

2,4,6-Tris(dimethylamino)-1,3,5-trimethylborazine

Mark A. Rodriguez^{a*} and
Theodore T. Borek^b^aPO Box 5800, MS 1411, Sandia National Laboratories, Albuquerque, NM 87185-1411, USA, and ^bPO Box 5800, MS 1411, Sandia National Laboratories, Albuquerque, NM 87185-0886, USA

Correspondence e-mail: marodri@sandia.gov

Key indicators

Single-crystal X-ray study

T = 173 K

Mean $\sigma(\text{N}-\text{B}) = 0.002 \text{ \AA}$

R factor = 0.045

wR factor = 0.139

Data-to-parameter ratio = 15.5

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the crystal structure of the title compound, $\text{C}_9\text{H}_{27}\text{B}_3\text{N}_6$, the borazine ring displays significant distortion away from a planar six-membered ring to a boat configuration. The distortion results in layered packing of the molecules to accommodate the distorted borazine rings.

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Comment

The title compound, (I), is a solid white material that melts at 337 K. It has been used as a starting material for further borazine transamination reactions as documented by Niedenzu *et al.* (1961) and its pyrolysis products have been modestly investigated by Toeniskoetter & Hall (1963).

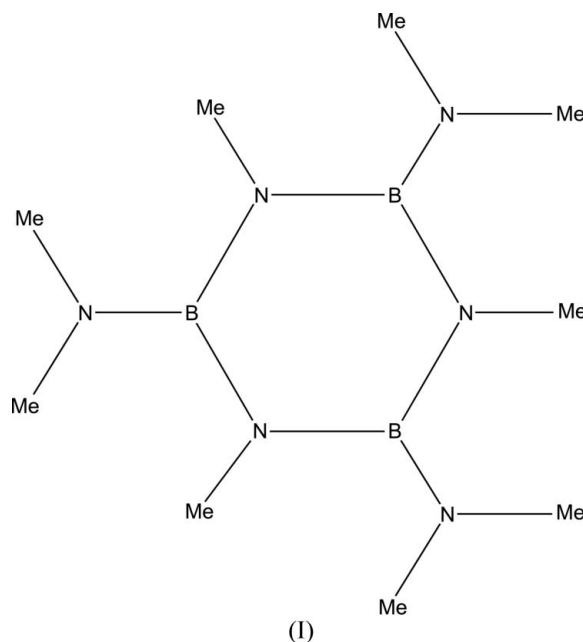


Fig. 1 shows the molecular structure of (I). This structure is similar to those of other amide-based borazine structures (Hess & Reiser, 1971; Cornu *et al.*, 1999; Toury *et al.*, 2001). In contrast to the planar borazine rings of these references, compound (I) shows significant distortion. Fig. 2 shows a side view of (I) with some of the ligands omitted for clarity. If one considers the plane formed by the central B1–N3 and B2–N2 atoms in the borazine ring as a reference plane, calculation of the plane defined by N2–B3–N3 combined with the amine group C9–N6–C8 yields a dihedral angle of $27.2 (1)^\circ$. A similar distortion occurs for the plane defined by the C1 methyl bound to the B1–N1–B2 section of the borazine ring that shows a tilt of $22.8 (1)^\circ$ (see Fig. 2). Thus, one observes a boat-like distortion of the borazine ring, which, while not

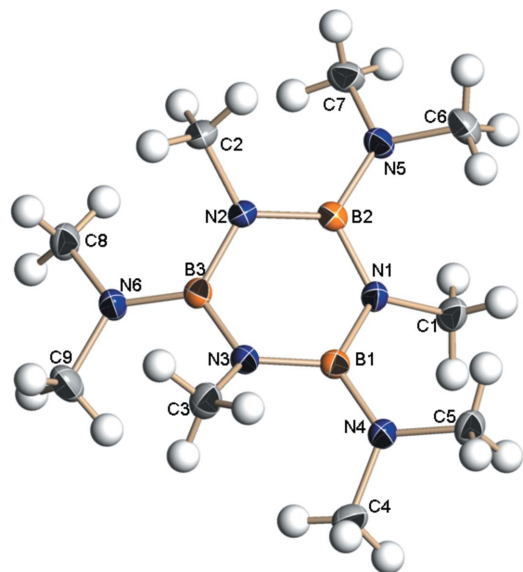


Figure 1
Displacement ellipsoid plot for (I) (50% probability level).

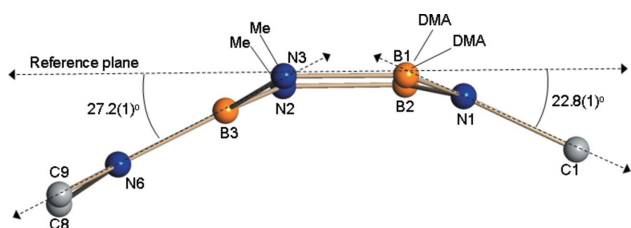


Figure 2
Side view of (I), to illustrate the degree of bending in the borazine ring. Some methyl (Me) and dimethylamine (DMA) ligands have been omitted for clarity of the tilt planes.

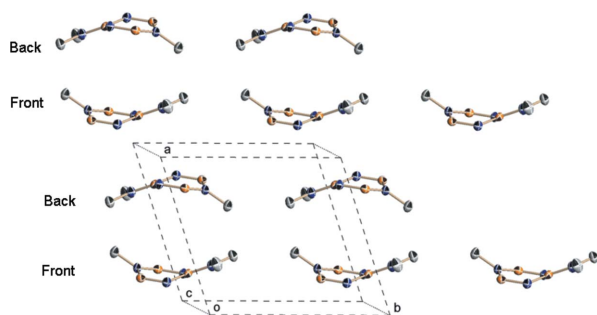


Figure 3
Packing diagram for (I), to illustrate the orientation, packing and relationship of distorted borazine rings within the crystal structure. Some methyl and dimethylamine ligands have been omitted so as to clarify the distorted borazine.

common, can occur (Habereider & Noth, 2001; Ma *et al.* 2002). Table 1 shows bond lengths for (I). Distortion of the borazine ring is mildly reflected in the B–N bond lengths.

Fig. 3 displays a packing diagram of (I). As in Fig. 2, some ligands have been omitted for clarity. From this figure it can be observed that (I) forms layers of like-oriented molecules where the layer stacking direction is along the *a* axis. In each layer, the molecules form head-to-toe rows that propagate along the *b* axis. Each subsequent stacking layer reverses the

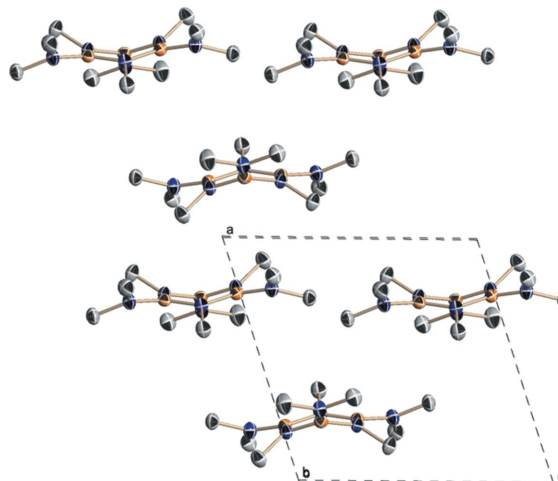


Figure 4
Packing diagram for (I), viewed down the *b* axis, to illustrate the staggering of alternating layers of molecules as they stack in the *a* axis direction.

head-to-toe direction along the *b* axis and also inverts the molecule. Although difficult to see in Fig. 3, each stacked layer is displaced by one-half of the *c* axis forward or backward from neighboring layers (hence, the labeling on the figure). The layer displacement is better illustrated by Fig. 4 which shows the stacking as viewed down the *b* axis. In this case, all atoms except H atoms are shown. This figure shows the relative shift of the molecules in each subsequent layer.

Experimental

Compound (I) was obtained using the published synthetic procedure of Toeniskoetter & Hall (1963), which reacts one equivalent of *B*-trichloroborazine in anhydrous toluene with six equivalents of anhydrous dimethylamine in anhydrous toluene at 195 K. The resulting mixture was filtered and the solvent removed by vacuum techniques. The crude product was extracted with additional anhydrous toluene and then vacuum sublimed with an 86% yield. Crystals of (I) were grown by recrystallization from hexane followed by vacuum sublimation; crystals were obtained off of the cold finger. Product purity was determined by nuclear magnetic resonance (^1H , ^{11}B , ^{13}C) and mass spectrometry (GC/MS and MALDI).

Crystal data

$\text{C}_9\text{H}_{27}\text{B}_3\text{N}_6$	$V = 749.2 (3) \text{ \AA}^3$
$M_r = 251.80$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.116 \text{ Mg m}^{-3}$
$a = 8.998 (2) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 10.043 (2) \text{ \AA}$	$\mu = 0.07 \text{ mm}^{-1}$
$c = 10.098 (2) \text{ \AA}$	$T = 173 (2) \text{ K}$
$\alpha = 119.045 (3)^\circ$	Irregular, colorless
$\beta = 100.581 (4)^\circ$	$0.39 \times 0.35 \times 0.31 \text{ mm}$
$\gamma = 99.473 (3)^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	5393 measured reflections
φ and ω scans	2664 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1999)	2404 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.97$, $T_{\max} = 0.98$	$R_{\text{int}} = 0.015$
	$\theta_{\text{max}} = 25.3^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.139$
 $S = 1.08$
 2664 reflections
 172 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0759P)^2 + 0.2627P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.15 \text{ e } \text{\AA}^{-3}$

Table 1

Selected bond lengths (Å).

B1—N4	1.435 (2)	N1—C1	1.471 (2)
B1—N3	1.442 (2)	N2—C2	1.467 (2)
B1—N1	1.448 (2)	N3—C3	1.469 (2)
B2—N5	1.433 (2)	N4—C4	1.449 (2)
B2—N2	1.439 (2)	N4—C5	1.453 (2)
B2—N1	1.450 (2)	N5—C6	1.449 (2)
B3—N6	1.417 (2)	N5—C7	1.450 (2)
B3—N2	1.462 (2)	N6—C8	1.457 (2)
B3—N3	1.466 (2)	N6—C9	1.458 (2)

All H atoms were positioned geometrically and treated as riding, with C—H distances of 0.98 Å and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 2001); program(s) used to solve structure: *SHELXTL* (Bruker, 1998); program(s) used to refine

structure: *XSHELL* (Bruker, 2000); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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