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Chiral Hydroxylamines. V.† (2*S*,3*S*)-*N*-Benzyl-*N*-[3-(*tert*-butoxycarbonylamino)-2-butyl]hydroxylamine‡

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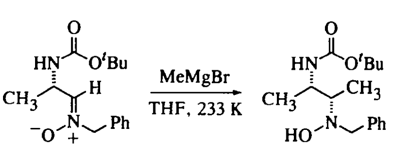
Abstract

The molecular structure of the title compound, $C_{16}H_{26}N_2O_3$, has been determined in order to establish the stereochemical effect of the addition of methyl magnesium bromide to the precursor nitrone. The molecular conformation is influenced by an intramolecular hydrogen-bonding interaction [O—H···O 2.714 (4) Å] and the packing in the crystal is mainly the result of van der Waals interactions.

Comment

In the course of our studies aimed at the synthesis of vicinal diamines, we have prepared the intermediate α-aminohydroxylamine (II) from the precursor nitrone (I), following our previously reported procedure (Merino *et al.*, 1997*a*). The structure of (I) had been confirmed previously by X-ray crystallographic analysis (Merino *et al.*, 1996*a*). The hydroxylamine (II) was obtained in 66% diastereoselectivity with 83% chemical yield and was fully characterized by means of spectroscopic data (NMR, IR and MS). The *ORTEP*II (Johnson, 1976) diagram of (II) with the atom-numbering scheme is shown in Fig. 1.



(I) (II)

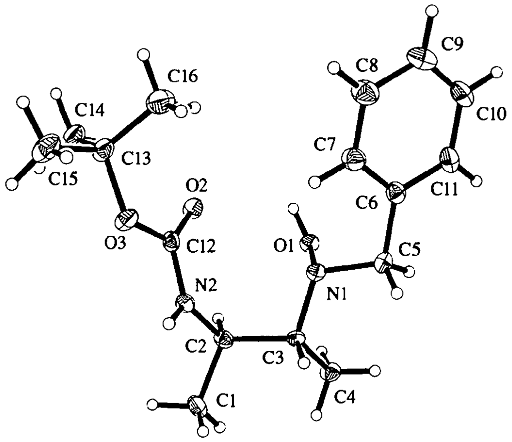


Fig. 1. The molecular structure of compound (II) showing the atom labelling. Displacement ellipsoids are shown at the 30% probability level.

We did not consider the absolute configuration of the title compound since the starting nitrone (I) was known to have an *S* configuration at the only chiral center [C2 in compound (II)]. The relative configuration at the second chiral atom, C3, is defined by the series of torsion angles given in Table 1.

As found in other α-(*tert*-butoxycarbonylamino)-hydroxylamines analyzed by X-ray crystallography (Merino *et al.*, 1996*b*, 1997*b*), an intramolecular hydrogen bond between the nitrone oxygen and the carbonyl group of the carbamate moiety is present, the distance between O1 and O2 being 2.714 (4) Å. The carbamate group is almost planar [N2—C12—O3—C13 −178.5 (4)°] as expected.

Experimental

The title compound was synthesized following our previously reported procedure for the addition of methylmagnesium bromide to α-amino nitrones (Merino *et al.*, 1997*a*). The hydroxylamine (II) was purified by column chromatography

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† Part IV: Merino, Merchan, Tejero & Franco (1997).

‡ Alternative name: *tert*-butyl *N*-{(2*S*,3*S*)-3-[*N*-(hydroxy)benzylamino]-2-butyl}carbamate.

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and then crystallized from a 3:2 mixture of hexane–diethyl ether. Crystals suitable for X-ray experiments were obtained by slow evaporation of the solvent. The melting point was found to be 397 K. $[\alpha]_D = -11.9$ (c 9.5 g dm^{-3}, chloroform).

Crystal data

$C_{16}H_{26}N_2O_3$
$M_r = 294.39$
Monoclinic
$P2_1$
$a = 6.358\,(5)$ Å
$b = 10.463\,(5)$ Å
$c = 12.842\,(5)$ Å
$\beta = 94.57\,(5)°$
$V = 851.6\,(9)$ Å^3
$Z = 2$
$D_x = 1.148$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 38 reflections
$\theta = 10.10$–$25.03°$
$\mu = 0.079$ mm^{-1}
$T = 173\,(2)$ K
Transparent block
$0.28 \times 0.12 \times 0.10$ mm
Colourless

Data collection

Siemens $P4$ diffractometer
$2\theta/\omega$ scans
Absorption correction: none
2349 measured reflections
1841 independent reflections
1458 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.068$

$\theta_{\rm max} = 25.49°$
$h = -1 \rightarrow 7$
$k = -1 \rightarrow 12$
$l = -15 \rightarrow 15$
3 standard reflections every 97 reflections intensity decay: 7.24%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.148$
$S = 1.025$
1749 reflections
195 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0889P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.198$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.235$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O2—C12	1.239 (4)	N2—C12	1.323 (5)
O1—N1	1.457 (4)	N2—C2	1.465 (6)
O3—C12	1.338 (5)	C4—C3	1.523 (6)
O3—C13	1.472 (6)	C3—C2	1.543 (5)
N1—C5	1.461 (5)	C2—C1	1.526 (6)
N1—C3	1.472 (5)		
O1—N1—C5	105.2 (3)	C1—C2—C3	111.8 (3)
O1—N1—C3	107.4 (3)	O2—C12—N2	125.6 (4)
C5—N1—C3	115.0 (3)	O2—C12—O3	123.8 (4)
N2—C2—C1	109.0 (3)	N2—C12—O3	110.5 (3)
N2—C2—C3	112.6 (3)		
C1—C2—C3—C4	−59.3 (4)	C1—C2—C3—N1	173.8 (3)
N1—C3—C2—N2	50.8 (4)	C4—C3—C2—N2	177.7 (3)

Data collection: *XSCANS* (Siemens, 1992). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXTL-Plus*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1318). Services for accessing these data are described at the back of the journal.

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Trimethyl Isocyanurate and Triethyl Isocyanurate†

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Abstract

The crystal structures of trimethyl isocyanurate, $C_6H_9N_3O_3$, (1), and triethyl isocyanurate, $C_9H_{15}N_3O_3$, (2), contain topologically similar C—H···O hydrogen-bonded networks. In (1), there are two symmetry-independent molecules and each forms its own layer structure. In (2), two of the ethyl groups point one way with respect to the heterocyclic ring, while the third points in the opposite direction.

Comment

The crystal structure of trimethyl isocyanurate, (1), has been reported previously by Belaj & Nachbaur (1987) to

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† Alternative names: 1,3,5-trimethyl-1,3,5-triazine-2,4,6(1H,3H,5H)-trione and 1,3,5-triethyl-1,3,5-triazine-2,4,6(1H,3H,5H)-trione.

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