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Tetrakis(2,3,5,5-tetramethylhexen-2-yl)-silane

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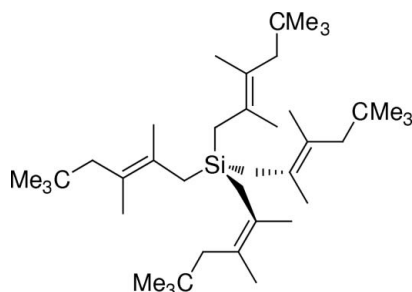
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.051; wR factor = 0.136; data-to-parameter ratio = 18.3.

In the title compound, $\text{C}_{40}\text{H}_{76}\text{Si}$, the Si atom is located on a special position of site symmetry $\bar{4}$. Thus, there is just a quarter of a molecule in the asymmetric unit. The $\text{C}=\text{C}$ double bonds exhibit a *trans* configuration. The Si atom and the *tert*-butyl group are located on the same side of the plane formed by the $\text{C}=\text{C}$ double bond and its four substituents. The crystal packing shows no short contacts between the molecules and despite the low crystal density (0.980 Mg m^{-3}), there are no significant voids in the structure.

Related literature

For information on the chemical background, see: Meyer-Wegner *et al.* (2011, 2014).



Experimental

Crystal data

$\text{C}_{40}\text{H}_{76}\text{Si}$
 $M_r = 585.10$
 Tetragonal, $I4_1/a$
 $a = 12.5780$ (11) Å
 $c = 25.053$ (3) Å
 $V = 3963.5$ (7) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.08 \text{ mm}^{-1}$
 $T = 173$ K
 $0.32 \times 0.28 \times 0.16 \text{ mm}$

Data collection

Stoe IPDS-II two-circle diffractometer
 Absorption correction: multi-scan (MULABS; Spek, 2009; Blessing, 1995)
 $T_{\min} = 0.963$, $T_{\max} = 0.988$

5117 measured reflections
 1742 independent reflections
 1301 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.045$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.136$
 $S = 1.08$
 1742 reflections

95 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$

Data collection: *X-Area* (Stoe & Cie, 2001); cell refinement: *X-RED32* (Stoe & Cie, 2001); data reduction: *X-Area*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97* and *pubCIF* (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: ZS2287).

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supporting information

Acta Cryst. (2014). E70, o376 [doi:10.1107/S1600536814004322]

Tetrakis(2,3,5,5-tetramethylhexen-2-yl)silane**Frank Meyer-Wegner, Michael Bolte and Hans-Wolfram Lerner****S1. Comment**

Very recently, we have investigated the reaction of Si_2Cl_6 with NR_3 ($R = \text{Me}, \text{Et}$) and we have been able to show that amine-complexed dichlorosilylenes represent the key intermediates in this disproportionation reaction (Meyer-Wegner *et al.*, 2011). In the course of these studies we isolated along with SiCl_4 the [4 + 1] cycloadduct of dichlorosilylene SiCl_2 with 2,3-dimethyl-1,3-butadiene (DMB) in good yield upon treatment of Si_2Cl_6 with catalytic amounts of NMe_3 or NMe_2Et in neat DMB (Meyer-Wegner *et al.*, 2014). In this study we report on the reaction of Si_2Cl_6 with catalytic amounts of NMe_2Et in the presence of DMB. Subsequent treatment of this reaction mixture with *t*BuLi yielded the title compound (I) which could be isolated (Fig. 1).

In the title compound (Fig. 2), the Si centre is located on a special position of site symmetry $\bar{4}$. Thus, there is just a quarter of a molecule in the asymmetric unit. The C=C double bonds show a *trans* configuration. It is interesting to note, that the Si centre and the *t*-butyl group are located on the same side of the plane formed by the C=C double bond and its four substituents. The crystal packing (Fig. 3) shows no short contacts between the molecules and despite the fact that the crystal density is rather low [0.980 g/cm³], there are no significant voids in the structure.

S2. Experimental

To Si_2Cl_6 (0.3 ml, 0.47 g, 1.74 mmol) in DMB (1.2 ml, 0.86 g, 10 mmol) a catalytic amount of NMe_2Et (0.012 g, 0.2 mmol) was added. Subsequent treatment of this mixture with a solution of *t*BuLi (16 mmol) in 10 ml of pentane yielded a colorless precipitate. After filtration all volatiles were removed *in vacuo*. The obtained residue was dissolved in benzene. This solution was stored at ambient temperature for a period of two days yielding single crystals of the title compound.

S3. Refinement

H atoms were geometrically positioned and refined using a riding model with fixed individual displacement parameters [$U(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ or $U(\text{H}) = 1.5 U_{\text{eq}}(\text{C}_{\text{methyl}})$] and with $\text{C}_{\text{methyl}}\text{—H} = 0.98 \text{ \AA}$ and $\text{C}_{\text{methylene}}\text{—H} = 0.99 \text{ \AA}$. The methyl groups attached to the double bond were allowed to rotate but not to tip.

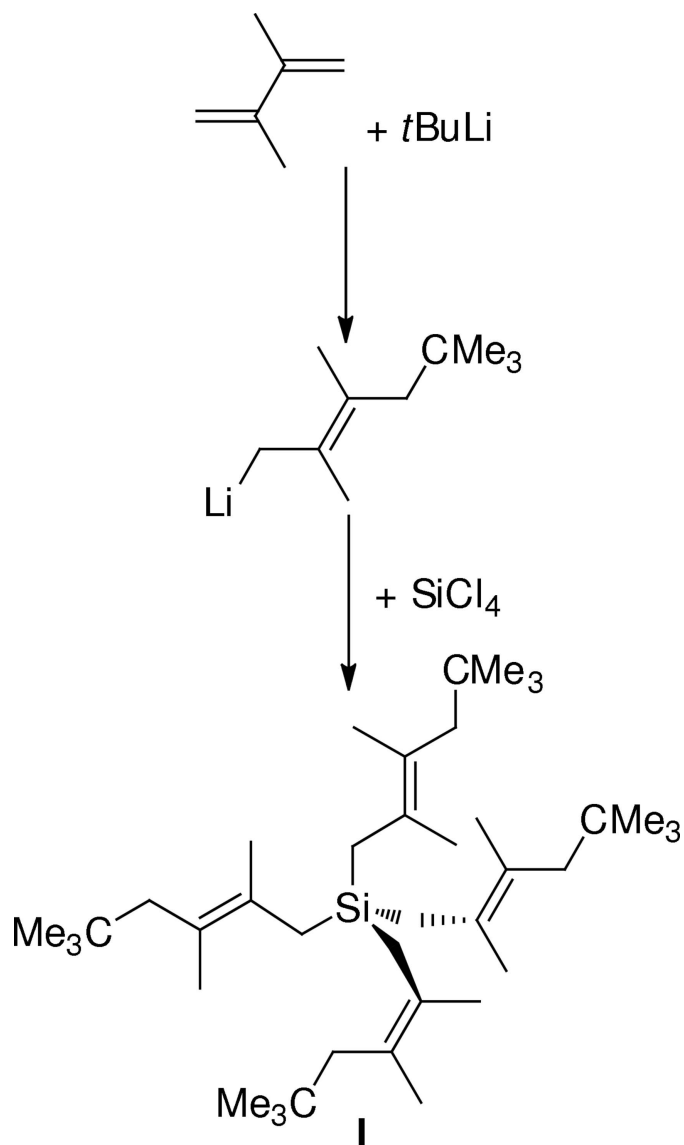
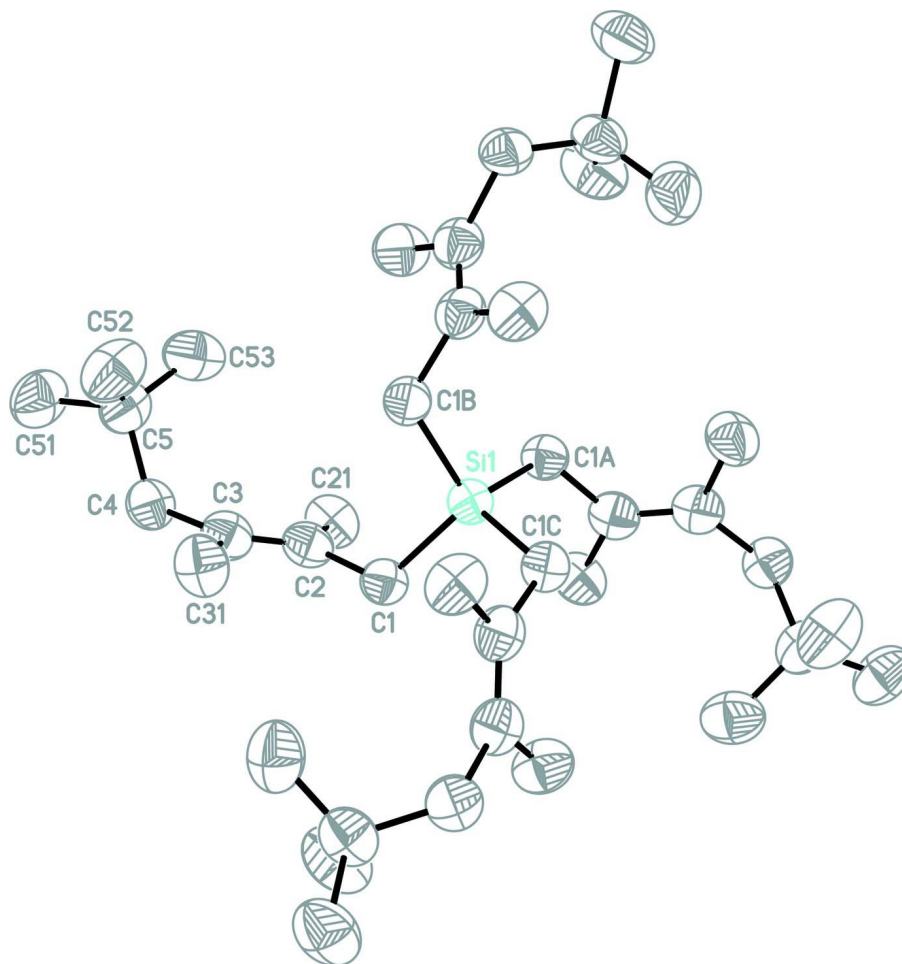


Figure 1

Reaction of SiCl_4 with $t\text{BuLi}$ in the presence of 2,3-dimethyl-1,3-butadiene.

**Figure 2**

Perspective view of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are omitted for clarity. Symmetry codes: (A) $-x + 1, -y + 3/2, z$; (B) $y - 1/4, -x + 5/4, -z + 1/4$; (C) $-y + 5/4, x + 1/4, -z + 1/4$.

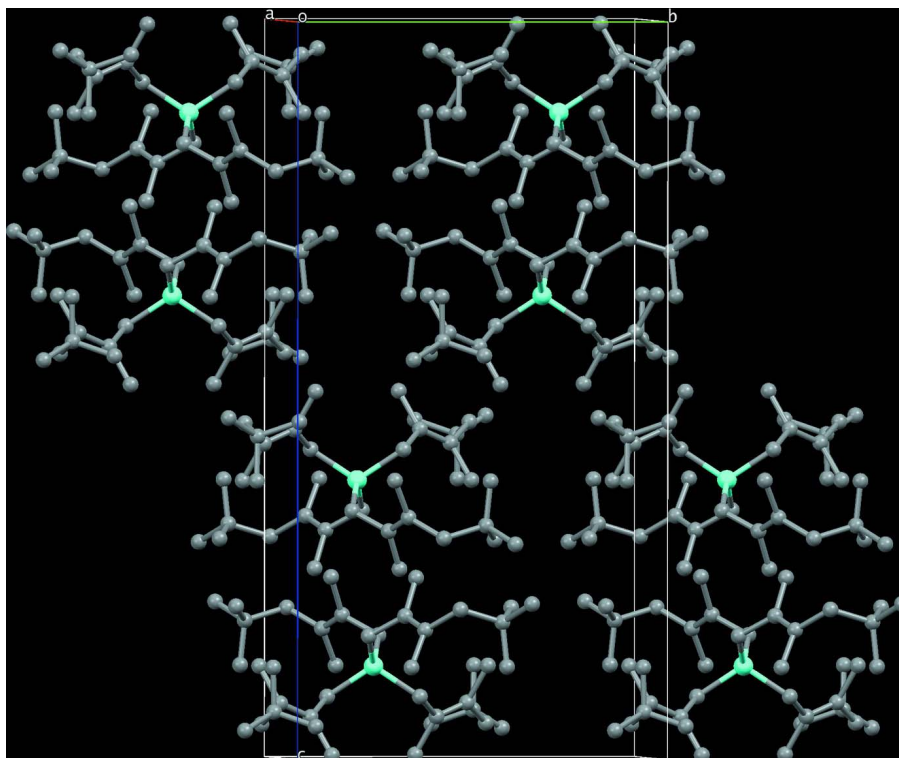


Figure 3

Packing diagram of the title compound; hydrogen atoms are omitted for clarity. The view direction is approximately down the *a* axis.

Tetrakis(2,3,5,5-tetramethylhexen-2-yl)silane

Crystal data

$C_{40}H_{76}Si$

$M_r = 585.10$

Tetragonal, $I4_1/a$

Hall symbol: $-I\ 4ad$

$a = 12.5780$ (11) Å

$c = 25.053$ (3) Å

$V = 3963.5$ (7) Å³

$Z = 4$

$F(000) = 1320$

$D_x = 0.980$ Mg m⁻³

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

Cell parameters from 4399 reflections

$\theta = 3.7$ – 25.9°

$\mu = 0.08$ mm⁻¹

$T = 173$ K

Block, colourless

$0.32 \times 0.28 \times 0.16$ mm

Data collection

Stoe IPDS-II two-circle
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan

(*MULABS*; Spek, 2009; Blessing, 1995)

$T_{\min} = 0.963$, $T_{\max} = 0.988$

5117 measured reflections

1742 independent reflections

1301 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 3.6^\circ$

$h = -8 \rightarrow 14$

$k = -14 \rightarrow 14$

$l = -25 \rightarrow 29$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.051$

$wR(F^2) = 0.136$

$S = 1.08$

1742 reflections

95 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

$w = 1/[\sigma^2(F_o^2) + (0.0654P)^2 + 0.753P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$

Special details

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Si1	0.5000	0.7500	0.1250	0.0457 (3)
C1	0.52419 (16)	0.62670 (16)	0.08358 (7)	0.0522 (5)
H1A	0.5766	0.6452	0.0556	0.063*
H1B	0.5581	0.5731	0.1070	0.063*
C2	0.43057 (17)	0.57385 (17)	0.05637 (8)	0.0564 (5)
C3	0.38412 (17)	0.48729 (17)	0.07519 (8)	0.0595 (6)
C4	0.29391 (17)	0.42817 (18)	0.04703 (8)	0.0588 (5)
H4A	0.3062	0.3510	0.0518	0.071*
H4B	0.2996	0.4432	0.0084	0.071*
C5	0.17836 (18)	0.45239 (19)	0.06442 (8)	0.0628 (6)
C21	0.3961 (2)	0.62822 (18)	0.00476 (8)	0.0652 (6)
H21A	0.4590	0.6459	-0.0165	0.098*
H21B	0.3570	0.6935	0.0133	0.098*
H21C	0.3500	0.5802	-0.0156	0.098*
C31	0.4258 (2)	0.43428 (18)	0.12553 (9)	0.0695 (6)
H31A	0.3791	0.3750	0.1353	0.104*
H31B	0.4274	0.4862	0.1547	0.104*
H31C	0.4979	0.4075	0.1191	0.104*
C51	0.1061 (2)	0.3674 (2)	0.03927 (11)	0.0833 (8)
H51A	0.1139	0.3691	0.0004	0.125*
H51B	0.0320	0.3818	0.0488	0.125*
H51C	0.1266	0.2970	0.0526	0.125*
C52	0.1662 (2)	0.4487 (3)	0.12469 (10)	0.0881 (8)
H52A	0.0924	0.4648	0.1343	0.132*
H52B	0.2136	0.5013	0.1410	0.132*

H52C	0.1848	0.3775	0.1376	0.132*
C53	0.1450 (2)	0.5620 (2)	0.04480 (11)	0.0825 (7)
H53A	0.1528	0.5654	0.0059	0.124*
H53B	0.1902	0.6162	0.0614	0.124*
H53C	0.0707	0.5749	0.0545	0.124*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0476 (4)	0.0476 (4)	0.0418 (5)	0.000	0.000	0.000
C1	0.0517 (11)	0.0526 (11)	0.0524 (10)	0.0025 (9)	-0.0010 (8)	-0.0013 (8)
C2	0.0612 (12)	0.0571 (12)	0.0508 (11)	0.0073 (10)	0.0031 (9)	-0.0075 (9)
C3	0.0636 (13)	0.0606 (13)	0.0543 (11)	0.0055 (10)	-0.0024 (9)	-0.0053 (9)
C4	0.0644 (13)	0.0599 (12)	0.0520 (10)	-0.0025 (10)	-0.0035 (9)	-0.0116 (9)
C5	0.0613 (13)	0.0744 (15)	0.0528 (11)	-0.0074 (11)	-0.0035 (10)	-0.0120 (10)
C21	0.0841 (16)	0.0659 (14)	0.0457 (10)	0.0062 (12)	-0.0018 (10)	0.0016 (9)
C31	0.0832 (15)	0.0641 (13)	0.0613 (12)	-0.0020 (12)	-0.0165 (11)	0.0081 (11)
C51	0.0674 (15)	0.0991 (19)	0.0833 (16)	-0.0160 (14)	-0.0095 (12)	-0.0227 (14)
C52	0.0807 (17)	0.122 (2)	0.0618 (13)	-0.0206 (16)	0.0092 (12)	-0.0160 (14)
C53	0.0744 (16)	0.0910 (19)	0.0821 (16)	0.0154 (14)	-0.0009 (12)	-0.0141 (13)

Geometric parameters (Å, °)

Si1—C1 ⁱ	1.8906 (19)	C5—C51	1.538 (3)
Si1—C1 ⁱⁱ	1.8906 (19)	C21—H21A	0.9800
Si1—C1	1.8907 (19)	C21—H21B	0.9800
Si1—C1 ⁱⁱⁱ	1.8907 (19)	C21—H21C	0.9800
C1—C2	1.514 (3)	C31—H31A	0.9800
C1—H1A	0.9900	C31—H31B	0.9800
C1—H1B	0.9900	C31—H31C	0.9800
C2—C3	1.323 (3)	C51—H51A	0.9800
C2—C21	1.526 (3)	C51—H51B	0.9800
C3—C31	1.520 (3)	C51—H51C	0.9800
C3—C4	1.529 (3)	C52—H52A	0.9800
C4—C5	1.548 (3)	C52—H52B	0.9800
C4—H4A	0.9900	C52—H52C	0.9800
C4—H4B	0.9900	C53—H53A	0.9800
C5—C52	1.518 (3)	C53—H53B	0.9800
C5—C53	1.522 (4)	C53—H53C	0.9800
C1 ⁱ —Si1—C1 ⁱⁱ	107.53 (6)	C2—C21—H21A	109.5
C1 ⁱ —Si1—C1	113.43 (12)	C2—C21—H21B	109.5
C1 ⁱⁱ —Si1—C1	107.53 (6)	H21A—C21—H21B	109.5
C1 ⁱ —Si1—C1 ⁱⁱⁱ	107.53 (6)	C2—C21—H21C	109.5
C1 ⁱⁱ —Si1—C1 ⁱⁱⁱ	113.43 (12)	H21A—C21—H21C	109.5
C1—Si1—C1 ⁱⁱⁱ	107.53 (6)	H21B—C21—H21C	109.5
C2—C1—Si1	118.82 (14)	C3—C31—H31A	109.5
C2—C1—H1A	107.6	C3—C31—H31B	109.5

Si1—C1—H1A	107.6	H31A—C31—H31B	109.5
C2—C1—H1B	107.6	C3—C31—H31C	109.5
Si1—C1—H1B	107.6	H31A—C31—H31C	109.5
H1A—C1—H1B	107.0	H31B—C31—H31C	109.5
C3—C2—C1	122.99 (19)	C5—C51—H51A	109.5
C3—C2—C21	123.0 (2)	C5—C51—H51B	109.5
C1—C2—C21	113.95 (18)	H51A—C51—H51B	109.5
C2—C3—C31	120.3 (2)	C5—C51—H51C	109.5
C2—C3—C4	124.31 (19)	H51A—C51—H51C	109.5
C31—C3—C4	115.20 (19)	H51B—C51—H51C	109.5
C3—C4—C5	118.12 (16)	C5—C52—H52A	109.5
C3—C4—H4A	107.8	C5—C52—H52B	109.5
C5—C4—H4A	107.8	H52A—C52—H52B	109.5
C3—C4—H4B	107.8	C5—C52—H52C	109.5
C5—C4—H4B	107.8	H52A—C52—H52C	109.5
H4A—C4—H4B	107.1	H52B—C52—H52C	109.5
C52—C5—C53	108.7 (2)	C5—C53—H53A	109.5
C52—C5—C51	109.1 (2)	C5—C53—H53B	109.5
C53—C5—C51	109.6 (2)	H53A—C53—H53B	109.5
C52—C5—C4	111.62 (19)	C5—C53—H53C	109.5
C53—C5—C4	110.24 (19)	H53A—C53—H53C	109.5
C51—C5—C4	107.60 (18)	H53B—C53—H53C	109.5
C1 ⁱ —Si1—C1—C2	-75.32 (15)	C1—C2—C3—C4	-176.17 (18)
C1 ⁱⁱ —Si1—C1—C2	165.92 (18)	C21—C2—C3—C4	2.6 (3)
C1 ⁱⁱⁱ —Si1—C1—C2	43.44 (12)	C2—C3—C4—C5	-96.9 (3)
Si1—C1—C2—C3	-101.5 (2)	C31—C3—C4—C5	88.3 (2)
Si1—C1—C2—C21	79.6 (2)	C3—C4—C5—C52	-48.5 (3)
C1—C2—C3—C31	-1.6 (3)	C3—C4—C5—C53	72.4 (2)
C21—C2—C3—C31	177.2 (2)	C3—C4—C5—C51	-168.2 (2)

Symmetry codes: (i) $-x+1, -y+3/2, z$; (ii) $-y+5/4, x+1/4, -z+1/4$; (iii) $y-1/4, -x+5/4, -z+1/4$.