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Hexaiododisilane

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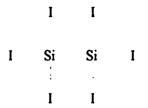
Abstract

The title compound, Si_2I_6, was prepared by the reaction of silicon tetraiodide with finely divided metallic silver and was subsequently purified by sublimation. The crystal structure can be regarded as an approximately dense packing of I atoms with the sequence *ABAC* with Si–Si dumb-bells occupying one-sixth of the octahedral sites. In an alternative description, the structure is composed of hexaiododisilane molecules with staggered conformation forming isolated, slightly distorted octahedra with Si–Si dumb-bells in the centres.

Comment

During the course of our investigations of silicon suboxides, the title compound was found to be a potential precursor for the preparation of silicon sesquioxide (Suresh & Padma, 1984; Belot *et al.*, 1991). The crystal structure of hexaiododisilane is the first of a hexahalogendisilane to be reported.



The hexaiododisilane molecules are arranged in a staggered conformation in which each Si atom is surrounded tetrahedrally by three I atoms and another Si atom. The I atoms form an *ABAC* layer structure where one-sixth of the octahedral sites are occupied by Si–Si dumb-bells. The site symmetry of hexaiododisilane is C_s and within the limits of experimental error the point-group symmetry is D_{3d}. The Si—I bond lengths in the title compound are in good agreement with the bond length reported for silicon tetraiodide (Hassel & Kringstad, 1931). The Si—Si bond length is in the same range as those of the analogous disilanes Si_2H_6 (Beagley *et al.*, 1972) and Si_2F_6 (Oberhammer, 1976), which were determined by gas-phase electron diffraction.

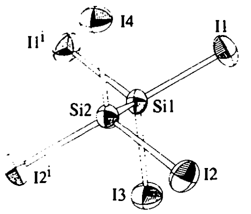


Fig. 1. A view of the staggered conformation of hexaiododisilane. Displacement ellipsoids are shown at the 50% probability level. [Symmetry code: (i) $x, \frac{1}{2} - y, z$.]

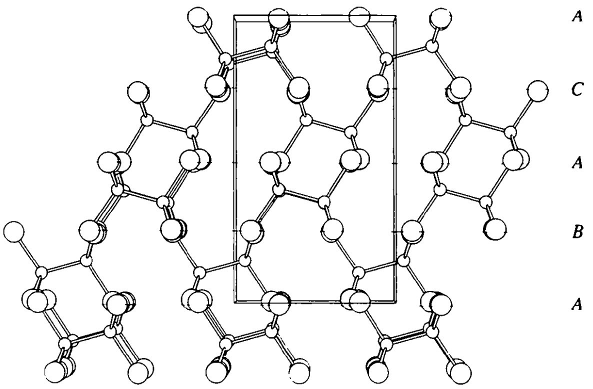


Fig. 2. Packing of I atoms with the sequence *ABAC* and Si–Si dumb-bells in one-sixth of the octahedral sites. Large circles are I atoms, small circles are Si atoms.

Experimental

Hexaiododisilane was prepared by a standard method (Friedel & Ladenburg, 1869; Schwarz & Pflugmacher, 1942; Petereit, 1964) in which silicon tetraiodide was reacted with finely divided metallic silver in an evacuated glass tube at 673 K for 3 h. Subsequent sublimation yields hexaiododisilane as colourless needles.

Crystal data

Si_2I_6
M_r = 817.58
Orthorhombic
Pnma
a = 13.702 (2) Å
b = 12.498 (2) Å
c = 7.8940 (10) Å
V = 1351.8 (4) Å^3
Z = 4
D_x = 4.017 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 21 reflections
θ = 0–30°
μ = 13.907 mm^{-1}
T = 293 (2) K
Needle
1 × 0.3 × 0.1 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
Variable $\omega/2\theta$ scans
Absorption correction: numerical (*SHELX76*; Sheldrick, 1976)
T_{min} = 0.0249, T_{max} = 0.0754
3665 measured reflections
1964 independent reflections

R_{int} = 0.0290
θ_{max} = 30.01°
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 11$
3 standard reflections
frequency: 60 min
intensity decay: none

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Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0608$
$wR(F^2) = 0.1650$
$S = 1.306$
1964 reflections
44 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0765P)^2 + 10.7170P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.75$ e Å^{-3}
$\Delta\rho_{\min} = -2.23$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993; Larson, 1970)
Extinction coefficient: 0.0019 (2)
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)*

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

	x	y	z	U_{eq}
I1	0.24321 (5)	0.41001 (5)	0.11088 (10)	0.0560 (3)
I2	0.00717 (5)	0.41080 (5)	−0.27616 (10)	0.0565 (3)
I3	−0.00093 (6)	1/4	0.22388 (13)	0.0553 (3)
I4	0.24813 (6)	1/4	−0.39933 (12)	0.0539 (3)
Si1	0.1479 (2)	1/4	0.0569 (4)	0.0349 (6)
Si2	0.1019 (2)	1/4	−0.2264 (4)	0.0339 (6)

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

Si1—I1	2.427 (2)	Si1—Si2	2.323 (4)
Si2—I2	2.425 (2)	Si1—I1^{i}	2.427 (2)
Si1—I3	2.428 (3)	Si2—I2^{i}	2.424 (2)
Si2—I4	2.424 (3)		
Si2—Si1—I1^{i}	108.37 (10)	Si1—Si2—I4	108.5 (2)
Si2—Si1—I1	108.37 (10)	Si1—Si2—I2^{i}	107.50 (10)
I1^{i}—Si1—I1	111.01 (13)	I4—Si2—I2^{i}	110.57 (9)
Si2—Si1—I3	107.2 (2)	Si1—Si2—I2	107.50 (10)
I1^{i}—Si1—I3	110.88 (9)	I4—Si2—I2	110.57 (9)
I1—Si1—I3	110.88 (9)	I2^{i}—Si2—I2	112.00 (13)

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

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software CELDIM*. Data reduction: *CAD*-4 *Software CADSHELL*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1971). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BR1117). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$K_2MoO_2As_2O_7$

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Abstract

The dipotassium molybdyl diarsenate(4−) structure is built up from MoO_6 octahedra and As_2O_7 groups sharing corners to form layers. The two O atoms of the molybdyl group remain unshared with short Mo—O distances (average 1.704 Å). The K^+ ions are located in the interlayer space. A relationship is found between the two-dimensional structure of the title compound and the three-dimensional melilite structure.

Commentaire

Dans le cadre de la recherche de nouveaux matériaux, à structure ouverte, nous avons précédemment explorés les systèmes *A*–*M*–As–O (*A* = alcalin, *M* = Nb, V, Al et Mo). Plusieurs composés ont été caractérisés et étudiés: $K_2Nb_2As_2O_{11}$ (Zid, Jouini, Jouini & Omezzine, 1988), $K_3NbAs_2O_9$ (Zid, Jouini, Jouini & Omezzine, 1989), KNb_4AsO_{13} (Haddad, Jouini, Jouini & Piffard, 1988), $NaVOAsO_4$ (Haddad, Jouini & Piffard, 1992), $RbAlAs_2O_7$ (Boughzala, Driss & Jouini, 1993), $NaAlAs_2O_7$ (Driss & Jouini, 1994) et $KMoO_2AsO_4$ (Zid & Jouini, 1995).

La comparaison de la structure de $K_2MoO_2As_2O_7$ avec celles de composés décrits antérieurement montre que ceux de formulation analogue connus $(NH_4)_2MoO_2P_2O_7$ (Averbuch-Pouchot, 1988) et $CsHMoO_2P_2O_7$ (Averbuch-Pouchot, 1989) ont la dimensionnalité 1.

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