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2,3,5,6-Tetrafluoro-1,4-bis(trimethylsilyl)benzene

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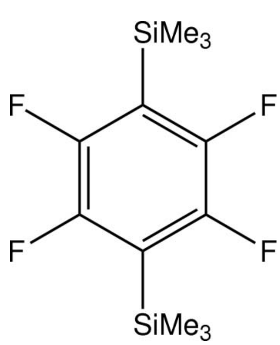
Key indicators: single-crystal X-ray study; T = 199 K; mean σ(C–C) = 0.002 Å; R factor = 0.027; wR factor = 0.068; data-to-parameter ratio = 14.0.

The asymmetric unit of the title compound, $C_{12}H_{18}F_4Si_2$, contains two independent molecules, both lying on inversion centers. The C_{arene}—Si distances are significantly longer than in the analogous non-fluorinated compound. The packing of the molecules results in a herringbone motif in the *ac* plane.

Related literature

For the synthesis and chemistry of 1,4-$(Me_3Si)_2$—C_6F_4, see: Fearon & Gilman (1967); Tamborski & Soloski (1969); Fields *et al.* (1970); Sartori & Frohn (1974); Bardin *et al.* (1991); Frohn *et al.* (1998); Kashiwabara & Tanaka (2006). For related structures see: Rehm *et al.* (1999); Sekiguchi *et al.* (2000); Haberecht *et al.* (2002, 2004); Krumm *et al.* (2005); Hanamoto *et al.* (2006).



Experimental

Crystal data

$C_{12}H_{18}F_4Si_2$
M_r = 294.44
Monoclinic, $P2_1/c$
a = 19.8389 (4) Å
b = 6.35013 (10) Å
c = 12.3827 (2) Å
β = 107.407 (2)°
V = 1488.53 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 199 K
0.25 × 0.22 × 0.20 mm

Data collection

Oxford Xcalibur Eos diffractometer
8888 measured reflections
2626 independent reflections
2496 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.068
S = 1.09
2626 reflections
187 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Si1—C2	1.9101 (15)	Si2—C8	1.9077 (15)

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2011); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: MW2055).

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2,3,5,6-Tetrafluoro-1,4-bis(trimethylsilyl)benzene

Maik Finze, Guido J. Reiss and Hermann-Josef Frohn

S1. Comment

The first synthesis of the title compound 1,4-bis(trimethylsilyl)tetrafluorobenzene, 1,4-$(Me_3Si)_2$—C_6F_4, was reported in 1967 starting from 1,2,4,5-tetrafluorobenzene, *n*-butyl lithium, and trimethylsilyl chloride (Fearon & Gilman 1967). Later, the compound was observed as a by-product in related reactions, improved methods for its selective synthesis were published and some reactions of 1,4-$(Me_3Si)_2$—C_6F_4 were described (Tamborski & Soloski, 1969; Fields *et al.* 1970; Sartori & Frohn, 1974; Bardin *et al.* 1991; Frohn *et al.* 1998; Kashiwabara & Tanaka, 2006). The 1,4-$(Me_3Si)_2$—C_6F_4 employed in the present study was prepared by a different route using poly(cadmium-2,3,5,6-tetrafluorobenzene), [1,4-Cd—$C_6F_4]_n$, and trimethylsilyl chloride as starting materials.

The title compound 1,4-bis(trimethylsilyl)tetrafluorobenzene (Figure 1) crystallizes in the monoclinic space group $P2_1/c$ with two independent molecules each of which is located on a center of symmetry. Both crystallographically independent molecules display very similar geometric parameters. The C–C, C_{methyl}–Si and C–F bond lengths are in the expected range. Both C_{arene}–Si distances are slightly longer than those found for the non-fluorinated analogue 1,4-$(Me_3Si)_2$—C_6H_4 [$d(C_{arene}$–Si) = 1.8817 (12) Å] (Haberecht *et al.* 2004) and other non-fluorinated Me_3Si–C_{arene} compounds (Haberecht *et al.* 2004; Rehm *et al.* 1999). In contrast, for related 1-trimethylsilyl-2,3,5,6-tetrafluoro benzene fragments similar values were reported (Sekiguchi *et al.* 2000, Krumm *et al.* 2005). In the closely related compound 1,2,4-$(i\mathrm{Pr}_3\mathrm{Si})_3$—$C_6F_3$ (Hanamoto *et al.* 2006) the $d(C_{arene}$–Si) of the $i\mathrm{Pr}_3\mathrm{Si}$ groups in *ortho* positions [d(C–Si) = 1.937 (2), 1.934 (2)] are significantly longer than those in the title compound whereas the third C_{arene}–Si distance [d(C–Si) = 1.914 (2) Å], which corresponds to the $i\mathrm{Pr}_3\mathrm{Si}$ group that has two F atoms in the *ortho* positions, is close to the values determined for 1,4-$(Me_3Si)_2$—C_6F_4.

The 1,4-bis(trimethylsilyl)tetrafluorobenzene molecules are arranged in the *ac* plane to form a herringbone structure (Figure 2). The trimethylsilyl groups are interlinked by van der Waals interactions with methyl groups of neighboring molecules.

S2. Experimental

The starting material poly(cadmium-2,3,5,6-tetrafluorobenzene), [1,4-Cd—$C_6F_4]_n$, was synthesized by thermolysis of Cd(1,4-O_2C—C_6F_4) at 270 °C under vacuum according to a literature procedure (Sartori & Frohn, 1974).

7.1 g (27.3 mmol) poly(cadmium-2,3,5,6-tetrafluorobenzene), [1,4-Cd—$C_6F_4]_n$, was charged into a Duran-glass Carius tube inside a glove box. 6.55 g (60.3 mmol) freshly distilled $(CH_3)_3SiCl$ was added under protection of dry nitrogen. The tube was sealed and shaken and heated inside an oven. The temperature was increased stepwise over 30 h to 222°C without visual change of the reaction components. Further heating from 230 to 250°C over 26 h was accompanied by a reduction of the liquid phase and a change of the color to light grey. The Carius tube was cooled stepwise to -78°C before opening under nitrogen protection. CAUTION: Handling of the sealed Carius tube should proceed behind a large protection screen with long-sleeve leather gloves. At 0°C 3.4 g (31.3 mmol) of $(CH_3)_3SiCl$ were recovered by condensation under high-vacuum. The dark grey solid residue, which contained the co-product $CdCl_2$, was extracted with boiling petrol ether (60–70°C fraction). After removing the solvent from the extract a slightly brownish oil remained which was sublimed under high vacuum. The colorless crystals were collected on a water cooled sublimation finger. Yield *ca* 80%; mp 46°C [31–32°C isomeric mixture (Fields *et al.*, 1970)]; NMR (20% CCl_4 solution): ^{1}H -0.39 p.p.m., ^{19}F -124.2 p.p.m.; MS (EI, 73 eV) $M^{+\cdot}$ 294, the fragment ions 81 (CH_3SiF_2), 77 ($(CH_3)_2SiF$), and 73 ($(CH_3)_3Si$) possessed higher intensities than the parent ion; IR (neat): 2946 (*m*), 2889 (*m*), 1575 (w), 1487 (w), 1400 (st, b), 1343 (w), 1334 (w), 1286 (w), 1240 (st), 1214 (st), 1183 (*m*), 1035 (w), 921 (st), 830 (st, b), 748 (st), 684 (*m*), 613 (*m*), 566 (w), 432 (w).

S3. Refinement

Methyl H atoms were identified in a difference map, idealized and refined using rigid groups allowed to rotate about the Si—C bond (AFIX 137 option of the *SHELXL97* program). All U_{iso}(H) values were refined unrestrictedly.

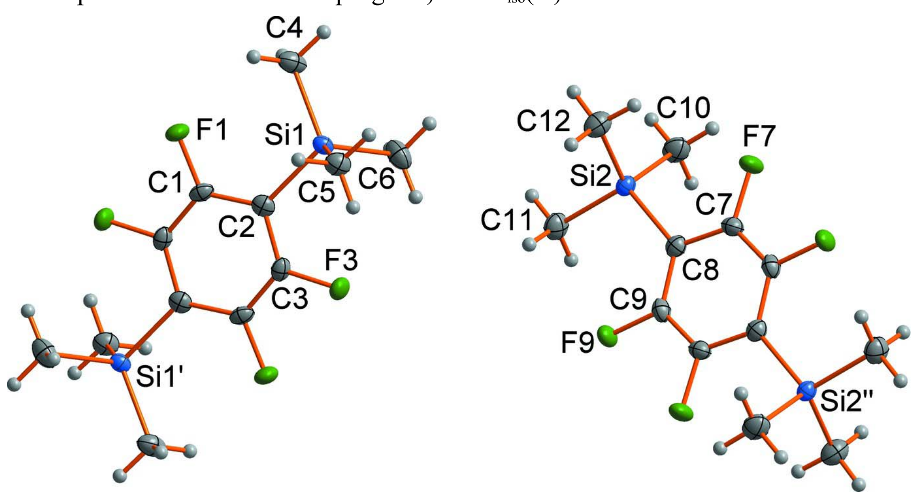

Figure 1

The two crystallographically independent molecules of the title compound (H-atoms are drawn with arbitrary radii; ′ = -*x*, 1 - *y*, -*z*; ″ = 1 - *x*, 2 - *y*, 1 - *z*).

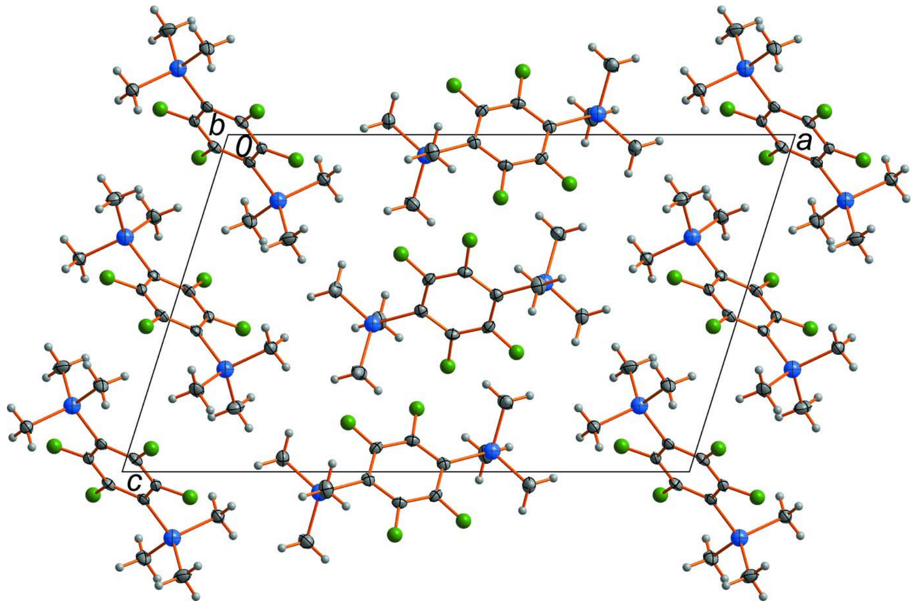

Figure 2

Molecular packing of the title compound viewed parallel to the *b* axis, showing the herringbone type motif (ball and stick type model with arbitrary atom radii).

2,3,5,6-Tetrafluoro-1,4-bis(trimethylsilyl)benzene

Crystal data

$C_{12}H_{18}F_4Si_2$
$M_r = 294.44$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 19.8389$ (4) Å
$b = 6.35013$ (10) Å
$c = 12.3827$ (2) Å
$\beta = 107.407$ (2)°
$V = 1488.53$ (5) Å^3
$Z = 4$

$F(000) = 616$
$D_x = 1.314$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8162 reflections
$\theta = 3.1$–28.3°
$\mu = 0.26$ mm^{-1}
$T = 199$ K
Block, colourless
0.25 × 0.22 × 0.20 mm

Data collection

Oxford Xcalibur Eos diffractometer
Radiation source: fine-focus sealed tube
Equatorial mounted graphite monochromator
Detector resolution: 16.2711 pixels mm^{-1}
ω–scan
8888 measured reflections

2626 independent reflections
2496 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.2°$
$h = -22 \rightarrow 23$
$k = -7 \rightarrow 7$
$l = -14 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.068$
$S = 1.09$
2626 reflections
187 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
$w = 1/[\sigma^2(F_o^2) + (0.0284P)^2 + 1.0425P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-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.

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Si1	0.12486 (2)	0.23395 (7)	0.19607 (3)	0.01427 (11)
C1	−0.01679 (8)	0.3116 (2)	0.03692 (12)	0.0145 (3)
F1	−0.03838 (4)	0.12566 (13)	0.06873 (7)	0.0203 (2)
C2	0.05291 (8)	0.3765 (2)	0.08187 (12)	0.0139 (3)
C3	0.06680 (7)	0.5691 (2)	0.04012 (12)	0.0141 (3)
F3	0.13382 (4)	0.64624 (14)	0.07568 (7)	0.0199 (2)
C4	0.08544 (8)	0.0143 (3)	0.25580 (13)	0.0224 (3)
H4A	0.0484	0.0679	0.2834	0.034 (5)*
H4B	0.1212	−0.0499	0.3170	0.037 (5)*
H4C	0.0663	−0.0886	0.1979	0.037 (5)*
C5	0.19335 (8)	0.1412 (3)	0.13233 (13)	0.0190 (3)
H5A	0.2119	0.2592	0.1019	0.033 (5)*
H5B	0.1727	0.0427	0.0728	0.030 (5)*
H5C	0.2309	0.0738	0.1894	0.037 (5)*
C6	0.16406 (9)	0.4291 (3)	0.30949 (13)	0.0241 (4)
H6A	0.1858	0.5406	0.2794	0.044 (6)*
H6B	0.1990	0.3611	0.3703	0.045 (6)*
H6C	0.1276	0.4864	0.3373	0.042 (6)*
Si2	0.35988 (2)	0.79571 (7)	0.56124 (3)	0.01519 (11)
C7	0.50606 (8)	0.8812 (2)	0.59251 (12)	0.0156 (3)
F7	0.51552 (5)	0.76178 (15)	0.68697 (7)	0.0227 (2)
C8	0.43772 (8)	0.9180 (2)	0.52347 (12)	0.0147 (3)
C9	0.43451 (7)	1.0409 (2)	0.42942 (12)	0.0152 (3)
F9	0.37103 (4)	1.08933 (14)	0.35422 (7)	0.0203 (2)
C10	0.36292 (9)	0.8818 (3)	0.70638 (13)	0.0235 (4)
H10A	0.3604	1.0326	0.7086	0.038 (6)*
H10B	0.4063	0.8348	0.7594	0.032 (5)*
H10C	0.3237	0.8222	0.7259	0.039 (6)*
C11	0.27524 (8)	0.8855 (3)	0.46024 (13)	0.0214 (3)
H11A	0.2727	0.8394	0.3853	0.031 (5)*
H11B	0.2729	1.0364	0.4617	0.030 (5)*
H11C	0.2365	0.8269	0.4816	0.034 (5)*

C12	0.36891 (9)	0.5045 (3)	0.55206 (14)	0.0235 (4)
H12A	0.3661	0.4662	0.4758	0.042 (6)*
H12B	0.3316	0.4365	0.5734	0.049 (6)*
H12C	0.4137	0.4610	0.6022	0.036 (5)*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0148 (2)	0.0168 (2)	0.0121 (2)	0.00203 (16)	0.00533 (16)	0.00124 (16)
C1	0.0186 (7)	0.0128 (7)	0.0152 (7)	−0.0018 (6)	0.0099 (6)	0.0001 (6)
F1	0.0192 (5)	0.0163 (5)	0.0258 (5)	−0.0034 (4)	0.0073 (4)	0.0062 (4)
C2	0.0158 (7)	0.0160 (7)	0.0121 (7)	0.0017 (6)	0.0074 (6)	−0.0015 (6)
C3	0.0114 (7)	0.0177 (7)	0.0144 (7)	−0.0023 (6)	0.0057 (6)	−0.0030 (6)
F3	0.0124 (4)	0.0215 (5)	0.0247 (5)	−0.0043 (4)	0.0041 (4)	0.0018 (4)
C4	0.0210 (8)	0.0264 (9)	0.0213 (8)	0.0043 (7)	0.0089 (6)	0.0093 (7)
C5	0.0189 (8)	0.0216 (8)	0.0186 (7)	0.0019 (6)	0.0085 (6)	0.0008 (6)
C6	0.0262 (9)	0.0271 (9)	0.0162 (8)	0.0050 (7)	0.0019 (7)	−0.0033 (7)
Si2	0.0138 (2)	0.0159 (2)	0.0169 (2)	−0.00165 (16)	0.00616 (16)	−0.00042 (16)
C7	0.0187 (8)	0.0144 (7)	0.0135 (7)	0.0006 (6)	0.0045 (6)	0.0025 (6)
F7	0.0190 (5)	0.0288 (5)	0.0191 (5)	−0.0001 (4)	0.0039 (4)	0.0117 (4)
C8	0.0156 (7)	0.0134 (7)	0.0156 (7)	−0.0005 (6)	0.0055 (6)	−0.0020 (6)
C9	0.0122 (7)	0.0159 (7)	0.0153 (7)	0.0020 (6)	0.0006 (6)	−0.0009 (6)
F9	0.0124 (4)	0.0260 (5)	0.0192 (4)	0.0008 (4)	0.0000 (3)	0.0064 (4)
C10	0.0260 (9)	0.0264 (9)	0.0209 (8)	−0.0044 (7)	0.0116 (7)	−0.0012 (7)
C11	0.0157 (8)	0.0254 (9)	0.0240 (8)	−0.0011 (6)	0.0075 (6)	−0.0006 (7)
C12	0.0237 (8)	0.0185 (8)	0.0299 (9)	−0.0019 (7)	0.0105 (7)	0.0003 (7)

Geometric parameters (Å, º)

Si1—C4	1.8574 (16)	Si2—C11	1.8579 (16)
Si1—C5	1.8595 (15)	Si2—C10	1.8620 (16)
Si1—C6	1.8602 (16)	Si2—C12	1.8644 (17)
Si1—C2	1.9101 (15)	Si2—C8	1.9077 (15)
C1—F1	1.3539 (17)	C7—F7	1.3591 (17)
C1—C3^{i}	1.379 (2)	C7—C9^{ii}	1.378 (2)
C1—C2	1.390 (2)	C7—C8	1.389 (2)
C2—C3	1.387 (2)	C8—C9	1.387 (2)
C3—F3	1.3605 (16)	C9—F9	1.3587 (16)
C3—C1^{i}	1.379 (2)	C9—C7^{ii}	1.378 (2)
C4—H4A	0.9600	C10—H10A	0.9600
C4—H4B	0.9600	C10—H10B	0.9600
C4—H4C	0.9600	C10—H10C	0.9600
C5—H5A	0.9600	C11—H11A	0.9600
C5—H5B	0.9600	C11—H11B	0.9600
C5—H5C	0.9600	C11—H11C	0.9600
C6—H6A	0.9600	C12—H12A	0.9600
C6—H6B	0.9600	C12—H12B	0.9600
C6—H6C	0.9600	C12—H12C	0.9600

C4—Si1—C5	112.29 (7)	C11—Si2—C10	108.76 (7)
C4—Si1—C6	109.29 (8)	C11—Si2—C12	110.31 (8)
C5—Si1—C6	109.76 (7)	C10—Si2—C12	111.96 (8)
C4—Si1—C2	109.89 (7)	C11—Si2—C8	110.18 (7)
C5—Si1—C2	108.36 (7)	C10—Si2—C8	108.80 (7)
C6—Si1—C2	107.11 (7)	C12—Si2—C8	106.81 (7)
F1—C1—C3^{i}	117.10 (13)	F7—C7—C9^{ii}	117.62 (13)
F1—C1—C2	120.37 (13)	F7—C7—C8	118.79 (13)
C3^{i}—C1—C2	122.53 (14)	C9^{ii}—C7—C8	123.58 (14)
C3—C2—C1	113.39 (13)	C9—C8—C7	113.74 (13)
C3—C2—Si1	120.39 (11)	C9—C8—Si2	126.70 (11)
C1—C2—Si1	126.13 (11)	C7—C8—Si2	119.55 (11)
F3—C3—C1^{i}	117.22 (13)	F9—C9—C7^{ii}	117.13 (13)
F3—C3—C2	118.72 (13)	F9—C9—C8	120.19 (13)
C1^{i}—C3—C2	124.06 (13)	C7^{ii}—C9—C8	122.68 (13)
Si1—C4—H4A	109.5	Si2—C10—H10A	109.5
Si1—C4—H4B	109.5	Si2—C10—H10B	109.5
H4A—C4—H4B	109.5	H10A—C10—H10B	109.5
Si1—C4—H4C	109.5	Si2—C10—H10C	109.5
H4A—C4—H4C	109.5	H10A—C10—H10C	109.5
H4B—C4—H4C	109.5	H10B—C10—H10C	109.5
Si1—C5—H5A	109.5	Si2—C11—H11A	109.5
Si1—C5—H5B	109.5	Si2—C11—H11B	109.5
H5A—C5—H5B	109.5	H11A—C11—H11B	109.5
Si1—C5—H5C	109.5	Si2—C11—H11C	109.5
H5A—C5—H5C	109.5	H11A—C11—H11C	109.5
H5B—C5—H5C	109.5	H11B—C11—H11C	109.5
Si1—C6—H6A	109.5	Si2—C12—H12A	109.5
Si1—C6—H6B	109.5	Si2—C12—H12B	109.5
H6A—C6—H6B	109.5	H12A—C12—H12B	109.5
Si1—C6—H6C	109.5	Si2—C12—H12C	109.5
H6A—C6—H6C	109.5	H12A—C12—H12C	109.5
H6B—C6—H6C	109.5	H12B—C12—H12C	109.5

F1—C1—C2—C3	179.45 (12)	F7—C7—C8—C9	−179.74 (13)
C3^{i}—C1—C2—C3	−1.0 (2)	C9^{ii}—C7—C8—C9	0.1 (2)
F1—C1—C2—Si1	−3.9 (2)	F7—C7—C8—Si2	−1.36 (19)
C3^{i}—C1—C2—Si1	175.56 (11)	C9^{ii}—C7—C8—Si2	178.52 (12)
C4—Si1—C2—C3	167.71 (11)	C11—Si2—C8—C9	−5.61 (16)
C5—Si1—C2—C3	−69.26 (13)	C10—Si2—C8—C9	−124.75 (14)
C6—Si1—C2—C3	49.09 (13)	C12—Si2—C8—C9	114.22 (14)
C4—Si1—C2—C1	−8.68 (15)	C11—Si2—C8—C7	176.25 (12)
C5—Si1—C2—C1	114.36 (13)	C10—Si2—C8—C7	57.10 (14)
C6—Si1—C2—C1	−127.29 (13)	C12—Si2—C8—C7	−63.93 (13)
C1—C2—C3—F3	−178.22 (12)	C7—C8—C9—F9	179.85 (13)
Si1—C2—C3—F3	4.96 (18)	Si2—C8—C9—F9	1.6 (2)

C1—C2—C3—C1^{i}	1.1 (2)	C7—C8—C9—C7^{ii}	−0.1 (2)
Si1—C2—C3—C1^{i}	−175.76 (11)	Si2—C8—C9—C7^{ii}	−178.38 (11)

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