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Structures of Chiral Nitrones. II. (*Z*)-*N*-[(2*S*)-2-(*tert*-Butoxycarbonylamino)propylidene]benzylamine *N*-Oxide

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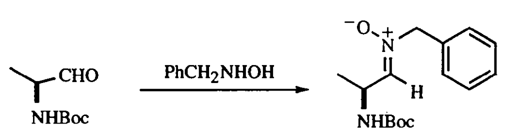
Abstract

The structure of the title compound, $C_{15}H_{22}N_2O_3$, was determined and the C—CH=N$^+$(O$^-$)C nitrone system shown to be planar. The configuration of the nitrone is confirmed to be *Z*. The molecular packing involves an intermolecular hydrogen bond of 2.915 (3) Å between the N atom of the carbonylamino group and the O atom of the *N*-oxide group.

Comment

During the course of a research program aimed at the synthesis of efficient nitrogen-containing electrophiles of synthetic utility, we have described general procedures for the preparation of both aldehyde- (Dondoni, Franco, Junquera, Merchan, Merino & Tejero, 1994) and ketone-derived nitrones (Franco, Merchan, Merino & Tejero, 1995). The title compound (II) was prepared from the α-aminoaldehyde (I) following these procedures. Its crystal structure was determined in order to confirm the configuration of the unsaturated nitrone system.

The absolute configuration of (II) was not considered since the starting aldehyde (I) was enantiomerically pure having an *S* configuration at the only asymmetric center. The molecular geometry and numbering scheme of (II) are shown in Fig. 1. The most interesting feature from a geometrical point of view is the planarity of the C—CH=N$^+$(O$^-$)C nitrone system which is confirmed to have a *Z* configuration; the O1—N1—C1—C2 and O1—N1—C1—H1 torsion angles are 5.0 (4) and −175°, respectively. The carbamate moiety is also planar [N2—C11—O2—O3 −177.8 (2)°]. No appreciable differences are observed between the bond lengths and angles of the nitrone function and those of other non-chiral (Bedford, Chaloner & Hitchcock, 1991) and chiral (Merino, Merchan, Tejero & Lanaspa, 1995) nitrones.



(I) (II)

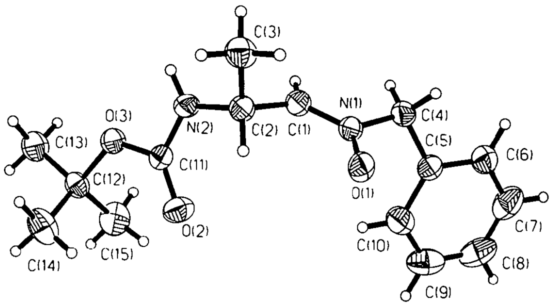


Fig. 1. Molecular geometry and the numbering scheme for the title compound. Displacement ellipsoids are plotted at the 30% probability level.

No intramolecular hydrogen bonds are observed but intermolecular hydrogen-bond interactions are present. An intermolecular hydrogen bond [N2—H···O1($-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$) 2.915 (3) Å] is observed. The packing in the crystal (shown in Fig. 2) is also influenced by van der Waals forces between the aromatic rings which are separated by 2.602 (4) Å.

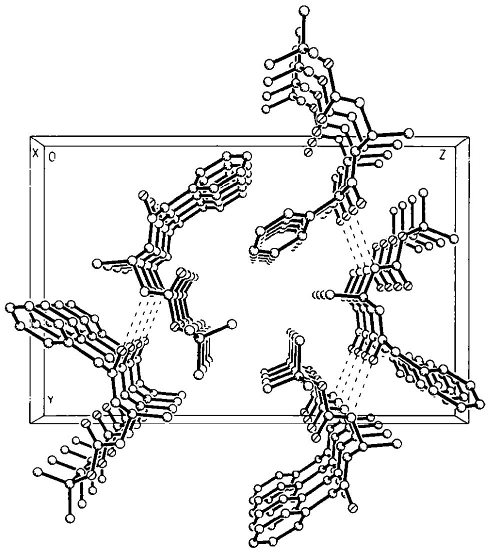


Fig. 2. Crystal packing of (II) along the shortest axis *a*, with the *c* axis horizontal.

Experimental

Compound (II) was synthesized by condensation of benzylhydroxylamine with *N*-(*tert*-butoxycarbonyl)-L-alaninal (I) (Dondoni, Franco, Junquera, Merchan, Merino & Tejero, 1994). Crystals suitable for X-ray analysis were obtained by slow evaporation of a solution of (II) in 1:5 hexane–ethyl acetate. Melting point 367 K, $[\alpha]_D$ +3.9° (*ca* 1.20 g in 100 ml^{-1} chloroform).

Crystal data

$C_{15}H_{22}N_2O_3$
$M_r = 278.35$
Orthorhombic
$P2_12_12_1$
$a = 5.9160\,(10)$ Å
$b = 13.112\,(3)$ Å
$c = 20.253\,(4)$ Å
$V = 1571.0\,(5)$ Å^3
$Z = 4$
$D_x = 1.177$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 39 reflections
$\theta = 12.237$–$25.136°$
$\mu = 0.082$ mm^{-1}
$T = 223\,(2)$ K
Transparent block
0.6 × 0.4 × 0.3 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$2\theta/\omega$ scans
Absorption correction: none
2193 measured reflections
2010 independent reflections
1729 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0419$

$\theta_{\rm max} = 24.98°$
$h = -1 \rightarrow 7$
$k = -1 \rightarrow 15$
$l = -1 \rightarrow 24$
3 standard reflections monitored every 97 reflections intensity decay: 7.68% (corrected)

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0416$
$wR(F^2) = 0.1078$
$S = 1.070$
2010 reflections
185 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0620P)^2 + 0.1528P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.179$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.193$ 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 (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O1	0.0114 (3)	0.23203 (12)	0.27315 (9)	0.0376 (4)
O2	0.4041 (3)	0.48101 (13)	0.35813 (8)	0.0426 (5)
N1	−0.1369 (4)	0.30125 (15)	0.28955 (9)	0.0306 (5)
O3	0.3358 (3)	0.65110 (12)	0.34419 (8)	0.0362 (4)
N2	0.1642 (4)	0.54074 (15)	0.27922 (10)	0.0353 (5)
C1	−0.1023 (5)	0.3986 (2)	0.28363 (12)	0.0336 (6)
C11	0.3097 (4)	0.5506 (2)	0.32999 (11)	0.0291 (5)
C2	0.1066 (5)	0.4411 (2)	0.25205 (12)	0.0328 (6)
C12	0.4912 (5)	0.6862 (2)	0.39605 (12)	0.0334 (6)
C5	−0.3538 (5)	0.1999 (2)	0.37511 (12)	0.0331 (6)
C4	−0.3612 (4)	0.2633 (2)	0.31265 (12)	0.0340 (6)
C10	−0.1776 (5)	0.2041 (2)	0.42059 (13)	0.0445 (7)
C13	0.4561 (6)	0.8010 (2)	0.39415 (14)	0.0470 (7)
C6	−0.5418 (5)	0.1405 (2)	0.38883 (14)	0.0443 (7)
C3	0.0678 (5)	0.4501 (2)	0.17726 (12)	0.0428 (7)
C9	−0.1943 (7)	0.1495 (3)	0.47945 (14)	0.0577 (9)
C14	0.7333 (5)	0.6591 (2)	0.3783 (2)	0.0543 (8)
C7	−0.5559 (6)	0.0855 (2)	0.4476 (2)	0.0582 (9)
C8	−0.3831 (7)	0.0911 (3)	0.4925 (2)	0.0601 (10)
C15	0.4221 (6)	0.6426 (2)	0.46255 (13)	0.0523 (8)

Table 2. *Selected geometric parameters (Å, °)*

O1—N1	1.306 (3)	C12—C14	1.518 (4)
O2—C11	1.213 (3)	C12—C13	1.520 (4)
N1—C1	1.298 (3)	C12—C15	1.519 (4)
N1—C4	1.492 (3)	C5—C6	1.386 (4)
O3—C11	1.357 (3)	C5—C10	1.392 (4)
O3—C12	1.470 (3)	C5—C4	1.515 (3)
N2—C11	1.347 (3)	C10—C9	1.394 (4)
N2—C2	1.458 (3)	C6—C7	1.394 (4)
C1—C2	1.499 (4)	C9—C8	1.380 (5)
C2—C3	1.537 (3)	C7—C8	1.369 (5)
C1—N1—O1	123.6 (2)	C14—C12—C13	110.8 (3)
C1—N1—C4	119.8 (2)	O3—C12—C15	110.3 (2)
O1—N1—C4	116.5 (2)	C14—C12—C15	112.0 (3)
C11—O3—C12	121.8 (2)	C13—C12—C15	111.0 (2)
C11—N2—C2	121.6 (2)	C6—C5—C10	119.4 (2)
N1—C1—C2	122.4 (2)	C6—C5—C4	116.9 (2)
O2—C11—N2	125.5 (2)	C10—C5—C4	123.5 (2)
O2—C11—O3	125.4 (2)	N1—C4—C5	114.8 (2)
N2—C11—O3	109.1 (2)	C5—C10—C9	119.5 (3)
N2—C2—C1	111.4 (2)	C5—C6—C7	120.6 (3)
N2—C2—C3	109.8 (2)	C8—C9—C10	120.4 (3)
C1—C2—C3	109.0 (2)	C8—C7—C6	119.6 (3)
O3—C12—C14	110.4 (2)	C7—C8—C9	120.5 (3)
O3—C12—C13	101.9 (2)		
O1—N1—C1—C2	5.0 (4)	C6—C7—C8—C9	−0.9 (5)
H1—C1—N1—C4	9	C7—C8—C9—C10	0.4 (5)
C4—N1—C1—C2	−171.4 (2)	N2—C11—O2—O3	179.2 (4)
C4—C5—C6—C7	−175.3 (2)	O1—N1—C1—H1	−175
C5—C6—C7—C8	0.6 (4)		

Data collection: *XSCANS* (Siemens, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXL*93. Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1197). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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