organic compounds

2,2,4,4-Tetrabromo-1,1,3,3-tetramethyl-cyclodiborazane

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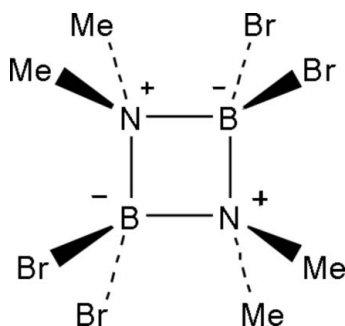Correspondence e-mail: dbouriss@chimie.ups-tlse.fr

The title compound, $C_4H_{12}B_2Br_4N_2$, contains an almost square four-membered ring that results from the head-to-tail dimerization of the dimethylaminodibromoborane. The dimer has almost *mmm* symmetry and does have $2/m$ crystallographic symmetry. The crystal structure involves C—H⋯Br hydrogen bonds.

Related literature

For related structures featuring an aminodibromoborane skeleton, see: Abu Ali *et al.* (2001); Klebe *et al.* (1984); Nie *et al.* (2005); Nöth *et al.* (1983). Aminohalogenoboranes are direct precursors for diboranes, see: Ishimaya *et al.* (2002).

Experimental

Crystal data

$C_4H_{12}B_2Br_4N_2$	V = 583.33 (12) Å^3
M_r = 429.42	Z = 2
Monoclinic, $C2/m$	Mo $K\alpha$ radiation
a = 11.1089 (13) Å	μ = 13.75 mm^{-1}
b = 8.8842 (10) Å	T = 173 (2) K
c = 6.9077 (8) Å	$0.08 \times 0.08 \times 0.03$ mm
β = 120.074 (2)°	

Data collection

Bruker APEXII CCD area-detector diffractometer	2689 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)	769 independent reflections
	683 reflections with $I > 2\sigma(I)$
T_{min} = 0.210, T_{max} = 0.662	R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032	34 parameters
$wR(F^2)$ = 0.085	H-atom parameters constrained
S = 1.04	$\Delta\rho_{max}$ = 0.74 e Å^{-3}
769 reflections	$\Delta\rho_{min}$ = −1.42 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C1—H1A⋯Br1^{iii}	0.98	3.02	3.982 (4)	167

Symmetry code: (iii) $-x + \frac{5}{2}, -y + \frac{1}{2}, -z + 1$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2006); software used to prepare material for publication: *SHELXTL*.

References

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supplementary materials

2,2,4,4-Tetrabromo-1,1,3,3-tetramethylcyclodiborazane

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Comment

The structure of the title compound is shown in Fig. 1. Head-to-tail dimerization of dimethylaminodibromoborane results in a NBNB four-membered ring only slightly deviating from square geometry. The shortest intermolecular contact between hydrogen and bromine from adjacent molecules is of 3.022 Å.

Experimental

The title compound was prepared by substituent redistribution from tris(dimethylamino)borane and tribromoborane. Suitable crystals were obtained upon allowing the crude reaction mixture to stand at 293 K.

Refinement

All H atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.97 Å and with U_{iso}(H) = 1.2U_{eq}(C).
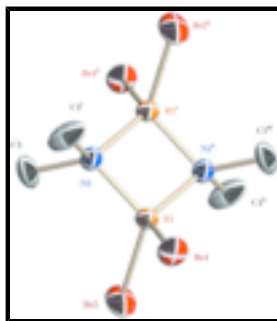
Figures

Fig. 1. The molecular structure of dimethylaminodibromoborane dimer, with the atom-labeling scheme. The B and Br atoms lie in the crystallographic mirror plane and the N atoms lie on the twofold axis. Displacement ellipsoids are drawn at the 50% probability level. H atoms omitted for clarity. Symmetry codes: (i) −x + 2, y, −z + 1; (ii) −x + 2, −y, −z + 1; (iii) x, −y, z.

2,2,4,4-Tetrabromo-1,1,3,3-tetramethylcyclodiborazane

Crystal data

$C_4H_{12}B_2Br_4N_2$	F_{000} = 400
M_r = 429.42	D_x = 2.445 Mg m^{-3}
Monoclinic, *C*2/*m*	Melting point: 340 K
Hall symbol: -C 2y	Mo *K*α radiation λ = 0.71073 Å
a = 11.1089 (13) Å	Cell parameters from 1609 reflections
b = 8.7842 (10) Å	θ = 3.1–28.2°

$c = 6.9077\ (8)$ Å

$\beta = 120.074\ (2)°$

$V = 583.33\ (12)$ Å^3

$Z = 2$

$\mu = 13.75$ mm^{-1}

$T = 173\ (2)$ K

Plate, colourless

$0.08 \times 0.08 \times 0.03$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer	769 independent reflections
Radiation source: fine-focus sealed tube	683 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.048$
$T = 173(2)$ K	$\theta_{max} = 28.2°$
φ and ω scans	$\theta_{min} = 3.1°$
Absorption correction: multi-scan (SADABS; Bruker, 2006)	$h = -14\rightarrow14$
$T_{min} = 0.210$, $T_{max} = 0.662$	$k = -10\rightarrow11$
2689 measured reflections	$l = -9\rightarrow9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.032$	H-atom parameters constrained
$wR(F^2) = 0.085$	$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2 + 1.911P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
769 reflections	$\Delta\rho_{max} = 0.74$ e Å^{-3}
34 parameters	$\Delta\rho_{min} = -1.42$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The crystal was made of many different layers so it was twinned. To solve this problem, we had to cut a very small plate (we couldn't even see it with the camera). This is why we measured a crystal that we were unable to index correctly for the absorption correction.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of F^2 > 2sigma(F^2) is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
Br1	1.17185 (5)	0.0000	0.31107 (9)	0.03261 (19)
Br2	1.28193 (6)	0.0000	0.83699 (9)	0.0429 (2)
N1	1.0000	0.1319 (4)	0.5000	0.0230 (8)
B1	1.1096 (5)	0.0000	0.5364 (8)	0.0205 (9)
C1	1.0398 (4)	0.2347 (5)	0.6963 (9)	0.0449 (10)
H1A	1.1199	0.2969	0.7227	0.067*
H1C	1.0644	0.1731	0.8293	0.067*
H1B	0.9613	0.3012	0.6651	0.067*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0359 (3)	0.0390 (3)	0.0348 (3)	0.000	0.0266 (2)	0.000
Br2	0.0411 (3)	0.0391 (4)	0.0231 (3)	0.000	−0.0028 (2)	0.000
N1	0.0233 (18)	0.0197 (18)	0.0300 (19)	0.000	0.0164 (16)	0.000
B1	0.018 (2)	0.023 (2)	0.022 (2)	0.000	0.0107 (18)	0.000
C1	0.042 (2)	0.037 (2)	0.069 (3)	−0.0175 (17)	0.037 (2)	−0.032 (2)

Geometric parameters (Å, °)

Br1—B1	1.995 (5)	N1—B1	1.606 (4)
Br2—B1	1.998 (5)	B1—N1^{ii}	1.606 (4)
N1—C1	1.499 (4)	C1—H1A	0.9800
N1—C1^{i}	1.499 (4)	C1—H1C	0.9800
N1—B1^{ii}	1.606 (4)	C1—H1B	0.9800
C1—N1—C1^{i}	106.0 (5)	N1^{ii}—B1—Br2	114.8 (2)
C1—N1—B1^{ii}	115.7 (2)	N1—B1—Br2	114.8 (2)
C1^{i}—N1—B1^{ii}	115.8 (3)	Br1—B1—Br2	106.5 (2)
C1—N1—B1	115.8 (3)	N1—C1—H1A	109.5
C1^{i}—N1—B1	115.7 (2)	N1—C1—H1C	109.5
B1^{ii}—N1—B1	87.7 (3)	H1A—C1—H1C	109.5
N1^{ii}—B1—N1	92.3 (3)	N1—C1—H1B	109.5
N1^{ii}—B1—Br1	114.2 (2)	H1A—C1—H1B	109.5
N1—B1—Br1	114.2 (2)	H1C—C1—H1B	109.5

Symmetry codes: (i) −x+2, y, −z+1; (ii) −x+2, −y, −z+1.

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···Br1^{iii}	0.98	3.02	3.982 (4)	167

Symmetry codes: (iii) −x+5/2, −y+1/2, −z+1.

Fig. 1