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Structures of Bicyclo[2.2.1] Systems IV. 6-Endohydroxy-3-Endo-Aminomethylbicyclo[2.2.1] Heptane-2-Endo-Carboxylic Acid Lactam

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STRUCTURES OF BICYCLO[2.2.1] SYSTEMS IV. 6-ENDO-HYDROXY-3-ENDO-AMINOMETHYLBICYCLO[2.2.1]HEPTANE-2-ENDO-CARBOXYLIC ACID LACTAM

Arthur J. Olson, David H. Templeton, and Lieselotte K. Templeton

December 1976

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For Reference

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LBL-5761

STRUCTURES OF BICYCLO[2.2.1] SYSTEMS IV.

6-ENDO-HYDROXY-3-ENDO-AMINOMETHYLBICYCLO[2.2.1]HEPTANE-2-ENDO-CARBOXYLIC

ACID LACTAM

By Arthur J. Olson, David H. Templeton and Lieselotte K. Templeton

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and

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Abstract. $C_9H_{13}NO_2$, monoclinic, $P2_1/c$; at 23°C, a = 10.870(3), b = 7.403(2), c = 10.721(4) Å, $\beta = 107.46(2)^\circ$, Z = 4, $D_x = 1.350(2)$, $D_m = 1.35(1)$ g cm⁻³. For 1169 reflections with I > σ , R = 0.055 and $R_w = 0.051$. The O(hydroxy1)-C(carbony1) distance is 2.832 Å, two hydrogen atoms are 2.00 Å from each other, and these distances would be even shorter except for distortion of some of the bond angles. Hydrogen bonds from the hydroxy1 and amide groups to the carbony1 oxygen atom link the molecules into chains.

Work done in part with support from the U.S. Energy Research and Development Administration.

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<u>Introduction</u>. To explore effects of orientation on rates of chemical reactions analogous to those catalyzed by enzymes, Koshland and co-workers have measured intramolecular reaction rates for norbornane molecules substituted with a variety of functional groups (Storm & Koshland, 1972). The title compound, a hydroxylactam, was synthesized by Hackney (1975) to serve, in its base catalyzed rearrangement to an aminolactone, as a model for the rate determining step in the hydrolysis of amides by chymotrypsin.



hydroxylactam

CH2NH2

aminolactone

We determined the crystal structure of this lactam as part of a program to establish the geometry of substituted norbornane molecules (Chapuis, Zalkin & Templeton, 1973, 197x, 197y). A sample which had been recrystallized from ethyl acetate (m.p. 185°C) was kindly supplied to us by Professor D. E. Koshland and Dr. D. D. Hackney.

A clear colorless crystal 0.18 x 0.12 x 0.15 mm in size was studied by photographic and diffractometer techniques. Laue symmetry 2/m and absent reflections h0l ($l \neq 2n$) and 0k0 ($k \neq 2n$) indicate space group P2₁/c. Setting angles for 12 reflections with 30° < 20 < 40° (MoK α_1 , λ 0.70926 Å) were used for least-squares adjustment of

-1-

the cell dimensions. The density was measured by flotation in solutions of CC1₄ and petroleum ether. Intensities were measured with graphite-monochromatized MoKa radiation and 0-20 scan technique for all the reflections in the half-sphere with ℓ nonnegative and 20 < 50°, many of them twice (a total of 4685 measurements). The scan extended 2° in 20 with a rate of 1°/min. Background was counted for 10 sec near each end of the scan. After equivalent measurements were averaged there were 1467 unique reflections, of which 1169 had I > σ . Absorption was small, $\mu = 0.57$ cm⁻¹, and no correction was made.

-2-

The crystal structure was solved by direct methods with the MULTAN program (Germain, Main & Woolfson, 1971). All hydrogen atoms were found in a ΔF map. The structure was refined with Zalkin's full matrix least squares program, modified (Olson, 1975) to use polar scattering factors for hydrogen (Stewart, Davidson & Simpson, 1965) so that the hydrogen coordinates more nearly represent the positions of the protons. For other atoms we used the scattering factors of Doyle & Turner (1968) with dispersion corrections from Cromer & Liberman (1970). Zero weight was assigned to reflections with I < σ ; otherwise, w = $(\sigma(F))^{-2}$; $\sigma(F)$ was derived from $\sigma(I) = [s^2 + (0.058 I)^2]^{1/2}$ where s^2 is the variance due to counting statistics. In the last cycle no parameter shifted more than 0.003 σ . The final residual R = $\Sigma |\Delta F| / \Sigma | F_o|$ was 0.055 for 1169 reflections and 0.078 for 1467 reflections including those of zero weight. The weighted residual minimized by the refinement was $R_w = [\Sigma w (\Delta F)^2 / \Sigma w F_o^2]^{1/2}$

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-3-

= 0.051. Final parameters are listed in Table 1 and 2.

Discussion. A view of the molecular structure, Fig. 1, shows the numbering of the atoms. The bond distances, Table 3, and most of the bond angles, Table 4, are normal. The angles C(2)-C(1)-C(6) =111.8° and C(3)-C(4)-C(5) = 110.7° are larger than any of the experimental or theoretical values reported for norbornane (Altona and Sundaralingam, 1972), and they indicate a distortion of the molecule in the direction which partially relieves the crowding of the substituent groups. Angle $C(1)-C(6)-O(2) = 115.9^{\circ}$ is large for the same reason. The distance O(2)-C(9) = 2.832(3) Å may be compared with 2.783 Å for an analogous distance in the sodium salt of 2-exo-methyl-6-endo-hydroxybicyclo[2.2.1]heptane-2-endo-carboxylic acid (Chapuis, et al., 197x) where the C(2)-C(1)-C(6) angle is 112.2° . In the present case the stress of the lactam ring results in a larger C(1)-C(2)-C(9) angle and thus a longer O(2)-C(9) distance with slightly less strain of the norbornane skeleton than in the salt of the 2,6,2-methylhydroxyacid. The enlargement of the C(3)-C(4)-C(5) angle probably results from the crowding of H(5) and H(10) which are at the abnormally short distance 2.00(4) Å. No other hydrogen atoms in the molecule are closer than 2.26 Å except those which are attached to a common carbon atom. In the methylhydroxyacid salt mentioned above

A listing of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. there was no corresponding crowding at the other end of the molecule, and the angle at C(4) was 107.6° .

-4-

For reference in discussion of the rates of conversion to the lactone (Hackney, 1975) in the context of the work reviewed by Bürgi (1975) we include in Table 4 some angles involving the O(2)-C(9) vector.

Figure 2 shows the molecular packing. Hydrogen bonds from the amide and alcohol groups to the carbonyl oxygen atom tie molecules together in strings along the screw axis. Table 5 gives distances and angles descriptive of these hydrogen bonds.

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-5-

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5815-5825.

Parameters for Heavy Atoms. The form of the temperature factor is $exp[-0.25(h^2a*^2B_{11}+2hka*b*B_{12}+...)]$. Table 1.

	x	У	z	^B 11	^B 22	^B 33	^B 12	^B 13	^B 23
C(1)	.2119(2)	.5819(3)	.1390(2)	2.13(9)	1.9(1)	3.5(1)	.10(8)	.88(8)	.51(9)
C(2)	.1548(2)	.3927(3)	.0960(2)	2.17(9)	2.4(1)	2.6(1)	14(8)	.63(9)	.02(8)
C(3)	.2756(2)	.2730(3)	.1080(3)	2.8(1)	2.5(1)	3.6(1)	04(8)	1.61(9)	3(1)
C(4)	.3876(2)	.4061(3)	.1661(3)	2.04(9)	2.8(1)	4.4(1)	.01(8)	1.45(9)	0(1)
C(5)	.3935(2)	.4582(4)	.3050(3)	2.2(1)	3.1(1)	4.0(1)	.17(9)	.54(9)	1(1)
C(6)	.2796(2)	.5904(3)	.2863(3)	2.4(1)	2.0(1)	3.7(1)	23(8)	.94(9)	24(9)
C(7)	.3304(3)	.5780(4)	.0893(3)	2.9(1)	2.9(1)	4.2(1)	65(9)	1.4(1)	.3(1)
C(8)	.2605(3)	.1071(4)	.1879(3)	3.4(1)	1.9(1)	5.3(2)	.17(9)	2.1(1)	.0(1)
C(9)	.0790(2)	.2932(3)	.1700(2)	2.37(9)	1.9(1)	3.5(1)	27(8)	1.02(8)	46(9)
0(1)	0292(1)	.3361(2)	.1782(2)	2.15(7)	2.53(8)	5.3(1)	16(6)	1.60(7)	26(7)
0(2)	.2032(2)	.5427(3)	.3680(2)	3.21(8)	3.23(8)	3.92(9)	.55(7)	1.61(7)	.19(7)
N(1)	.1405(2)	.1408(3)	.2191(2)	2.93(9)	1.95(9)	4,9(1)	.01(7)	2.03(8)	.71(8)

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	factor is exp(-	$B\lambda^{-2}\sin^2\theta$).	,	
ant an	x	у	Z	В
H(1)	.147(2)	.690(3)	.105(2)	2.3(5)
H(2)	.098(2)	.396(3)	002(2)	1.3(4)
H(3)	.278(2)	.232(4)	.013(3)	3.2(6)
H(4)	.480(2)	.361(3)	.153(2)	2,6(5)
H(5)	.377(2)	. 344 (4)	.360(2)	3.0(6)
H(6)	.483(3)	.521(4)	.358(3)	4.5(7)
H(7)	.316(2)	.728(3)	.309(2)	2.4(5)
H(8)	.301(2)	.564(3)	020(3)	2.5(5)
H(9)	. 390(2)	.698(3)	.121(2)	3.1(5)
H(10)	.343(3)	.093(4)	.290(3)	3.7(6)
H(11)	.248(2)	010(4)	.130(3)	3.6(6)
H(12)	.103(3)	.051(4)	.268(3)	4.7(7)
H(13)	.139(4)	.635(5)	.361(4)	6.7(10)

Table 2. Parameters for Hydrogen Atoms. The form of the temperature factor is $\exp(-B\lambda^{-2}\sin^2\theta)$.

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-9-

Table 3. Bond Lengths (A)

C(1)-C(2)	1.545(3)	C(1)-H(1)	1.06(2)
C(1)-C(6)	1.530(3)	C(2)-H(2)	1.05(2)
C(1)-C(7)	1.533(3)	C(3)-H(3)	1.08(3)
C(2)-C(3)	1.557(3)	C(4)-H(4)	1.11(2)
C(2)-C(9)	1.499(3)	С(5)-Н(5)	1.08(3)
C(3)-C(4)	1.545(3)	С(5)-Н(6)	1.07(3)
C(3)-C(8)	1.534(4)	С(6)-Н(7)	1.09(3)
C(4)-C(5)	1.521(4)	С(7)-Н(8)	1.13(3)
C(4)-C(7)	1.539(4)	С(7)-Н(9)	1.09(3)
C(5)-C(6)	1.544(3)	C(8)-H(10)	1.19(3)
0(1)-C(9)	1.246(3)	C(8)-H(11)	1.05(3)
O(2)-C(6)	1.420(3)	N(1)-H(12)	1.00(3)
N(1)-C(8)	1.462(3)	O(2)-H(13)	0.97(4)
N(1)-C(9)	1.336(3)		

Table 4.

Bond Angles (°)

C(2)-C(1)-C(6)	111.8(2)	C(1)-C(6)-C(5)	103.1(2)
C(2)-C(1)-C(7)	100.6(2)	C(1)-C(6)-O(2)	115.9(2)
C(6) - C(1) - C(7)	99.4(2)	C(5)-C(6)-O(2)	110.9(2)
C(1) - C(2) - C(3)	103.8(2)	C(1)-C(7)-C(4)	94.5(2)
C(1) - C(2) - C(9)	121.5(2)	C(3) - C(8) - N(1)	104.1(2)
C(3)-C(2)-C(9)	105.1(2)	C(2) - C(9) - N(1)	109.5(2)
C(2)-C(3)-C(4)	102.3(2)	C(2)-C(9)-O(1)	126.3(2)
C(2)-C(3)-C(8)	105.9(2)	O(1)-C(9)-N(1)	124.0(2)
C(4)-C(3)-C(8)	119.3(2)	C(8)-N(1)-C(9)	115.5(2)
C(3) - C(4) - C(5)	110.7(2)	C(6)-O(2)C(9)	86.3(1)
C(3)-C(4)-C(7)	99.8(2)	C(2)-C(9)0(2)	82.1(1)
C(5)-C(4)-C(7)	101.7(2)	0(1)-C(9)0(2)	91.6(2)
C(4) - C(5) - C(6)	103.7(2)	N(1)-C(9)0(2)	100.7(2)

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-11-

Table 5. Hydrogen Bonding

Bond (X-HY)	d(XY)	d(HY)	Х-Н-Ү
0(2)-H(13)0(1 ⁱ)	2.824(2) Å	1.87(4) Å	168(3)°
$N(1)-H(12)0(1^{ii})$	2.926(3)	1.95(3)	164(3)

Symmetry Code:

(i) -x, 1/2 + y, 1/2 - z

(ii) -x, -1/2 + y, 1/2 - z

Figure Captions

-12-

Fig. 1. Perspective view of a molecule with 50% probability thermal ellipsoids, drawn with Johnson's ORTEP. Size of hydrogen atoms is arbitrary.

Fig. 2. Stereoscopic view of molecular packing, viewed along the \underline{b} axis.





Fig. 1



XBL 762-471

Fig. 2

0000 470 4 7 0 4 3 4 3

OBSERVED STRUCTURE FACTORS, STANDARD DEVIATIONS, AND DIFFERENCES (ALL X10.0) C9 H13 N 02 F(0,0,0) = 3601

FOB AND FCA ARE THE OBSERVED AND CALCULATED STRUCTURE FACTORS. SG = ESTIMATED STANDARD DEVIATION OF FOB. DEL = /FOB/ - /FCA/. * INDICATES ZERO WEIGHTED DATA.

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3 4 5 6 ° 110 - 110 - 8 7 6 5 4 3 2 - 0 1	16936 2626 2637 211 186 209 2638 209 268 378 50 35	316 21127 165212 3960 10559	-5* 10* 5 10* -1 10* -3 -7* -12 -3 -3 1 9	1 2 3 4 5 6 7 8 9 10 11 -11 -11 -9 -8 -7	K + 226 2 577 1890 2 207 1428 1465 7 4 168 577 1428 1427 4 8 694 222 1222	373367945406344453	0 4 -28 5 142 -2 -22 -2 -23 -4 -23 -4 -00 0	$ \begin{array}{r} 8\\ 9\\ 10\\ -12\\ -11\\ -18\\ -9\\ -8\\ -7\\ -6\\ -5\\ -4\\ -3\\ -2\\ -1\\ 0\\ 1 \end{array} $	41 103 55 28 58 118 57 233 26 104 338 240	7 5 9 3 1 3 0 8 4 6 4 3 7 3 7 3 7 10 7	-48-135 -15144-222168-1955 -1955	-6	75 163 26 107 236 39 10 181 189 30 14 87 88 162 12 57 57 57 57 57 57 57 57 57 57 57 57 57	3534724766€054723 254725	44 -0 -7 -6 -7 -4 -3 -6 -6 -6 -6 -6 -6 -6 -6 -6 -5 -6 -6 -5 -6 -7 -6 -7 -7 -6 -7 -7 -7 -7 -7 -7 -7 -7 -7 -7	-110987654321012345	<pre>< +L= 51482 10554722150 1654722150 14392 2150644493</pre>	3738224565737966564 14	-8 -4* 9 -2 -6 -5** -6 -8 -7 22
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3 4 5 6 ° 110 9 8 7 6 5 4 3 2 1 0 1 2 3 4 	1693 26±67 2118 2092 138 2092 138 2052 288 2052 288 2052 288 2052 288 2052 288 2052 288 2052 288 2052 288 287 287 287 287 287 287 287 287 28	316 211227652239626619593206	-5^{+} -0^{+} 9^{+} -1^{+} 10^{+} -3^{-} -1^{-} -3^{-} 1^{-} -3^{-} -1^{-} -3^{-} -1^{-} -3^{-} -1^{-} -3^{-} -1^{-} -3^{-} -1^{-} -1^{-} -3^{-} -1^{-} -3^{-} -1^{-} -3^{-} -1^{-} -3^{-}	123456789011 -10987654	K + 226 4267790 21721267790 14685774 1468574 122726 1227261 106942226 10610 10600 10600 10600 10600 10600 10600 10600 10600 10600 10600 10600 10600 10600 10600 106000 106000 106000 106000 106000 10600000000000000000000000000000000000	373367945406344453444	04285514242 24221234 3400313	8910 -121-199-765-43-21 -119-87-65-43-21 -123-4 -123-4	41 1035 55 288 1896 7336 109 2964 1380 201 3401 201 47 47	759330846437373707654	-481354422221681955417 -1-2-1681955417	-6543210123456789 11098 -1098	75 163 26 107 2368 39 10 181 189 30 181 189 30 147 88 16 12 x 7 0 89 10 147 88 16 12 x 7 0 89 10 147 88 16 12 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 7 236 10 10 10 10 10 10 10 10 10 10 10 10 10	3534724766€054723 22547225269	44 -07 -77 -72 -32 -34 -34 -34 -34 -34 -34 -34 -34 -34 -34		<pre>L=614825472150644933=4</pre>	373824565737966564934 1934	-1 -4* 9 -2 -6 -52* -6 -8 -7 21 9 -10*
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This report was done with support from the United States Energy Research and Development Administration. Any conclusions or opinions expressed in this report represent solely those of the author(s) and not necessarily those of The Regents of the University of California, the Lawrence Berkeley Laboratory or the United States Energy Research and Development Administration. TECHNICAL INFORMATION DIVISION LAWRENCE BERKELEY LABORATORY UNIVERSITY OF CALIFORNIA BERKELEY, CALIFORNIA 94720