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THE SYNTHESIS OF ALIPHATIC DIMERIC N-ISOPROPYL HYDROXAMIC ACIDS AND THE CRYSTAL AND MOLECULAR STRUCTURE OF N,N' -DIHYDROXY-N,N' - DIISOPROPYLHEXANEDIAMIDE: A HYDROXAMIC ACID IN THE TRANS CONFORMATION

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William L. Smith and Kenneth N. Raymond

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The Synthesis of Aliphatic Dimeric N-Isopropyl Hydroxamic
Acids and the Crystal and Molecular Structure of N,N'-
Dihydroxy-N,N'-diisopropylhexanediamide: A Hydroxamic
Acid in the trans Conformation

By

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Running Title: A Hydroxamic Acid in the trans Conformation

Abstract

A series of dimeric N-isopropylhydroxamic acids, separated by $(\text{CH}_2)_n$ units ($n = 4, 6, 8$ and 10), have been synthesized from the acid chlorides and N-isopropylhydroxylamine. The structure of the adipoyl derivative ($n = 4$) has been determined from single-crystal x-ray diffraction data collected by counter methods. In contrast to all previous hydroxamic acids that have been structurally characterized, the planar hydroxamate groups are trans, with $\text{C}(\text{O})\text{-N}$ and $\text{C}=\text{O}$ distances similar to those found in amides. Strong intermolecular hydrogen bonds between the hydroxyl and carbonyl groups hold the molecules in pleated planes perpendicular to the b-axis and compensate for the loss of the intramolecular hydrogen bond of the cis conformation. The intermolecular hydrogen bonds appear to be stronger than the intramolecular hydrogen bonds of the cis conformation, but the latter are entropically favored in solution. The molecules are located on a center of inversion in the space group Pbca with $a = 9.3859(3)$, $b = 18.7156(2)$ and $c = 8.4180(2)$ Å. Full-matrix least-squares refinement on 1217 independent data with $F_o^2 > 3\sigma(F_o^2)$ and all atoms (including hydrogen) anisotropic led to an R factor of 3.8 percent.

Introduction

As part of the development of actinide-specific chelating agents,¹ a series of dimeric N-isopropylhydroxamic acids, which are connected by even-numbered carbon chains of four to ten, have been synthesized from the acid chlorides and N-isopropylhydroxylamine. Unsubstituted dimeric hydroxamic acids separated by up to eight carbon atoms previously have been prepared from the esters and basic hydroxylamine.²⁻⁴ Acid chlorides are more reactive but require a N-substituted hydroxylamine to prevent the formation of cyclic N-diacylhydroxylamines. A series of dimeric N-phenylhydroxamic acids has been prepared from the acid chlorides,⁵ but the N-phenyl group may impart undesirable redox properties to the ligand as observed in the oxidation of U(IV) to the uranyl ion by N-phenylbenzohydroxamic acid.⁶

Because of the anomalously high melting point and extremely low carbonyl stretching frequency, the structure of N,N'-diisopropyladipohydroxamic acid, compound **1**, was of interest to us. Although no other structures of N-substituted hydroxamic acids are known, the planar trans conformation of the hydroxamic acid group found in **1** is seen only in N-hydroxyurea,⁷ whose geometry is influenced by hydrogen bonding of the amino group. The structures of aceto-,⁸ benzo-⁹ and salicylo-¹⁰ hydroxamic acids and the IR spectrum of formohydroxamic acid¹¹ show a planar cis conformation. This conformation is retained in the structures determined for a series of four O-benzoylbenzohydroxamic acids.¹² Thus all solid-state structures of

hydroxamic acids are cis, although the trans isomer is expected to be the more stable in the absence of intramolecular hydrogen bonding. In fact, reports of such isomerization have appeared for the related hydroxamic acids $\left[\begin{array}{c} R_2-O \quad OH \\ | \quad | \\ R_1-C=N \end{array} \right]$, for $R_1 = R_2 = CH_3$ ¹³ and $R_1 = Ph, R_2 = Et$ ¹⁴, where no intramolecular hydrogen bonding is possible. With the expectation that perhaps the anomalous properties of compound 1 could be explained by such isomerization, the structure analysis reported here was begun.

Experimental

Infrared spectra were obtained on a Perkin-Elmer 283 or a Beckman IR-9 spectrophotometer. ¹H-NMR spectra were recorded on a Varian EM 390 spectrophotometer. Melting points were determined in open capillaries with a Buchi apparatus and are uncorrected. Microanalyses were performed by the Analytical Services, Chemistry Department, University of California, Berkeley.

The dicarboxylic acids, which were used as obtained, were converted into their acid chlorides by refluxing thionyl chloride followed by fractional vacuum distillation. N-isopropylhydroxylamine was prepared by the reduction of 2-nitropropane with zinc dust.¹⁵

N,N'-Dihydroxy-N,N'-diisopropylhexanediamide, (1). A suspension of 0.22 mol of isopropylhydroxylamine and 0.3 mol of NaHCO_3 in 250 ml ether and 25 ml of water was cooled to 0°C and 0.1 mol of adipoylchloride, diluted with an equal volume of ether, was added dropwise with stirring. After stirring overnight, the reaction mixture was filtered and the precipitate was crystallized from methanol. The resulting solid was washed with 25 ml of 0.01 M HCl followed by 3 x 25 ml of water. These washings exhibited a positive FeCl_3 test indicating the presence of the monohydroxamic acid, probably formed by the hydrolysis of an eight-membered cyclic N,O-diacylhydroxylamine (see Ref. 2). Compound 1 was obtained as a white powder after recrystallization from methanol.

Anal. Calcd for $\text{C}_{12}\text{H}_{24}\text{N}_2\text{O}_4$: C, 55.36; H, 9.29; N, 10.76
Found: C, 55.26; H, 9.21; N, 10.67

N,N'-Dihydroxy-N,N'-diisopropyloctane-, -decane- and -dodecanediamide, (2 - 4). Using the above procedure, these compounds were obtained as white plates after recrystallizing the first precipitate twice from methanol.

Anal. Calcd for $\text{C}_{14}\text{H}_{28}\text{N}_2\text{O}_4$: C, 58.31; H, 9.79; N, 9.71.
Found: C, 58.35; H, 9.78; N, 9.68.

Anal. Calcd for $\text{C}_{16}\text{H}_{32}\text{N}_2\text{O}_4$: C, 60.73; H, 10.19; N, 8.85.
Found: C, 63.02; H, 9.98; N, 8.83.

Anal. Calcd for $\text{C}_{18}\text{H}_{36}\text{N}_2\text{O}_4$: C, 62.76; H, 10.53; N, 8.13.
Found: C, 63.02; H, 10.17; N, 8.08.

The yields and physicochemical properties of the above hydroxamic acids are summarized in Table I.

X-ray Diffraction Studies. Colorless rhombic needles of N,N'-diisopropyladipohydroxamic acid, $\mathbf{1}$, were obtained by the slow evaporation of a methanol solution. A piece of crystal of dimensions 0.18 x 0.23 x 0.48 mm was cleaved from a long needle using a scalpel and was sealed in a thin-walled glass capillary. Precession photographs and diffraction data showed orthorhombic symmetry and the following systematic absences: $0k\ell$, $k \neq 2n$; $h0\ell$, $\ell \neq 2n$; $hk0$, $h \neq 2n$; consistent with the space group Pbca. Graphite-monochromatized Cu K_{α} radiation was used for the precise measurement of the cell constants and for data collection on a Nonius CAD-4 automated four-circle diffractometer.¹⁶ Cell parameters obtained by a least-squares fit to the measured coordinates of 23 reflections ranging in 2θ from 58 to 96° are listed in Table II.

The intensities of two equivalent forms, h , k , $\pm\ell$, of all reflections with $2\theta \leq 150^\circ$ were measured using a $\theta - 2\theta$ scan and processed as previously described.^{17,18} An absorption correction¹⁹ gave a maximum range of 1.12 to 1.25 for the correction factor and so was not applied. Equivalent forms were averaged and, to avoid overweighting the strong reflections, a parameter p equal to 0.02 (the R factor for averaging F^2) was introduced as previously described.²⁰

Structure Determination and Refinement. Phases were determined by MULTAN²¹ using the 80 largest E values. All nonhydrogen atoms were located from the E-map with the highest figure of merit among the eight phase sets generated by three special reflections. After least-squares refinement of these atoms, the hydrogen atoms were found in a difference Fourier.

Final full-matrix, least-squares refinement²² on 1217 reflections with $F_o^2 > 3\sigma(F_o^2)$ and 191 variables led to convergence with $R = 0.038$, $R_w = 0.049$ and an error in an observation of unit weight of 2.48.²³ All atoms were given anisotropic temperature factors and scattering factors for the neutral atoms.²⁴ The unusual crystal perfection gave rise to severe secondary extinction, and a correction was applied.²⁵ A final difference Fourier showed maximum residual peaks of $\pm 0.13 \text{ e } \text{\AA}^{-3}$. Refinement using isotropic hydrogen temperature factors led to convergence with $R = 0.044$, $R_w = 0.061$ and an error in an observation of unit weight of 2.96; justifying the use of anisotropic hydrogen temperature factors at the 99.9% level.²⁶

Description of the Structure

The unusual trans conformation of the hydroxamic acid group is shown in Figure 1. The carbon, nitrogen and oxygen atoms of the hydroxamic acid group have an average deviation of $0.014 \text{ \AA}$ from their least-squares plane. The O-H bond

and C5-C6 vector of the isopropyl group are almost perpendicular to this plane. The substituent carbons, C2 and C4, are located 0.1 and 0.2 Å from opposite sides of the plane. Since the molecule is situated on a crystallographic inversion center, the carbon chain connecting the hydroxamic acid groups is planar. This plane forms a dihedral angle of 70.3(2)° with the hydroxamic acid plane.

Bond lengths and angles are shown in Figure 2. The N-C(=O), C=O and C-C(=O) bond lengths are in good agreement with those found in amides [1.333(5), 1.235(5) and 1.506(5) Å]²⁷ and in acetohydroxamic acid [1.321(6), 1.234(6) and 1.498(6) Å].⁸ The N-C(=O) bond is slightly shorter, and the C=O distance is longer, in arylhydroxamic acids,^{9,10} possibly as a result of conjugation. The N-O bond length, 1.396(2) Å, is identical to that found in N-hydroxyurea⁷ and acetohydroxamic acid.⁸ The C-C bonds in the carbon chain are shorter than normal, [1.537(5) Å],²⁷ but are similar to those found in the carbon chains of the aliphatic diacids²⁸ and their diamides.^{29,30} Although the N-C4 bond is shorter than normally found in amines, [1.472(5) Å],²⁷ it agrees with the C-N distance of 1.457(2) Å found in [16]aneN₄.³¹ Because of thermal motion the isopropyl C-C bond lengths are shortened. Assuming that the methyl carbons ride on the methyldyne carbon, corrected bond distances³² of 1.537(2) and 1.535(2) Å are obtained.

As is normally observed in x-ray diffraction studies, the average³³ C-H distance of 0.99(5) Å is shorter than that found by neutron diffraction³⁴ due to the polarization of electron density of the H atom toward the C-H bond. However, the O-H distance of 0.93(3) Å is in good agreement with the neutron diffraction values of 0.95(2) Å found in hydroxylamine hydrochloride and 0.94(3) Å found in pentaerythritol.³⁴ This may be a result of a strong hydrogen bond which prevents the polarization of charge toward the oxygen. The average³³ H-C-H and C-C-H angles of 109(2)° and 109(3)° are as expected for tetrahedral carbon atoms.

As seen in Figure 3, each molecule of $\mathbf{I}$ is hydrogen bonded to four others forming pleated planes perpendicular to the b axis. The short intermolecular O1 - O2 distance of 2.617(2) Å and the nearly linear O1-H-O2 angle of 175(2)°, indicates a strong hydrogen bond.³⁴ This is manifested by the high melting point and low carbonyl stretch of $\mathbf{I}$ as compared to the other members of the series. In addition to a band at 3130 cm⁻¹, compound $\mathbf{I}$ also exhibits several hydroxyl bands between 2500 and 2900 cm⁻¹, which are characteristics of strongly hydrogen bonded carboxylic acids,³⁵ and which are not found in the IR spectra of its congeners.

Discussion

The planarity of the hydroxamic acid group and its bond lengths indicate partial N-C(O) double bond character as

found in amides. The existence of stable conformations of the hydroxamic acid group and hindered rotation about the N-C(O) bond is supported by the 15(1) kcal mol⁻¹ barrier to rotation measured in N,O-diacetylmethylhydroxylamine by NMR.³⁶ This barrier to rotation could be increased by hydrogen bonding of the hydroxyl group in hydroxamic acids. That the carbonyl stretching frequencies of $\mathbf{2} - \mathbf{4}$ are 30 cm⁻¹ higher than that found in $\mathbf{1}$ indicate that $\mathbf{2} - \mathbf{4}$ assume the cis conformation normally found for hydroxamic acids in the solid state.⁸⁻¹⁰ As a result of steric interactions, the cis-conformation forms weaker intermolecular hydrogen bonds thus exhibiting a higher carbonyl stretching frequency than found in the trans conformation.

As seen in Table I, the carbonyl stretching frequencies shift to higher frequencies upon dissolution of the hydroxamic acids in methanol. The identical methanolic frequencies show that $\mathbf{1} - \mathbf{4}$ exhibit the same structure in solution. The carbonyl shift of 53 cm⁻¹ seen for $\mathbf{1}$ is more than double the shift seen for its congeners, which are similar to those reported to occur between the solid state and carbon tetrachloride solution for several other hydroxamic acids.³⁷ With the exception of $\mathbf{1}$, all known solid-state structures of hydroxamic acids show a cis-planar conformation,⁸⁻¹⁰ supporting the assigned cis conformation for $\mathbf{2} - \mathbf{4}$. In all cases, the hydroxamic acid groups are intermolecularly hydrogen-bonded in the solid state.⁸⁻¹⁰ Upon dissolution,

weaker intramolecular hydrogen-bonds (which are entropically favored over the dimeric or polymeric intermolecularly bonded species) or hydrogen-bonds to solvent molecules would account for the increased carbonyl stretching frequency. Strong evidence for the intramolecular hydrogen-bond, which requires a cis conformation, has been presented by the additional 44 cm^{-1} increase of the carbonyl stretching frequency upon methylation of the hydroxyl group.³⁷ The cis conformation is also supported by dipole measurements of benzene and dioxane solutions of hydroxamic acids.³⁸

In summary, with the exception of λ , the cis conformation is retained in the solid state.⁸⁻¹⁰ The stabilization of the trans conformer of λ in the solid state is ascribed to the very strong intermolecular hydrogen bonding present in this particular structure - which also gives rise to the unusually ideal crystallographic properties of the compound. The infrared spectra of these compounds are a reliable diagnostic test of their conformation.

Acknowledgment

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Supplementary Material Available

A listing of the observed and calculated structure factors for λ . Ordering information is available on any current masthead page.

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16. Programs used by the PDP 8/E were written by Enraf-Nonius Corporation.
17. A maximum of 90 s were used to scan a reflection through a variable scan angle of $0.6 + 0.14 \tan \theta$, where θ was calculated using $(\lambda_{K\alpha_1} + \lambda_{K\alpha_2})/2$. The background was estimated by extending the scan 25% on each side. An aperture with a variable width of $2.0 + \tan \theta$ mm and a height of 4 mm was located 173 mm from the crystal. An attenuator was employed when the intensity exceeded 5×10^4 cps. The orientation was checked every 250 reflections and recalculated if any of the setting angles changed more than 0.1%. Three intensity standards were measured every 7200 s.
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21. Machine computations were performed on a CDC 7600 computer using, in addition to locally written programs: Zalkin's FORDAP Fourier program; Ibers' NUCLS group least-squares version of the Busing-Levy ORFLS; ORFFE, a function and error program by Busing and Levy; Johnson's ORTEP, a thermal ellipsoid plot program; MULTAN, a program series for direct-methods phase determination by Germain, Main and Woolfson.
22. The function minimized in all refinements is: $\sum w(|F_o| - |F_c|)^2$ where $w = 4 F_o^2 / \sigma^2(F_o^2)$.
23. Definitions of indicators are: $R = \sum ||F_o| - |F_c|| / \sum |F_o|$; $R_w = [\sum w(|F_o| - |F_c|)^2 / \sum w F_o^2]^{1/2}$ and the error in an observation of unit weight = $[\sum w(|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$, where N_o is the number of observations and N_v is the number of refined variables.
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Table I. Synthesis and characterization of aliphatic dimeric N-isopropylhydroxamic acids.

n	Mw	Mp, °C	IR, cm ⁻¹		¹ H NMR (d ⁶ -Me ₂ CO-Me ₄ Si) δ, ppm (J, Hz)						% Yield
			KBr pellet OH	MeOH soln C=O	(CH ₃) ₂ C<, 12H doublet	-(CH ₂) _{n-2} multiplet	-C(O)CH ₂ -, 4H triplet	≥CH, 2H septet	-OH, ^a 2H singlet		
4	260.3	162-3	3130 ^b	1570	1623	1.07(7)	1.50, 4H	2.35(7)	4.58(7)	9.53	52
6	288.4	135-6	3160	1600	1622	1.07(7)	1.44, 8H	2.30(7)	4.49(7)	9.10	43
8	316.4	137-8	3165	1600	1621	1.08(7)	1.40, 12H	2.31(7)	4.54(7)	9.10	74
10	344.5	138-9	3165	1600	1622	1.05(7)	1.36, 16H	2.30(7)	4.48(7)	9.10	76

^aConcentration dependent, disappears with D₂O.

^bSeveral bands between 2500-2900 cm⁻¹.

Table II. Crystal data for N,N'-dihydroxy-N,N'-diisopropyl-hexanediamide

Molecular formula	$C_{12}H_{24}N_2O_4$
Molecular weight	260.33
Space group	Pbca (D_{2h}^{15})
Cell constants ^a	
a	9.3859(3) Å
b	18.7156(5) Å
c	8.4180(2) Å
Volume	1478.73(7) Å ³
Formula units/cell	4
Calculated density	1.169 g cm ⁻³
Observed density ^b	1.16 g cm ⁻³
Linear absorption coefficient, $\mu_{CuK\alpha}$	6.85 cm ⁻¹

^a 23.8 ± .5°C; CuK α_1 radiation, λ = 1.540562 Å.

^b Measured by flotation in CCl₄-pentane.

Table III. Positional and anisotropic thermal parameters^a ($\times 10^3$) for compound 1.

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
O ₁	.9100(1)	.91720(6)	.9564(1)	12.3(1)	3.89(4)	15.8(2)	1.81(5)	-1.3(1)	-2.35(6)
O ₂	.2163(1)	.87200(6)	.7433(1)	15.3(2)	4.76(4)	19.6(2)	2.06(6)	5.7(1)	3.58(7)
N	.0277(1)	.87878(6)	.9015(1)	10.8(1)	3.01(3)	12.4(1)	1.10(5)	.1(1)	.34(5)
C ₁	.0185(2)	.96503(8)	.5412(2)	18.6(2)	2.87(4)	12.4(2)	.86(9)	-2.3(2)	.71(7)
C ₂	.0685(3)	.97479(9)	.7092(2)	23.3(3)	2.94(5)	13.0(2)	.92(10)	-2.7(2)	.86(7)
C ₃	.1089(1)	.90534(7)	.7866(1)	13.8(2)	3.02(4)	11.5(2)	.58(7)	-1.0(1)	.81(6)
C ₄	.0655(2)	.81659(8)	.9976(2)	14.0(2)	2.78(4)	12.5(2)	.46(7)	1.3(1)	.68(6)
C ₅	.9416(3)	.7658(1)	.0125(3)	22.3(3)	3.95(7)	20.7(3)	-2.83(13)	3.0(3)	-1.14(12)
C ₆	.1236(3)	.8397(1)	.1569(2)	22.3(3)	4.17(6)	15.7(3)	.00(13)	-5.4(2)	1.31(10)
HO ₁	.837(3)	.902(1)	.890(3)	26.(4)	7.0(11)	27.(5)	2.0(18)	9.(3)	-8.1(19)
H _{1A}	.093(3)	.938(1)	.476(2)	30.(4)	5.8(9)	15.(3)	8.8(17)	4.(3)	1.1(13)
H _{1B}	.931(3)	.933(1)	.543(3)	31.(4)	3.4(7)	46.(6)	-5.5(16)	-20.(5)	6.9(18)
H _{2A}	.004(3)	.999(1)	.769(2)	43.(6)	4.2(8)	11.(3)	9.3(20)	-6.(3)	1.1(12)
H _{2B}	.168(4)	.005(1)	.700(3)	42.(6)	3.9(8)	53.(8)	-4.3(22)	-13.(6)	11.9(21)
H ₄	.140(2)	.795(1)	.939(2)	20.(3)	3.9(6)	12.(3)	.8(12)	-4.(2)	2.6(10)
H _{5A}	.901(3)	.753(2)	.906(3)	42.(6)	8.8(13)	18.(4)	-12.8(23)	-5.(4)	.4(18)
H _{5B}	.969(3)	.721(1)	.067(3)	35.(5)	4.8(9)	35.(6)	-2.7(20)	-2.(4)	5.5(19)
H _{5C}	.867(3)	.791(1)	.076(3)	25.(4)	4.5(8)	37.(5)	-1.5(15)	8.(4)	-5.1(18)
H _{6A}	.204(3)	.873(1)	.141(3)	32.(5)	5.1(8)	29.(5)	-6.5(19)	-7.(4)	4.9(16)
H _{6B}	.152(2)	.795(1)	.215(2)	29.(4)	5.6(8)	22.(4)	2.3(16)	0.(3)	3.7(15)
H _{6C}	.045(2)	.861(1)	.212(2)	22.(4)	4.8(8)	21.(4)	-.1(16)	-1.(3)	.1(14)

^aThe form of the anisotropic temperature factor is $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$.

Figure Captions

Figure 1. Stereopair of N,N'-diisopropyladipohydroxamic acid with nonhydrogen thermal ellipsoids at the 30% probability level.

Figure 2. Bond lengths and angles in N,N'-diisopropyladipohydroxamic acid. Unless specified in parentheses, esd's are 0.002 Å and 0.2°.

Figure 3. Packing diagram of N,N'-diisopropyladipohydroxamic acid viewed down the a axis with the b axis vertical. The O-H hydrogen atom ellipsoid is solid black to distinguish it from the other atoms.

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