Acta Cryst. (1997). **C53**, 1951–1952

1,3-Bis[bis(dimethylamino)]-*N*-methyldiborylamine

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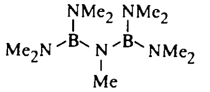
(*Received 19 May 1997; accepted 1 August 1997*)

Abstract

The structure of the title compound, $C_9H_{27}B_2N_5$, shows twofold symmetry and the boron–nitrogen framework of this molecule is planar.

Comment

As part of an ongoing investigation of reductive dehalogenation of boron halides (Maringgele, Dielkus *et al.*, 1994; Maringgele, Heine *et al.*, 1994; Maringgele, Noltemeyer & Meller, 1997), the title compound, (I), was synthesized as an intermediate. It was characterized both spectroscopically and by X-ray crystallography.



$(Me_2N)(NMe_2)B{-}N(Me){-}B(NMe_2)(NMe_2)$

(I)

The molecule lies on a twofold axis which passes through the central N atom and the attached methyl group.

The central B—NMe—B unit is planar as required by space group symmetry. The environment of the B atom is also nearly planar [the B atom lies 0.009 (1) Å from the plane through the three N atoms bound to it], whereas the terminal N atoms show slight but significant deviations from planarity; N1 and N2 are 0.015 (1) and 0.057 (1) Å from their respective surrounding atoms.

The average bond length from the B atom to the terminal N atoms is 1.432 Å, which lies in the range 1.415–1.448 Å found for aminoalkylboranes in the Cambridge Structural Database (Allen & Kennard, 1993). In contrast, the bond length to the bridging N atom is significantly longer (1.455 Å), as expected since the lone pair on that N atom is delocalized over two B atoms instead of just one.

As a result of the steric repulsion of the methyl groups, the NMe_2 groups are not coplanar with the B—NMe—B plane. The interplanar angles range from 24.0 to 35.3°. This is similar to the range found in the compound $B(NMe_2)_3$ (Schmid, Boese & Bläser, 1982), which should have quite similar steric requirements.

The methyl groups envelop the molecule and give it high stability against nucleophilic attack. This compound can be handled in air and hydrolyzes only after several hours.

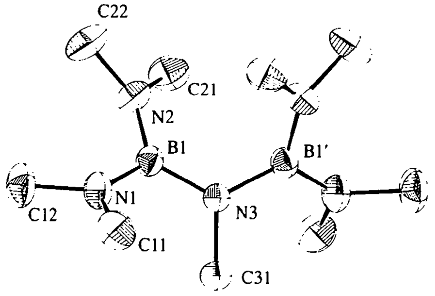


Fig. 1. The molecular structure of (I) showing 50% probability ellipsoids. H atoms have been omitted for clarity.

Experimental

Chlorobis(dimethylamino)borane (61.8 g, 0.46 mol) (Nöth, Schick & Meister 1964) was added to bis(trimethylsilyl)-methylamine (40.0 g, 0.23 mol) (Wannagat & Gerschler, 1957) while cooling with an ice bath. The reaction mixture was heated to 383 K and the Me_3SiCl formed during reaction removed by distillation. When no more Me_3SiCl distilled over, the target compound was purified by vacuum distillation to yield a clear liquid. On standing overnight, large well formed needles were obtained which were suitable for X-ray diffraction studies. Yield 33.7 g (61 %); b.p. 325–327 K, 10^{-3} Torr (1 Torr = 133.322 Pa); m.p. 288–300 K. ^{1}H NMR: 2.47 (*s*, 3H, NMe_2), 2.54 (*s*, 24 H, NMe). ^{13}C NMR: 34.58 (NMe_2), 39.37 (NMe). ^{11}B NMR: 28.7 ($h_{1/2}$ = 150 Hz). EI–MS: 227 (3) [M^+], 183 (100) [$M^+ - NMe_2$]. FI–MS: 227 (100) [M^+].

Crystal data

$C_9H_{27}B_2N_5$
$M_r = 226.98$
Monoclinic
$C2/c$
$a = 10.349(4)$ Å
$b = 13.981(6)$ Å
$c = 10.247(4)$ Å
$\beta = 102.46(6)°$
$V = 1447.8(10)$ Å^3
$Z = 4$
$D_x = 1.041$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 23 reflections
$\theta = 5–15°$
$\mu = 0.064$ mm^{-1}
$T = 193(2)$ K
Monoclinic prism
0.6 × 0.6 × 0.6 mm
Colourless

Data collection

Stoe–Siemens AED four-circle diffractometer
ω–2θ scans (Clegg, 1981*a*,*b*)
Absorption correction: none
4595 measured reflections
2117 independent reflections
1805 reflections with $I > 2\sigma(I)$

$R_{int} = 0.046$
$\theta_{max} = 30.08°$
$h = -14 \rightarrow 14$
$k = -19 \rightarrow 19$
$l = -12 \rightarrow 14$
3 standard reflections
frequency: 90 min
intensity decay: none

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Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.162$
$S = 1.068$
2117 reflections
80 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0955P)^2 + 0.346P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.355$ e Å^{-3}
$\Delta\rho_{min} = -0.201$ e Å^{-3}
Extinction correction: *SHELXL*96
Extinction coefficient: 0.014 (4)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
B1	0.12738 (10)	0.39998 (8)	0.27444 (10)	0.0310 (2)
N1	0.23964 (8)	0.44035 (7)	0.36486 (9)	0.0399 (3)
C11	0.23101 (13)	0.49365 (10)	0.48373 (12)	0.0500 (3)
C12	0.37380 (11)	0.43480 (11)	0.34414 (14)	0.0545 (4)
N2	0.14302 (9)	0.31287 (6)	0.20691 (9)	0.0398 (3)
C21	0.05408 (14)	0.28068 (9)	0.08588 (12)	0.0496 (3)
C22	0.24049 (15)	0.24075 (10)	0.26201 (16)	0.0601 (4)
N3†	0	0.44841 (8)	1/4	0.0300 (3)
C31†	0	0.55328 (10)	1/4	0.0386 (3)

† Site occupancy = 0.5.

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

B1—N2	1.4271 (14)	B1—N3	1.4548 (12)
B1—N1	1.4361 (15)		
N2—B1—N1	119.18 (9)	N1—B1—N3	120.34 (9)
N2—B1—N3	120.47 (9)	B1—N3—B1^i	124.53 (11)

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

H atoms were included in calculated positions using a riding model. The torsion angle of the methyl groups was determined by a circular difference Fourier synthesis. The isotropic displacement parameter was set to $1.5U_{iso}$ of the parent atom. The H atoms of the methyl group, C31, are disordered about the twofold axis.

Data collection: *DIF*4 (Stoe & Cie, 1991*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1991*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996). Molecular graphics: *ZORTEP* (Zsolnai, Pritzkow & Huttner, 1996). Software used to prepare material for publication: *SHELXL*96.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1012). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). C**53**, 1952–1954

Diphenyl (−)-(*R*)-1-[(*S*)-Isoleucyl]pyrrolidine-2-phosphonate Hydrochloride

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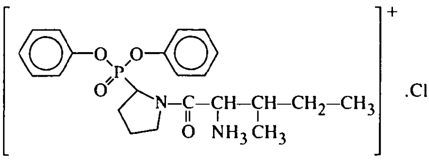
(*Received 29 April 1997; accepted 6 June 1997*)

Abstract

The crystal structure of the title compound, $C_{22}H_{30}N_2O_4P^+.Cl^-$, was determined in order to ascertain its absolute configuration. The pyrrolidine ring is markedly flattened and adopts a conformation intermediate between an envelope and a half-chair.

Comment

The title compound, (I), belongs to a series of *L-AA*–Pro^P dipeptides (*L-AA* is an amino acid residue and Pro^P is proline diphenyl phosphonate). These compounds were synthesized as potential inhibitors of proline-specific serine proteases. Only the (−) diastereomers acted as inhibitors (Belyaev *et al.*, 1995). To obtain the absolute configuration of (I), its crystal structure has been determined from single-crystal diffraction data.



(I)