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1,4-Dibromo-1,4-disilabutane

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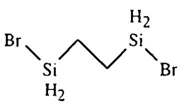
Abstract

Molecules of the title compound, $C_2H_8Br_2Si_2$, have a crystallographic centre of inversion. The Si—C—C—Si unit is thus planar and has a *trans* conformation. The Br—Si—C—C moieties have almost ideal *gauche* conformations with Si—Br bond lengths of 2.2362 (12) Å.

Comment

In the course of studies on cyclic silicon–nitrogen compounds (Mitzel, Bissinger, Riede, Dreihäupl & Schmidbaur, 1993; Mitzel, Riede, Schier, Angermaier & Schmidbaur, 1995), 1,4-dibromo-1,4-disilabutane, (1), served as a synthon for the $H_2Si—CH_2—CH_2—SiH_2$ unit. Structural information on simple molecules containing this structural element appeared desirable in order to obtain a basis for an understanding of the decomposition pathways of such molecules, which are probed as single-source precursors for the chemical vapour deposition (CVD) of epitaxially grown β-SiC (Kunstmann *et al.*, 1995).



(1)

In the crystal, the molecule of (1) has a crystallographic centre of inversion (Fig. 1). Similar results were obtained for all other open chain molecules with a symmetrical substitution pattern at the Si—C—C—Si unit studied to date (Shibayeva, Atovmyan, Rozenberg & Stryukov, 1983; Tacke, Niedner, Frohnecke, Ernst & Sheldrick, 1980; Ovchinnikov, Shklover, Struchkov, Polyakov & Guselnikov, 1985).

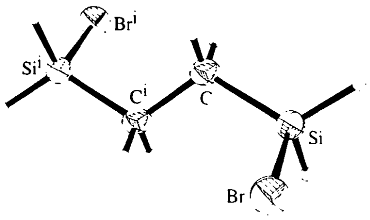


Fig. 1. A view of one molecule of (1) with the atom-numbering scheme. Displacement ellipsoids enclose 50% probability surfaces and H atoms are shown as spheres of arbitrary radii.

The C—C and Si—C distances, as well as the angle Si—C—C, are very similar to those reported for the reference molecules. The Si—Br distance [2.2362 (12) Å] fits well into the range established by only four examples of Si—Br compounds with four-coordinate Si atoms studied in the solid state so far [according to a search in the Cambridge Crystal Structure Database and the Inorganic Crystal Structure Database (Gurkova, Gusev, Sharapov, Gar & Alexeev, 1979; Kratky, Hengge, Stüger & Rheingold, 1985; Schubert & Steib, 1982)].

The most interesting structural feature of (1) is the *gauche* conformation of each Br—Si—C—C unit. The corresponding torsion angle [−61.7 (5)°] is close to the ideal value for the *gauche* conformation of interpenetrating tetrahedra. The overall *gauche-trans-gauche* conformation has also been found by Ovchinnikov *et al.* (1985) in 2,5-dichloro-2,5-dimethyl-2,5-disilahexane and by Tacke *et al.* (1980) for $(\{[Me_3N(CH_2)_3]Me_2SiCH_2\}_2)I_2$, taking the most electronegative substituent at silicon as a reference. This conformational preference appears to be an inherent characteristic of this class of compounds, although in the present case it may also be affected by Si···Br and Br···Br intermolecular interactions. There are some weak intermolecular interactions as indicated by the shortest lattice contacts Si···Br [3.741 (1) Å] and Br···Br [3.839 (1) Å], which are very close to the corresponding sums of the van der Waals radii (Si···Br

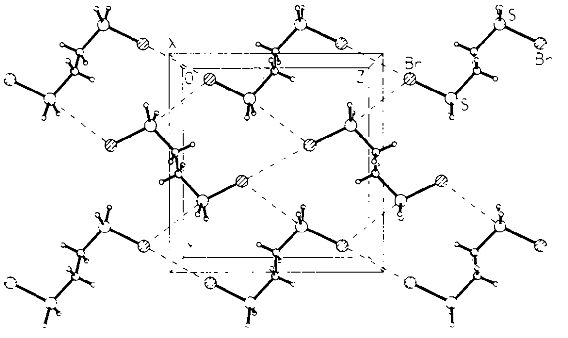


Fig. 2. Packing plot showing the Si···Br and Br···Br contacts in crystals of (1), and the unit cell.

3.95, Br···Br 3.90 Å). This Br···Si interaction causes a contraction of the Br—Si—H angles and a subsequent widening of the C—Si—H angles (see Fig. 2). The Br—Si···Br (x, $\frac{1}{2} - y$, $z - \frac{1}{2}$) angle is 167.65 (4)°.

We are presently investigating the solid-state and gas-phase structures of $H_3Si—CH_2—CH_2—SiH_3$ and $Cl_3Si—CH_2—CH_2—SiCl_3$, mainly with respect to their molecular conformations.

Experimental

1,4-Dibromo-1,4-disilabutane, (1), was prepared by a modified literature procedure (Schmidbaur & Dörzbach, 1987). Crystals of (1) were grown by slowly cooling the melt, diluted with a minimum amount of pentane, to 243 K. Owing to the air sensitivity and the low melting point of (1), the crystals were transferred to a glass capillary and sealed under an atmosphere of dry argon at 223 K.

Crystal data

$C_2H_8Br_2Si_2$
$M_r = 248.08$
Orthorhombic
Pbca
$a = 8.104\,(1)$ Å
$b = 9.975\,(1)$ Å
$c = 9.986\,(2)$ Å
$V = 807.2\,(2)$ Å^3
$Z = 4$
$D_x = 2.041$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 15$–$19°$
$\mu = 10.231$ mm^{-1}
$T = 195\,(3)$ K
Block
0.40 × 0.35 × 0.30 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–θ scans with ω width $(1.0 + 0.35\tan\theta)°$
Absorption correction: ψ scans (North, Phillips & Mathews, 1968) and refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{min} = 0.016$, $T_{max} = 0.039$
884 measured reflections
880 independent reflections

699 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.202$ (on 4 reflections only)
$\theta_{max} = 26.96°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 12$
3 standard reflections monitored every 100 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0315$
$wR(F^2) = 0.0779$
$S = 1.168$
876 reflections
45 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0357P)^2 + 0.1370P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.446$ e Å^{-3}
$\Delta\rho_{min} = -0.437$ e Å^{-3}
Extinction correction: *SHELXL93* (Sheldrick, 1993)
Extinction coefficient: 0.0009 (4)
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 isotropic or equivalent isotropic displacement parameters (Å^2)*

U_{iso} for H atoms; $U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for all others.

	x	y	z	U_{eq}
Br	0.02221 (5)	0.41173 (4)	0.83016 (4)	0.0353 (2)
Si	0.03694 (12)	0.31442 (11)	0.62877 (11)	0.0261 (3)
C	0.0706 (5)	0.4484 (4)	0.5031 (4)	0.0292 (8)
H1	−0.104 (5)	0.250 (4)	0.611 (4)	0.038 (11)
H2	0.156 (5)	0.220 (4)	0.640 (3)	0.036 (11)
H3	0.084 (6)	0.408 (4)	0.4135 (5)	0.045 (13)
H4	0.174 (6)	0.492 (4)	0.520 (4)	0.050 (13)

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

Br—Si	2.2362 (12)	Si—H1	1.32 (4)
Si—C	1.853 (4)	Si—H2	1.36 (4)
C—C^i	1.541 (8)		
C—Si—Br	107.68 (14)	Br—Si—H1	106.5 (15)
C^i—C—Si	113.6 (4)	Br—Si—H2	105.4 (14)
C—Si—H1	112.8 (16)	C—Si—H2	116.9 (15)
Br—Si—C—C^i	−61.7 (5)	Si^i—C^i—C—Si	180

Symmetry code: (i) $-x$, $1 - y$, $1 - z$.

Initial ψ-scan-based absorption corrections (North, Phillips & Mathews, 1968) were augmented by an empirical *DIFABS* ΔF-based correction (Walker & Stuart, 1983) in view of high residual electron density features (close to the Br atom).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: local programs. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1989). Software used to prepare material for publication: *SHELXL93*.

The authors are indebted to the Daresbury Laboratory for access to the Cambridge Structural Database. The Deutsche Forschungsgemeinschaft is thanked for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1041). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-Amino-1,2-dimethylimidazole

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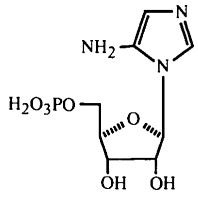
Abstract

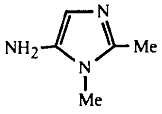
The determination of the structure of the title compound, $C_5H_9N_3$, provides the first geometrical data for 5-amino-4-unsubstituted imidazoles. The amino group is planar and inclined at 45 (3)° to the plane of the imidazole ring. The N3—C4 bond is slightly longer than that in imidazole and significantly longer than those in 5-nitroimidazoles. There is intermolecular hydrogen bonding between the amino substituents and the ring N atoms at position 3.

Comment

5-Aminoribonucleotide (AIR), (1), is an intermediate in the biosynthesis of purine ribonucleotides and thiamin (Mueller, Meyer, Rudolph, Davisson & Stubbe, 1994). Until recently, simple 5-aminoimidazoles were regarded as too unstable to be isolated and the properties of this fundamental heterocyclic system, including its structural features, remained unexplored (Lythgoe & Ramsden, 1994). In earlier studies we have described the preparation and isolation of several 5-amino-4-unsubstituted imidazoles (Al-Shaar, Gilmour, Lythgoe, McClenaghan & Ramsden, 1992; Al-Shaar, Chambers *et al.*, 1992; Humphries & Ramsden, 1995). We now report the molecular structure of the title compound, (2), and compare the results with those previously calculated (Al-Shaar, Gilmour *et al.*, 1992) using the semi-empirical AM1 method (Dewar, Zoebisch, Healy & Stewart, 1985).



(1)



(2)

Fig. 1 shows a perspective view of (2). A comparison of the bond lengths with those in antibacterial 1,2-dialkyl-5-nitroimidazoles (Blaton, Peeters & De Ranter, 1979*a*,*b*; Goldberg, 1982; Chasseaud, Henrick, Matthews, Scott & Wood, 1984; De Bondt, Blaton, Peeters & De Ranter, 1991; Germain, Declercq, Van Meerssche & Koch, 1977) shows that most bond lengths are similar. There is one significant difference: the N3—C4 bond in the 5-nitroimidazoles varies between 1.343 and 1.367 Å (average 1.356 Å) whereas in the 5-aminoimidazole (2) it is 1.400 (4) Å. The significantly shorter N3—C4 bond lengths in the nitroimidazoles, which are slightly shorter than the mean value for imidazoles (1.377 Å) found in the Cambridge Structural Database (Allen *et al.*, 1987), can be rationalized in terms of partial double-bond character resulting from resonance between the nitro substituent and N1. In the amino derivative (2), resonance between ring and substituent is not favoured. The amino N atom is displaced 0.044 (6) Å from the plane of the imidazole ring and the N6—H6*a*—H6*b* plane is inclined at 45 (3)° to the ring plane. This results in a structure which has bond lengths closer to those of imidazole (N3—C4 1.375 Å; McMullan, Epstein, Ruble & Craven, 1979). With one exception, the ring bond lengths in the crystal structure of (2) are 0.02–0.06 Å shorter than the values calculated by the AM1 method, and, as suggested in our earlier paper (Al-Shaar, Gilmour *et al.*, 1992), we believe that this is a result of the AM1 method overestimating the core-repulsion energies for multi-nitrogen heterocycles. Interestingly, the exception is the N3—C4 bond length which is calculated (1.388 Å) to be 0.012 Å shorter than observed [1.400 (4) Å]. The reason for this difference is not clear.

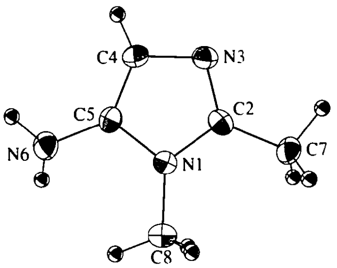


Fig. 1. Structure of (2) showing 50% probability displacement ellipsoids. H atoms are shown as circles of arbitrary radii.

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