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Crystal Structures of 2-Bromo-1,1,1,3,3,3-Hexamethyl-2-(Trimethylsilyl)Trisilane and 2-Bromo-1,1,1,3,3,3-Hexaisopropyl-2-(Triisopropylsilyl)Trisilane

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Crystal structures of 2-bromo-1,1,1,3,3,3-hexamethyl-2-(trimethylsilyl)trisilane and 2-bromo-1,1,1,3,3,3-hexaisopropyl-2-(triisopropylsilyl)trisilane

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CCDC references: 1854536; 1854535

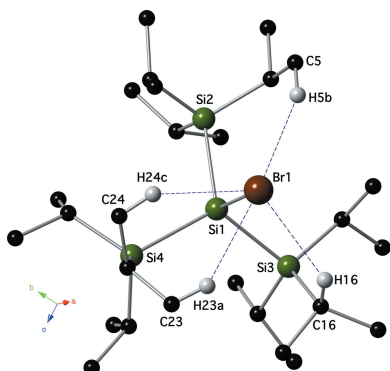
Supporting information: this article has supporting information at journals.iucr.org/e

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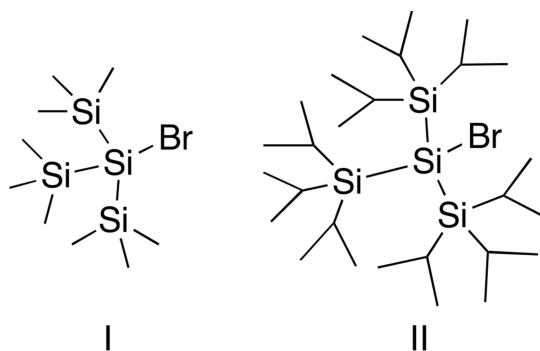
The synthesis and crystal structures of two tris(trialkylsilyl)silyl bromide compounds, C₉H₂₇BrSi₄ (**I**, HypSiBr) and C₂₇H₆₃BrSi₄ (**II**, TipSiBr), are described. Compound **I** was prepared in 85% yield by free-radical bromination of 1,1,1,3,3,3-hexamethyl-2-(trimethylsilyl)trisilane using bromobutane and 2,2'-azobis(2-methylpropionitrile) as a radical initiator at 333 K. The molecule possesses threefold rotational symmetry, with the central Si atom and the Br atom being located on the threefold rotation axis. The Si–Br bond distance is 2.2990 (12) Å and the Si–Si bond lengths are 2.3477 (8) Å. The Br–Si–Si bond angles are 104.83 (3)° and the Si–Si–Si bond angles are 113.69 (2)°, reflecting the steric hindrance inherent in the three trimethylsilyl groups attached to the central Si atom. Compound **II** was prepared in 55% yield by free-radical bromination of 1,1,1,3,3,3-hexaisopropyl-2-(triisopropylsilyl)trisilane using *N*-bromosuccinimide and 2,2'-azobis(2-methylpropionitrile) as a radical initiator at 353 K. Here the Si–Br bond length is 2.3185 (7) Å and the Si–Si bond lengths range from 2.443 (1) to 2.4628 (9) Å. The Br–Si–Si bond angles range from 98.44 (3) to 103.77 (3)°, indicating steric hindrance between the three triisopropylsilyl groups.

1. Chemical context

The steric and electronic effects of the tris(trimethylsilyl)silane group have been exploited for the synthesis and study of a variety of reactive centers including silylenes (Wendel *et al.*, 2017) and silylanions (Kayser *et al.*, 2002; Mechtler *et al.*, 2004; Zirngast *et al.*, 2008; Marschner, 2015). This sterically hindered group has been shown to lead to lower coordination by solvent when it is attached to organolithium compounds (Feil & Harder, 2003). It has also been used in organic synthesis to produce highly stereoselective aldol reactions leading to unique reactivity (Gati & Yamamoto, 2016). For this research we prepared tris(trimethylsilyl)silyl bromide (HypSiBr) as a precursor to vinyltris(trimethylsilyl)silane. The even bulkier tris(triisopropylsilyl)silyl bromide (TipSiBr) was prepared as a potential precursor to methoxytris(triisopropylsilyl)silane. Herein, we report on the crystal structures of these two sterically hindered silyl bromides 2-bromo-1,1,1,3,3,3-hexamethyl-2-(trimethylsilyl)trisilane (**I**), and 2-bromo-1,1,1,3,3,3-hexaisopropyl-2-(triisopropylsilyl)trisilane (**II**).



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2. Structural commentary

The molecular structure of compound **I** (HypSiBr), is shown in Fig. 1, and selected geometrical parameters are given in Table 1. The asymmetric unit is composed of one trimethylsilyl group, with the central silicon atom Si1 and the bromine atom Br1 lying on a threefold rotation axis. This supersilylbromide crystallized in the cubic space group $Pa\bar{3}$ with a central 4-coordinate silicon atom, Si1, that deviates slightly from an ideal tetrahedron due to the steric bulk of the attached trimethylsilyl (TMS) groups. The τ_4 descriptor for fourfold coordination around Si1 is 0.94 (where, for extreme forms, $\tau_4 = 0.00$ for square-planar, 1.00 for tetrahedral and 0.85 for trigonal-pyramidal; Yang *et al.*, 2007). Interestingly, the τ_4 descriptor for fourfold coordination around the TMS atom Si2 is 0.99, which demonstrates an ideal tetrahedral geometry around this silicon atom. The Si2–Si1–Si2^{i,ii} bond angle is 113.69 (2)° while the Br1–Si1–Si2 bond angle is 104.83 (3)°,

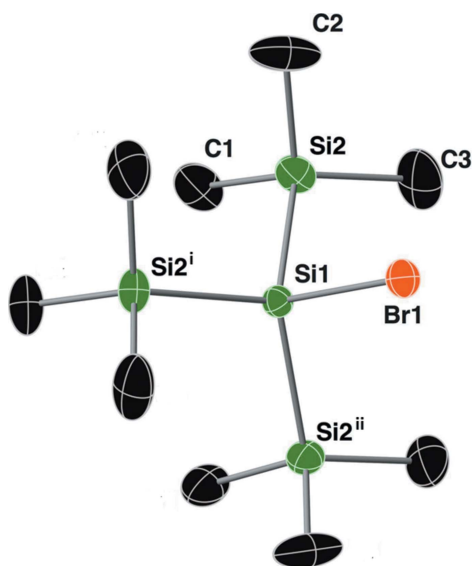


Figure 1

A view of the molecular structure of compound **I**, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen atoms have been omitted for clarity. Unlabelled atoms are related to the labelled atoms by threefold rotation symmetry [symmetry codes: (i) z, x, y ; (ii) y, z, x].

indicating that the trimethylsilyl groups are forced away from one another. The Si1–Br1 bond length is 2.2990 (12) Å. As for Si2, the C–Si2–C bond angles range from 107.1 (2) to 110.55 (17)°, while the C–Si2–Si1 bond angles range from 108.61 (10) to 110.16 (11)°.

The asymmetric unit of compound **II** (TipSiBr), is shown in Fig. 2, and selected geometrical parameters are given in Table 1. This compound crystallized in the triclinic space group $P\bar{1}$ with a central four-coordinate silicon atom, Si1, that deviates from the ideal tetrahedron as shown from its τ_4 descriptor for fourfold coordination of 0.90. The Br1–Si1–Si2/Si3/Si4 bond angles range from 98.44 (3) to 103.77 (3)°, and the Si1–Br1 bond distance is 2.3185 (7) Å, which is longer than that of compound **I** [2.2990 (12) Å]. The τ_4 descriptor values for atoms Si2, Si3 and Si4 (the silicon atoms of the triisopropylsilyl groups) are 0.96, 0.97 and 0.95, respectively, indicating that their coordination geometry is closest to an ideal tetrahedron.

3. Supramolecular features

There are no significant intermolecular contacts, other than weak van der Waals interactions, present in the crystals of compounds **I** or **II**. Compound **II**, however, contains four intramolecular C–H...Br hydrogen bonds (Table 2, Fig. 3). These hydrogen bonds contain $D\cdots A$ distances that range

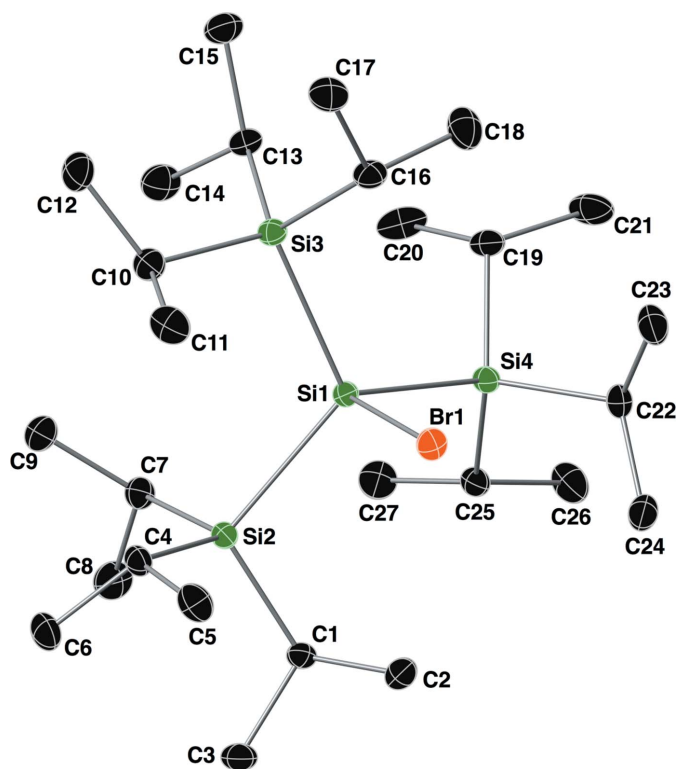


Figure 2

A view of the molecular structure of compound **II**, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level, and all hydrogen atoms have been omitted for clarity.

Table 1

Selected bond lengths (Å), bond angles (°) and the fourfold coordination descriptor, τ_4 ,^a for compounds **I** (HypSiBr), **II** (TipSiBr), **III** (HypSiCl) and **IV** (TipSiH).

Compound	I (HypSiBr)	II (TipSiBr)	III (HypSiCl) ^b	IV (TipSiH) ^c
Si1—X ^d	2.2990 (12)	2.3185 (7)	2.1248 (9)	1.608 (1)
Si1—Si ^e	2.3477 (8)	2.4430 (10), 2.4448 (10), 2.4628 (9)	2.3406 (6)	2.405 (1)
Si2—Si1—Si ^f	113.69 (2)	115.02 (4), 116.55 (3), 116.59 (4)	113.13 (2)	117.9 (1)
Si2—Si1—X ^d	104.83 (3)	98.44 (3) to 103.77 (3)	105.51 (2)	98.3 (1)
τ_4 of Si1	0.94	0.90	0.95	0.88

Notes: (a) Yang *et al.* (2007); (b) Kuzora *et al.* (2009); (c) X-ray data (Gaspar *et al.*, 1999); (d) X = Br for **I** and **II**, Cl for **III**, and H for **IV**; (e) $n = 2$ for **I**, **III** and **IV**, and 2, 3 and 4 for **II**; (f) $n = 2^i$ and 2^{ii} for **I**, **III** and **IV** [symmetry codes: (i) = z, x, y ; (ii) = y, z, x], and 2, 3 and 4 for **II**.

Table 2

Hydrogen-bond geometry (Å, °) for **II**.

D—H...A	D—H	H...A	D...A	D—H...A
C5—H5b...Br1	0.98	2.80	3.711 (3)	155
C16—H16...Br1	1.00	2.84	3.584 (3)	131
C23—H23a...Br1	0.98	2.87	3.685 (3)	142
C24—H24c...Br1	0.98	2.93	3.726 (3)	139

from from 3.584 (3) to 3.726 (3) Å, and D—H...A bond angles that range from 131 to 155°.

4. Database survey

The Cambridge Structural Database (CSD, version 5.39, February 2018; Groom *et al.*, 2016) contains 1398 structures containing a Si₃Si group. Of these, there are only 42 structures where the central silicon atom is bonded directly to a halogen.

Of particular interest to this work is the structure of tris(trimethylsilyl)chlorosilane (**III**, HypSiCl) [CSD refcode

QULWEA; Kuzora *et al.*, 2009], the isotopic chloro derivative of compound **I**, and the structure of (iPr₃Si)₃SiH (**IV**, TipSiH), isotopic with compounds **I** and **III**. The analysis of **IV** by both X-ray and neutron diffraction has been described by Gaspar *et al.* (1999). Table 1 contains pertinent bond lengths and bond angles for compounds **I**, **II**, **III** (HypSiCl) and **IV** (TipSiH).

For compounds **I** and **III** the Si—X bond lengths follow the expected trend with the Si1—Cl bond length of QULWEA at 2.1248 (9) Å compared to the Si1—Br1 bond length of 2.2990 (12) Å for compound **I**. The Si1—Si2 bond length of the bromo derivative **I** reported here is 2.3477 (8) Å, which is slightly longer than the Si1—Si2 bond length of the chloro derivative at 2.3406 (6) Å. The central silicon atom of the chloro derivative appears less sterically hindered with an Si2—Si1—Cl1 bond angle of 105.508 (18)° and Si2—Si1—Si2^{i,ii} bond angles of 113.126°, versus a smaller Si2—Si1—Br1 bond angle of 104.83 (3)° and a larger Si2—Si1—Si2^{i,ii} bond angle of 113.69 (2)° for compound **I** [symmetry codes: (i) z, x, y ; (ii) y, z, x]. The protio derivative (HypSiH) is a liquid at room temperature, and the structure of the iodo derivative (HypSiI) has not been deposited in the CSD.

The X-ray data for compound **IV** (TipSiH) was not found in the CSD, but the journal article (Gaspar *et al.*, 1999) contains all pertinent structural data to allow for a comparison with (iPr₃Si)₃SiBr, viz. compound **II** (TipSiBr). Like compounds **I** and **III**, compound **IV** crystallizes in the cubic space group $Pa\bar{3}$, and the molecule possesses threefold rotation symmetry. The presence of a small hydrogen atom bonded to the central silicon atom Si1 allows the three (iPr₃Si)— groups to push further away from one another, resulting in Si2—Si1—Si2^{i,ii} bond angles of 117.9 (1)° and Si2^{i,ii}—Si1—H bond angles of 98.3 (1)° [symmetry codes: (i) z, x, y ; (ii) y, z, x]. In **II**, the corresponding Si—Si—Si bond angles range from 115.02 (4) to 116.59 (4)° and the Si—Si—Br bond angles vary from 98.44 (3) to 103.77 (3)°.

5. Synthesis and crystallization

Compound I: Tris(trimethylsilyl)silane (2.0 g, 8.0 mmol) was added to an oven-dried nitrogen-flushed 250 ml Schlenk flask with a stir-bar. Bromobutane (2.0 g, 14.6 mmol) was filtered through a plug of silica gel in a Pasteur pipette and was transferred into the Schlenk flask. AIBN [2,2-azobis(2-methylpropionitrile); 20 mg] was then added to the flask, and the reaction was heated to 333 K using an oil bath and then

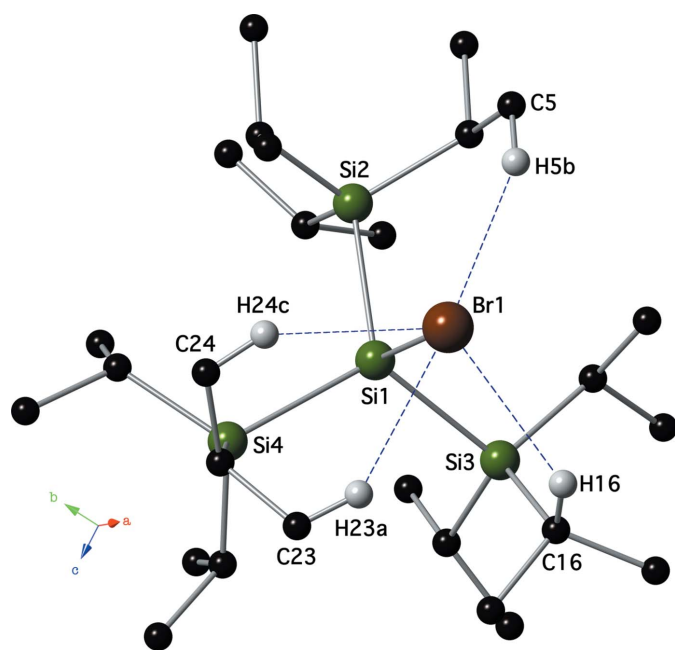


Figure 3

Intramolecular C—H...Br hydrogen bonds (blue dashed lines; see Table 2) present in compound **II**. For clarity, only the hydrogen atoms involved in a hydrogen bonding are shown.

Table 3
Experimental details.

	I	II
Crystal data		
Chemical formula	C ₉ H ₂₇ BrSi ₄	C ₂₇ H ₆₃ BrSi ₄
<i>M_r</i>	327.57	580.04
Crystal system, space group	Cubic, <i>Pa</i> $\bar{3}$	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	173	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	15.6497 (19), 15.6497 (19), 15.6497 (19)	8.4412 (4), 11.1336 (6), 18.8477 (10)
α , β , γ (°)	90, 90, 90	92.565 (4), 90.527 (4), 108.718 (4)
<i>V</i> (Å ³)	3832.8 (14)	1675.44 (15)
<i>Z</i>	8	2
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	2.37	1.38
Crystal size (mm)	0.45 × 0.24 × 0.14	0.38 × 0.10 × 0.02
Data collection		
Diffractometer	Bruker APEXII CCD	Bruker SMART APEX CCD area detector
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2014)	Multi-scan (<i>SADABS</i> ; Bruker, 2014)
<i>T</i> _{min} , <i>T</i> _{max}	0.571, 0.745	0.554, 0.674
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	11038, 1174, 953	21505, 6628, 4752
<i>R</i> _{int}	0.046	0.057
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.601	0.622
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> [<i>F</i> ²], <i>S</i>	0.028, 0.075, 1.05	0.037, 0.075, 1.01
No. of reflections	1174	6628
No. of parameters	46	307
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.31, -0.16	0.36, -0.28

Computer programs: *APEX2*, *SMART* and *SAINT* (Bruker, 2014), *SHELXS97* and *SHELXTL* (Sheldrick, 2008), *SHELXT2013* (Sheldrick, 2015a), *SHELXL2017* (Sheldrick, 2015b), *OLEX2* (Dolomanov *et al.*, 2009; Bourhis *et al.*, 2015) and *CrystalMaker* (Palmer, 2007).

heating was stopped. After stirring the reaction overnight at room temperature, GC–MS analysis of a sample indicated incomplete reaction and more AIBN (11 mg) was added to the flask. The reaction was heated once more to 333 K for 1 h. Analysis by GC–MS now indicated that the reaction was complete. The flask was placed in a freezer at 243 K and colourless block-like crystals of **I** formed overnight. Removal of the solvent *in vacuo* yielded 2.2 g (85%). ¹H NMR (300 MHz, chloroform-*d*) δ 0.24 (*s*, 27H); ¹³C NMR (75 MHz, chloroform-*d*) δ -0.51 ppm; GC–MS: 11.24 min, *m/z* = 328, base peak: 73.

Compound II: Tris(triisopropylsilyl)silane (110 mg, 0.22 mmol) was dissolved in freshly distilled benzene (10 ml) along with NBS (45 mg) and AIBN (2 mg, initiator). The mixture was heated using an oil bath at 333 K for 30 min, when GC–MS analysis indicated that no reaction had occurred. At this point the solution was heated with a heat gun until the reaction mixture turned slightly yellow. The yellow colour dissipated in less than 1 min. Analysis of the reaction mixture by ¹H NMR indicated that only 60% of the starting material had been consumed. An additional amount of NBS (*N*-bromosuccinimide; 20 mg) was added to the reaction flask, and the solution was again heated with a heat gun. The product was isolated by removing the solvent *in vacuo* and extracting the product from the crude reaction mixture with pentane. The pentane solution was filtered through glass wool, concentrated and weighed (135 mg). Analysis of the product with ¹H NMR indicated this was 90% pure. The product was further purified by dissolving this solid in 1 ml pentane,

cooling to 195 K and isolating the colourless needle-like crystals of **II** by removing the solvent with a syringe, washing with pentane and drying *in vacuo* (yield 62 mg, 55%). ¹H NMR (300 MHz, C₆D₆) δ 1.34 (*d*, *J* = 7.3 Hz, 54H), 1.66 (*heptet*, *J* = 7.4 Hz, 9H); ¹³C NMR (75 MHz, chloroform-*d*) δ 16.4, 21.6; HRMS for C₁₇H₆₃BrSi₄ calculated 535.2642 (*M* – C₃H₇), found 535.2641.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. For both compounds the hydrogen atoms were placed in calculated positions and refined using a riding model: C–H = 0.98–1.00 Å with *U*_{iso}(H) = 1.5*U*_{eq}(C–methyl) and 1.2*U*_{eq}(C) for other H atoms.

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supporting information

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Crystal structures of 2-bromo-1,1,1,3,3,3-hexamethyl-2-(trimethylsilyl)trisilane and 2-bromo-1,1,1,3,3,3-hexaisopropyl-2-(triisopropylsilyl)trisilane

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Computing details

Data collection: *APEX2* (Bruker, 2014) for (I); *SMART* (Bruker, 2014) for (II). For both structures, cell refinement: *SAINTE* (Bruker, 2014); data reduction: *SAINTE* (Bruker, 2014). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008) for (I); *SHELXT2013* (Sheldrick, 2015a) for (II). Program(s) used to refine structure: *SHELXL2017* (Sheldrick, 2015b) for (I); *SHELXL2014* (Sheldrick, 2015b) for (II). Molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009; Bourhis *et al.*, 2015) for (I); *SHELXTL* (Sheldrick, 2008) for (II). Software used to prepare material for publication: *CrystalMaker* (Palmer, 2007) for (I); *SHELXTL* (Sheldrick, 2008) for (II).

2-Bromo-1,1,1,3,3,3-hexamethyl-2-(trimethylsilyl)trisilane (I)

Crystal data

$C_9H_{27}BrSi_4$

$M_r = 327.57$

Cubic, $Pa\bar{3}$

$a = 15.6497$ (19) Å

$V = 3832.8$ (14) Å³

$Z = 8$

$F(000) = 1376$

$D_x = 1.135$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3863 reflections

$\theta = 2.3$ – 25.3°

$\mu = 2.37$ mm⁻¹

$T = 173$ K

Block, colourless

$0.45 \times 0.24 \times 0.14$ mm

Data collection

Bruker APEXII CCD
diffractometer

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2014)

$T_{\min} = 0.571$, $T_{\max} = 0.745$

11038 measured reflections

1174 independent reflections

953 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.046$

$\theta_{\max} = 25.3^\circ$, $\theta_{\min} = 2.3^\circ$

$h = -18 \rightarrow 16$

$k = -18 \rightarrow 17$

$l = -15 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.075$

$S = 1.05$

1174 reflections

46 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0349P)^2 + 1.4333P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$

$$\Delta\rho_{\max} = 0.31 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$$

Special details

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

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.74011 (2)	0.74011 (2)	0.74011 (2)	0.04337 (17)
Si1	0.65529 (4)	0.65529 (4)	0.65529 (4)	0.0327 (3)
Si2	0.69238 (5)	0.51472 (4)	0.69229 (5)	0.0473 (2)
C1	0.6249 (2)	0.43945 (18)	0.6286 (2)	0.0734 (10)
H1A	0.6331	0.4506	0.5675	0.110*
H1B	0.5646	0.4478	0.6434	0.110*
H1C	0.6415	0.3805	0.6414	0.110*
C2	0.8079 (2)	0.49575 (19)	0.6707 (3)	0.0943 (14)
H2A	0.8227	0.4370	0.6866	0.141*
H2B	0.8422	0.5358	0.7044	0.141*
H2C	0.8194	0.5044	0.6098	0.141*
C3	0.6752 (3)	0.49746 (19)	0.8090 (2)	0.0953 (13)
H3A	0.6142	0.5032	0.8222	0.143*
H3B	0.7076	0.5401	0.8414	0.143*
H3C	0.6946	0.4401	0.8247	0.143*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.04337 (17)	0.04337 (17)	0.04337 (17)	-0.00621 (10)	-0.00621 (10)	-0.00621 (10)
Si1	0.0327 (3)	0.0327 (3)	0.0327 (3)	-0.0008 (3)	-0.0008 (3)	-0.0008 (3)
Si2	0.0529 (5)	0.0317 (4)	0.0573 (5)	-0.0013 (3)	-0.0106 (4)	0.0008 (3)
C1	0.087 (2)	0.0426 (16)	0.091 (2)	-0.0092 (16)	-0.0304 (19)	-0.0051 (16)
C2	0.056 (2)	0.0494 (19)	0.178 (4)	0.0145 (16)	-0.007 (2)	0.001 (2)
C3	0.168 (4)	0.052 (2)	0.066 (2)	0.006 (2)	-0.019 (2)	0.0183 (17)

Geometric parameters (\AA , $^\circ$)

Br1—Si1	2.2990 (12)	Si2—C1	1.870 (3)
Si1—Si2 ⁱ	2.3478 (8)	Si2—C2	1.862 (3)
Si1—Si2	2.3477 (8)	Si2—C3	1.866 (3)
Si1—Si2 ⁱⁱ	2.3478 (8)		
Br1—Si1—Si2	104.83 (3)	C1—Si2—Si1	108.61 (10)
Br1—Si1—Si2 ⁱⁱ	104.83 (3)	C2—Si2—Si1	110.16 (11)
Br1—Si1—Si2 ⁱ	104.83 (3)	C2—Si2—C1	110.55 (17)

Si2—Si1—Si2 ⁱⁱ	113.69 (2)	C2—Si2—C3	107.12 (19)
Si2—Si1—Si2 ⁱ	113.69 (3)	C3—Si2—Si1	109.96 (10)
Si2 ⁱ —Si1—Si2 ⁱⁱ	113.69 (2)	C3—Si2—C1	110.45 (16)

Symmetry codes: (i) *y*, *z*, *x*; (ii) *z*, *x*, *y*.

2-Bromo-1,1,1,3,3,3-hexaisopropyl-2-(triisopropylsilyl)trisilane (II)

Crystal data

$C_{27}H_{63}BrSi_4$	$Z = 2$
$M_r = 580.04$	$F(000) = 632$
Triclinic, $P\bar{1}$	$D_x = 1.150 \text{ Mg m}^{-3}$
$a = 8.4412 (4) \text{ \AA}$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$b = 11.1336 (6) \text{ \AA}$	Cell parameters from 8192 reflections
$c = 18.8477 (10) \text{ \AA}$	$\theta = 2.0\text{--}26.0^\circ$
$\alpha = 92.565 (4)^\circ$	$\mu = 1.38 \text{ mm}^{-1}$
$\beta = 90.527 (4)^\circ$	$T = 173 \text{ K}$
$\gamma = 108.718 (4)^\circ$	Needles, colourless
$V = 1675.44 (15) \text{ \AA}^3$	$0.38 \times 0.10 \times 0.02 \text{ mm}$

Data collection

Bruker SMART APEX CCD area detector diffractometer	21505 measured reflections
Radiation source: sealed tube	6628 independent reflections
Detector resolution: 8 pixels mm^{-1}	4752 reflections with $I > 2\sigma(I)$
ω and ϕ scans	$R_{\text{int}} = 0.057$
Absorption correction: multi-scan (SADABS; Bruker, 2014)	$\theta_{\text{max}} = 26.3^\circ$, $\theta_{\text{min}} = 1.1^\circ$
$T_{\text{min}} = 0.554$, $T_{\text{max}} = 0.674$	$h = -10 \rightarrow 10$
	$k = -13 \rightarrow 13$
	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.037$	H-atom parameters constrained
$wR(F^2) = 0.075$	$w = 1/[\sigma^2(F_o^2) + (0.0274P)^2 + 0.3128P]$
$S = 1.01$	where $P = (F_o^2 + 2F_c^2)/3$
6628 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
307 parameters	$\Delta\rho_{\text{max}} = 0.36 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.28 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

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

Refinement. All H atoms were positioned geometrically and refined using a riding model with C—H = 0.95–0.99 \AA and $wA_{\text{H}} U_{\text{iso}}(\text{H}) = 1.2$ (1.5 for methyl groups) times $U_{\text{eq}}(\text{C})$.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	1.06221 (3)	0.19737 (3)	0.24790 (2)	0.02768 (9)

Si1	0.77723 (8)	0.16573 (6)	0.25116 (3)	0.01926 (16)
Si2	0.72101 (8)	0.26469 (7)	0.14487 (4)	0.02137 (17)
Si3	0.67829 (9)	-0.06634 (7)	0.24791 (4)	0.02310 (17)
Si4	0.74993 (9)	0.27556 (7)	0.36447 (4)	0.02177 (17)
C1	0.8269 (3)	0.4443 (2)	0.15761 (13)	0.0249 (6)
H1	0.7549	0.4757	0.1905	0.030*
C2	1.0018 (3)	0.4866 (3)	0.19270 (15)	0.0349 (7)
H2A	1.0777	0.4575	0.1626	0.052*
H2B	0.9962	0.4498	0.2393	0.052*
H2C	1.0435	0.5794	0.1987	0.052*
C3	0.8371 (4)	0.5176 (3)	0.08928 (14)	0.0335 (7)
H3A	0.8753	0.6091	0.1015	0.050*
H3B	0.7262	0.4931	0.0660	0.050*
H3C	0.9162	0.4972	0.0570	0.050*
C4	0.7935 (3)	0.1980 (3)	0.06084 (12)	0.0266 (6)
H4	0.7464	0.1034	0.0618	0.032*
C5	0.9832 (3)	0.2301 (3)	0.05683 (14)	0.0371 (7)
H5A	1.0101	0.1830	0.0161	0.056*
H5B	1.0268	0.2065	0.1006	0.056*
H5C	1.0345	0.3214	0.0513	0.056*
C6	0.7282 (4)	0.2347 (3)	-0.00855 (13)	0.0388 (7)
H6A	0.7814	0.3257	-0.0146	0.058*
H6B	0.6067	0.2155	-0.0066	0.058*
H6C	0.7549	0.1862	-0.0487	0.058*
C7	0.4836 (3)	0.2270 (2)	0.13810 (14)	0.0287 (6)
H7	0.4439	0.2136	0.1879	0.034*
C8	0.4264 (4)	0.3368 (3)	0.11280 (17)	0.0429 (8)
H8A	0.4536	0.3500	0.0627	0.064*
H8B	0.4841	0.4146	0.1414	0.064*
H8C	0.3053	0.3155	0.1182	0.064*
C9	0.3878 (3)	0.1029 (3)	0.09537 (15)	0.0394 (7)
H9A	0.2692	0.0772	0.1073	0.059*
H9B	0.4342	0.0360	0.1073	0.059*
H9C	0.3991	0.1169	0.0444	0.059*
C10	0.6700 (3)	-0.1257 (3)	0.15145 (13)	0.0304 (6)
H10	0.6105	-0.0769	0.1245	0.036*
C11	0.8446 (4)	-0.0949 (3)	0.11986 (15)	0.0402 (8)
H11A	0.9038	-0.1481	0.1406	0.060*
H11B	0.9078	-0.0052	0.1305	0.060*
H11C	0.8337	-0.1118	0.0683	0.060*
C12	0.5697 (4)	-0.2671 (3)	0.13666 (15)	0.0423 (8)
H12A	0.5798	-0.2910	0.0866	0.063*
H12B	0.4517	-0.2814	0.1470	0.063*
H12C	0.6138	-0.3189	0.1669	0.063*
C13	0.4641 (3)	-0.1251 (2)	0.28895 (14)	0.0278 (6)
H13	0.4741	-0.0754	0.3352	0.033*
C14	0.3289 (4)	-0.0951 (3)	0.24525 (16)	0.0426 (8)
H14A	0.3107	-0.1440	0.1997	0.064*

H14B	0.3651	-0.0041	0.2367	0.064*
H14C	0.2244	-0.1178	0.2714	0.064*
C15	0.4016 (4)	-0.2660 (3)	0.30713 (16)	0.0402 (7)
H15A	0.2967	-0.2836	0.3327	0.060*
H15B	0.4859	-0.2842	0.3371	0.060*
H15C	0.3827	-0.3196	0.2632	0.060*
C16	0.8392 (3)	-0.1171 (2)	0.29934 (14)	0.0292 (6)
H16	0.9513	-0.0608	0.2850	0.035*
C17	0.8383 (4)	-0.2537 (3)	0.28398 (15)	0.0383 (7)
H17A	0.9229	-0.2705	0.3143	0.057*
H17B	0.8637	-0.2649	0.2340	0.057*
H17C	0.7277	-0.3129	0.2938	0.057*
C18	0.8317 (4)	-0.0933 (3)	0.38032 (15)	0.0461 (8)
H18A	0.7258	-0.1494	0.3976	0.069*
H18B	0.8392	-0.0047	0.3909	0.069*
H18C	0.9252	-0.1109	0.4039	0.069*
C19	0.5967 (3)	0.1584 (2)	0.42272 (13)	0.0290 (6)
H19	0.6217	0.0766	0.4166	0.035*
C20	0.4130 (4)	0.1287 (3)	0.40018 (18)	0.0467 (8)
H20A	0.3450	0.0522	0.4228	0.070*
H20B	0.4012	0.1145	0.3484	0.070*
H20C	0.3751	0.2003	0.4149	0.070*
C21	0.6171 (4)	0.1952 (3)	0.50277 (14)	0.0486 (9)
H21A	0.5929	0.2749	0.5118	0.073*
H21B	0.7321	0.2064	0.5185	0.073*
H21C	0.5392	0.1280	0.5290	0.073*
C22	0.9557 (3)	0.3395 (2)	0.41777 (13)	0.0286 (6)
H22	0.9246	0.3756	0.4631	0.034*
C23	1.0335 (4)	0.2414 (3)	0.44175 (15)	0.0397 (7)
H23A	1.0703	0.2024	0.4002	0.059*
H23B	0.9504	0.1757	0.4669	0.059*
H23C	1.1299	0.2831	0.4736	0.059*
C24	1.0893 (3)	0.4526 (3)	0.38677 (14)	0.0364 (7)
H24A	1.1806	0.4887	0.4218	0.055*
H24B	1.0390	0.5175	0.3750	0.055*
H24C	1.1334	0.4236	0.3437	0.055*
C25	0.6831 (3)	0.4212 (2)	0.34999 (13)	0.0258 (6)
H25	0.7695	0.4766	0.3192	0.031*
C26	0.6873 (4)	0.5007 (3)	0.41963 (14)	0.0450 (8)
H26A	0.6597	0.5772	0.4092	0.067*
H26B	0.7994	0.5251	0.4417	0.067*
H26C	0.6053	0.4502	0.4522	0.067*
C27	0.5152 (4)	0.3988 (3)	0.31189 (15)	0.0392 (7)
H27A	0.4243	0.3572	0.3434	0.059*
H27B	0.5082	0.3445	0.2688	0.059*
H27C	0.5050	0.4803	0.2990	0.059*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02088 (14)	0.03279 (18)	0.02927 (15)	0.00846 (11)	0.00155 (11)	0.00149 (12)
Si1	0.0195 (4)	0.0190 (4)	0.0191 (4)	0.0060 (3)	0.0012 (3)	0.0007 (3)
Si2	0.0215 (4)	0.0220 (4)	0.0204 (4)	0.0066 (3)	0.0003 (3)	0.0017 (3)
Si3	0.0256 (4)	0.0192 (4)	0.0247 (4)	0.0076 (3)	0.0019 (3)	0.0009 (3)
Si4	0.0255 (4)	0.0205 (4)	0.0195 (4)	0.0075 (3)	0.0028 (3)	0.0008 (3)
C1	0.0272 (14)	0.0233 (15)	0.0229 (14)	0.0060 (12)	0.0038 (11)	0.0043 (11)
C2	0.0326 (16)	0.0281 (17)	0.0391 (17)	0.0031 (13)	-0.0053 (13)	0.0016 (13)
C3	0.0416 (18)	0.0255 (16)	0.0336 (16)	0.0103 (13)	0.0054 (13)	0.0078 (13)
C4	0.0325 (15)	0.0280 (16)	0.0199 (13)	0.0110 (12)	-0.0005 (11)	-0.0002 (11)
C5	0.0398 (18)	0.049 (2)	0.0266 (15)	0.0205 (15)	0.0080 (13)	0.0018 (14)
C6	0.0447 (18)	0.047 (2)	0.0228 (15)	0.0122 (15)	-0.0018 (13)	-0.0007 (14)
C7	0.0234 (15)	0.0325 (17)	0.0306 (15)	0.0091 (12)	-0.0023 (12)	0.0033 (13)
C8	0.0280 (16)	0.044 (2)	0.059 (2)	0.0136 (14)	-0.0057 (15)	0.0083 (16)
C9	0.0287 (16)	0.0406 (19)	0.0425 (18)	0.0022 (14)	-0.0059 (13)	0.0037 (14)
C10	0.0392 (17)	0.0255 (16)	0.0284 (15)	0.0138 (13)	0.0009 (13)	-0.0026 (12)
C11	0.053 (2)	0.0409 (19)	0.0339 (16)	0.0245 (15)	0.0101 (15)	0.0006 (14)
C12	0.055 (2)	0.0358 (19)	0.0371 (17)	0.0182 (16)	-0.0091 (15)	-0.0096 (14)
C13	0.0281 (15)	0.0207 (15)	0.0328 (15)	0.0053 (12)	0.0030 (12)	0.0029 (12)
C14	0.0303 (17)	0.044 (2)	0.052 (2)	0.0098 (14)	0.0021 (14)	0.0088 (15)
C15	0.0404 (18)	0.0301 (17)	0.0447 (18)	0.0030 (14)	0.0061 (14)	0.0067 (14)
C16	0.0312 (15)	0.0220 (16)	0.0371 (16)	0.0119 (12)	-0.0010 (12)	0.0045 (12)
C17	0.0431 (18)	0.0310 (18)	0.0456 (18)	0.0180 (14)	0.0007 (15)	0.0072 (14)
C18	0.062 (2)	0.042 (2)	0.0410 (18)	0.0268 (17)	-0.0133 (16)	-0.0041 (15)
C19	0.0364 (16)	0.0218 (16)	0.0287 (15)	0.0086 (12)	0.0091 (12)	0.0038 (12)
C20	0.0369 (18)	0.0338 (19)	0.069 (2)	0.0089 (14)	0.0210 (16)	0.0101 (16)
C21	0.075 (2)	0.036 (2)	0.0324 (17)	0.0132 (17)	0.0229 (16)	0.0069 (14)
C22	0.0327 (16)	0.0310 (17)	0.0212 (14)	0.0097 (13)	-0.0035 (12)	-0.0036 (12)
C23	0.0491 (19)	0.042 (2)	0.0312 (16)	0.0197 (15)	-0.0109 (14)	-0.0033 (14)
C24	0.0348 (17)	0.0362 (18)	0.0322 (16)	0.0041 (14)	-0.0043 (13)	-0.0040 (13)
C25	0.0347 (16)	0.0231 (15)	0.0211 (13)	0.0117 (12)	0.0018 (12)	-0.0006 (11)
C26	0.077 (2)	0.0380 (19)	0.0302 (16)	0.0336 (17)	-0.0023 (16)	-0.0064 (14)
C27	0.0388 (18)	0.0384 (19)	0.0464 (18)	0.0213 (14)	-0.0010 (14)	0.0007 (15)

Geometric parameters (\AA , $^\circ$)

Br1—Si1	2.3185 (7)	C12—H12B	0.9800
Si1—Si2	2.4430 (10)	C12—H12C	0.9800
Si1—Si3	2.4448 (10)	C13—C14	1.531 (4)
Si1—Si4	2.4628 (9)	C13—C15	1.541 (4)
Si2—C4	1.908 (2)	C13—H13	1.0000
Si2—C7	1.913 (3)	C14—H14A	0.9800
Si2—C1	1.914 (3)	C14—H14B	0.9800
Si3—C10	1.899 (3)	C14—H14C	0.9800
Si3—C13	1.899 (3)	C15—H15A	0.9800
Si3—C16	1.903 (3)	C15—H15B	0.9800

Si4—C22	1.909 (3)	C15—H15C	0.9800
Si4—C25	1.910 (3)	C16—C17	1.533 (4)
Si4—C19	1.911 (3)	C16—C18	1.543 (4)
C1—C2	1.532 (3)	C16—H16	1.0000
C1—C3	1.543 (4)	C17—H17A	0.9800
C1—H1	1.0000	C17—H17B	0.9800
C2—H2A	0.9800	C17—H17C	0.9800
C2—H2B	0.9800	C18—H18A	0.9800
C2—H2C	0.9800	C18—H18B	0.9800
C3—H3A	0.9800	C18—H18C	0.9800
C3—H3B	0.9800	C19—C20	1.530 (4)
C3—H3C	0.9800	C19—C21	1.539 (4)
C4—C5	1.528 (4)	C19—H19	1.0000
C4—C6	1.536 (4)	C20—H20A	0.9800
C4—H4	1.0000	C20—H20B	0.9800
C5—H5A	0.9800	C20—H20C	0.9800
C5—H5B	0.9800	C21—H21A	0.9800
C5—H5C	0.9800	C21—H21B	0.9800
C6—H6A	0.9800	C21—H21C	0.9800
C6—H6B	0.9800	C22—C23	1.525 (4)
C6—H6C	0.9800	C22—C24	1.538 (4)
C7—C9	1.543 (4)	C22—H22	1.0000
C7—C8	1.544 (4)	C23—H23A	0.9800
C7—H7	1.0000	C23—H23B	0.9800
C8—H8A	0.9800	C23—H23C	0.9800
C8—H8B	0.9800	C24—H24A	0.9800
C8—H8C	0.9800	C24—H24B	0.9800
C9—H9A	0.9800	C24—H24C	0.9800
C9—H9B	0.9800	C25—C27	1.525 (4)
C9—H9C	0.9800	C25—C26	1.543 (3)
C10—C11	1.534 (4)	C25—H25	1.0000
C10—C12	1.541 (4)	C26—H26A	0.9800
C10—H10	1.0000	C26—H26B	0.9800
C11—H11A	0.9800	C26—H26C	0.9800
C11—H11B	0.9800	C27—H27A	0.9800
C11—H11C	0.9800	C27—H27B	0.9800
C12—H12A	0.9800	C27—H27C	0.9800
Br1—Si1—Si2	103.77 (3)	C10—C12—H12C	109.5
Br1—Si1—Si3	98.44 (3)	H12A—C12—H12C	109.5
Si2—Si1—Si3	116.55 (3)	H12B—C12—H12C	109.5
Br1—Si1—Si4	102.65 (3)	C14—C13—C15	109.5 (2)
Si2—Si1—Si4	115.02 (4)	C14—C13—Si3	112.65 (19)
Si3—Si1—Si4	116.59 (4)	C15—C13—Si3	116.31 (18)
C4—Si2—C7	108.70 (11)	C14—C13—H13	105.9
C4—Si2—C1	111.49 (12)	C15—C13—H13	105.9
C7—Si2—C1	109.69 (11)	Si3—C13—H13	105.9
C4—Si2—Si1	112.06 (9)	C13—C14—H14A	109.5

C7—Si2—Si1	106.40 (9)	C13—C14—H14B	109.5
C1—Si2—Si1	108.35 (8)	H14A—C14—H14B	109.5
C10—Si3—C13	111.22 (12)	C13—C14—H14C	109.5
C10—Si3—C16	109.44 (12)	H14A—C14—H14C	109.5
C13—Si3—C16	111.50 (12)	H14B—C14—H14C	109.5
C10—Si3—Si1	107.75 (9)	C13—C15—H15A	109.5
C13—Si3—Si1	109.78 (8)	C13—C15—H15B	109.5
C16—Si3—Si1	106.98 (9)	H15A—C15—H15B	109.5
C22—Si4—C25	104.78 (12)	C13—C15—H15C	109.5
C22—Si4—C19	106.42 (12)	H15A—C15—H15C	109.5
C25—Si4—C19	111.59 (12)	H15B—C15—H15C	109.5
C22—Si4—Si1	112.92 (8)	C17—C16—C18	108.9 (2)
C25—Si4—Si1	111.74 (8)	C17—C16—Si3	116.29 (19)
C19—Si4—Si1	109.24 (8)	C18—C16—Si3	112.56 (18)
C2—C1—C3	107.7 (2)	C17—C16—H16	106.1
C2—C1—Si2	115.52 (18)	C18—C16—H16	106.1
C3—C1—Si2	114.54 (18)	Si3—C16—H16	106.1
C2—C1—H1	106.1	C16—C17—H17A	109.5
C3—C1—H1	106.1	C16—C17—H17B	109.5
Si2—C1—H1	106.1	H17A—C17—H17B	109.5
C1—C2—H2A	109.5	C16—C17—H17C	109.5
C1—C2—H2B	109.5	H17A—C17—H17C	109.5
H2A—C2—H2B	109.5	H17B—C17—H17C	109.5
C1—C2—H2C	109.5	C16—C18—H18A	109.5
H2A—C2—H2C	109.5	C16—C18—H18B	109.5
H2B—C2—H2C	109.5	H18A—C18—H18B	109.5
C1—C3—H3A	109.5	C16—C18—H18C	109.5
C1—C3—H3B	109.5	H18A—C18—H18C	109.5
H3A—C3—H3B	109.5	H18B—C18—H18C	109.5
C1—C3—H3C	109.5	C20—C19—C21	109.0 (2)
H3A—C3—H3C	109.5	C20—C19—Si4	113.92 (19)
H3B—C3—H3C	109.5	C21—C19—Si4	114.62 (19)
C5—C4—C6	108.5 (2)	C20—C19—H19	106.2
C5—C4—Si2	114.13 (17)	C21—C19—H19	106.2
C6—C4—Si2	114.24 (19)	Si4—C19—H19	106.2
C5—C4—H4	106.5	C19—C20—H20A	109.5
C6—C4—H4	106.5	C19—C20—H20B	109.5
Si2—C4—H4	106.5	H20A—C20—H20B	109.5
C4—C5—H5A	109.5	C19—C20—H20C	109.5
C4—C5—H5B	109.5	H20A—C20—H20C	109.5
H5A—C5—H5B	109.5	H20B—C20—H20C	109.5
C4—C5—H5C	109.5	C19—C21—H21A	109.5
H5A—C5—H5C	109.5	C19—C21—H21B	109.5
H5B—C5—H5C	109.5	H21A—C21—H21B	109.5
C4—C6—H6A	109.5	C19—C21—H21C	109.5
C4—C6—H6B	109.5	H21A—C21—H21C	109.5
H6A—C6—H6B	109.5	H21B—C21—H21C	109.5
C4—C6—H6C	109.5	C23—C22—C24	110.6 (2)

H6A—C6—H6C	109.5	C23—C22—Si4	116.47 (19)
H6B—C6—H6C	109.5	C24—C22—Si4	115.61 (18)
C9—C7—C8	109.7 (2)	C23—C22—H22	104.1
C9—C7—Si2	115.43 (18)	C24—C22—H22	104.1
C8—C7—Si2	114.49 (19)	Si4—C22—H22	104.1
C9—C7—H7	105.4	C22—C23—H23A	109.5
C8—C7—H7	105.4	C22—C23—H23B	109.5
Si2—C7—H7	105.4	H23A—C23—H23B	109.5
C7—C8—H8A	109.5	C22—C23—H23C	109.5
C7—C8—H8B	109.5	H23A—C23—H23C	109.5
H8A—C8—H8B	109.5	H23B—C23—H23C	109.5
C7—C8—H8C	109.5	C22—C24—H24A	109.5
H8A—C8—H8C	109.5	C22—C24—H24B	109.5
H8B—C8—H8C	109.5	H24A—C24—H24B	109.5
C7—C9—H9A	109.5	C22—C24—H24C	109.5
C7—C9—H9B	109.5	H24A—C24—H24C	109.5
H9A—C9—H9B	109.5	H24B—C24—H24C	109.5
C7—C9—H9C	109.5	C27—C25—C26	108.5 (2)
H9A—C9—H9C	109.5	C27—C25—Si4	117.05 (18)
H9B—C9—H9C	109.5	C26—C25—Si4	112.35 (18)
C11—C10—C12	110.3 (2)	C27—C25—H25	106.0
C11—C10—Si3	112.34 (19)	C26—C25—H25	106.0
C12—C10—Si3	115.08 (19)	Si4—C25—H25	106.0
C11—C10—H10	106.1	C25—C26—H26A	109.5
C12—C10—H10	106.1	C25—C26—H26B	109.5
Si3—C10—H10	106.1	H26A—C26—H26B	109.5
C10—C11—H11A	109.5	C25—C26—H26C	109.5
C10—C11—H11B	109.5	H26A—C26—H26C	109.5
H11A—C11—H11B	109.5	H26B—C26—H26C	109.5
C10—C11—H11C	109.5	C25—C27—H27A	109.5
H11A—C11—H11C	109.5	C25—C27—H27B	109.5
H11B—C11—H11C	109.5	H27A—C27—H27B	109.5
C10—C12—H12A	109.5	C25—C27—H27C	109.5
C10—C12—H12B	109.5	H27A—C27—H27C	109.5
H12A—C12—H12B	109.5	H27B—C27—H27C	109.5
C13—Si3—C10—C11	-172.26 (19)	C10—Si3—C13—C14	-52.9 (2)
C16—Si3—C10—C11	-48.6 (2)	C16—Si3—C13—C14	-175.32 (19)
Si1—Si3—C10—C11	67.4 (2)	Si1—Si3—C13—C14	66.3 (2)
C13—Si3—C10—C12	-44.9 (2)	C10—Si3—C13—C15	74.7 (2)
C16—Si3—C10—C12	78.8 (2)	C16—Si3—C13—C15	-47.8 (2)
Si1—Si3—C10—C12	-165.25 (18)	Si1—Si3—C13—C15	-166.17 (17)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C5—H5B \cdots Br1	0.98	2.80	3.711 (3)	155
C16—H16 \cdots Br1	1.00	2.84	3.584 (3)	131

C23—H23A···Br1	0.98	2.87	3.685 (3)	142
C24—H24C···Br1	0.98	2.93	3.726 (3)	139
