4 21.00 usec  
P5 21.00 usec  
PZ9 40000.00 usec  
PL1 1.66 dB  
SFO1 500.2210449 MHz  
SFO2 60.000000 MHz  
SFO3 99.626039 MHz  
SFO4 0.00 Hz  
SFO5 0.00 Hz  
SFO6 0.00 Hz  
SFO7 0.00 Hz  
SFO8 0.00 Hz  
SFO9 0.00 Hz  
SFOF9 0.00 Hz

==== GRABEDIT CHANNEL ====  
SFO1 100.626039 MHz  
SFO2 100.626039 MHz  
SFO3 100.626039 MHz  
SFO4 100.626039 MHz  
SFO5 100.626039 MHz  
SFO6 100.626039 MHz  
SFO7 100.626039 MHz  
SFO8 100.626039 MHz  
SFO9 100.626039 MHz  
SFOF9 100.626039 MHz

F2 - Processing parameters  
SI 65536  
SF 500.2210449 MHz  
WDW EM  
SSB 0  
GB 0  
LB 0.00 Hz  
PC 1.00  
ID NMR plot parameters  
CX 22.88 cm  
CY 50.00 cm  
CZ 40.00 cm  
EP 480.000000 ppm  
FP 480.000000 ppm  
FZP -0.500000 ppm  
FZ -250.11 Hz  
PRGCM 0.41667 ppm/cm  
HCN 208.42500 Hz/cm



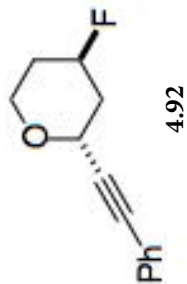
1H spectrum



Current Data Parameters  
 USER KKH-II-014-1-D  
 NAME KKH-II-014-1-D  
 EXNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20191209  
 Time 16:45  
 PROBNM 0727140  
 PROCNO 5  
 PULPROG zgpg30  
 TD 48074  
 SOLVENT CDCl3  
 DS 9  
 SFO 8012.820 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.3398677 sec  
 RG 62.400 usec  
 DE 6.00 usec  
 TE 298.2 K  
 O1 0.1000000 sec  
 MCHRES 0.1000000 sec  
 MCOREK 0.01500000 sec  
 ===== CHANNEL f1 =====  
 NUC1 13C  
 P1 1.50 usec  
 PL1 1.66 dB  
 SFO1 500.2235015 MHz  
 F2 - Processing Parameters  
 SI 65536  
 SF 500.2200000 MHz  
 WDW no  
 GB 0.00 Hz  
 CB 0  
 PC 1.00  
 ID Wdr Plot Parameters  
 CY 22.80 cm  
 CX 15.00 cm  
 FIP 9.000 ppm  
 F1 4301.308 Hz  
 F2 -250.11 Hz  
 FREQM 0.41667 ppm/cm  
 HZCM 206.44500 Hz/cm

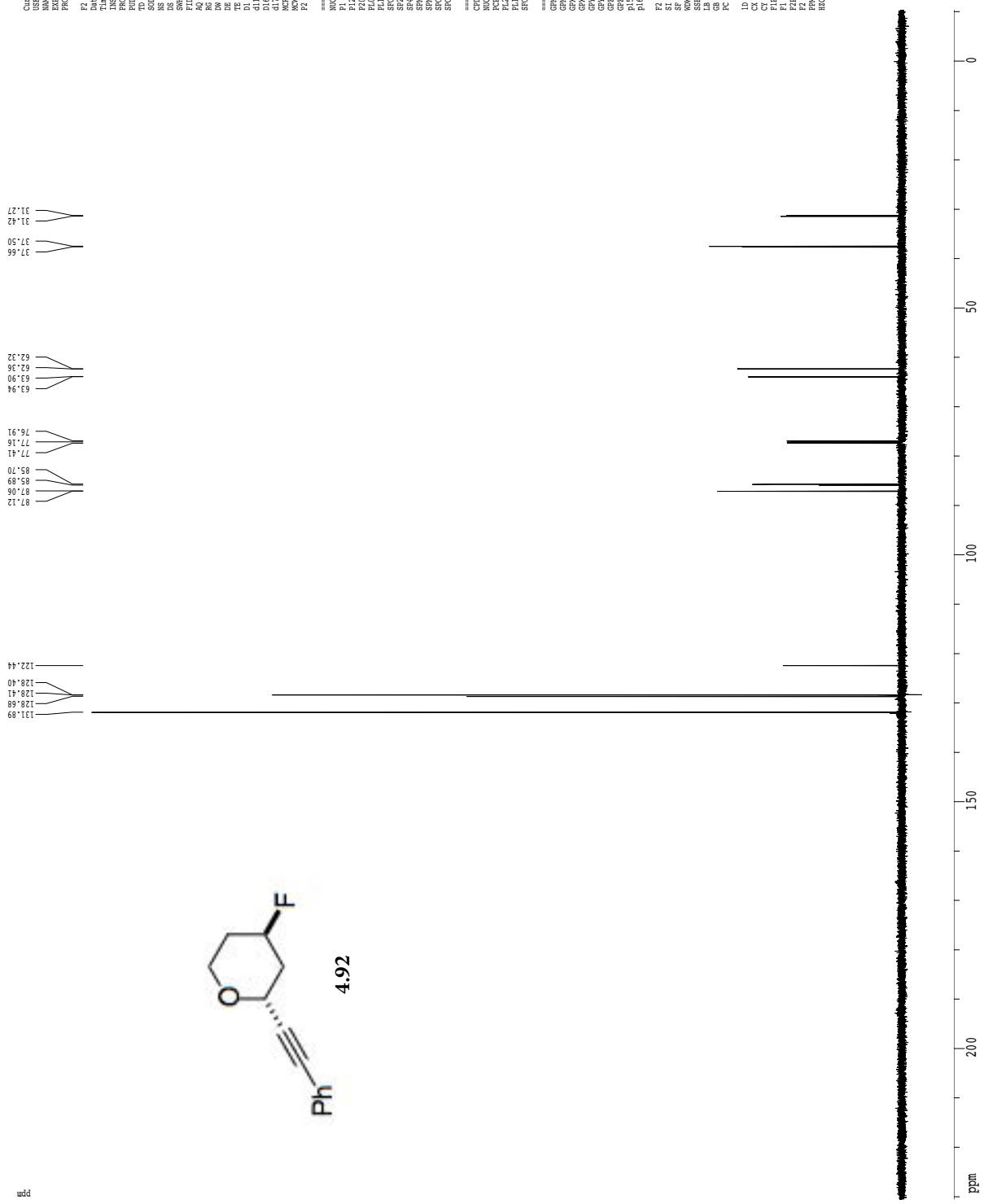
Z-restored spin-echo 13C spectrum with 1H decoupling

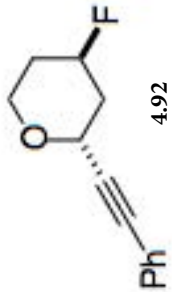
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Current Data Parameters
=====
USER           Kzwattl
NAME           KM-111-11-1-2D
PROCNO         1
PROG           1
F2 - Acquisition Parameters
=====
Date_         20181015
Time_         11:01
INSTRUM       cryo500
PULPROG       zgpg30
PROBHD        5 mm cryo500
PDETECTORS    rd
AQ            4.65536 sec
RG            6538
SOLVENT       CDCl3
NS            16
DS            4
SWH           30803.801 Hz
FIDRES       0.462208 Hz
AQ           4.65536 sec
RG           6538
RG            6538 sec
DQ           16.500 usec
DE           6.00 usec
D1           0.25000000 sec
D11          0.03000000 sec
D12          0.03000000 sec
D13          0.03000000 sec
D14          0.03000000 sec
D15          0.03000000 sec
D16          0.03000000 sec
DELTA        0.03500000 sec
MCOREST      0.03500000 sec
F2           31.0 usec
=====
===== CHANNEL f1 =====
NUC1          13C
P1           15.00 usec
PL1          0.00 dB
PL2          2000.00 usec
P20          500.00 usec
P21          120.00 dB
P22          120.00 dB
SFO1         125.7642448 MHz
SFO2         2.70 GHz
SFO3         2.70 GHz
SFO4         Ccp66,0.5,20.1
SFO5         0.00 Hz
SFOF2        0.00 Hz
SFOF4        0.00 Hz
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          13C
P2           100.00 usec
PL2          1.60 dB
PL12         500.2120011 MHz
SFO2         125.7642448 MHz
===== GRADIENT CHANNEL =====
GPRM1        SFR1100
SFR1100      3.00 usec
SFR1100      6.00 V
GPR2         0.00 V
GPR3         0.00 V
GPR4         0.00 V
GPR5         30.00 V
GPR6         50.00 V
GPR7         100.00 usec
GPR8         100.00 usec
F2 - Processing parameters
=====
SI           125.7642448 MHz
SF           0.00 Hz
SD           0.00 Hz
SSB          0.00 Hz
GB           0.00 Hz
PC           2.00
=====
ID_NMR proc parameters
CX           22.80 cm
CY           15.95 cm
CZ           15.95 cm
F1           2.0016498 ppm
F2           -10.487 ppm
F3           -104.87 ppm
GPRM1       125.7642448 MHz
GPRM2       125.7642448 MHz
=====
  
```





4.92

gcosy60

```

Current Data Parameters
=====
USER          khewit1
NAME          KAH-III-014-1-2D
PROCNO       1
PRGNO        1

F2 - Acquisition Parameters
=====
Date_         20191209
Time         16:48
EXPNO        2
PROCNO       5
PROBHD       5 mm CRY1 1H-
PULPROG      zgpg30
TD           2048
SOLVENT      CDCl3
NS           16
DS           4
SWH          4734.849 Hz
FIDRES      2.311938 Hz
AQ          0.2163188 sec
RG          105.601
WDW          EM
SSB          0
LB           6.00 usec
TE           298.0 K
DQ           0.0000300 sec
DE           1.0000000 sec
DI           0.0000000 sec
DL           0.0000000 sec
E2           0.0000000 sec
AQ2          0.0000000 sec
DQ2          0.0000000 sec
===== CHANNEL f1 =====
NUC1          1H
P1           7.00 usec
PL1          1.60 dB
SFO1         500.221259 MHz

===== GRADIENT CHANNEL =====
GRAMI1       Sine-100
GXM1Z        Sine-100
GPX2         0.00 %
GPT1         0.00 %
GPT2         0.00 %
GPT3         0.00 %
GPT4         17.00 %
GPT5         17.00 %
GPT6         17.00 %
PL6          1000.00 usec

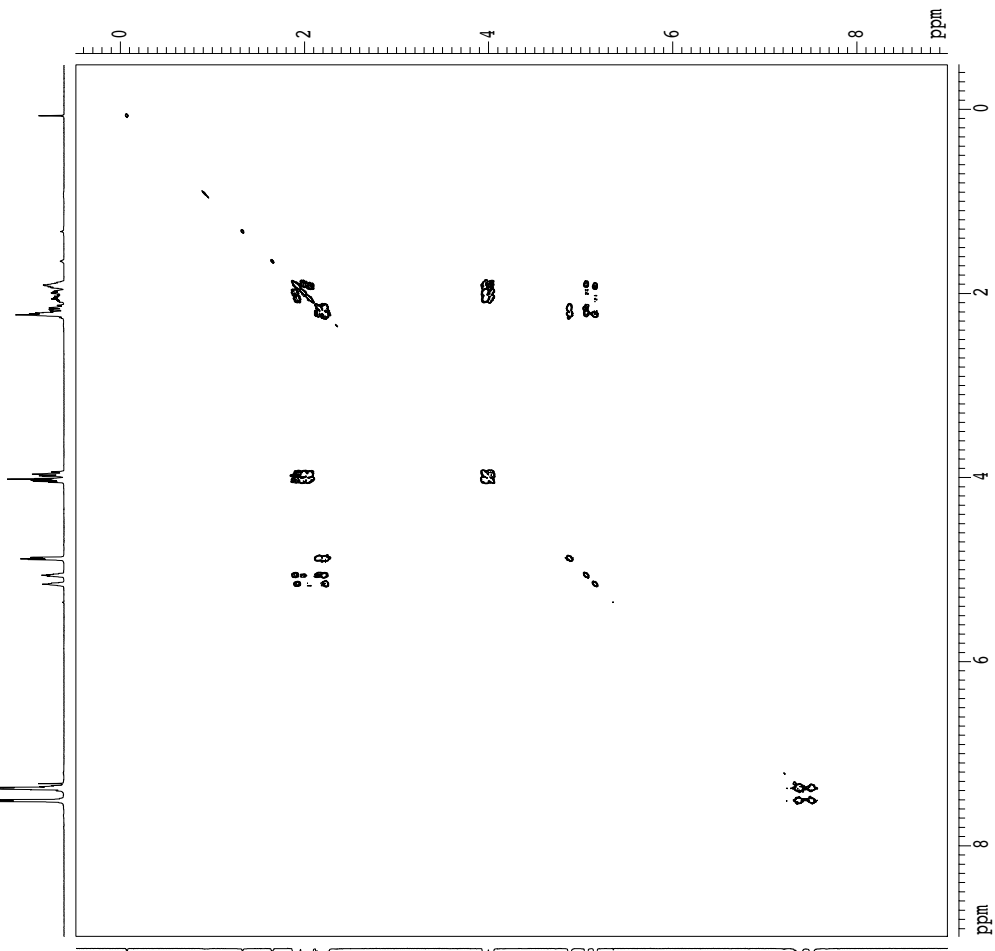
F1 - Acquisition Parameters
=====
NUC1          13C
P1           5.00 usec
PL1          1.90 dB
SFO1         125.761155 MHz

===== GRADIENT CHANNEL =====
GRAMI1       Sine-100
GXM1Z        Sine-100
GPX2         0.00 %
GPT1         0.00 %
GPT2         0.00 %
GPT3         0.00 %
GPT4         17.00 %
GPT5         17.00 %
GPT6         17.00 %
PL6          1000.00 usec

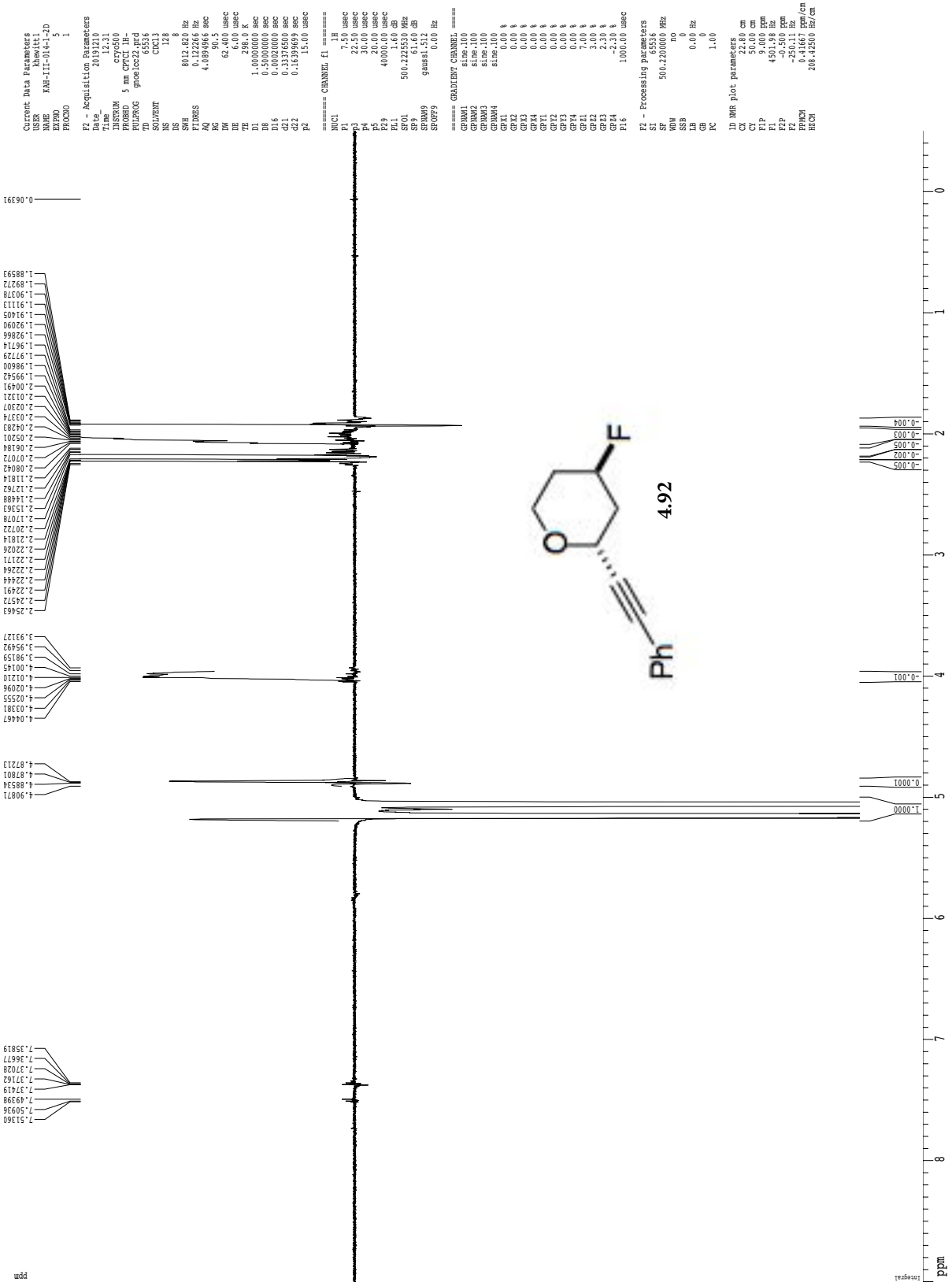
F2 - Processing parameters
=====
SI           1024
SF           500.2200000 MHz
WDW          EM
SSB          0
LB           0.00 Hz
GB           0
PC           1.00

F1 - Processing parameters
=====
SI           1024
SF           500.2200000 MHz
WDW          EM
SSB          0
LB           0.00 Hz
GB           0

2D NMR plot parameters
=====
CX2          15.00 cm
F2FLO        8.583 ppm
F2AQ         4493.36 Hz
F2NUC        13C
F2P1         -0.483 ppm
F2P2         -241.49 Hz
F2FPO        8.583 ppm
F2FLO        4493.36 Hz
F2P1         -0.483 ppm
F2P2         -241.49 Hz
F2FPO        8.583 ppm
F2FPCN       0.63104 ppm/cm
F2FPCN       315.65659 Hz/cm
F2FPCN       0.63104 ppm/cm
F2FPCN       315.65659 Hz/cm
  
```

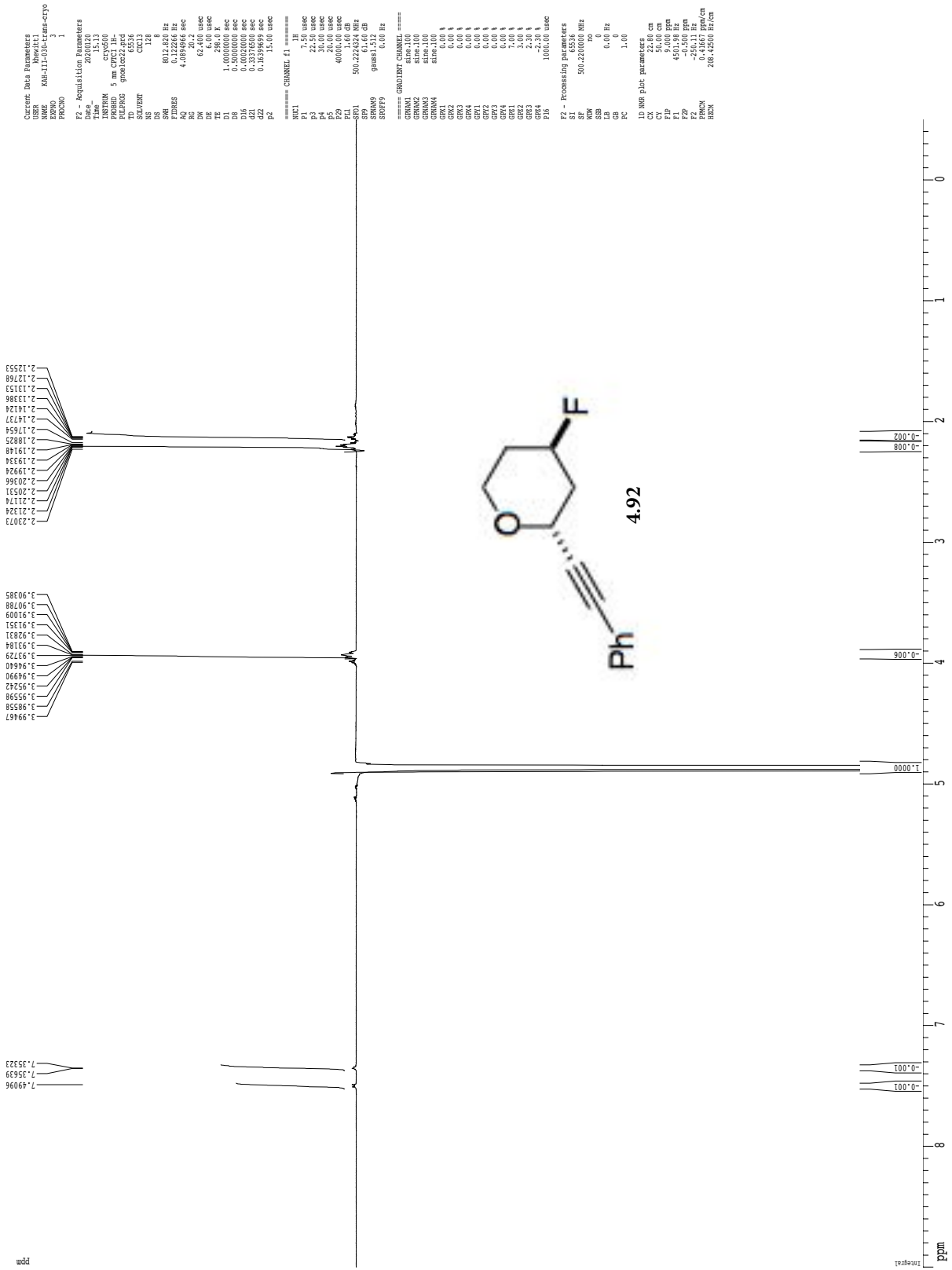


gnoe



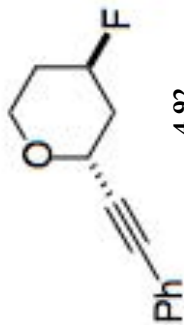
gnoe

ppm



f19.c

8



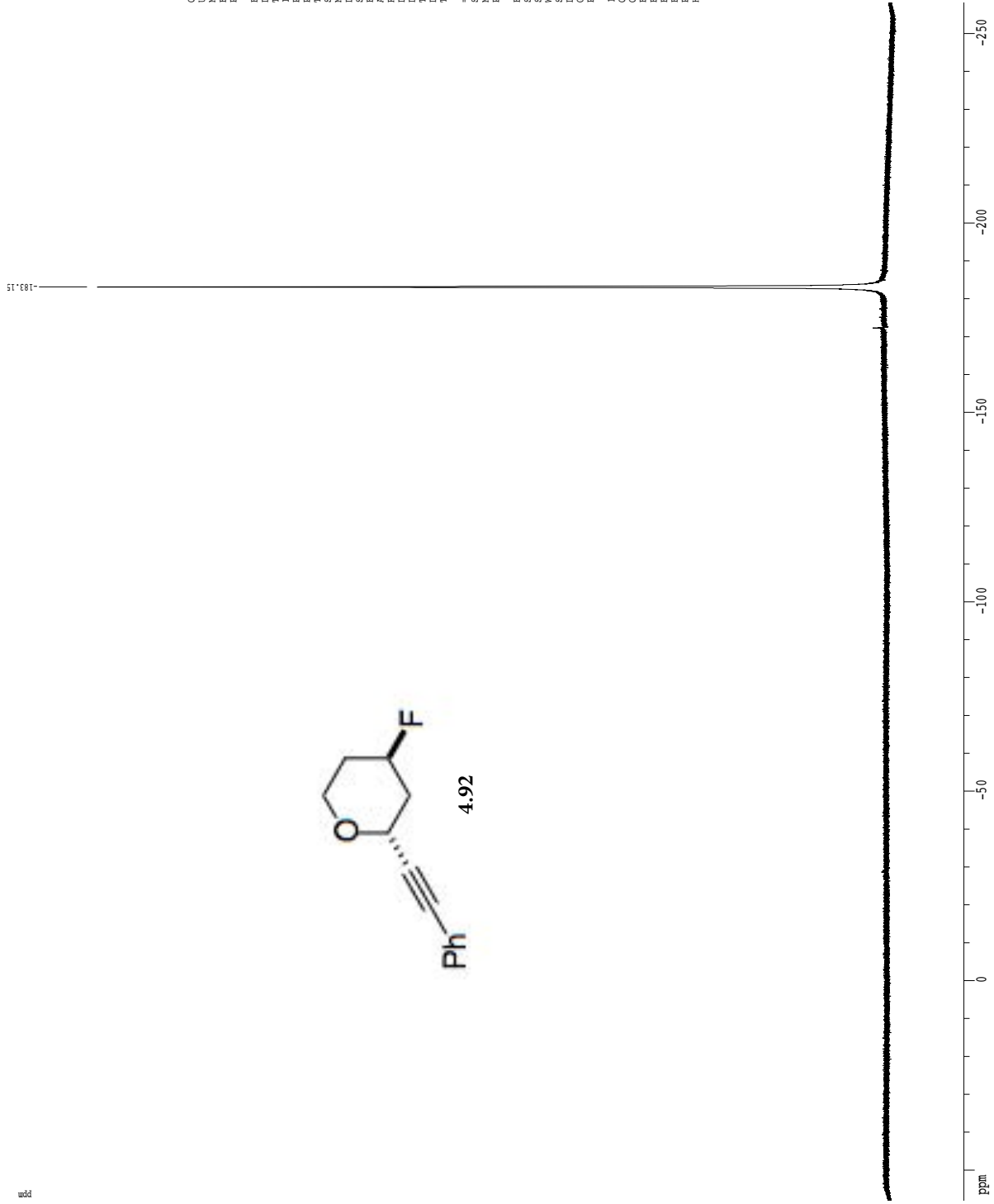
Current Data Parameters  
USER khschittl  
NAME KM-III-191P  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20160908  
Time 15:16  
INSTRUM av600  
PROBHD 5 mm CPBBO BB-  
PULPROG zgpg30  
F1 125.132  
SOLVENT CDCl3  
NS 16  
DS 2  
SHF 175.71422 MHz  
NUC1 13C  
MAG 0.320931686 G  
RG 515  
DM 2.800 usec  
DE 18.000 usec  
TE 300.2 K  
D1 3.0000000 sec  
TD0 1

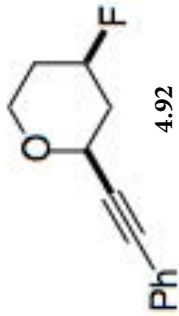
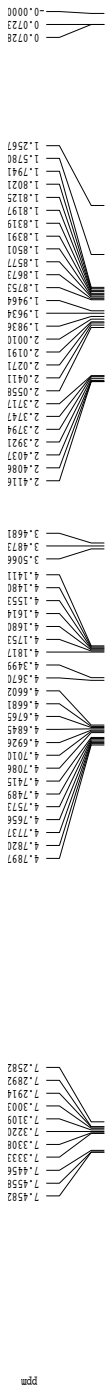
===== CHANNEL f1 =====  
SFO 125.132 MHz  
NUC1 13C  
P1 18.25 usec

F2 - Processing parameters  
SI 32768  
SF 564.686426 MHz  
WDW no  
SSB 0  
GB 0  
PC 1.00

IDMR plot parameters  
CT 2.00 cm  
CX 15.00 cm  
FLP 58.955 ppm  
F1 32762.68 Hz  
F2P -259.176 ppm  
FRANW 13.86912 Hz/cm  
HZCN 7832.08657 Hz/cm



h1.c



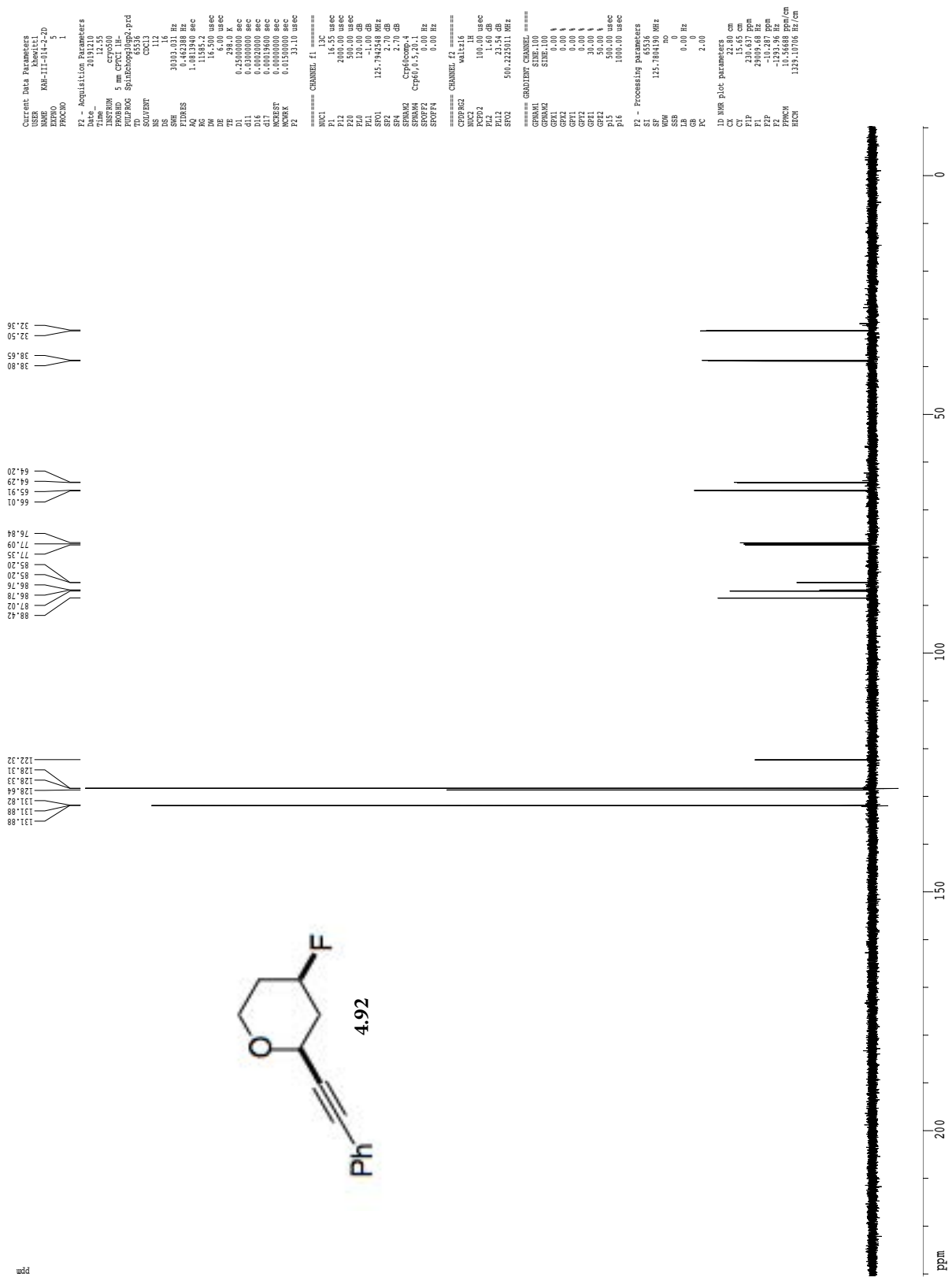
Current Data Parameters  
USER Khowatit  
EXPNO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20101010  
Time 10:32  
INSTRUM spect  
PROBHD 5 mm CPBBO  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 3  
DS 2  
SWH 9635.385 Hz  
FIDRES 0.498842 Hz  
AQ 5.0998979 sec  
RG 327.5  
DM 52.400 us/pt  
DE 14.23 us/pt  
TE 298.0 K  
TD0 0.100000000 sec

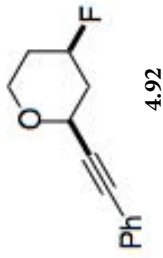
==== CHANNEL f1 =====  
NUC1 60.1324019 MHz  
P1 9.50 us/pt

F2 - Processing parameters  
SI 32768  
SF 600.1300533 MHz  
WDW EM  
SSB 0  
GB 0  
PC 1.00  
LB\_NMR plot parameters  
CY 22.80 cm  
CZ 15.00 cm  
FLP 9.000 ppm  
SFO 540.000 Hz  
FZ 3000.000 ppm  
PZ -3000.000 Hz  
PPMCM 0.41667 ppm/cm  
RECON 250.05518 Hz/cm



Z-restored spin-echo 13C spectrum with 1H decoupling





```

Current Data Parameters
=====
USER      NAME      ECHO     PROCNO
=====
          KAH-III-014-2-20
          6
          1

F2 - Acquisition Parameters
=====
Date_     Time_
-----
20010811 11:25
INSFREQ   CYSO500
PROBHD    5 mm CPCL IH-
PULPROG   zgpg30
SOLVENT   CDCl3
NS        16
DS        4
SWH        8012.820 Hz
FIDRES     3.912510 Hz
AQ         0.116832 sec
RG         62.400 usec
DK         62.400 usec
DE         6.00 usec
TE         298.0 K
AQ0        0.0000300 sec
RG0        1.0000000 usec
D13        0.0000300 sec
D16        0.00020000 sec
IN0        0.00012480 sec

===== CHANNEL f1 =====
NUC1      P1      P2
----      --      --
13C       7.50 usec
PLL       1.60 dB
SFO1      500.2235015 MHz

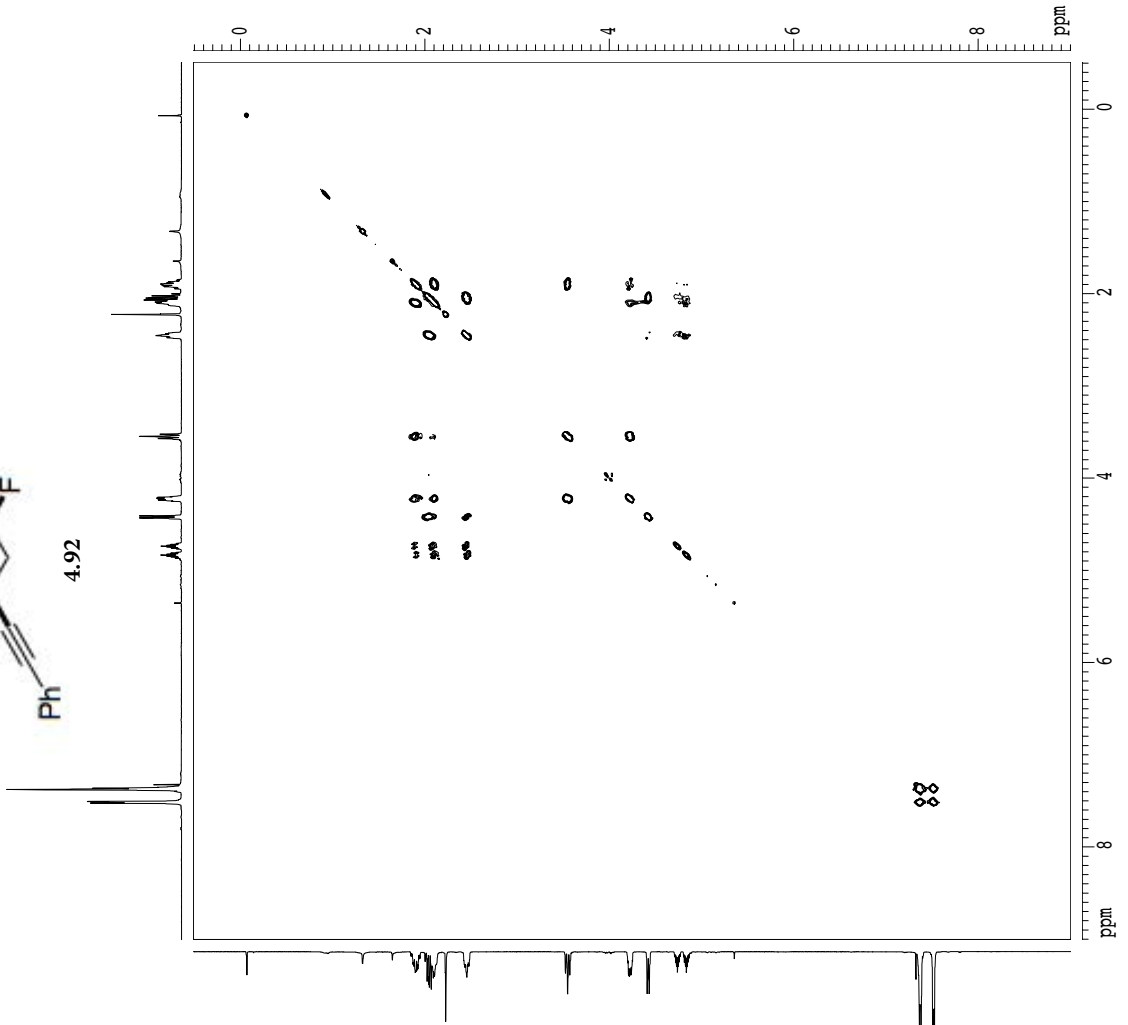
===== GRABBER CHANNEL =====
GRABMG    SFO1
-----
GPX1      0.00 %
GPX2      0.00 %
GPF1      0.00 %
GPF2      0.00 %
GPR1      17.00 %
GPR2      17.00 %
P16       1000.00 usec

F1 - Acquisition parameters
=====
NUC1      P1      P2
----      --      --
13C       7.50 usec
SFO1      500.2235 MHz
FIDRES    15.650040 Hz
SN        16.018 ppm
FNOISE    QF

F2 - Processing parameters
=====
SI        1024
SF        500.2200000 MHz
SSB       0
GB        0
PC        1.00

F1 - Processing parameters
=====
SI        1024
SF        500.2200000 MHz
SSB       0
GB        0
PC        1.00

2D NMR plot parameters
=====
CX2       15.00 cm
CY2       15.00 cm
FZLO      9.002 ppm
FZHI      49.508 ppm
FZL1      -0.509 ppm
FZL2      -254.47 Hz
FZLO      9.002 ppm
FZL1      450.114 Hz
FZL2      -254.905 ppm
FZHCN     0.65407 ppm/cm
FZHCN     317.17416 Hz/cm
FZPCN     0.65407 ppm/cm
FZPCN     317.17416 Hz/cm
  
```



gcosy60

```

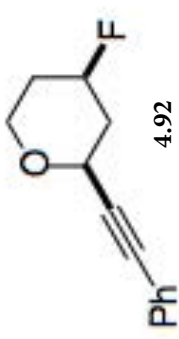
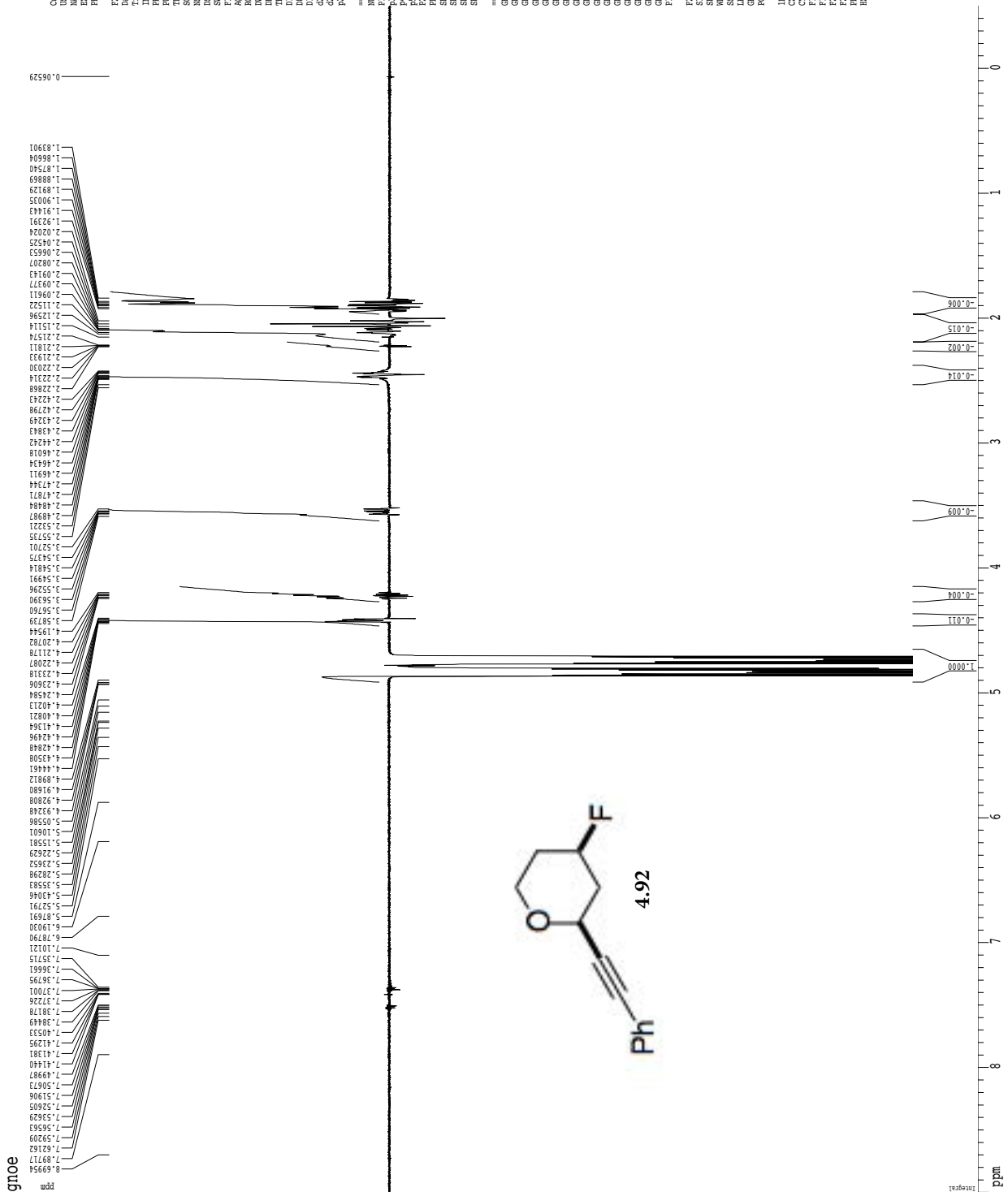
Current Data Parameters
USER      Krewitt1
EXPNO    4
PROCNO   1
PROC3D   0

F2 - Acquisition Parameters
Date_    20191210
Time     07:45:01
INSTRUM  cpdcp1
PROBHD   5 mm CPYCI 1H
PULPROG  groe.cg22.prd
TD       65536
SFO      500.136260 MHz
AQ       0.1839899 sec
RG       655
DS       4
SS       8
SWH      8012.822 Hz
FIDRES   0.122266 Hz
AQRES    4.191966 sec
RGRES    101.6
DSRES    62.400 usec
DE        6.00 usec
TE        300.2 K
D1        1.0020000 sec
D11       0.5000000 sec
D12       0.0020000 sec
dZ1       0.33376500 sec
dZ2       0.1839899 sec
dZ        15.00 usec

===== CHANNEL f1 =====
NUC1      1H
P1        12.00 usec
PL1       0.00 dB
P2        21.50 usec
PL2       0.00 dB
P3        34.00 usec
PL3       0.00 dB
P4        24.00 usec
PL4       0.00 dB
P5        40000.00 usec
PL5       0.00 dB
SFO1      500.136260 MHz
WDW       EM
SSB       0
GB        0
PC        1.00
===== GRADIENT CHANNEL =====
GDMAX1    sine.100
GDMAX2    sine.100
GDMAX3    sine.100
GDMAX4    sine.100
GDX1      0.00 k
GDX2      0.00 k
GDX3      0.00 k
GDX4      0.00 k
GDX5      0.00 k
GDX6      0.00 k
GDX7      0.00 k
GDX8      0.00 k
GDX9      0.00 k
GDX10     0.00 k
GDX11     0.00 k
GDX12     0.00 k
GDX13     0.00 k
GDX14     0.00 k
GDX15     0.00 k
GDX16     0.00 k
===== Processing parameters =====
SI        65536
SF        500.136260 MHz
WDW       EM
SSB       0
GB        0
PC        1.00

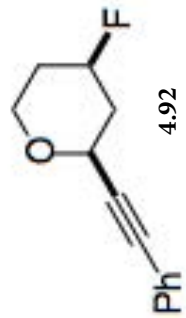
ID NMR file parameters
CX        22.86 cm
CY        54.00 cm
FIP       9.000 ppm
F2P       4.920 ppm
F2        -250.11 Hz
F2PCH     0.4166 ppm/cm
RECCH     208.4250 Hz/cm

```



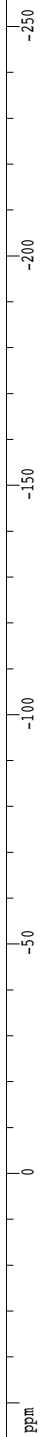
f19.c

1

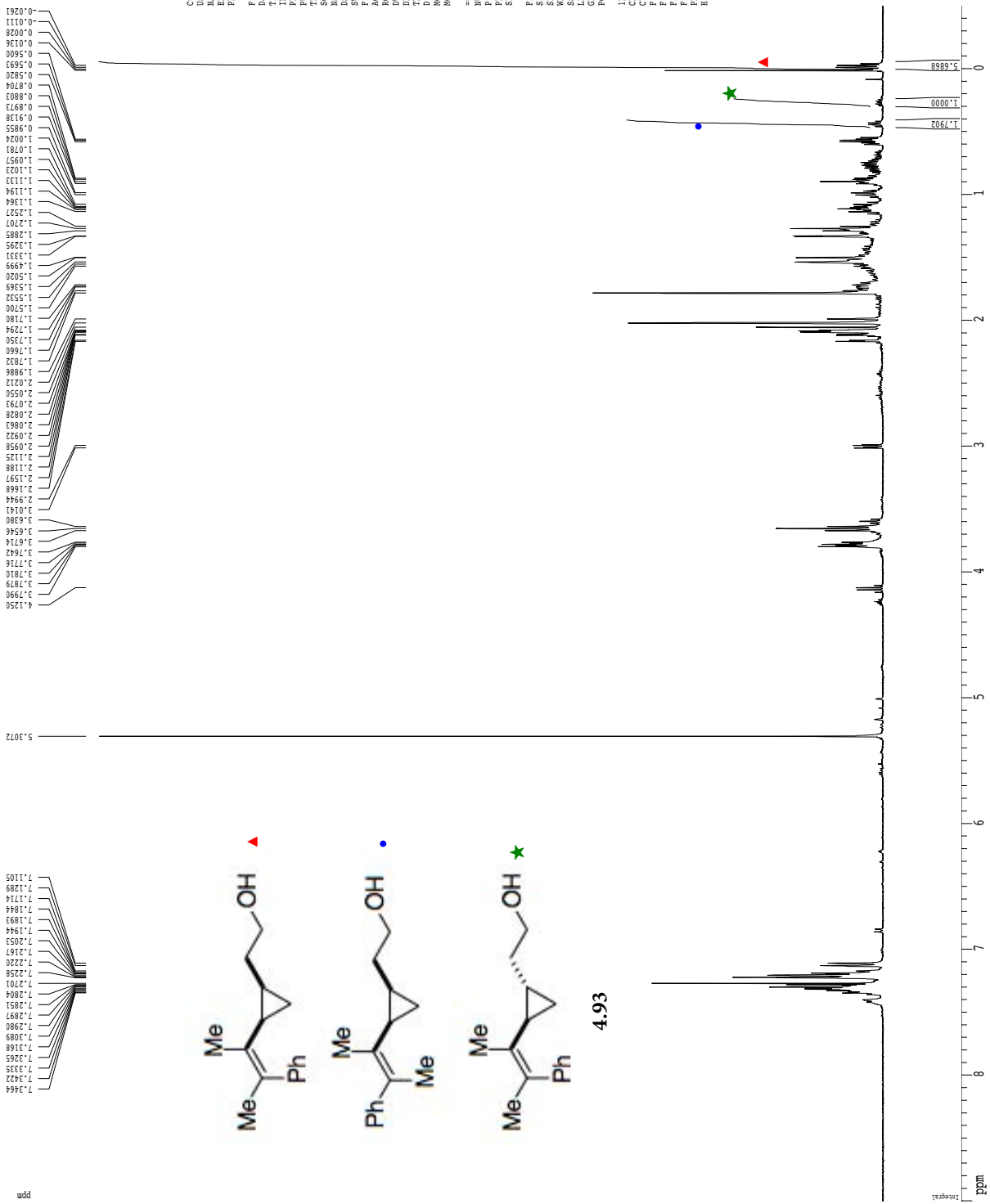


-172.44  
-172.35

Current Data Parameters  
USER Rhowittl  
EXPNO 1  
PROCNO 1  
PROCNO 1  
Date\_ 20010630  
Time 10.38  
INSTRUM av600  
PROBHD 5 mm CPBBO  
PULPROG zgpg30  
TD 131072  
SOLVENT CDCl3  
AQ 179571.422 Hz  
FIDRES 1.862392 Hz  
AQ 0.3670516 sec  
RG 2.800 USFC  
DE 18.00 USFC  
TE 298.0 K  
TD0 3.0000000 sec  
===== CHANNEL f1 =====  
NUC1 564.66915 MHz  
NUC2  
PC1 18.25 USFC  
F2 - Processing parameters  
SF 564.664315 MHz  
WDW no  
SSB 0  
GB 0  
PC 1.00  
LD\_MW\_plot\_parameters  
CX 22.80 cm  
CY 15.00 cm  
FLP 88.457 ppm  
FZ 2.68 Hz  
F2 -1453917.58 Hz  
FREQM 13.866379 ppm/cm  
FREQH 7032.08057 Hz/cm

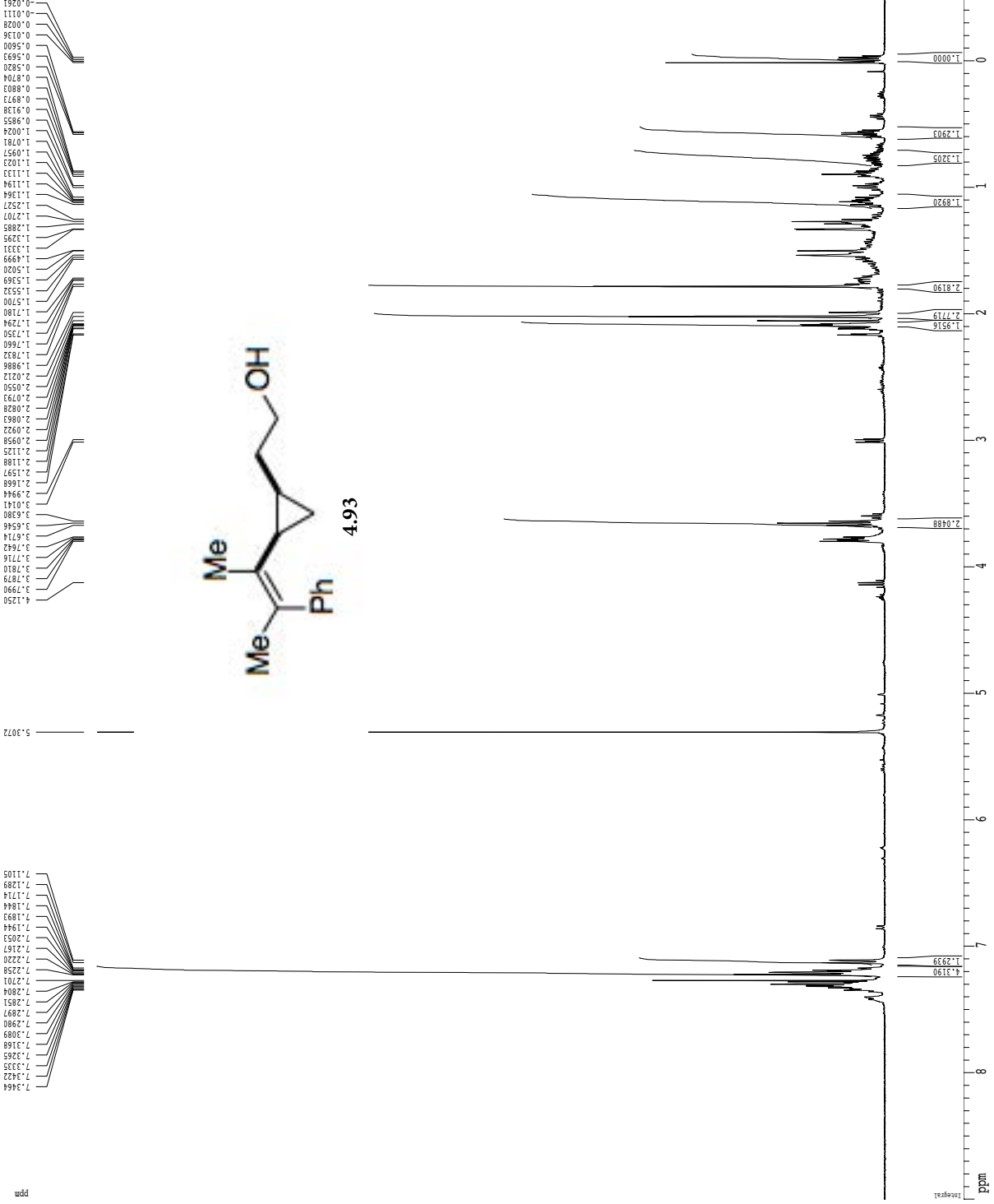


1H spectrum



Current Data Parameters  
 USER KAMRITT  
 NAME KAM-11-30-1  
 EXNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20191208  
 Time 14.30  
 INSTRUM spect  
 PROBHD 5 mm BBO  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 DS 9  
 SFO 6410.258 Hz  
 FIDRES 0.166673 Hz  
 AQ 2.199229 sec  
 RG 327.8  
 DW 78.000 usec  
 DE 4.50 usec  
 TE 297.8 K  
 T1 0.100000 sec  
 MCBST 0.000000 sec  
 MCORE 0.0150000 sec  
 ===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.132809 MHz  
 F2 - Processing Parameters  
 SI 65536  
 SF 400.1300175 MHz  
 WDW no  
 GB 0.00 Hz  
 PC 2.00  
 ID WDR file parameters  
 CY 22.80 cm  
 CV 15.00 cm  
 FIP 9.000 ppm  
 FL 360.10 Hz  
 F2 -2000.00 Hz  
 F3 -2000.00 Hz  
 PPMCM 0.41667 ppm/cm  
 HZCM 166.72084 Hz/cm

1H spectrum



Current Data Parameters  
 USER KAWAII  
 NAME KAN-IT-30-1  
 EXNO 1  
 PROCNO 1

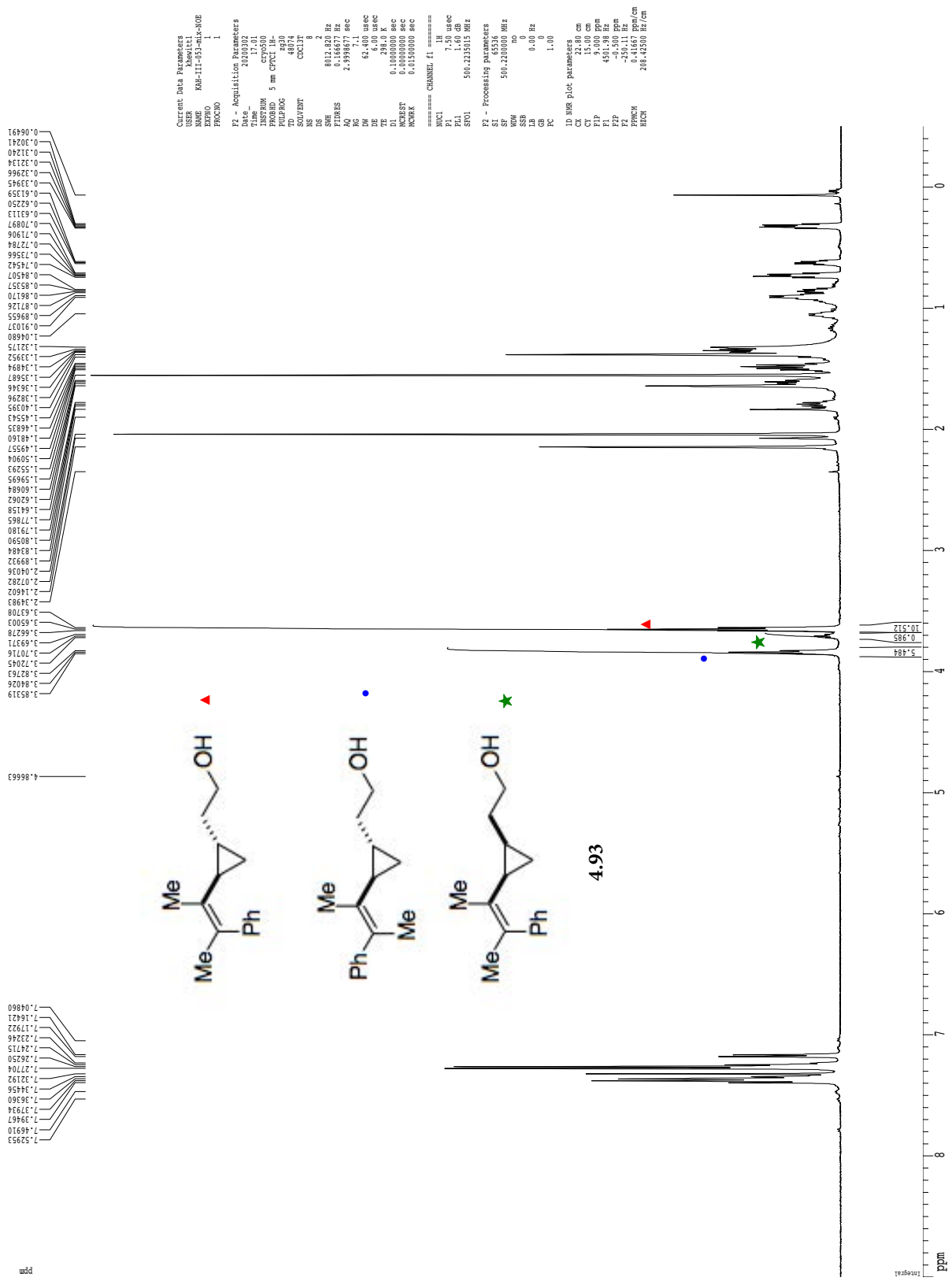
F2 - Acquisition Parameters  
 Date\_ 20191208  
 Time 14.30  
 PROBN 1314400  
 PROCNO 5  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 DS 9  
 SFO 6410.258 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.199229 sec  
 RG 327.5  
 DW 78.000 usec  
 DE 4.50 usec  
 TE 297.2 K  
 T1 0.1000000 sec  
 T2 0.0000000 sec  
 MCRST 0.0000000 sec  
 MCRBK 0.0000000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.00 usec  
 PL1 -1.10 dB  
 SFO1 400.1328009 MHz

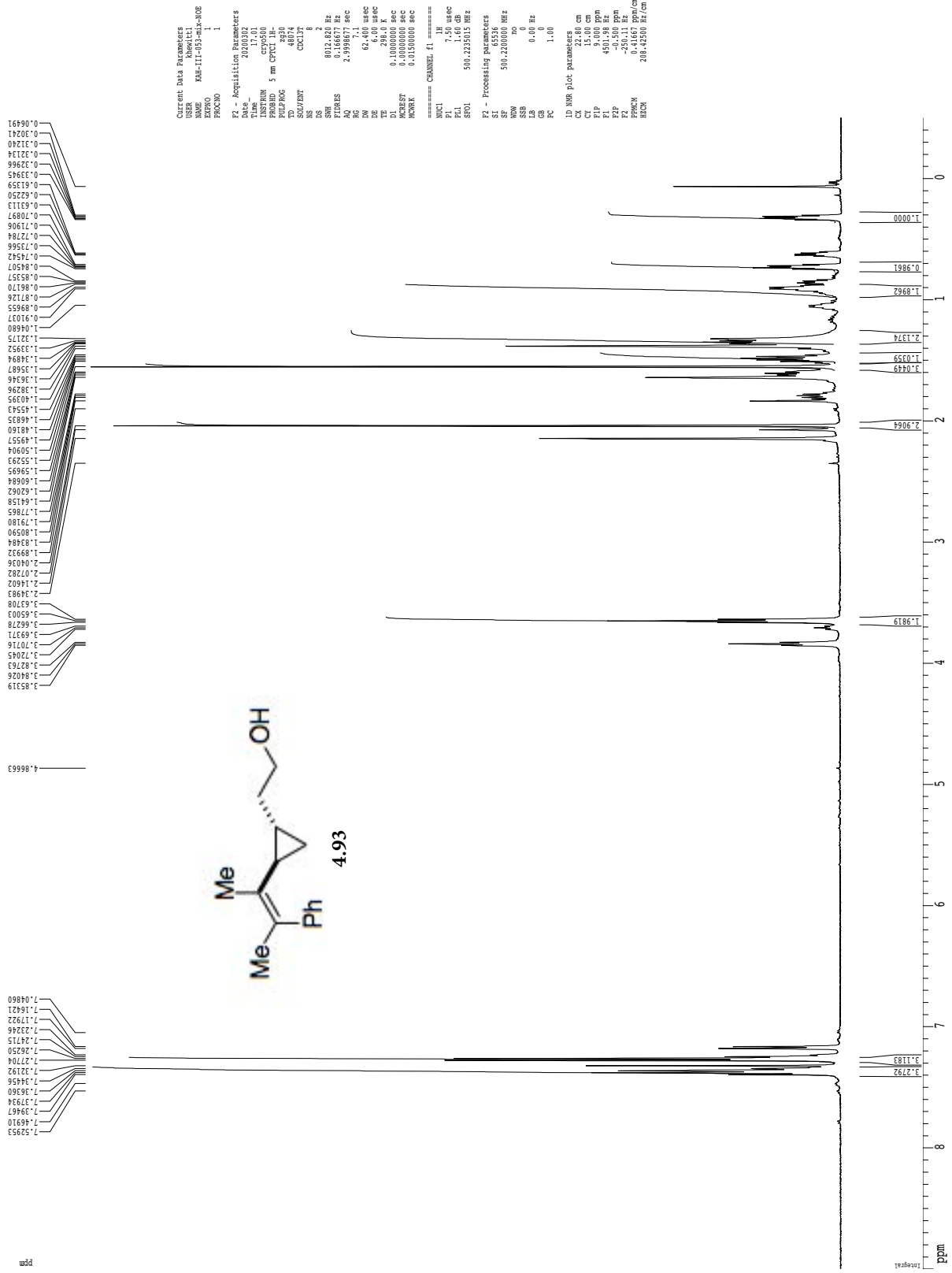
F2 - Processing Parameters  
 SI 65536  
 SF 400.1300175 MHz  
 WDM no  
 LB 0.00 Hz  
 GB 0  
 PC 2.00

DJ NR Parameters  
 CY 22.80 cm  
 CV 15.00 cm  
 FIP 9.000 ppm  
 FL 3600.10 Hz  
 F2 -200.00 Hz  
 F2 0.41667 ppm/cm  
 HZCM 166.72084 Hz/cm

<sup>1</sup>H spectrum



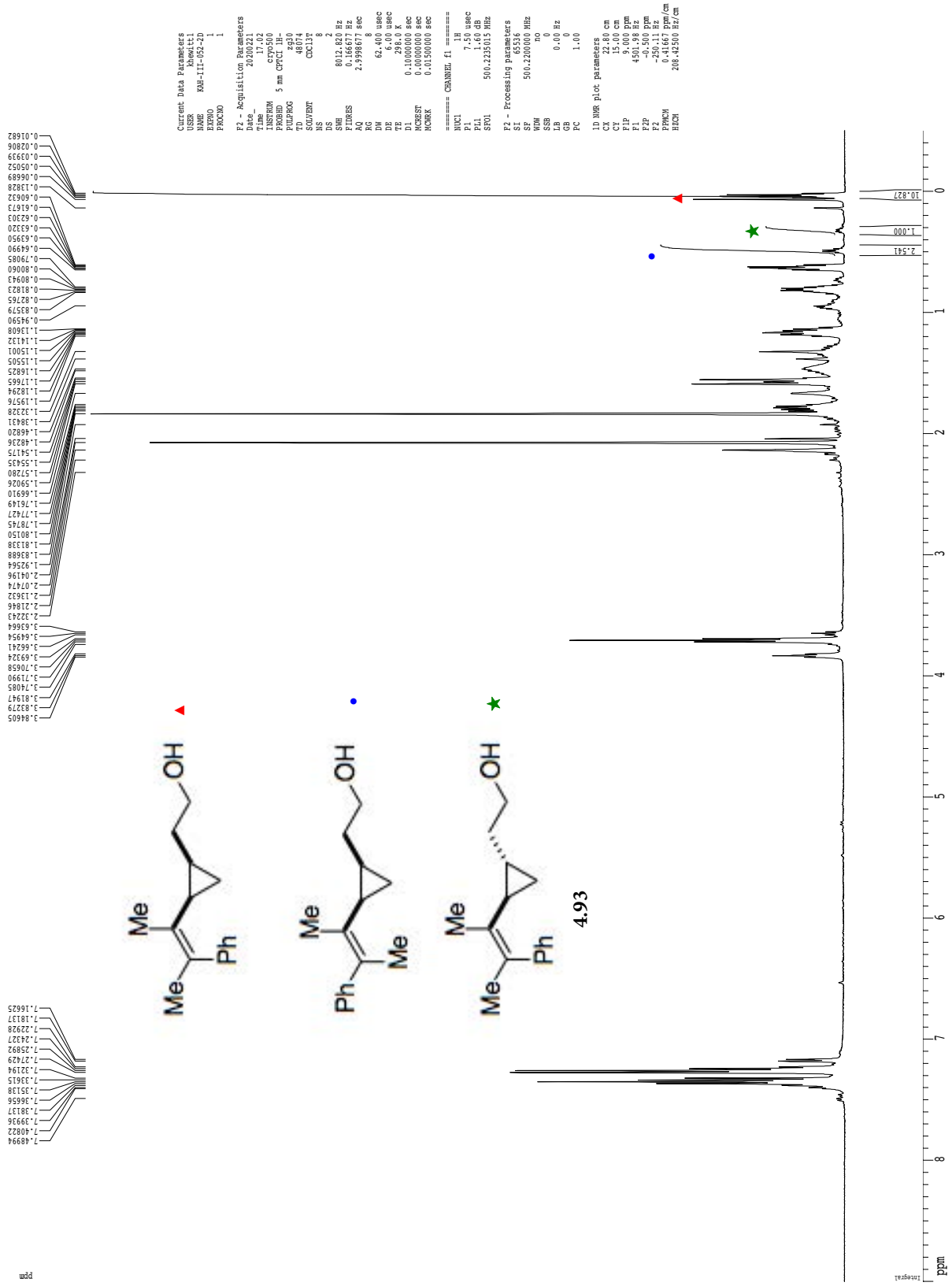
1H spectrum



Current Data Parameters  
 USER kowatt1  
 NAME RM-III-051-mls-NOE  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 201012  
 Time\_ 11:02  
 INSTRUM crys500  
 PROBRD 5 mm CPCL H-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT COCLIT  
 NS 8  
 DS 8  
 SWH 8012.820 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.399677 sec  
 RG 327.500  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 300.2 K  
 D1 0.10000000 sec  
 MCREST 0.00000000 sec  
 MORK 0.01500000 sec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.50 usec  
 PL1 0.00 dB  
 SFO1 500.235015 MHz  
 F2 - Processing parameters  
 SI 32768  
 SF 500.235000 MHz  
 WDW no  
 GB 0  
 CB 0  
 PC 1.00  
 ID: NMR 3/04 parameters  
 CX 22.80 cm  
 CY 15.00 cm  
 F1 4998.50 Hz  
 F2 -500.000000 Hz  
 F3 250.11 Hz  
 FWHM 4.43 Hz  
 EQW 204.42500 Hz/cm

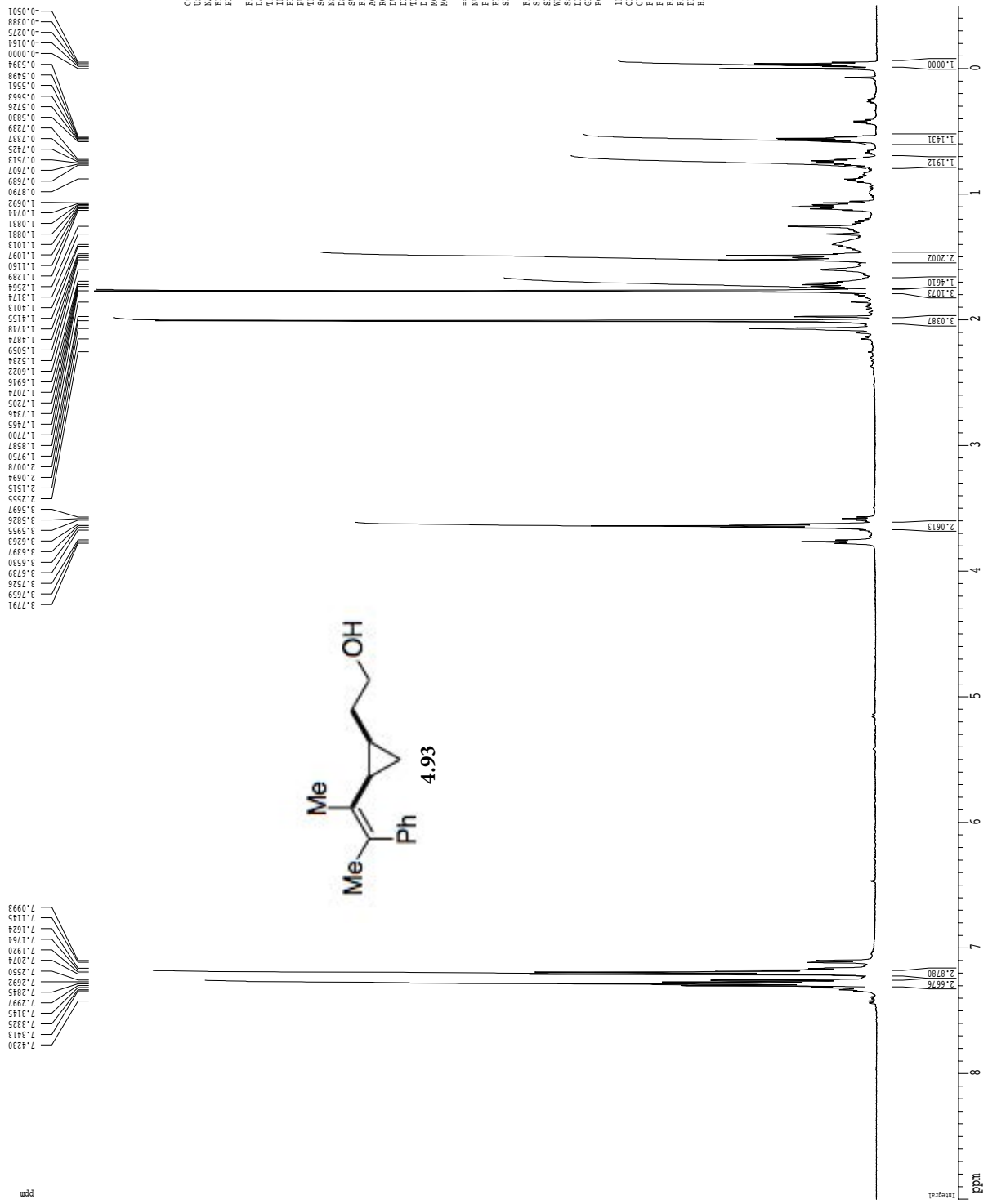


1H spectrum



Current Data Parameters  
 USER: KMH-III-052-ZD  
 NAME: KMH-III-052-ZD  
 EXPNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20200221  
 Time: 17:02  
 PROBNM: 07270100  
 PULPROG: zgpg30  
 PROCNO: 5  
 TD: 48074  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 8012.820 Hz  
 FIDRES: 0.16667 Hz  
 AQ: 2.3398677 sec  
 RG: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 T1: 0.10000000 sec  
 MCHRES: 0.00000000 sec  
 ACQRES: 0.00500000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 1.50 usec  
 PL1: 1.68 dB  
 SFO1: 500.2235015 MHz  
 F2 - Processing parameters  
 SI: 65536  
 SF: 500.2200000 MHz  
 WDW: no  
 SSB: 0.00 Hz  
 GB: 0  
 PC: 1.00  
 ID: NMR file parameters  
 CY: 22.80 cm  
 CX: 15.00 cm  
 FIP: 9.000 ppm  
 F1: 4300.136 Hz  
 F2: -250.11 Hz  
 PPRM1: 0.41667 ppm/cm  
 HZHM: 206.44500 Hz/cm

1H spectrum



Current Data Parameters  
 USER: Kowalski  
 NAME: KNU-III-052-2D  
 EXNO: 1  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20200221  
 Time: 17.02  
 INSTR: spect  
 PULPROG: zgpg30  
 TD: 48074  
 SOLVENT: CDCl3  
 DS: 9  
 SFO: 8012.820 Hz  
 FIDRES: 0.166677 Hz  
 AQ: 2.3998677 sec  
 RG: 62.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 SCANS: 0.10000000 sec  
 ACQRES: 0.00000000 sec  
 NUC1: 13C  
 P1: 1.50 usec  
 PL1: 1.66 dB  
 SFO1: 500.2235015 MHz

F2 - Processing Parameters  
 SI: 65536  
 SF: 500.2203315 MHz  
 WDW: no  
 SSB: 0.00 Hz  
 GB: 0  
 PC: 1.00

D0 NMR file parameters  
 CY: 22.80 cm  
 C1: 15.00 cm  
 P1P: 9.000 ppm  
 F1: 4300.98 Hz  
 F2: -250.11 Hz  
 PPRCM: 0.41667 ppm/cm  
 HZCM: 206.44502 Hz/cm

Current Data Parameters  
 USER Meeatcl  
 EXPNO RM-111-503-C02  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20200219  
 Time 12:02:00  
 INSTRUM spect  
 PROBHD 5 mm CPCL 1H-  
 PULPROG cosyg60.prd  
 TD 2048  
 SOLVENT CDCl3  
 NS 16  
 DS 16  
 SWH 4734.849 Hz  
 FIDRES 2.311938 Hz  
 AQ 0.2163188 sec  
 RG 1024  
 RW 105.600 usec  
 DE 105.600 usec  
 TE 298.0 K  
 d0 0.0000300 sec  
 d1 1.0000000 sec  
 d13 0.0000300 sec  
 d16 0.0000300 sec  
 d20 0.0001129 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*

NUC1 1H  
 P1 7.50 usec  
 PL1 0.00 dB  
 SF01 500.2212129 MHz

\*\*\*\*\* GRADIENT CHANNEL \*\*\*\*\*

GRNM1 sine.100  
 GRNM2 sine.100  
 GRP1 0.00 %  
 GRP2 0.00 %  
 GRP3 0.00 %  
 GRP4 0.00 %  
 GRP5 0.00 %  
 GRP6 0.00 %  
 GRP7 0.00 %  
 GRP8 0.00 %  
 GRP9 0.00 %  
 GRP10 0.00 %  
 GRP11 0.00 %  
 GRP12 0.00 %  
 GRP13 0.00 %  
 GRP14 0.00 %  
 GRP15 0.00 %  
 GRP16 0.00 %  
 P16 1000.00 usec

F1 - Acquisition parameters

NU0 1  
 TD 512  
 SFO1 500.2221 MHz  
 FIDRES 9.247751 Hz  
 AQC 3.4 ppm  
 P0 0.00 usec  
 PC 1.00

F2 - Processing parameters

SI 1024  
 SF 500.2200000 MHz  
 SFO 511.625 MHz  
 SSF 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters

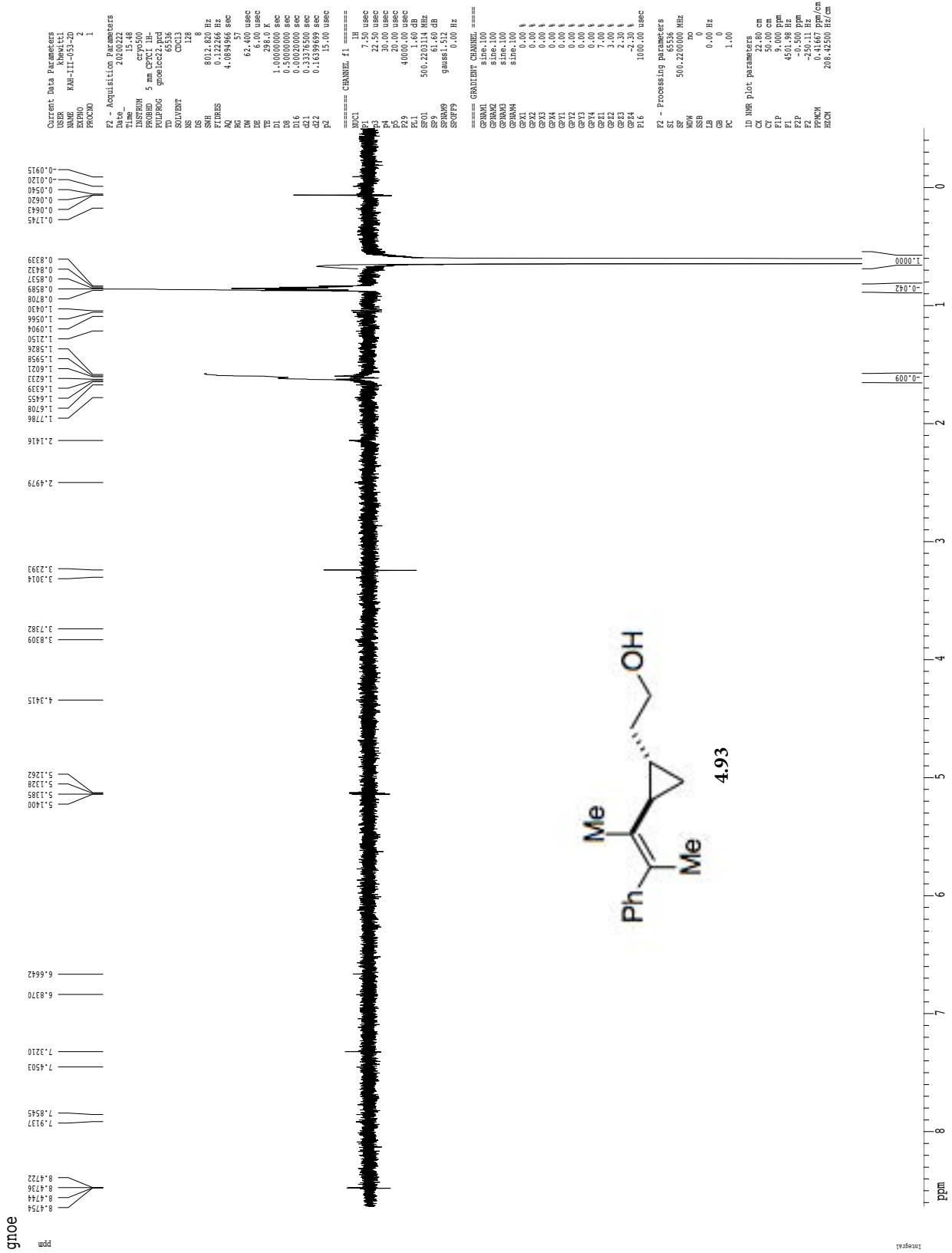
SI 1024  
 SF 500.2200000 MHz  
 SFO 511.625 MHz  
 SSF 0  
 LB 0.00 Hz  
 GB 0

2D NMR Plot parameters

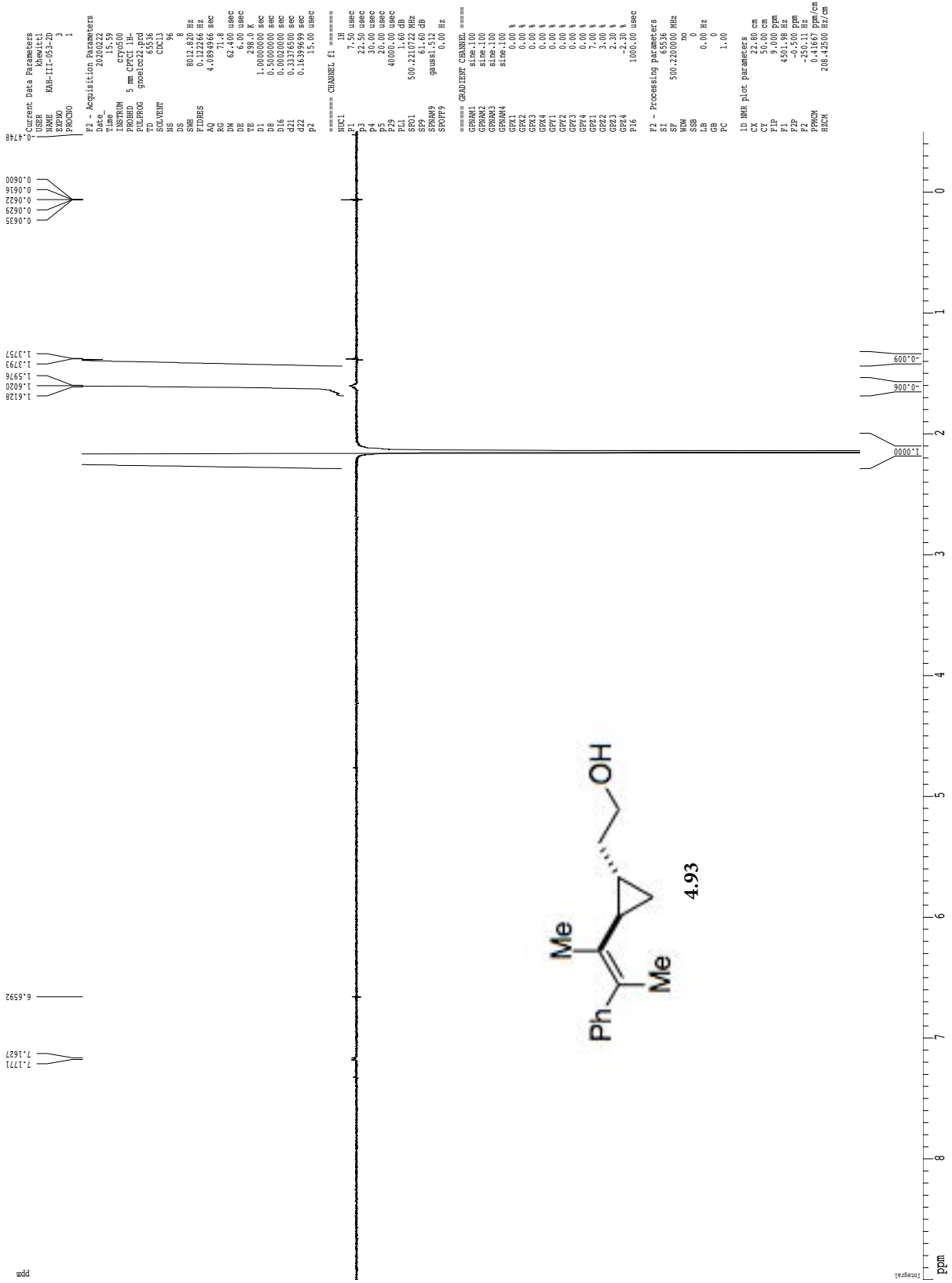
CX2 15.00 cm  
 CX1 15.00 cm  
 ZF2 4493.86 Hz  
 FZPHI -0.463 ppr  
 F2H1 -241.49 Hz  
 F2L0 8.983 ppr  
 F2L1 4493.36 Hz  
 F2H2 -241.49 ppr  
 F2L2 8.983 ppr  
 F2PHI 0.463 ppr  
 F2PCMC 0.63104 ppr/cm  
 F2PHCM 315.65659 Hz/cm  
 F2PCCH 0.63104 ppr/cm  
 F2PHCN 315.65659 Hz/cm

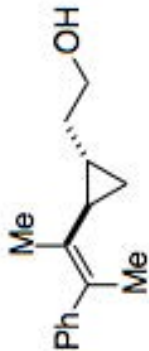


gnoe



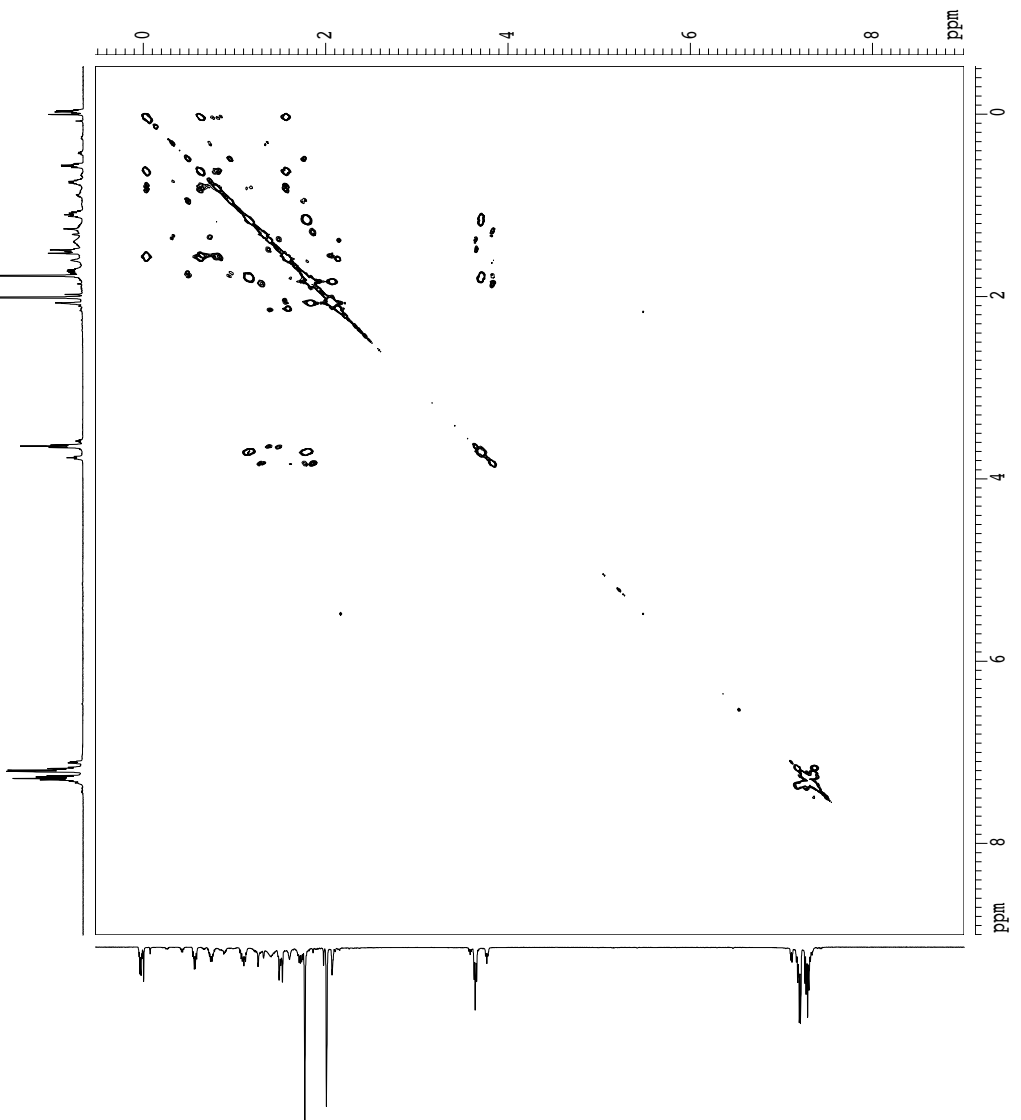
gnoe





gcosy60

4.93



Current Data Parameters  
 USER khsvit1  
 NAME RAN-III-052-2D  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20200221  
 Time 17.04  
 INSTRUM cryo500  
 PULPROG zgpg30  
 PROCNO 2048  
 SOLVENT CDCl3  
 NS 1  
 DS 16  
 SWH 8013.81 Hz  
 FIDRES 3.912510 Hz  
 AQ 0.1278452 sec  
 RG 181  
 DW 62.400 usec  
 DE 298.0 usec  
 TE 298.0 usec  
 D0 0.0000300 sec  
 D1 1.0000000 sec  
 d13 0.0000300 sec  
 D16 0.0002000 sec  
 D10 0.0001480 sec

==== CHANNEL f1 =====  
 NU11 1H  
 P1 7.50 usec  
 PL 0.00 dB  
 SFO1 500.225015 MHz

==== GRADIENT CHANNEL =====  
 GPM11 sine,100  
 GPM12 sine,100  
 GPZ 0.00 %  
 GPY 0.00 %  
 GPX 0.00 %  
 GPZ 0.00 %  
 GPY 0.00 %  
 GPX 0.00 %  
 G16 1000.00 usec

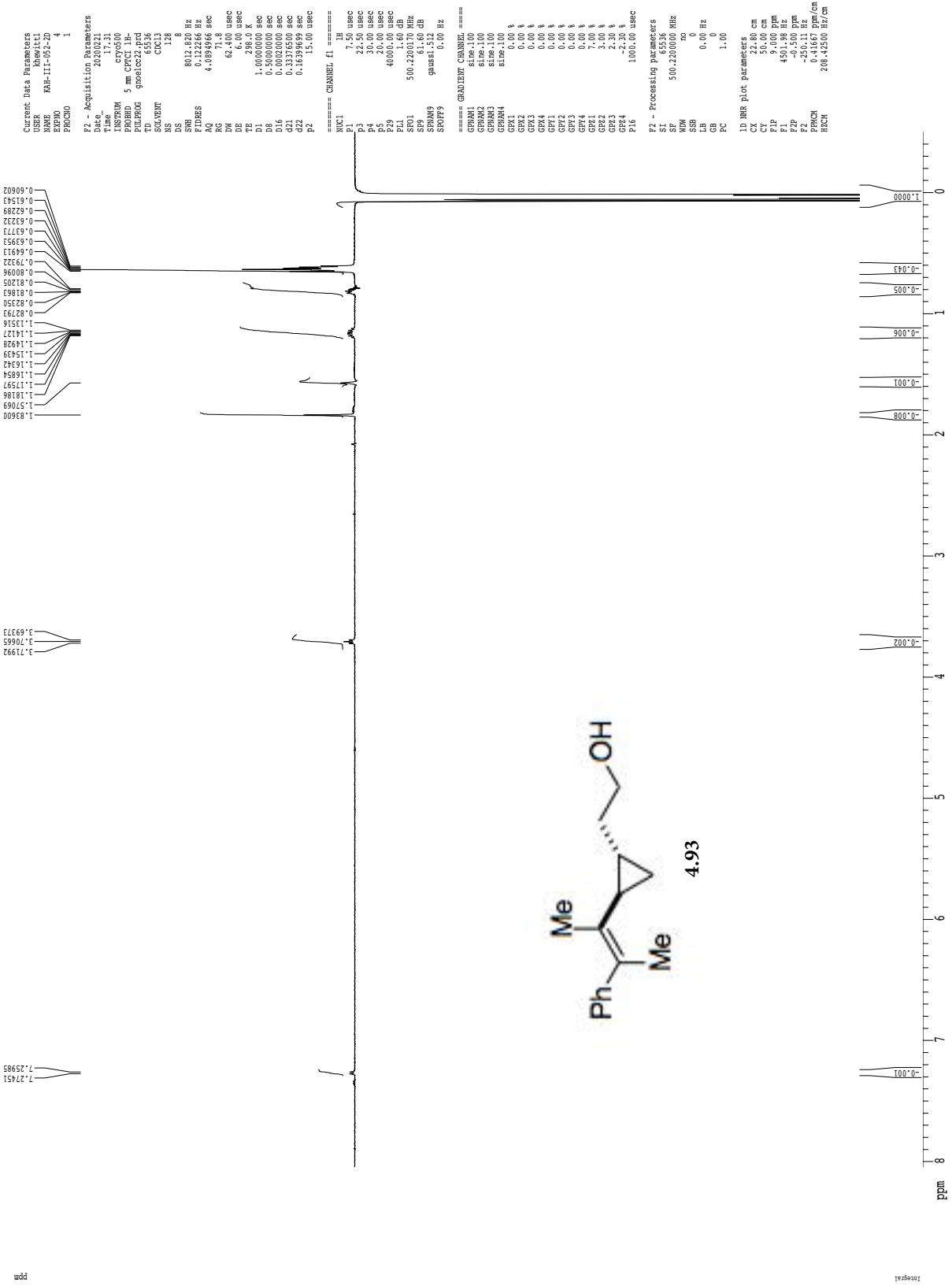
F1 - Acquisition parameters  
 ND0 1  
 NS 16  
 SWH 8013.81 Hz  
 SFO1 500.225015 MHz  
 FIDRES 3.912510 Hz  
 SW 16.018 ppm  
 FMODE GF

F2 - Processing parameters  
 SI 32768  
 SF 500.2200000 MHz  
 WDM 1.0000000  
 SSB 0  
 CB 0.00 Hz  
 GC 0.00 Hz  
 PC 1.00

F1 - Processing parameters  
 SI 32768  
 SF 500.2200000 MHz  
 WDM 1.0000000  
 SSB 0  
 LB 0.00 Hz  
 GB 0

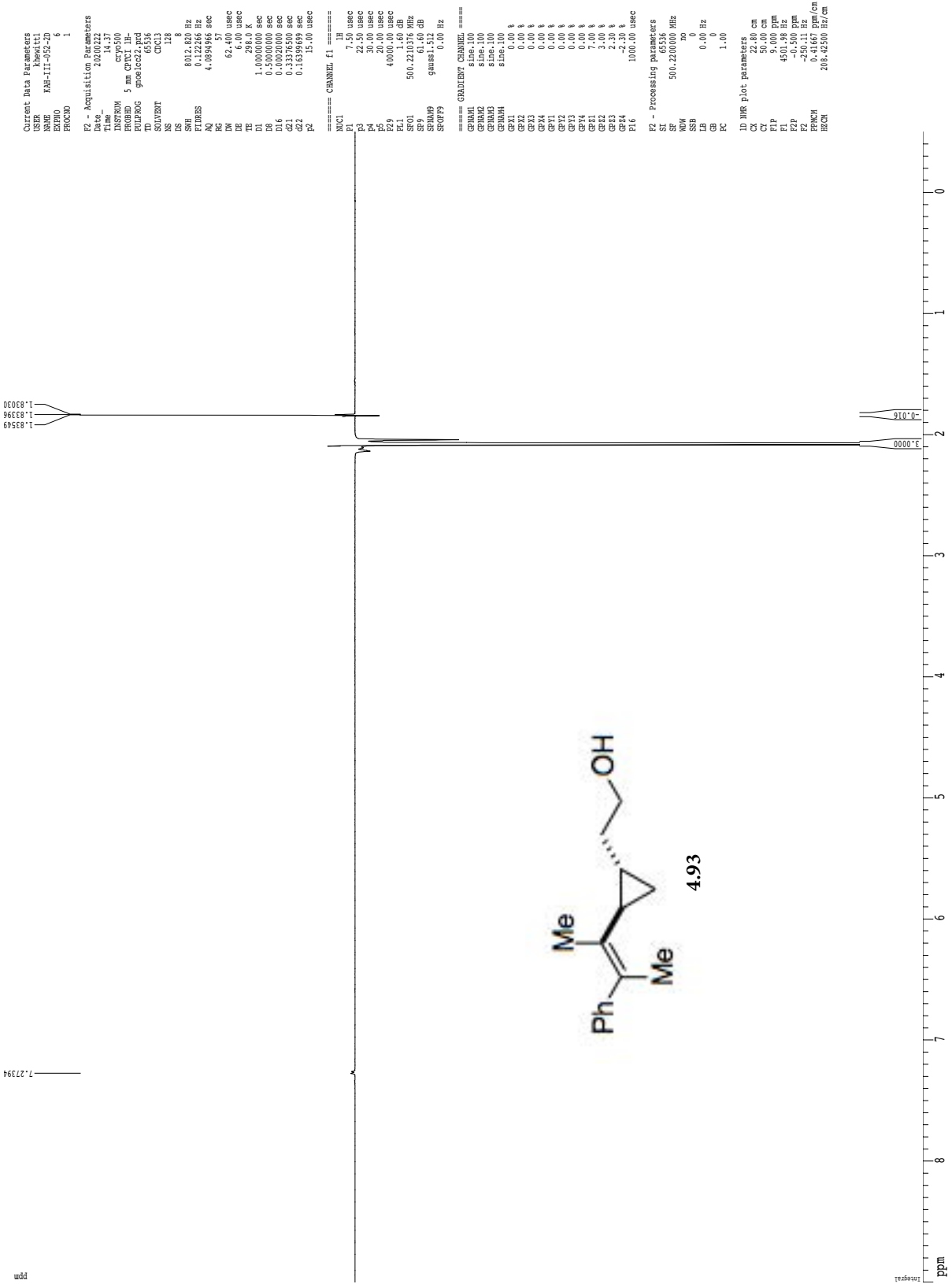
2D NMR Plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 5.002 ppm  
 F2P20 45.000 ppm  
 F2P10 45.524 ppm  
 F2HI -262.29 Hz  
 F1PLO 9.002 ppm  
 F1LO 4503.14 Hz  
 F1PHI 0.524 ppm  
 F1PCW 2.000 ppm  
 F2PCW 0.63511 ppm/cm  
 F2RCM 317.69583 Hz/cm  
 F1PCW 0.63511 ppm/cm  
 F1RCM 317.69583 Hz/cm

gnoe



gnoe

ppm





gc0sy60

```

Current Data Parameters
USER          khevit11
NAME          KAH-III-053-COSY
EXPNO        2
PROCNO       1

F2 - Acquisition Parameters
Date_        201013
Time         1013
INSTRUM      cryo500
PROBHD       5 mm CPXI 1H
PULPROG      cosyprg0.prd
TD           2048
SOLVENT      CDCl3
NS           1
DS           16
SFR          4734.64 Hz
FIDRES       2.311938 Hz
AQ           0.2163188 sec
RG           1024
DW           105.600 usec
DE           6.00 usec
TE           298.0 K
d0           0.00000300 sec
d1           1.00000000 sec
d11          0.00000000 sec
d16          0.00020000 sec
d18          0.00021200 sec
IN0          0.00021200 sec

===== CHANNEL f1 =====
NUC1         1H
P1           7.50 usec
PL1          1.60 dB
SFO1         500.2221259 MHz

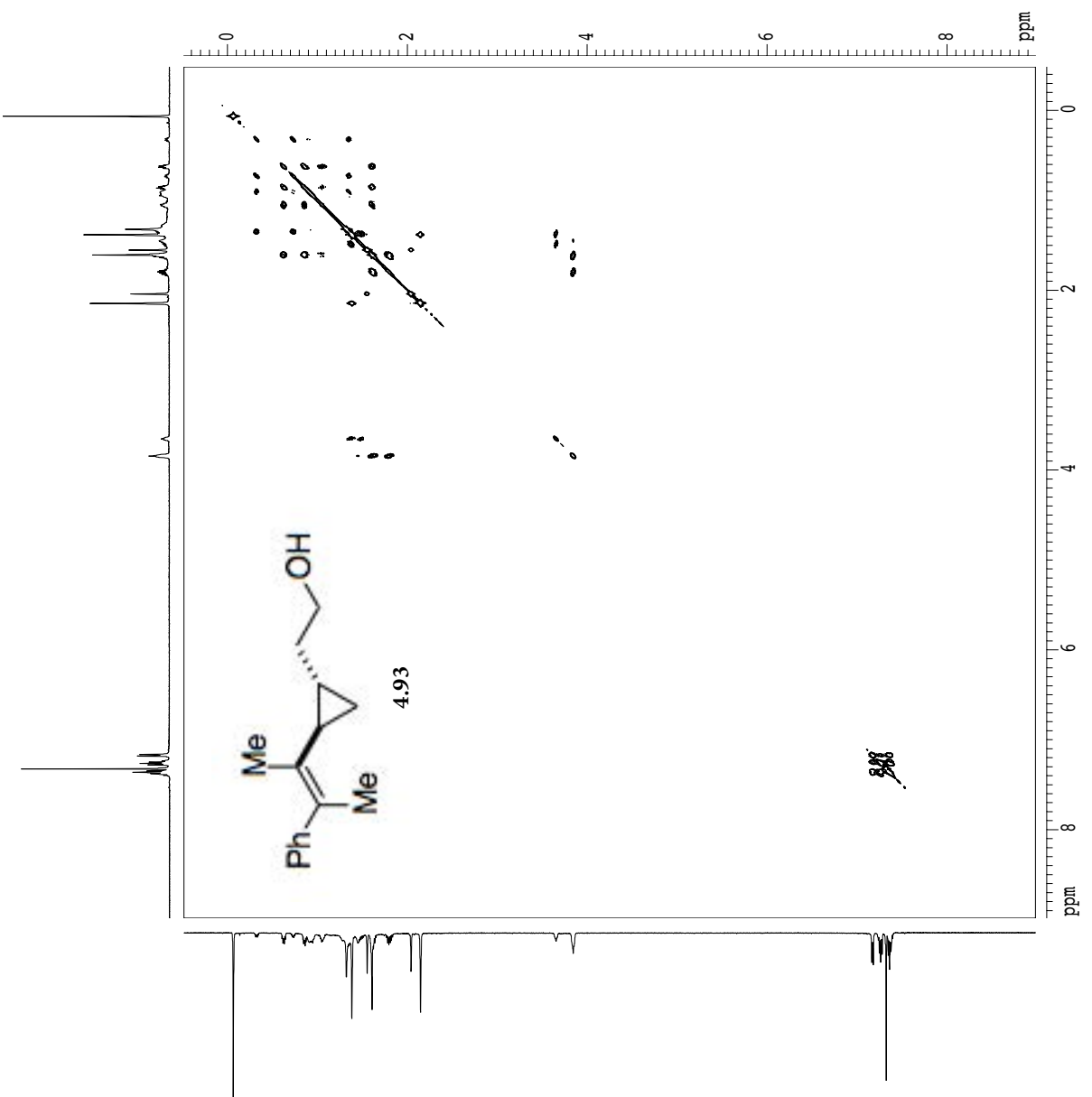
===== GRADIENT CHANNEL =====
GPRAM1       sine.100
GFX1         0.00 %
GFX2         0.00 %
GFY1         0.00 %
GFY2         0.00 %
GZ1          17.00 %
GZ2          17.00 %
P16         1000.00 usec

F1 - Acquisition parameters
ND0          1
TD           512
SFO1         500.2221 MHz
FIDRES       9.247751 Hz
SFO2         9.465 ppm
P16000      OF

F2 - Processing parameters
SI           1024
SF           500.2200000 MHz
WDW          SINE
SSB          0
LB           0.00 Hz
GB           0
PC           1.00

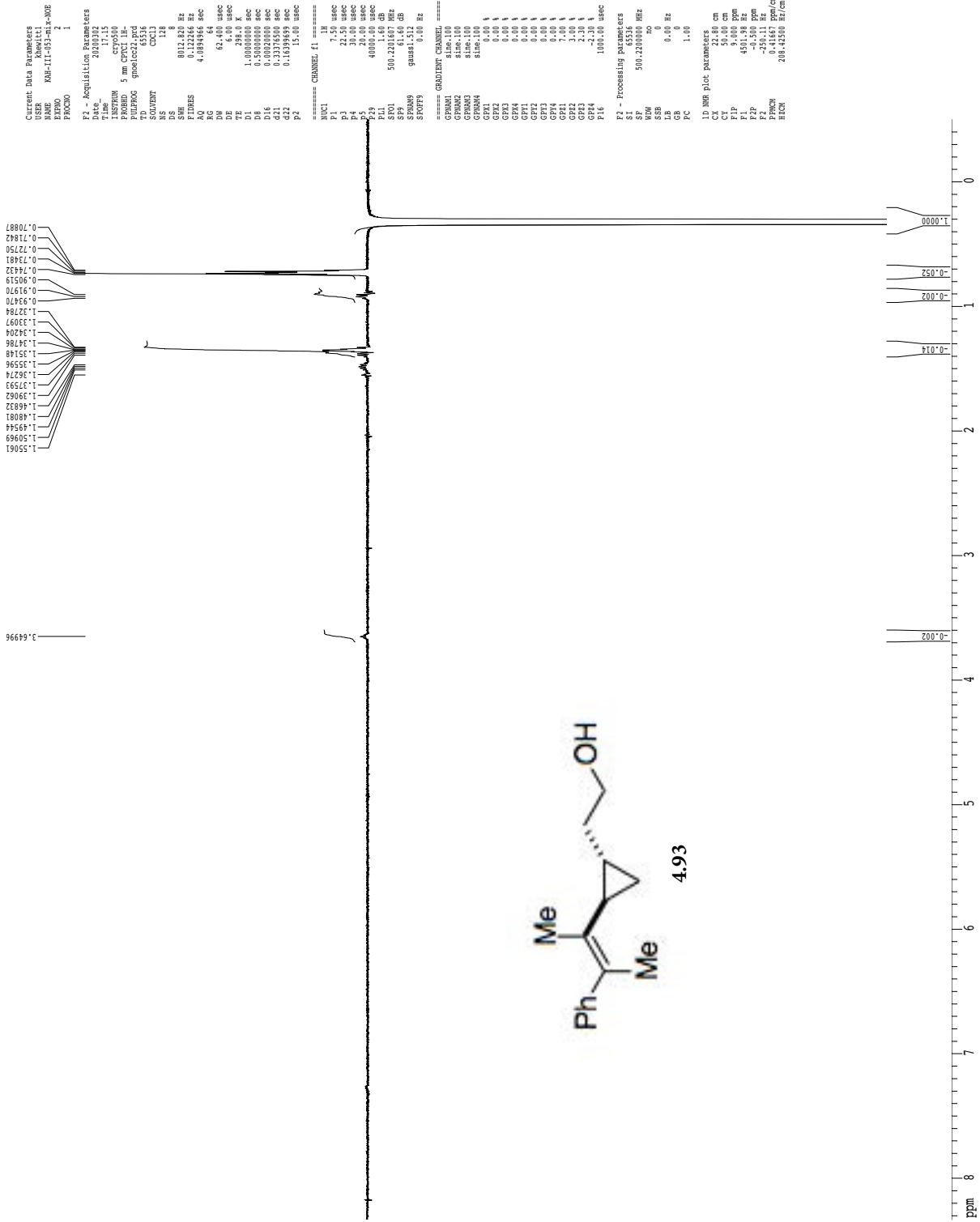
F1 - Processing parameters
SI           1024
MC2          OF
SF           500.2200000 MHz
WDW          SINE
SSB          0
LB           0.00 Hz
GB           0

2D NMR plot parameters
CX2          15.00 cm
CX1          15.00 cm
F2PLO        8.983 ppm
F2FLO        4493.36 Hz
F2PHI        -0.483 ppm
F2H1         -241.49 Hz
F2H2         -241.49 Hz
F2LO         4493.36 ppm
F2H1         -0.483 ppm
F2H2         -241.49 Hz
F2PPMCM     0.65104 ppm/cm
F2FPCMCN    315.65659 Hz/cm
F2HPCMCN    0.65104 ppm/cm
F2FHCNCN    315.65659 Hz/cm
  
```



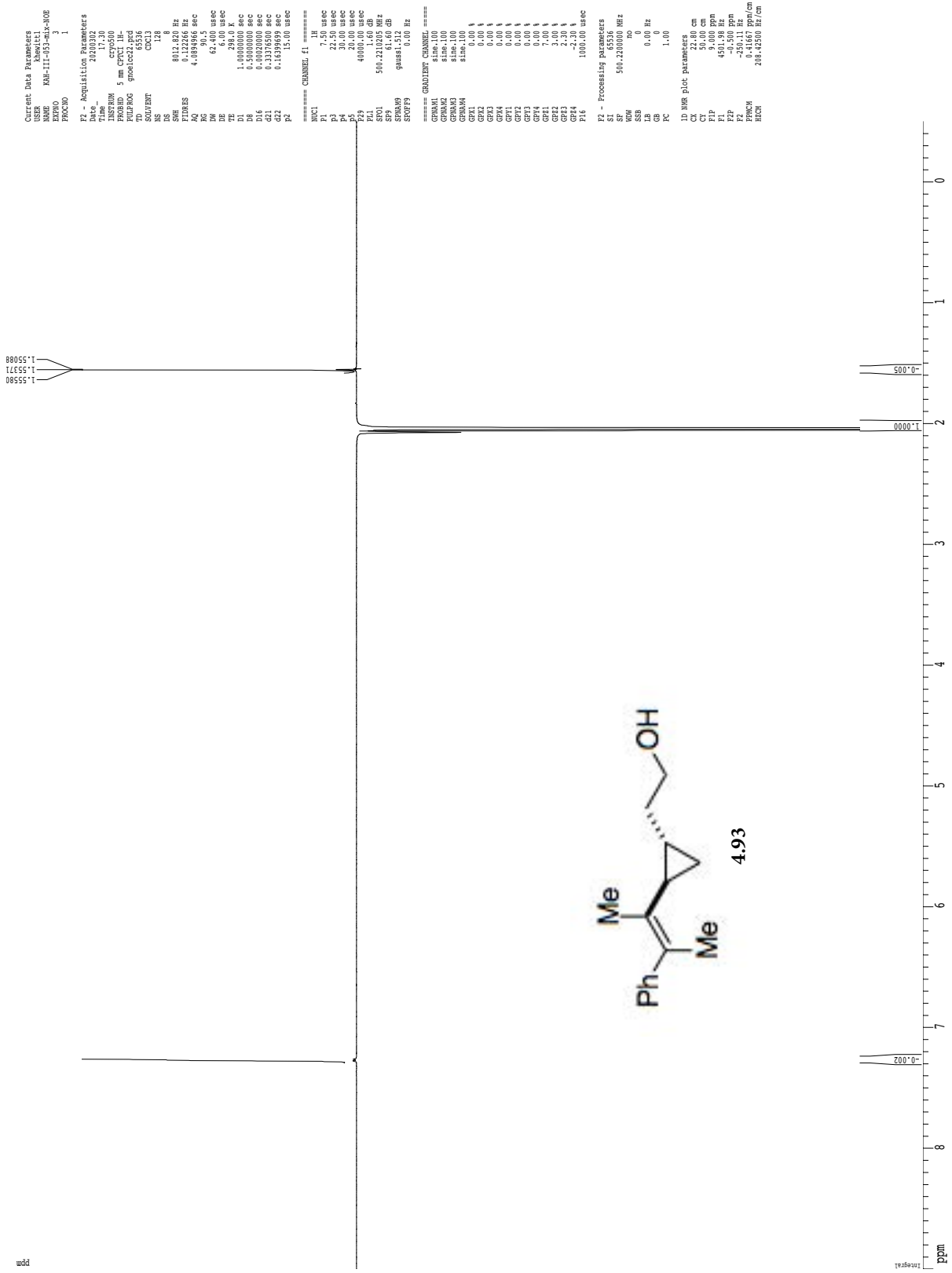
gnoc

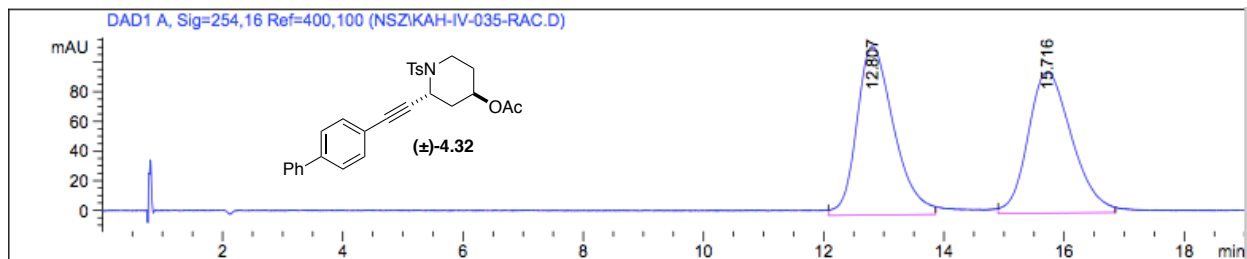
ppm



gnoc

ppm

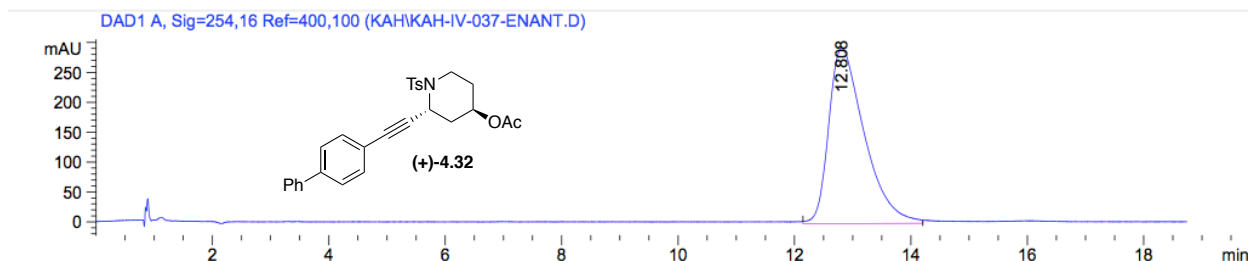




Signal 1: DAD1 A, Sig=254,16 Ref=400,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.807	VV	0.5061	4867.51563	113.55048	50.3655
2	15.716	VV	0.5935	4796.87646	95.08912	49.6345

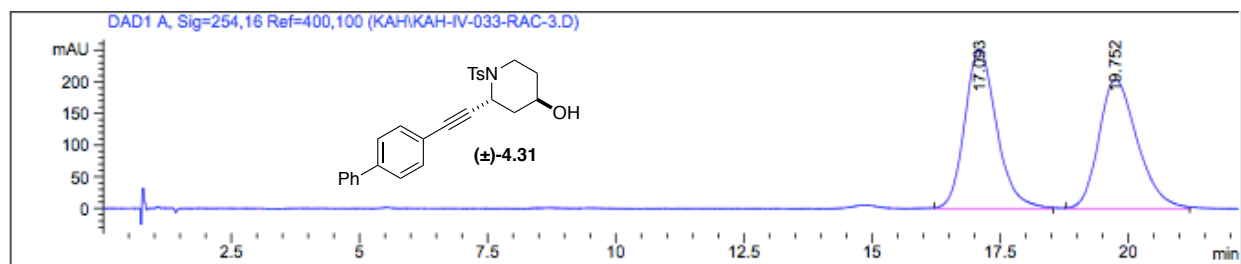
Totals : 9664.39209 208.63960



Signal 1: DAD1 A, Sig=254,16 Ref=400,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.808	VV	0.5042	1.24940e4	293.16055	100.0000

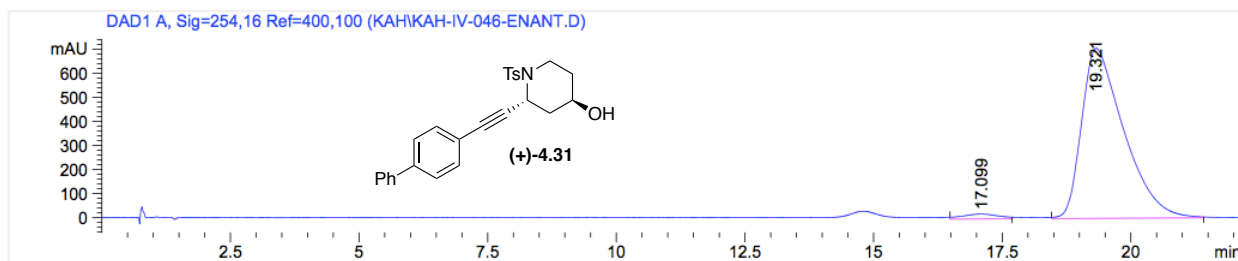
Totals : 1.24940e4 293.16055



Signal 1: DAD1 A, Sig=254,16 Ref=400,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.093	VV	0.6216	1.09056e4	252.66681	50.5481
2	19.752	VV	0.7028	1.06691e4	204.44299	49.4519

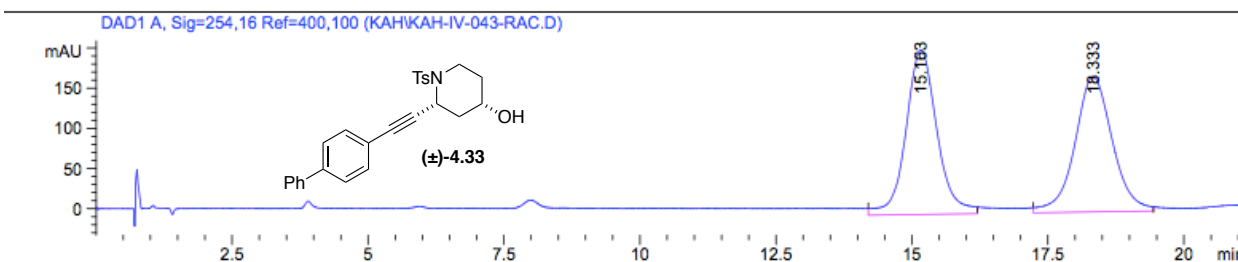
Totals : 2.15747e4 457.10980



Signal 1: DAD1 A, Sig=254,16 Ref=400,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.099	VV	0.5787	1028.28625	20.91209	2.4860
2	19.321	VB	0.6717	4.03350e4	707.90094	97.5140

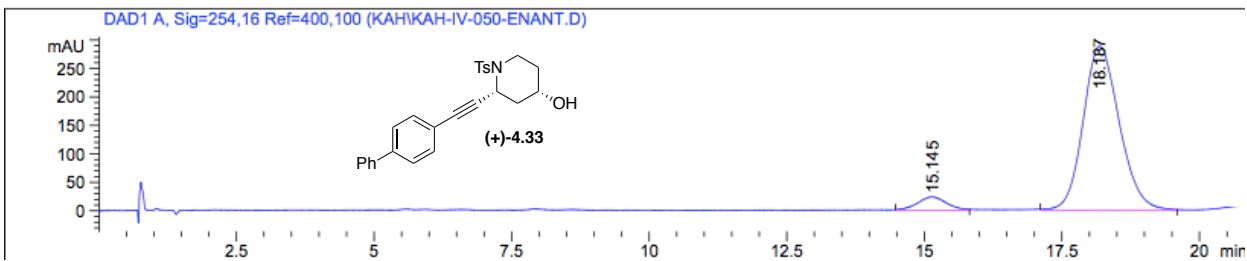
Totals : 4.13632e4 728.81303



Signal 1: DAD1 A, Sig=254,16 Ref=400,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.163	VB	0.5563	8158.00439	205.00032	50.3242
2	18.333	VV	0.5825	8052.88818	168.53467	49.6758

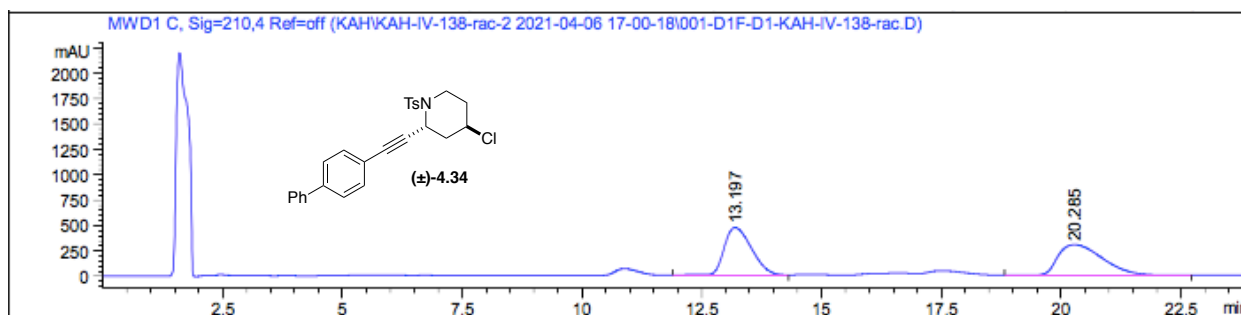
Totals : 1.62109e4 373.53499



Signal 1: DAD1 A, Sig=254,16 Ref=400,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.145	VV	0.4335	856.49738	23.32073	6.1296
2	18.187	VV	0.6261	1.31167e4	288.13208	93.8704

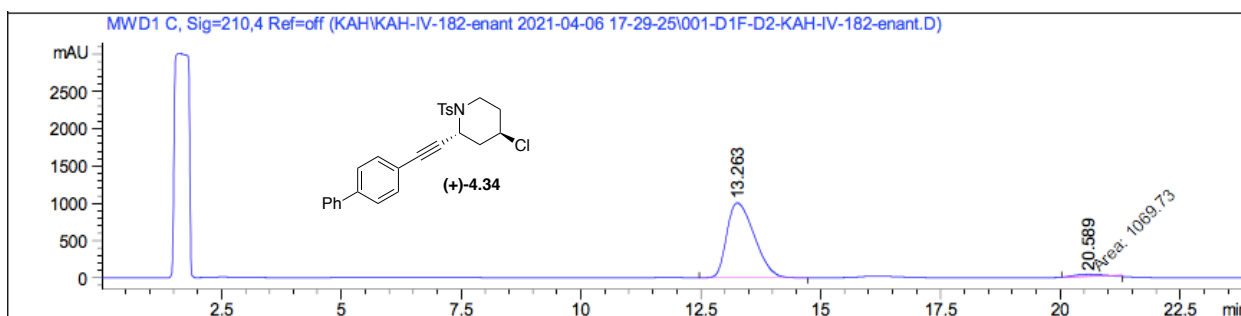
Totals : 1.39732e4 311.45281



Signal 3: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.197	VB R	0.6269	1.85477e4	467.39673	49.0205
2	20.285	BB	1.0257	1.92889e4	300.15744	50.9795

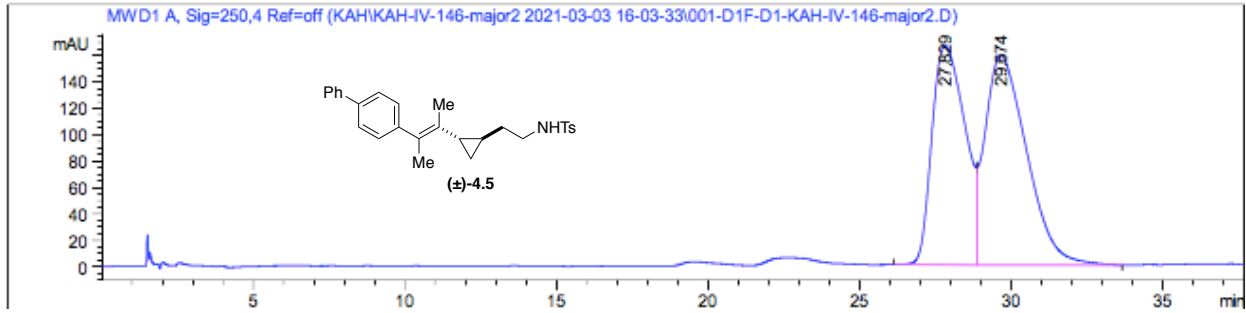
Totals : 3.78366e4 767.55417



Signal 3: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.263	VB	0.6339	3.99296e4	1004.20569	97.3909
2	20.589	MM	0.6448	1069.72583	27.65214	2.6091

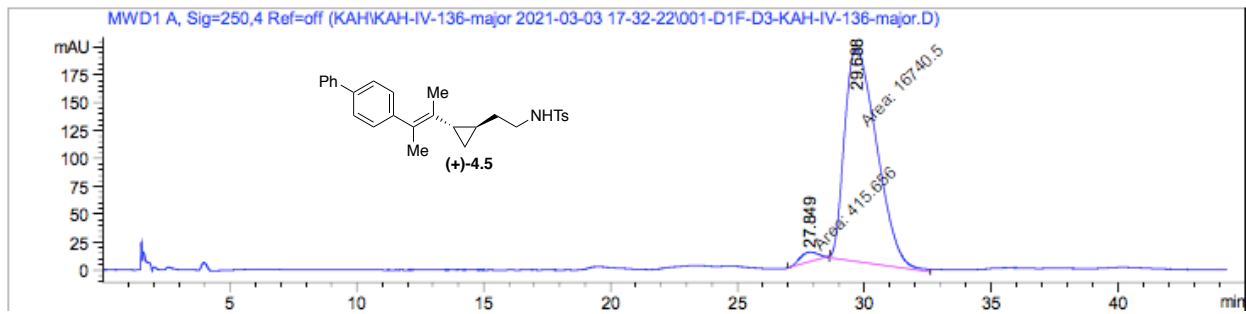
Totals : 4.09993e4 1031.85783



Signal 1: MWD1 A, Sig=250,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.829	VV R	1.0909	1.25773e4	166.57452	45.1638
2	29.674	VV R	1.3485	1.52708e4	159.27989	54.8362

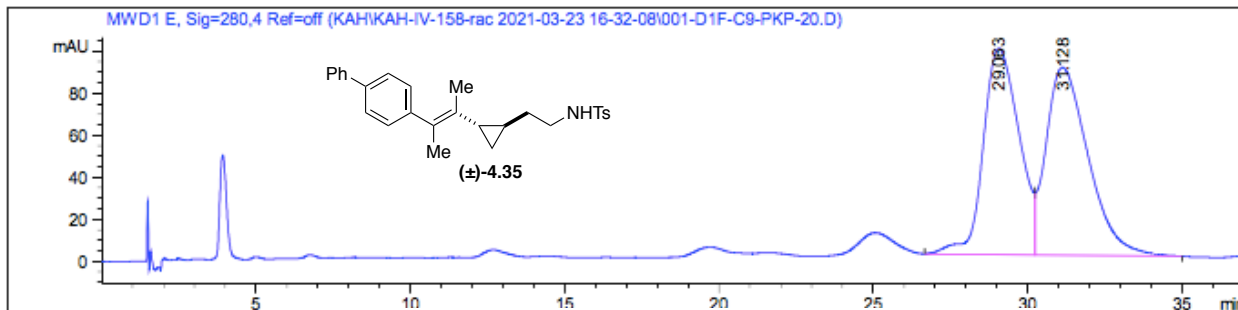
Totals : 2.78481e4 325.85442



Signal 1: MWD1 A, Sig=250,4 Ref=off

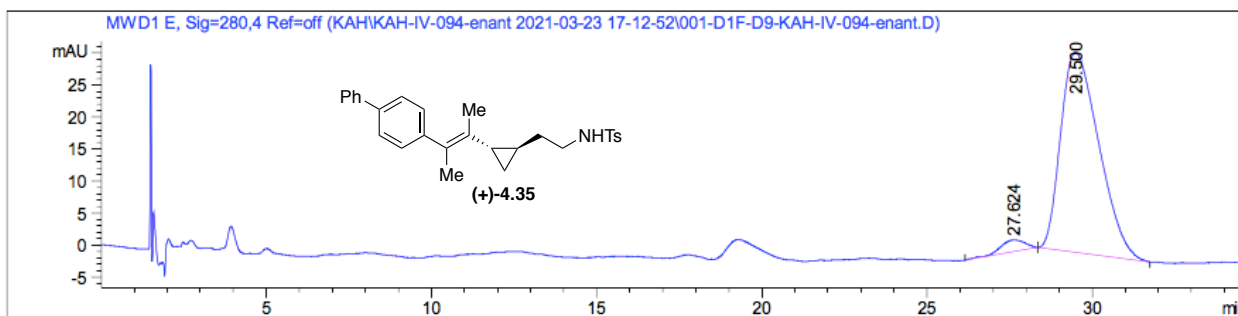
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.849	MM	0.5898	415.65552	8.55463	2.4228
2	29.688	MM	1.4600	1.67405e4	191.09866	97.5772

Totals : 1.71562e4 199.65329



Signal 5: MWD1 E, Sig=280,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.063	BV	1.1694	7931.67773	98.08938	48.2122
2	31.128	VB	1.3026	8519.92676	89.34296	51.7878
Totals :				1.64516e4	187.43233	



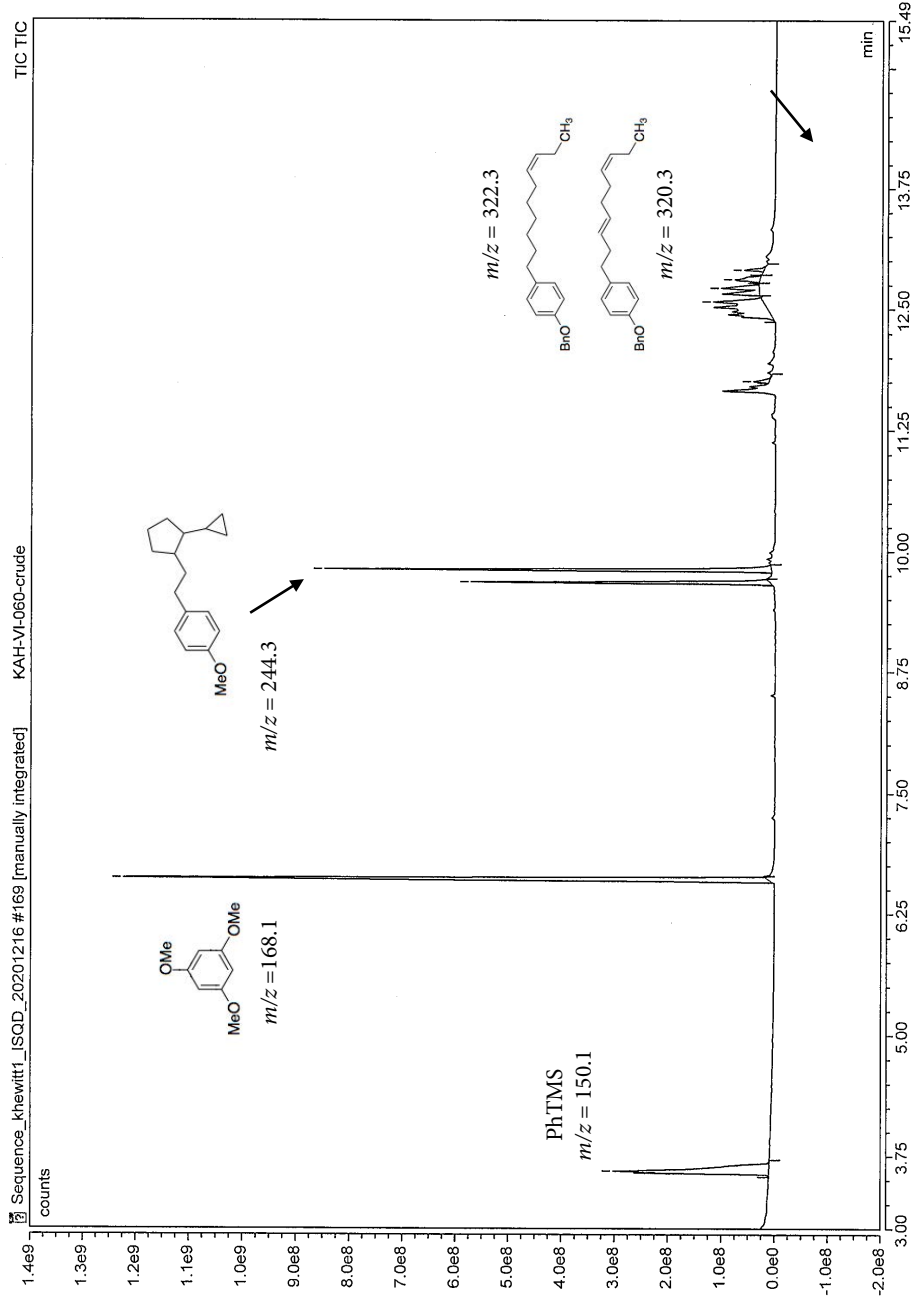
Signal 5: MWD1 E, Sig=280,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.624	VB R	0.6253	97.29442	1.85081	3.7297
2	29.500	BB	1.0528	2511.35840	31.27596	96.2703
Totals :				2608.65282	33.12677	

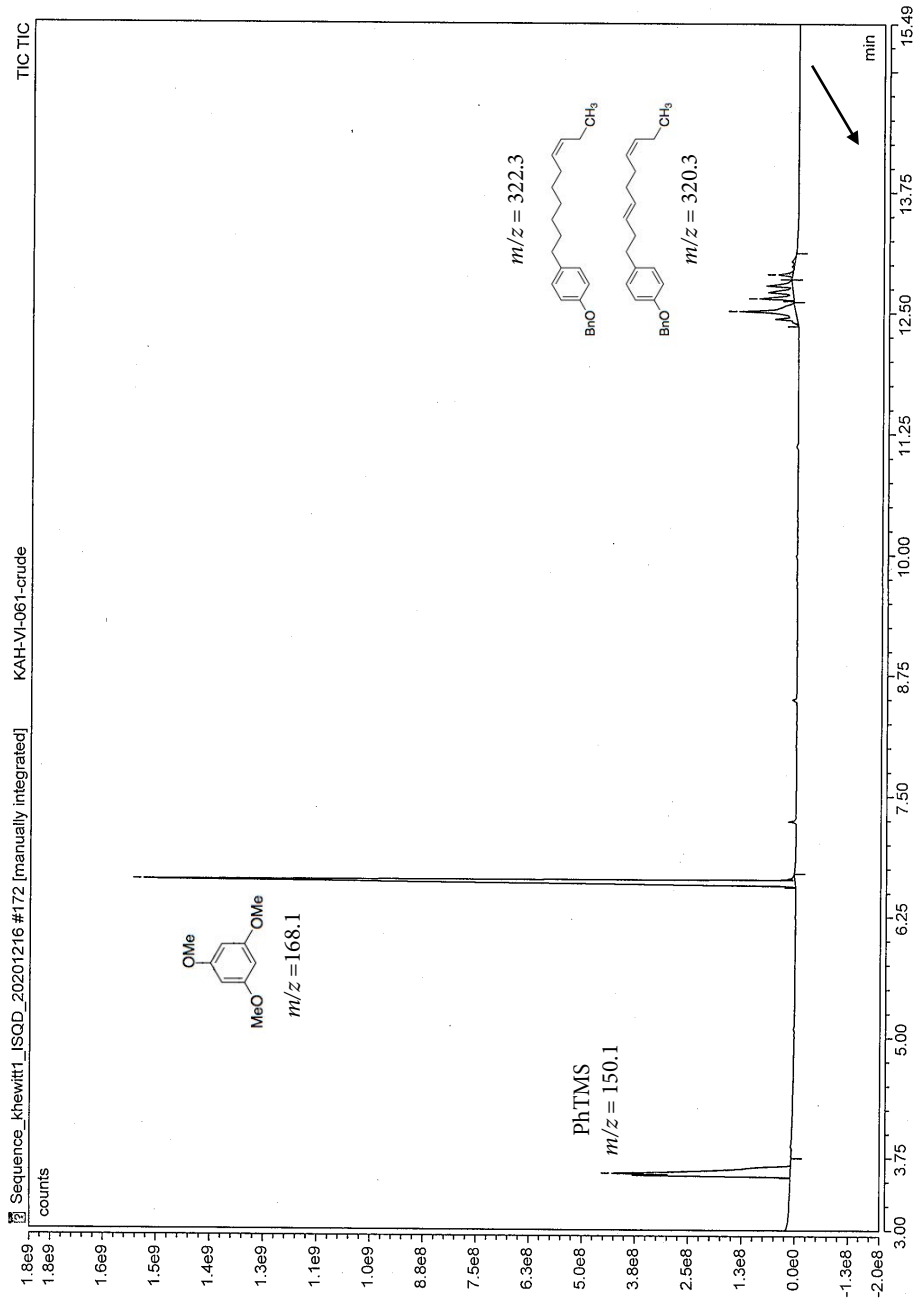


APPENDIX A.4

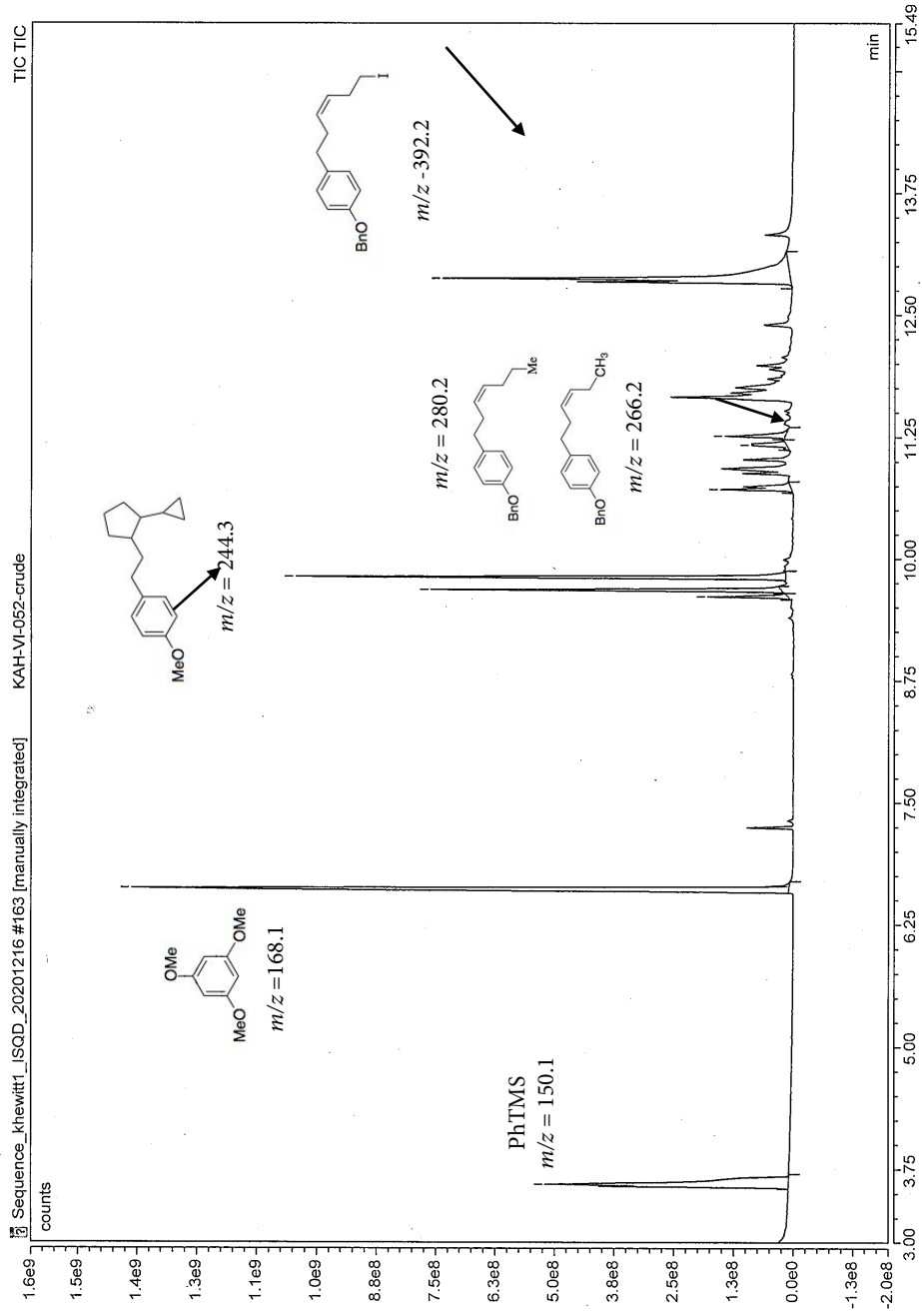
$^1\text{H}$ ,  $^{13}\text{C}$ , COSY, HMQC NMR SPECTRA, GCMS SPECTRA FOR CHAPTER 5



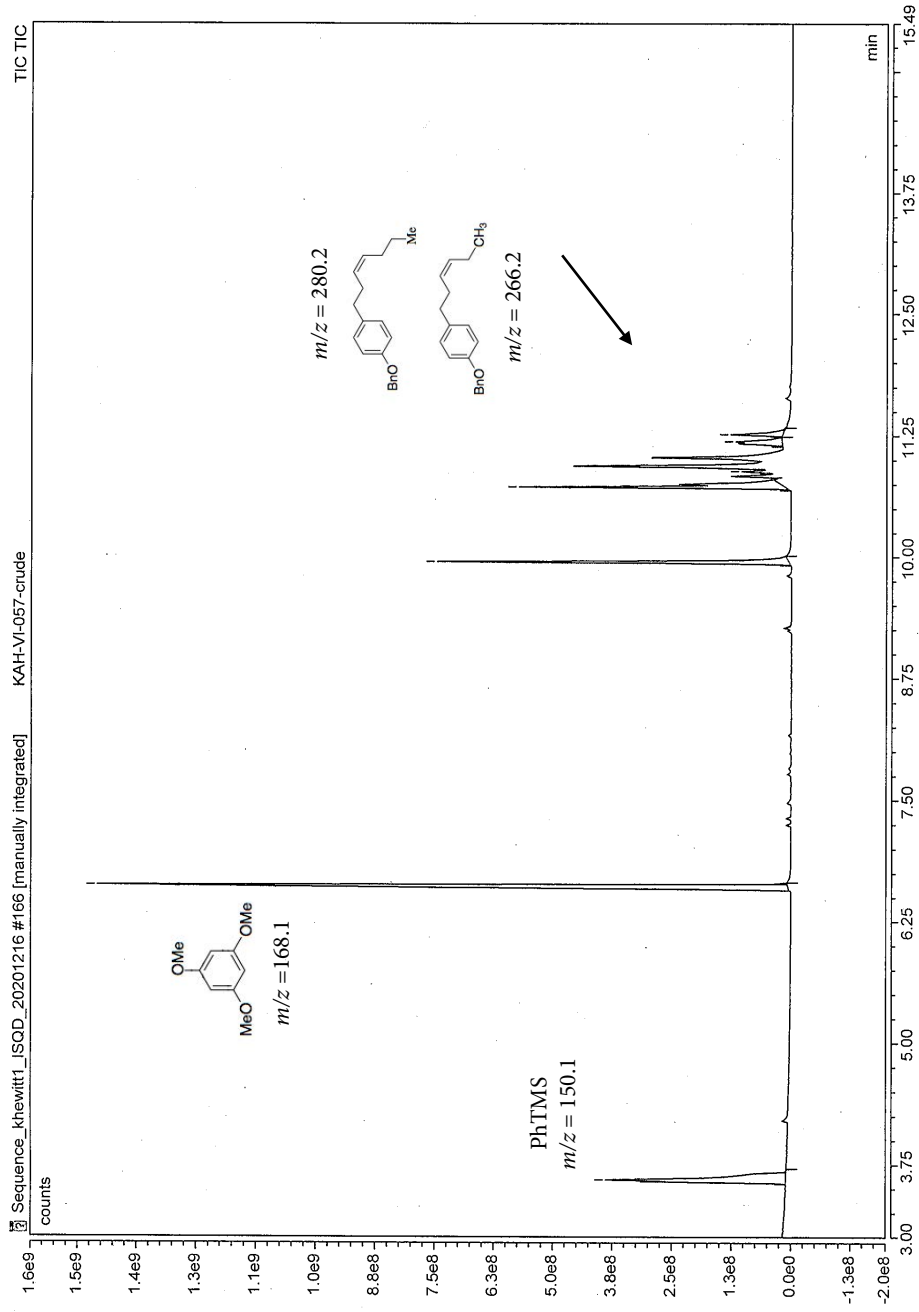
Sequence: Sequence\_khewitt1\_ISQD\_20201216  
Injection #172: KAH-VI-061-crude



Sequence: Sequence\_khewitt1\_ISQD\_20201216  
Injection #163: KAH-VI-052-crude

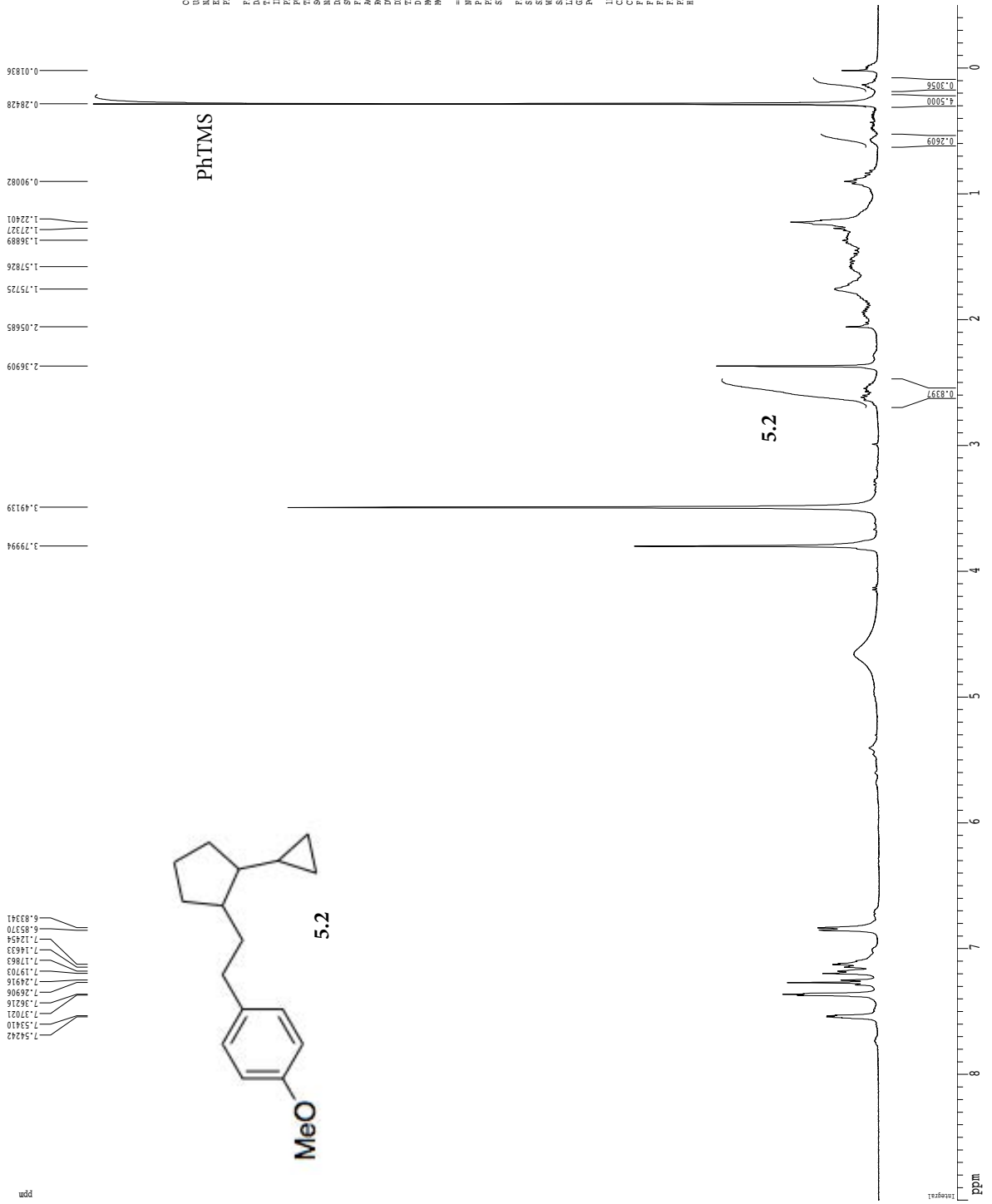
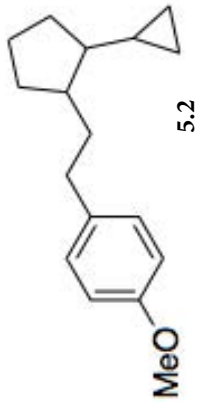


Sequence: Sequence\_khewitt1\_ISOD\_20201216  
Injection # 166: KAH-VI-057-crude



Sequence\_khewitt1\_ISOD\_20201216 #166 [manually integrated] KAH-VI-057-crude

1H spectrum



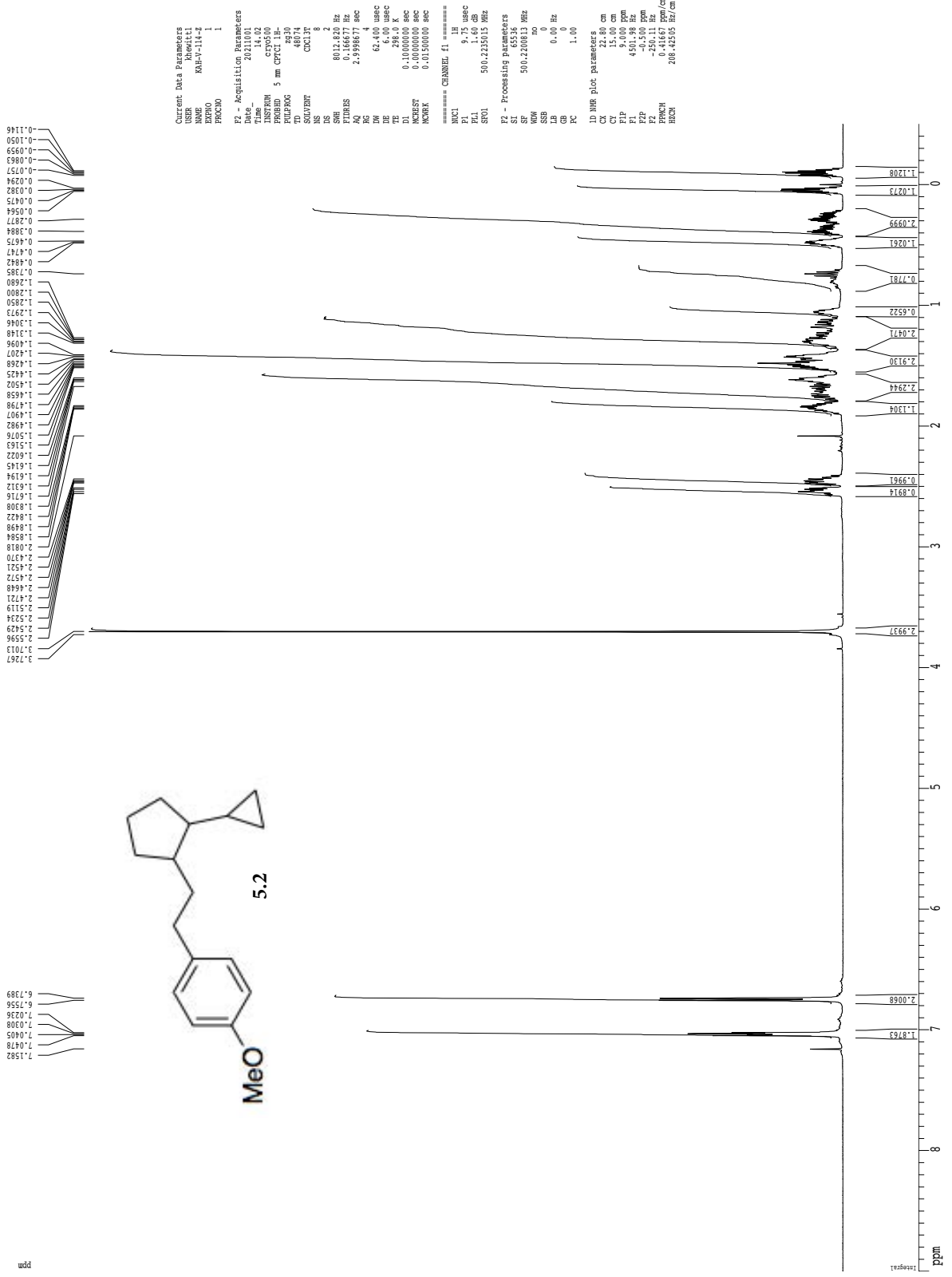
Current Data Parameters  
 USER hewitt1  
 NAME 10H-III-04 crude  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 201211  
 Time 14:31  
 INSTRUM dx400  
 PROBDI 3 mm QNP H/F/P  
 PULPROG zgpg30  
 TD 38460  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6410.256 Hz  
 FIDRES 0.146673 Hz  
 AQ 2.995929 sec  
 RG 718.000  
 IN 718.000 MHz  
 DE 4.50 MHz  
 TE 298.1 K  
 F2 400.130000 MHz  
 F1 100.626126 MHz  
 NUC1 13C  
 NUC2 1H  
 P1 12.00 MHz  
 PL 0.00 dB  
 SFO1 400.1328009 MHz

F2 - Processing parameters  
 SI 65536  
 SF 400.1300175 MHz  
 DS 8  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 2.00

1D NMR plot parameters  
 CX 22.80 cm  
 CY 15.00 cm  
 CZ 9.000 cm  
 FI 360.117 Hz  
 F2 -0.500 Hz  
 F3 0.41667 MHz/cm  
 PRGCMV 166.72884 Hz/cm  
 HZCN

1H spectrum



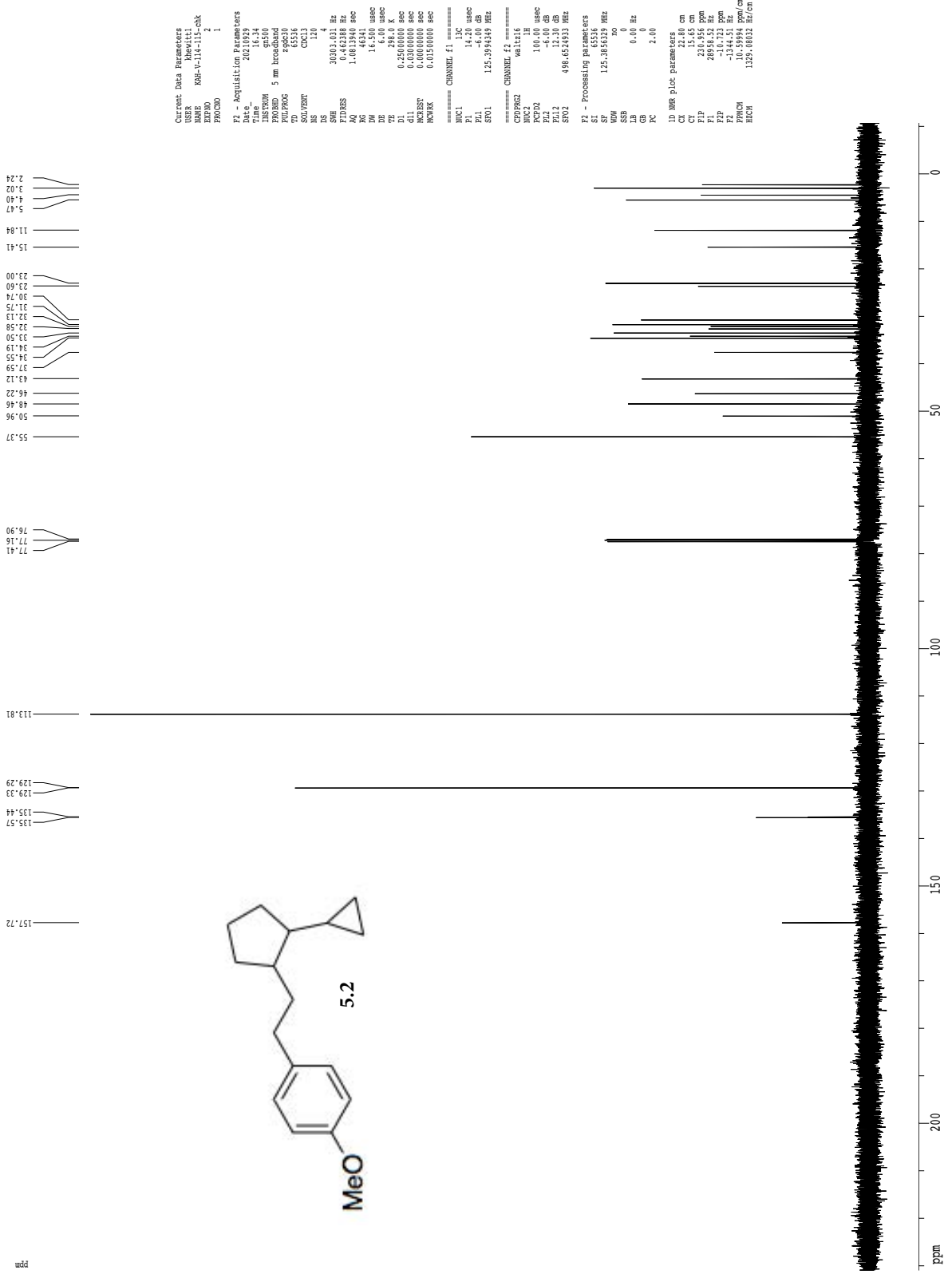
Current Data Parameters  
 USER hewlett1  
 NAME RAH-7-114-2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20210101  
 Time 12:00:00  
 INSTRUM cty0500  
 PROBEID 5 mm CPCLP 1H-  
 PULPROG zgpg30  
 PC 4.22  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6073.82 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.9998677 sec  
 RG 4  
 DW 62.400 usec  
 DE 1.900 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 MCREST 0.00000000 sec  
 PCNMR 0.01000000 sec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 9.40 usec  
 PL1 0.00 dB  
 SFO1 500.225015 MHz

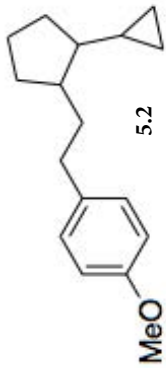
F2 - Processing parameters  
 SI 65536  
 SF 500.2250813 MHz  
 WDW no  
 SSB 0  
 CB 0.0 Hz  
 GB 0  
 PC 1.00

ID MRB plot parameters  
 CT 15.00 cm  
 CY 15.00 cm  
 FIP 9.000 ppm  
 FI 4501.88 Hz  
 F2 -750.31 Hz  
 F2 0.41667 ppm/cm  
 HZCM 208.42505 Hz/cm

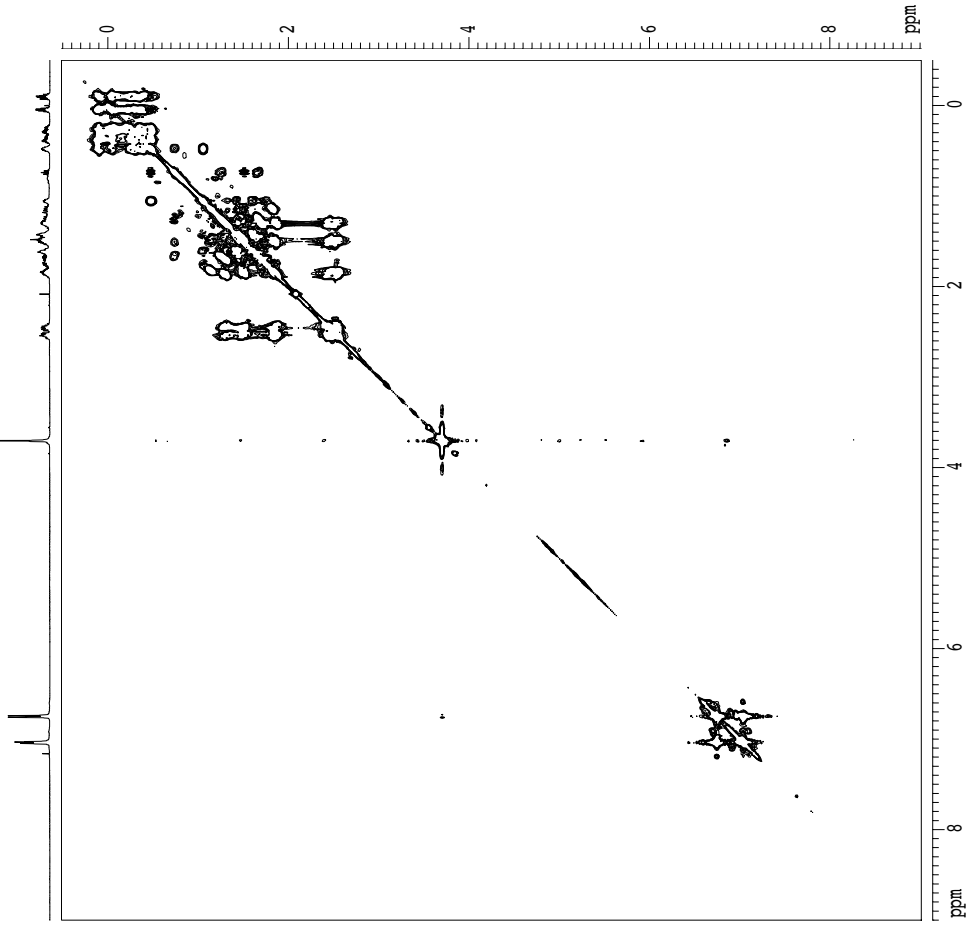
13C spectrum with 1H decoupling







gcossy60



Current Data Parameters  
 AcqNo: 1  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 201100  
 Time 14.04  
 INSTRUM crys500  
 PROBD 5 mm CPCLH-  
 PULPROG conjugate2d  
 SOLVENT CDCl3  
 NS 1  
 DS 16  
 SWH 8012.820 Hz  
 FIDRES 0.1278452 Hz  
 AQ 0.1278452 Hz  
 RG 35.9  
 DW 62.400 usec  
 DE 6.00 usec  
 DS 16  
 AS 0.00000000 sec  
 AD 0.00000000 sec  
 D1 1.00000000 sec  
 d13 0.00000000 sec  
 D16 0.00020000 sec  
 INO 0.0001480 sec

==== CHANNEL f1 =====  
 NUC1 1H  
 P1 9.75 usec  
 PL1 1.00 dB  
 SFO1 500.125015 MHz

==== GRADIENT CHANNEL =====  
 GPMAX SINGLO.100  
 GPMWZ SINGLO.100  
 GPZ1 0.00 %  
 GPZ2 0.00 %  
 GPZ3 0.00 %  
 GPZ4 0.00 %  
 GPZ5 17.00 %  
 GPZ6 17.00 %  
 P16 1000.000 usec

F1 - Acquisition parameters  
 RD0 61  
 SFO1 500.125 MHz  
 F1RES 15.655040 Hz  
 SW 16.018 ppm  
 FWHMDE QF

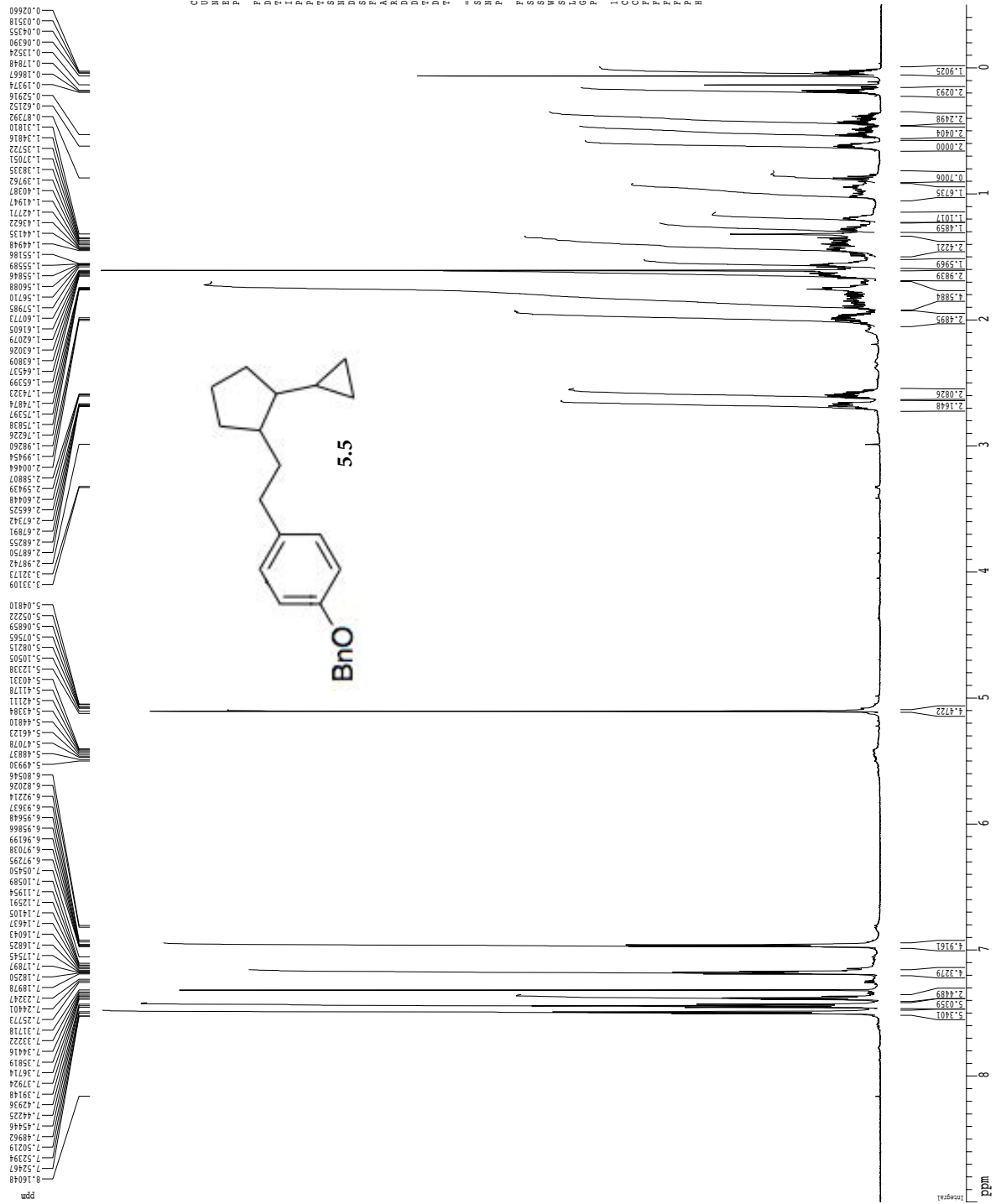
F2 - Processing parameters  
 SI 102  
 SF 500.1200813 MHz  
 SINE SINE  
 WDW 0  
 SSB 0  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 102  
 SF 500.1200813 MHz  
 SINE SINE  
 WDW 0  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 ZC0 0  
 ZC1 450.98 Hz  
 FZ0 0  
 FZ1 -0.500 ppm  
 FZ2HI -250.11 Hz  
 F1LO 9.012 ppm  
 F1HI 460.98 Hz  
 F1LO -0.515 ppm  
 F1HI -251.49 Hz  
 F2PAPCH 0.63333 ppm/cm  
 F2PACH 316.80606 Hz/cm  
 F1PAPCH 0.56351 ppm/cm  
 F1PACH 317.09350 Hz/cm



1H spectrum



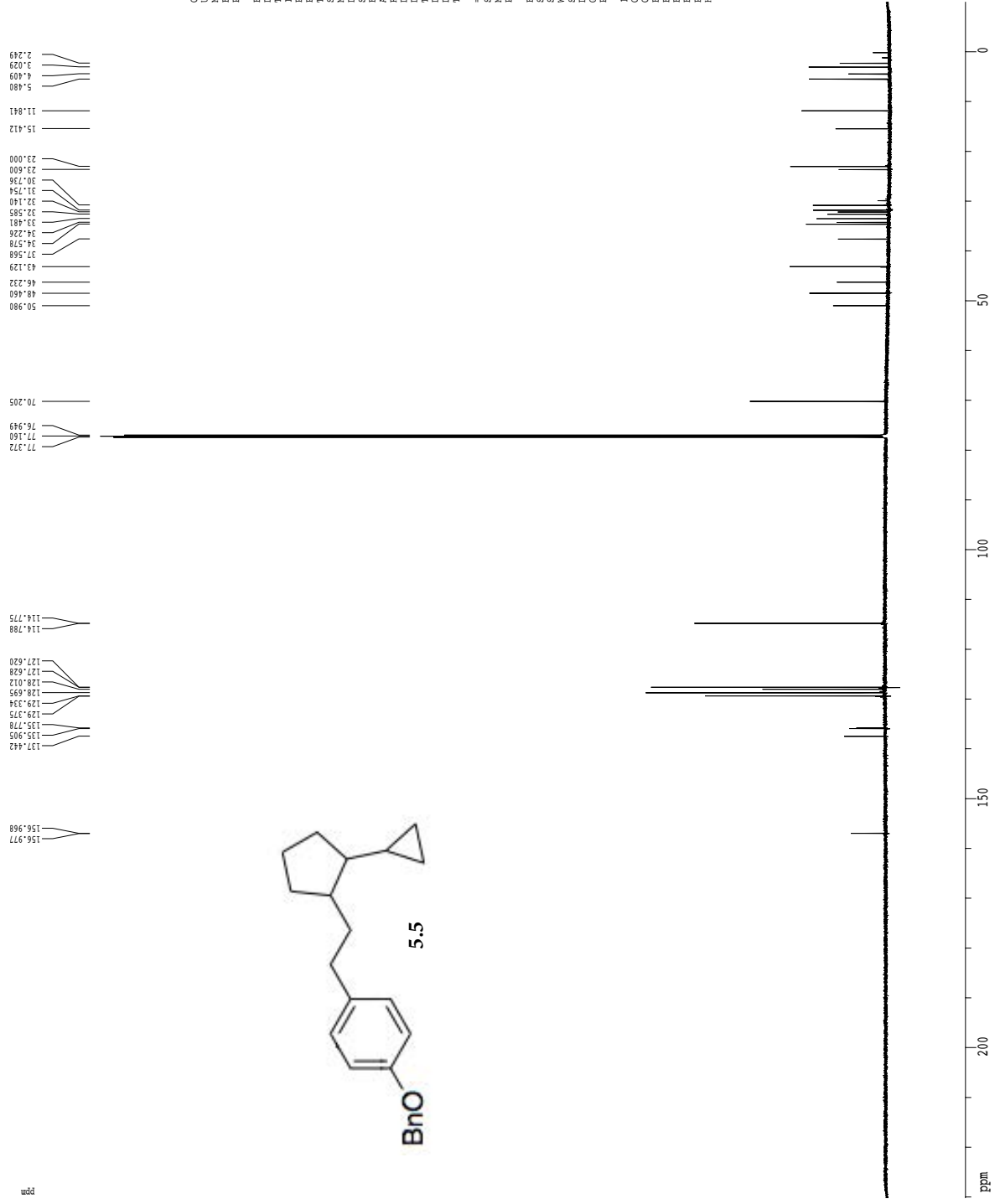
Current Data Parameters  
 Date\_ 20220313  
 Time\_ 16:50  
 INSTRUM av600  
 PULPROG zgpg30  
 FIDRES 0.098442 Hz  
 AQ 5.0998979 sec  
 DD 52.000 usec  
 DE 14.23 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 D2 1.00000000 sec  
 T20 1.00000000 sec

==== CHANNEL f1 =====  
 SFO1 600.1342009 MHz  
 P1 9.50 usec  
 PL 0.00 dB

F2 - Processing Parameters  
 SI 6556  
 SF 600.1300000 MHz  
 NWDW 00  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

ID NMR Plot parameters  
 CX 22.86 cm  
 F1 5403.17 Hz  
 F2 5403.17 Hz  
 F3 5403.17 Hz  
 F4 5403.17 Hz  
 F5 5403.17 Hz  
 F6 5403.17 Hz  
 F7 5403.17 Hz  
 F8 5403.17 Hz  
 F9 5403.17 Hz  
 F10 5403.17 Hz  
 F11 5403.17 Hz  
 F12 5403.17 Hz  
 F13 5403.17 Hz  
 F14 5403.17 Hz  
 F15 5403.17 Hz  
 F16 5403.17 Hz  
 F17 5403.17 Hz  
 F18 5403.17 Hz  
 F19 5403.17 Hz  
 F20 5403.17 Hz

13C spectrum



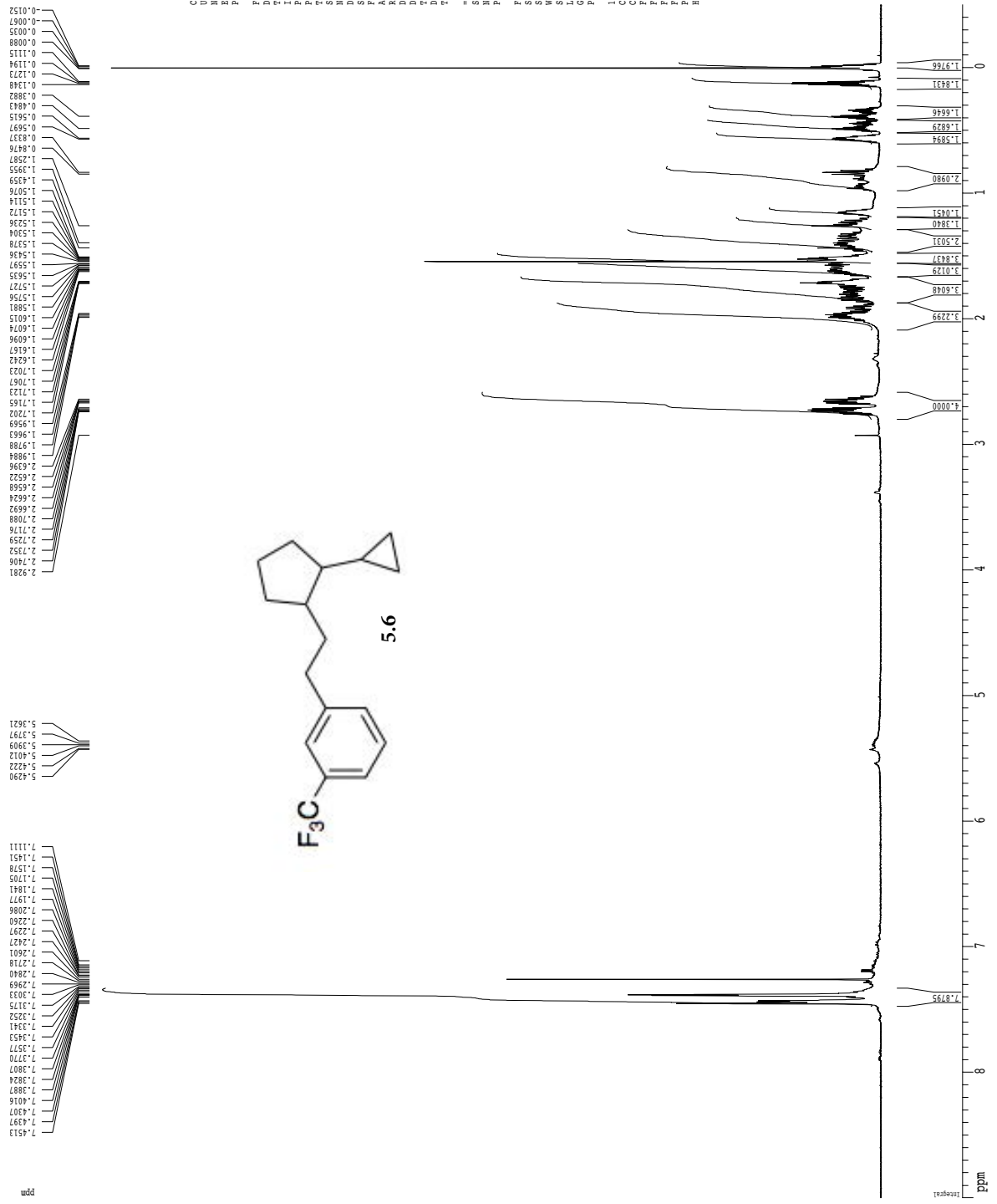
Current Data Parameters  
 Date\_ 20220313  
 Time\_ 16:56  
 INSTRUM av600  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 814  
 DS 4  
 SFO 36231.883 Hz  
 FIDRES 0.552855 Hz  
 AQ 0.504468 sec  
 RG 260  
 DE 13.000 usec  
 TE 297.5 K  
 D1 0.4000001 sec  
 D11 0.5000001 sec  
 TDO 1

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 10.10 usec

F2 - Processing parameters  
 SF 150.907542 MHz  
 SFO 36231.883 Hz  
 MM no  
 SSB 0  
 GB 0.00 Hz  
 PC 1.00

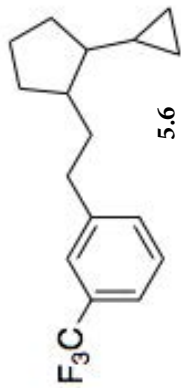
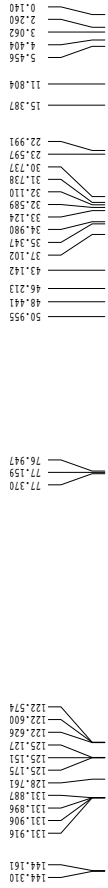
ID NMR Plot parameters  
 X 25.00 cm  
 Y 15.00 cm  
 FIP 230.147 ppm  
 F1 34724.77 Hz  
 F2 -150.91 ppm  
 FWHM 10.55074 ppm/cm  
 RECN 1589.11786 Hz/cm

h1.c



c13.c

ppm

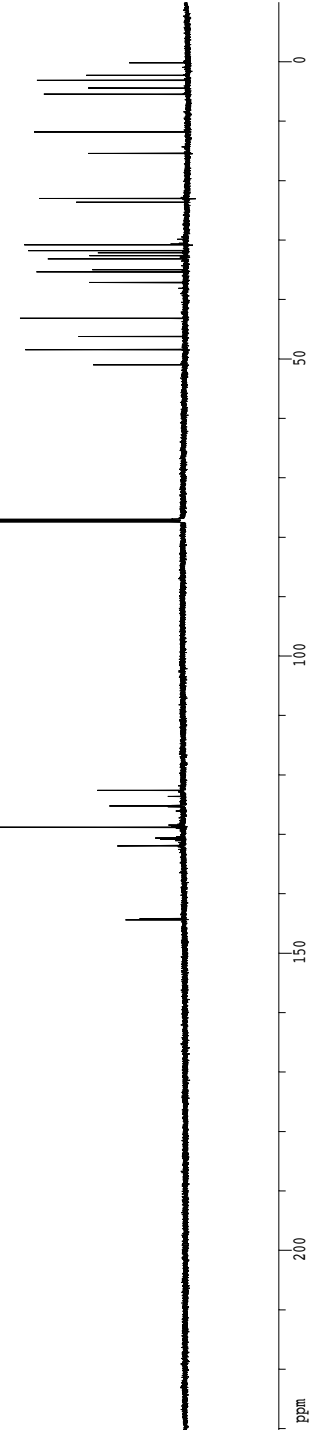


Current Data Parameters  
Date\_ 20210802  
Time\_ 16:48  
INSTRUM av600  
PROBHD 5 mm CPBPR0  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
DS 500  
SWH 36231.883 Hz  
AQ 0.552855 sec  
RG 260  
DE 13.000 usec  
TE 298.0 K  
D1 0.40000001 sec  
DELTA 1.00000001 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 130.319400 MHz  
PC 10.10 usec

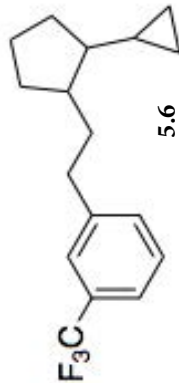
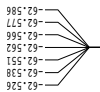
F2 - Processing parameters  
SI 150.9027931 MHz  
SF 150.9027931 MHz  
WDW no  
SSB 0  
GB 0.00 Hz  
PC 1.00

ID NMR Plot parameters  
X 25.00 cm  
Y 15.00 cm  
Z 15.00 cm  
FID 230.154 ppm  
F1 34730.87 Hz  
F2 -150.91 ppm  
F3 -150.91 ppm  
PRNCH 10.53074 ppm/cm  
RECN 1589.11768 Hz/cm

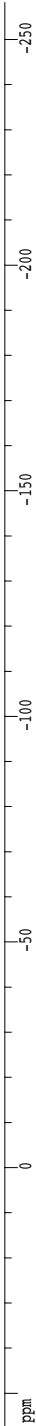


f19.c

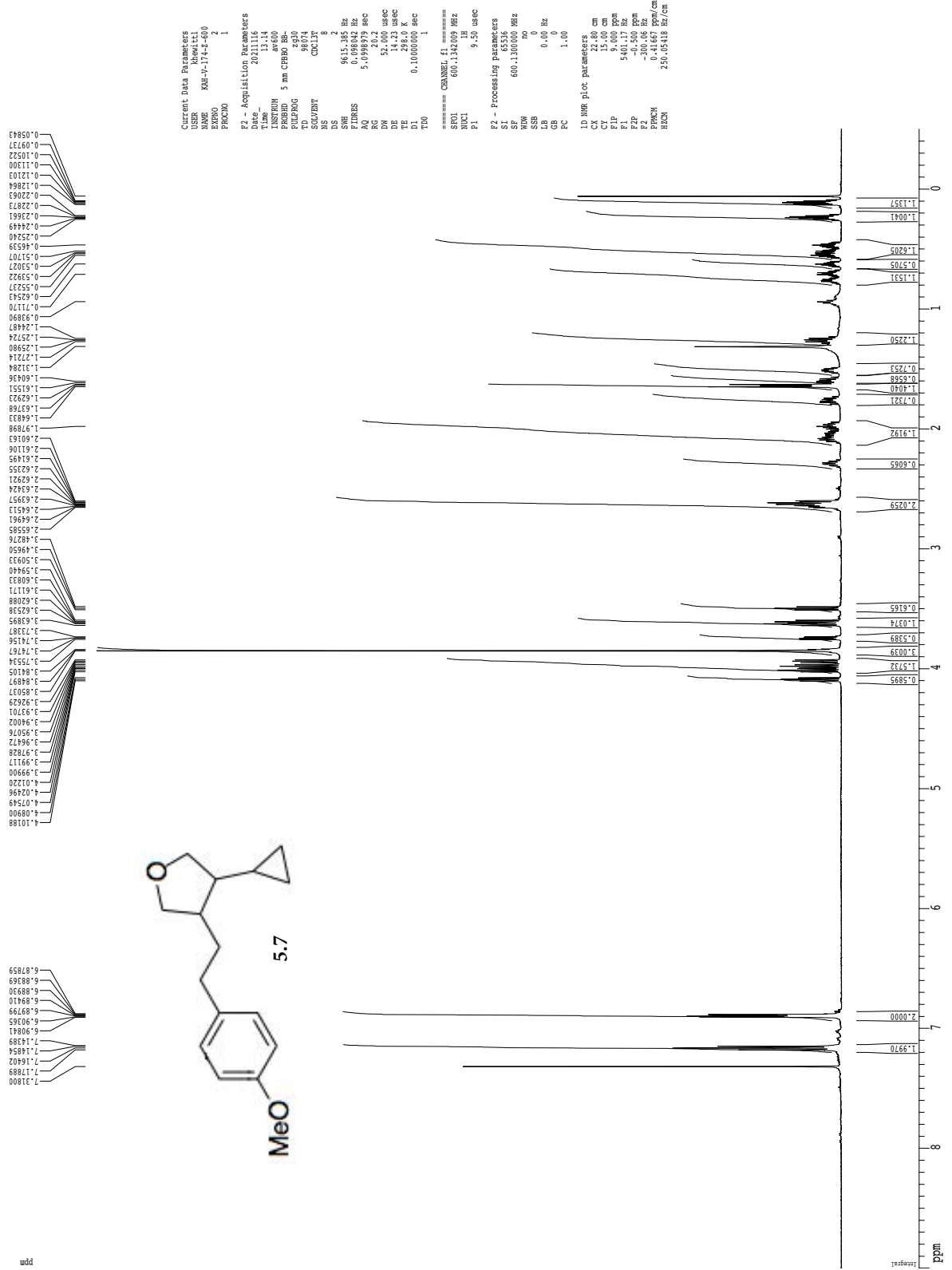
Hz



Current Data Parameters  
Date\_ 20210802  
Time\_ 16:43  
INSTRUM av600  
PROBHD 5 mm QNP1H30  
PULPROG zgpg30  
TD 331072  
SOLVENT CDCl3  
DS 6  
SS 6  
SFO 178571.422 Hz  
FIDRES 1.362392 Hz  
AQ 0.3670516 sec  
RG 327.500  
DE 2.800 usec  
TE 298.0 K  
D1 3.0000000 sec  
D2 1.0000000 sec  
===== CHANNEL f1 =====  
SFO1 564.629196 MHz  
NUC1 13C  
P1 18.75 usec  
===== F2 - Processing Parameters =====  
SI 331072  
SF 564.629196 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00  
===== ID NMR Plot parameters =====  
CX 22.80 cm  
FID 1.0000000000000000  
F1P 58.053 ppm  
F1 32783.488 Hz  
F2P -238.178 ppm  
F2 -145789.578 Hz  
FREQH 7832.06008 MHz/cm  
FREQV 7832.06008 MHz/cm

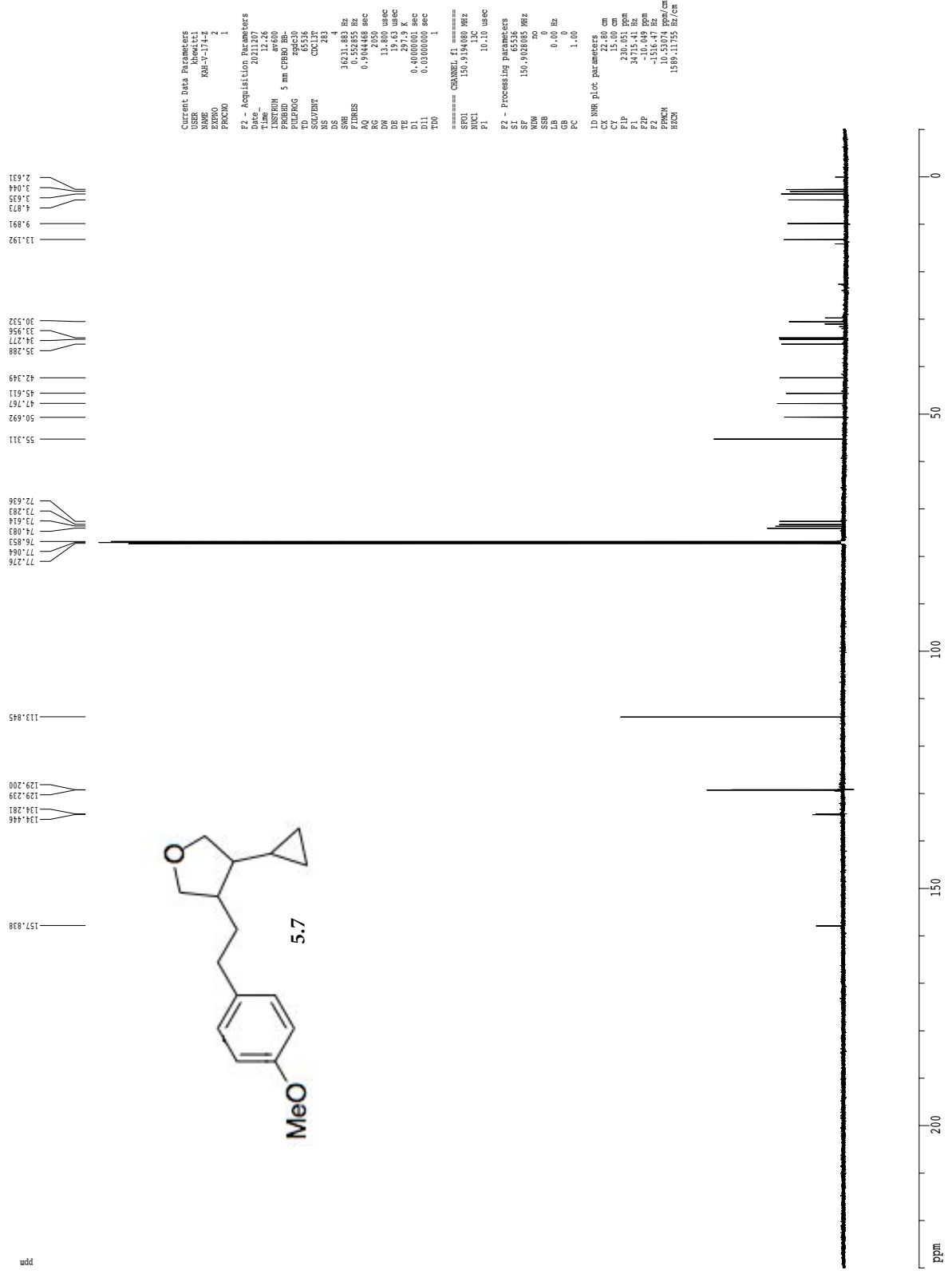


1H spectrum

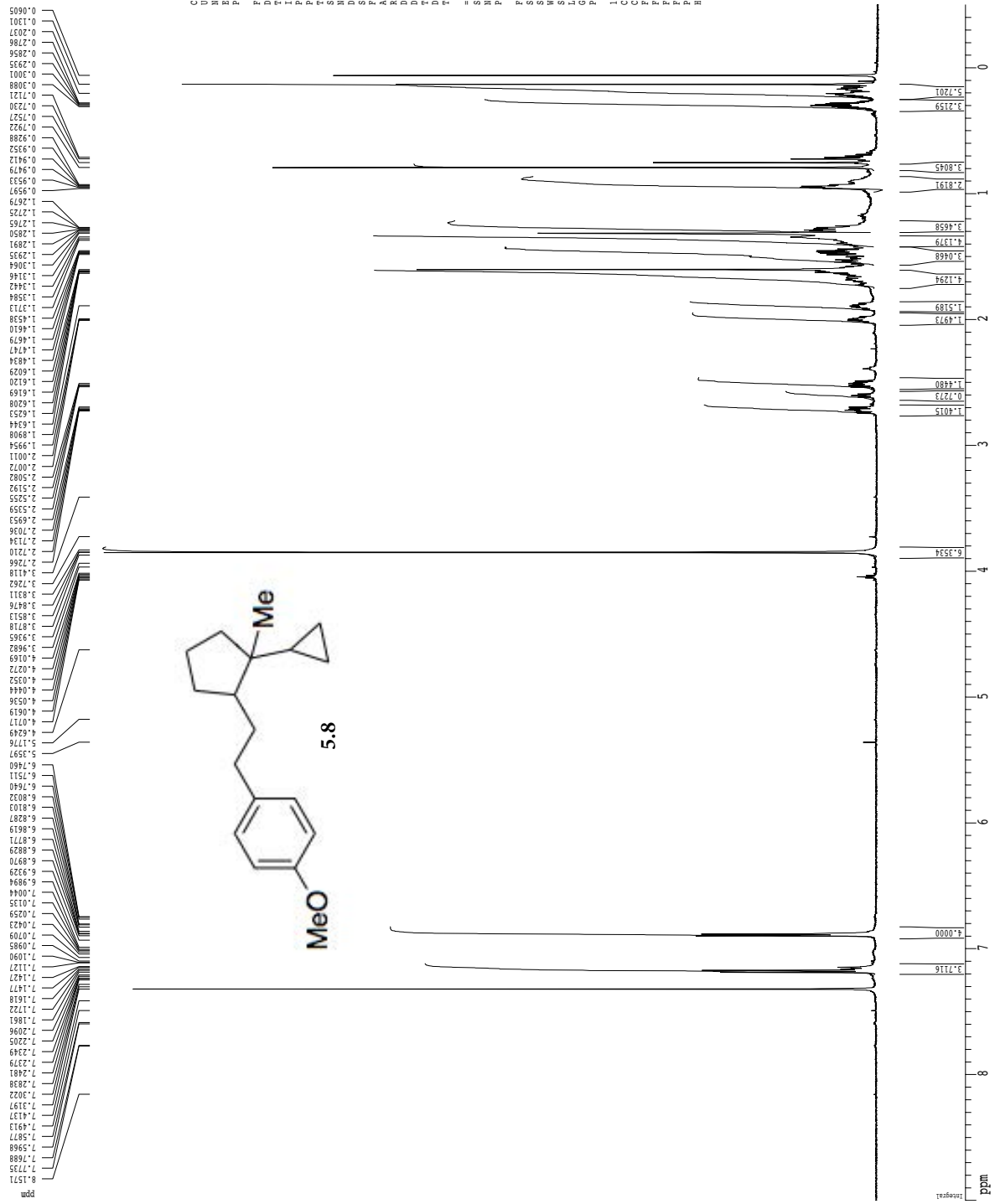




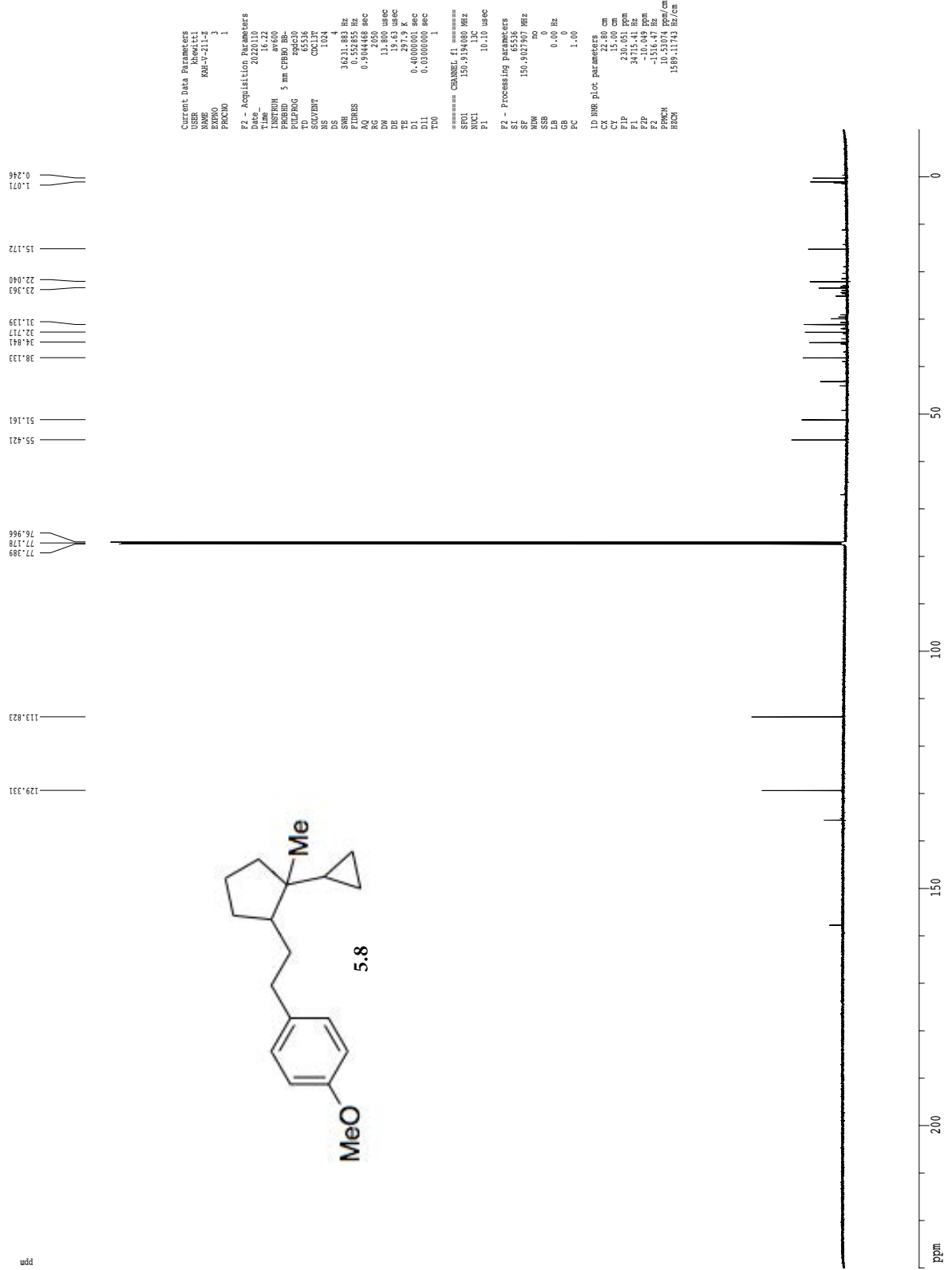
13C spectrum



1H spectrum



13C spectrum



Current Data Parameters  
 USER khewitt1  
 NAME KAH-V-211-2D  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters

Date 20220111  
 Time 11.06  
 INSTRUM cryo500  
 PROBDH 5 mm CPCL1 H-  
 PULPROG cosygp60.prd  
 TD 2048  
 SOLVENT CDCl3  
 NS 16  
 SNR 8012.820 Hz  
 FIDRES 3.912510 Hz  
 AQ 0.12778452 sec  
 RG 724.1  
 DM 62.400 usec  
 DE 6.00 usec  
 HB 29.00 usec  
 HD 0.0000300 sec  
 DI 1.0000000 sec  
 d13 0.0000300 sec  
 D16 0.0002000 sec  
 INO 0.0012480 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*

NUC1 P1 9.75 usec  
 P2 1.60 dB  
 SFO1 500.2235015 MHz

\*\*\*\*\* GRADIENT CHANNEL \*\*\*\*\*

GPRM1 SHSQ10-100  
 GPRM2 SHSQ10-100  
 GPX1 0.00 %  
 GPY1 0.00 %  
 GPZ1 0.00 %  
 GPZ2 17.00 %  
 P16 1000.00 usec

F1 - Acquisition parameters

NUO 512  
 TD 512  
 SFO1 500.2235 MHz  
 FIDRES 15.650040 Hz  
 SN 16.018 ppm  
 FMODE QF

F2 - Processing parameters

SI 1024  
 SF 500.2200307 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters

SI 1024  
 MC2 QF  
 SF 500.2200307 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters

CX1 15.00 cm  
 CX2 15.00 cm  
 F2PLO 9.004 ppm  
 F2LO 4503.78 Hz  
 F2HI 4512.66 Hz  
 F1PLO 9.004 ppm  
 F1LO 4503.78 Hz  
 F1HI 4512.66 Hz  
 F1PHI -0.523 ppm  
 F1PI -261.66 Hz  
 F2PMCH 0.63511 ppm/cm  
 F2PCH 317.69560 Hz/cm  
 F1PCH 0.63511 ppm/cm  
 F1CHC 317.69560 Hz/cm



Current Data Parameters  
 USER Rmwtll  
 NAME R4E-121117.D  
 EXPRNO 1  
 PROCNO 1

F1 - Acquisition Parameters  
 DATE\_ 20220117  
 TIME 16:45  
 INSTRUM spect  
 PULPROG zgpg30  
 TD 2048  
 SFO1 501.2221566 MHz  
 F2 501.2221566 MHz  
 F1 501.2221566 MHz  
 DS 16  
 SS 100.0000000 sec  
 AQ 0.2064884 sec  
 RG 26008  
 IN 100.0000000 sec  
 TE 298.2 K

===== CHANNEL f1 =====  
 NS1 8  
 NS2 8  
 PC 1.60 dB  
 SFO1 501.2221566 MHz

===== CHANNEL f2 =====  
 CPDPRG2 gMPC  
 NS1 8  
 NS2 8  
 PC 1.60 dB  
 SFO1 501.2221566 MHz

===== GRADIENT CHANNEL =====  
 GFRMAG 18.15 uT/m  
 GPC1 0.00 A  
 GPC2 0.00 A  
 GPC3 0.00 A  
 GPC4 0.00 A  
 GPC5 0.00 A  
 GPC6 0.00 A  
 GPC7 0.00 A  
 GPC8 0.00 A  
 GPC9 0.00 A  
 GPC10 0.00 A  
 GPC11 30.00 A  
 GPC12 18.00 A  
 GPC13 18.00 A  
 GPC14 18.00 A  
 GPC15 18.00 A  
 GPC16 1000.00 uT/m

===== CHANNEL f1 =====  
 NS1 8  
 NS2 8  
 PC 1.60 dB  
 SFO1 501.2221566 MHz

===== CHANNEL f2 =====  
 NS1 8  
 NS2 8  
 PC 1.60 dB  
 SFO1 501.2221566 MHz

===== GRADIENT CHANNEL =====  
 GFRMAG 18.15 uT/m  
 GPC1 0.00 A  
 GPC2 0.00 A  
 GPC3 0.00 A  
 GPC4 0.00 A  
 GPC5 0.00 A  
 GPC6 0.00 A  
 GPC7 0.00 A  
 GPC8 0.00 A  
 GPC9 0.00 A  
 GPC10 0.00 A  
 GPC11 30.00 A  
 GPC12 18.00 A  
 GPC13 18.00 A  
 GPC14 18.00 A  
 GPC15 18.00 A  
 GPC16 1000.00 uT/m

===== CHANNEL f1 =====  
 NS1 8  
 NS2 8  
 PC 1.60 dB  
 SFO1 501.2221566 MHz

===== CHANNEL f2 =====  
 NS1 8  
 NS2 8  
 PC 1.60 dB  
 SFO1 501.2221566 MHz

===== GRADIENT CHANNEL =====  
 GFRMAG 18.15 uT/m  
 GPC1 0.00 A  
 GPC2 0.00 A  
 GPC3 0.00 A  
 GPC4 0.00 A  
 GPC5 0.00 A  
 GPC6 0.00 A  
 GPC7 0.00 A  
 GPC8 0.00 A  
 GPC9 0.00 A  
 GPC10 0.00 A  
 GPC11 30.00 A  
 GPC12 18.00 A  
 GPC13 18.00 A  
 GPC14 18.00 A  
 GPC15 18.00 A  
 GPC16 1000.00 uT/m

===== CHANNEL f1 =====  
 NS1 8  
 NS2 8  
 PC 1.60 dB  
 SFO1 501.2221566 MHz

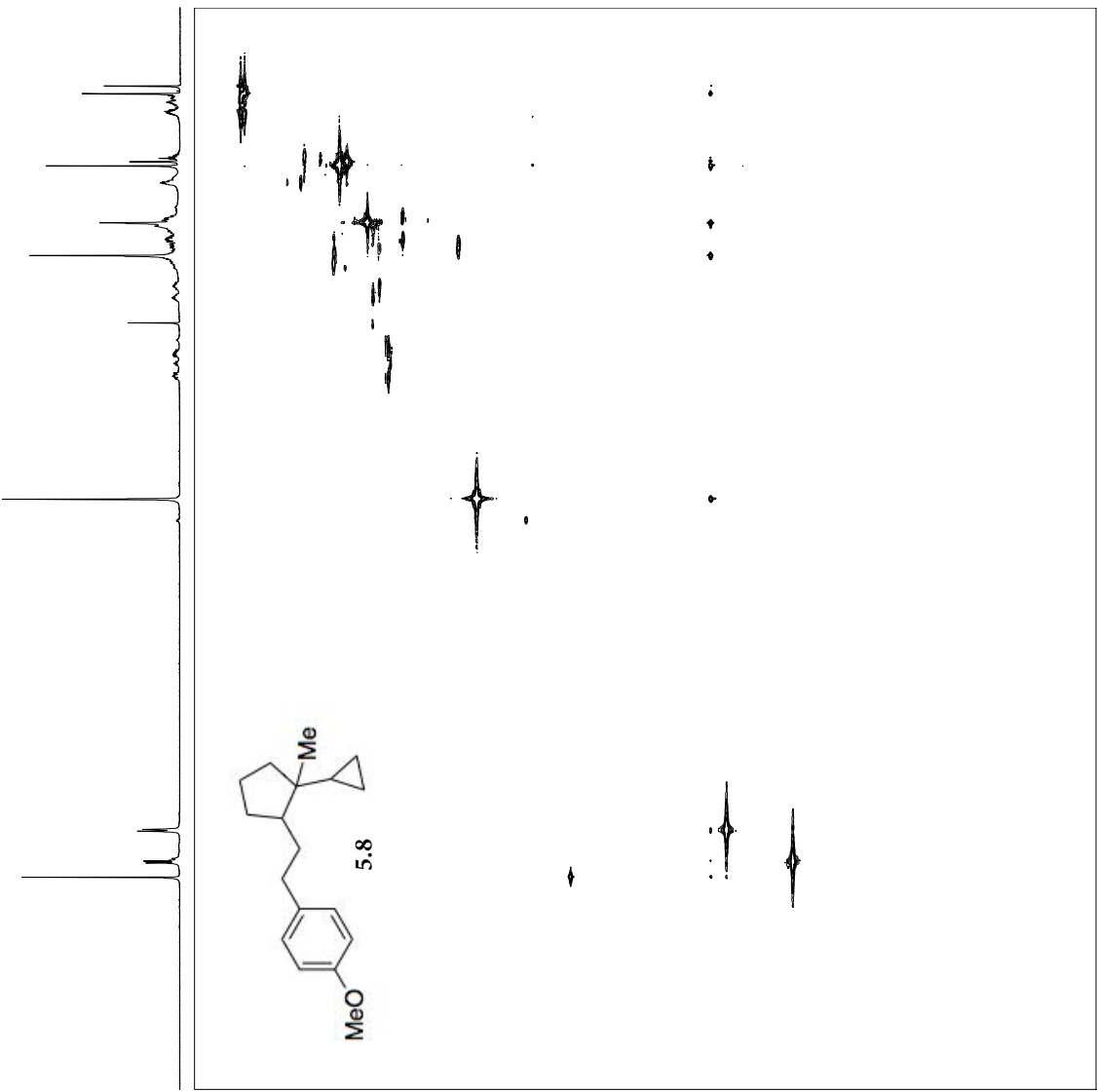
===== CHANNEL f2 =====  
 NS1 8  
 NS2 8  
 PC 1.60 dB  
 SFO1 501.2221566 MHz

===== GRADIENT CHANNEL =====  
 GFRMAG 18.15 uT/m  
 GPC1 0.00 A  
 GPC2 0.00 A  
 GPC3 0.00 A  
 GPC4 0.00 A  
 GPC5 0.00 A  
 GPC6 0.00 A  
 GPC7 0.00 A  
 GPC8 0.00 A  
 GPC9 0.00 A  
 GPC10 0.00 A  
 GPC11 30.00 A  
 GPC12 18.00 A  
 GPC13 18.00 A  
 GPC14 18.00 A  
 GPC15 18.00 A  
 GPC16 1000.00 uT/m

===== CHANNEL f1 =====  
 NS1 8  
 NS2 8  
 PC 1.60 dB  
 SFO1 501.2221566 MHz

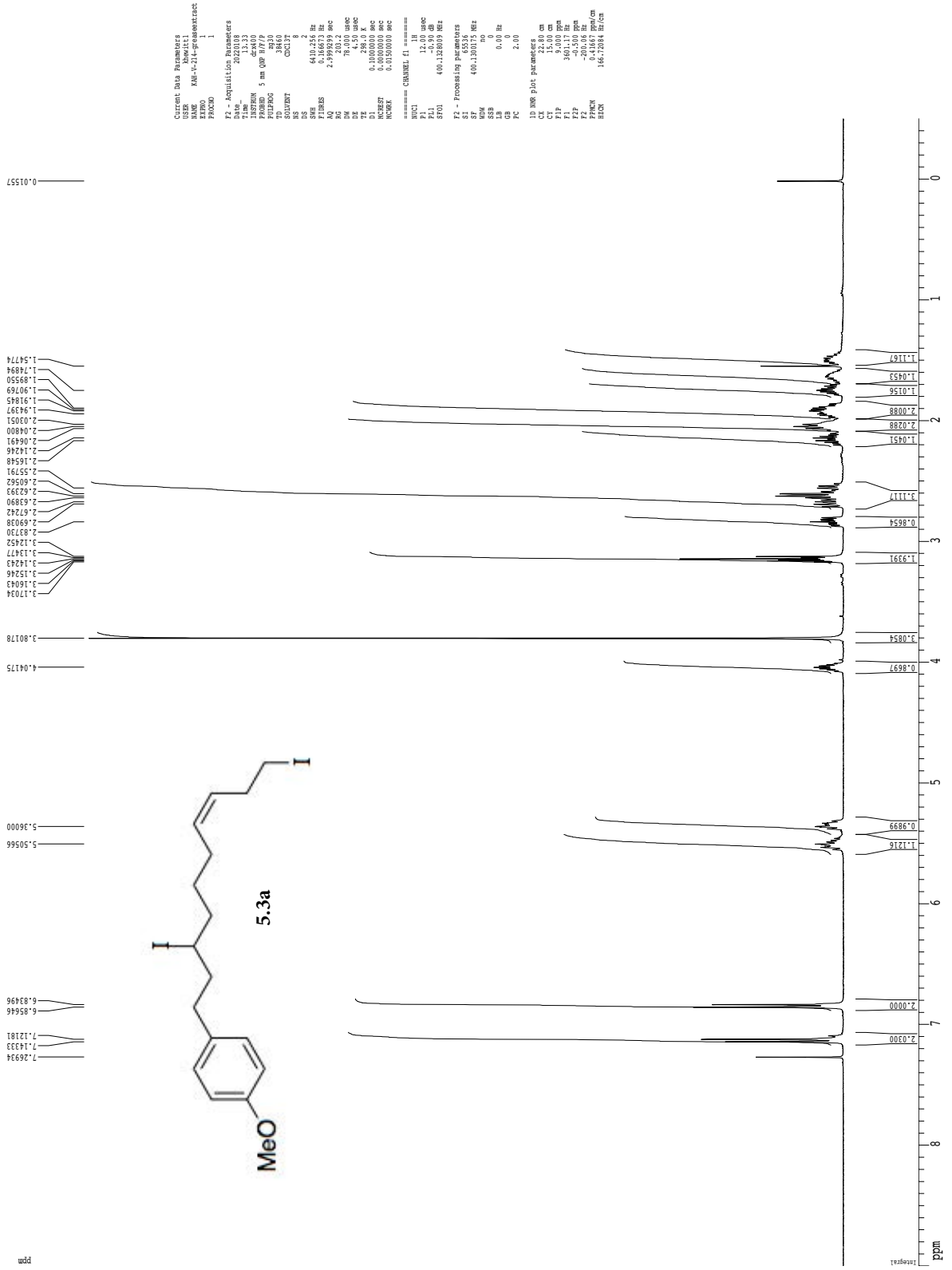
===== CHANNEL f2 =====  
 NS1 8  
 NS2 8  
 PC 1.60 dB  
 SFO1 501.2221566 MHz

===== GRADIENT CHANNEL =====  
 GFRMAG 18.15 uT/m  
 GPC1 0.00 A  
 GPC2 0.00 A  
 GPC3 0.00 A  
 GPC4 0.00 A  
 GPC5 0.00 A  
 GPC6 0.00 A  
 GPC7 0.00 A  
 GPC8 0.00 A  
 GPC9 0.00 A  
 GPC10 0.00 A  
 GPC11 30.00 A  
 GPC12 18.00 A  
 GPC13 18.00 A  
 GPC14 18.00 A  
 GPC15 18.00 A  
 GPC16 1000.00 uT/m

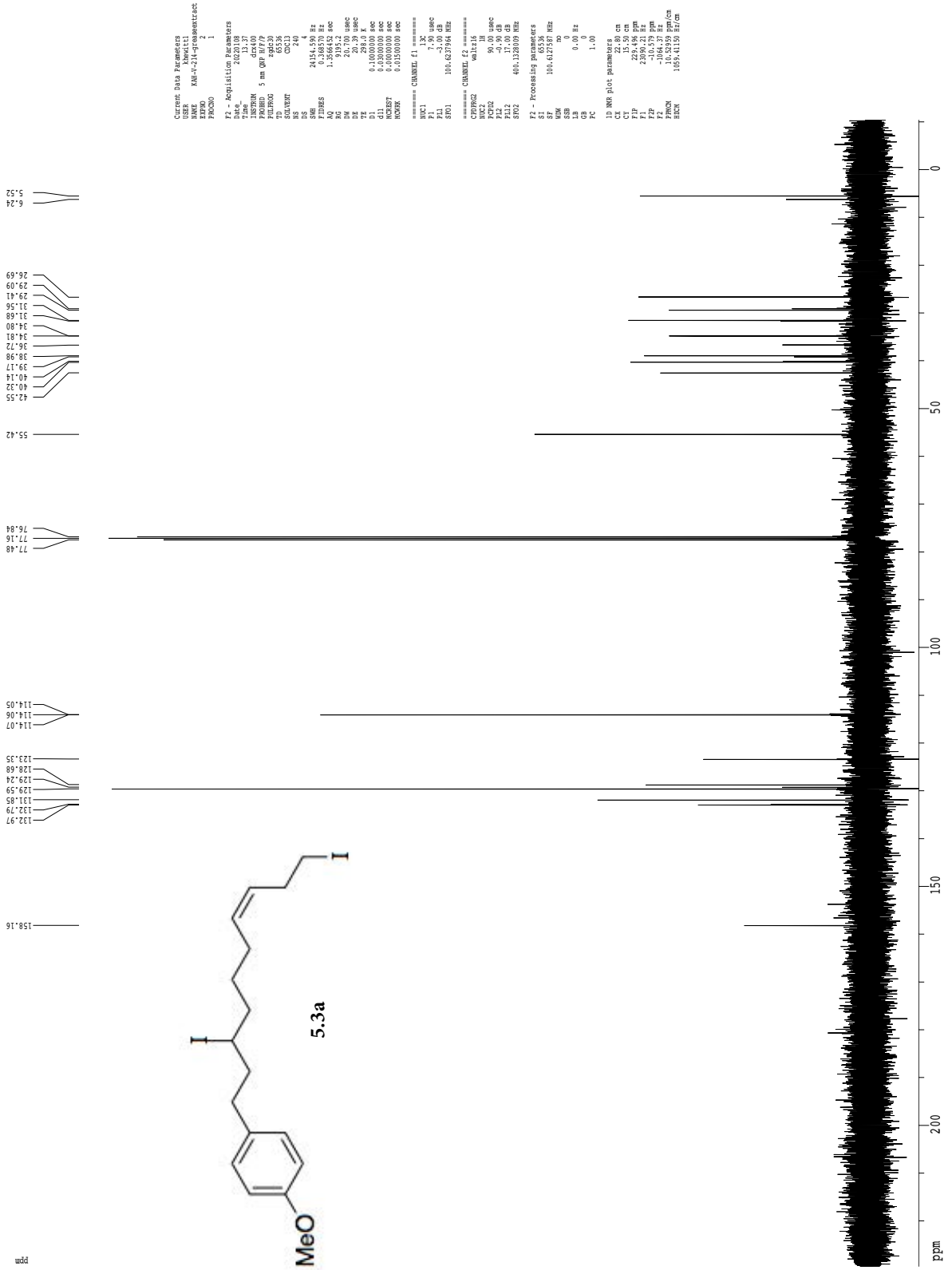


gfmqc

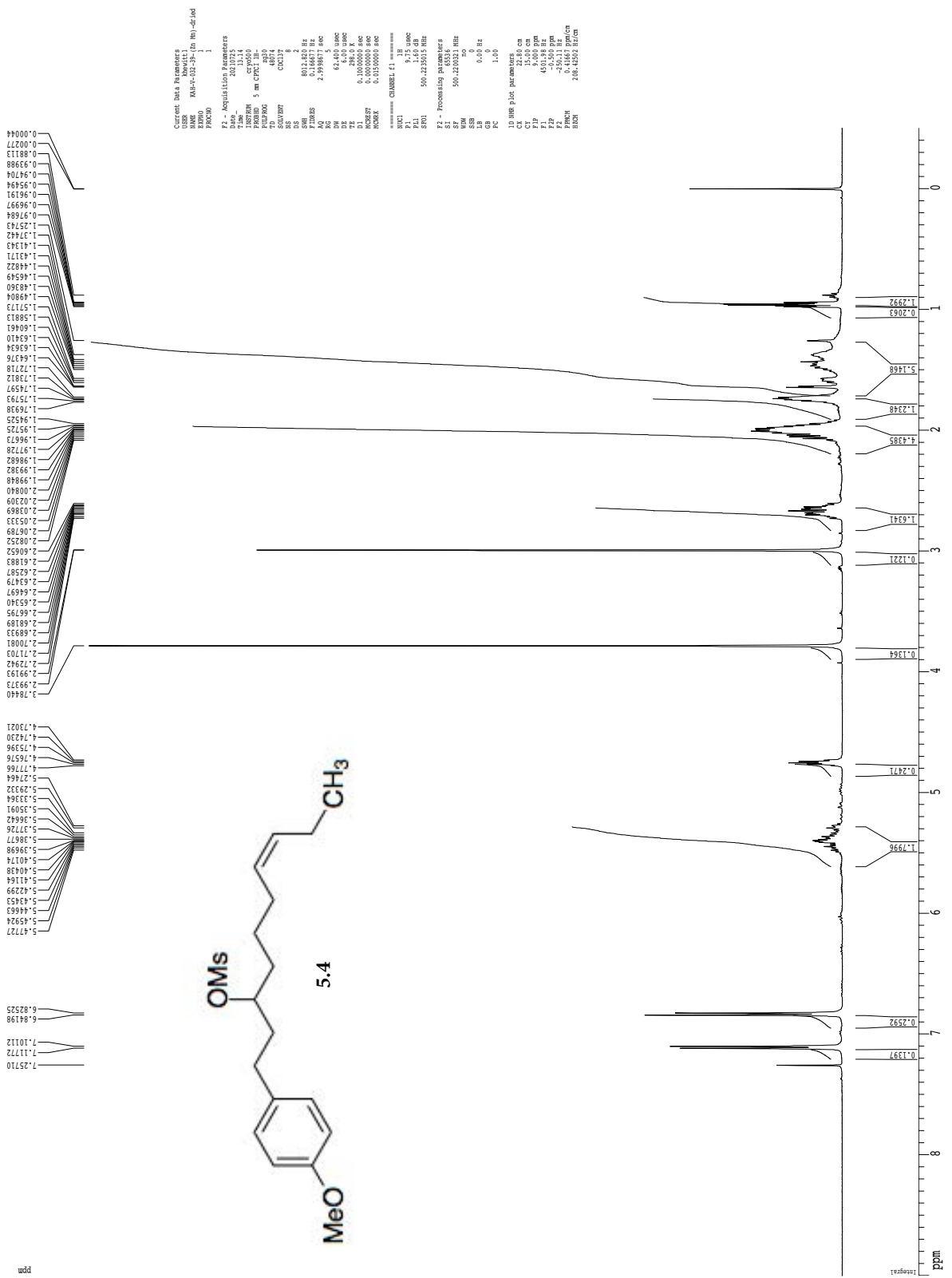
1H spectrum



**<sup>13</sup>C spectrum with <sup>1</sup>H decoupling**

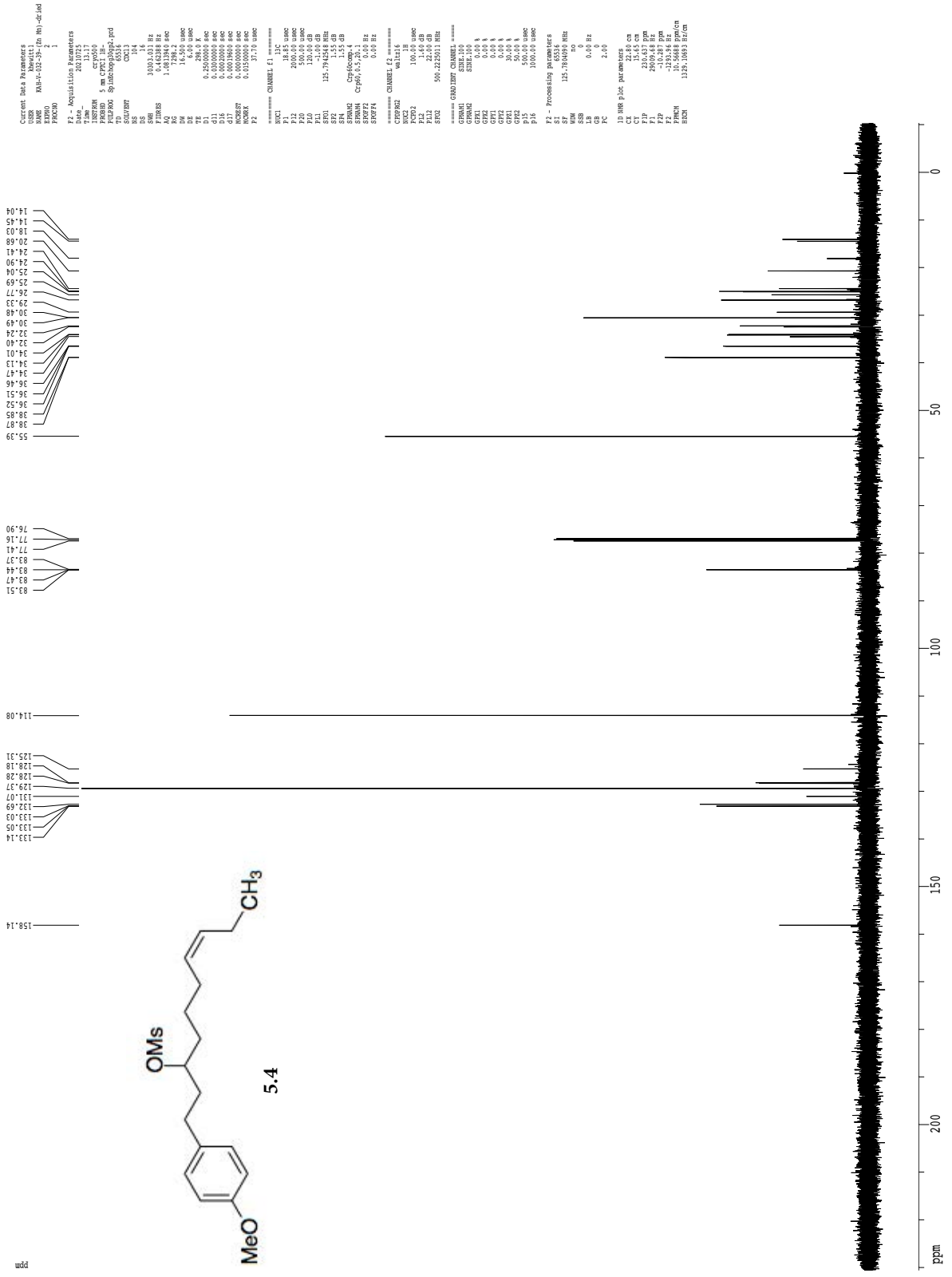


<sup>1</sup>H spectrum

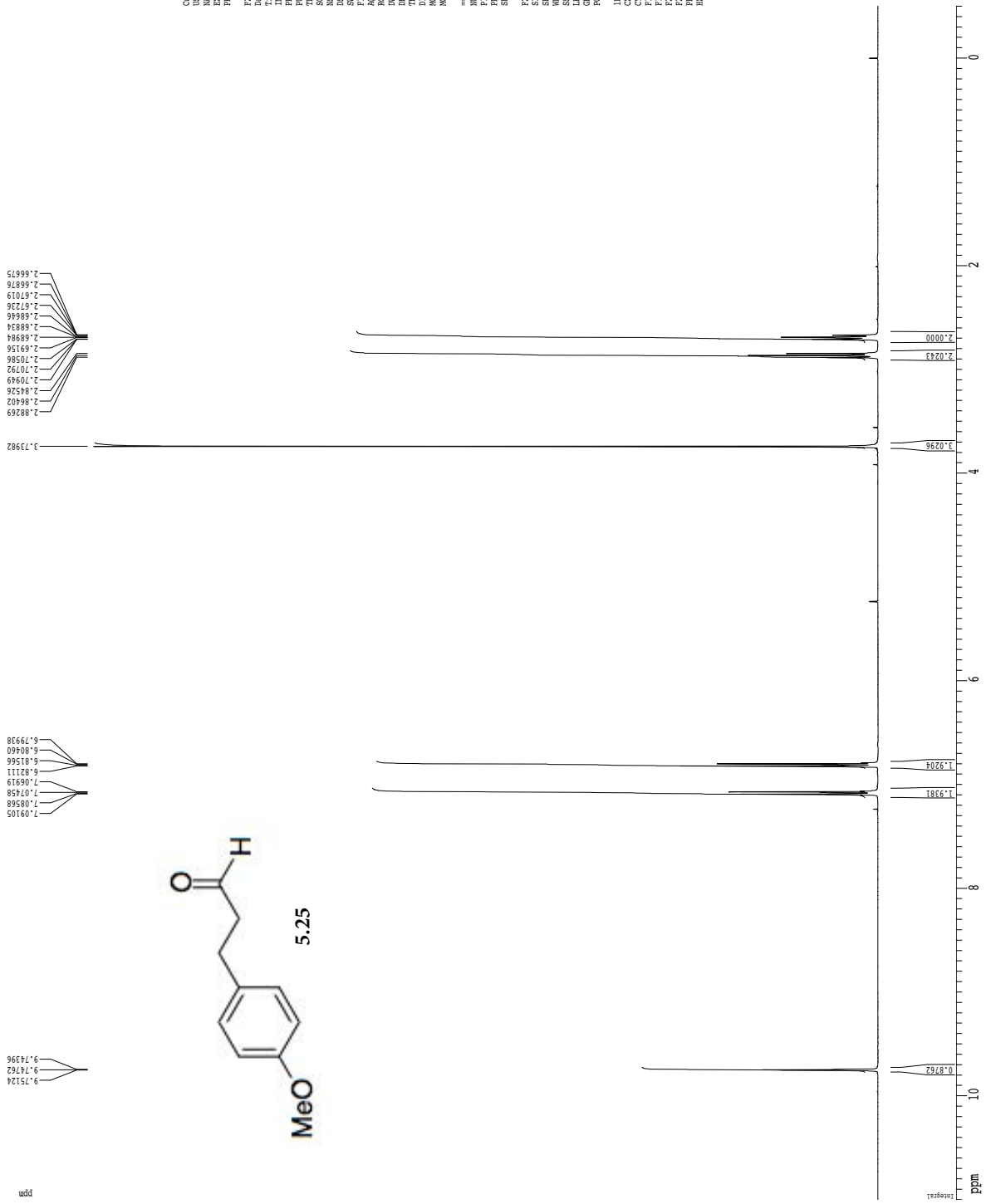




Z-restored spin-echo 13C spectrum with 1H decoupling



1H spectrum



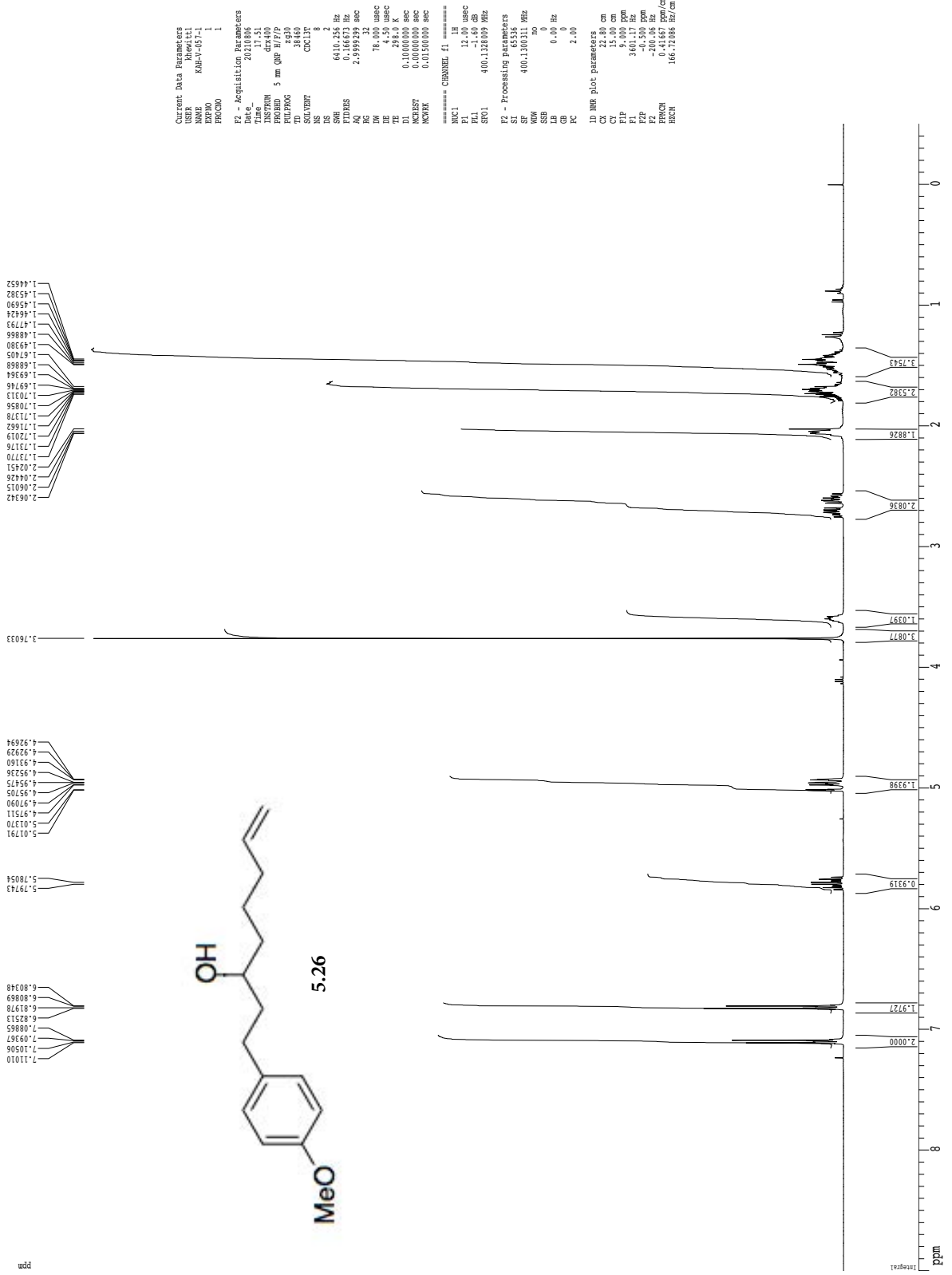
Current Data Parameters  
USER hewlett  
NAME RAH-7-054-1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 2/21/05  
Time 11:00  
INSTRUM dxt400  
PROBHD 5 mm QNP H/P/P  
PULPROG zgpg  
AQ 3.20  
SOLVENT CDCl3  
MS 8  
DS 2  
SWH 615.20 Hz  
FIDRES 0.166653 Hz  
AQ 2.9999259 sec  
RG 32  
DM 76.00 usec  
DE 19.00 usec  
TE 298.0 K  
D1 0.1000000 sec  
PCREST 0.0000000 sec  
PCW 0.0100000 sec  
===== CHANNEL f1 =====  
NUC1 1H  
P1 12.00 usec  
PL1 -1.00 dB  
SFO1 400.1324809 MHz

F2 - Processing parameters  
SI 32768  
SF 400.130101 MHz  
WDW po  
SSB 0  
GB 0.0 Hz  
PC 2.00

ID MR plot parameters  
CT 40.00 cm  
CY 15.00 cm  
FIP 11.000 ppm  
F1 4401.43 Hz  
F2 100.625 MHz  
F3 -700.00 Hz  
FPMCH 0.50439 ppm/cm  
HMCH 20.61998 Hz/cm

1H spectrum



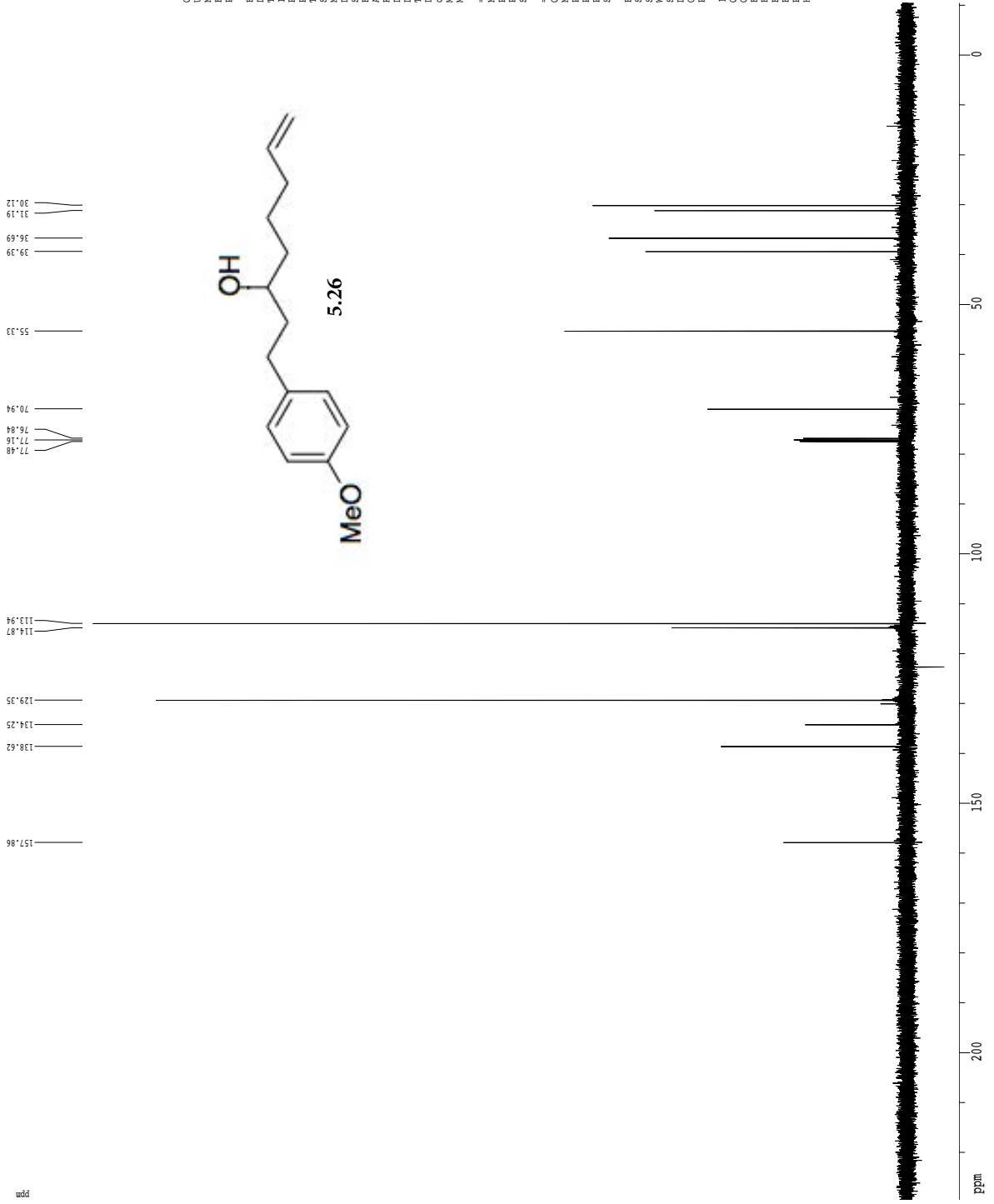
Current Data Parameters  
 USER hweilt1  
 NAME F45-057-1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 2/21/06  
 Time 11:56  
 INSTRUM dzt400  
 PROBDW 5 mm QNP H/F/P  
 PULPROG zgpg30  
 PC 30  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 616.35 Hz  
 FIDRES 0.16653 Hz  
 AQ 2.9959259 sec  
 RG 32  
 DW 76.000 usec  
 DE 1.500 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 MCHST 0.00000000 sec  
 MCHCK 0.01000000 sec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -1.00 dB  
 SFO1 400.1324009 MHz

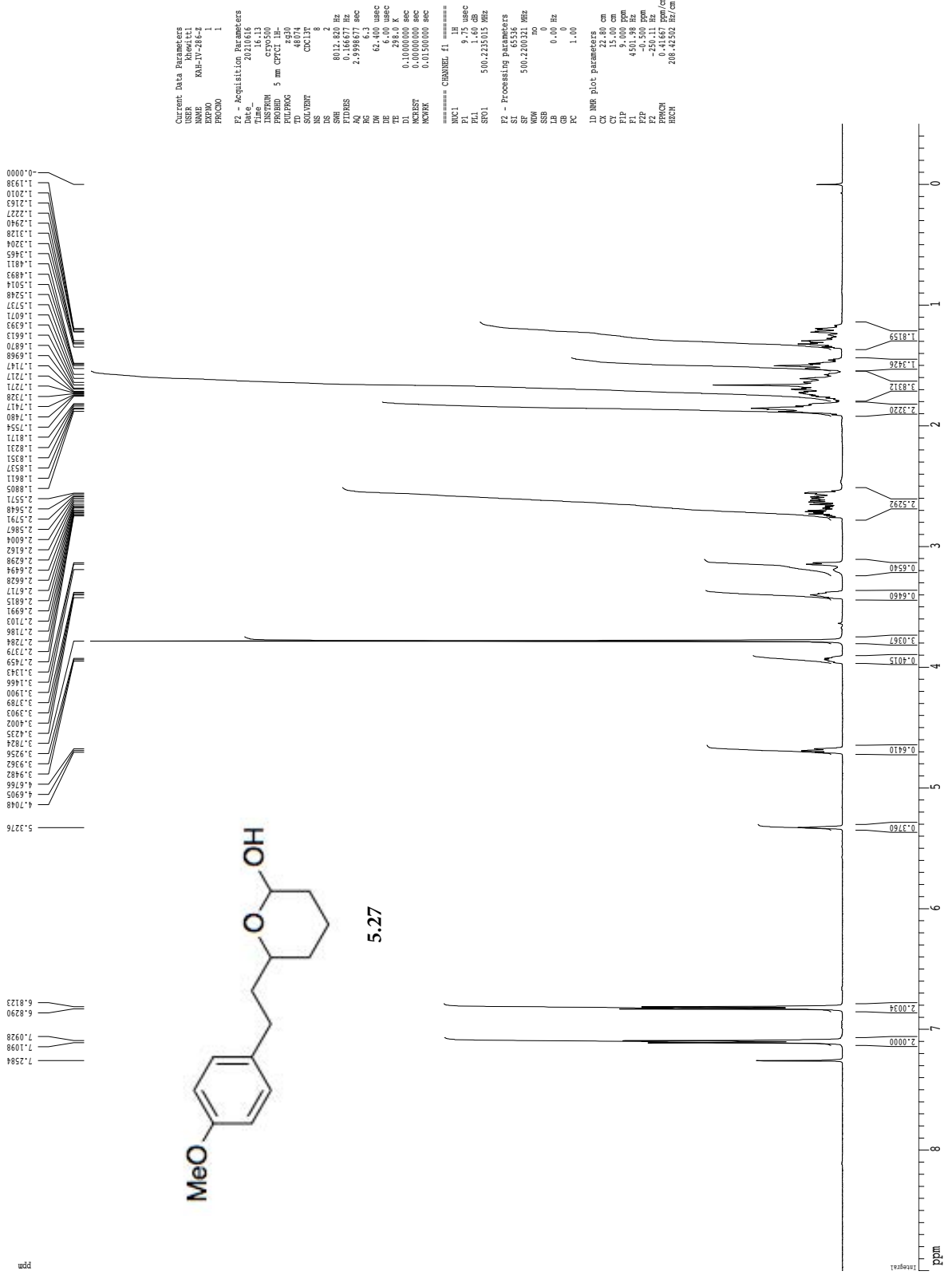
F2 - Processing parameters  
 SI 32768  
 SF 400.130311 MHz  
 WDM no  
 SSB 0  
 GB 0.0 Hz  
 PC 2.00

ID MR plot parameters  
 CT 15.00 cm  
 CY 15.00 cm  
 FIP 9.000 ppm  
 FI 3601.17 Hz  
 F2 -700.36 Hz  
 F2 0.41667 ppm/cm  
 HZCM 166.72066 Hz/cm

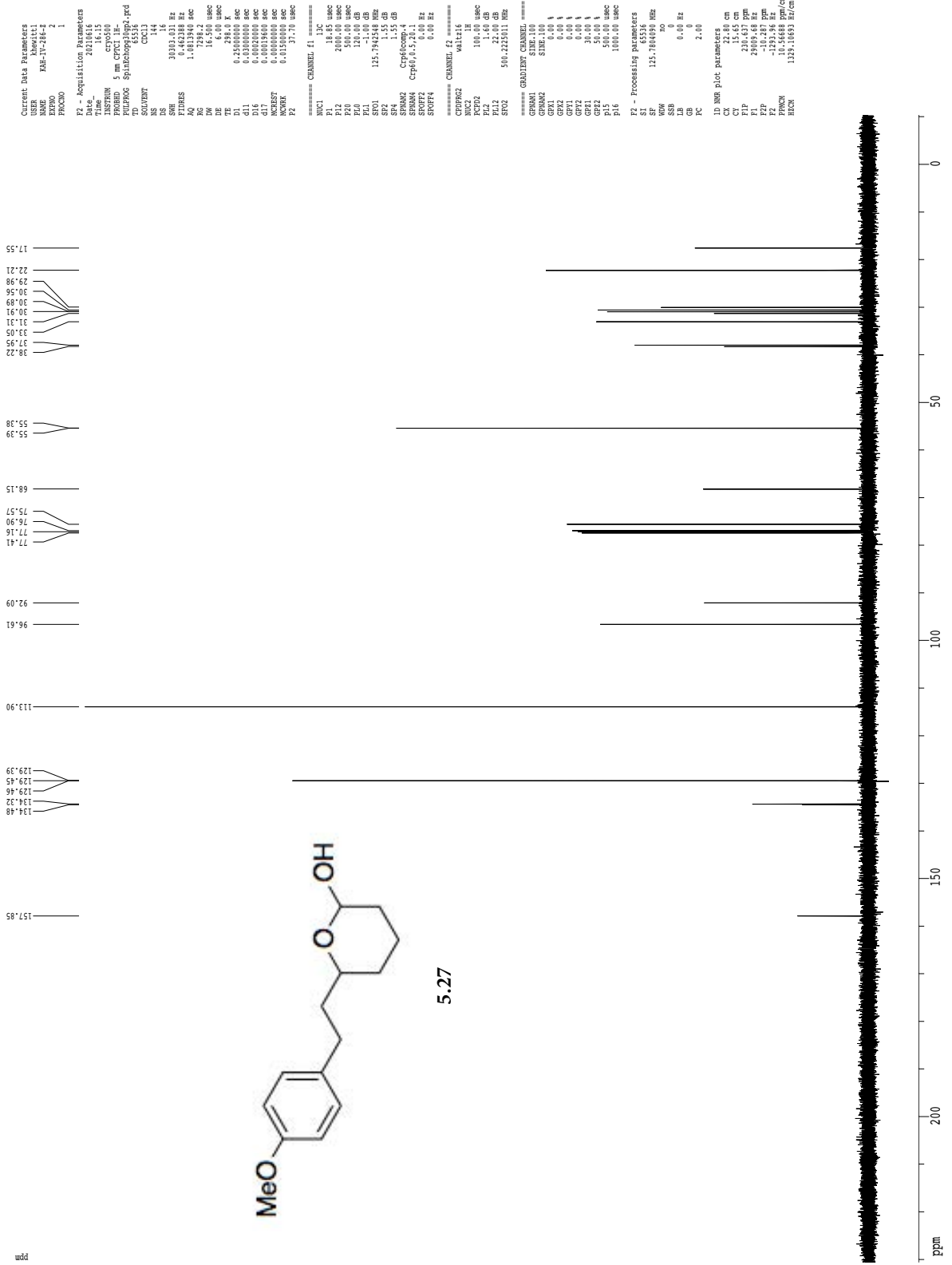
13C spectrum with 1H decoupling



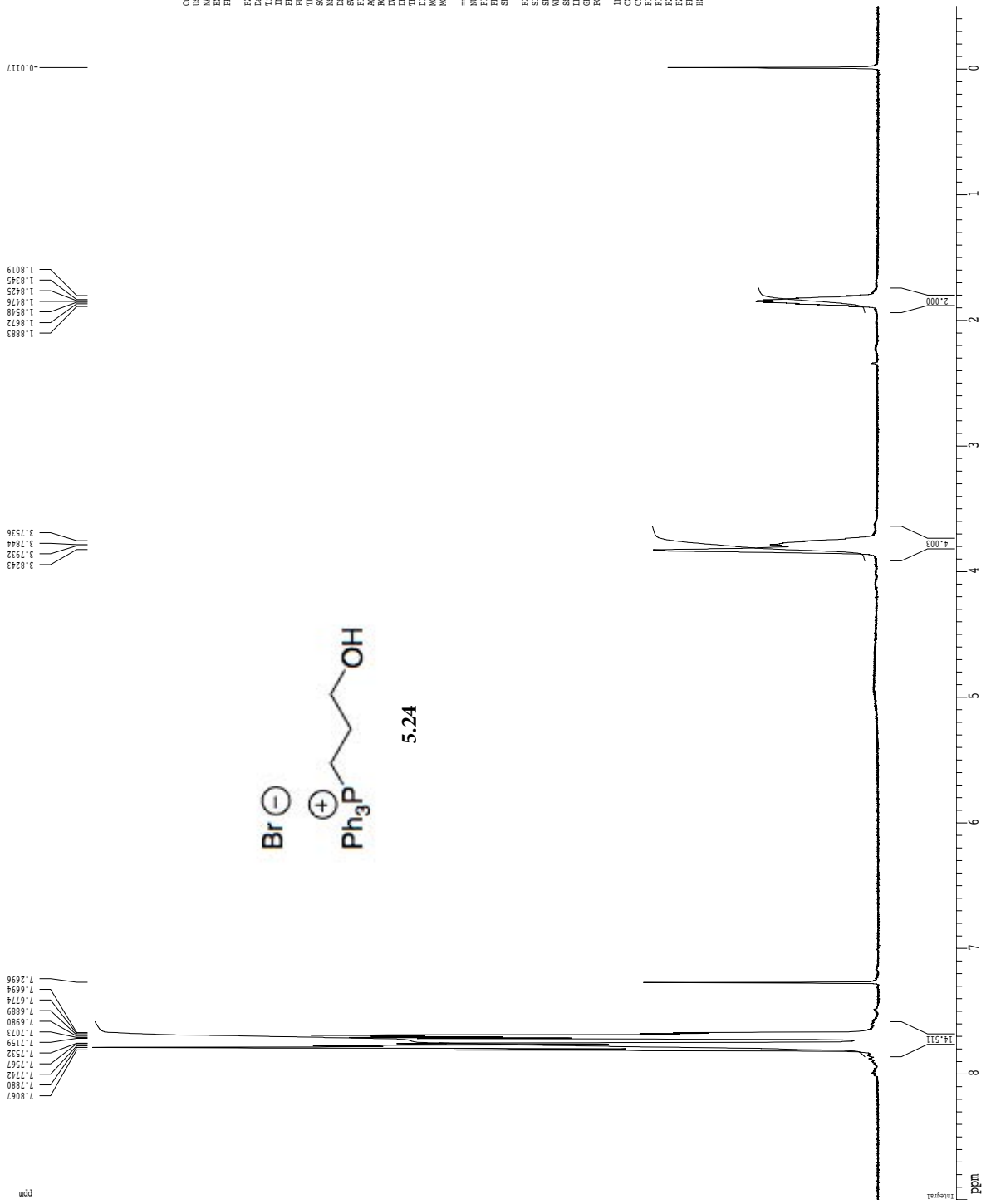
1H spectrum



Z-restored spin-echo 13C spectrum with 1H decoupling



1H spectrum



Current Data Parameters  
USER hweilt1  
NAME KH-17-29-1  
PROCNO 1

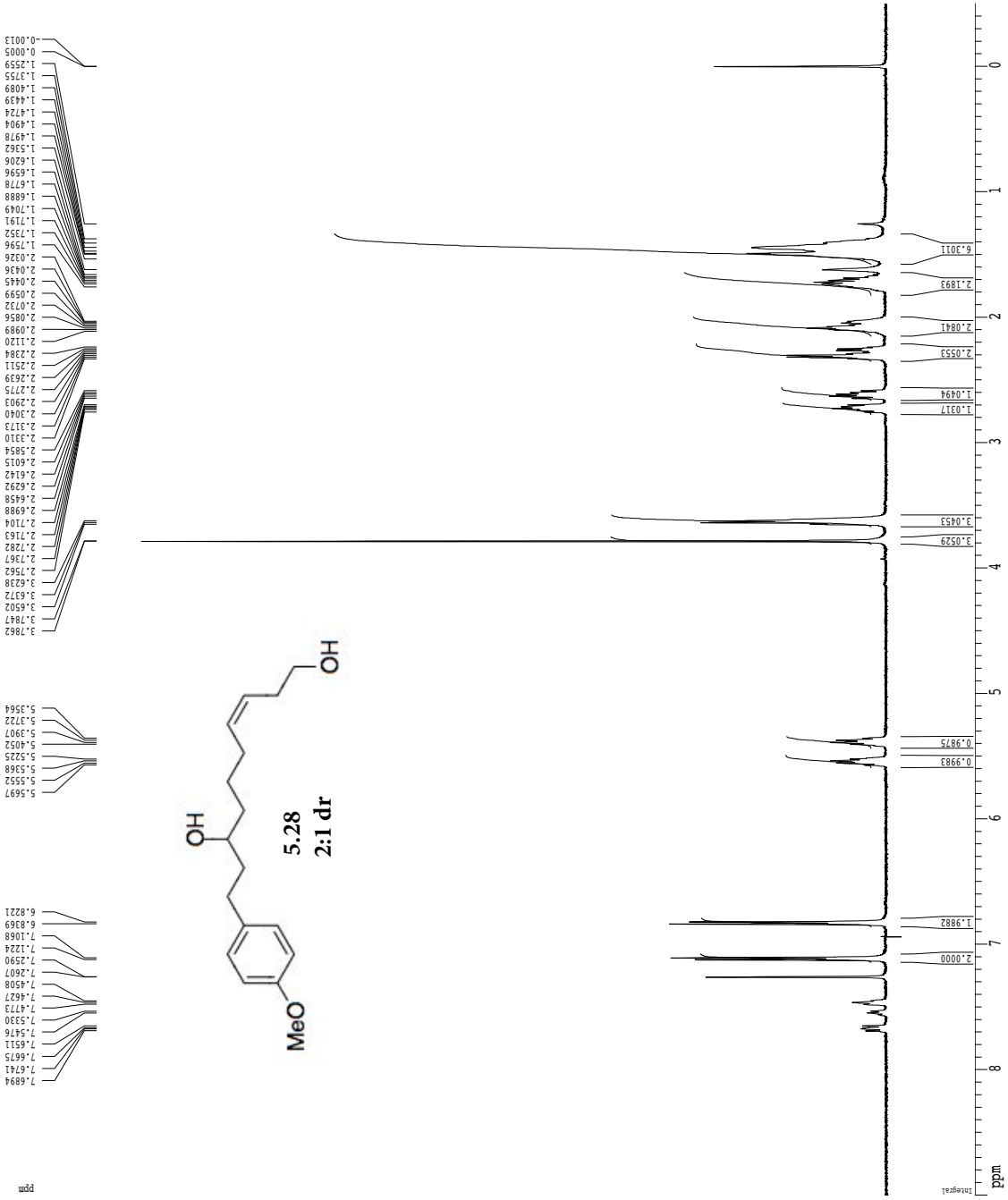
F2 - Acquisition Parameters  
Date\_ 2/2/02  
Time 12:00:00  
INSTRUM dr400  
PROBHD 5 mm QNP H/P/P  
PULPROG zgpg  
PC 320  
SOLVENT CDCl3  
NS 8  
DS 2  
SWH 6412.90 Hz  
FIDRES 0.166653 Hz  
AQ 2.9999299 sec  
RG 362  
DM 78.000 usec  
DE 19.000 usec  
TE 298.0 K  
D1 0.10000000 sec  
ICREST 0.00000000 sec  
PCWATK 0.01000000 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 12.00 usec  
PL1 -1.00 dB  
SFO1 400.1324809 MHz

F2 - Processing parameters  
SI 65536  
SF 400.130175 MHz  
WDW mo  
SSB 0  
GB 0.0 Hz  
PC 2.00

ID MR plot parameters  
CT 15.00 cm  
CY 15.00 cm  
PIF 9.000 ppm  
F1 3601.17 Hz  
F2 90.000 MHz  
PP4CH 0.41667 ppm/cm  
PPMCH 0.41667 ppm/cm  
HSCN 166.72084 Hz/cm

1H spectrum



Current Data Parameters  
 USER khewittl  
 NAME KAH-IV-288-CHK  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date 20210621  
 Time 14.21  
 INSTRUM gn500  
 PROBHD 5 mm broadband  
 PULPROG zg30  
 TD 48074  
 SOLVENT CDCl3T  
 NS 8  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.9998677 sec  
 RG 1448.2  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 MCREST 0.00000000 sec  
 MCWRK 0.01500000 sec

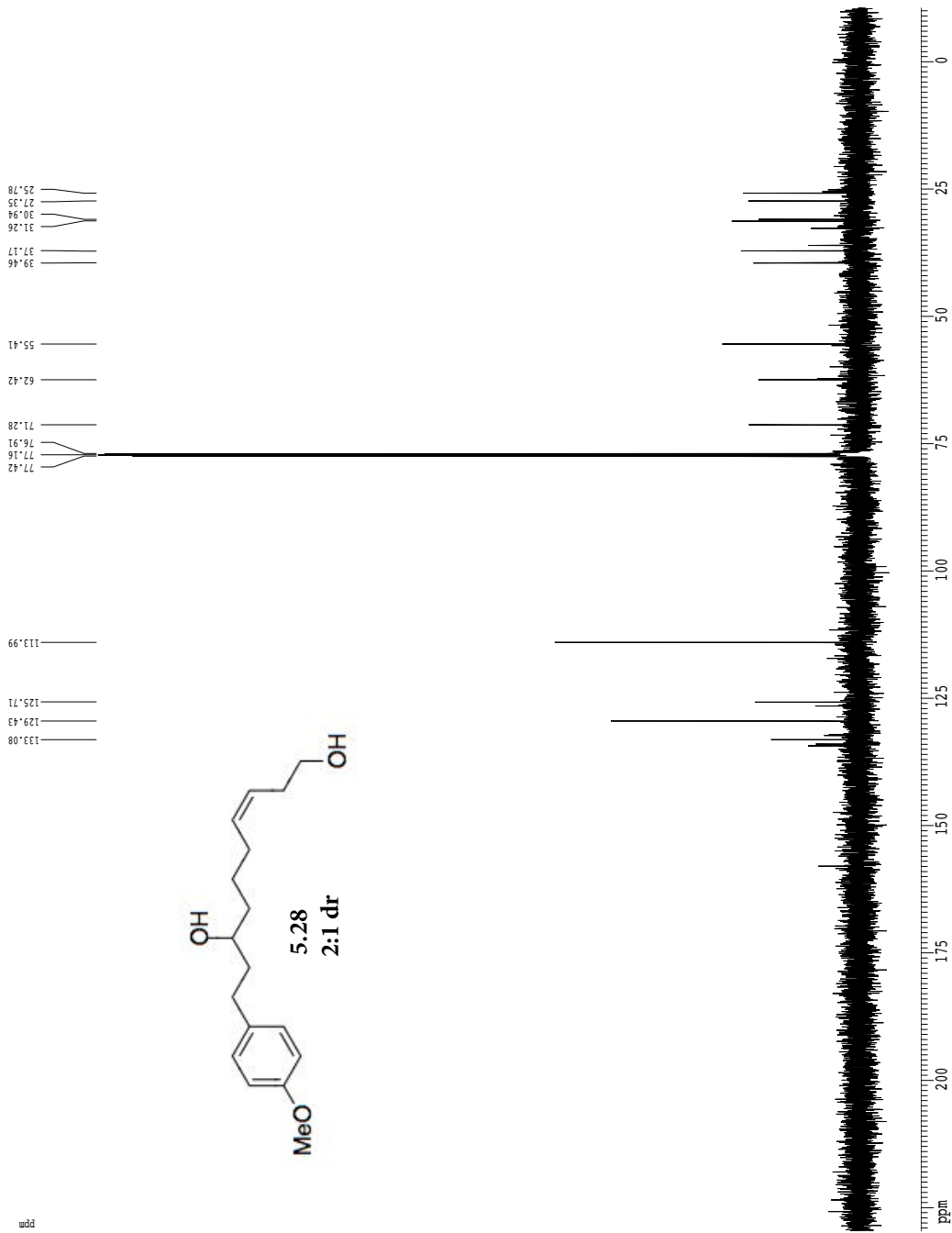
===== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -6.00 dB  
 SFO1 498.7534913 MHz

F2 - Processing parameters  
 SI 65536  
 SF 498.7500314 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 CY 12.50 cm  
 F1P 9.000 ppm  
 F1 4488.75 Hz  
 F2P -0.500 ppm  
 F2 -249.38 Hz  
 PPMCM 0.47500 ppm/cm  
 HZCM 236.90627 Hz/cm



13C spectrum with 1H decoupling



Current Data Parameters  
 USER khewitt1  
 NAME KAH-IV-288-CHK  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20210621  
 Time\_ 14.23  
 INSTRUM gn500  
 PROBHD 5 mm broadband  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 144  
 DS 4  
 SWH 30303.031 Hz  
 FIDRES 0.462388 Hz  
 AQ 1.0813940 sec  
 RG 5792.6  
 DW 16.500 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.25000000 sec  
 d11 0.03000000 sec  
 INCRST 0.00000000 sec  
 MCWRK 0.01500000 sec

==== CHANNEL f1 =====

NUC1 13C  
 P1 14.20 usec  
 PL1 -6.00 dB  
 SF01 125.4245824 MHz

==== CHANNEL f2 =====

CPDPRG2 waitz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 -6.00 dB  
 PL12 12.30 dB  
 SF02 498.7524937 MHz

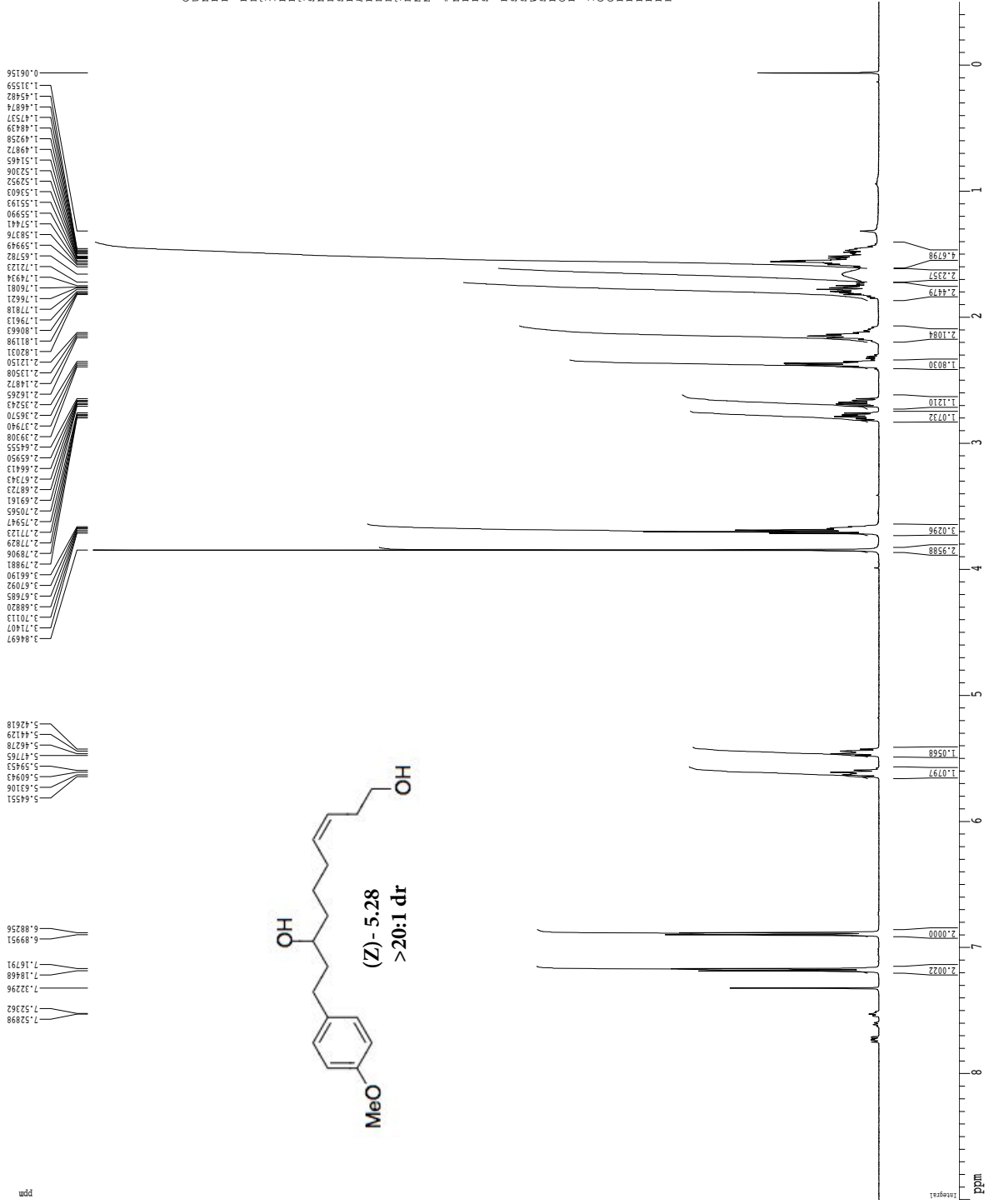
F2 - Processing parameters

SI 65536  
 SF 125.4107757 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 2.00

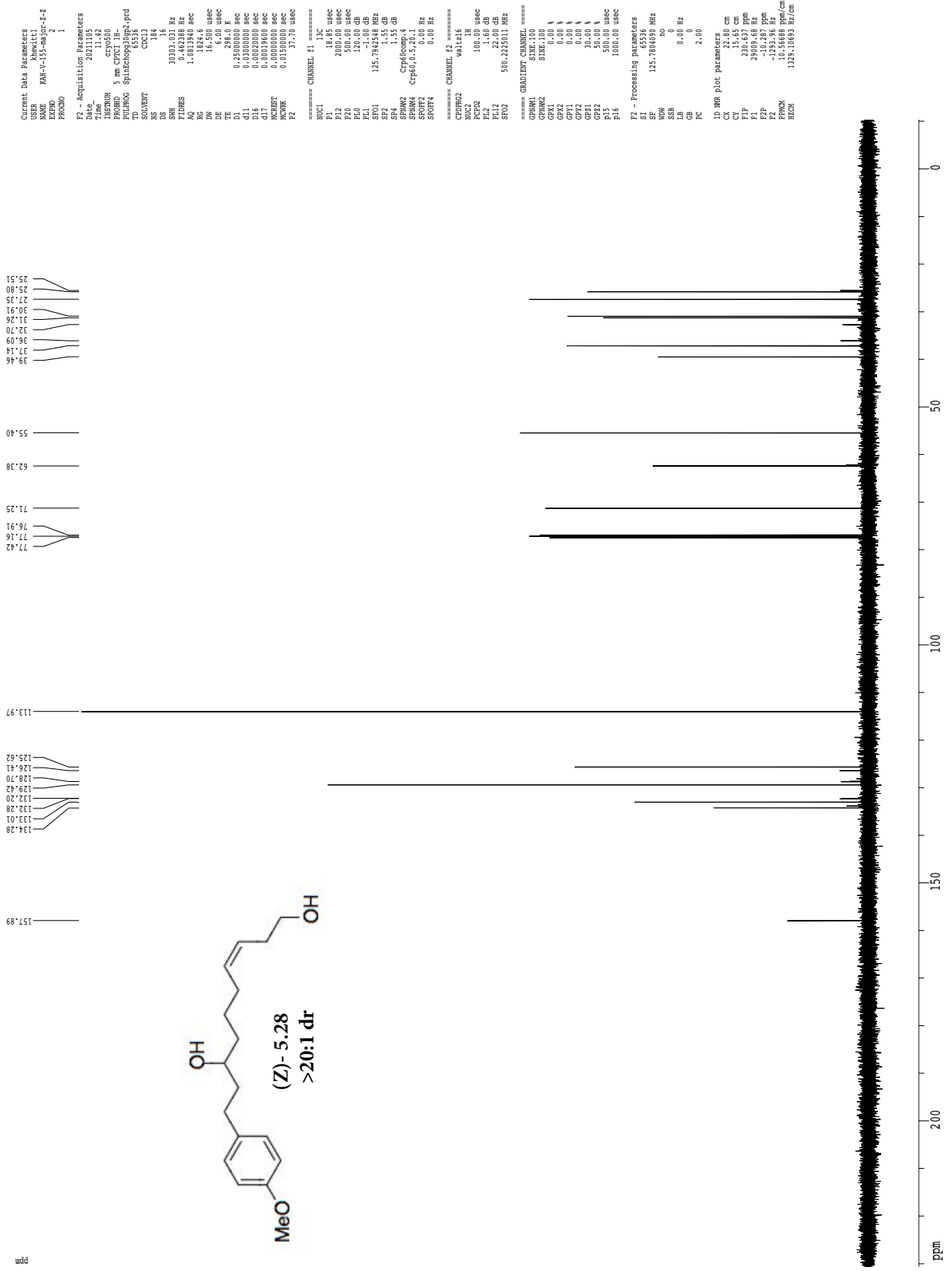
1D NMR plot parameters

CX 20.00 cm  
 CY 12.50 cm  
 F1P 229.520 ppm  
 F1 28784.24 Hz  
 F2P -10.507 ppm  
 F2 -1317.64 Hz  
 PPMCM 12.00131 ppm/cm  
 HZCM 1505.09399 Hz/cm

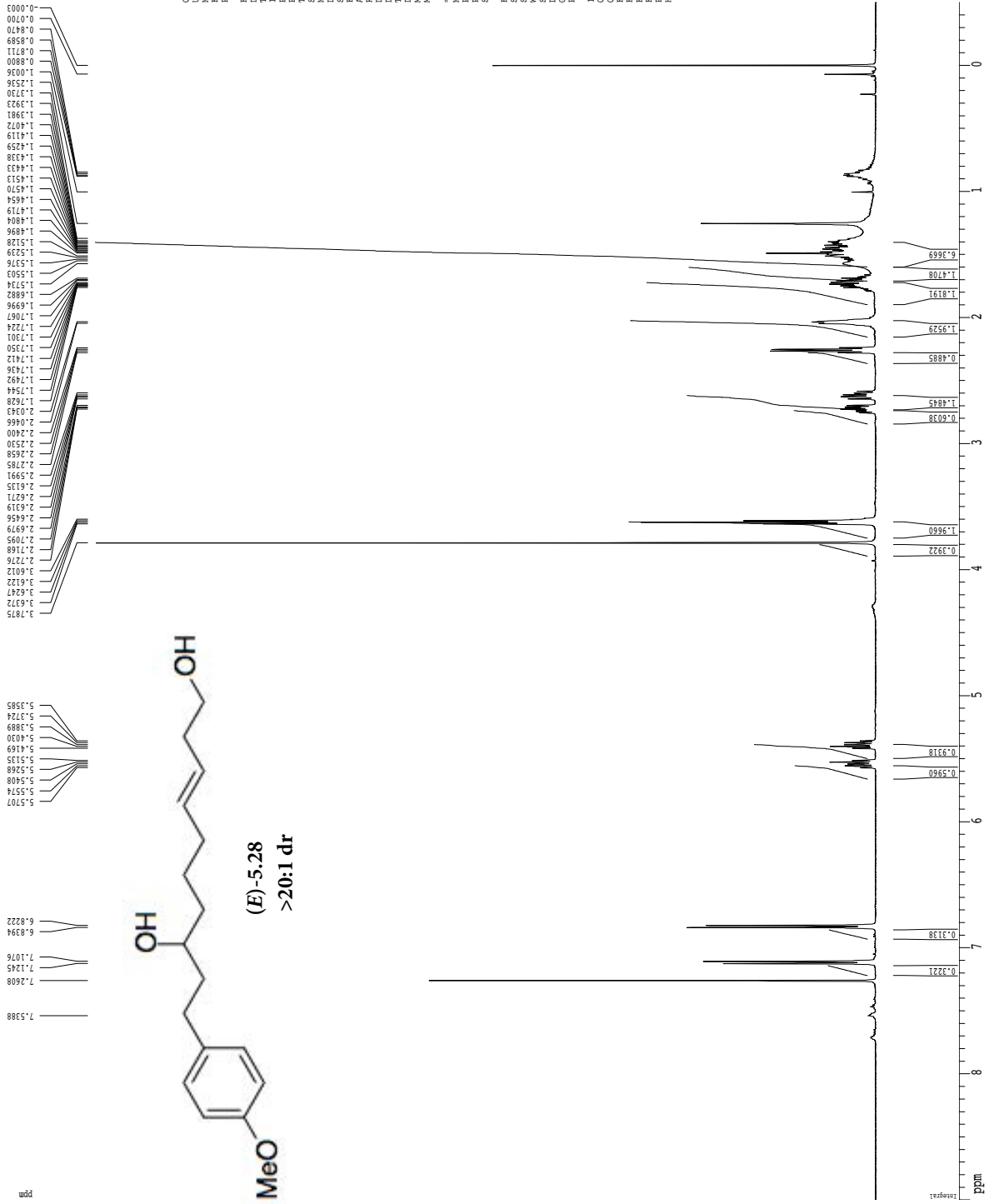
1H spectrum



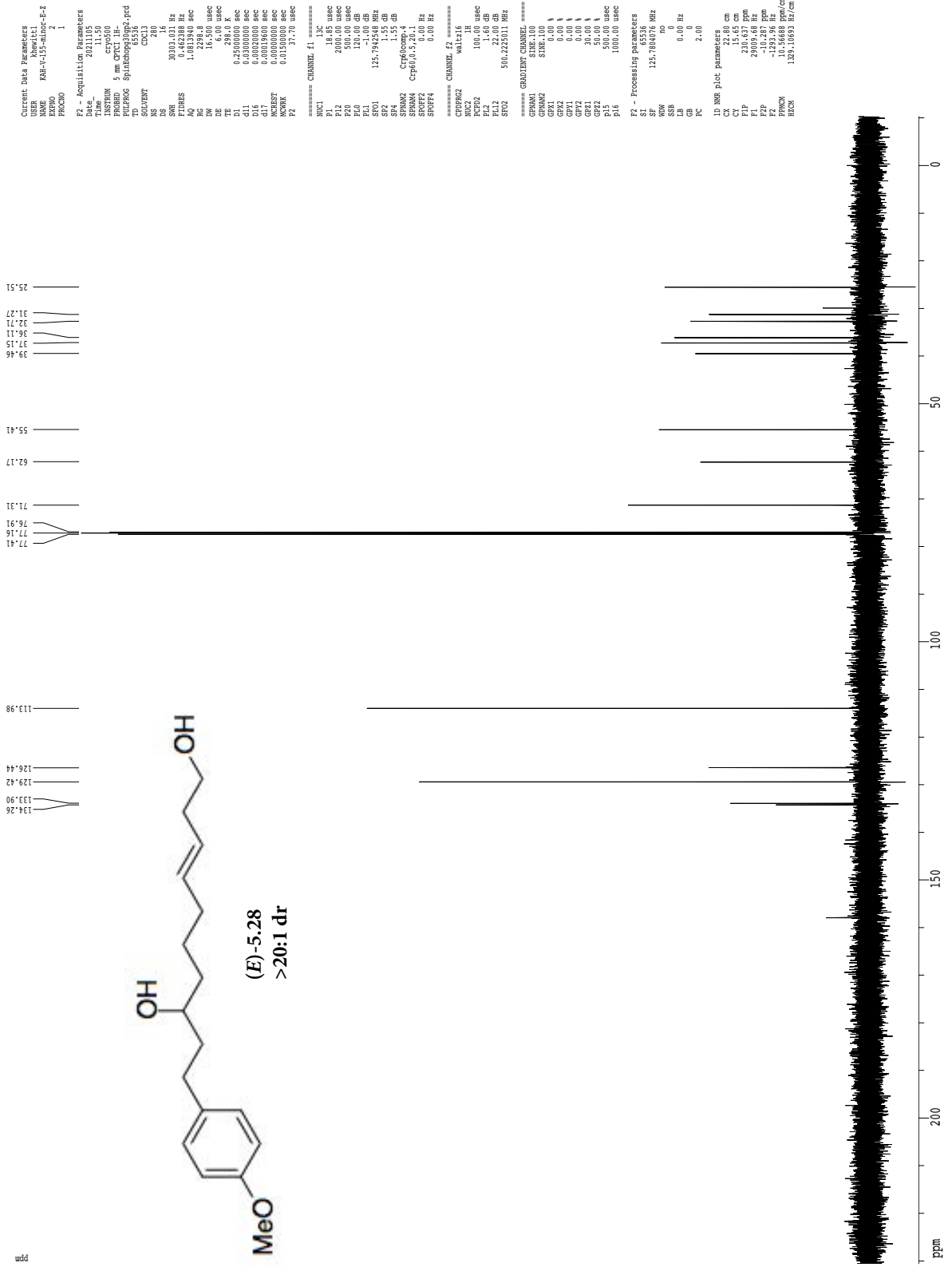
Z-restored spin-echo 13C spectrum with 1H decoupling



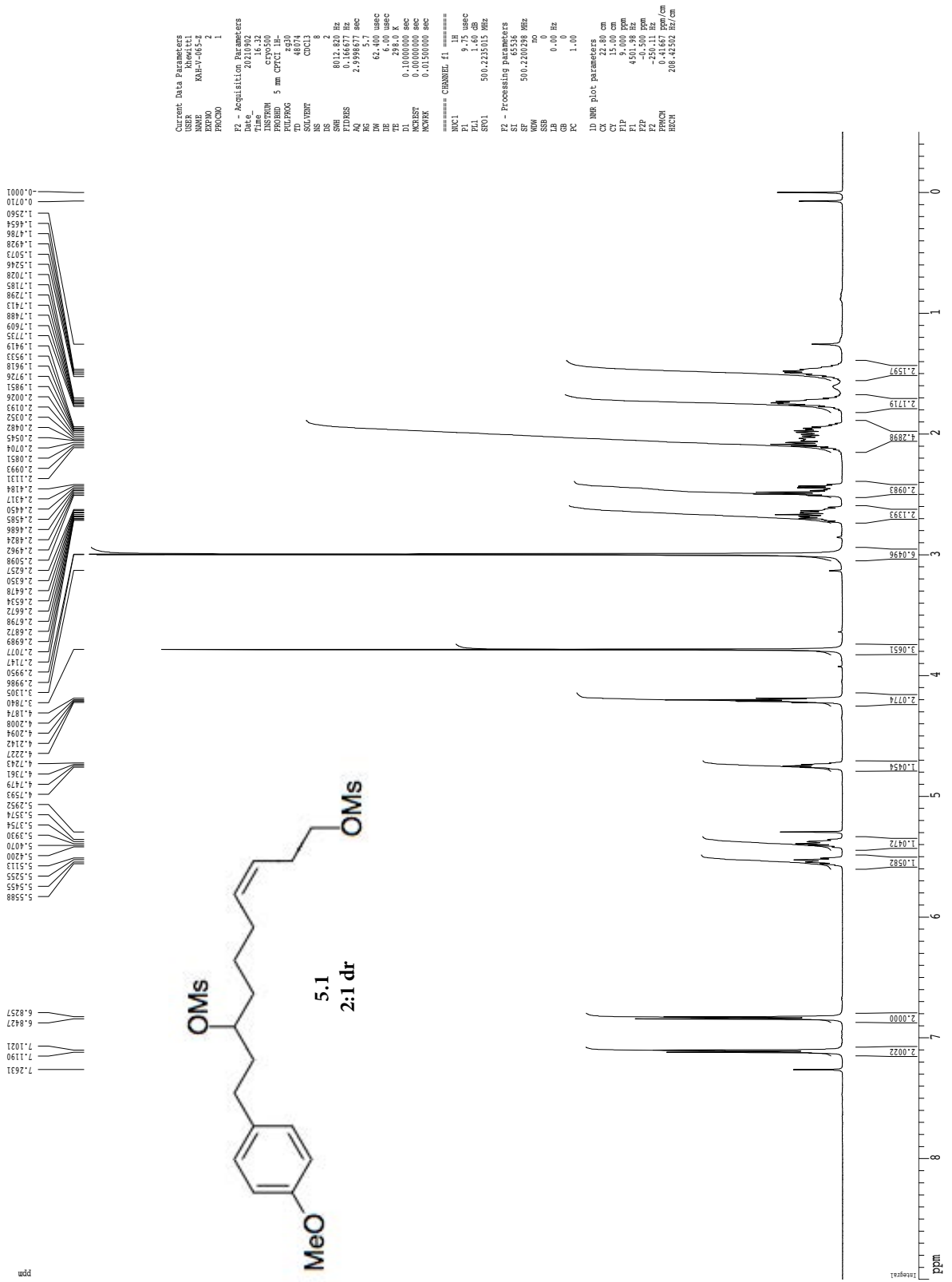
1H spectrum



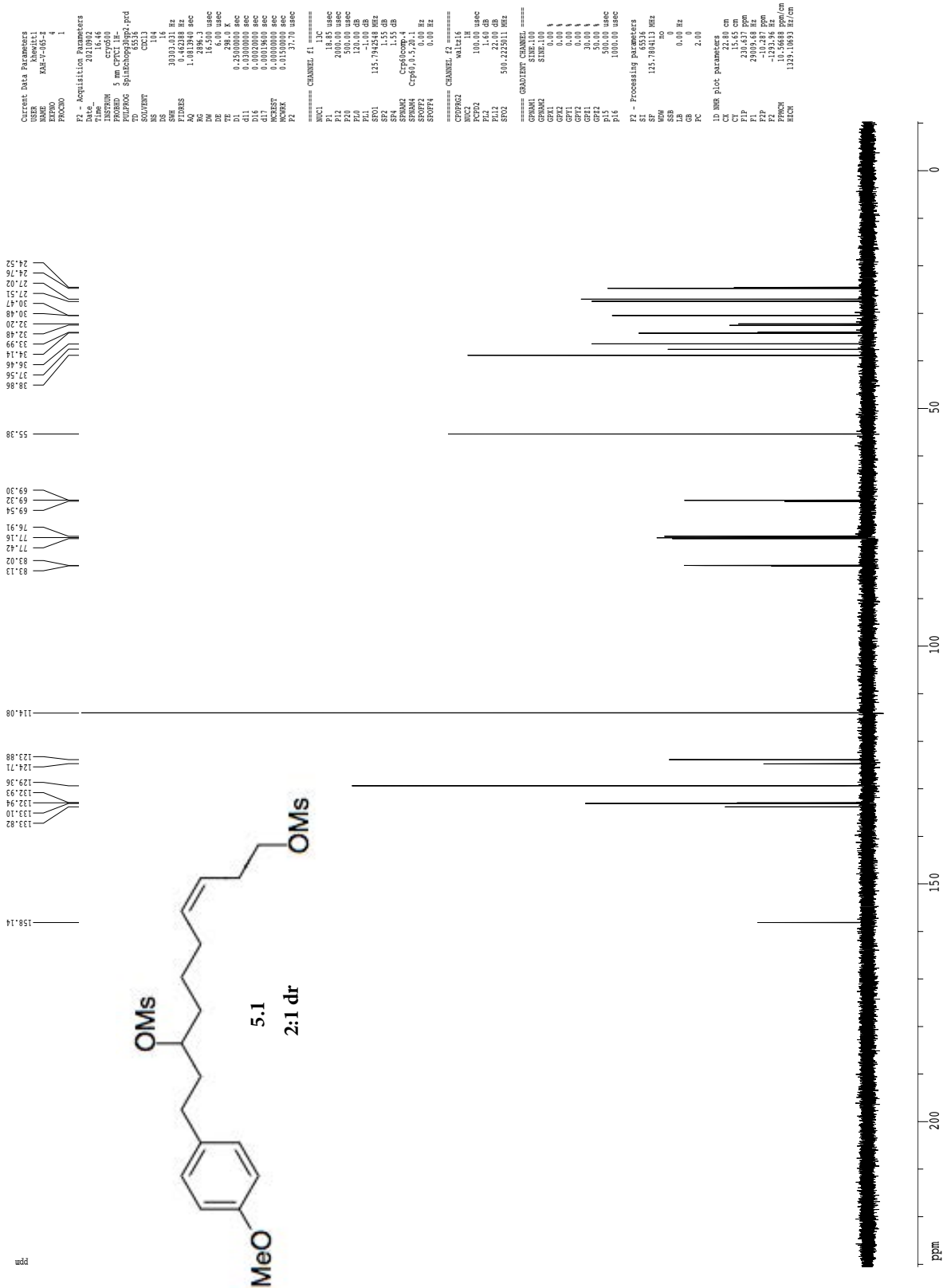
Z-restored spin-echo 13C spectrum with 1H decoupling

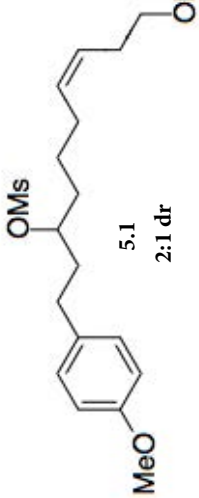


1H spectrum

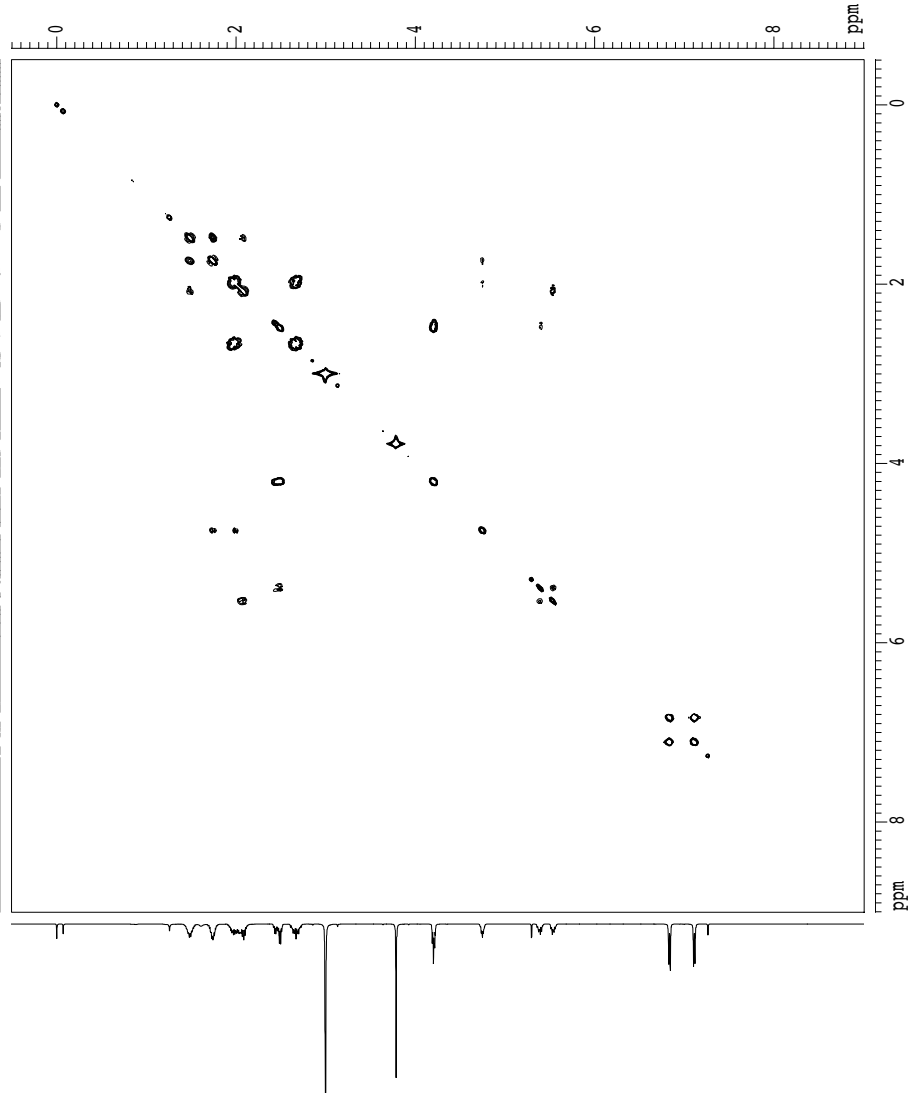


Z-restored spin-echo 13C spectrum with 1H decoupling





gcosy60



Current Data Parameters  
 USER khewit1  
 NAME FAN-Y-06-2  
 EXPNO 3  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20100902  
 Time 16:34  
 PROBN 00000000  
 PULPROG zgpg30  
 TD 2048  
 SOLVENT CDCl3  
 DS 4  
 SWH 8012.820 Hz  
 FIDRES 3.812510 Hz  
 AQ 0.177852 sec  
 SFO1 500.2235015 MHz  
 DM 62.00 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D0 0.0000000 sec  
 D1 1.0000000 sec  
 d13 0.0000000 sec  
 D16 0.0002000 sec  
 D30 0.0001240 sec

==== CHANNEL f1 =====  
 NUC1 1H  
 P1 9.75 usec  
 PL1 1.60 dB  
 SFO1 500.2235015 MHz

==== CHANNEL CHANNEL =====  
 GPMW1 SWSQ10.100  
 GPMW2 SWSQ10.100  
 GPC1 0.00 %  
 GPC2 0.00 %  
 GPC3 0.00 %  
 GPC4 0.00 %  
 GPC5 0.00 %  
 GPC6 17.00 %  
 P16 1000.00 usec

F1 - Acquisition Parameters  
 MD0 1  
 TD 512  
 SFO1 500.2235 MHz  
 FIDRES 15.46018 ppm  
 SW 16.4018 ppm  
 FWHM 0.6

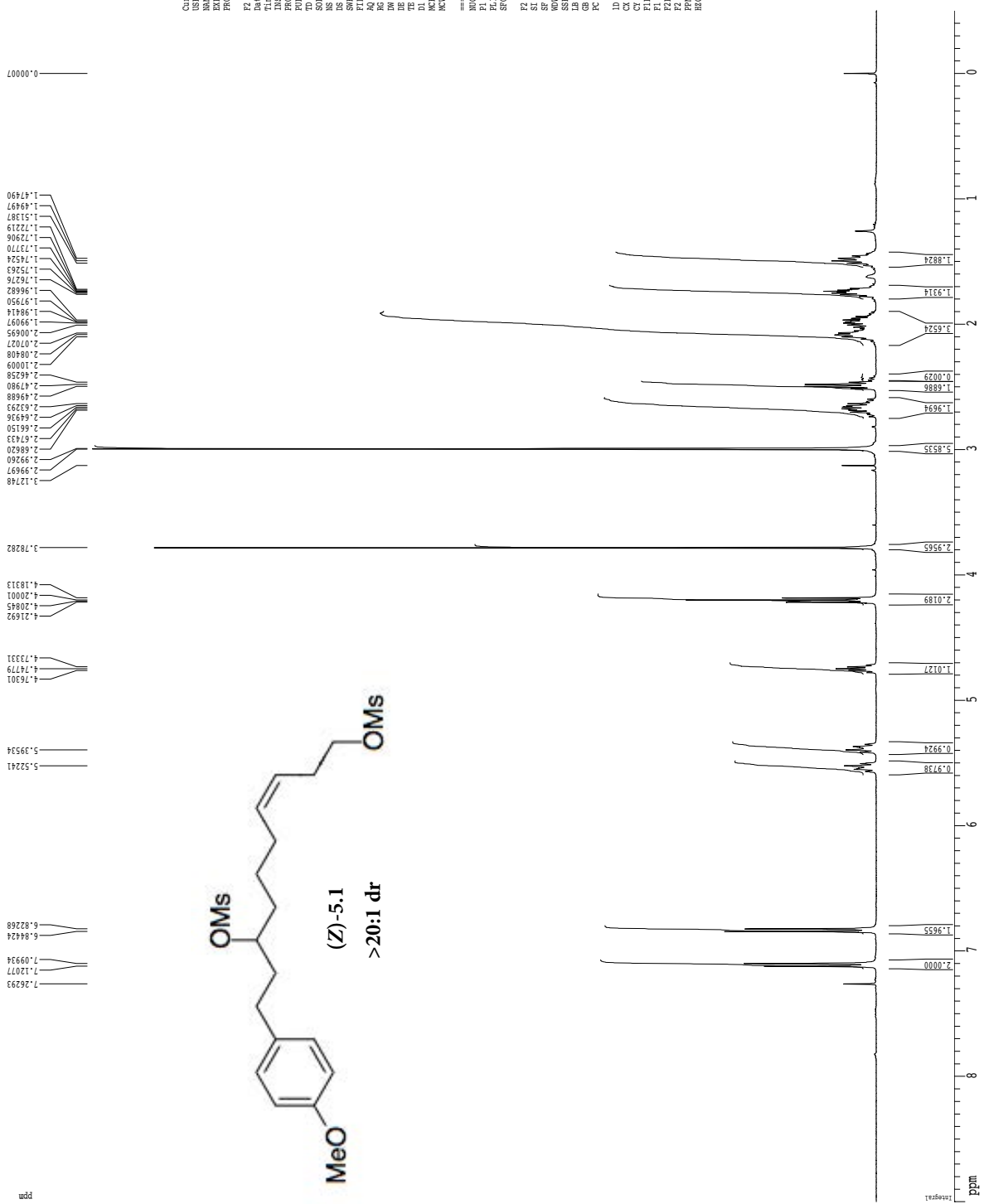
F2 - Processing parameters  
 SI 32768  
 SF 500.2200988 MHz  
 WDM 1  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 1024  
 SF 500.2200988 MHz  
 WDM 1  
 SSB 0  
 LB 0.00 Hz  
 GB 0

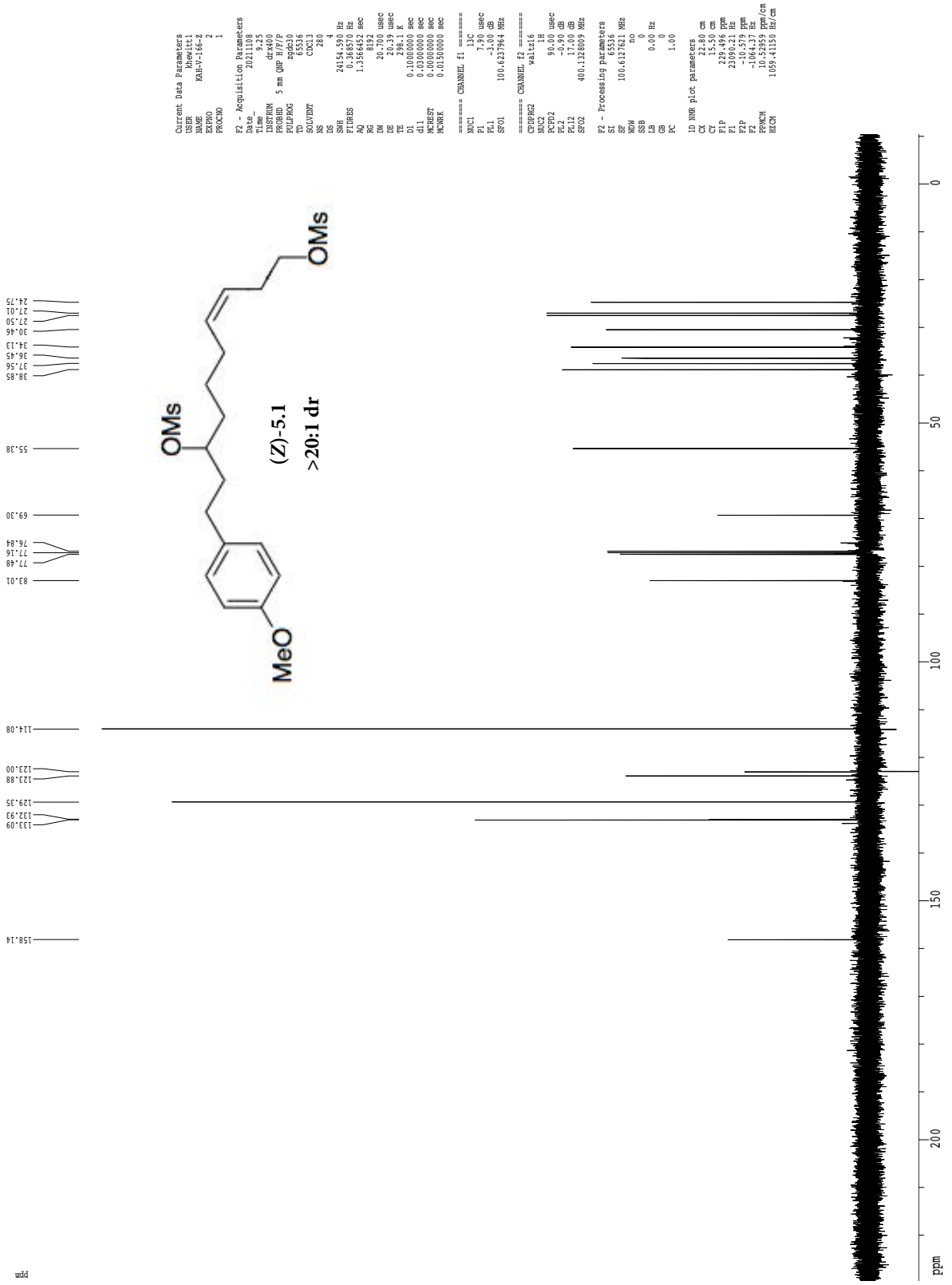
2D NMR plot parameters  
 CZ 15.00 cm  
 CX 15.00 cm  
 FZLO 450.84 Hz  
 FZHO 450.84 Hz  
 FZPH -0.506 ppm  
 FZPI -252.97 Hz  
 FZPO 9.005 ppm  
 FZLO 450.84 Hz  
 FZHO 450.84 Hz  
 FZPH -0.506 ppm  
 FZPI -252.97 Hz  
 FZPO 9.005 ppm  
 FZPMCH 0.63407 ppm/cm  
 FZPCMH 317.17413 Hz/cm  
 FZSCMH 0.64531 ppm/cm  
 FZPCSH 317.17413 Hz/cm



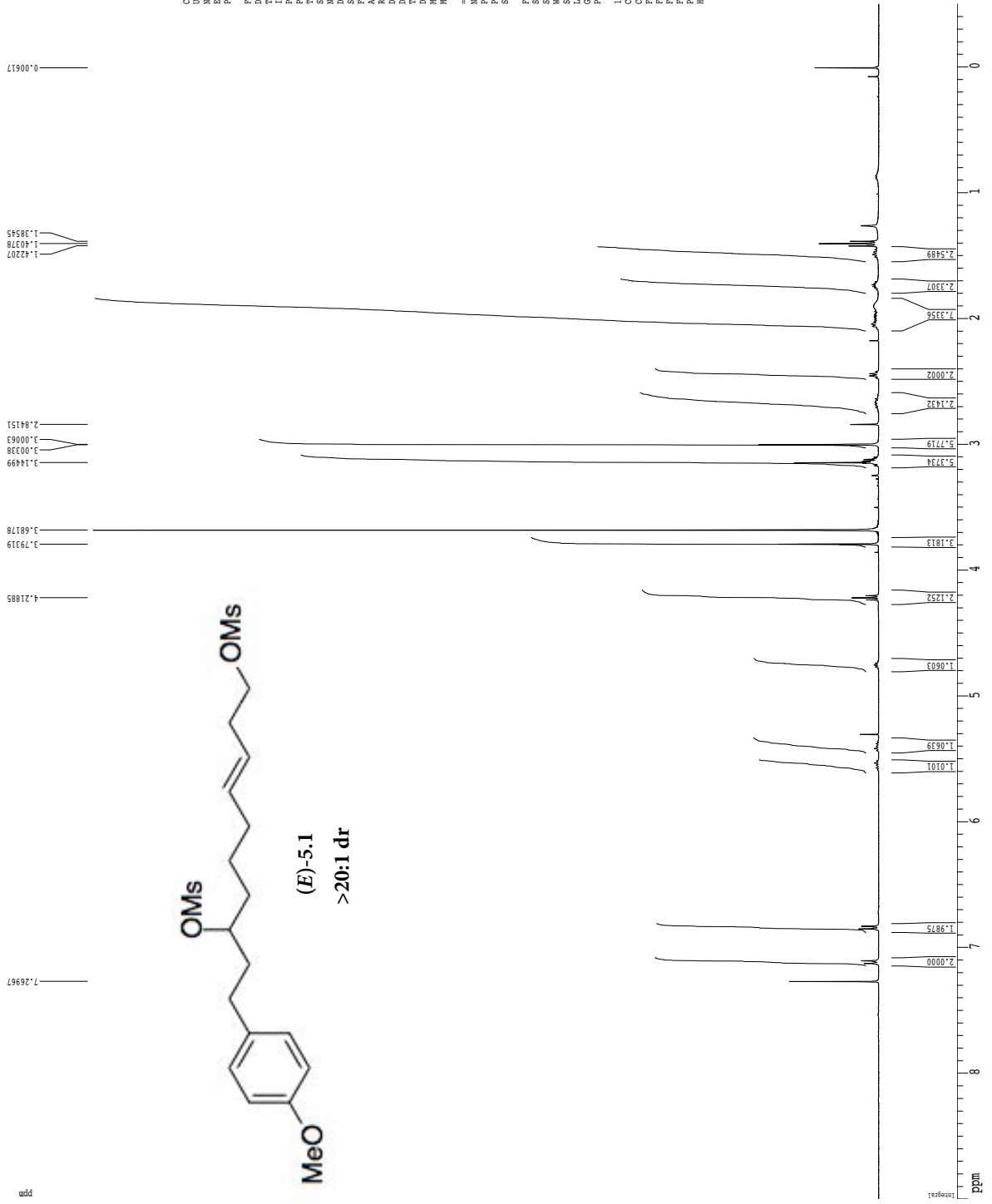
1H spectrum



<sup>13</sup>C spectrum with <sup>1</sup>H decoupling

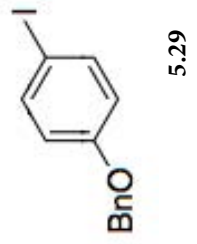
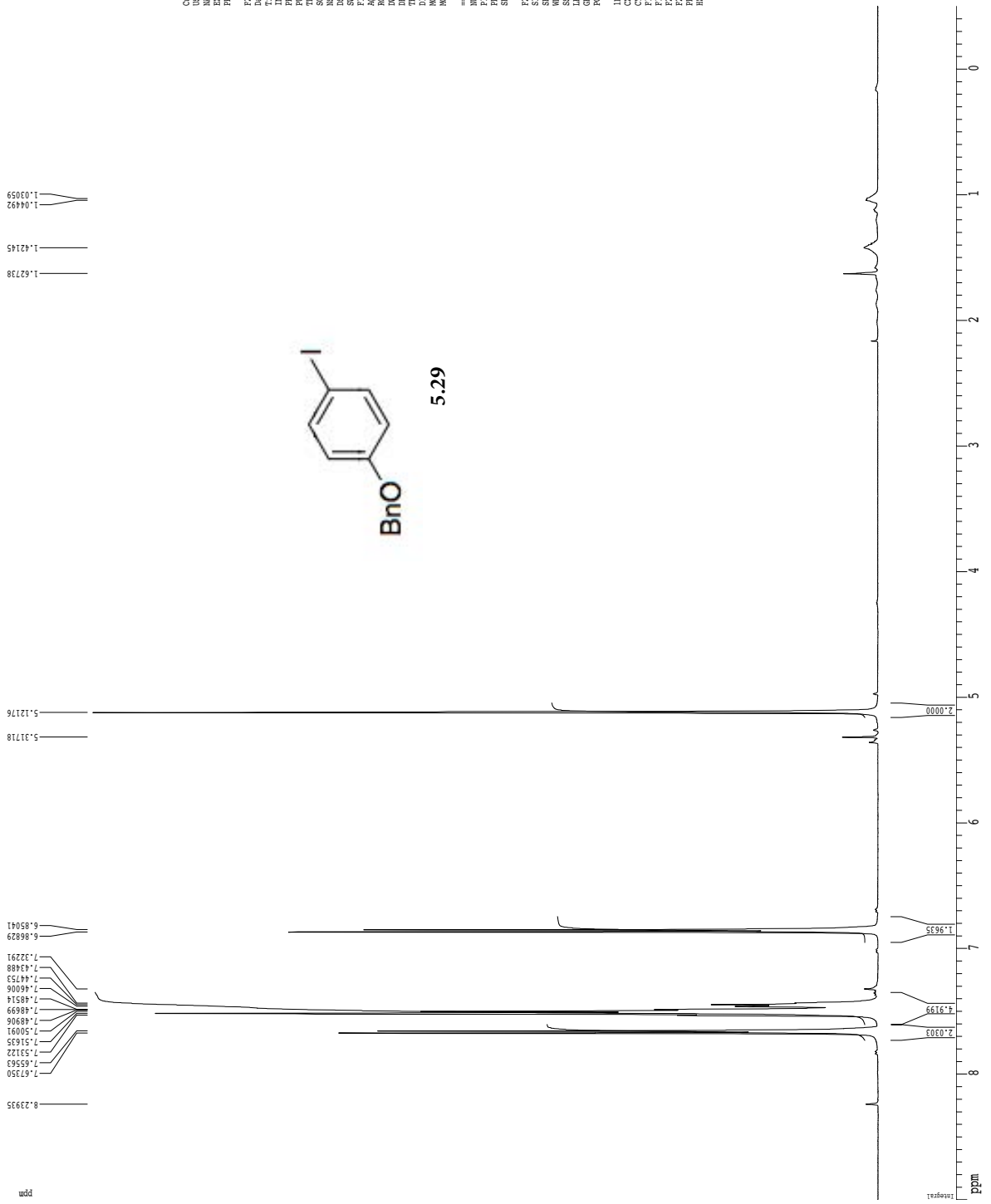


1H spectrum



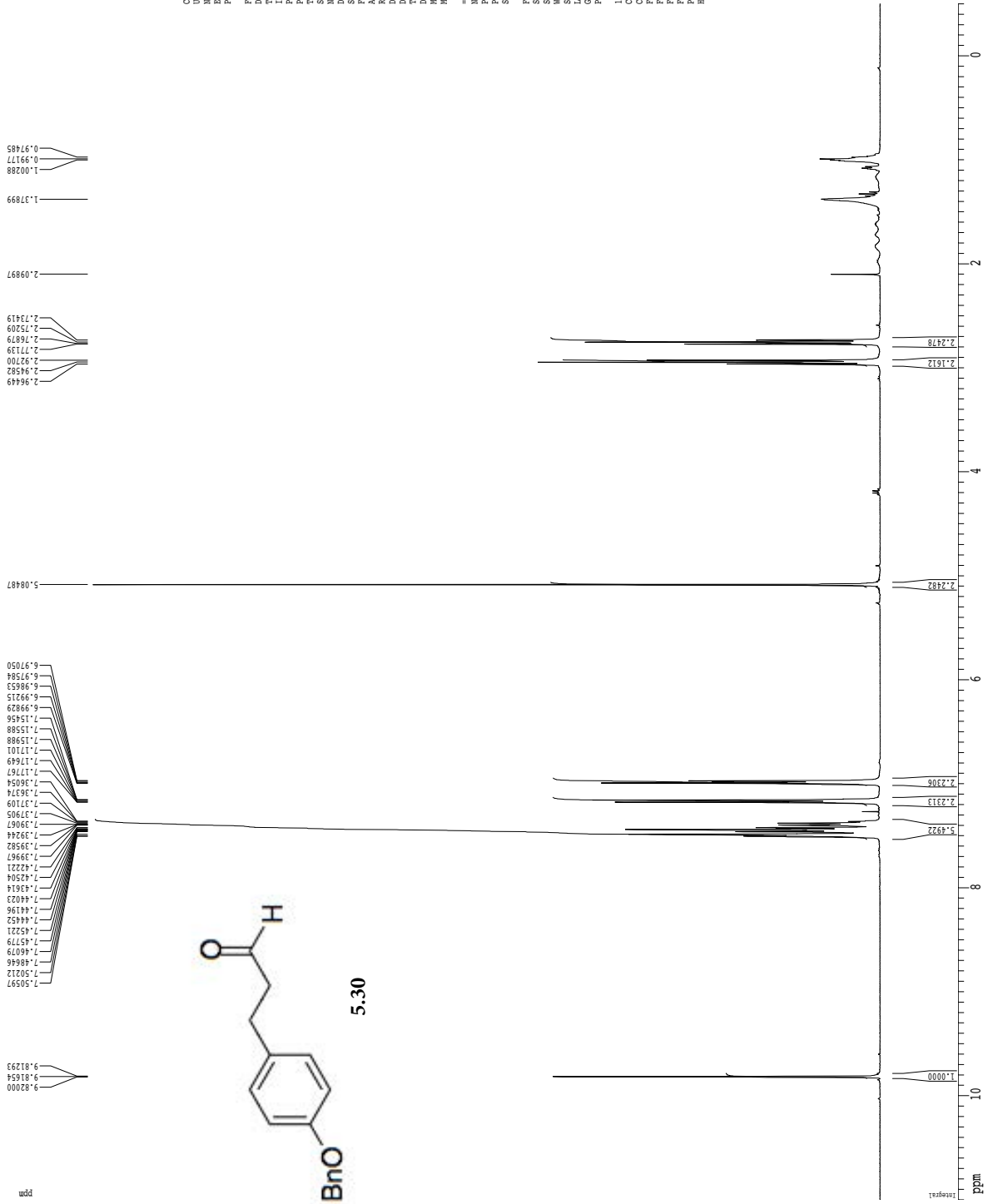


1H spectrum



Current Data Parameters  
USER hweiltl  
NAME KH-17-276-1  
PROCNO 1  
PROCDO 1  
F2 - Acquisition Parameters  
Date\_ 20210605  
Time 11:00:00  
INSTRUM cryo500  
PROBHD 5 mm CPCLP-1H-  
PULPROG zgpg30  
PC 4.00  
SOLVENT CDCl3  
NS 8  
DS 2  
SWH 6015.82 Hz  
FIDRES 0.166677 Hz  
AQ 2.9998677 sec  
RG 3.6  
DM 62.400 usec  
DE 1.900 usec  
TE 298.0 K  
D1 0.1000000 sec  
ACQRES 0.0000000 sec  
PCWAT 0.0100000 sec  
===== CHANNEL f1 =====  
NUC1 1H  
P1 9.00 usec  
PL1 0.00 dB  
SFO1 500.2235015 MHz  
F2 - Processing parameters  
SI 32768  
SF 500.2235000 MHz  
WDW no  
SSB 0  
GB 0  
CB 0.0 Hz  
PC 1.00  
ID MR plot parameters  
CT 15.00 cm  
CY 15.00 cm  
FIP 9.000 ppm  
FL 4501.88 Hz  
FR 20.000 ppm  
FZ -250.31 Hz  
PPMCH 0.41667 ppm/cm  
HZCH 208.42500 Hz/cm

1H spectrum



Current Data Parameters  
USER: Khe Wittl  
NAME: KKH-9-265-1  
PROCNO: 1  
PRUNO: 1  
PRCNO: 1

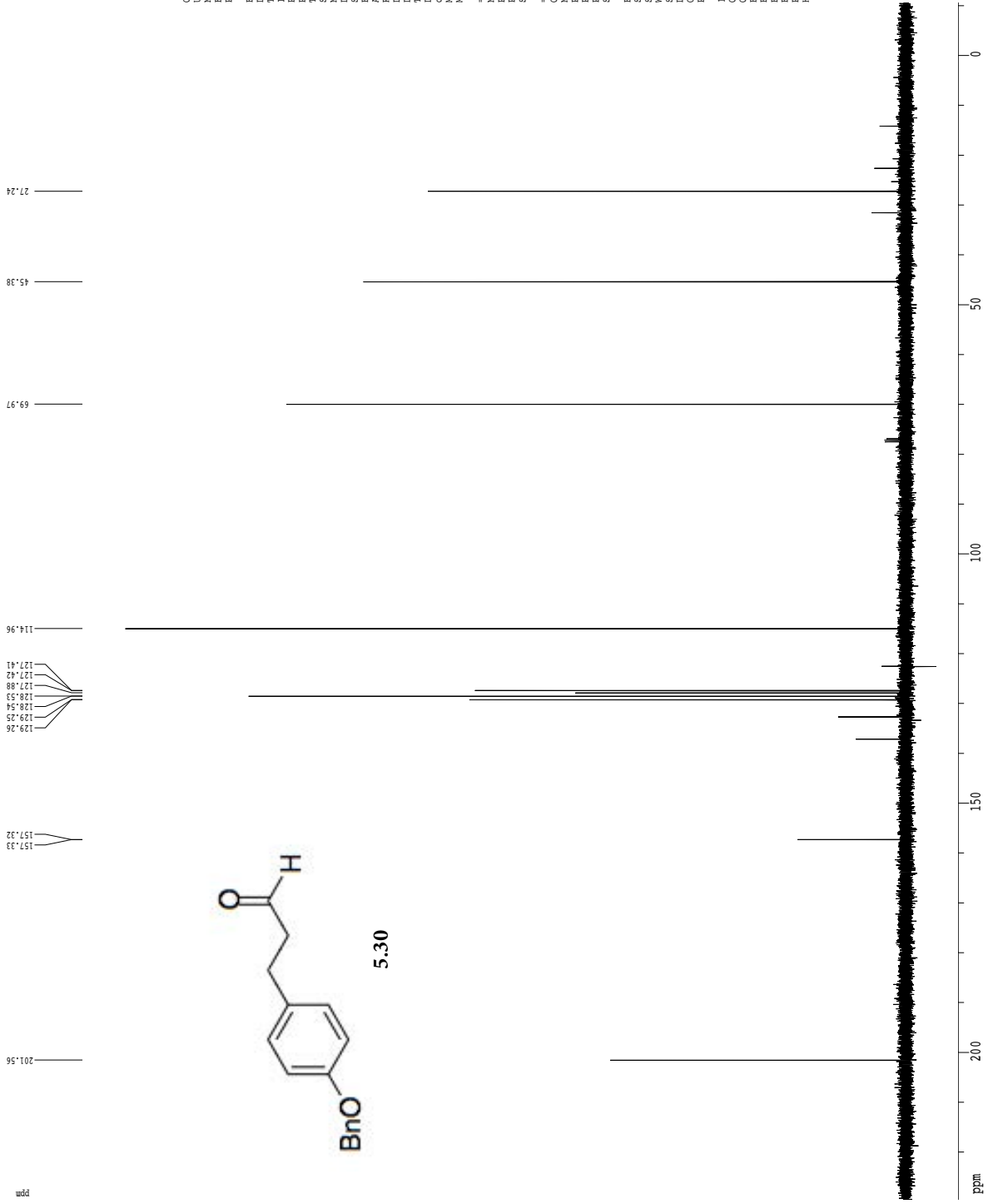
F2 - Acquisition Parameters  
Date\_: 2/22/05  
Time\_: 11:00  
INSTRUM: dcr400  
PROBHD: 5 mm QNP H/P/P  
PULPROG: zgpg30  
SOLVENT: CDCl3  
NS: 8  
DS: 2  
SWH: 640.252 MHz  
FIDRES: 0.149713 Hz  
AQ: 5.1118578 sec  
RG: 32  
DW: 78.000 usec  
DE: 1.900 usec  
TE: 298.2 K  
D1: 0.10000000 sec  
MCREST: 0.00000000 sec  
MCWXA: 0.01500000 sec

==== CHANNEL f1 =====  
NUC1: 1H  
P1: 12.00 usec  
PL1: -1.00 dB  
SFO1: 400.1328000 MHz

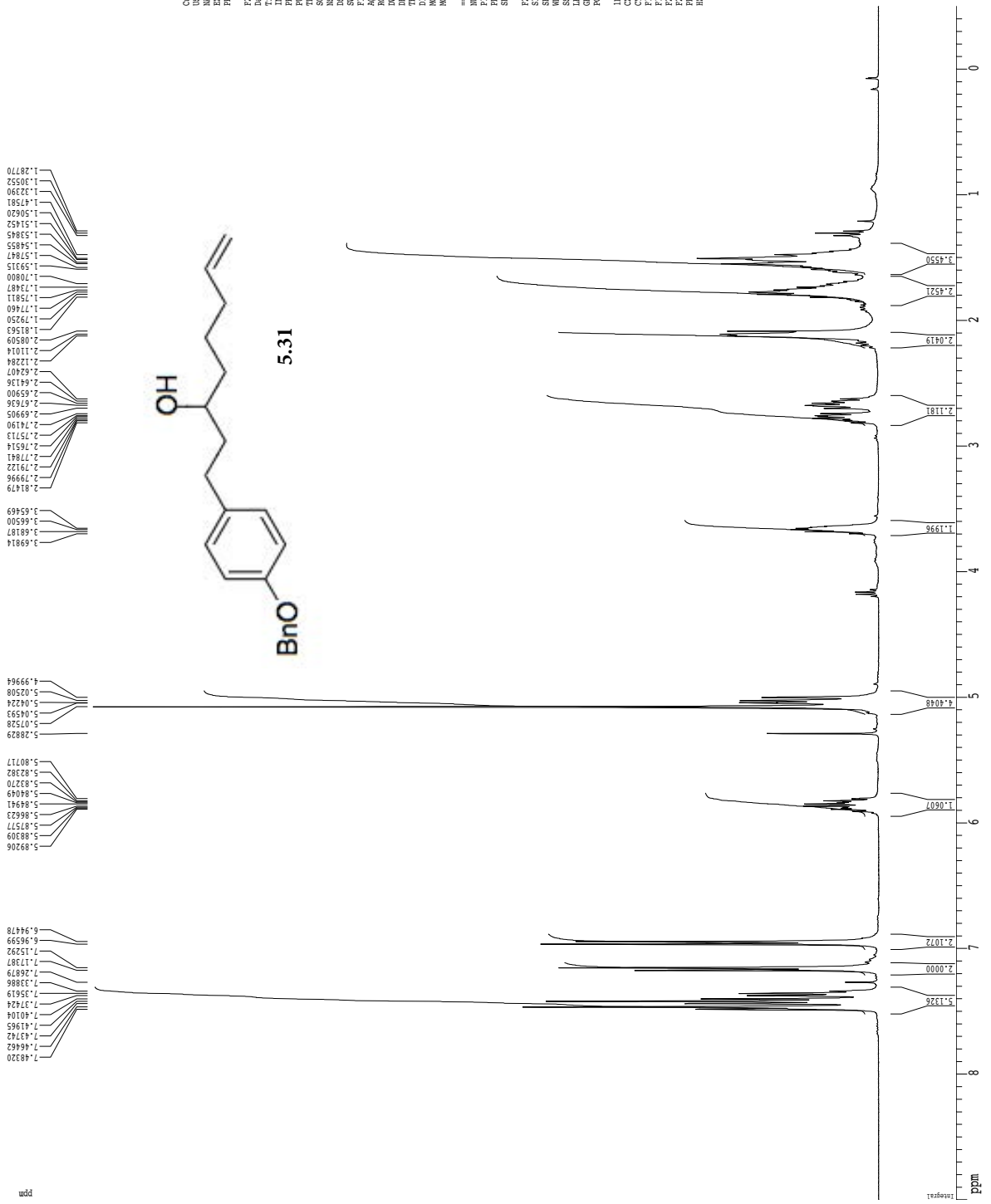
F2 - Processing parameters  
SI: 32768  
SF: 400.130175 MHz  
WDW: no  
SSB: 0  
GB: 0.00 Hz  
PC: 2.00

ID WDR Plot parameters  
CY: 4  
CX: 15.00 cm  
FIP: 11.000 ppm  
FI: 4401.48 Hz  
F2: 10000.000 ppm  
FZ: -200.00 Hz  
PRMCN: 0.50439 ppm/cm  
RMCN: 201.81996 Hz/cm

13C spectrum with 1H decoupling



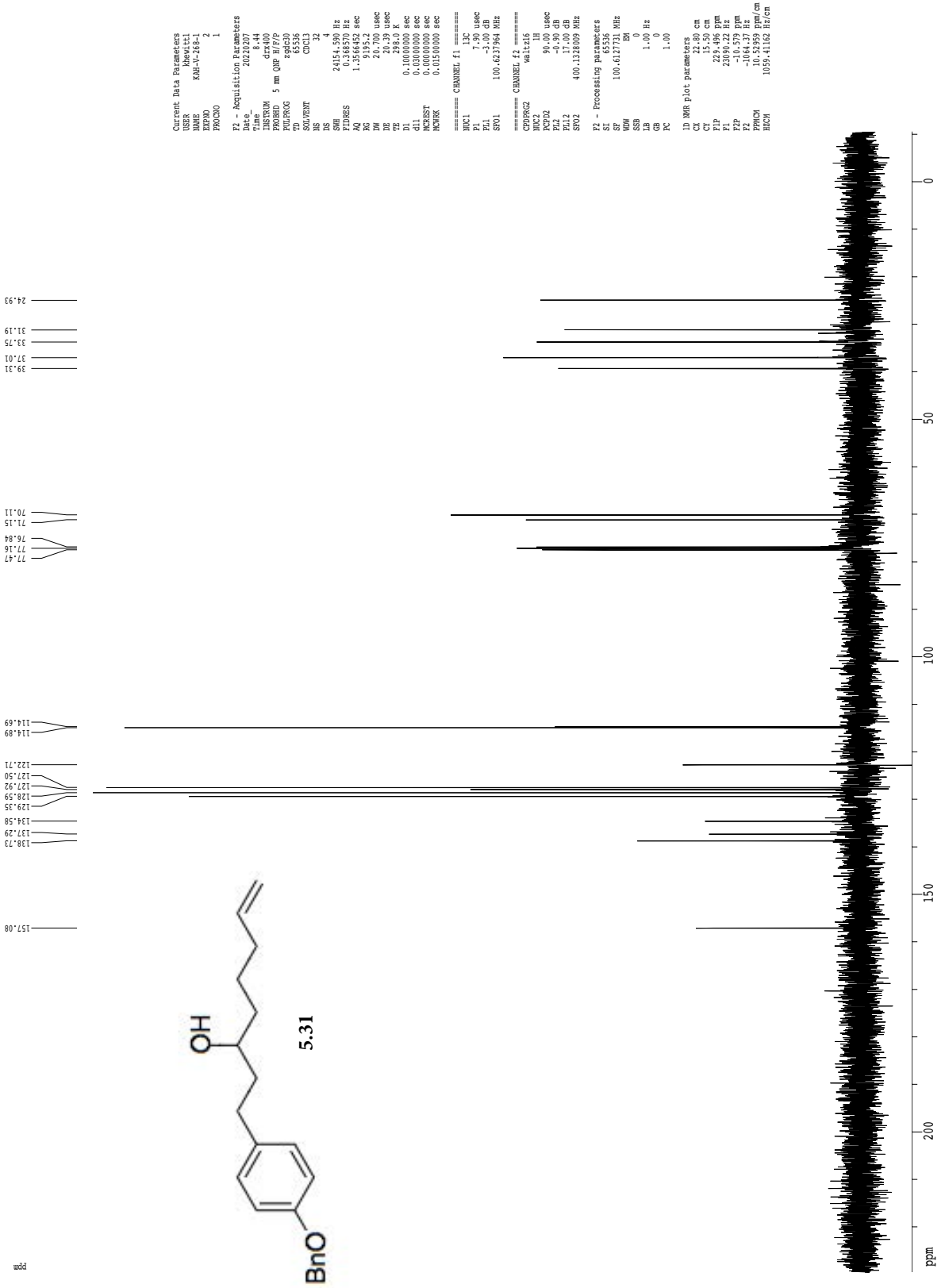
1H spectrum



Current Data Parameters  
 USER hewlett  
 NAME RAH-7-268-1  
 PROCNO 1  
 PULPROG zgpg30  
 F2 - Acquisition Parameters  
 Date\_ 2/22/07  
 Time\_ 12:00:00  
 INSTRUM dm400  
 PROBD 5 mm QNP H/P/P  
 PULPROG zgpg30  
 PC 32  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6132.00 Hz  
 FIDRES 0.166653 Hz  
 AQ 2.9999259 sec  
 RG 32  
 DW 78.000 usec  
 DE 19.000 usec  
 TE 298.2 K  
 D1 0.1000000 sec  
 MCHST 0.0000000 sec  
 PCNKK 0.0100000 sec  
 ===== CHANNEL f1 =====  
 NUCL1 1H  
 P1 12.00 usec  
 PL1 -0.00 dB  
 SFO1 400.1328009 MHz  
 F2 - Processing parameters  
 SI 65536  
 SF 400.1301175 MHz  
 WDM no  
 SSB 0  
 GB 0  
 CB 0.0 Hz  
 PC 2.00  
 ID MR plot parameters  
 CT 40  
 CY 15.00 cm  
 FIP 9.000 ppm  
 FI 3601.17 Hz  
 F2 65536.000 Hz  
 FZ -2702.06 Hz  
 FPMCH 0.41667 ppm/cm  
 HZCM 166.72084 Hz/cm



13C spectrum with 1H decoupling



Current Data Parameters  
 USER Rkwelttl  
 NAME KM-V-268-1  
 PROBA 2  
 PROCIO 1

F2 - Acquisition Parameters  
 Date\_ 20220707  
 Time\_ 14:46  
 INSTRUM dr400  
 PROBHD 5 mm QNP H/FP  
 PULPROG zgpg30  
 CD 6536  
 ACQPRG1 C0512  
 NS 32  
 DS 4  
 SM 24154.590 Hz  
 TD 65536  
 FIDRES 0.156470 Hz  
 RG 9195.2  
 AC 20.700 MHz  
 DM 20.700 MHz  
 DE 20.39 MHz  
 TE 300.2 K  
 D1 0.1000000 sec  
 d11 0.0300000 sec  
 ACRESF 0.0000000 sec  
 ACWBK 0.0150000 sec

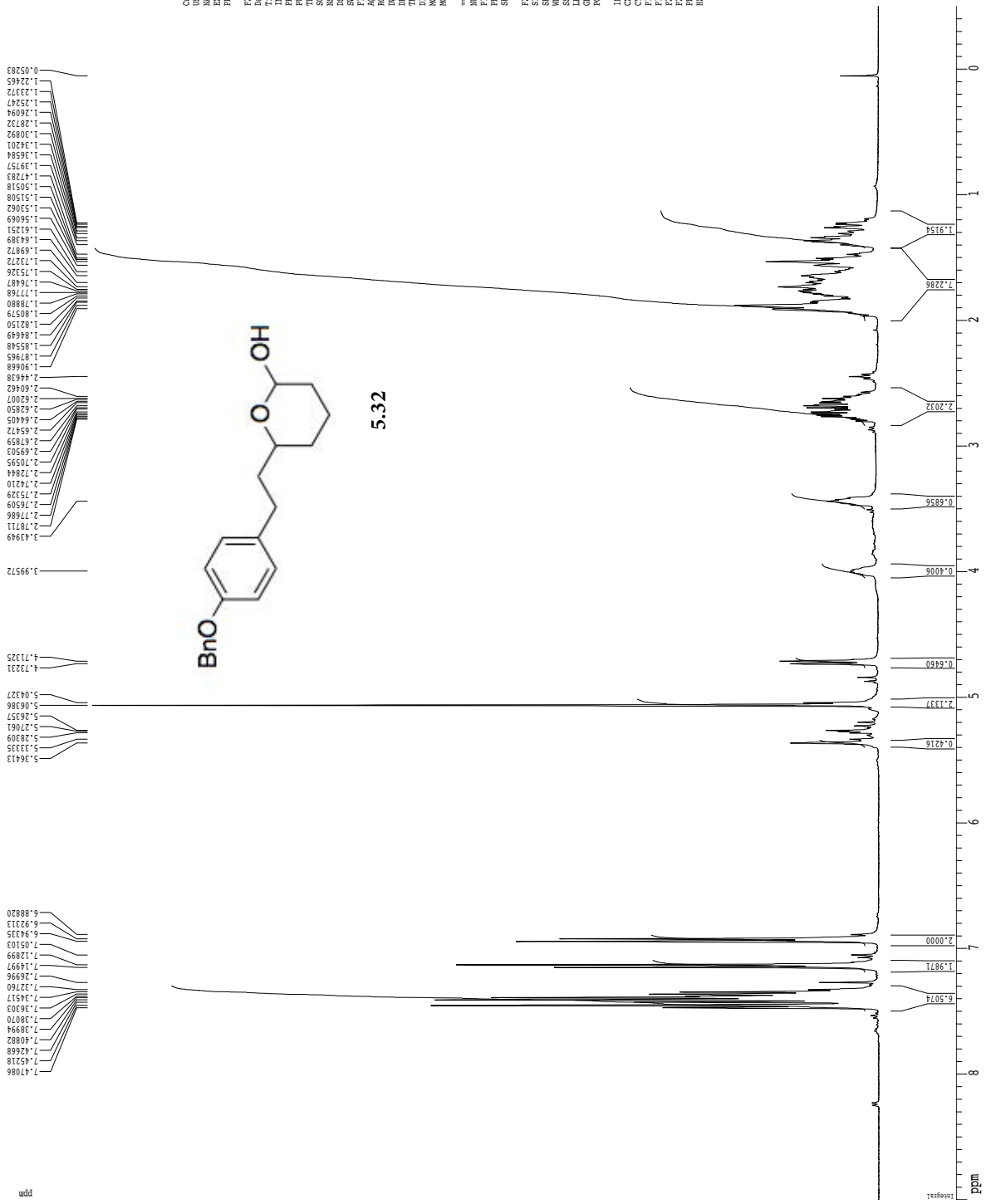
===== CHANNEL f1 =====  
 NU1 13C  
 P1 7.50 MHz  
 PL 2.00 dB  
 SFO1 100.627904 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NU2 13C  
 P2 90.00 MHz  
 PL2 -6.50 dB  
 PL12 17.00 dB  
 SFO2 400.1328009 MHz

F2 - Processing parameters  
 SI 65536  
 SF 100.6127731 MHz  
 DS 65536  
 AS 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

1D NMR Post parameters  
 CX 22.50 cm  
 CY 15.50 cm  
 FIP 228.496 ppm  
 F1 25.00 MHz  
 F2 -1064.37 Hz  
 PRGCM 10.53959 ppm/cm  
 HZCM 1039.41162 Hz/cm

1H spectrum

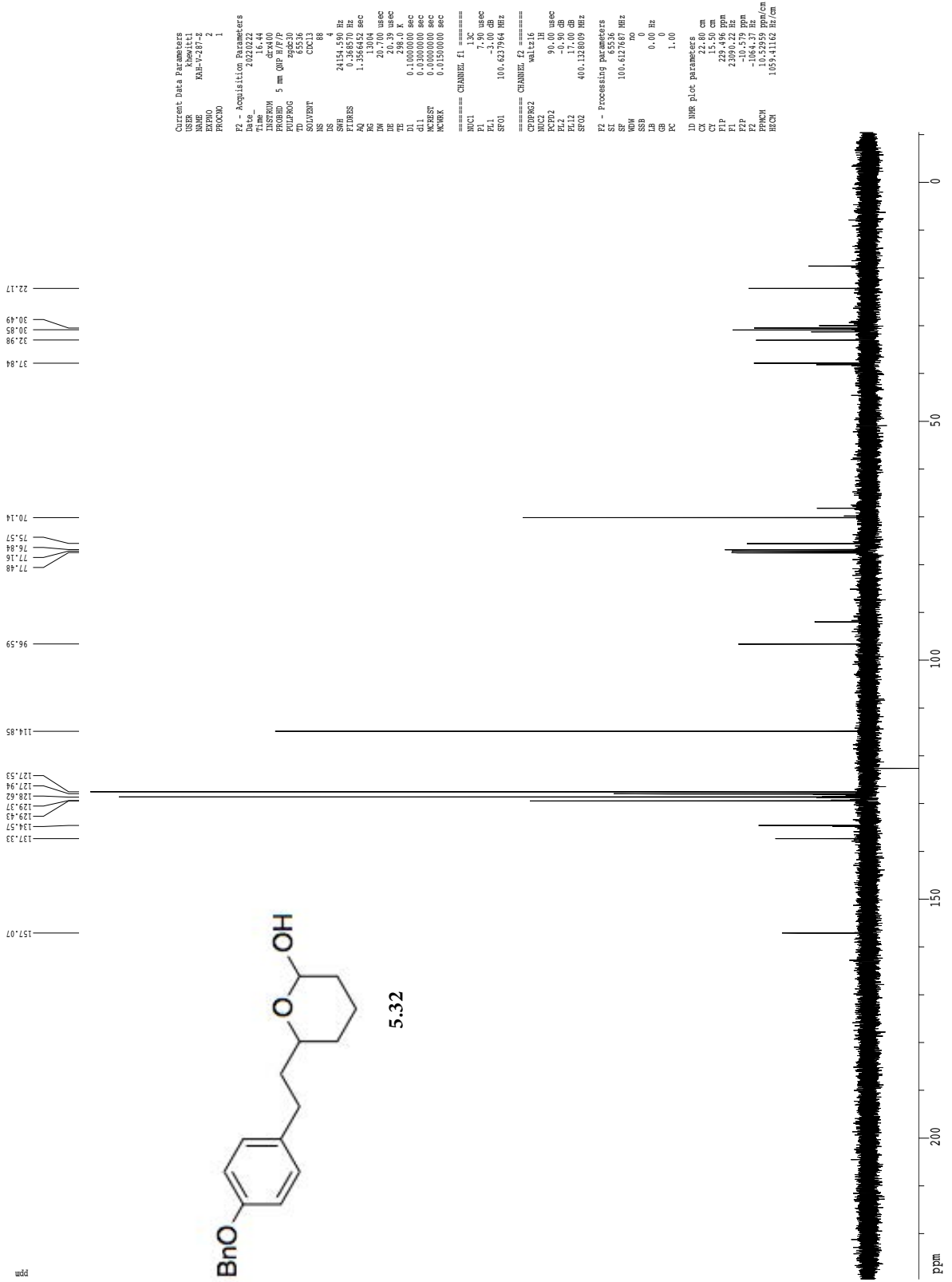


Current Data Parameters  
 USER hweiltl  
 NAME RAH-7-87-2  
 PROCNO 1  
 PULPROG zgpg30  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6412.90 Hz  
 FIDRES 0.166653 Hz  
 AQ 2.9999259 sec  
 RG 57  
 DW 78.000 usec  
 DE 238.0 KHz  
 TE 298.0 K  
 DL 0.1000000 sec  
 ACQRES 0.0000000 sec  
 PCW 0.0130000 sec  
 ===== CHANNEL f1 =====  
 NUCL1 1H  
 P1 12.00 usec  
 PL1 -0.00 dB  
 SFO1 400.1328009 MHz

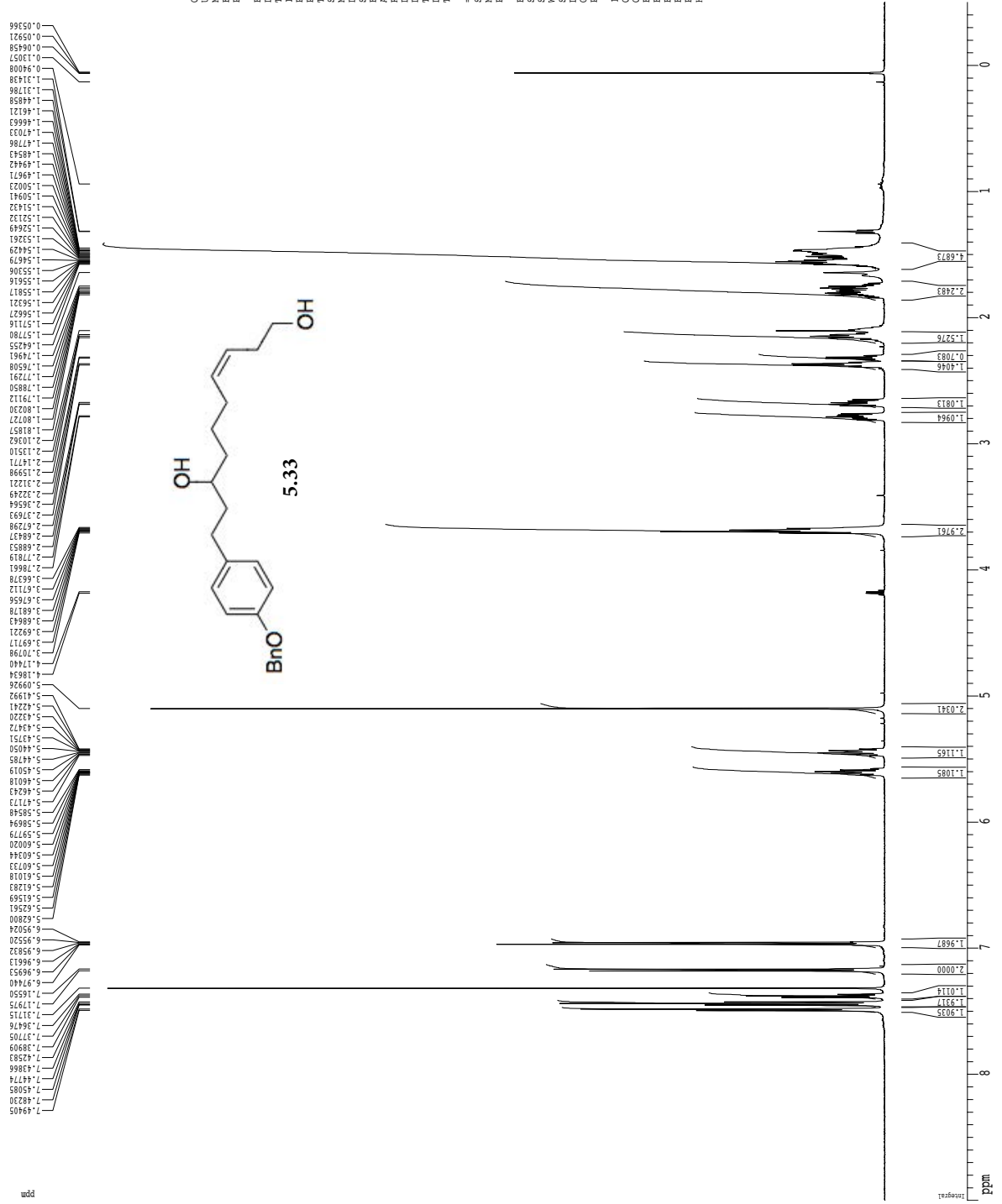
F2 - Processing parameters  
 SI 32768  
 SF 400.130175 MHz  
 WDW mo  
 SSB 0  
 GB 0  
 CB 0.0 Hz  
 PC 2.00

ID MRB plot parameters  
 CT 15.00 cm  
 CY 15.00 cm  
 FIP 9.000 ppm  
 FI 3601.17 Hz  
 F2 -700.06 Hz  
 F2 0.41667 ppm/cm  
 HZCM 166.72084 Hz/cm

13C spectrum with 1H decoupling

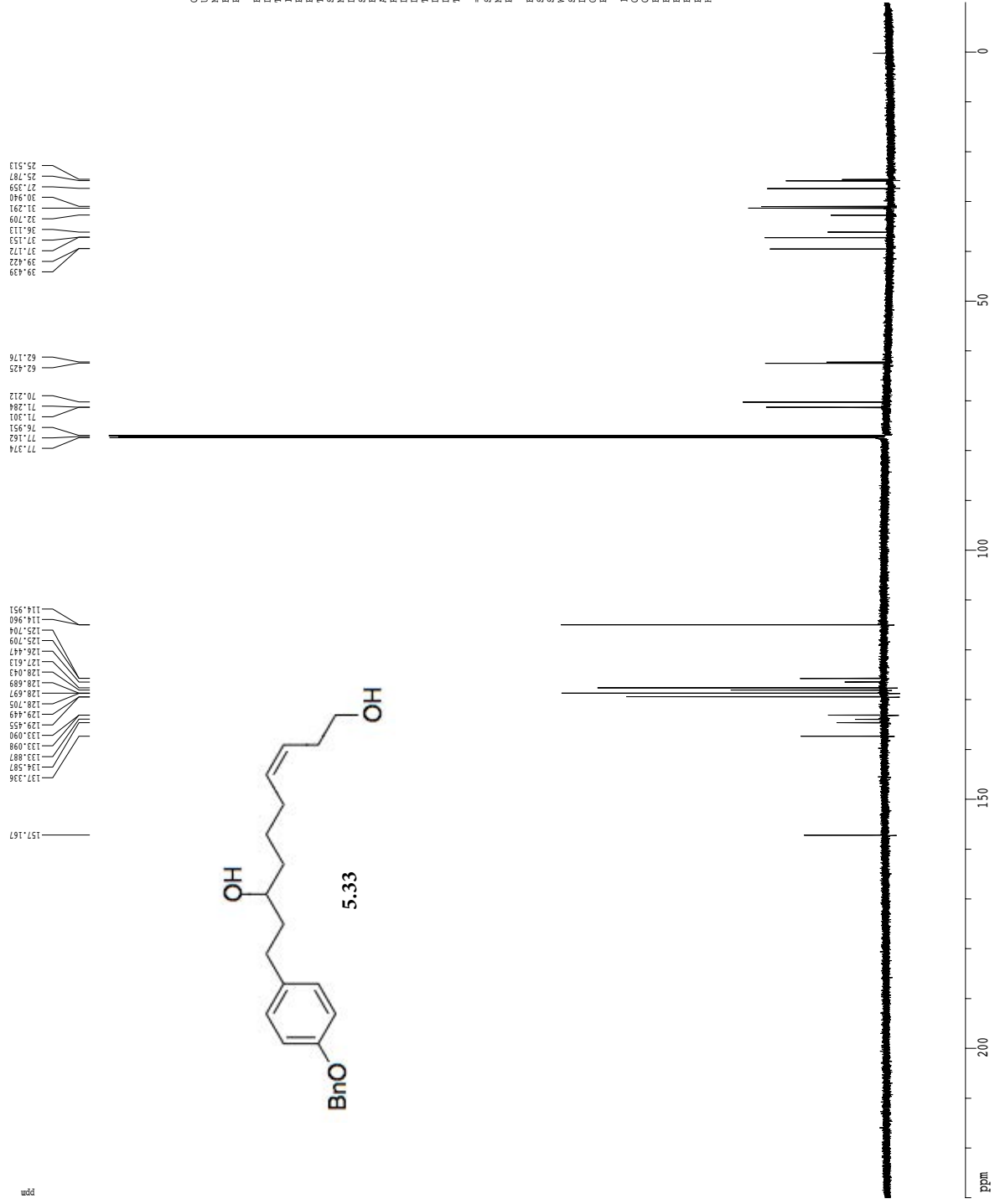


1H spectrum



Current Data Parameters  
NAME K01-VZ-201-6101-2  
EXPNO 2  
PROCNO 1  
P2 - Acquisition Parameters  
Date\_ 20220225  
Time 14.35  
INSTRUM av600  
PROBHD 5 mm CRES  
PULPROG zgpg30  
TD 98074  
SOLVENT CDCl3  
DS 8  
SS 2  
SF 9615.385 Hz  
FIDRES 0.098042 Hz  
AQ 5.0998979 sec  
RG 256  
DD 52.000 usec  
DE 14.23 usec  
TE 297.5 K  
D1 0.1000000 sec  
D20 1  
===== CHANNEL f1 =====  
SFO1 600.1362000 MHz  
NUC1 13C  
PC 9.50 usec  
P2 - Processing parameters  
SI 6556  
SF 600.1362000 MHz  
WDW 500  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00  
ID INR Plot parameters  
CX 22.80 cm  
CY 11.40 cm  
F1 5.000 ppm  
F2 5403.17 Hz  
F3 -0.500 ppm  
F4 0.000 Hz  
F5 0.000 Hz  
F6 0.000 Hz  
F7 0.000 Hz  
F8 0.000 Hz  
F9 250.05418 Hz/cm

13C spectrum



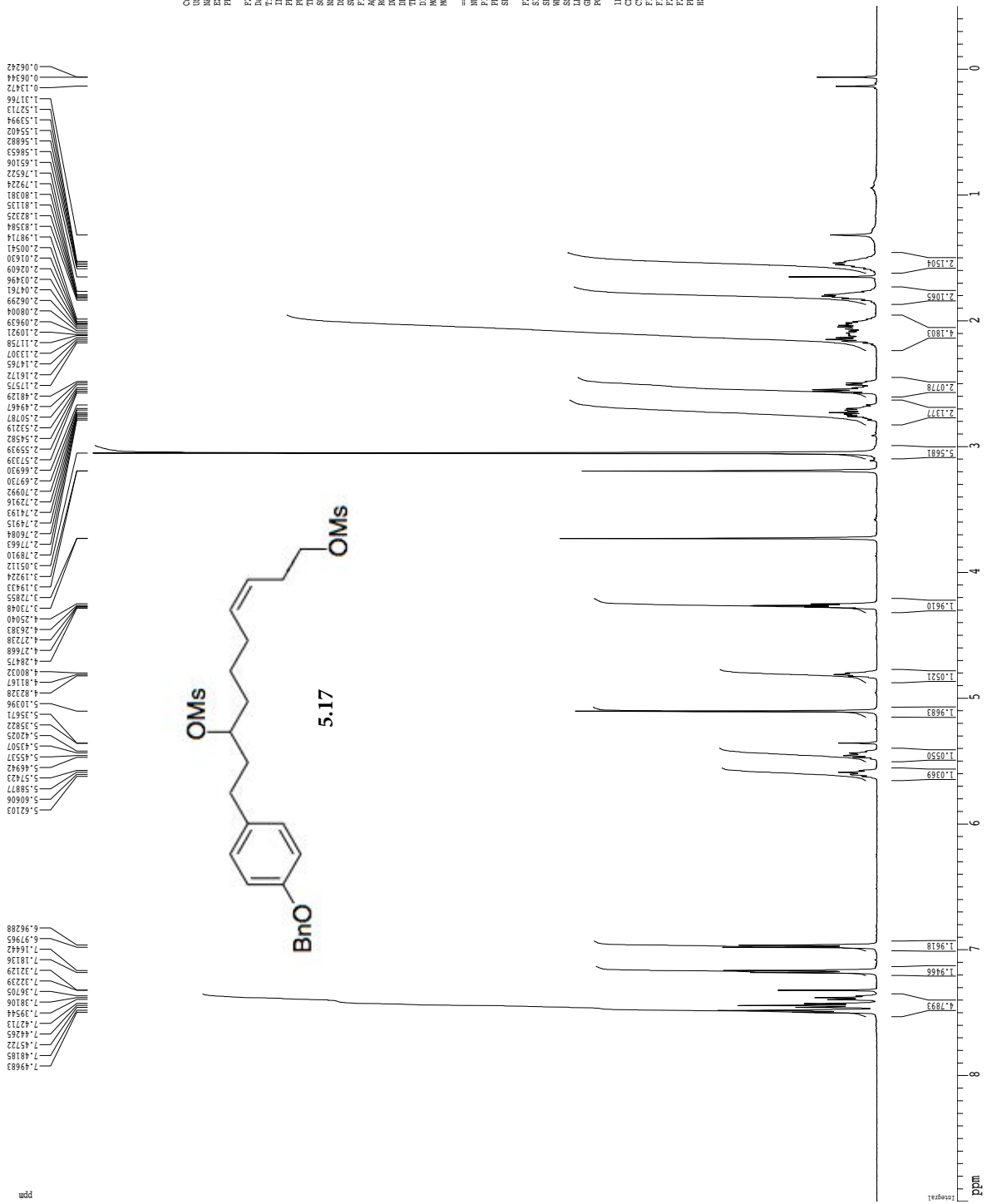
Current Data Parameters  
 Date\_ 20220225  
 Time\_ 14:29  
 INSTRUM av600  
 PULPROG zgpg30  
 FIDRES 0.5044468 sec  
 TD 65536  
 SOLVENT CDCl3  
 DS 180  
 SFO 36231.883 Hz  
 AQ 0.552855 Hz  
 DE 19.453 usec  
 TE 297.5 K  
 D1 0.40000001 sec  
 D11 0.50000001 sec  
 TDO 1

===== CHANNEL f1 =====  
 NUC1 13C  
 NUCL1 13C  
 P1 10.10 usec

F2 - Processing parameters  
 SF 150.907542 MHz  
 WFM no  
 SSB 0  
 GB 0.00 Hz  
 PC 1.00

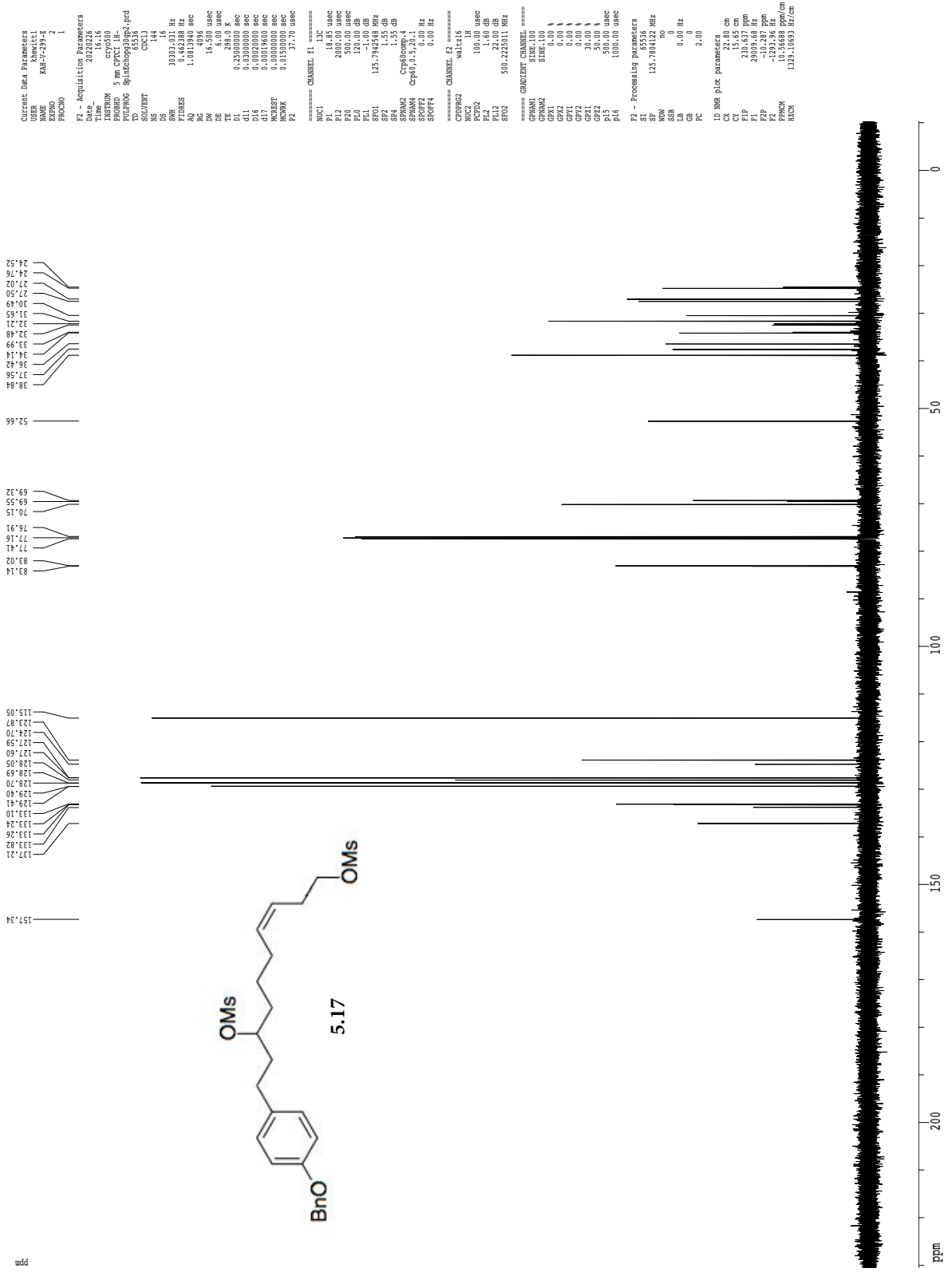
ID MR Plot parameters  
 X 25.00 cm  
 Y 15.00 cm  
 FIP 230.000 Dpm  
 F1 34707.64 Hz  
 F2 -1500.00 Dpm  
 FZ -1500.00 Dpm  
 PPRQ1 10.52632 Dpm/cm  
 RECN 1588.45068 Hz/cm

1H spectrum

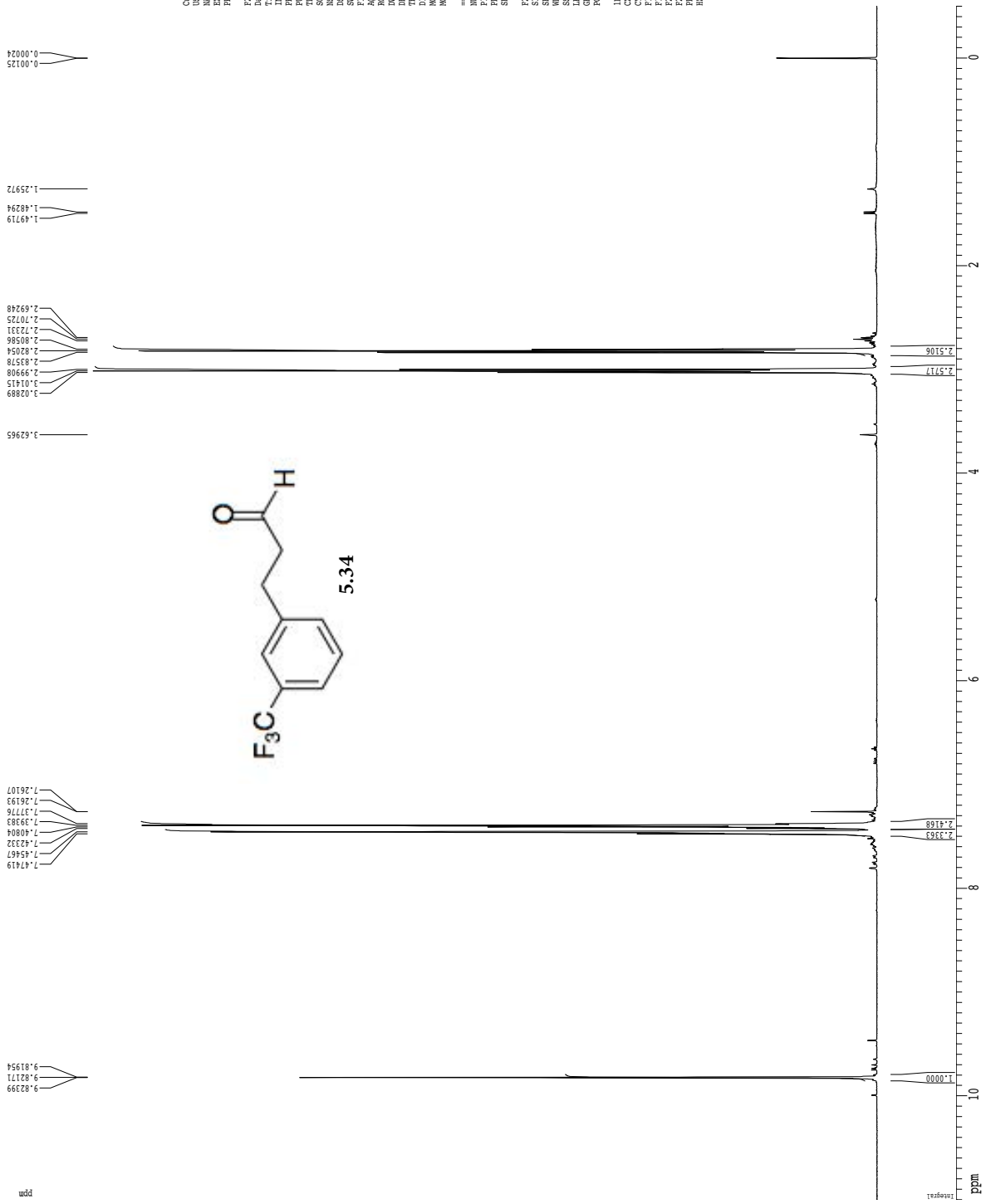


Current Data Parameters  
 USER hewlett1  
 NAME RAH-7-29-2  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 2/22/24  
 Time\_ 12:22:24  
 INSTRUM cryo500  
 PROBEID 5 mm CPCLP 1H-  
 PULPROG zgpg30  
 AQ 4.00  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6013.62 Hz  
 FIDRES 0.166677 Hz  
 AQ 2.9998677 sec  
 RG 5  
 DW 62.400 usec  
 DE 1.900 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 MCHST 0.00000000 sec  
 MCHNK 0.01500000 sec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 0.00 dB  
 SFO1 500.2235015 MHz  
 F2 - Processing parameters  
 SI 32768  
 SF 500.2235000 MHz  
 WDW EM  
 SSB 0  
 GB 0.00 Hz  
 CB 1.00  
 PC 1.00  
 ID MR plot parameters  
 CT 40  
 CY 15.00 cm  
 FIP 9.000 ppm  
 FI 4501.88 Hz  
 F2 -750.11 Hz  
 FPCW 0.41667 ppm/cm  
 HZCN 208.42500 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling

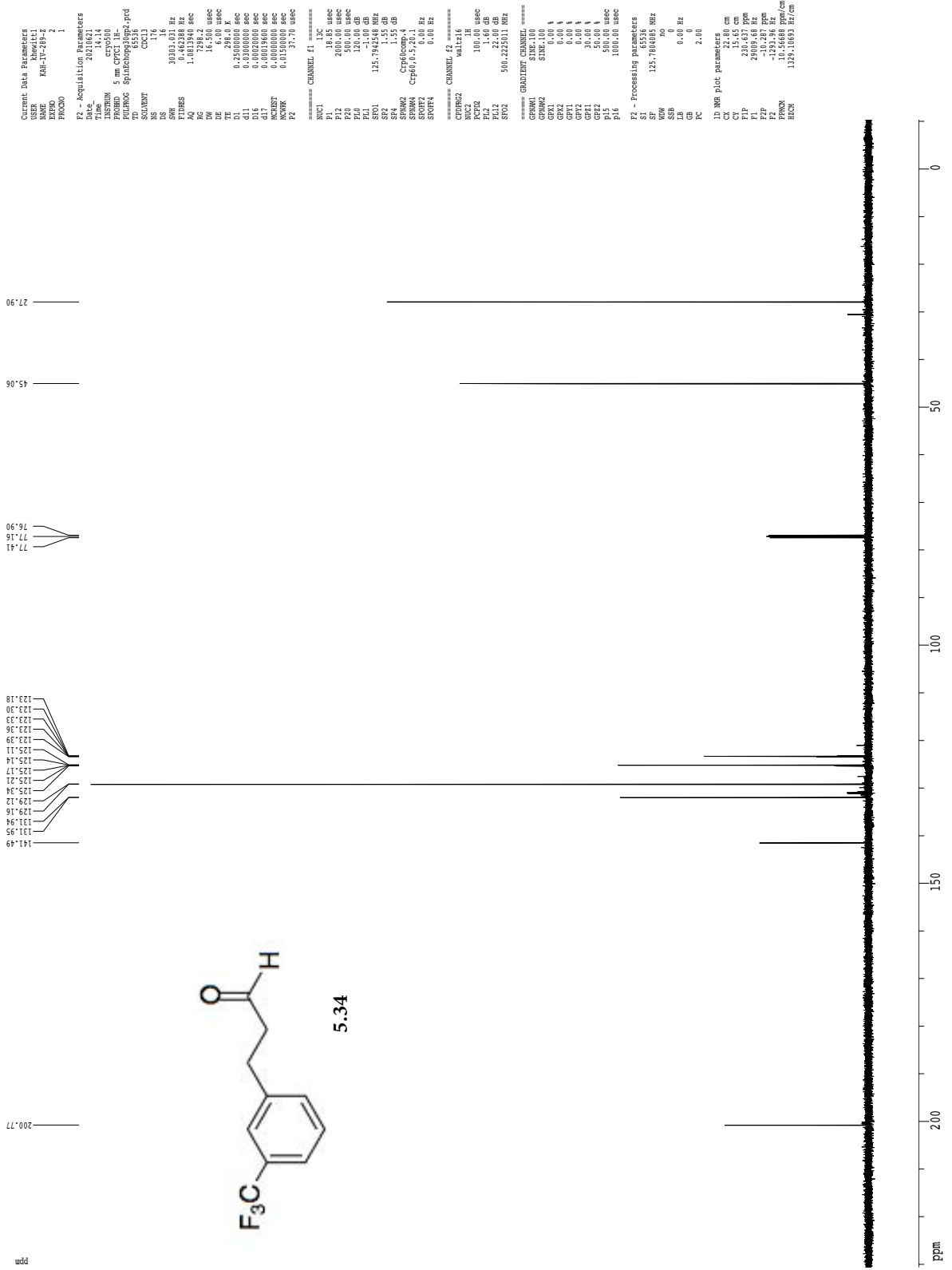


1H spectrum





Z-restored spin-echo 13C spectrum with 1H decoupling



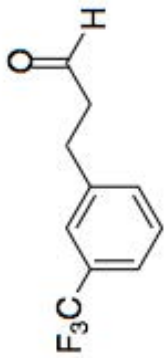
```

Current Data Parameters
USER      khewitt1
EXPNO    2
PROCNO   1
Date_    20231021
Time     14.14
INSTRUM  spect
PROBHD   5 mm CPY 1H
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        177
DS        16
SFO1     503.2021 Hz
SFO2     125.764380 MHz
AQ        1.0813946 sec
RG        7289.2
DE        16.000000 usec
TE        298.2 K
D1        0.25000000 sec
d11       0.00100000 sec
D16       0.00220000 sec
d17       0.00139000 sec
ACQBR    0.00100000 sec
SFO3     100.628150 MHz
SFO4     37.70 usec
===== CHANNEL f1 =====
NUC1     13C
P1       18.85 usec
P2       200.00 usec
PL1     0.00 dB
PL2     120.00 dB
PL3     -1.00 dB
SFO5     125.794380 MHz
SFO6     1.55 dB
SFO7     Cpdecoupl-4
SFO8     Cp16.15.0.0 Hz
SFO9     0.00 Hz
SFO10    0.00 Hz
===== CHANNEL f2 =====
CPROG2   waltz16
NUC2     1H
P1       100.00 usec
PL1     0.00 dB
PL2     22.00 dB
SFO11    500.225011 MHz
===== GRADIENT CHANNEL =====
GPM1     SINE.100
GPM2     SINE.100
GPR1     0.00 V
GPR2     0.00 V
GPR3     0.00 V
GPR4     30.00 V
GPR5     30.00 V
P15      50.00 usec
P16      100.00 usec
F2 - Processing parameters
SI        0
SF        125.764380 MHz
WDW       no
SSB       0
GB        0
PC        2.00
ID_NMR   plot Parameters
CX        22.80 cm
CT        15.65 cm
FI        2400.00 Hz
F2        -10.287 Ppm
F3        1.291536 Hz
F4        1.000000 Hz
HSCN     1329.10659 Hz/cm
  
```

f19.c

1

0.19729



5.34

Current Data Parameters  
Date\_ 20210621  
Time\_ 14:54  
INSTRUM av600  
PROBHD 5 mm QNP1H  
PULPROG zgpg30  
TD 131072  
SOLVENT CDCl3  
DS 6  
SS 6  
SFO 178571.422 Hz  
FIDRES 1.362392 Hz  
AQ 0.1670516 sec  
RG 2.800 usec  
DE 18.400 usec  
TE 298.0 K  
D1 3.0000000 sec  
TD 1  
===== CHANNEL f1 =====  
SFO1 564.629196 MHz  
NUC1 13C  
P1 18.75 usec  
===== CHANNEL f2 =====  
SFO2 131.072 MHz  
NUC2 13C  
P2 0.00 Hz  
GB 0  
LB 0.00 Hz  
PC 1.00  
===== CHANNEL f3 =====  
SFO3 22.80 MHz  
NUC3 1H  
P3 58.060 usec  
F1 32785.75 Hz  
F2 -288.171 ppm  
F3 -143.85471 ppm/cm  
F4 7832.06008 Hz/cm

ppm

-50

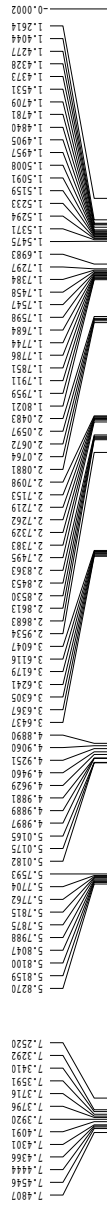
-100

-150

-200

-250

h1.c

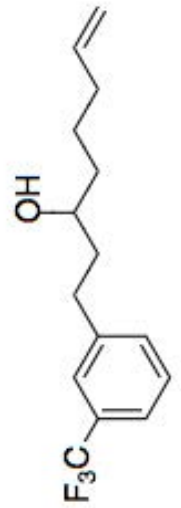


Current Data Parameters  
Date 20210703  
Time 12.00  
INSTRUM av600  
PROBHD 5 mm CPBPR  
PULPROG zgpg30  
TD 98074  
SOLVENT CDCl3  
DS 8  
SS 2  
SF 9615.385 Hz  
AQ 0.098842 Hz  
FIDRES 5.0998975 sec  
RG 64  
DE 14.23 usec  
TE 298.0 K  
D1 0.1000000 sec  
TD 1

==== CHANNEL f1 =====  
SFO1 600.134000 MHz  
NUC1 13C  
P1 9.50 usec  
PL 0.00 dB

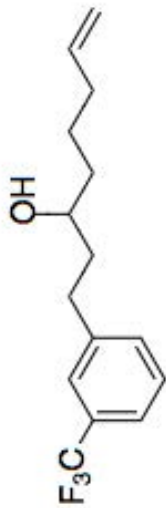
F2 - Processing parameters  
SI 6556  
SF 600.134000 MHz  
WDW EM  
SSB 0  
GB 0  
LB 0.00 Hz  
PC 1.00

ID NMR Plot parameters  
CX 22.80 cm  
FL 1.50 mm  
FID 9.000 ppm  
F1 5403.17 Hz  
F2P -0.500 ppm  
F3P 0.000 Hz  
F4P 0.000 Hz  
F5P 0.000 Hz  
REFC 250.05420 Hz/cm

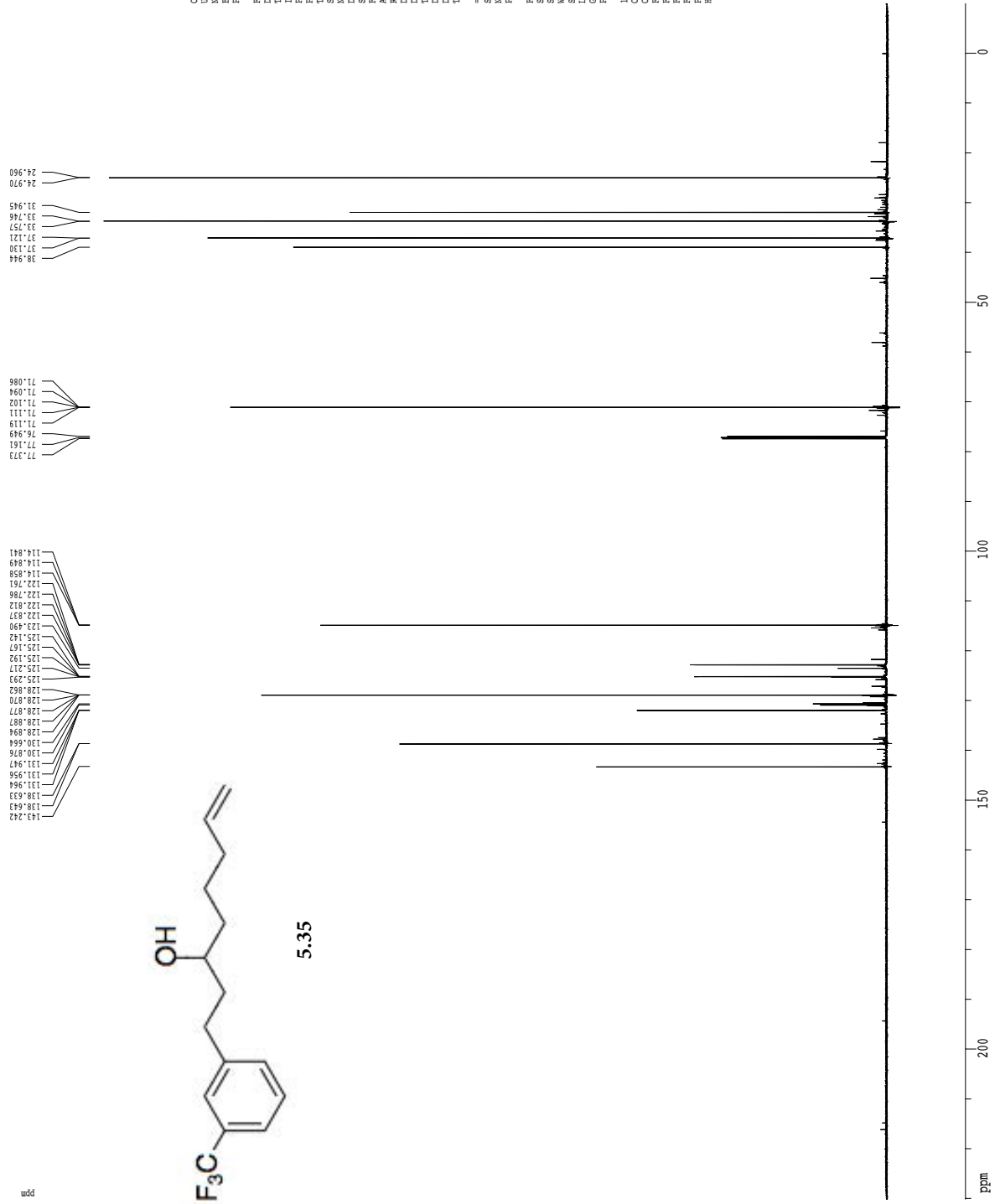


13C.c

ppm



5.35



Current Data Parameters  
NAME KSH-IV-244-2  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210703  
Time 11.56  
INSTRUM spect  
PROBHD 5 mm CBO-1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 12  
DS 4  
SWH 36231.883 Hz  
FIDRES 0.552855 Hz  
AQ 0.5004468 sec  
RG 662  
DM 13.00 usec  
DE 19.63 usec  
TE 298.0 K  
DT 0.4000001 sec  
D1 0.2300001 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 150.9194186 MHz  
NUC2  
PC 10.10 usec

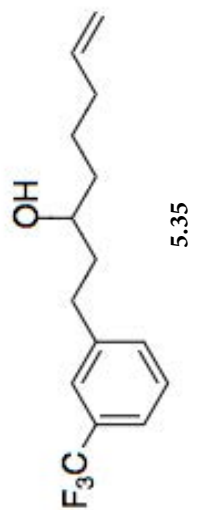
F2 - Processing parameters  
SI 32768  
SF 150.9077866 MHz  
WDW no  
SSB 0  
LB 0.0 Hz  
GB 0  
PC 1.00

ID NMR plot parameters  
CX 15.00 cm  
CY 15.00 cm  
FLP 230.117 ppm  
F1 34725.34 Hz  
F2 69450.68 Hz  
F3 104176.02 Hz  
FPMCH 10.53074 ppm/cm  
HZCM 1589.11780 Hz/cm

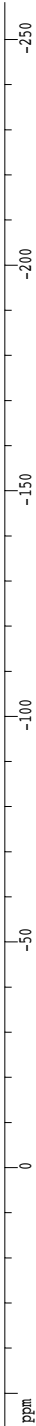
f19.c

1H

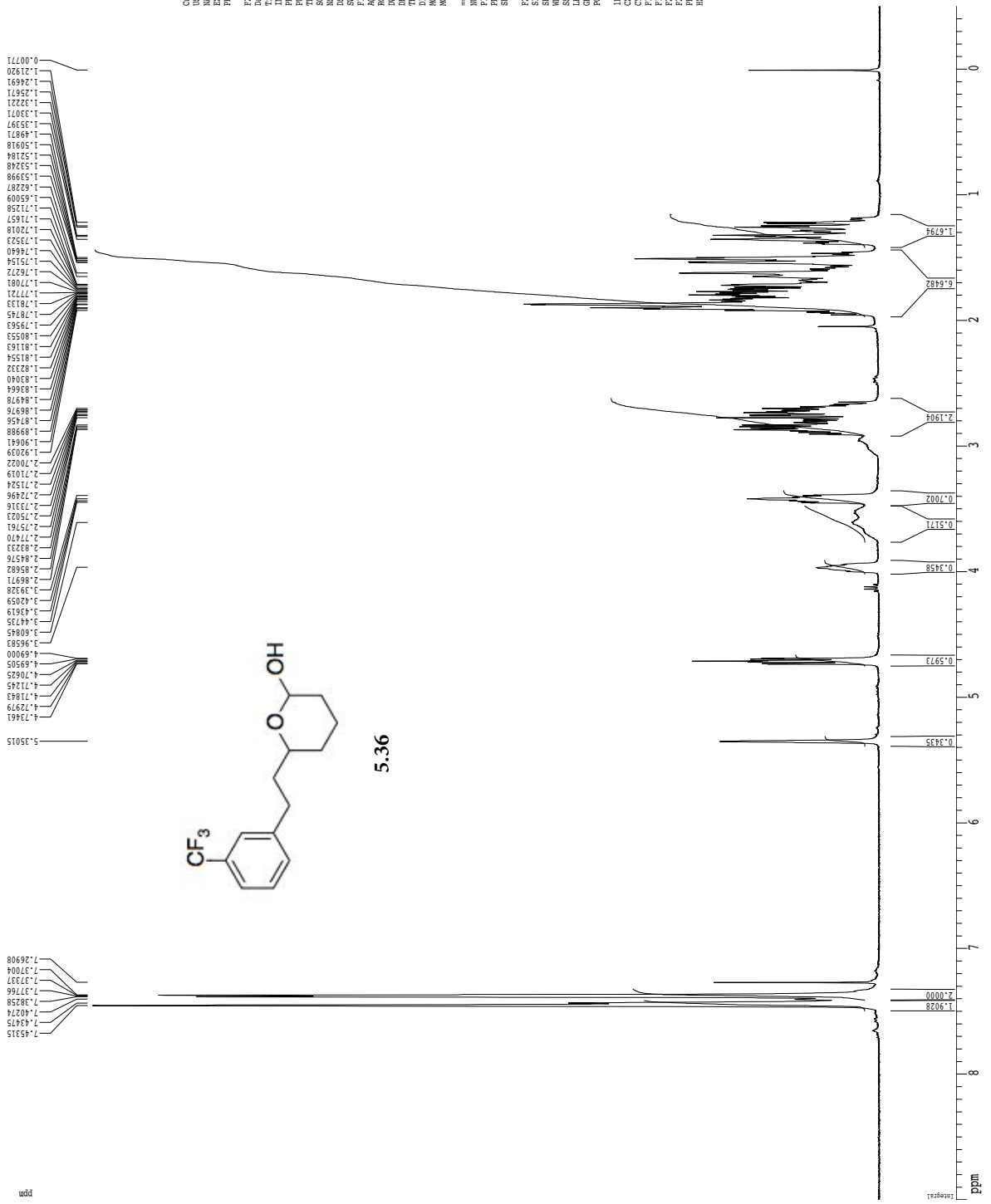
62.56  
56.29



Current Data Parameters  
Date\_ 20210703  
Time\_ 12.04  
INSTRUM av600  
PROBHD 5 mm QNP 1H/13  
PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
DS 4  
SS 16  
SF 178571.422 Hz  
FIDRES 1.362392 Hz  
AQ 0.3670516 sec  
RG 327.68  
DE 2.800 usec  
TE 298.0 K  
D1 3.0000000 sec  
D2 1.0000000 sec  
===== CHANNEL f1 =====  
SFO1 561.629196 MHz  
NUC1 13C  
P1 18.75 usec  
===== CHANNEL f2 =====  
SFO2 125.761154 MHz  
NUC2 1H  
P2 13.00 usec  
===== CHANNEL f3 =====  
SFO3 400.146201 MHz  
NUC3 1H  
P3 13.00 usec  
===== CHANNEL f4 =====  
SFO4 101.626125 MHz  
NUC4 13C  
P4 13.00 usec  
===== CHANNEL f5 =====  
SFO5 400.146201 MHz  
NUC5 1H  
P5 13.00 usec  
===== CHANNEL f6 =====  
SFO6 101.626125 MHz  
NUC6 13C  
P6 13.00 usec  
===== CHANNEL f7 =====  
SFO7 400.146201 MHz  
NUC7 1H  
P7 13.00 usec  
===== CHANNEL f8 =====  
SFO8 101.626125 MHz  
NUC8 13C  
P8 13.00 usec  
===== CHANNEL f9 =====  
SFO9 400.146201 MHz  
NUC9 1H  
P9 13.00 usec  
===== CHANNEL f10 =====  
SFO10 101.626125 MHz  
NUC10 13C  
P10 13.00 usec  
===== CHANNEL f11 =====  
SFO11 400.146201 MHz  
NUC11 1H  
P11 13.00 usec  
===== CHANNEL f12 =====  
SFO12 101.626125 MHz  
NUC12 13C  
P12 13.00 usec  
===== CHANNEL f13 =====  
SFO13 400.146201 MHz  
NUC13 1H  
P13 13.00 usec  
===== CHANNEL f14 =====  
SFO14 101.626125 MHz  
NUC14 13C  
P14 13.00 usec  
===== CHANNEL f15 =====  
SFO15 400.146201 MHz  
NUC15 1H  
P15 13.00 usec  
===== CHANNEL f16 =====  
SFO16 101.626125 MHz  
NUC16 13C  
P16 13.00 usec  
===== CHANNEL f17 =====  
SFO17 400.146201 MHz  
NUC17 1H  
P17 13.00 usec  
===== CHANNEL f18 =====  
SFO18 101.626125 MHz  
NUC18 13C  
P18 13.00 usec  
===== CHANNEL f19 =====  
SFO19 400.146201 MHz  
NUC19 1H  
P19 13.00 usec  
===== CHANNEL f20 =====  
SFO20 101.626125 MHz  
NUC20 13C  
P20 13.00 usec  
===== CHANNEL f21 =====  
SFO21 400.146201 MHz  
NUC21 1H  
P21 13.00 usec  
===== CHANNEL f22 =====  
SFO22 101.626125 MHz  
NUC22 13C  
P22 13.00 usec  
===== CHANNEL f23 =====  
SFO23 400.146201 MHz  
NUC23 1H  
P23 13.00 usec  
===== CHANNEL f24 =====  
SFO24 101.626125 MHz  
NUC24 13C  
P24 13.00 usec  
===== CHANNEL f25 =====  
SFO25 400.146201 MHz  
NUC25 1H  
P25 13.00 usec  
===== CHANNEL f26 =====  
SFO26 101.626125 MHz  
NUC26 13C  
P26 13.00 usec  
===== CHANNEL f27 =====  
SFO27 400.146201 MHz  
NUC27 1H  
P27 13.00 usec  
===== CHANNEL f28 =====  
SFO28 101.626125 MHz  
NUC28 13C  
P28 13.00 usec  
===== CHANNEL f29 =====  
SFO29 400.146201 MHz  
NUC29 1H  
P29 13.00 usec  
===== CHANNEL f30 =====  
SFO30 101.626125 MHz  
NUC30 13C  
P30 13.00 usec  
===== CHANNEL f31 =====  
SFO31 400.146201 MHz  
NUC31 1H  
P31 13.00 usec  
===== CHANNEL f32 =====  
SFO32 101.626125 MHz  
NUC32 13C  
P32 13.00 usec  
===== CHANNEL f33 =====  
SFO33 400.146201 MHz  
NUC33 1H  
P33 13.00 usec  
===== CHANNEL f34 =====  
SFO34 101.626125 MHz  
NUC34 13C  
P34 13.00 usec  
===== CHANNEL f35 =====  
SFO35 400.146201 MHz  
NUC35 1H  
P35 13.00 usec  
===== CHANNEL f36 =====  
SFO36 101.626125 MHz  
NUC36 13C  
P36 13.00 usec  
===== CHANNEL f37 =====  
SFO37 400.146201 MHz  
NUC37 1H  
P37 13.00 usec  
===== CHANNEL f38 =====  
SFO38 101.626125 MHz  
NUC38 13C  
P38 13.00 usec  
===== CHANNEL f39 =====  
SFO39 400.146201 MHz  
NUC39 1H  
P39 13.00 usec  
===== CHANNEL f40 =====  
SFO40 101.626125 MHz  
NUC40 13C  
P40 13.00 usec  
===== CHANNEL f41 =====  
SFO41 400.146201 MHz  
NUC41 1H  
P41 13.00 usec  
===== CHANNEL f42 =====  
SFO42 101.626125 MHz  
NUC42 13C  
P42 13.00 usec  
===== CHANNEL f43 =====  
SFO43 400.146201 MHz  
NUC43 1H  
P43 13.00 usec  
===== CHANNEL f44 =====  
SFO44 101.626125 MHz  
NUC44 13C  
P44 13.00 usec  
===== CHANNEL f45 =====  
SFO45 400.146201 MHz  
NUC45 1H  
P45 13.00 usec  
===== CHANNEL f46 =====  
SFO46 101.626125 MHz  
NUC46 13C  
P46 13.00 usec  
===== CHANNEL f47 =====  
SFO47 400.146201 MHz  
NUC47 1H  
P47 13.00 usec  
===== CHANNEL f48 =====  
SFO48 101.626125 MHz  
NUC48 13C  
P48 13.00 usec  
===== CHANNEL f49 =====  
SFO49 400.146201 MHz  
NUC49 1H  
P49 13.00 usec  
===== CHANNEL f50 =====  
SFO50 101.626125 MHz  
NUC50 13C  
P50 13.00 usec  
===== CHANNEL f51 =====  
SFO51 400.146201 MHz  
NUC51 1H  
P51 13.00 usec  
===== CHANNEL f52 =====  
SFO52 101.626125 MHz  
NUC52 13C  
P52 13.00 usec  
===== CHANNEL f53 =====  
SFO53 400.146201 MHz  
NUC53 1H  
P53 13.00 usec  
===== CHANNEL f54 =====  
SFO54 101.626125 MHz  
NUC54 13C  
P54 13.00 usec  
===== CHANNEL f55 =====  
SFO55 400.146201 MHz  
NUC55 1H  
P55 13.00 usec  
===== CHANNEL f56 =====  
SFO56 101.626125 MHz  
NUC56 13C  
P56 13.00 usec  
===== CHANNEL f57 =====  
SFO57 400.146201 MHz  
NUC57 1H  
P57 13.00 usec  
===== CHANNEL f58 =====  
SFO58 101.626125 MHz  
NUC58 13C  
P58 13.00 usec  
===== CHANNEL f59 =====  
SFO59 400.146201 MHz  
NUC59 1H  
P59 13.00 usec  
===== CHANNEL f60 =====  
SFO60 101.626125 MHz  
NUC60 13C  
P60 13.00 usec  
===== CHANNEL f61 =====  
SFO61 400.146201 MHz  
NUC61 1H  
P61 13.00 usec  
===== CHANNEL f62 =====  
SFO62 101.626125 MHz  
NUC62 13C  
P62 13.00 usec  
===== CHANNEL f63 =====  
SFO63 400.146201 MHz  
NUC63 1H  
P63 13.00 usec  
===== CHANNEL f64 =====  
SFO64 101.626125 MHz  
NUC64 13C  
P64 13.00 usec  
===== CHANNEL f65 =====  
SFO65 400.146201 MHz  
NUC65 1H  
P65 13.00 usec  
===== CHANNEL f66 =====  
SFO66 101.626125 MHz  
NUC66 13C  
P66 13.00 usec  
===== CHANNEL f67 =====  
SFO67 400.146201 MHz  
NUC67 1H  
P67 13.00 usec  
===== CHANNEL f68 =====  
SFO68 101.626125 MHz  
NUC68 13C  
P68 13.00 usec  
===== CHANNEL f69 =====  
SFO69 400.146201 MHz  
NUC69 1H  
P69 13.00 usec  
===== CHANNEL f70 =====  
SFO70 101.626125 MHz  
NUC70 13C  
P70 13.00 usec  
===== CHANNEL f71 =====  
SFO71 400.146201 MHz  
NUC71 1H  
P71 13.00 usec  
===== CHANNEL f72 =====  
SFO72 101.626125 MHz  
NUC72 13C  
P72 13.00 usec  
===== CHANNEL f73 =====  
SFO73 400.146201 MHz  
NUC73 1H  
P73 13.00 usec  
===== CHANNEL f74 =====  
SFO74 101.626125 MHz  
NUC74 13C  
P74 13.00 usec  
===== CHANNEL f75 =====  
SFO75 400.146201 MHz  
NUC75 1H  
P75 13.00 usec  
===== CHANNEL f76 =====  
SFO76 101.626125 MHz  
NUC76 13C  
P76 13.00 usec  
===== CHANNEL f77 =====  
SFO77 400.146201 MHz  
NUC77 1H  
P77 13.00 usec  
===== CHANNEL f78 =====  
SFO78 101.626125 MHz  
NUC78 13C  
P78 13.00 usec  
===== CHANNEL f79 =====  
SFO79 400.146201 MHz  
NUC79 1H  
P79 13.00 usec  
===== CHANNEL f80 =====  
SFO80 101.626125 MHz  
NUC80 13C  
P80 13.00 usec  
===== CHANNEL f81 =====  
SFO81 400.146201 MHz  
NUC81 1H  
P81 13.00 usec  
===== CHANNEL f82 =====  
SFO82 101.626125 MHz  
NUC82 13C  
P82 13.00 usec  
===== CHANNEL f83 =====  
SFO83 400.146201 MHz  
NUC83 1H  
P83 13.00 usec  
===== CHANNEL f84 =====  
SFO84 101.626125 MHz  
NUC84 13C  
P84 13.00 usec  
===== CHANNEL f85 =====  
SFO85 400.146201 MHz  
NUC85 1H  
P85 13.00 usec  
===== CHANNEL f86 =====  
SFO86 101.626125 MHz  
NUC86 13C  
P86 13.00 usec  
===== CHANNEL f87 =====  
SFO87 400.146201 MHz  
NUC87 1H  
P87 13.00 usec  
===== CHANNEL f88 =====  
SFO88 101.626125 MHz  
NUC88 13C  
P88 13.00 usec  
===== CHANNEL f89 =====  
SFO89 400.146201 MHz  
NUC89 1H  
P89 13.00 usec  
===== CHANNEL f90 =====  
SFO90 101.626125 MHz  
NUC90 13C  
P90 13.00 usec  
===== CHANNEL f91 =====  
SFO91 400.146201 MHz  
NUC91 1H  
P91 13.00 usec  
===== CHANNEL f92 =====  
SFO92 101.626125 MHz  
NUC92 13C  
P92 13.00 usec  
===== CHANNEL f93 =====  
SFO93 400.146201 MHz  
NUC93 1H  
P93 13.00 usec  
===== CHANNEL f94 =====  
SFO94 101.626125 MHz  
NUC94 13C  
P94 13.00 usec  
===== CHANNEL f95 =====  
SFO95 400.146201 MHz  
NUC95 1H  
P95 13.00 usec  
===== CHANNEL f96 =====  
SFO96 101.626125 MHz  
NUC96 13C  
P96 13.00 usec  
===== CHANNEL f97 =====  
SFO97 400.146201 MHz  
NUC97 1H  
P97 13.00 usec  
===== CHANNEL f98 =====  
SFO98 101.626125 MHz  
NUC98 13C  
P98 13.00 usec  
===== CHANNEL f99 =====  
SFO99 400.146201 MHz  
NUC99 1H  
P99 13.00 usec  
===== CHANNEL f100 =====  
SFO100 101.626125 MHz  
NUC100 13C  
P100 13.00 usec



1H spectrum



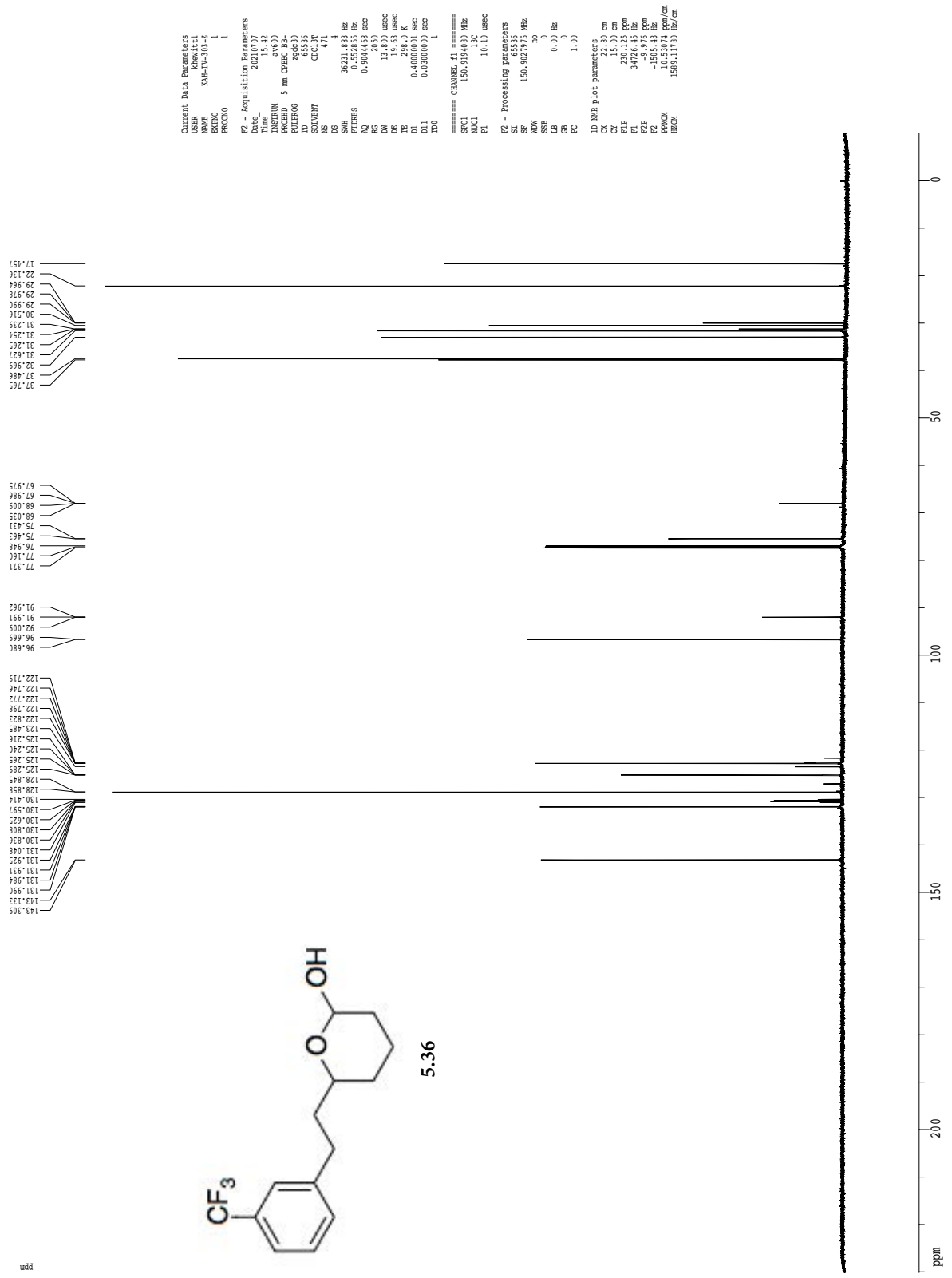
Current Data Parameters  
 USER hewlett1  
 NAME RAH-VU-312-CHK  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 2/21/07  
 Time 12:00:00  
 INSTRUM dxt400  
 PROBED 5 mm QNP H/F/P  
 PULPROG zgpg30  
 CQZ 3  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6416.97 Hz  
 FIDRES 0.057653 Hz  
 AQ 5.1118579 sec  
 RG 101.6  
 DW 78.000 usec  
 DE 298.0 K  
 TE 298.0 K  
 DL 0.1000000 sec  
 ACQRES 0.0000000 sec  
 PCNTX 0.0100000 sec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -1.00 dB  
 SFO1 400.1324809 MHz

F2 - Processing parameters  
 SI 32768  
 SF 400.1301175 MHz  
 WDW no  
 SSB 0  
 GB 0.0 Hz  
 PC 2.00

ID MR plot parameters  
 CT 15.00 cm  
 CY 15.00 cm  
 FIP 9.000 ppm  
 FI 3601.17 Hz  
 F2 -700.00 Hz  
 F2 0.41647 ppm/cm  
 HZCM 166.72084 Hz/cm

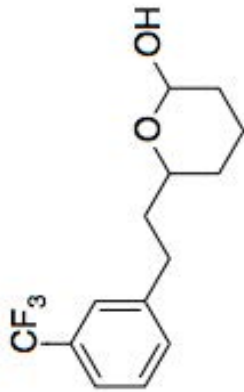
C13.c



f19.c

8

62.560  
62.572



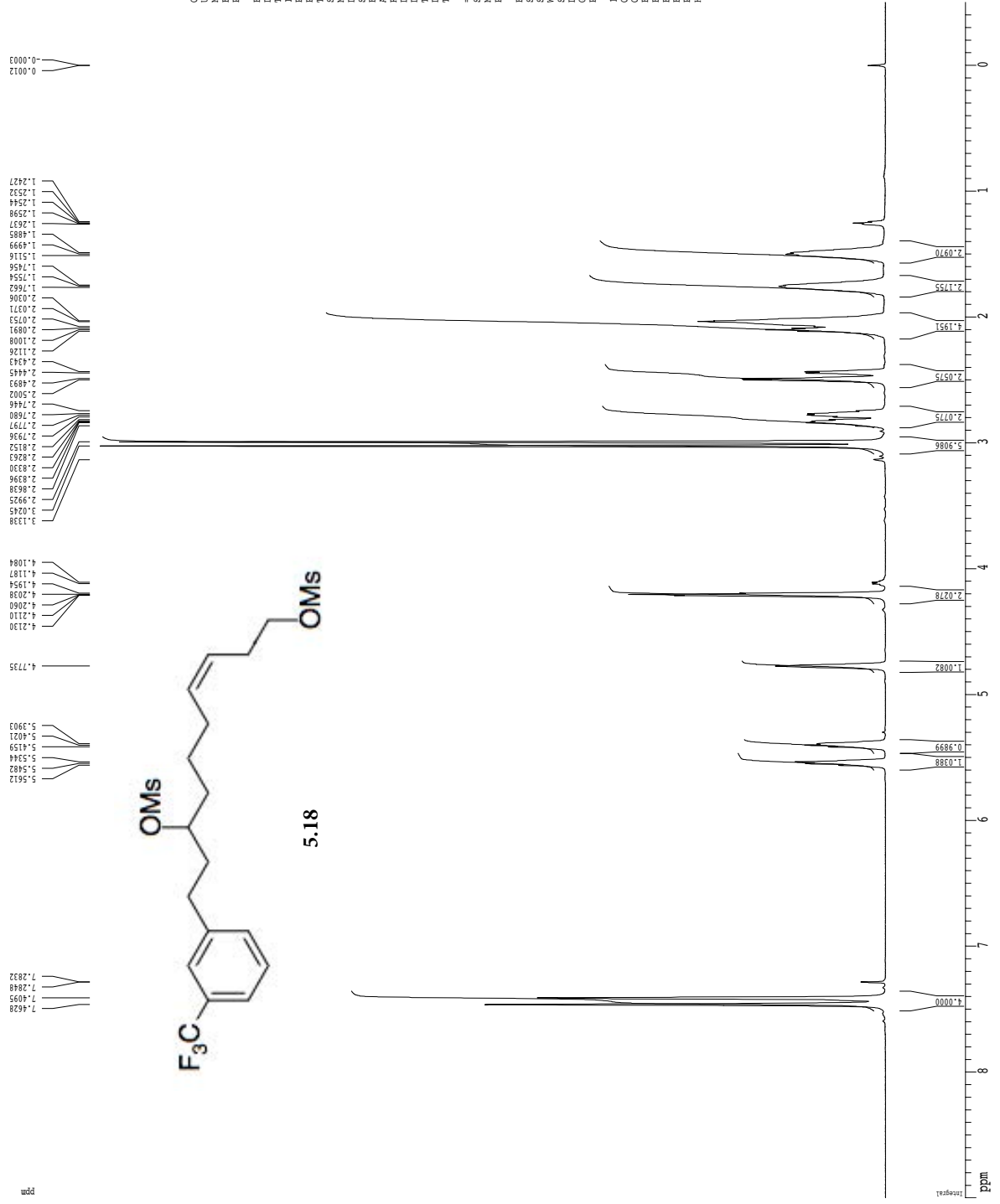
5.36

Current Data Parameters  
Date\_ 20210707  
Time\_ 15:59  
INSTRUM av600  
PROBHD 5 mm QNP 1H/13  
PULPROG zgpg30  
TD 331072  
SOLVENT CDCl3  
DS 5  
SS 5  
SFO 178571.422 Hz  
FIDRES 1.362392 Hz  
AQ 0.3670516 sec  
RG 327.500  
DE 2.800 usec  
TE 298.0 K  
D1 3.0000000 sec  
D2 1.0000000 sec  
===== CHANNEL f1 =====  
SFO1 564.629196 MHz  
NUC1 13C  
P1 18.75 usec  
F2 - Processing Parameters  
SI 331072  
SF 564.629196 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00  
ID MR Plot parameters  
CX 22.80 cm  
CT 1.00 cm  
F1 58.061 ppm  
F2 327866.34 Hz  
F3 -238.170 ppm  
F4 -145.8259 Hz  
F5 7832.06057 Hz/cm  
F6 7832.06057 Hz/cm

ppm -250 -200 -150 -100 -50 0



h1.c



Current Data Parameters  
Date\_ 20210713  
Time\_ 14.04  
INSTRUM av600  
PROBHD 5 mm CPBPR  
PULPROG zgpg30  
TD 66074  
SOLVENT CDCl3  
DS 8  
SS 2  
SFO 9615.385 Hz  
FIDRES 0.098842 Hz  
AQ 5.0998979 sec  
RG 652  
DE 14.23 usec  
TE 298.0 K  
D1 0.1000000 sec  
TD0 1

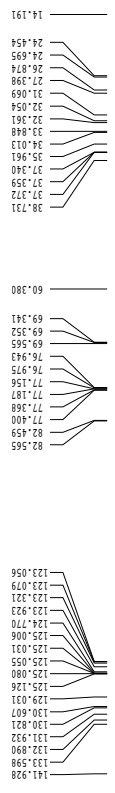
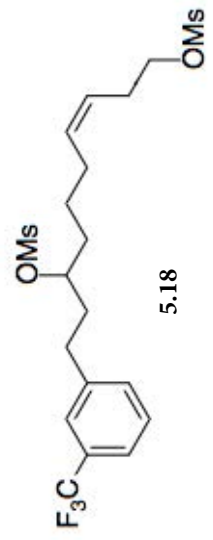
==== CHANNEL f1 =====  
SFO1 600.134009 MHz  
NUC1 13C  
P1 9.50 usec  
PL 0

F2 - Processing parameters  
SI 6536  
SF 600.134009 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

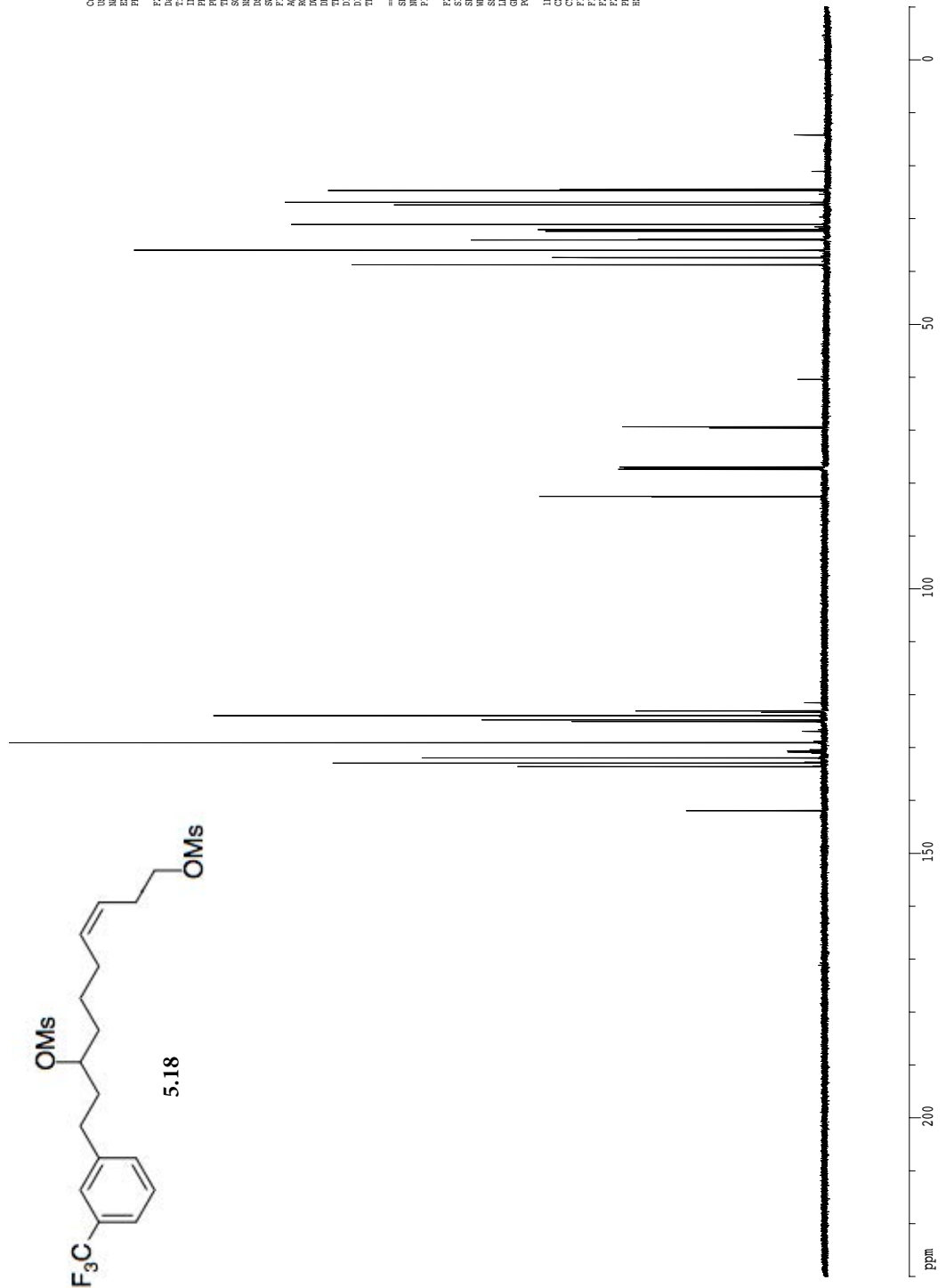
ID NMR plot parameters  
CX 22.80 cm  
FL 5.00 mm  
FID 9.000 ppm  
F1 5403.17 Hz  
F2 -0.500 ppm  
F3 0.000 Hz  
F4 0.000 Hz  
F5 0.000 Hz  
F6 0.000 Hz  
F7 0.000 Hz  
F8 0.000 Hz  
F9 0.000 Hz  
F10 0.000 Hz  
F11 0.000 Hz  
F12 0.000 Hz  
F13 0.000 Hz  
F14 0.000 Hz  
F15 0.000 Hz  
F16 0.000 Hz  
F17 0.000 Hz  
F18 0.000 Hz  
F19 0.000 Hz  
F20 0.000 Hz  
F21 0.000 Hz  
F22 0.000 Hz  
F23 0.000 Hz  
F24 0.000 Hz  
F25 0.000 Hz  
F26 0.000 Hz  
F27 0.000 Hz  
F28 0.000 Hz  
F29 0.000 Hz  
F30 0.000 Hz  
F31 0.000 Hz  
F32 0.000 Hz  
F33 0.000 Hz  
F34 0.000 Hz  
F35 0.000 Hz  
F36 0.000 Hz  
F37 0.000 Hz  
F38 0.000 Hz  
F39 0.000 Hz  
F40 0.000 Hz  
F41 0.000 Hz  
F42 0.000 Hz  
F43 0.000 Hz  
F44 0.000 Hz  
F45 0.000 Hz  
F46 0.000 Hz  
F47 0.000 Hz  
F48 0.000 Hz  
F49 0.000 Hz  
F50 0.000 Hz  
F51 0.000 Hz  
F52 0.000 Hz  
F53 0.000 Hz  
F54 0.000 Hz  
F55 0.000 Hz  
F56 0.000 Hz  
F57 0.000 Hz  
F58 0.000 Hz  
F59 0.000 Hz  
F60 0.000 Hz  
F61 0.000 Hz  
F62 0.000 Hz  
F63 0.000 Hz  
F64 0.000 Hz  
F65 0.000 Hz  
F66 0.000 Hz  
F67 0.000 Hz  
F68 0.000 Hz  
F69 0.000 Hz  
F70 0.000 Hz  
F71 0.000 Hz  
F72 0.000 Hz  
F73 0.000 Hz  
F74 0.000 Hz  
F75 0.000 Hz  
F76 0.000 Hz  
F77 0.000 Hz  
F78 0.000 Hz  
F79 0.000 Hz  
F80 0.000 Hz  
F81 0.000 Hz  
F82 0.000 Hz  
F83 0.000 Hz  
F84 0.000 Hz  
F85 0.000 Hz  
F86 0.000 Hz  
F87 0.000 Hz  
F88 0.000 Hz  
F89 0.000 Hz  
F90 0.000 Hz  
F91 0.000 Hz  
F92 0.000 Hz  
F93 0.000 Hz  
F94 0.000 Hz  
F95 0.000 Hz  
F96 0.000 Hz  
F97 0.000 Hz  
F98 0.000 Hz  
F99 0.000 Hz  
F100 0.000 Hz

c13.c

ppm

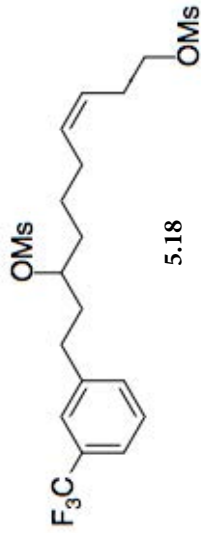


```
Current Data Parameters
NAME          KRM-N-035-2
EXPNO        1
PROCNO       1
F2 - Acquisition Parameters
Date_         20210713
Time         13.57
INSTRUM      av600
PROBHD       5 mm CPBPR0
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3T
NS           128
DS           4
SWH          36231.883 Hz
AQ           0.553855 Hz
RG           0.5904468 sec
RG2          0.5904468 sec
DE           13.800 usec
TE           298.2 K
D1           0.40000001 sec
D11          0.50000001 sec
D12          0.50000001 sec
D13          0.50000001 sec
TD0          1
===== CHANNEL f1 =====
NUC1         13C
P1           10.10 usec
===== CHANNEL f2 =====
NUC2         13C
P2           10.10 usec
F2 - Processing Parameters
SI           156.9026113 MHz
SF           156.9026113 MHz
WDW          no
SSB          0
GB           0.00 Hz
PC           1.00
ID MR ELOC parameters
CX          15.00 cm
CY          15.00 cm
CZ          15.00 cm
F1P         230.033 ppm
F1          34712.62 Hz
F2P         -155.8 ppm
F2          -155.8 ppm
FREQ(CM)    10.53074 ppm/cm
FREQ(HZ)    1989.11768 Hz/cm
```



19F Spectrum

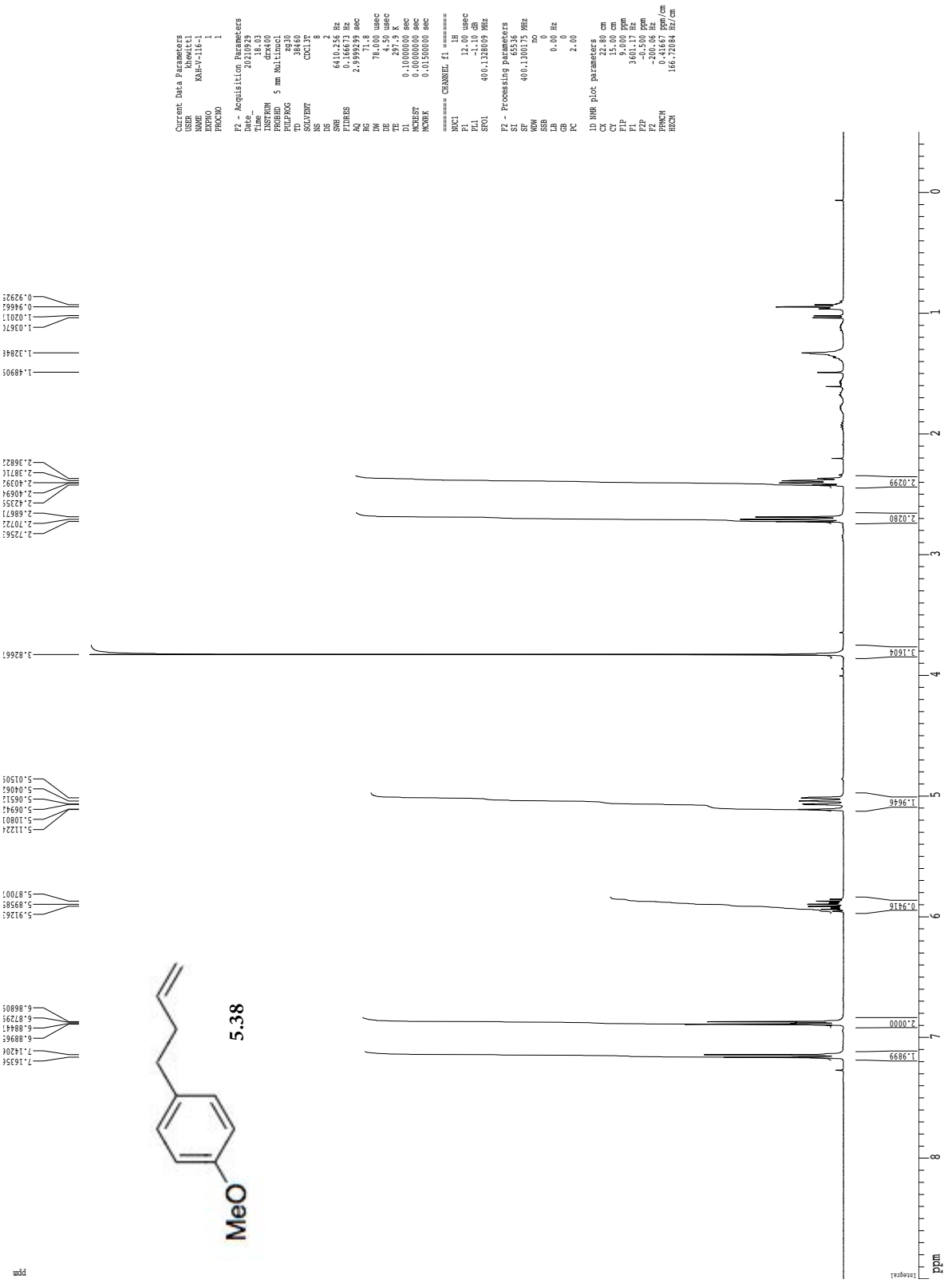
62.532



Current Data Parameters  
NAME KM-14-051-F  
EXPNO 2  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20220117  
Time 17.13  
INSTRUM spect  
PROBHD 5 mm QNP 1H/13  
PULPROG zgpg30  
TD 131072  
SOLVENT CDCl3  
DS 6  
SS 6  
SFO 178571.422 Hz  
FIDRES 1.362392 Hz  
AQ 0.3670516 sec  
RG 327.500  
DE 2.800 usec  
DD 18.400 usec  
TE 298.0 K  
D1 3.0000000 sec  
D2 1  
===== CHANNEL f1 =====  
SFO1 564.6295196 MHz  
NUC1 19F  
PC1 18.75 usec  
F2 - Processing Parameters  
SI 131072  
SF 564.6295196 MHz  
WDW 100  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00  
ID NMR Plot parameters  
CX 22.80 cm  
CY 15.00 cm  
FIP 58.077 ppm  
F1 32795.21 Hz  
F2P -238.154 ppm  
F3P -145.7822 Hz  
FREQN 7832.06057 MHz/cm  
RECN 7832.06057 MHz/cm

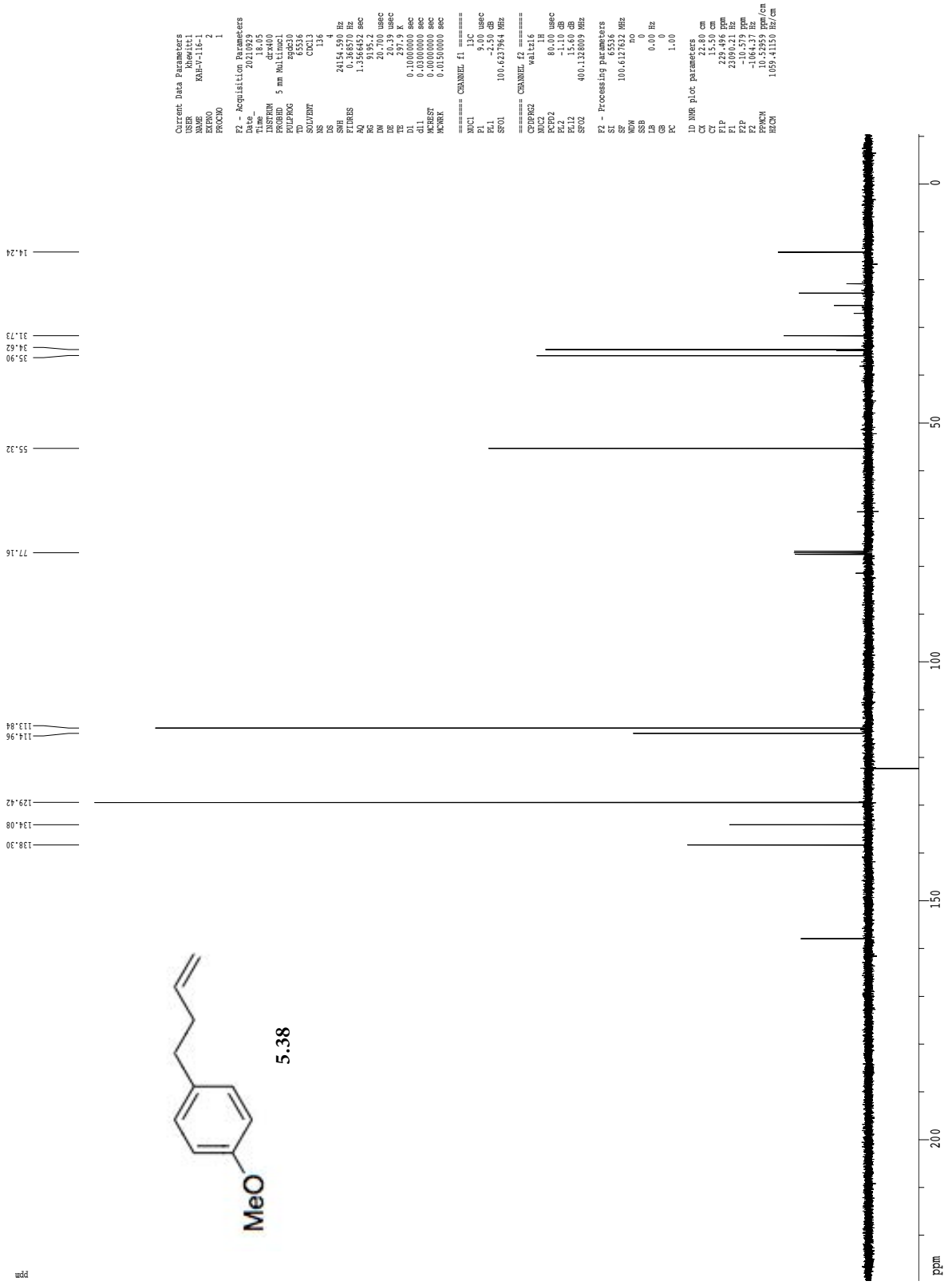
ppm -250 -200 -150 -100 -50 0

1H spectrum

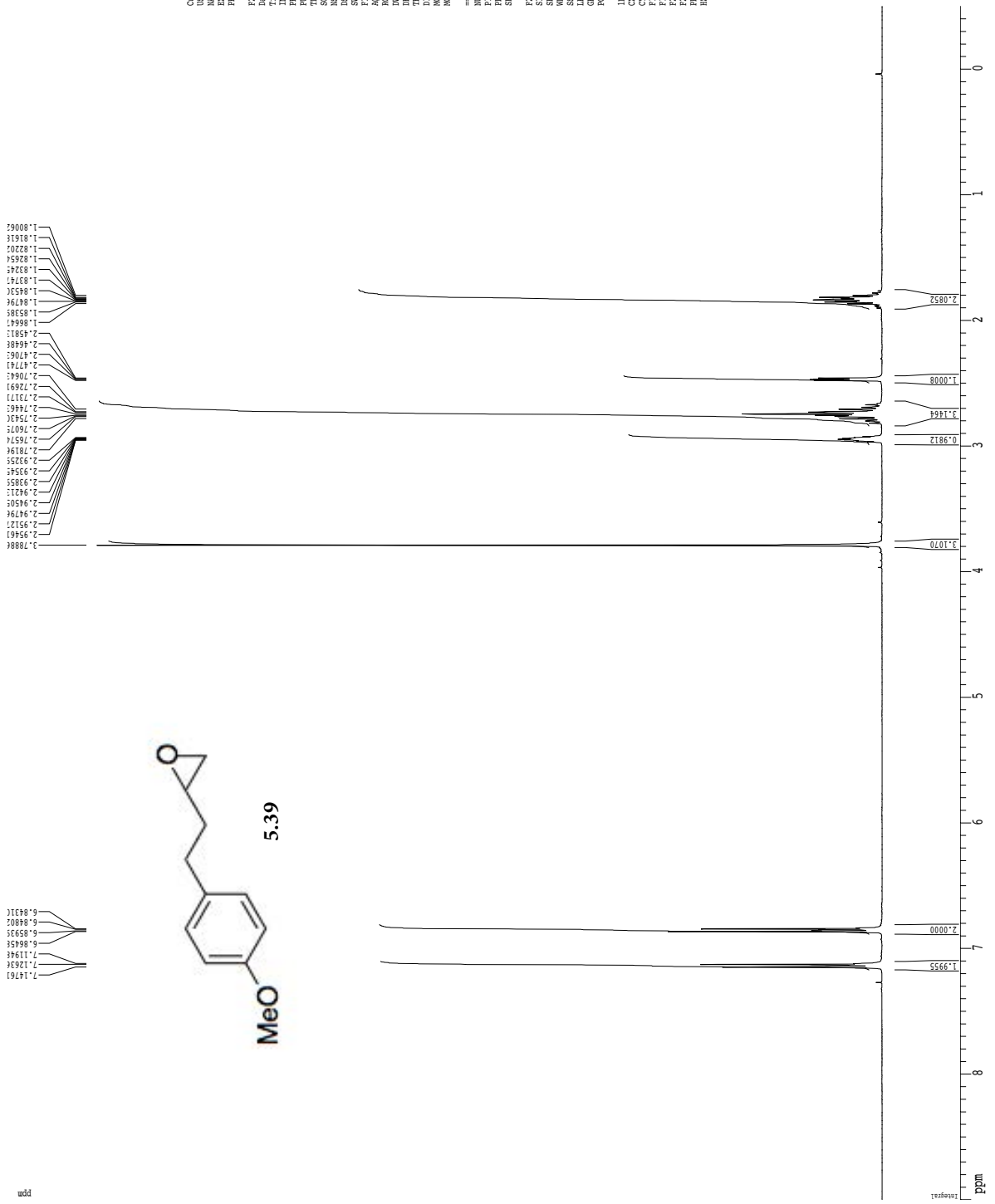


Current Data Parameters  
 USER hewitt1  
 NAME RAH-7-116-1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20210929  
 Time 17:42:00  
 INSTRUM dx400  
 PROBEID 5 mm Multinuc1  
 PULPROG zgpg30  
 PC 3.82  
 SOLVENT CDCl3T  
 NS 8  
 DS 2  
 SWH 6410.20 Hz  
 FIDRES 0.166653 Hz  
 AQ 2.9599259 sec  
 RG 71.8  
 DW 78.000 usec  
 DE 1.200 usec  
 TE 297.9 K  
 DL 0.1000000 sec  
 ACQRES 0.0000000 sec  
 PCW 0.0130000 sec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -1.00 dB  
 SFO1 400.1328009 MHz  
 F2 - Processing parameters  
 SI 65536  
 SF 400.130175 MHz  
 WDW po  
 SSB 0  
 GB 0.0 Hz  
 CB 0.0 Hz  
 PC 2.00  
 ID MRB plot parameters  
 CT 15.00 cm  
 CY 15.00 cm  
 FIP 9.000 ppm  
 FI 3601.17 Hz  
 F2 -700.06 Hz  
 FPMCH 0.41667 ppm/cm  
 HZCM 166.72084 Hz/cm

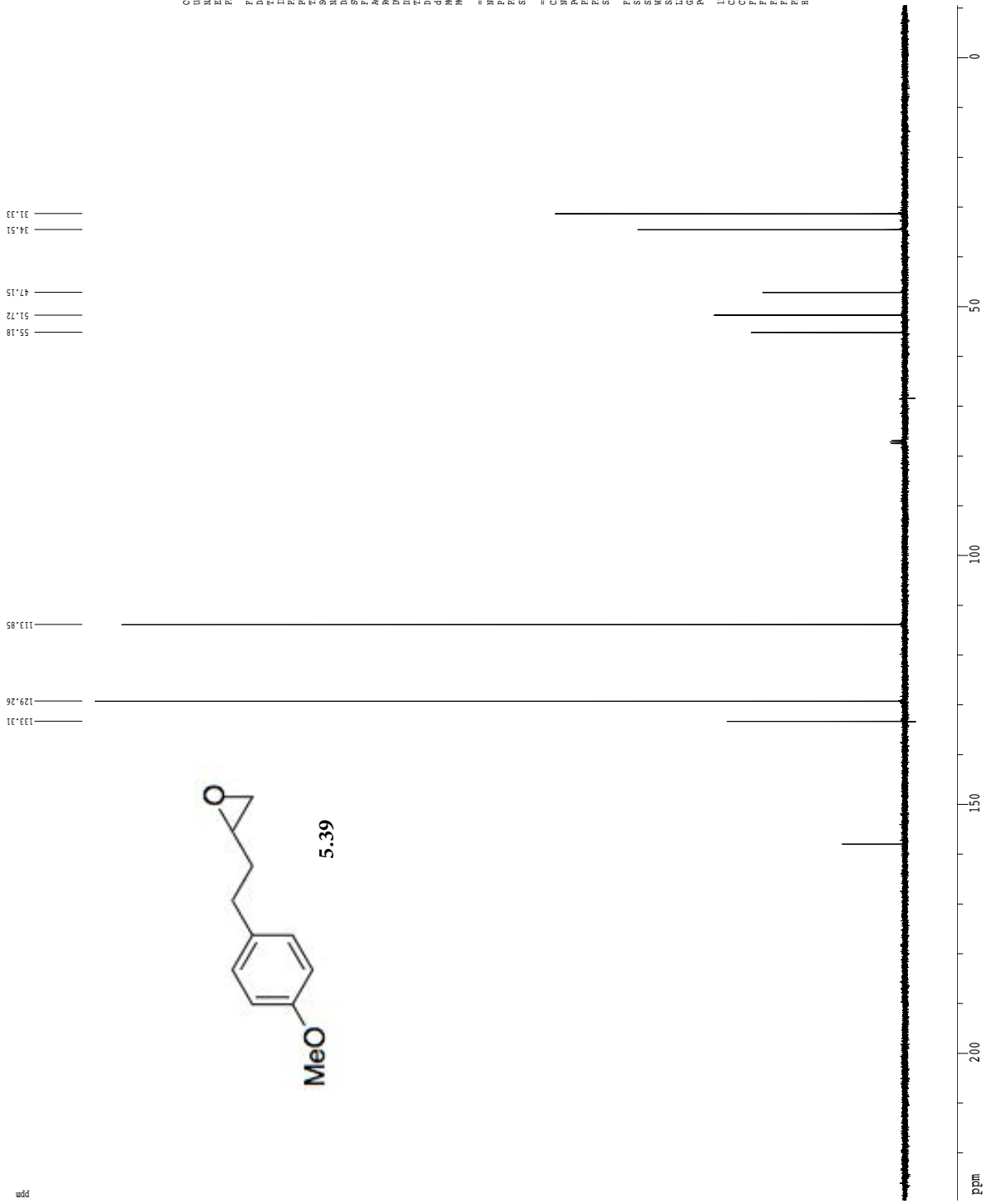
13C spectrum with 1H decoupling



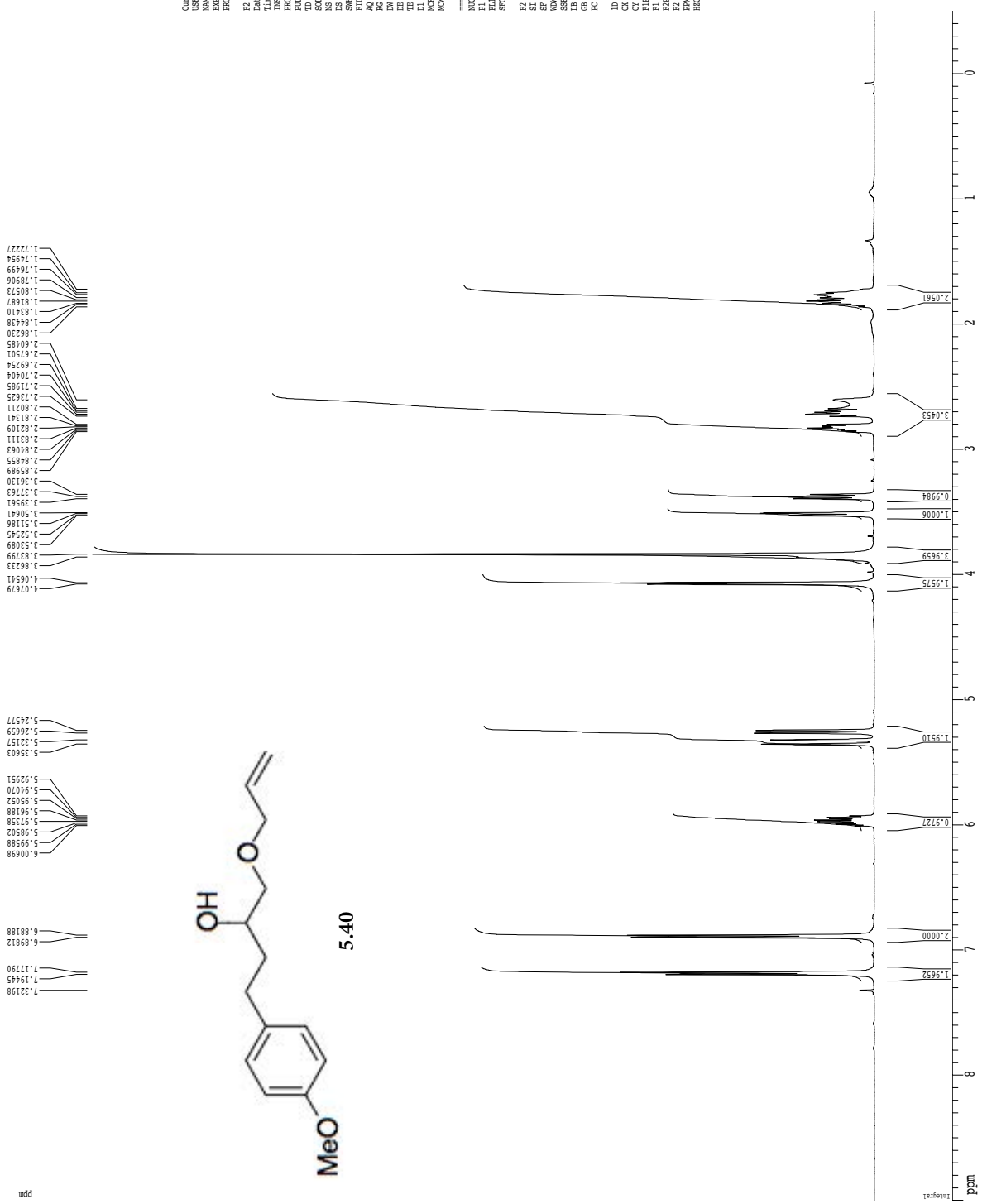
1H spectrum



<sup>13</sup>C spectrum with <sup>1</sup>H decoupling



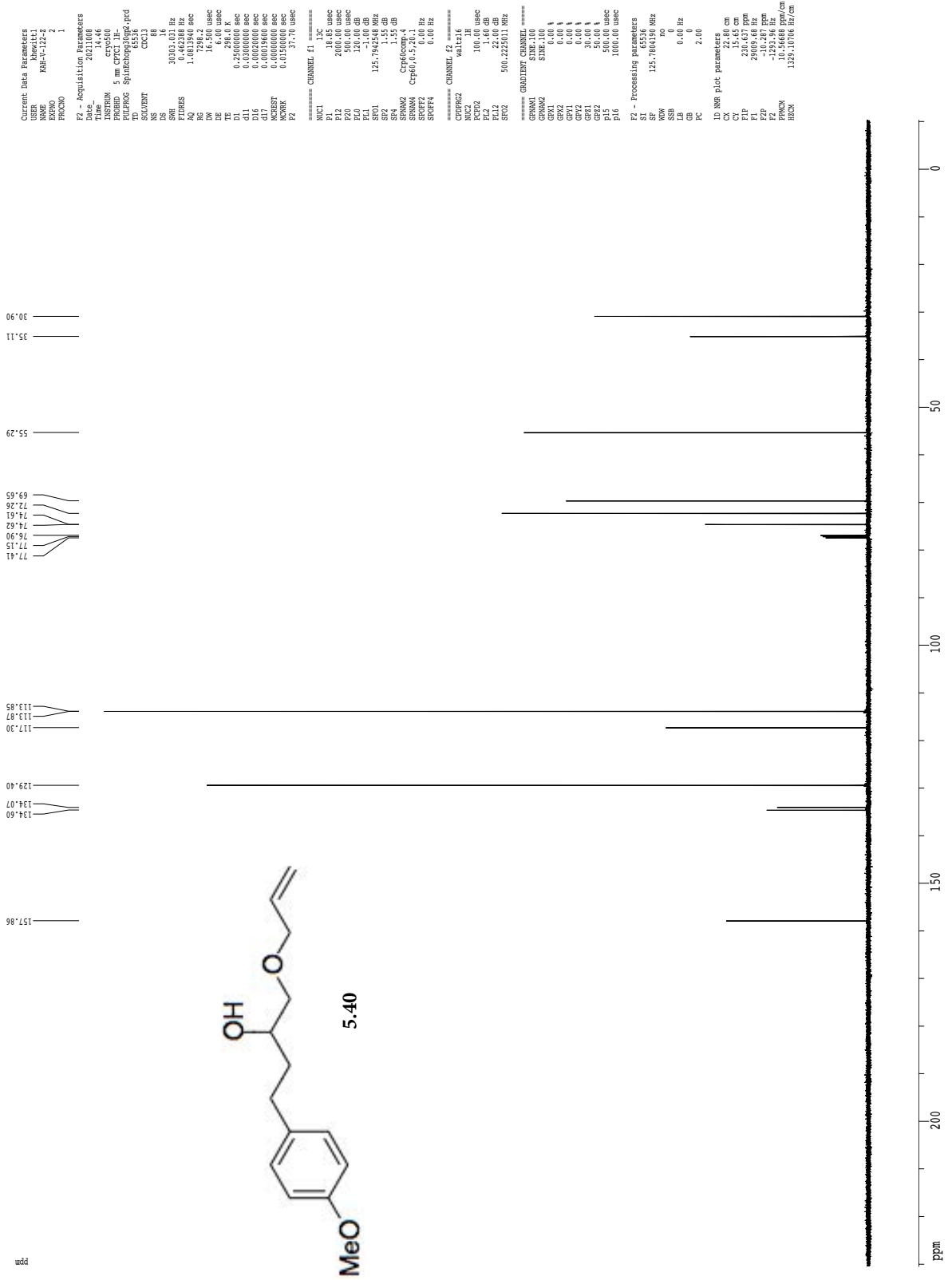
1H spectrum



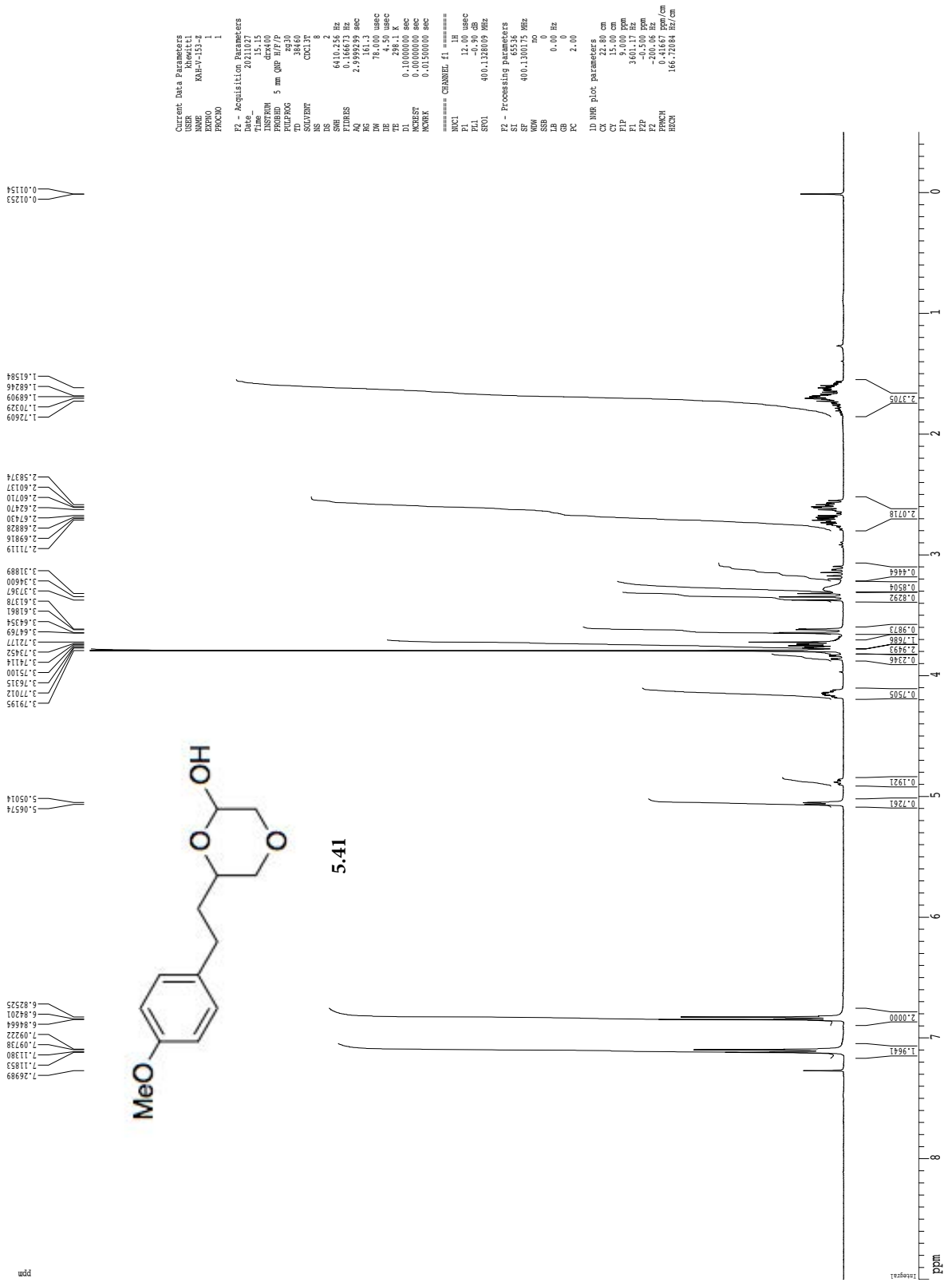
Current Data Parameters  
 USER khowl11  
 NAME KHM-V12.2-2  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 201108  
 Time\_ 11:00  
 INSTRUM crys500  
 PROBRN 5 mm QNP1H-  
 PULPROG zgpg30  
 F2 250  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6012.0 Hz  
 FIDRES 0.068843 Hz  
 AQ 5.0988774 sec  
 RG 41.5  
 DW 62.400 usec  
 DE 1.900 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 MCHYST 0.00000000 sec  
 PCNMR 0.01000000 sec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 0.10 sec  
 PL1 1.40 dB  
 SFO1 500.225015 MHz  
 F2 - Processing parameters  
 SI 32768  
 SF 500.220000 MHz  
 WDW no  
 SSB 0  
 GB 0  
 PC 1.00  
 IDMR plot parameters  
 X 128  
 Y 128  
 CT 15.00 cm  
 FIP 5.000 ppm  
 FI 4501.88 Hz  
 FP 2.50000000 ppm  
 F2 -250.11 Hz  
 FWHM 0.41667 ppm/cm  
 HZCN 208.42500 Hz/cm



Z-restored spin-echo 13c spectrum with 1H decoupling

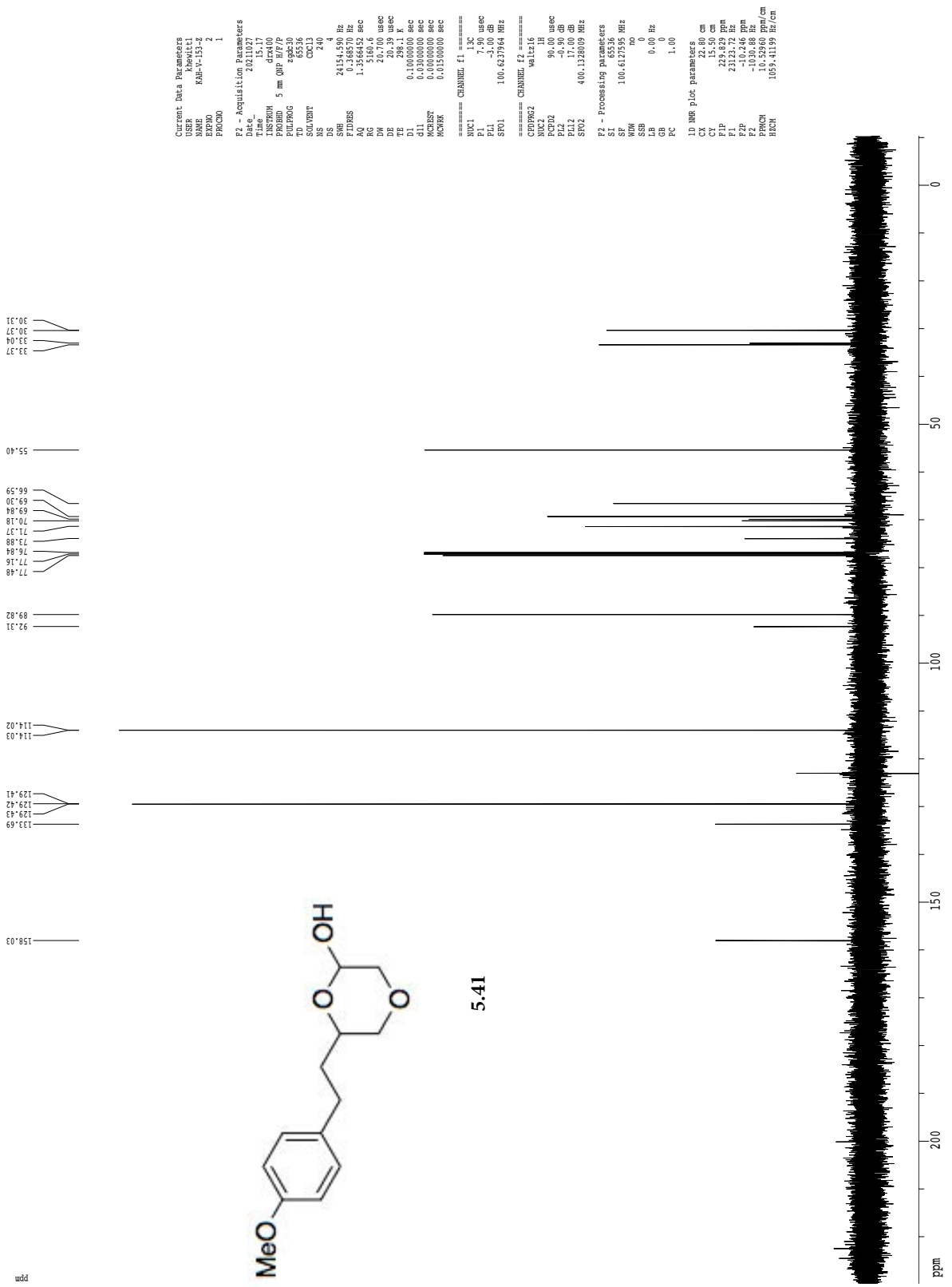


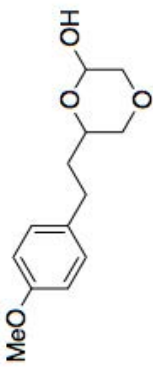
1H spectrum



Current Data Parameters  
 USER hweiltl  
 NAME RAH-7-13-2  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 201111  
 Time\_ 12:11:11  
 INSTRUM dxt400  
 PROBD 5 mm QNP H/F/P  
 PULPROG zgpg  
 SFO1 400.1328009 MHz  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6412.90 Hz  
 FIDRES 0.166653 Hz  
 AQ 2.9999259 sec  
 RG 161.3  
 DW 78.000 usec  
 DE 1.90 usec  
 TE 298.15 K  
 D1 0.1000000 sec  
 ICHRES 0.0000000 sec  
 PCNMR 0.0100000 sec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -0.00 dB  
 SFO1 400.1328009 MHz  
 F2 - Processing parameters  
 SI 65536  
 SF 400.130175 MHz  
 WDW po  
 SSB 0  
 GB 0.0 Hz  
 PC 2.00  
 ID MR plot parameters  
 SI 65536  
 CT 15.00 cm  
 FIP 9.000 ppm  
 FI 3601.17 Hz  
 F2 -700.06 Hz  
 FPCW 0.41667 ppm/cm  
 HZCN 166.72084 Hz/cm

<sup>13</sup>C spectrum with <sup>1</sup>H decoupling





gcosy60

5.41

```

Current Data Parameters
=====
USER          Krewatt1
EXPNO        164-01-2
PROCNO       1
PROCNO       1

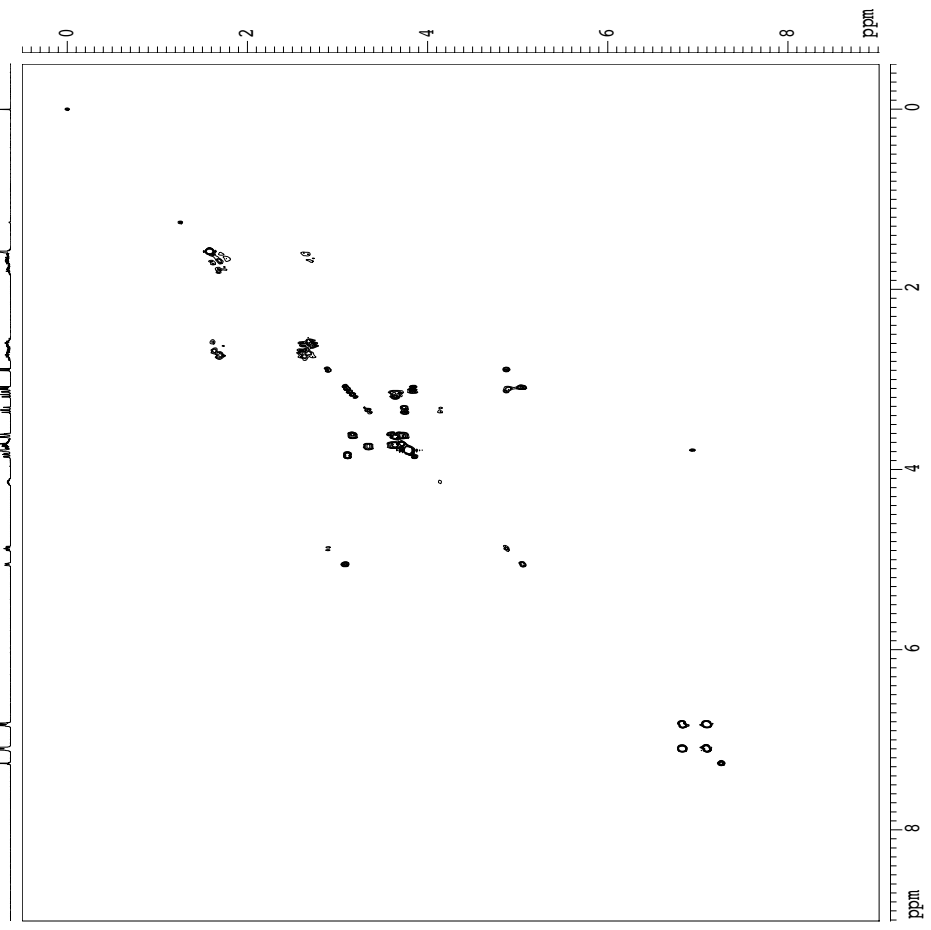
F2 - Acquisition Parameters
=====
Date_         20111111
Time         13:18
INSTRUM      spect
PROBHD       5 mm QNP B/F/P
PULPROG      zgpg30
TD           65536
AQ           2.048
RG           327.5
DS           16
SFO          400.1362609 MHz
SF           400.1362609 MHz
WDW          EM
SSB          0
GB           0
PC           2.00
===== CHANNEL f1 =====
NUC1          1H
P1           12.00 usec
PL1          -0.50 dB
SFO1         400.1362609 MHz
===== GRADIENT CHANNEL =====
GPRM1        SWSQ10-100
GPRM2        SWSQ10-100
GZ1          0.00 %
GZ2          0.00 %
GZ3          0.00 %
GZ4          0.00 %
GZ5          10.00 %
GZ6          10.00 %
F16          1000.00 usec

F1 - Acquisition parameters
=====
TD           1
SI           32768
SF           400.1362609 MHz
WDW          EM
SSB          0
GB           0
PC           2.00

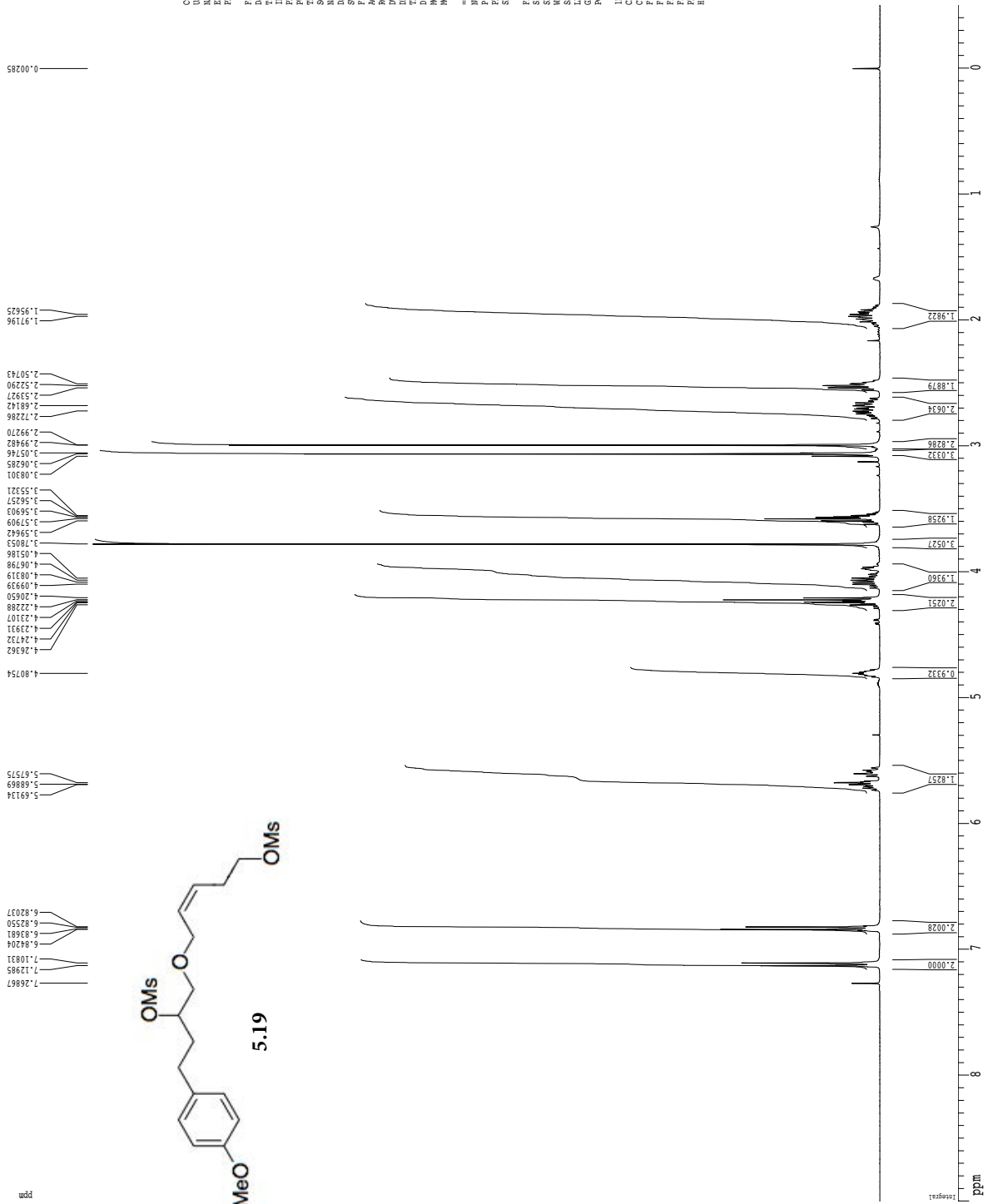
F2 - Processing parameters
=====
SI           32768
SF           400.1362609 MHz
WDW          EM
SSB          0
GB           0
PC           2.00

F1 - Processing parameters
=====
SI           32768
SF           400.1362609 MHz
WDW          EM
SSB          0
GB           0
PC           2.00

ZD NMR plot parameters
=====
CX2          15.00 cm
CX1          15.00 cm
CX0          15.00 cm
FZ0          3665.86 Hz
FZ1          -0.5000 ppm
FZ2          -200.23 Hz
F1F0         9.012 ppm
F1F1         3.9556 Hz
F1F2         -200.23 Hz
F1F3         0.5414 ppm/cm
F2F0PACH    255.7393 Hz/cm
F2F1PACH    0.6583 ppm/cm
F2F2PACH    255.7393 Hz/cm
F1F3PACH    255.7393 Hz/cm
  
```



1H spectrum



Current Data Parameters  
 USER khewitt1  
 NAME KAH-V-171-4  
 RNOV 1  
 PROCNO 1

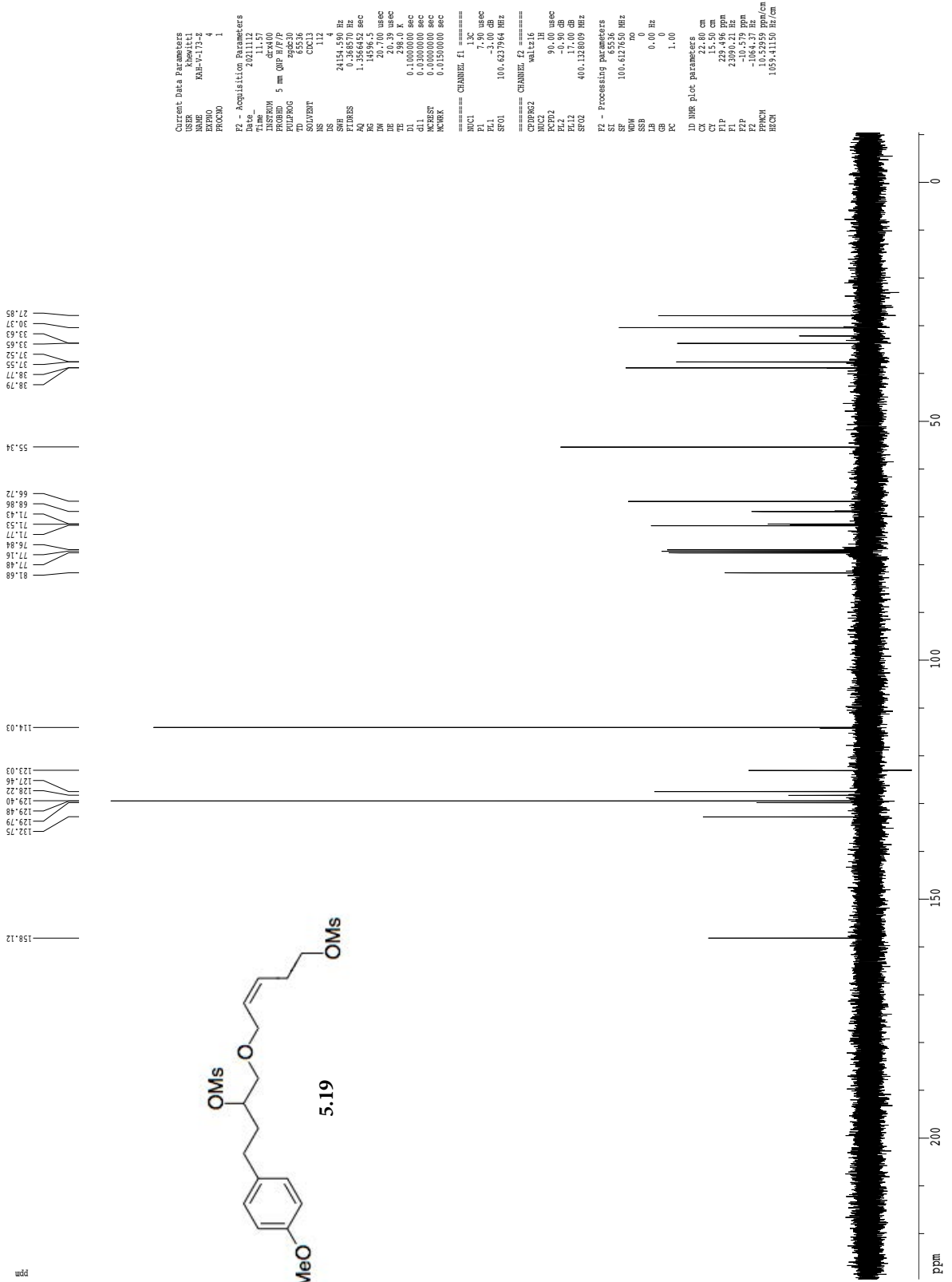
F2 - Acquisition Parameters  
 Date\_ 2/11/12  
 Time\_ 11:14  
 INSTRUM dxs400  
 PROBRD 5 mm QNP H/F/P  
 PULPROG zgpg30  
 SOLVENT CDCl3  
 NS 8  
 DS 4  
 SWH 6410.72 Hz  
 FIDRES 0.164673 Hz  
 AQ 2.5995259 sec  
 RG 90.5  
 DW 78.000 usec  
 DE 1.000 usec  
 TE 298.0 K  
 D1 0.1000000 sec  
 ACQST 0.0000000 sec  
 ACWA 0.0150000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -1.50 dB  
 SFO1 400.132609 MHz

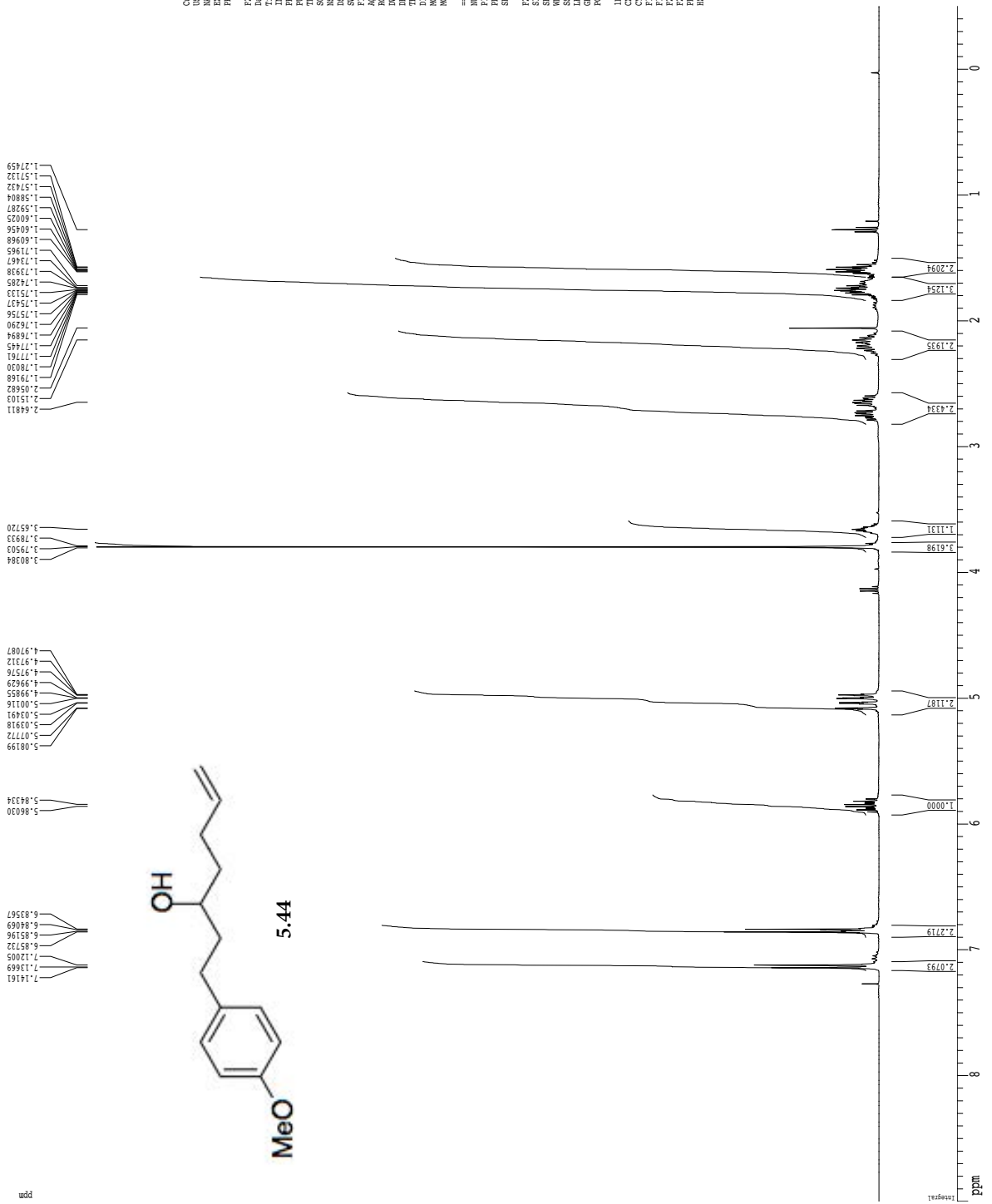
F2 - Processing parameters  
 SI 32768  
 SF 400.130015 MHz  
 WDW no  
 SSB 0  
 GB 0  
 PC 2.00

D0 MR FID parameters  
 CT 0  
 CD 0  
 CF 15.00 cm  
 FIP 9.000 ppm  
 FL 360.17 Hz  
 FZ 0.0000000 ppm  
 EQ -20.000 Hz  
 FPGM 0.41667 ppm/cm  
 HCN 166.72084 Hz/cm

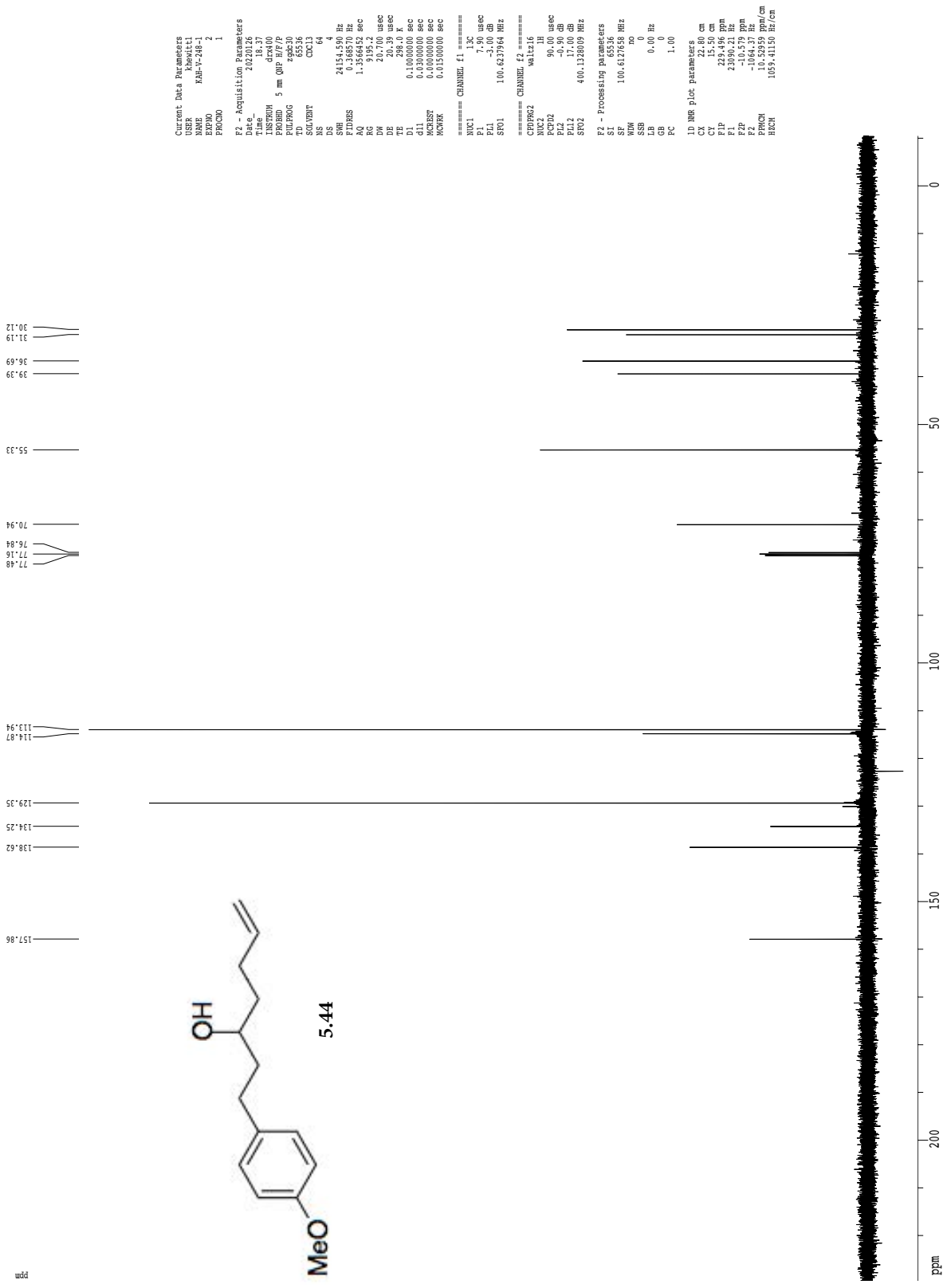
13C spectrum with 1H decoupling



1H spectrum

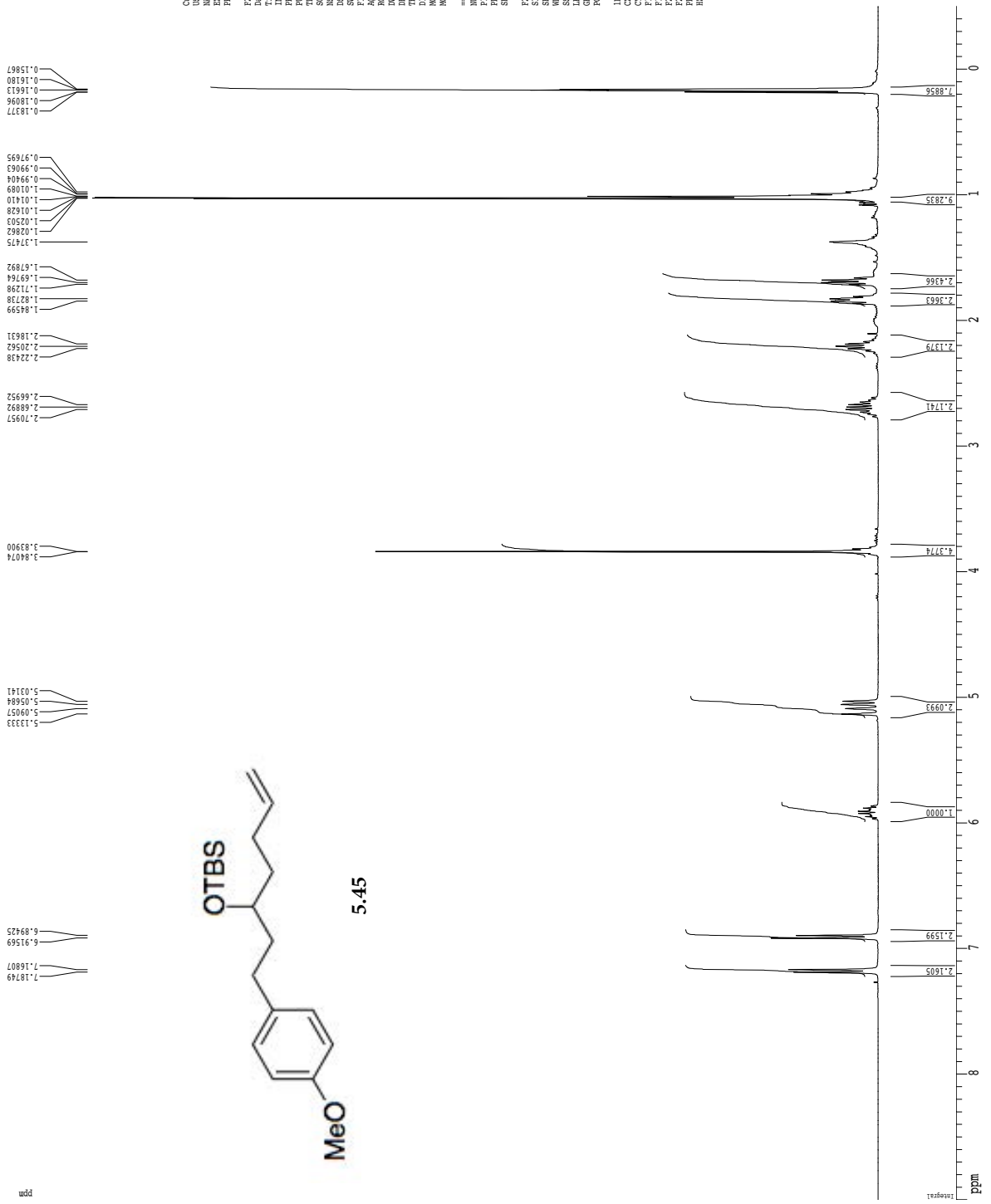


13C spectrum with 1H decoupling





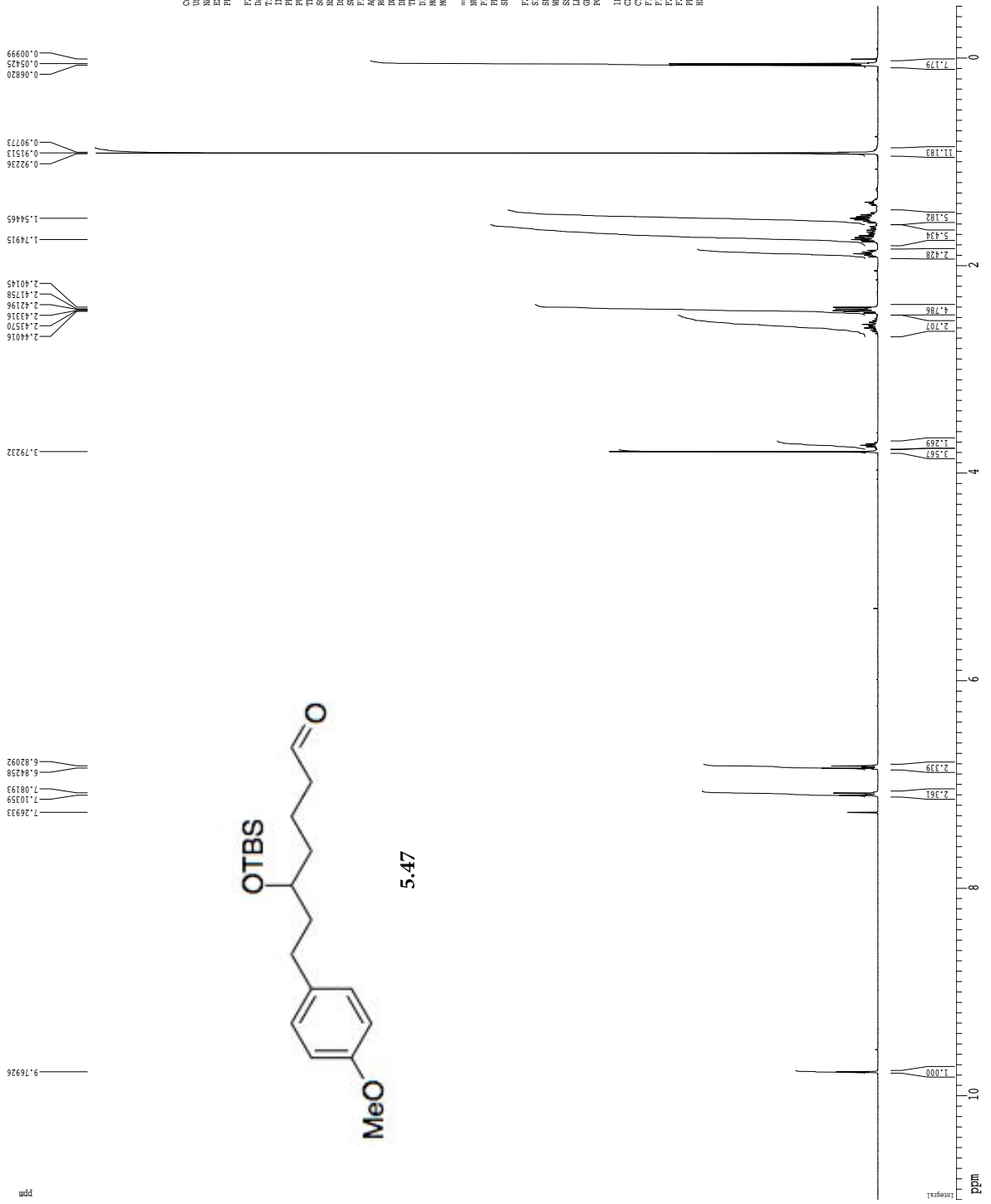
1H spectrum



Current Data Parameters  
 USER hewlett1  
 NAME RAH-7-159-1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 2/21/08  
 Time 14:42  
 INSTRUM dxt400  
 PROBEID 5 mm QNP H/F/P  
 PULPROG zgpg30  
 PC 32  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6412.90 Hz  
 FIDRES 0.166653 Hz  
 AQ 2.9599259 sec  
 RG 11.3  
 DW 78.000 usec  
 DE 1.10  
 TE 298.0 K  
 TD 65536  
 SFO1 400.132809 MHz  
 F2 - Processing parameters  
 SI 65536  
 SF 400.130175 MHz  
 WDW mo  
 SSB 0  
 GB 0  
 PC 2.00  
 ID MR plot parameters  
 CT 40  
 CY 15.00 cm  
 FIP 9.000 ppm  
 FI 3601.17 Hz  
 F2 -700.99 Hz  
 F2 0.41647 ppm/cm  
 F2CH 166.72084 Hz/cm



1H spectrum



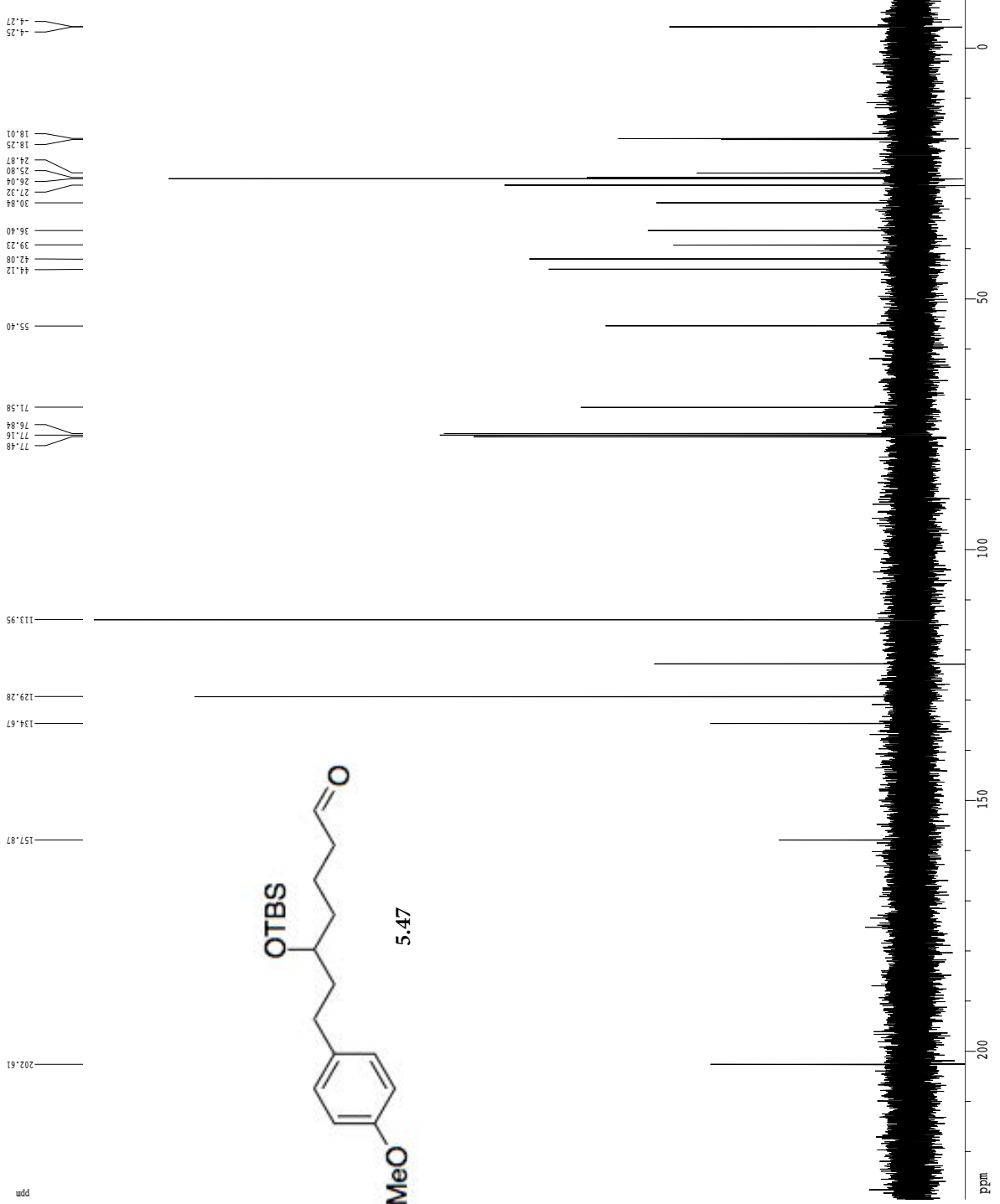
Current Data Parameters  
USER hewlett1  
NAME RAH-7-259-1  
PROCNO 1  
PROCXX 1

F2 - Acquisition Parameters  
Date\_ 2/27/02  
Time 14:05:00  
INSTRUM dxt400  
PROBHD 5 mm QNP H/F/FP  
PULPROG zgpg30  
AQ 3.90  
SOLVENT CDCl3  
NS 8  
DS 2  
SWH 6142.97 Hz  
FIDRES 0.166653 Hz  
AQ 2.9999249 sec  
RG 114  
DM 78.000 usec  
DE 19.000 usec  
TE 298.2 K  
D1 0.10000000 sec  
ICREST 0.00000000 sec  
PCWATK 0.01000000 sec  
===== CHANNEL f1 =====  
NUC1 1H  
P1 12.00 usec  
PL1 -0.00 dB  
SFO1 400.1324809 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300175 MHz  
WDW mo  
SSB 0  
GB 0  
PC 2.00

ID MR plot parameters  
CT 40.00 cm  
CY 15.00 cm  
FIP 11.000 ppm  
FL 4401.43 Hz  
F2 -700.00 Hz  
F3 -200.00 Hz  
FPMCH 0.50439 ppm/cm  
HMCH 20.41996 Hz/cm

<sup>13</sup>C spectrum with <sup>1</sup>H decoupling



```

Current Data Parameters
USER          khwitll
NAME          RAH-V-28-1
PROCNO       2
PROBHD       5 mm QNP B/B/P
PULPROG      zgpg30
TD            65536
AQ            0.19110000
RG            327.5
DS            4
SWH          24154.590 Hz
FIDRES       0.2866570 Hz
AQRES        1.102452
RG           10324.3
DE           20.700 us/c
DM           20.39 us/c
d11          0.10000000 sec
d12          0.03000000 sec
d13          0.00000000 sec
d14          0.00000000 sec
d15          0.00000000 sec
d16          0.00000000 sec
d17          0.00000000 sec
d18          0.00000000 sec
d19          0.00000000 sec
d20          0.00000000 sec
d21          0.00000000 sec
d22          0.00000000 sec
d23          0.00000000 sec
d24          0.00000000 sec
d25          0.00000000 sec
d26          0.00000000 sec
d27          0.00000000 sec
d28          0.00000000 sec
d29          0.00000000 sec
d30          0.00000000 sec
d31          0.00000000 sec
d32          0.00000000 sec
d33          0.00000000 sec
d34          0.00000000 sec
d35          0.00000000 sec
d36          0.00000000 sec
d37          0.00000000 sec
d38          0.00000000 sec
d39          0.00000000 sec
d40          0.00000000 sec
d41          0.00000000 sec
d42          0.00000000 sec
d43          0.00000000 sec
d44          0.00000000 sec
d45          0.00000000 sec
d46          0.00000000 sec
d47          0.00000000 sec
d48          0.00000000 sec
d49          0.00000000 sec
d50          0.00000000 sec
d51          0.00000000 sec
d52          0.00000000 sec
d53          0.00000000 sec
d54          0.00000000 sec
d55          0.00000000 sec
d56          0.00000000 sec
d57          0.00000000 sec
d58          0.00000000 sec
d59          0.00000000 sec
d60          0.00000000 sec
d61          0.00000000 sec
d62          0.00000000 sec
d63          0.00000000 sec
d64          0.00000000 sec
d65          0.00000000 sec
d66          0.00000000 sec
d67          0.00000000 sec
d68          0.00000000 sec
d69          0.00000000 sec
d70          0.00000000 sec
d71          0.00000000 sec
d72          0.00000000 sec
d73          0.00000000 sec
d74          0.00000000 sec
d75          0.00000000 sec
d76          0.00000000 sec
d77          0.00000000 sec
d78          0.00000000 sec
d79          0.00000000 sec
d80          0.00000000 sec
d81          0.00000000 sec
d82          0.00000000 sec
d83          0.00000000 sec
d84          0.00000000 sec
d85          0.00000000 sec
d86          0.00000000 sec
d87          0.00000000 sec
d88          0.00000000 sec
d89          0.00000000 sec
d90          0.00000000 sec
d91          0.00000000 sec
d92          0.00000000 sec
d93          0.00000000 sec
d94          0.00000000 sec
d95          0.00000000 sec
d96          0.00000000 sec
d97          0.00000000 sec
d98          0.00000000 sec
d99          0.00000000 sec
d100         0.00000000 sec

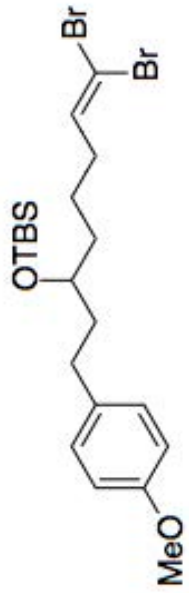
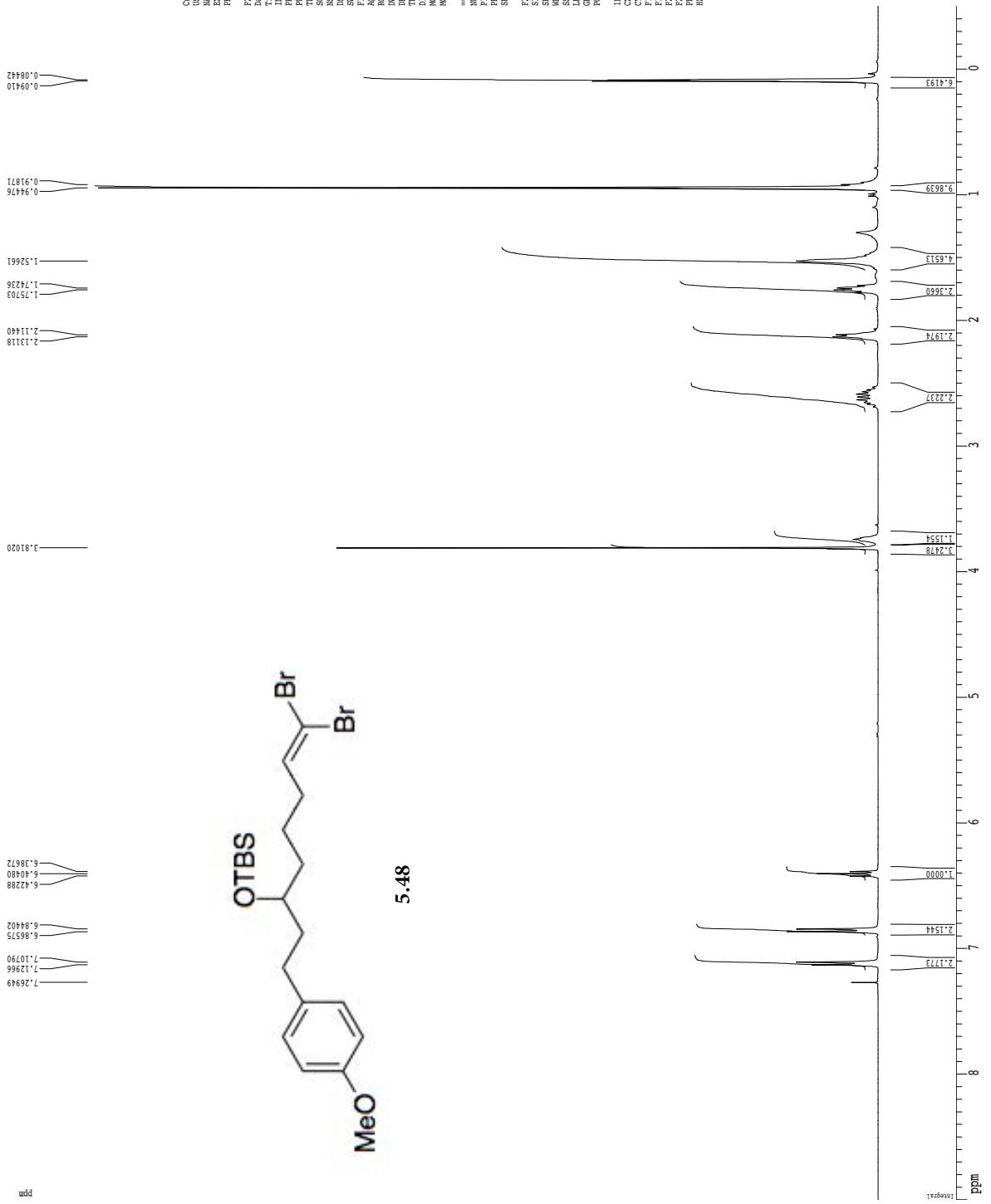
===== CHANNEL f1 =====
NUC1         13C
P1           7.50 us/c
PL1          7.00 dB
SFO1         100.627384 MHz

===== CHANNEL f2 =====
CPDPRG2     waltz16
NUC2         13C
P2           9.00 us/c
PL2          -0.90 dB
SFO2         400.132609 MHz

F2 - Processing Parameters
SI           65536
WDW          0
GB           0
GB1          0.00 Hz
GB2          0
PC           1.00

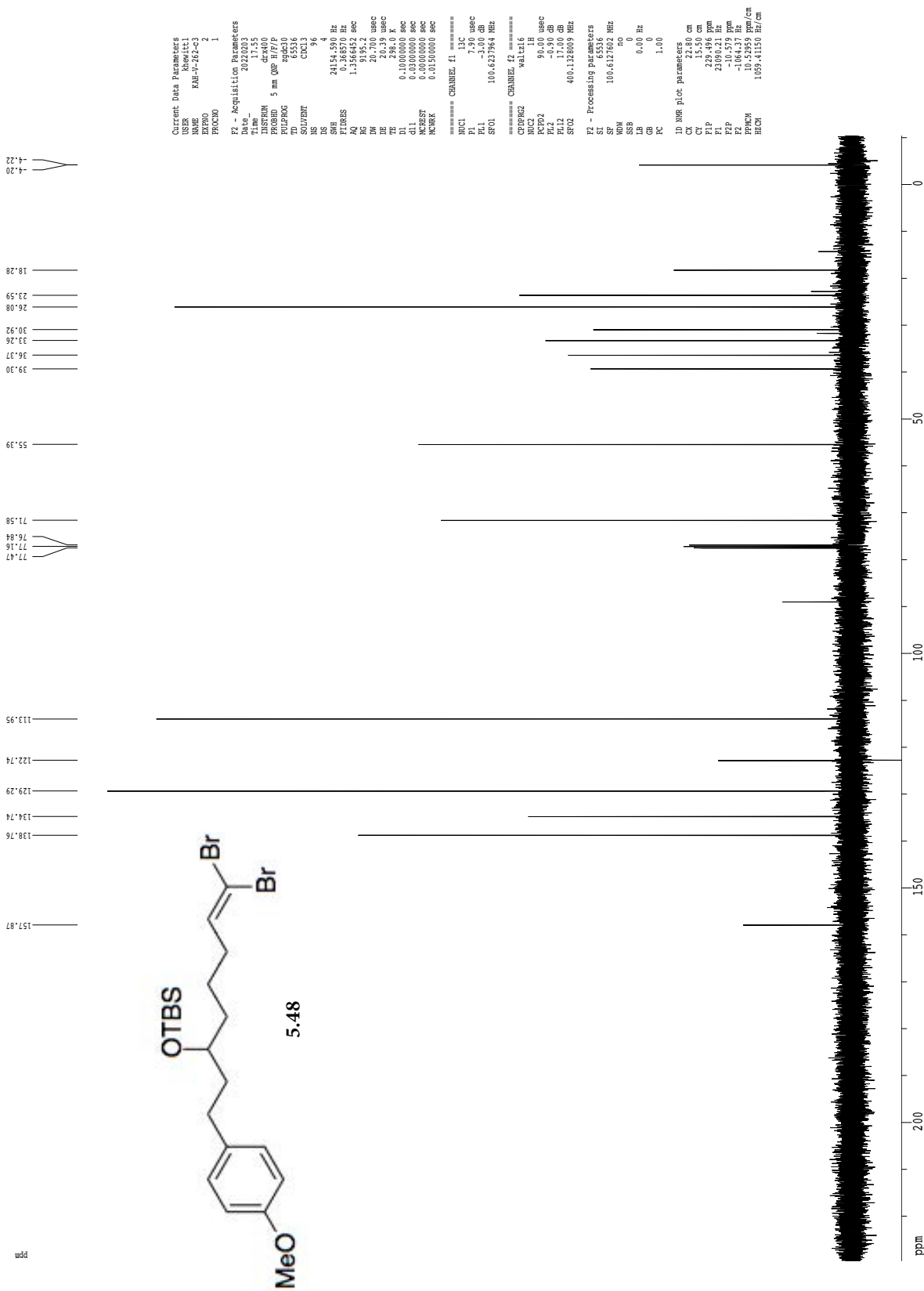
ID IMP Plot parameters
CX           22.80 cm
CY           15.50 cm
F1P         229.846 ppm
F2P         230.126 ppm
F3P         10.576 MHz
F4P         -1064.37 Hz
PRCKW       10.52859 ppm/cm
RZCN        1053.41150 Hz/cm
    
```

1H spectrum

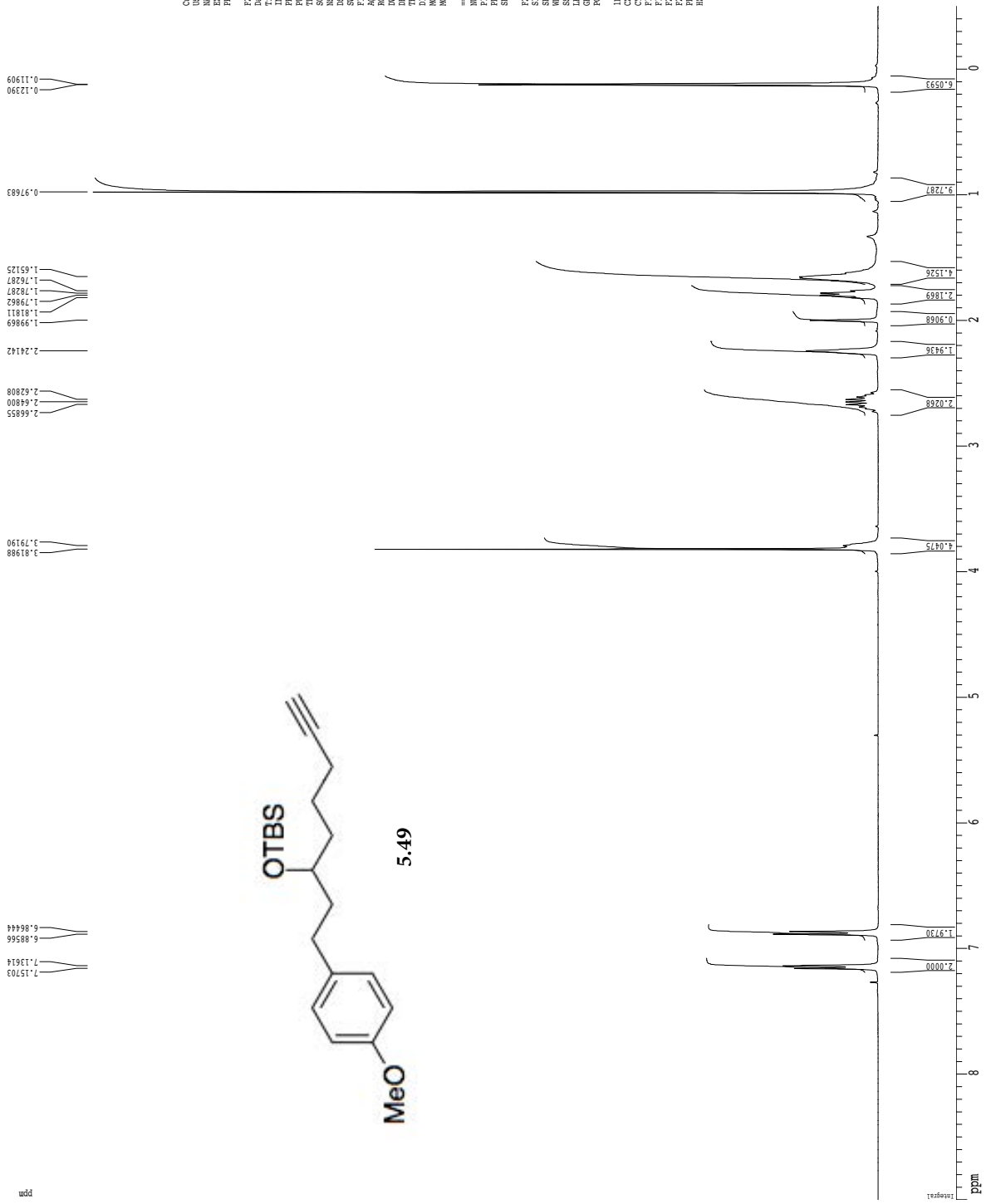


Current Data Parameters  
 USER hweatl1  
 NAME KAU-V-262-c3  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 2/22/03  
 Time 14:53  
 INSTRUM drr400  
 PROBD 5 mm QNP H/P/P  
 PULPROG zgpg30  
 SFO 400.130175  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6412.90 Hz  
 FIDRES 0.166653 Hz  
 AQ 2.9599259 sec  
 RG 57  
 DW 78.000 usec  
 DE 1.900 usec  
 TE 298.0 K  
 D1 0.1000000 sec  
 ACQRES 0.0000000 sec  
 PCW 0.0130000 usec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -0.90 dB  
 SFO1 400.1328009 MHz  
 F2 - Processing parameters  
 SF 400.130175 MHz  
 DS 2  
 WDW mo  
 SSB 0  
 GB 0  
 CB 0.0 Hz  
 PC 2.00  
 ID MR plot parameters  
 CT 15.00 cm  
 CY 15.00 cm  
 FIP 9.000 ppm  
 FI 3601.17 Hz  
 F2 -700.06 Hz  
 F3 0.000000 Hz  
 FPMCH 0.41667 ppm/cm  
 HZCM 166.72084 Hz/cm

13C spectrum with 1H decoupling

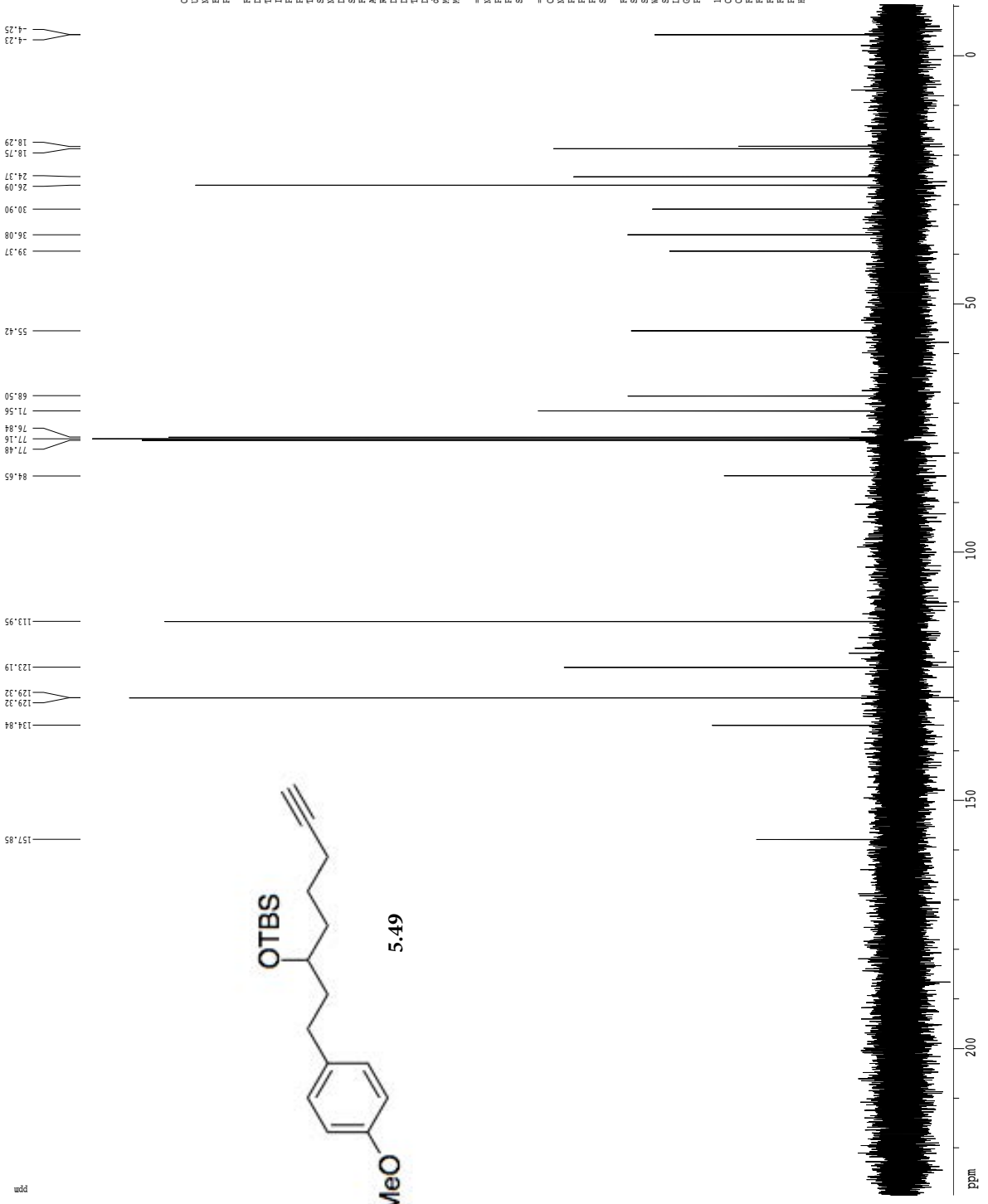


1H spectrum



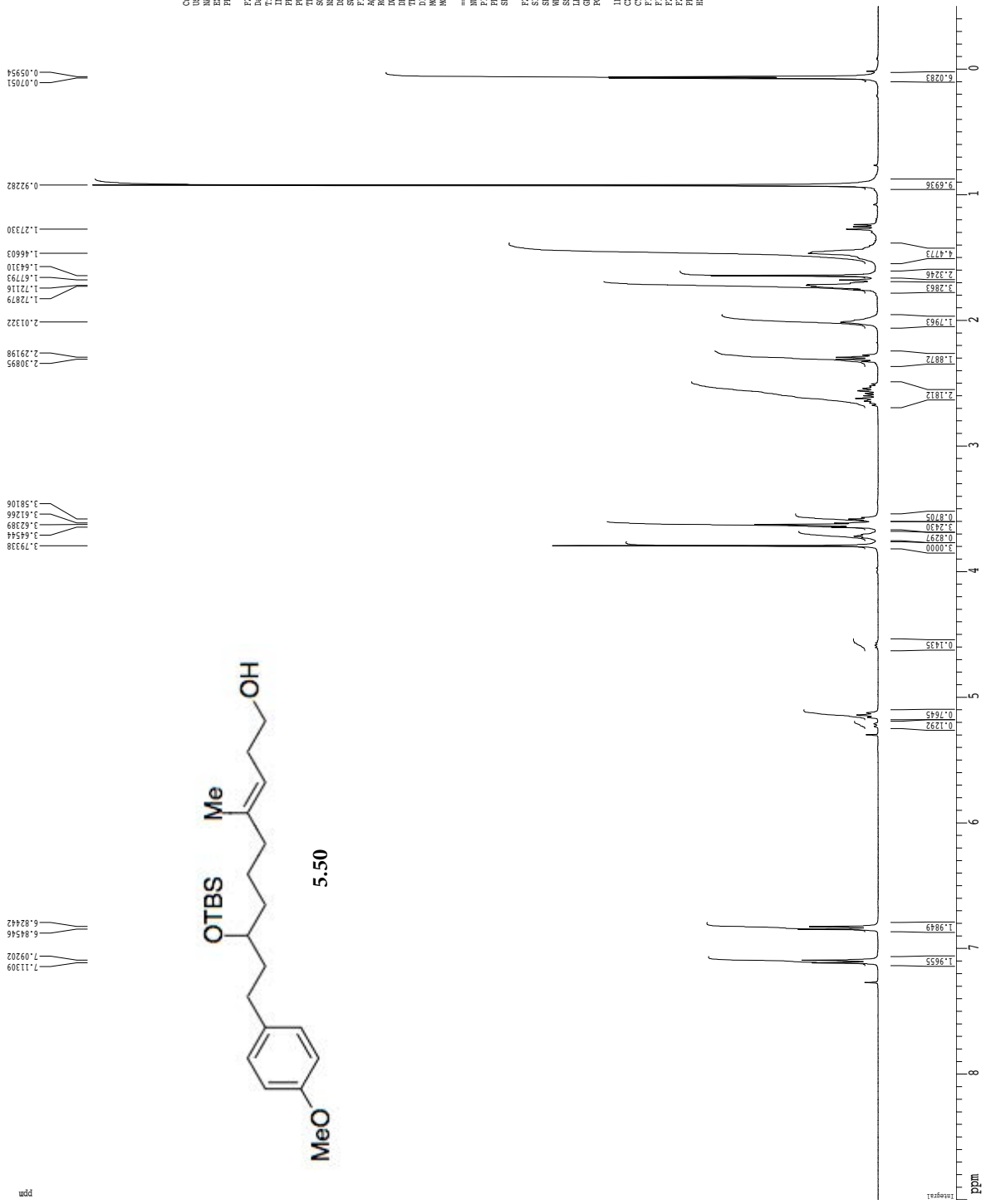
Current Data Parameters  
 USER hweilt1  
 NAME RAH-7-185-1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 202102  
 Time 14:00  
 INSTRUM dxt400  
 PROBEID 5 mm QNP H/F/P  
 PULPROG zgpg30  
 SFO1 400.1324009 MHz  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6412.91 Hz  
 FIDRES 0.166653 Hz  
 AQ 2.9999259 sec  
 RG 16  
 DW 78.000 usec  
 DE 19.00 usec  
 TE 298.0 K  
 D1 0.1000000 sec  
 ICHRES 0.0000000 sec  
 ACQMSK 0.0100000 sec  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.00 usec  
 PL1 -0.90 dB  
 SFO1 400.1324009 MHz  
 F2 - Processing parameters  
 SI 65536  
 SF 400.130175 MHz  
 WDW mo  
 SSB 0  
 GB 0.0 Hz  
 PC 2.00  
 ID MRB plot parameters  
 CT 15.00 cm  
 CY 15.00 cm  
 FIP 9.000 ppm  
 FI 3601.17 Hz  
 F2 -700.66 Hz  
 F3 -416.67 Hz/cm  
 FPMCH 166.72084 Hz/cm

13C spectrum with 1H decoupling





1H spectrum



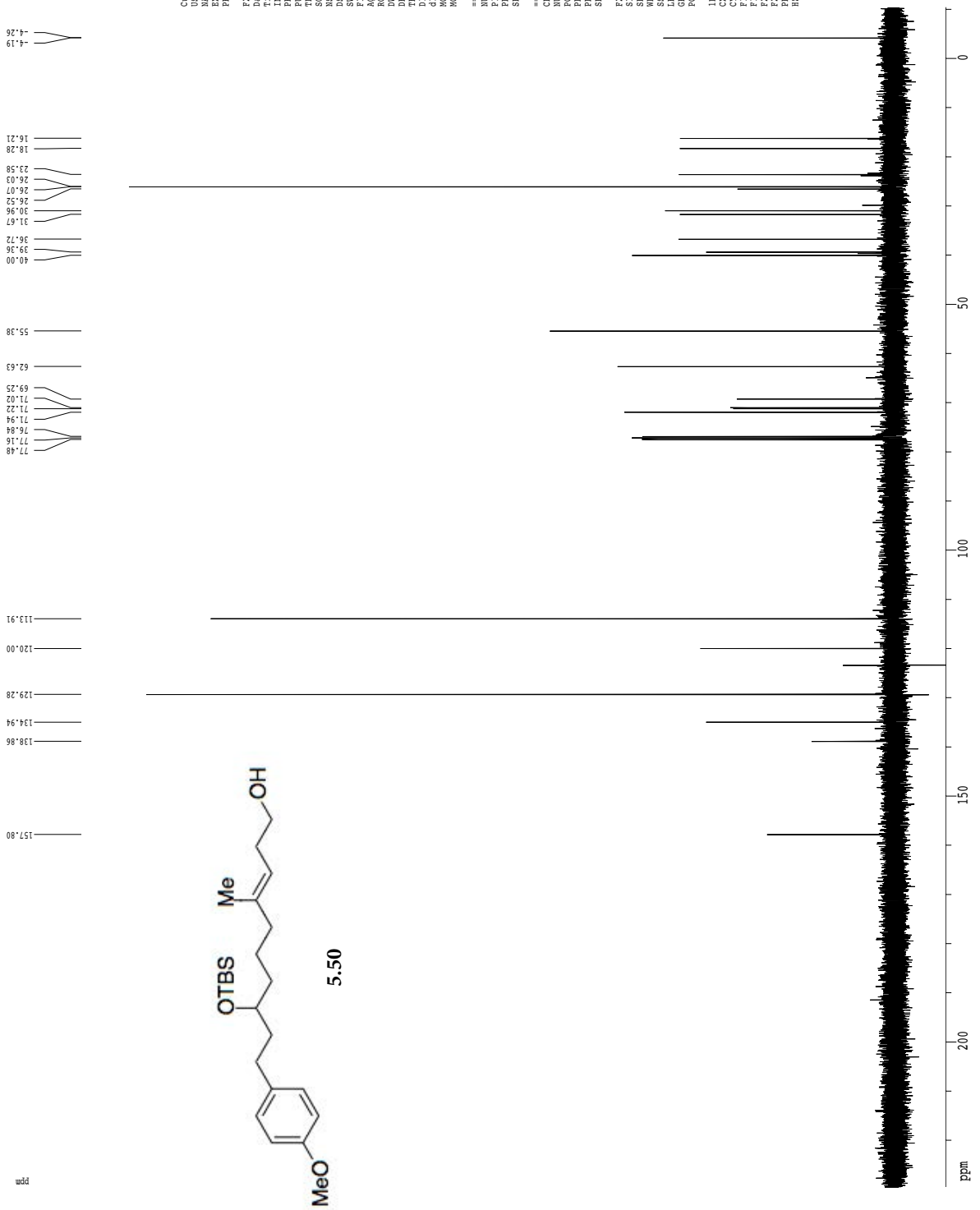
Current Data Parameters  
 USER hewlett  
 NAME RM-V-133-CHK  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 2/21/09  
 Time 12:40:00  
 INSTRUM dxt400  
 PROBEID 5 mm QNP H/P/P  
 PULPROG zgpg30  
 PCPRG23  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6142.90 Hz  
 FIDRES 0.166653 Hz  
 AQ 2.9999259 sec  
 RG 50.8  
 DW 76.000 usec  
 DE 19.00 usec  
 TE 298.0 K  
 TD 65536  
 SFO 400.1328000 MHz  
 FREQ 400.1328000 MHz  
 CHANNEL f1

F2 - Processing parameters  
 SI 32768  
 SF 400.1300175 MHz  
 WDM no  
 SSB 0  
 GB 0  
 CB 0.0 Hz  
 PC 2.00

ID MR plot parameters  
 CT 15.00 cm  
 CY 15.00 cm  
 FIP 9.000 ppm  
 FI 3601.17 Hz  
 F2 -700.66 Hz  
 F2 0.41667 ppm/cm  
 HZCM 166.72084 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters  
 USER Hewlett  
 NAME RM-193-cbk  
 PROC 7  
 PRCD 1

F2 - Acquisition Parameters  
 Date\_ 20211209  
 Time\_ 11:00:00  
 INSTRM dm40  
 PROBHD 5 mm QNP H/F/P  
 PULPROG zgpg30  
 CD 65536  
 CONVPT 0  
 NS 200  
 DS 4  
 SFO 24154.590 Hz  
 FIDRES 0.486432 Hz  
 AQ 1.566432 sec  
 RG 9195.2  
 DW 20.700 usec  
 DE 20.39 usec  
 TE 300.2 K  
 D1 0.1000000 sec  
 d11 0.0300000 sec  
 ACQRES 0.0000000 sec  
 ACQBRK 0.0150000 sec

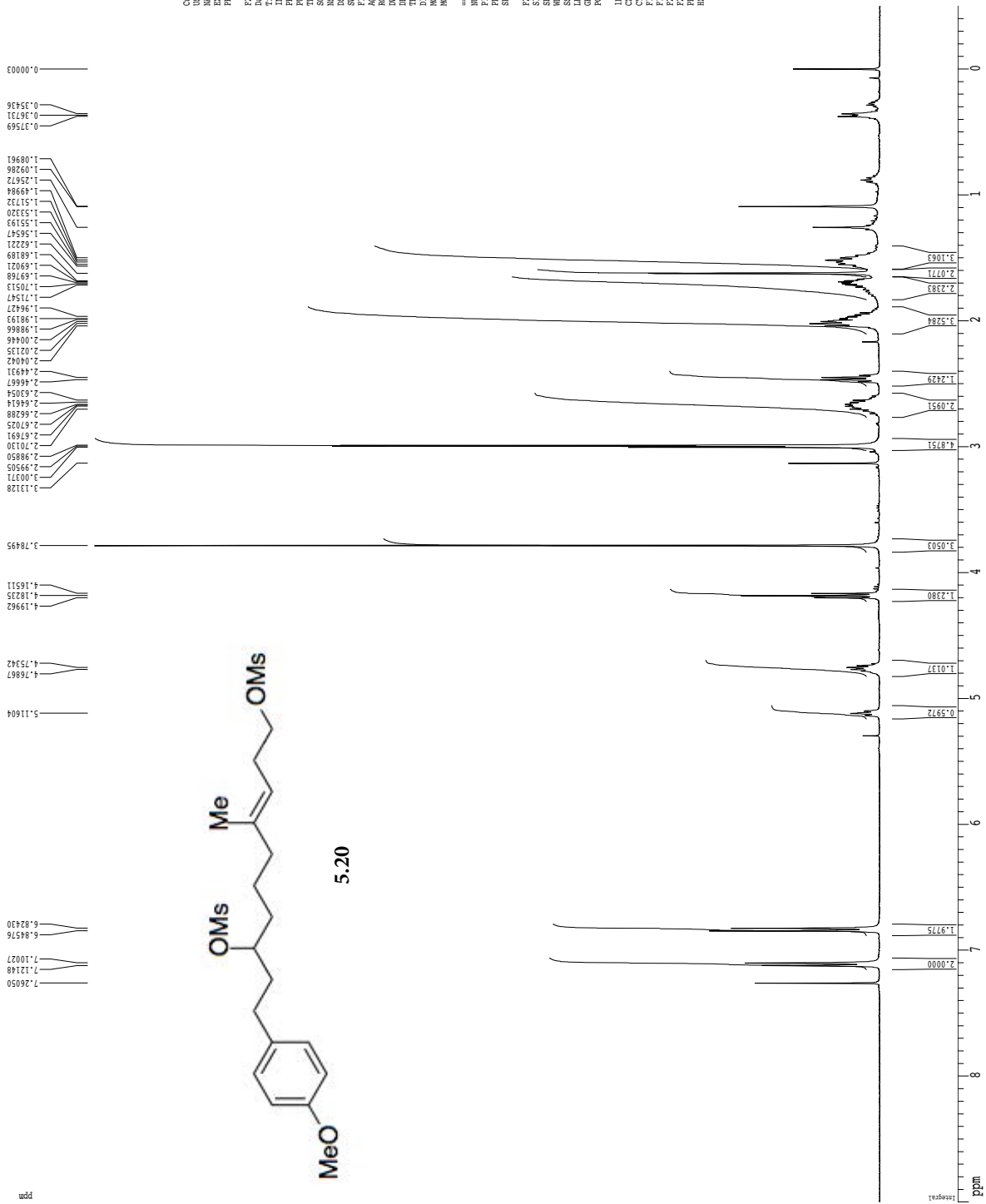
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 7.50 usec  
 PL1 -5.00 dB  
 SFO1 100.627594 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 13C  
 P2 90.00 usec  
 PL2 -0.90 dB  
 PL12 17.00 dB  
 SFO2 400.132809 MHz

F2 - Processing parameters  
 SI 65536  
 SF 100.612759 MHz  
 DS 4  
 LB 0.00 Hz  
 GB 0  
 FC 1.00

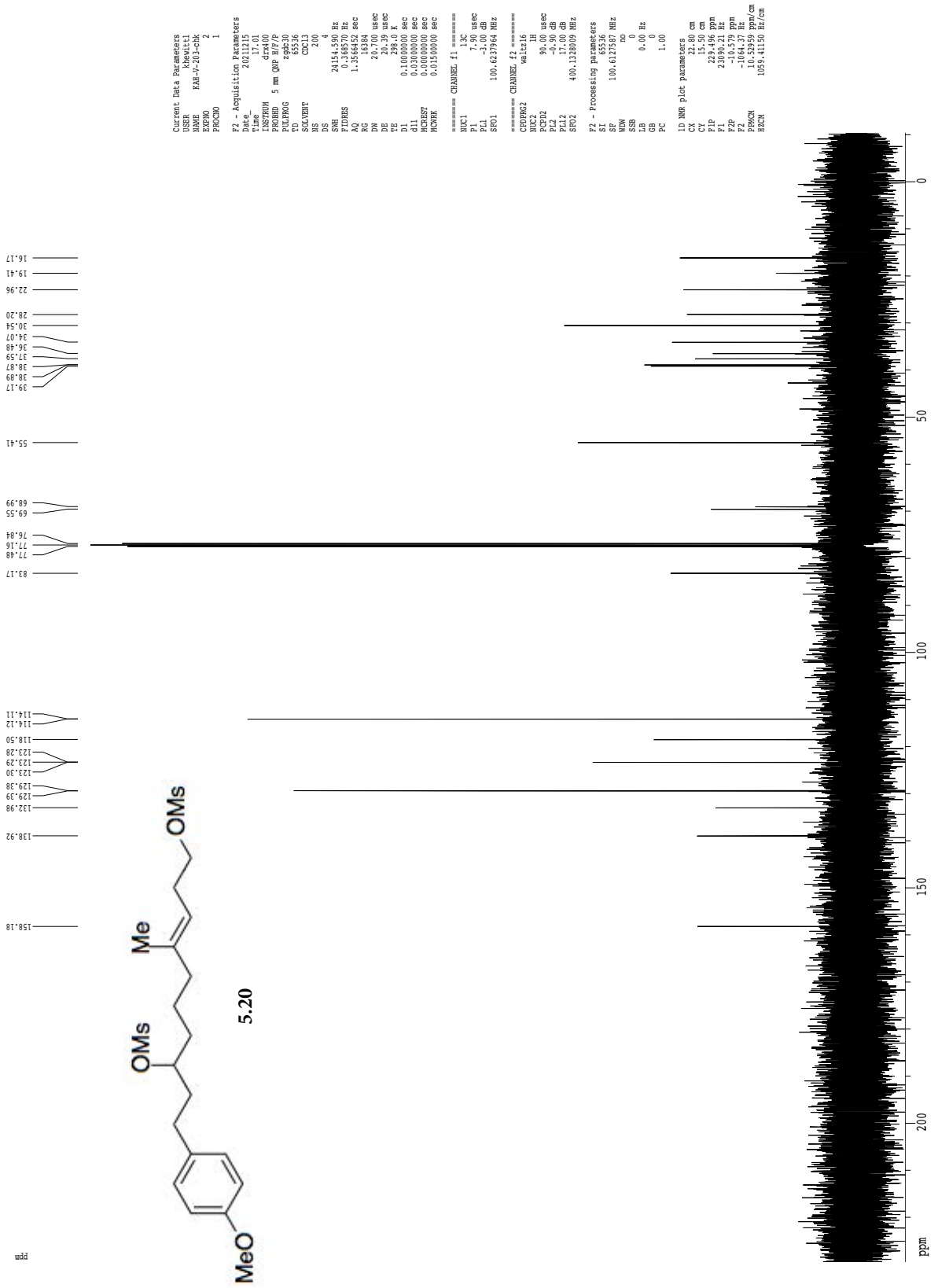
1D NMR Plot parameters  
 CX 22.80 cm  
 CY 15.50 cm  
 F1P 228.496 ppm  
 F2P 100.627594 MHz  
 F2 -1064.37 Hz  
 PPMCN 10.52959 ppm/cm  
 HZCN 1035.41150 Hz/cm

1H spectrum

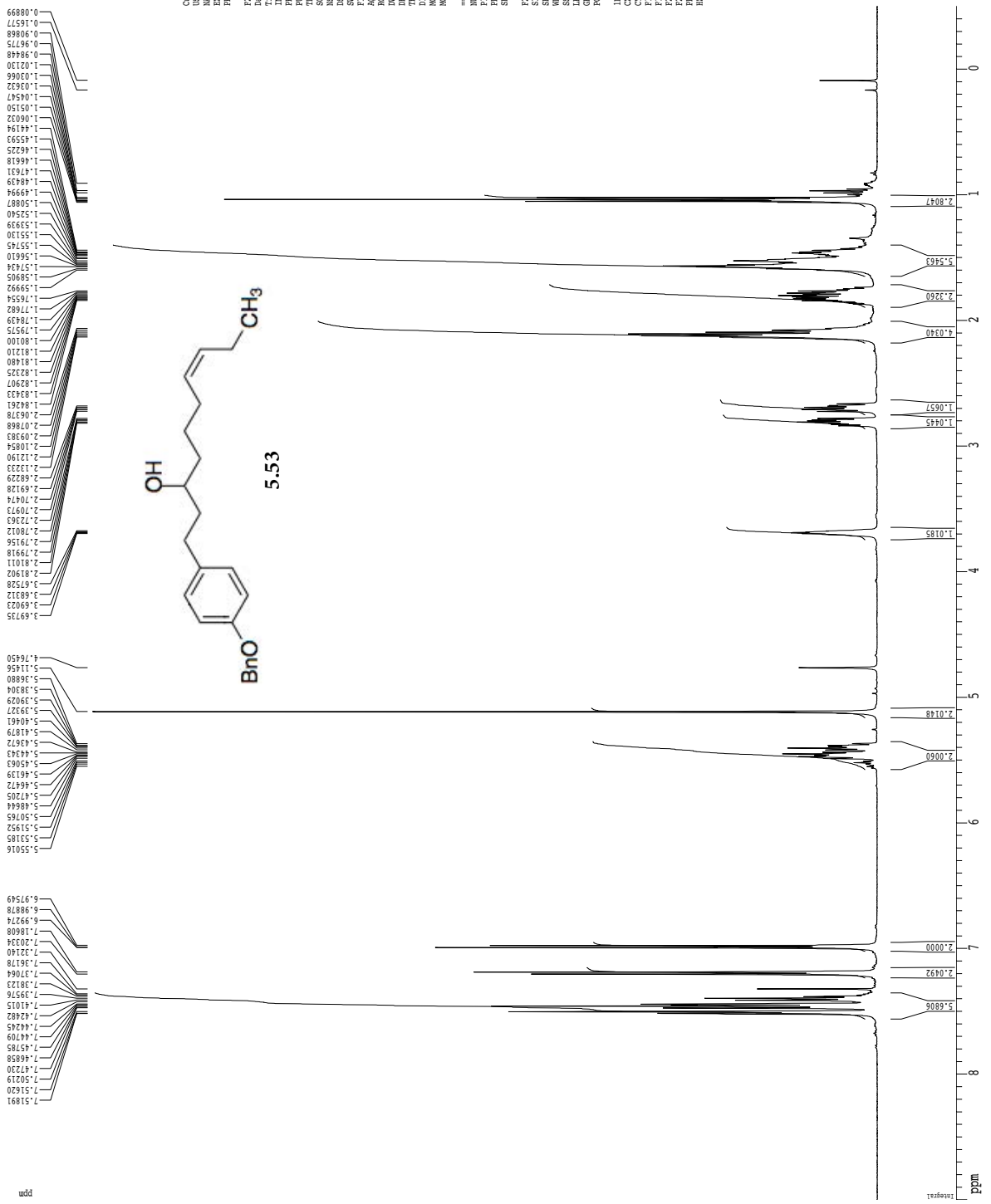


Current Data Parameters  
 USER khowl1  
 NMRE RMH-V-20-cbk  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20111118  
 Time\_ 11:42:11  
 INSTRUM dxs400  
 PROBHD 5 mm QNP 1H/13C  
 PULPROG zgpg30  
 CHLOROP CHLORO  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 6410.27 Hz  
 FIDRES 0.166673 Hz  
 AQ 2.9999299 sec  
 RG 181  
 DW 78.000 usec  
 DE 1.0000000 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 ACQSTRT 0.00000000 sec  
 ACQSTP 0.01000000 sec  
 ===== CHANNEL f1 =====  
 NUC1 13C  
 P1 15.00 usec  
 PL 0.00 dB  
 SFO1 400.1328009 MHz  
 F2 - Processing parameters  
 SI 32768  
 SF 400.1300211 MHz  
 WDW no  
 SSB 0  
 GB 0  
 PC 2.00  
 ID NMR plot parameters  
 SI 32768  
 CF 15.00 cm  
 FID 15.00 cm  
 F1P 5.000 ppm  
 F1 3601.17 Hz  
 F2P -200.06 ppm  
 F2 -200.06 Hz  
 FWHM 0.41667 ppm/cm  
 HZCN 166.72886 Hz/cm

<sup>13</sup>C spectrum with <sup>1</sup>H decoupling

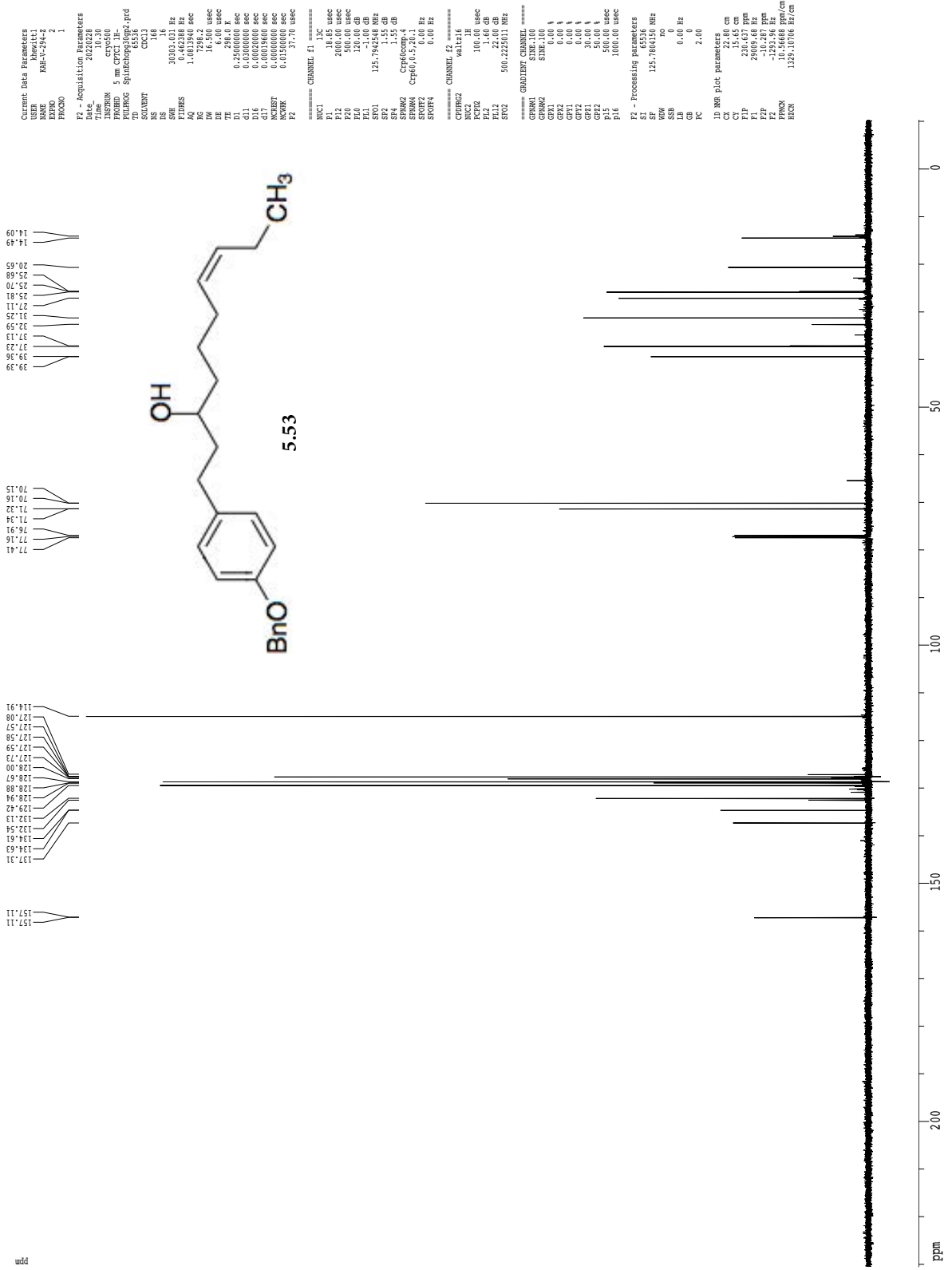


**<sup>1</sup>H spectrum**

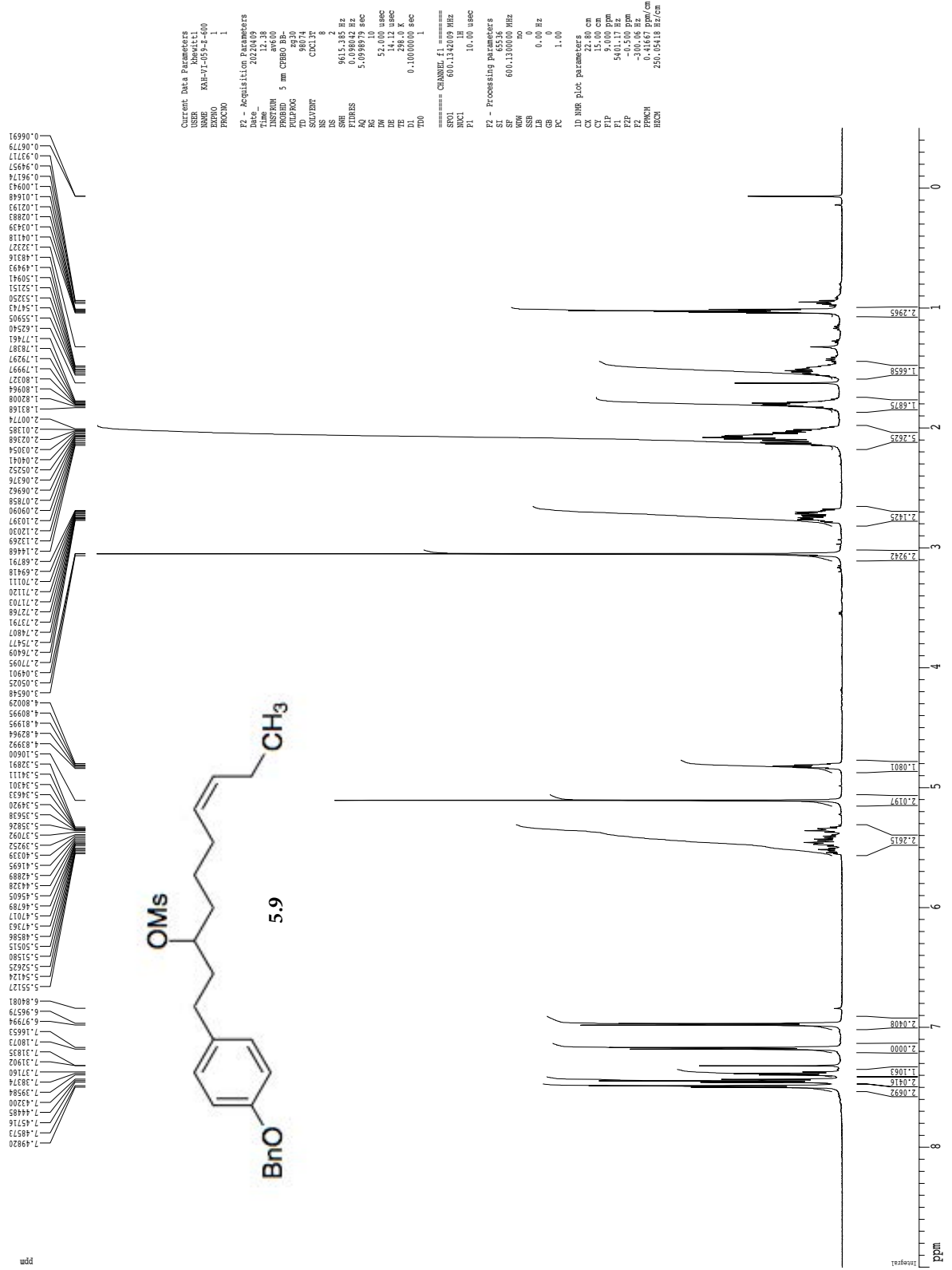


Current Data Parameters  
 USER: hewitt1  
 NAME: RAH-7-294-2  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 2/22/28  
 Time: 16:06  
 INSTRUM: cryo500  
 PROBEHD: 5 mm CPCLP 1H-  
 PULPROG: zg30  
 AQC: 6.00  
 SOLVENT: CDCL3T  
 NS: 8  
 DS: 2  
 SWH: 6073.67 Hz  
 FIDRES: 0.166677 Hz  
 AQ: 2.9998677 sec  
 RG: 4.5  
 DW: 62.400 usec  
 DE: 1.900 usec  
 TE: 298.0 K  
 D1: 0.1000000 sec  
 MCHRES: 0.0000000 sec  
 PCWAK: 0.0150000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 1H  
 P1: 12.00 usec  
 PL1: 0.00 dB  
 SFO1: 500.2235015 MHz  
 F2 - Processing parameters  
 SI: 32768  
 SF: 500.220000 MHz  
 WDM: no  
 SSB: 0  
 GB: 0.0 Hz  
 PC: 1.00  
 ID: MMR plot parameters  
 CT: 40  
 CY: 15.00 cm  
 FIP: 9.000 ppm  
 FI: 4501.88 Hz  
 F2: -750.31 Hz  
 FPMCH: 0.41667 ppm/cm  
 HZCM: 208.42500 Hz/cm

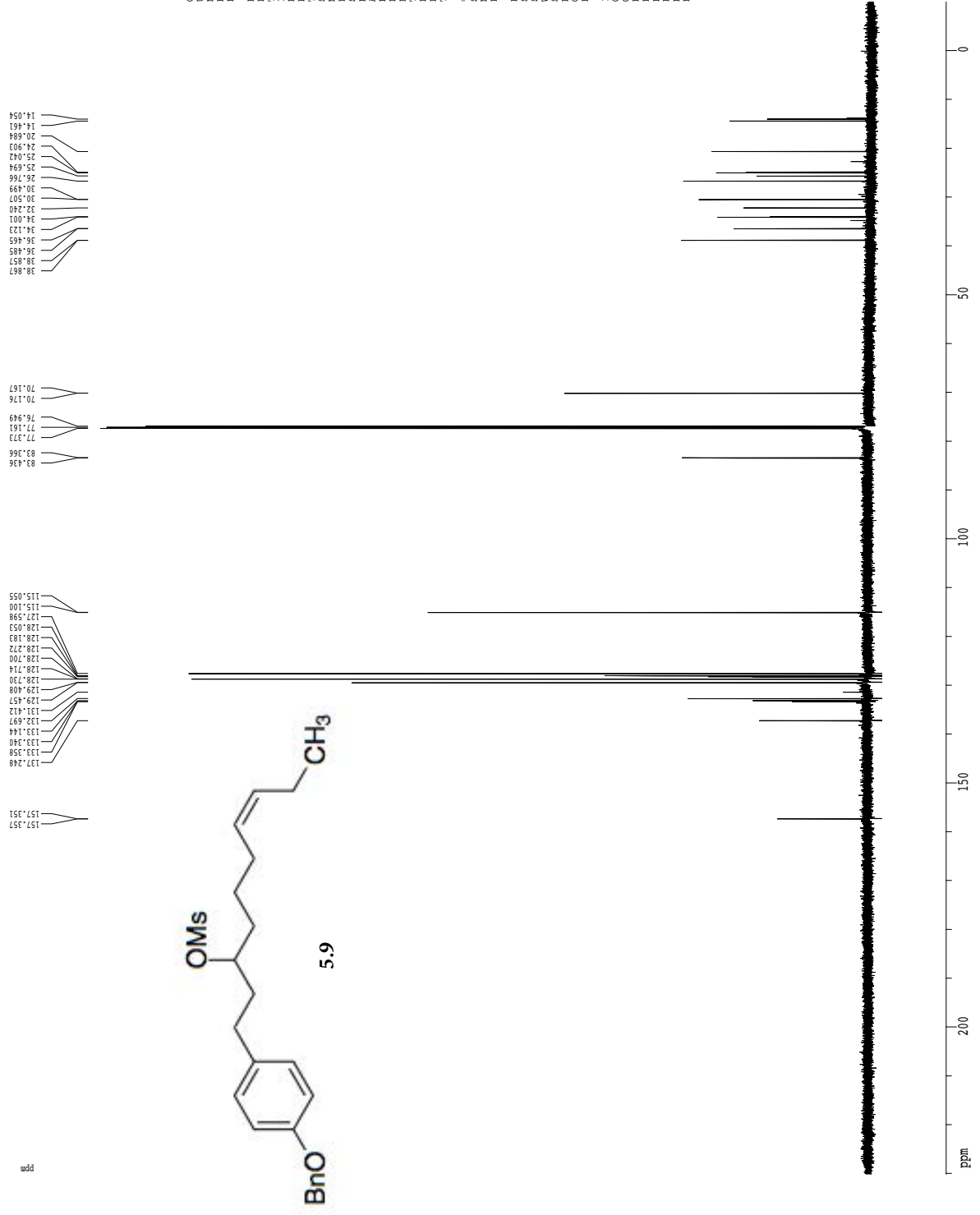
Z-restored spin-echo 13C spectrum with 1H decoupling



1H spectrum



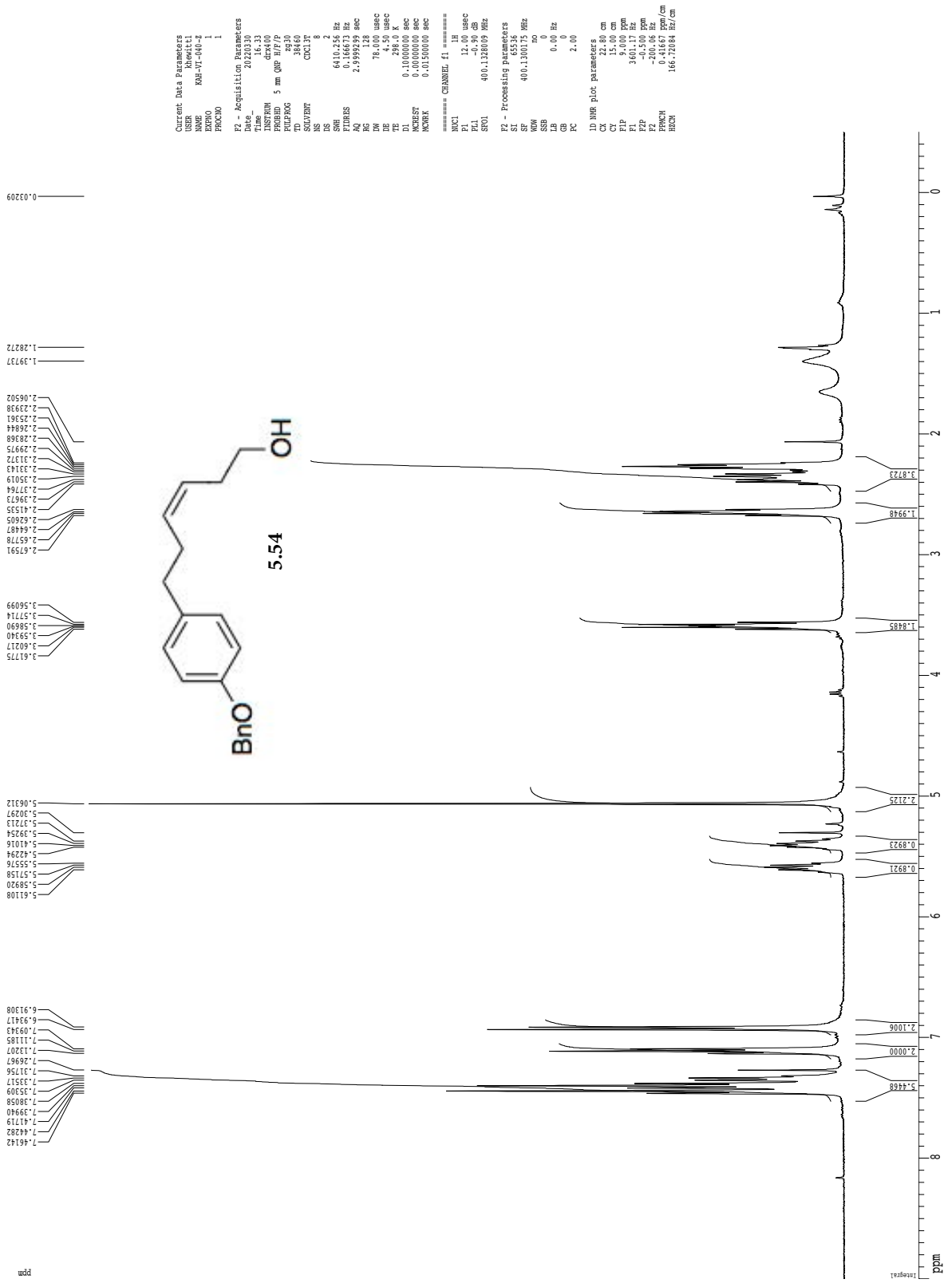
13C spectrum



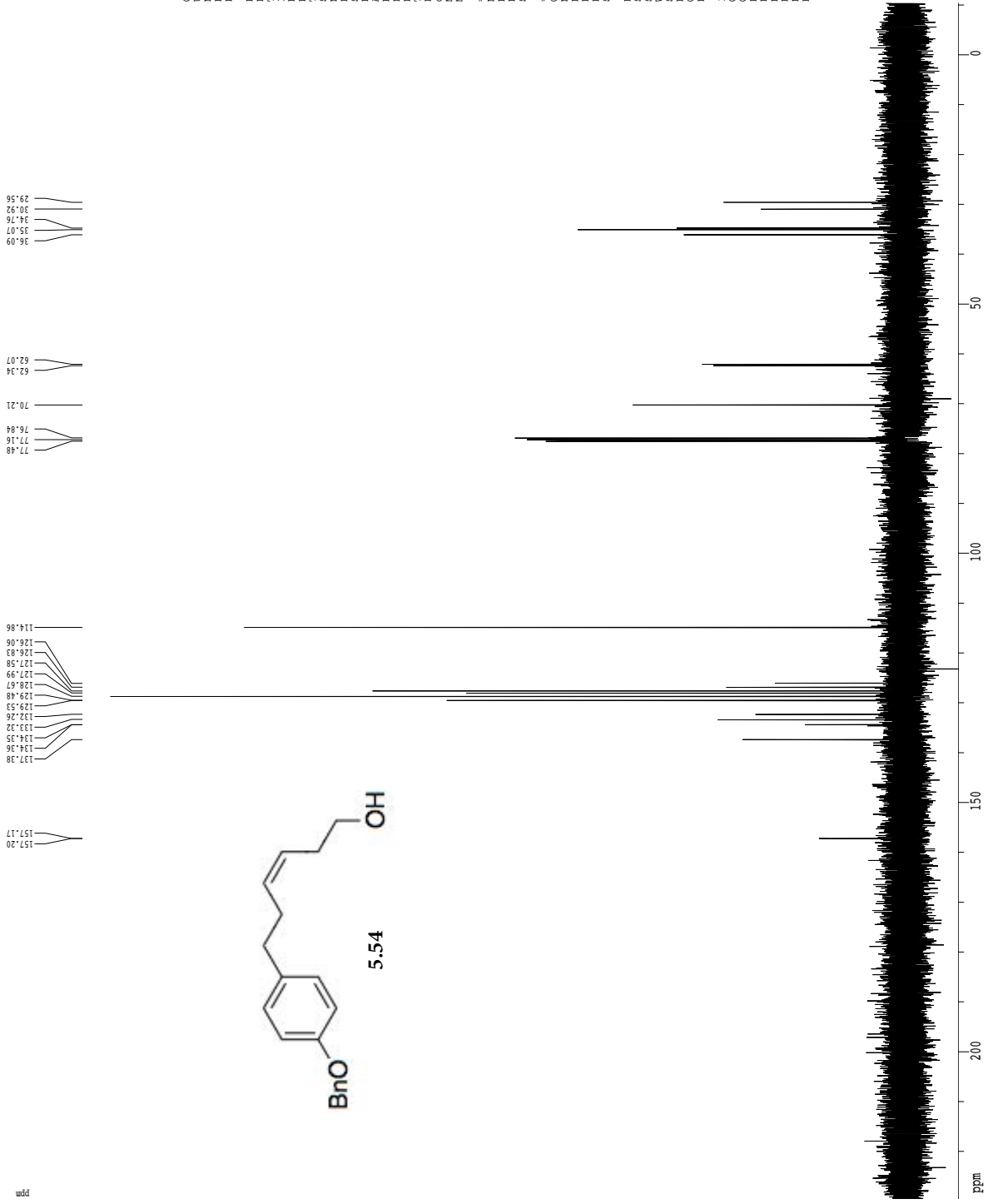
Current Data Parameters  
 NAME K2H-VI-059-P-610  
 EXPNO 2  
 PROCNO 1  
 P2 - Acquisition Parameters  
 Date\_ 20220409  
 Time 12:43  
 Operator  
 PULPROG 5 mm CDEPR0  
 PCPRG05  
 TD 65536  
 SOLVENT CDCl3  
 NS 4  
 DS 4  
 SWH 36231.883 Hz  
 FIDRES 0.552855 Hz  
 AQ 0.339786 sec  
 RG 7855  
 DW 13.800 usec  
 DE 19.63 usec  
 TE 300.2 K  
 D1 0.4000001 sec  
 D11 0.0300000 sec  
 TDO 1  
 ===== CHANNEL f1 =====  
 SFO1 150.9194080 MHz  
 NUC1 13C  
 P1 10.10 usec  
 P2 - Processing parameters  
 SI 65536  
 SF 150.9027875 MHz  
 DS 0  
 SW 0.00 Hz  
 GB 0  
 PC 1.00  
 ID NMR Plot parameters  
 CX 22.80 cm  
 CY 15.00 cm  
 CZ 0.00 cm  
 FL 34726.45 Hz  
 F2 34726.45 Hz  
 F3 0.00 Hz  
 F4 -9.976 ppm  
 F5 -1.90544 Hz  
 F6 0.00000 Hz  
 F7 0.00000 Hz  
 F8 0.00000 Hz  
 F9 0.00000 Hz  
 F10 0.00000 Hz  
 F11 0.00000 Hz  
 F12 0.00000 Hz  
 F13 0.00000 Hz  
 F14 0.00000 Hz  
 F15 0.00000 Hz  
 F16 0.00000 Hz  
 F17 0.00000 Hz  
 F18 0.00000 Hz  
 F19 0.00000 Hz  
 F20 0.00000 Hz



1H spectrum



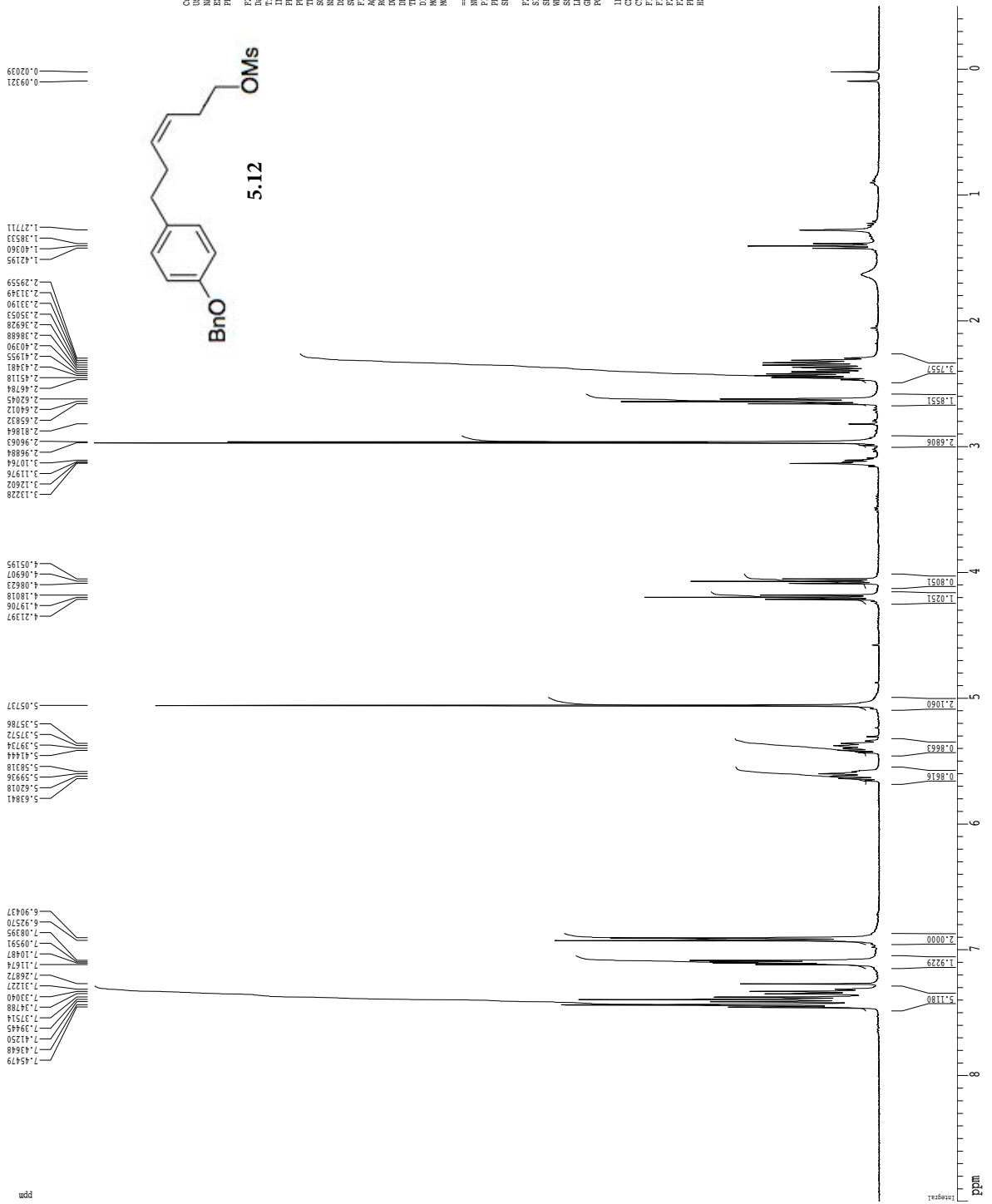
13C spectrum with 1H decoupling



```

Current Data Parameters
USER      khwa111
NAME      F4H-VI-041-2
EXPNO     2
PROCNO    1
F2 - Acquisition Parameters
Date_     202010
Time      14:05
INSTRUM   direct
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
TD         65536
SFO2      400.128009 MHz
AQ         0.1715
RG         315
DS         4
SHH        24154.500 Hz
NUC1       13C
NUC2       1H
F1         7.50 usec
P1         -3.00 dB
SFO1      100.627384 MHz
===== CHANNEL f1 =====
CPDPRG2   waltz16
NUC1       13C
NUC2       1H
PCPD2      90.00 usec
PL2        -0.90 dB
PL12       17.00 dB
SFO2      400.128009 MHz
F2 - Processing parameters
SI         65536
SF         100.627610 MHz
WDW        no
SSB        0
GB         0
CB         1.00
PC         1.00
ID_NMR     plot parameters
CX         22.80 cm
CY         15.50 cm
F1_P       229.496 ppm
F2_P       10.519 ppm
F2         -1.66437 Hz
PP4CH     10.52859 ppm/cm
BECON     1059.41150 Hz/cm
    
```

1H spectrum



**<sup>13</sup>C spectrum with <sup>1</sup>H decoupling**

