

C38	0.9101 (5)	0.2589 (6)	0.9588 (4)	0.042 (3)
C39	0.5014 (5)	0.3797 (5)	0.8780 (4)	0.049 (3)
C40	0.6399 (6)	0.1076 (6)	0.7736 (4)	0.078 (4)
C41	1.0626 (5)	0.3851 (6)	1.0595 (4)	0.067 (3)
C42	1.1410 (5)	0.0626 (6)	1.1442 (5)	0.078 (4)

Table 2. Selected geometric parameters (Å, °)

O1—C14	1.201 (10)	N3—C15	1.377 (9)
O2—C15	1.219 (10)	N3—C16	1.471 (9)
O3—C25	1.219 (9)	N4—C25	1.352 (10)
O4—C24	1.223 (10)	N4—C26	1.465 (8)
N1—C13	1.459 (9)	N4—C41	1.466 (10)
N1—C14	1.362 (10)	N5—C24	1.392 (9)
N1—C39	1.464 (9)	N5—C25	1.396 (10)
N2—C14	1.400 (11)	N5—C42	1.463 (10)
N2—C15	1.386 (9)	N6—C23	1.483 (8)
N2—C40	1.468 (10)	N6—C24	1.359 (10)
N3—C13	1.481 (8)	N6—C26	1.466 (9)
C13—N1—C14	120.5 (6)	N3—C13—C12	111.6 (5)
C13—N1—C39	117.0 (6)	C1—C13—C12	101.3 (5)
C14—N1—C39	116.7 (6)	O1—C14—N1	124.2 (8)
C14—N2—C15	124.8 (7)	O1—C14—N2	121.0 (8)
C14—N2—C40	118.0 (6)	N1—C14—N2	114.8 (7)
C15—N2—C40	117.1 (7)	O2—C15—N2	120.6 (7)
C13—N3—C15	118.2 (6)	O2—C15—N3	123.2 (7)
C13—N3—C16	117.5 (5)	N2—C15—N3	116.1 (7)
C15—N3—C16	115.7 (6)	N3—C16—C17	116.7 (5)
C25—N4—C26	123.0 (6)	N6—C23—C22	115.6 (5)
C25—N4—C41	116.1 (6)	O4—C24—N5	119.8 (7)
C26—N4—C41	116.4 (6)	O4—C24—N6	122.9 (7)
C24—N5—C25	122.2 (6)	N5—C24—N6	117.4 (7)
C24—N5—C42	118.7 (7)	O3—C25—N4	122.9 (8)
C25—N5—C42	117.6 (6)	O3—C25—N5	120.5 (7)
C23—N6—C24	115.9 (6)	N4—C25—N5	116.7 (6)
C23—N6—C26	116.5 (5)	N4—C26—N6	108.2 (5)
C24—N6—C26	121.9 (6)	N4—C26—C27	110.0 (6)
N1—C13—N3	107.7 (5)	N4—C26—C38	112.3 (5)
N1—C13—C1	112.1 (6)	N6—C26—C27	111.8 (5)
N1—C13—C12	113.2 (5)	N6—C26—C38	113.6 (6)
N3—C13—C1	110.9 (5)		

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
C5—H5...O3 ⁱ	0.95	2.37	3.288 (9)	163.1
C9—H9...O4 ⁱⁱ	0.95	2.44	3.265 (9)	145.1

Symmetry codes: (i) $x - 1, y, z$; (ii) $1 - x, -y, 2 - z$.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *ORTEP* (Johnson, 1965) in *TEXSAN*. Software used to prepare material for publication: *TEXSAN FINISH*, *PLATON* (Spek, 1990).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: FG1188). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Packing of Four Independent Molecules: 1-Methyl-*N,N'*-bis(salicylidene)-2,4-phenylenediamine

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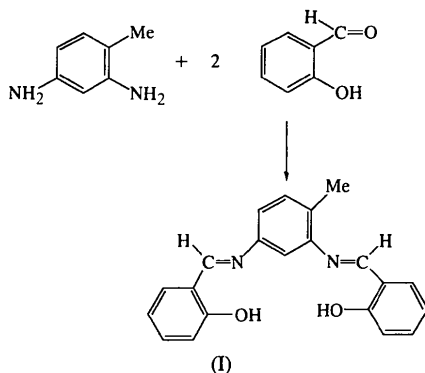
Abstract

The four independent molecules in the asymmetric unit of the title compound, C₂₁H₁₈N₂O₂, share similar geometry with two coplanar rings and the third making an angle of 20–40° with the others. In the crystal, pairs of molecules are aligned parallel, apart from the diverging third rings.

Comment

The title compound (I) was prepared for evaluation as a potential ligand, and the crystal structure of the uncomplexed molecule was determined for comparative purposes. The dimensions appear standard, though few closely similar structures have been reported (Allen *et al.*, 1991); the central toluene–nitrogen core was reported by Zhou, Chen & Shi (1984). The correspond-

ing 3,4-substituted derivative has been reported briefly by Mederos, Medina, Gili, Manrique & Nurmez (1986). In the absence of coordinates, no full comparison is



possible, but the 3,4-derivative does not have an unusual number of molecules per asymmetric unit, and so probably has less internal steric interaction than the present compound. In this, the mean C—O, C(ph)—N and C=N distances are 1.357 (4), 1.411 (6) and 1.282 (6) Å, respectively (e.s.d. values for means). The four independent molecules in the asymmetric unit (Fig. 1) are closely similar in geometry with the terminal ring (1) (the ring *trans* to the toluene methyl group) virtually parallel to the central ring (2), but the third ring (3) making variable dihedral angles with the other two (Table 2); values of *ca* 22 (twice), 30 and 38° are found. This deviation from planarity probably relieves steric interactions between the methyl group and O(2X) (on ring 3); the alternative orientation of ring 3 would introduce more severe interactions with C(17X). The orientations of the hydroxy groups suggest the formation of internal hydrogen bonding to the adjacent N atoms, as the deviations from planarity arise by twisting about the C(ph)—N bonds rather than the C(ph)—C bonds. Thus, the N atom remains in the ring plane, as close as possible to the O atom.

The independent molecules pack in pairs with the two coplanar rings closely parallel and the third rings

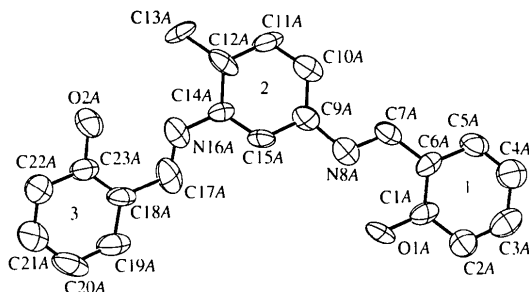
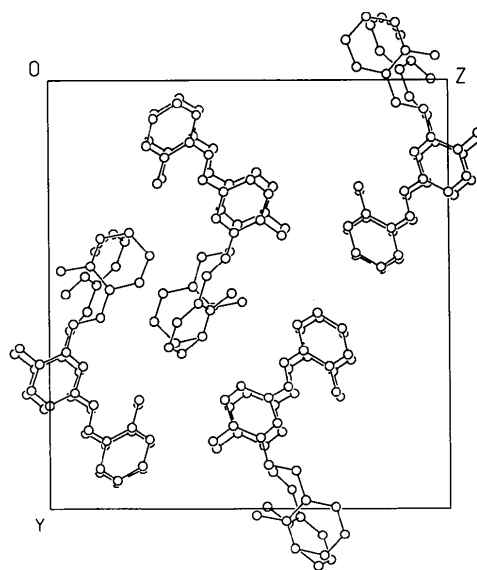
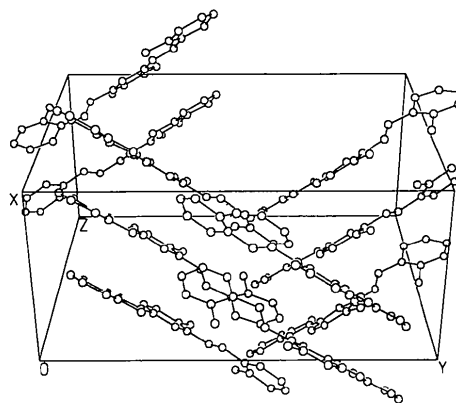


Fig. 1. View of one molecule showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level. The remaining molecules are numbered identically, with B, C and D suffixes. H atoms are omitted for clarity.



(a)



(b)

Fig. 2. (a) Packing viewed down *a*; (b) packing viewed approximately down $10\bar{1}$.

diverging. Fig. 2(a) shows the packing viewed down *a*, with the molecules overlapped, and Fig. 2(b) is the corresponding edge-on view (approximately down $10\bar{1}$).

The rationalization of packing interactions in crystals with several independent molecules is difficult (Karthé, Sadasivan & Gautham, 1993), but can sometimes be treated as equivalent to the co-crystallization of distinct species. In the present case, two alternative favourable pairwise interactions probably arise (*A* + *C*; *B* + *D*), followed by these two equally stable sub-units crystallizing together with herring-bone aromatic-aromatic contacts [Fig. 2(b)].

Experimental

2,4-Diaminotoluene (4.0 g, 32.7 mmol) in methanol (30 cm³) was added dropwise to a chilled (273 K) solution of salicylaldehyde in methanol (7.9 g, 64.7 mmol). The resulting yellow

solution was stirred at room temperature for 24 h. Complete removal of the solvent provided a light yellow solid which was washed with diethyl ether (2 × 25 cm³) and pumped dry *in vacuo*. Recrystallization from 50:50 methanol/dichloromethane provided the title compound as light yellow needles (yield 7.8 g 74%). (Found: C 76.2, H 5.5, N 9.6%; $M^* = 330$, C₂₁H₁₈O₂N₂ requires C 76.3, H 5.5, N 9.7%, $M = 330$.) NMR δ_H (CDCl₃) 2.48 (3H, s, CH₃), 6.91–7.46 (11H, m, aromatic ring), 8.62 (1H, s, OH), 8.66 (1H, s, OH), 13.25 (1H, s, CH), 13.32 (1H, s, CH).

Crystal data

C₂₁H₁₈N₂O₂ $M_r = 330.37$

Monoclinic

P2₁ $a = 9.553$ (9) Å $b = 19.20$ (3) Å $c = 18.607$ (18) Å $\beta = 101.76$ (7)° $V = 3341$ (6) Å³ $Z = 8$ $D_x = 1.313$ Mg m⁻³ D_m not measuredMo $K\alpha$ radiation $\lambda = 0.71073$ Å

Cell parameters from 20

reflections

 $\theta = 7$ –9° $\mu = 0.085$ mm⁻¹ $T = 293$ (2) K

Needle

0.78 × 0.19 × 0.13 mm

Light yellow

Data collection

Siemens R3m diffractometer

 ω -2 θ scans

Absorption correction:

none

5229 measured reflections

4893 independent reflections

1900 observed reflections

 $[I > 2\sigma(I)]$ $R_{int} = 0.0386$ $\theta_{max} = 22.61^\circ$ $h = 0 \rightarrow 10$ $k = -1 \rightarrow 20$ $l = -20 \rightarrow 19$

3 standard reflections

monitored every 197

reflections

intensity decay: 4%

Refinement

Refinement on F^2 $R[F > 4\sigma(F)] = 0.1058$ $wR(F^2) = 0.4000$ $S = 0.998$

4890 reflections

913 parameters

H atoms riding

 $w = 1/[\sigma^2(F_o^2) + (0.2409P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{max} = 0.824$ $\Delta\rho_{max} = 0.378$ e Å⁻³ $\Delta\rho_{min} = -0.384$ e Å⁻³

Extinction correction: none

Atomic scattering factors

from *International Tables*for *Crystallography* (1992,

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
O1A	-0.0078 (19)	0.7387 (8)	0.7263 (8)	0.083 (5)
C1A	-0.078 (2)	0.6782 (8)	0.7084 (8)	0.053 (6)
C2A	-0.163 (2)	0.6512 (10)	0.7540 (10)	0.071 (7)
C3A	-0.227 (2)	0.5894 (10)	0.7363 (10)	0.071 (7)
C4A	-0.207 (2)	0.5526 (9)	0.6757 (11)	0.076 (7)
C5A	-0.135 (3)	0.5807 (9)	0.6281 (10)	0.075 (7)
C6A	-0.0615 (19)	0.6437 (7)	0.6457 (8)	0.047 (5)
C7A	0.024 (2)	0.6719 (9)	0.5977 (10)	0.051 (6)
N8A	0.0889 (18)	0.7307 (8)	0.6128 (8)	0.052 (5)
C9A	0.173 (2)	0.7581 (9)	0.5617 (8)	0.050 (5)
C10A	0.180 (2)	0.7305 (9)	0.4936 (9)	0.062 (6)
C11A	0.257 (2)	0.7629 (9)	0.4493 (9)	0.067 (7)
C12A	0.331 (2)	0.8246 (8)	0.4697 (8)	0.059 (6)
C13A	0.408 (2)	0.8600 (11)	0.4176 (9)	0.063 (6)
C14A	0.3186 (19)	0.8536 (8)	0.5365 (7)	0.044 (5)
C15A	0.2427 (19)	0.8208 (9)	0.5805 (8)	0.049 (5)
N16A	0.395 (2)	0.9142 (8)	0.5577 (8)	0.063 (5)
C17A	0.364 (3)	0.9557 (10)	0.6046 (11)	0.074 (7)
C18A	0.4470 (19)	1.0200 (8)	0.6259 (9)	0.059 (6)
C19A	0.417 (2)	1.0632 (9)	0.6797 (10)	0.078 (7)
C20A	0.491 (3)	1.1217 (10)	0.7004 (10)	0.092 (9)
C21A	0.608 (3)	1.1369 (9)	0.6690 (11)	0.089 (9)
C22A	0.643 (2)	1.0961 (9)	0.6159 (10)	0.097 (10)
C23A	0.564 (2)	1.0352 (9)	0.5962 (9)	0.067 (7)
O2A	0.5997 (19)	0.9958 (7)	0.5431 (8)	0.090 (5)
O1B	0.518 (2)	0.2271 (7)	0.2841 (9)	0.087 (6)
C1B	0.588 (2)	0.1657 (8)	0.3007 (9)	0.061 (7)
C2B	0.670 (2)	0.1396 (9)	0.2533 (9)	0.065 (6)
C3B	0.733 (3)	0.0774 (10)	0.2675 (11)	0.082 (8)
C4B	0.726 (2)	0.0411 (9)	0.3293 (10)	0.074 (7)
C5B	0.649 (2)	0.0684 (9)	0.3789 (9)	0.073 (7)
C6B	0.5763 (18)	0.1297 (8)	0.3638 (8)	0.048 (5)
C7B	0.497 (2)	0.1576 (10)	0.4136 (9)	0.052 (6)
N8B	0.4251 (18)	0.2145 (7)	0.4031 (8)	0.046 (4)
C9B	0.349 (2)	0.2404 (9)	0.4526 (9)	0.050 (5)
C10B	0.339 (2)	0.2131 (9)	0.5211 (9)	0.057 (6)
C11B	0.254 (2)	0.2461 (8)	0.5616 (8)	0.054 (6)
C12B	0.1844 (19)	0.3072 (8)	0.5414 (8)	0.051 (5)
C13B	0.101 (2)	0.3436 (10)	0.5914 (9)	0.072 (7)
C14B	0.190 (2)	0.3346 (7)	0.4722 (8)	0.053 (6)
C15B	0.276 (2)	0.3002 (8)	0.4312 (7)	0.047 (5)
N16B	0.118 (2)	0.3975 (8)	0.4530 (8)	0.071 (6)
C17B	0.071 (2)	0.4106 (9)	0.3837 (8)	0.057 (6)
C18B	0.019 (2)	0.4805 (8)	0.3603 (7)	0.052 (5)
C19B	-0.030 (2)	0.4954 (9)	0.2860 (8)	0.065 (6)
C20B	-0.058 (3)	0.5610 (9)	0.2637 (9)	0.076 (7)
C21B	-0.073 (3)	0.6117 (9)	0.3155 (11)	0.107 (10)
C22B	-0.048 (2)	0.5963 (10)	0.3876 (11)	0.092 (9)
C23B	-0.004 (3)	0.5295 (9)	0.4100 (8)	0.071 (7)
O2B	0.036 (2)	0.5163 (8)	0.4833 (6)	0.104 (6)
O1C	-0.7283 (18)	0.2461 (6)	0.7756 (7)	0.073 (5)
C1C	-0.674 (2)	0.3107 (8)	0.7938 (9)	0.053 (6)
C2C	-0.589 (2)	0.3389 (9)	0.7493 (8)	0.054 (6)
C3C	-0.523 (3)	0.4006 (10)	0.7677 (11)	0.085 (8)
C4C	-0.539 (2)	0.4344 (8)	0.8302 (11)	0.072 (7)
C5C	-0.625 (2)	0.4070 (9)	0.8739 (10)	0.077 (7)
C6C	-0.691 (2)	0.3427 (8)	0.8572 (8)	0.054 (6)
C7C	-0.780 (2)	0.3168 (8)	0.9033 (10)	0.057 (6)
N8C	-0.8441 (19)	0.2576 (9)	0.8903 (8)	0.055 (5)
C9C	-0.928 (2)	0.2297 (7)	0.9361 (8)	0.042 (5)
C10C	-0.951 (2)	0.2593 (8)	0.9999 (9)	0.060 (6)
C11C	-1.022 (2)	0.2233 (8)	1.0451 (9)	0.056 (6)
C12C	-1.0890 (19)	0.1608 (8)	1.0244 (8)	0.049 (5)
C13C	-1.171 (2)	0.1230 (9)	1.0734 (9)	0.058 (6)
C14C	-1.076 (2)	0.1329 (9)	0.9570 (8)	0.057 (6)
C15C	-0.995 (2)	0.1661 (8)	0.9156 (8)	0.057 (6)
N16C	-1.144 (2)	0.0671 (8)	0.9388 (8)	0.061 (5)
C17C	-1.187 (2)	0.0519 (9)	0.8709 (9)	0.057 (6)
C18C	-1.237 (2)	-0.0133 (8)	0.8471 (7)	0.065 (6)
C19C	-1.279 (2)	-0.0298 (8)	0.7733 (8)	0.062 (6)
C20C	-1.315 (2)	-0.0933 (10)	0.7470 (9)	0.075 (7)
C21C	-1.315 (3)	-0.1471 (10)	0.7981 (11)	0.119 (11)
C22C	-1.277 (3)	-0.1337 (9)	0.8705 (10)	0.113 (11)
C23C	-1.237 (3)	-0.0682 (9)	0.8959 (8)	0.094 (9)
O2C	-1.196 (2)	-0.0567 (10)	0.9690 (6)	0.121 (7)
O1D	0.2368 (19)	0.7565 (7)	0.2221 (7)	0.083 (5)
C1D	0.173 (2)	0.8197 (9)	0.2055 (9)	0.076 (8)
C2D	0.091 (2)	0.8466 (10)	0.2516 (8)	0.068 (7)
C3D	0.022 (2)	0.9069 (10)	0.2330 (9)	0.078 (8)
C4D	0.026 (2)	0.9396 (9)	0.1681 (9)	0.072 (7)
C5D	0.113 (2)	0.9159 (8)	0.1246 (8)	0.055 (6)
C6D	0.181 (2)	0.8522 (8)	0.1398 (8)	0.049 (6)
C7D	0.264 (3)	0.8247 (9)	0.0900 (11)	0.068 (7)
N8D	0.332 (2)	0.7672 (9)	0.1042 (8)	0.064 (6)
C9D	0.406 (2)	0.7382 (9)	0.0537 (8)	0.058 (6)
C10D	0.420 (2)	0.7665 (9)	-0.0136 (9)	0.064 (7)
C11D	0.500 (2)	0.7329 (8)	-0.0568 (9)	0.074 (8)
C12D	0.569 (2)	0.6716 (9)	-0.0364 (8)	0.060 (6)
C13D	0.659 (2)	0.6367 (10)	-0.0859 (10)	0.081 (8)
C14D	0.563 (2)	0.6440 (9)	0.0318 (8)	0.059 (6)

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°)

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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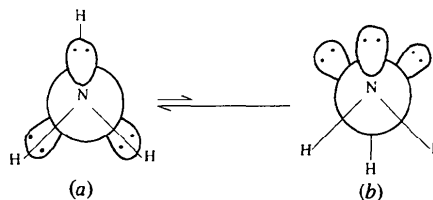
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Abstract

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 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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