

C15D	0.483 (2)	0.6783 (8)	0.0751 (8)	0.060 (7)
N16D	0.6352 (16)	0.5822 (8)	0.0528 (8)	0.049 (4)
C17D	0.596 (2)	0.5385 (10)	0.0957 (9)	0.059 (6)
C18D	0.6724 (19)	0.4764 (8)	0.1190 (8)	0.058 (6)
C19D	0.627 (2)	0.4325 (9)	0.1687 (9)	0.063 (6)
C20D	0.696 (2)	0.3729 (9)	0.1914 (10)	0.070 (6)
C21D	0.820 (2)	0.3558 (10)	0.1631 (10)	0.080 (7)
C22D	0.8655 (19)	0.3969 (9)	0.1159 (9)	0.055 (5)
C23D	0.787 (2)	0.4565 (8)	0.0904 (9)	0.057 (6)
O2D	0.8395 (14)	0.4976 (8)	0.0425 (6)	0.072 (4)

Table 2. Dihedral angles ( $^{\circ}$ ) between ring planes (*e.s.d.* for each value  $1^{\circ}$ )

Ring	A1	A2	A3	B1	B2	B3	C1	C2	C3	D1	D2
A2	6										
A3	18	22									
B1	58	60	64								
B2	61	63	68	5							
B3	56	55	71	32	30						
C1	1	7	17	56	60	56					
C2	3	3	21	58	61	54	4				
C3	39	35	57	55	55	30	40	36			
D1	59	61	65	1	4	32	58	59	55		
D2	69	63	68	4	0	30	60	61	55	4	
D3	63	67	61	22	25	54	61	64	73	22	25

The crystals diffracted very weakly, accounting for the poor final *R* value. However, the final geometry has been unequivocally determined. Systematic absences indicated that the space group was either  $P2_1/m$  or  $P2_1$ . Initial attempts using the former having failed, the structure was solved using *TREF* (Sheldrick, 1990) and refined in  $P2_1$ . H atoms were added at calculated positions and refined using a riding model. Methyl groups were treated as rigid bodies, free to rotate about the C—C bond. Anisotropic temperature factors were used for all non-H atoms; H atoms were given isotropic temperature factors,  $U = 0.08 \text{ \AA}^2$ . Weak restraints were applied to equalize the C—N distances and the dimensions of corresponding rings. Refinement of the absolute structure parameter did not give a statistically significant result.

The crystals show pseudo-centres of inversion at approximately  $\frac{1}{8}$ ,  $\frac{1}{2}$ ,  $\frac{1}{4}$  and  $\frac{3}{8}$ ,  $\frac{1}{2}$ ,  $\frac{1}{4}$  which are identifiable on Fig. 2; these centres are only approximate, as is demonstrated by the variations in dihedral angles (Table 2). Also, because they lie neither on the  $2_1$  axis nor at  $\frac{1}{4}$  from it, they do not generate either  $2_1/m$  or  $2_1/c$  symmetry.

Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993).

We wish to acknowledge the use of the EPSRC's Chemical Database Service at Daresbury.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SK1003). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## *O*-Acetyl-*N*-(*o*-hydroxybenzyl)-*N*-isopropylhydroxylamine

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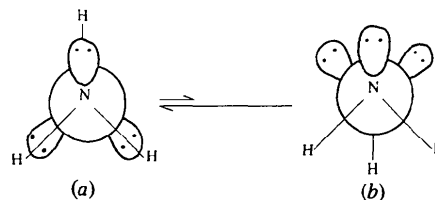
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## Abstract

Although the solution chemistry of hydroxylamine derivatives is well documented, literature on the conformational aspects in the solid state is rather scarce. The structure of the title compound,  $C_{12}H_{17}NO_3$ , revealed an N4—O1—C15—O2 torsion angle of  $-6.0(7)^{\circ}$  indicating a *cis* and planar disposition of N—O and C—O bonds. This is the most stable and only observable conformer, as opposed to the *trans* and planar arrangement.

## Comment

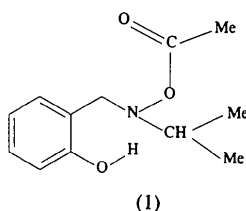
Owing to their unique conformational properties, hydroxylamine derivatives are very important in the field of conformational analysis (Riddle, 1981). The discovery that, unlike other simple organic species such as ethane, methylamine and methanol, hydroxylamine has the lone pair on nitrogen and the hydroxyl bond formally eclipsed was a great surprise for chemists. Thus, not only is (a) the only observable conformer, it is at the same time highly reluctant to flip over the very high energy barriers (*ca*  $50 \text{ kJ mol}^{-1}$ ), either through nitrogen inversion or N—O bond rotation (Riddle, 1981), in order to attain the less stable conformer shown as (b).



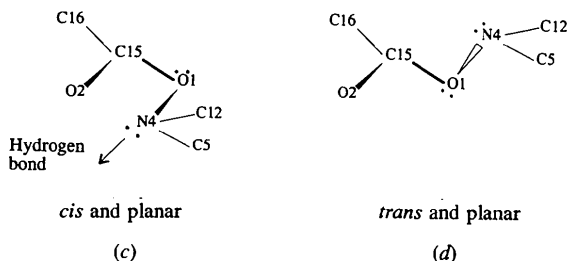
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These unique properties have led to the use of hydroxylamine derivatives in investigating several interesting and otherwise intractable conformational problems (Riddle, 1981; Ali & Wazeer, 1992). Even though the solution chemistry of hydroxylamine derivatives is well documented, literature on the conformational aspects in the solid state is rather scarce (Bushfield *et al.*, 1986).

Our objective was to study the effects of sterically crowded substituents and intramolecular hydrogen bonding on the conformational effects around the N—O bond in the title compound, (1). It was also our intention to clarify the effects the electron-withdrawing conjugative CH<sub>3</sub>C=O group (attached to the oxygen) and hydrogen bonding have on the high inversion/rotation barrier (61 kJ mol<sup>-1</sup>; Ali, 1995) observed in the title compound. With this in mind the crystal structure of (1) was determined.



The N4—O1—C15—O2 torsion angle is  $-6.0(7)^\circ$  indicating a *cis* and planar disposition of N4—O1 with respect to the O2—C15 bonds allowing intramolecular hydrogen bonding as shown in structure (c). This disposition is opposed to the other possibility, a *trans* and planar arrangement (d) in which no hydrogen bonding is possible.



As a consequence of the *cis* and planar arrangement, O2 is in close proximity to N4 at a distance of 2.574(4) Å. This arrangement also leads to intramolecular N4...H3—O3 hydrogen-bonding interactions. The C15—O1—N4—C5 and C15—O1—N4—C12 torsion angles are 106.8(4) and  $-128.9(4)^\circ$ , respectively, showing normal tetrahedral geometry for N4. The C6—C11—O3—H3 torsion angle is 0.3(6)° indicating that the O3—H3 hydroxyl group and the phenyl ring are coplanar. The N4...O3 hydrogen-bonding distance is 2.756(4) Å which is the normally observed distance for an R(N...O) type hydrogen bond. The O3...O2 distance is 3.006(4) Å.

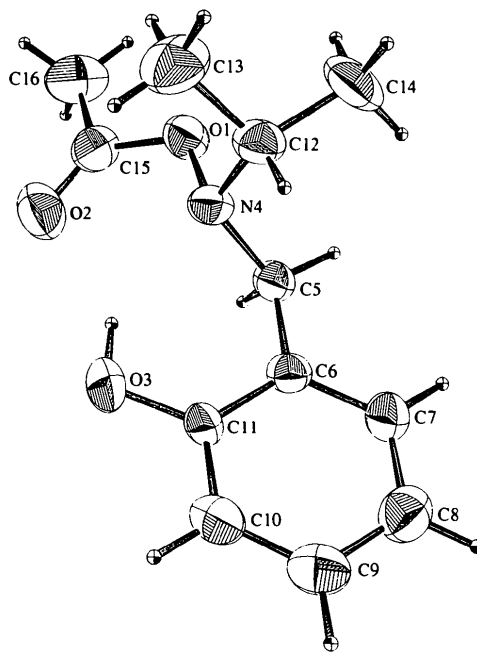


Fig. 1. ORTEP (Johnson, 1965) view of the molecule showing the atom-numbering scheme and the *cis* and planar disposition of N—O and C—O bonds in the molecule. Displacement ellipsoids are drawn at the 35% probability level.

## Experimental

The compound was prepared by the procedure reported earlier (Ali & Wazeer, 1992). Crystals for X-ray studies were obtained by slow evaporation of the solution of the crude reaction product in ether/hexane mixture. Crystals (m.p. 226–227 K) of the title compound were obtained after about an hour.

### Crystal data

C<sub>12</sub>H<sub>17</sub>NO<sub>3</sub>  
*M<sub>r</sub>* = 223.27  
 Triclinic  
*P* $\bar{1}$   
*a* = 6.930(4) Å  
*b* = 9.597(6) Å  
*c* = 10.169(7) Å  
 $\alpha$  = 80.10(3)°  
 $\beta$  = 89.94(6)°  
 $\gamma$  = 73.22(2)°  
*V* = 637.0 Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 1.16 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

### Data collection

Enraf–Nonius CAD-4  
 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction:  
 none  
 1350 measured reflections  
 1145 independent reflections  
 854 observed reflections  
 [*F* > 3.0σ(*F*)]

Mo *K*α radiation  
 $\lambda$  = 0.71073 Å  
 Cell parameters from 25  
 reflections  
 $\theta$  = 2.37–31.15°  
 $\mu$  = 0.08 mm<sup>-1</sup>  
*T* = 296 K  
 Rectangular  
 0.64 × 0.64 × 0.41 mm  
 Colourless

*R*<sub>int</sub> = 0.041  
 $\theta_{\max}$  = 31.65°  
*h* = -6 → 0  
*k* = -9 → 8  
*l* = -9 → 9  
 3 standard reflections  
 monitored every 97  
 reflections  
 intensity decay: none

## Refinement

Refinement on  $F^2$  $R(F) = 0.038$  $wR(F^2) = 0.045$  $S = 1.05$ 

854 reflections

145 parameters

H-atom parameters not refined

 $w = 4F_o^2/\sigma^2(F_o^2)$  $(\Delta/\sigma)_{\max} = 0.03$  $\Delta\rho_{\max} = 0.30 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$ 

Extinction correction: none

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C) and Cromer & Waber (1974)Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$B_{\text{eq}} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$B_{\text{eq}}$
O1	0.0771 (5)	0.7336 (4)	0.6421 (4)	5.6 (1)
O2	-0.1659 (5)	0.6273 (4)	0.6164 (4)	8.1 (1)
O3	0.1017 (5)	0.3274 (4)	0.7436 (4)	6.5 (1)
N4	0.1837 (6)	0.5943 (4)	0.7247 (4)	4.8 (1)
C5	0.3430 (7)	0.5178 (5)	0.6458 (5)	4.8 (1)
C6	0.4302 (7)	0.3612 (5)	0.7135 (5)	4.4 (1)
C7	0.6375 (7)	0.2981 (6)	0.7311 (6)	5.3 (1)
C8	0.7174 (8)	0.1511 (7)	0.7901 (6)	6.6 (2)
C9	0.5916 (8)	0.0667 (6)	0.8344 (6)	6.2 (2)
C10	0.3881 (8)	0.1302 (6)	0.8177 (6)	6.1 (2)
C11	0.3051 (6)	0.2728 (5)	0.7589 (5)	4.2 (1)
C12	0.2545 (8)	0.6280 (6)	0.8500 (6)	5.8 (2)
C13	0.070 (1)	0.6945 (8)	0.9249 (7)	9.2 (2)
C14	0.3934 (9)	0.7248 (7)	0.8298 (7)	8.8 (2)
C15	-0.1060 (8)	0.7334 (6)	0.5986 (6)	6.2 (2)
C16	-0.216 (1)	0.8836 (8)	0.5188 (7)	9.0 (2)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O1—N4	1.443 (3)	C6—C11	1.405 (7)
O1—C15	1.345 (7)	C7—C8	1.384 (7)
O2—C15	1.193 (8)	C10—C9	1.363 (7)
O3—C11	1.354 (5)	C10—C11	1.351 (6)
N4—C5	1.461 (6)	C8—C9	1.378 (9)
N4—C12	1.484 (8)	C15—C16	1.511 (8)
C5—C6	1.489 (6)	C12—C13	1.521 (8)
C6—C7	1.387 (6)	C12—C14	1.511 (9)
N4—O1—C15	111.7 (4)	C9—C10—C11	122.4 (5)
O1—N4—C5	107.1 (3)	O3—C11—C6	121.2 (4)
O1—N4—C12	107.3 (4)	O3—C11—C10	119.0 (5)
C5—N4—C12	115.2 (4)	C6—C11—C10	119.8 (4)
N4—C5—C6	109.7 (4)	N4—C12—C13	108.1 (5)
C5—C6—C7	120.8 (5)	N4—C12—C14	114.4 (5)
C5—C6—C11	121.0 (4)	C13—C12—C14	111.9 (5)
C7—C6—C11	118.2 (4)	O1—C15—O2	124.1 (4)
C6—C7—C8	120.4 (5)	O1—C15—C16	109.9 (5)
C7—C8—C9	120.3 (5)	O2—C15—C16	126.0 (5)
C8—C9—C10	118.8 (5)		

Table 3. Contact distances ( $\text{\AA}$ )

O2...N4	2.574 (4)	O2...O3	3.006 (4)
O3...N4	2.756 (5)		

A small colourless crystal was mounted on a glass fibre in a random orientation for data collection. A linear decay correction was applied but no absorption correction was made. The structure was solved using direct methods and refined by full-matrix least-squares methods. The weights were further modified using a sharpening factor of 20.0 (Dunitz & Seiler, 1973). Anomalous dispersion effects were included in  $F_c$  (Ibers & Hamilton, 1964); the values for  $\Delta f'$  and  $\Delta f''$  were those of Cromer (1974). The standard deviation of an observation of unit weight was 1.05. H atoms were placed in idealized positions (C—H = 0.96  $\text{\AA}$ ) with isotropic displacement parameters,  $U = 0.05 \text{\AA}^2$ .

All computations were carried out on a Compaq-PC using a combination of the PC-DOS versions of *SHELXS86* (Sheldrick, 1990) and *SDP* software (Frenz, 1988, 1991).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1977). Cell refinement: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SDP* (Frenz, 1988, 1991). Molecular graphics: *ORTEP* (Johnson, 1965).

We are thankful to KFUPM for supporting this work. Thanks are also due to Professor Sk. A. Ali for supplying the crystals and useful discussions.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1059). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 3-Hydroxyanthranilic Acid Hydrochloride

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## Abstract

In the structure of the title compound, 2-carboxy-6-hydroxyanilinium chloride,  $\text{C}_7\text{H}_8\text{NO}_3^+\text{Cl}^-$ , the amino group is protonated and the plane of the carboxyl group