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Tetrakis[3,5-bis(trifluoromethyl)phenyl]silane

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.051
wR factor = 0.158
Data-to-parameter ratio = 14.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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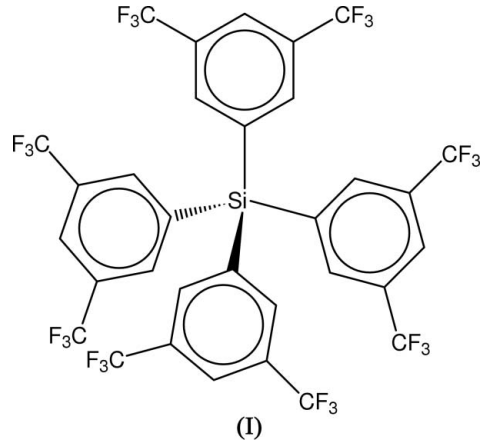
The title compound, tetrakis[3,5-bis(trifluoromethyl)phenyl]-silane ($SiAr'''_4$, $C_{32}H_{12}F_{24}Si$), is a minor product from the reaction of silicon(IV) bromide with lithiated 1,3-bis(trifluoromethyl)benzene (Ar′H). The structure crystallizes with two half-molecules in the asymmetric unit, with each central Si atom positioned on a twofold axis in a pseudo-tetrahedral environment, with Si—C bond lengths in the range 1.873 (3)–1.879 (3) Å.

Comment

The reactions of silicon(IV) chloride in a 1:2 ratio with lithiated trifluoromethyl-substituted aromatic hydrocarbons are complex and interesting (Buijink *et al.*, 1993; Braddock-Wilking *et al.*, 1995; Batsanov *et al.*, 2003). With lithiated 1,3,5-tris(trifluoromethyl)benzene (ArH), the only product identified was Ar_2SiF_2, which was fully characterized crystallographically (Buijink *et al.*, 1993). This result was subsequently confirmed by Batsanov *et al.* (2003). Similarly, $HSiCl_3$ reacts with ArLi to form Ar_2SiHF, again involving chlorine–fluorine exchange; this has also been characterized by single-crystal X-ray diffraction (Braddock-Wilking *et al.*, 1995). With 1,3-bis(trifluoromethyl)benzene, Ar′H, the system is more complicated because there are various lithiation positions (Bartle *et al.*, 1973; Batsanov *et al.*, 2002, 2003; Cornet *et al.*, 2003). These are *ortho* to both CF_3 groups, giving 2,6-bis(trifluoromethyl)phenyl (Ar′) derivatives, *ortho* to one CF_3 group and *para* to the other, yielding 2,4-bis(trifluoromethyl)-phenyl (Ar″) species, and, much less likely, *meta* to both CF_3 groups, giving 3,5-bis(trifluoromethyl)phenyl (Ar‴) derivatives. An analytical gas–liquid chromatography study, following carboxylation of the organolithium compounds and subsequent esterification with diazomethane, showed *ca* 60% of the 2,4-isomer, 40% of the 2,6-isomer and less than 1% of a third component, presumed to be the 3,5-isomer (Bartle *et al.*, 1973). With $SiCl_4$, four of the possible disubstituted products, once F/Cl exchange is taken into account, have been observed spectroscopically, *viz.* Ar'_2SiCl_2, Ar'_2SiF_2, Ar''_2SiCl_2 and Ar''_2SiF_2; two of these, Ar'_2SiF_2 and Ar''_2SiCl_2, have been characterized by single-crystal X-ray diffraction at 120 K (Batsanov *et al.*, 2003). The results suggested that the F/Cl exchange rate decreased in the order Ar > Ar′ > Ar″ (Batsanov *et al.*, 2003). It was therefore of considerable interest to extend this work to reactions of silicon(IV) bromide in a 1:2 molar ratio with the lithium derivatives of ArH and Ar′H.

Not surprisingly, the only product observed from $SiBr_4$ and ArLi was Ar_2SiF_2; Si—Br bonds are weaker than Si—Cl bonds, so facile exchange could reasonably be expected. The ^{19}F NMR data are given in Table 2, with literature data for

comparison (Batsanov *et al.*, 2003). With lithiated Ar′H and $SiBr_4$, the products Ar'_2SiF_2 (Batsanov *et al.*, 2003), Ar''_2SiBr_2 and Ar''_2SiBrF were identified by ^{19}F NMR solution state spectroscopy (Table 2). The results thus lend support to the idea that halogen exchange is slowest in the Ar″ species. After the mixture had been left to stand for some time, a few crystals were isolated, and proved to be of the fully substituted silane with no *ortho* CF_3 groups, *i.e.* tetrakis[3,5-bis(trifluoromethyl)-phenyl]silane. Since there is little or no steric hindrance around silicon, further substitution beyond the disubstituted product is clearly more favourable for 3,5-derivatives than for 2,4- or 2,6-compounds. Nevertheless this product is a surprising one, in view of the work of Bartle *et al.* (1973).



(I)

The compound tetrakis[3,5-bis(trifluoromethyl)phenyl]-silane, (I), crystallizes in the monoclinic space group *P*2/*c*, with two half molecules in the asymmetric unit and the molecular structure is shown in Fig. 1. Selected bond distances and angles are listed in Table 1. The central Si atom is in a tetrahedral environment, with bond angles around Si between 106.27 (16) and 110.97 (11)°. The Si—C bond lengths are all very similar, between 1.873 (3) and 1.877 (3) Å. The CF_3 groups are all ordered, presumably due to the weak F···F interactions (Fig. 2 and Table 1). While this species has not been reported previously, the structurally similar tetrakis[3,5-bis(trifluoro-methyl)phenyl]borate ion has been widely used in recent years, since the first report by Nishida *et al.* (1984), as a large lipophilic stable counter-ion for a variety of cationic complexes; it features in 291 structures in the Cambridge Structural Database (November 2004 edition; Allen, 2002). The structures of neutral tris[3,5-bis(trifluoromethyl)phenyl]-phosphane (Jessop *et al.*, 2002) and -arsane (Dietzel & Jansen, 2004) have also been described. However, these species were all made directly from 3,5-bis(trifluoromethyl)phenyl-substituted aromatic starting materials.

Experimental

All manipulations of air- and/or moisture-sensitive compounds were performed either under an inert atmosphere of dry nitrogen or *in vacuo*, using standard Schlenk and cannula techniques, or in a nitrogen-filled glovebox. ^{19}F NMR spectra were recorded on a Varian Unity 300 Fourier transform spectrometer at 282.2 MHz; chemical shifts were measured relative to external $CFCl_3$. A solution of $SiBr_4$ (1.7 ml, 13.5 mmol) in diethyl ether was added dropwise, *via* a cannula, to a solution of ArLi (8 ml, 27 mmol) in diethyl ether at 195 K [the lithiated solutions of both ArH and Ar'H were prepared as described previously by Batsanov *et al.* (2002)]. White fumes were evolved. The mixture was allowed to warm to room temperature and stirred for 5 h, giving a pale-yellow oil. The presence of Ar_2SiF_2 as the only major silicon-containing component was confirmed by ^{19}F NMR solution state spectroscopy (Table 2). Similarly, a solution of $SiBr_4$ (0.7 ml, 5.6 mmol) in diethyl ether was added slowly, *via* a cannula, to

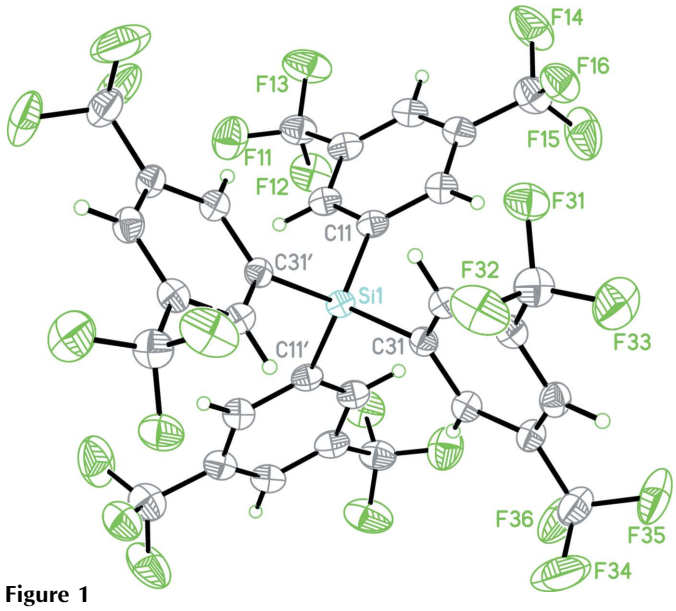


Figure 1
View of one of the independent molecules of (I) with selected atoms labelled. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code for primed and unlabelled atoms: $1-x, y, \frac{1}{2}-z$.] The other molecule is very similar.

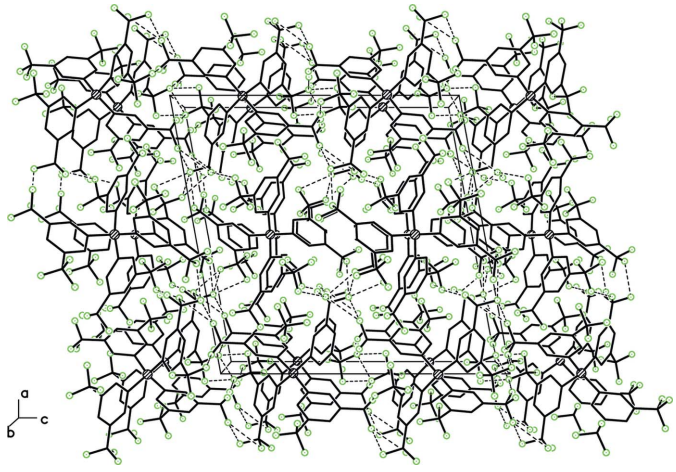


Figure 2
Crystal packing in the title compound, viewed down the *b* axis, showing short intermolecular F···F contacts as dashed lines. H atoms have been omitted for clarity.

a solution of lithiated Ar′H (4 ml, 11.3 mmol) in diethyl ether at 195 K. The mixture was allowed to reach room temperature and stirred overnight, giving a pale-brown solution. Analysis by ^{19}F NMR spectroscopy indicated three main components (Table 2). When the mixture was allowed to stand for some weeks, a few crystals appeared; these were isolated, and analysed by single-crystal X-ray diffraction. As indicated above, they proved to be of tetrakis[3,5-bis(trifluoromethyl)phenyl]silane.

Crystal data

$C_{32}H_{12}F_{24}Si$
M_r = 880.51
Monoclinic, $P2/c$
a = 18.3760 (4) Å
b = 9.5325 (2) Å
c = 18.7776 (4) Å
β = 100.388 (1)°
V = 3235.34 (12) Å^3
Z = 4
D_x = 1.808 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 120 (2) K
Block, colourless
0.20 × 0.12 × 0.10 mm

Data collection

Bruker SMART-6000 CCD diffractometer
ω scans
Absorption correction: integration (*XPREP* in *SHELXTL*; Sheldrick, 1997*b*)
T_{min} = 0.957, T_{max} = 0.977
28959 measured reflections
7404 independent reflections
5078 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.158
S = 1.02
7404 reflections
515 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0779P)^2 + 2.8011P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Si1—C11	1.873 (3)	Si2—C41	1.876 (3)
Si1—C31	1.877 (3)	Si2—C21	1.879 (3)
F12···F14^{i}	2.847 (3)	F21···F44^{iii}	2.861 (3)
F13···F31^{ii}	2.919 (3)	F31···F41	2.901 (3)
F16···F42	2.997 (3)	F33···F26	2.969 (3)
F16···F43	2.848 (3)	F33···F41	2.852 (3)
C11^{iv}—Si1—C11	106.27 (16)	C41^{v}—Si2—C41	105.22 (16)
C11^{iv}—Si1—C31^{iv}	110.63 (11)	C41^{v}—Si2—C21	111.80 (11)
C11—Si1—C31^{iv}	110.97 (11)	C41—Si2—C21	110.87 (11)
C11^{iv}—Si1—C31	110.97 (11)	C41^{v}—Si2—C21^{v}	110.87 (11)
C11—Si1—C31	110.63 (11)	C41—Si2—C21^{v}	111.80 (11)
C31^{iv}—Si1—C31	107.40 (16)	C21—Si2—C21^{v}	106.39 (16)

Symmetry codes: (i) $-x+1, y, -z+\frac{1}{2}$; (ii) $-x, y, -z+\frac{1}{2}$; (iii) $-x+1, -y, -z+1$; (iv) $-x+1, -y+1, -z+1$; (v) $x, -y+1, z-\frac{1}{2}$.

Table 2
^{19}F NMR (p.p.m., Hz) data for reaction products.

Group	No. of Fs	δ ^{19}F	$^5J_{FF}$	δ ^{19}F^a	$^5J_{FF}$^{a}
Ar_2SiF_2					
o-CF_3	12	−57.7	*t*, 12.4	−57.3	*t*, 12.8
p-CF_3	6	−63.8	*s*	−64.2	*s*
Si—F	2	−125.7	*m*, NR^b	−124.5	*m*, 12.8
Ar'_2SiF_2					
o-CF_3	12	−57.5	*t*, 12.8	−57.5	*t*, 12.3
Si—F	2	−125.4	*m*, 12.8	−125.5	*m*, 12.5
Ar''_2SiBr_2					
o-CF_3	6	−57.6	*s*		
p-CF_3	6	−64.5	*s*		
Ar''_2SiBrF					
o-CF_3	6	−59.6	*d*, 12.8		
p-CF_3	6	−64.6	*s*		
Si—F	1	−158.4	*m*, 12.8		

Notes: (*a*) literature data from Batsanov *et al.* (2003); (*b*) not resolved.

All H atoms were positioned geometrically (C—H = 0.95 Å) and refined using a riding model with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART-NT* (Bruker, 2000); cell refinement: *SMART-NT*; data reduction: *SAINT-NT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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supporting information

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Crystal data

$C_{32}H_{12}F_{24}Si$
$M_r = 880.51$
Monoclinic, $P2/c$
Hall symbol: -P 2yc
$a = 18.3760$ (4) Å
$b = 9.5325$ (2) Å
$c = 18.7776$ (4) Å
$\beta = 100.388$ (1)°
$V = 3235.34$ (12) Å^3
$Z = 4$
$F(000) = 1736$
$D_x = 1.808$ Mg m^{-3}
Melting point: not measured K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5856 reflections
$\theta = 2.4$–$26.8°$
$\mu = 0.24$ mm^{-1}
$T = 120$ K
Block, colourless
0.20 × 0.12 × 0.10 mm

Data collection

Bruker SMART-6000 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8 pixels mm^{-1}
ω scans
Absorption correction: integration (XPREP in SHELXTL; Sheldrick, 1997b)
$T_{min} = 0.957$, $T_{max} = 0.977$
28959 measured reflections
7404 independent reflections
5078 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.1°$
$h = -23 \rightarrow 23$
$k = -12 \rightarrow 12$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.158$
$S = 1.02$
7404 reflections
515 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
$w = 1/[\sigma^2(F_o^2) + (0.0779P)^2 + 2.8011P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.62$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}

Special details

Experimental. The data collection nominally covered full sphere of reciprocal Space, by a combination of 5 sets of ω scans each set at different φ and/or 2θ angles and each scan (15 s exposure) covering 0.3° in ω. Crystal to detector distance 5.81 cm.

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Si1	0.5000	0.51518 (10)	0.2500	0.0226 (2)
C11	0.50382 (15)	0.3973 (3)	0.33037 (14)	0.0265 (5)
C12	0.55556 (16)	0.2873 (3)	0.33934 (15)	0.0306 (6)
H12	0.5873	0.2748	0.3051	0.037*
C13	0.56073 (16)	0.1963 (3)	0.39802 (15)	0.0324 (6)
C14	0.51587 (17)	0.2136 (3)	0.44805 (15)	0.0328 (6)
H14	0.5202	0.1519	0.4883	0.039*
C15	0.46426 (17)	0.3205 (3)	0.44026 (15)	0.0332 (6)
C16	0.45809 (16)	0.4127 (3)	0.38142 (15)	0.0307 (6)
H16	0.4225	0.4860	0.3763	0.037*
C17	0.61350 (19)	0.0747 (3)	0.40342 (16)	0.0399 (7)
F11	0.67404 (11)	0.1038 (2)	0.37646 (12)	0.0562 (5)
F12	0.58198 (12)	−0.03549 (18)	0.36466 (11)	0.0524 (5)
F13	0.63480 (14)	0.0299 (2)	0.47074 (11)	0.0641 (6)
C18	0.4136 (2)	0.3379 (3)	0.49352 (18)	0.0446 (7)
F14	0.44268 (14)	0.2839 (2)	0.55899 (11)	0.0691 (7)
F15	0.34864 (13)	0.2778 (3)	0.47187 (14)	0.0729 (7)
F16	0.40028 (11)	0.4729 (2)	0.50652 (10)	0.0485 (5)
C31	0.41640 (14)	0.6318 (3)	0.23931 (13)	0.0242 (5)
C32	0.41887 (14)	0.7540 (3)	0.28073 (14)	0.0269 (5)
H32	0.4625	0.7763	0.3143	0.032*
C33	0.35807 (15)	0.8435 (3)	0.27335 (14)	0.0281 (5)
C34	0.29410 (15)	0.8129 (3)	0.22503 (15)	0.0295 (6)
H34	0.2528	0.8743	0.2200	0.035*
C35	0.29090 (14)	0.6912 (3)	0.18393 (14)	0.0289 (6)
C36	0.35160 (14)	0.6021 (3)	0.19073 (14)	0.0267 (5)
H36	0.3488	0.5198	0.1618	0.032*
C37	0.36451 (17)	0.9769 (3)	0.31688 (16)	0.0369 (6)
F31	0.40129 (10)	0.95860 (18)	0.38421 (9)	0.0418 (4)
F32	0.40239 (13)	1.07538 (18)	0.28782 (11)	0.0561 (6)
F33	0.29914 (11)	1.0315 (2)	0.32191 (12)	0.0571 (5)
C38	0.22219 (17)	0.6590 (3)	0.13071 (18)	0.0421 (7)
F34	0.21457 (13)	0.7411 (3)	0.07311 (13)	0.0871 (9)
F35	0.16130 (10)	0.6791 (2)	0.15935 (13)	0.0622 (6)
F36	0.21858 (11)	0.5268 (2)	0.10818 (13)	0.0665 (7)
Si2	0.0000	0.85198 (10)	0.2500	0.0226 (2)

C21	0.06268 (14)	0.9701 (3)	0.20832 (14)	0.0263 (5)
C22	0.06995 (15)	0.9610 (3)	0.13596 (14)	0.0274 (5)
H22	0.0441	0.8899	0.1062	0.033*
C23	0.11462 (15)	1.0547 (3)	0.10653 (14)	0.0292 (6)
C24	0.15327 (15)	1.1580 (3)	0.14924 (15)	0.0314 (6)
H24	0.1842	1.2209	0.1291	0.038*
C25	0.14687 (15)	1.1696 (3)	0.22082 (15)	0.0306 (6)
C26	0.10142 (15)	1.0782 (3)	0.25045 (15)	0.0294 (6)
H26	0.0964	1.0887	0.2996	0.035*
C27	0.11787 (18)	1.0511 (3)	0.02752 (16)	0.0382 (7)
F21	0.08780 (13)	0.9363 (2)	−0.00568 (10)	0.0557 (5)
F22	0.07939 (13)	1.1592 (2)	−0.00753 (10)	0.0579 (5)
F23	0.18555 (11)	1.0634 (3)	0.01409 (11)	0.0633 (6)
C28	0.18495 (18)	1.2886 (3)	0.26534 (17)	0.0392 (7)
F24	0.14555 (13)	1.40746 (18)	0.25369 (11)	0.0576 (6)
F25	0.25153 (11)	1.3164 (2)	0.24927 (12)	0.0593 (6)
F26	0.19520 (11)	1.26399 (19)	0.33628 (9)	0.0465 (4)
C41	0.05500 (15)	0.7325 (3)	0.31883 (13)	0.0257 (5)
C42	0.13058 (14)	0.7488 (3)	0.34453 (13)	0.0281 (5)
H42	0.1561	0.8258	0.3284	0.034*
C43	0.16907 (15)	0.6534 (3)	0.39354 (14)	0.0305 (6)
C44	0.13299 (16)	0.5395 (3)	0.41750 (14)	0.0312 (6)
H44	0.1594	0.4748	0.4511	0.037*
C45	0.05818 (15)	0.5215 (3)	0.39184 (14)	0.0284 (5)
C46	0.01943 (15)	0.6164 (3)	0.34351 (14)	0.0273 (5)
H46	−0.0320	0.6029	0.3268	0.033*
C47	0.25038 (17)	0.6700 (4)	0.41920 (16)	0.0394 (7)
F41	0.27414 (10)	0.7993 (2)	0.41015 (11)	0.0542 (5)
F42	0.28932 (11)	0.5855 (2)	0.38304 (12)	0.0609 (6)
F43	0.27010 (10)	0.6407 (2)	0.49010 (10)	0.0556 (5)
C48	0.01947 (17)	0.3948 (3)	0.41478 (16)	0.0373 (7)
F44	0.04750 (12)	0.3538 (2)	0.48211 (11)	0.0567 (6)
F45	−0.05241 (11)	0.41571 (19)	0.41221 (11)	0.0502 (5)
F46	0.02479 (12)	0.28522 (19)	0.37153 (12)	0.0571 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0238 (5)	0.0191 (4)	0.0240 (5)	0.000	0.0023 (4)	0.000
C11	0.0300 (13)	0.0216 (12)	0.0261 (12)	−0.0028 (10)	0.0002 (10)	−0.0013 (10)
C12	0.0342 (14)	0.0259 (13)	0.0300 (13)	−0.0013 (11)	0.0012 (11)	−0.0003 (10)
C13	0.0395 (16)	0.0231 (12)	0.0316 (14)	−0.0008 (11)	−0.0016 (12)	−0.0017 (10)
C14	0.0438 (16)	0.0243 (13)	0.0288 (13)	−0.0022 (12)	0.0025 (12)	0.0009 (10)
C15	0.0393 (16)	0.0307 (14)	0.0294 (13)	−0.0026 (12)	0.0055 (12)	−0.0002 (11)
C16	0.0343 (15)	0.0251 (13)	0.0324 (14)	−0.0010 (11)	0.0054 (12)	0.0007 (11)
C17	0.0497 (19)	0.0342 (15)	0.0338 (15)	0.0078 (13)	0.0023 (13)	0.0045 (12)
F11	0.0446 (11)	0.0508 (12)	0.0728 (14)	0.0135 (9)	0.0097 (10)	0.0108 (10)
F12	0.0702 (14)	0.0297 (9)	0.0570 (12)	0.0055 (9)	0.0110 (10)	−0.0044 (8)

F13	0.0912 (17)	0.0581 (13)	0.0391 (10)	0.0369 (12)	0.0015 (10)	0.0116 (9)
C18	0.054 (2)	0.0382 (16)	0.0433 (17)	0.0010 (15)	0.0127 (15)	0.0105 (14)
F14	0.0957 (18)	0.0727 (15)	0.0466 (11)	0.0325 (13)	0.0336 (12)	0.0274 (11)
F15	0.0619 (14)	0.0767 (16)	0.0898 (17)	−0.0275 (12)	0.0391 (13)	−0.0178 (13)
F16	0.0595 (12)	0.0453 (10)	0.0436 (10)	0.0083 (9)	0.0168 (9)	0.0003 (8)
C31	0.0252 (13)	0.0226 (12)	0.0253 (12)	−0.0013 (10)	0.0061 (10)	0.0036 (9)
C32	0.0273 (13)	0.0240 (12)	0.0286 (12)	−0.0010 (10)	0.0029 (10)	0.0010 (10)
C33	0.0325 (14)	0.0245 (12)	0.0286 (13)	0.0011 (11)	0.0088 (11)	0.0015 (10)
C34	0.0284 (13)	0.0261 (12)	0.0347 (14)	0.0045 (11)	0.0077 (11)	0.0031 (11)
C35	0.0239 (13)	0.0306 (13)	0.0318 (13)	0.0025 (10)	0.0043 (11)	0.0002 (11)
C36	0.0270 (13)	0.0238 (12)	0.0291 (13)	−0.0005 (10)	0.0046 (10)	−0.0012 (10)
C37	0.0422 (17)	0.0294 (14)	0.0390 (16)	0.0031 (12)	0.0065 (13)	−0.0023 (12)
F31	0.0523 (11)	0.0376 (9)	0.0345 (9)	0.0000 (8)	0.0052 (8)	−0.0085 (7)
F32	0.0899 (16)	0.0301 (9)	0.0505 (11)	−0.0173 (10)	0.0182 (11)	−0.0029 (8)
F33	0.0523 (12)	0.0458 (11)	0.0707 (14)	0.0176 (9)	0.0041 (10)	−0.0218 (10)
C38	0.0303 (15)	0.0437 (17)	0.0491 (18)	0.0064 (13)	−0.0014 (13)	−0.0071 (14)
F34	0.0638 (15)	0.124 (2)	0.0598 (14)	−0.0178 (15)	−0.0259 (12)	0.0330 (15)
F35	0.0242 (9)	0.0650 (13)	0.0958 (17)	−0.0018 (9)	0.0061 (10)	−0.0259 (12)
F36	0.0408 (11)	0.0643 (13)	0.0837 (15)	0.0096 (10)	−0.0173 (10)	−0.0399 (12)
Si2	0.0253 (5)	0.0212 (4)	0.0210 (4)	0.000	0.0033 (4)	0.000
C21	0.0250 (13)	0.0247 (12)	0.0290 (13)	0.0038 (10)	0.0046 (10)	0.0030 (10)
C22	0.0278 (13)	0.0264 (12)	0.0274 (12)	0.0003 (10)	0.0034 (10)	0.0001 (10)
C23	0.0297 (14)	0.0271 (13)	0.0303 (13)	0.0012 (11)	0.0044 (11)	0.0009 (10)
C24	0.0285 (14)	0.0273 (13)	0.0388 (15)	0.0006 (11)	0.0070 (12)	0.0033 (11)
C25	0.0314 (14)	0.0243 (13)	0.0348 (14)	0.0017 (11)	0.0023 (11)	−0.0014 (11)
C26	0.0309 (14)	0.0259 (13)	0.0317 (14)	0.0009 (11)	0.0059 (11)	0.0003 (10)
C27	0.0433 (17)	0.0391 (16)	0.0336 (15)	−0.0081 (13)	0.0103 (13)	−0.0016 (12)
F21	0.0835 (15)	0.0508 (11)	0.0351 (10)	−0.0203 (11)	0.0170 (10)	−0.0103 (8)
F22	0.0821 (15)	0.0550 (12)	0.0359 (10)	0.0079 (11)	0.0083 (10)	0.0104 (9)
F23	0.0449 (12)	0.1063 (18)	0.0417 (11)	−0.0164 (12)	0.0161 (9)	−0.0057 (11)
C28	0.0448 (17)	0.0297 (14)	0.0427 (17)	−0.0048 (13)	0.0066 (14)	−0.0036 (12)
F24	0.0805 (15)	0.0269 (9)	0.0592 (12)	0.0046 (9)	−0.0041 (11)	−0.0078 (8)
F25	0.0559 (13)	0.0569 (12)	0.0673 (13)	−0.0292 (10)	0.0169 (10)	−0.0174 (10)
F26	0.0570 (11)	0.0397 (10)	0.0393 (10)	−0.0081 (9)	−0.0009 (8)	−0.0093 (8)
C41	0.0301 (13)	0.0250 (12)	0.0221 (11)	0.0009 (10)	0.0048 (10)	−0.0009 (9)
C42	0.0279 (13)	0.0311 (13)	0.0247 (12)	−0.0008 (11)	0.0035 (10)	−0.0012 (10)
C43	0.0291 (14)	0.0348 (14)	0.0268 (13)	0.0007 (11)	0.0028 (11)	−0.0015 (11)
C44	0.0348 (15)	0.0293 (13)	0.0282 (13)	0.0053 (11)	0.0022 (11)	0.0026 (11)
C45	0.0321 (14)	0.0250 (12)	0.0272 (13)	−0.0004 (11)	0.0031 (11)	−0.0005 (10)
C46	0.0265 (13)	0.0266 (12)	0.0277 (12)	0.0003 (10)	0.0022 (10)	0.0001 (10)
C47	0.0313 (15)	0.0528 (18)	0.0327 (15)	0.0047 (14)	0.0024 (12)	0.0040 (13)
F41	0.0339 (10)	0.0646 (13)	0.0612 (12)	−0.0148 (9)	0.0006 (9)	0.0076 (10)
F42	0.0369 (11)	0.0805 (15)	0.0651 (13)	0.0157 (10)	0.0088 (10)	−0.0065 (11)
F43	0.0354 (10)	0.0860 (15)	0.0400 (10)	−0.0051 (10)	−0.0078 (8)	0.0137 (10)
C48	0.0412 (17)	0.0293 (14)	0.0391 (16)	−0.0026 (12)	0.0009 (13)	0.0060 (12)
F44	0.0675 (13)	0.0451 (11)	0.0503 (11)	−0.0140 (10)	−0.0087 (10)	0.0236 (9)
F45	0.0430 (11)	0.0400 (10)	0.0689 (13)	−0.0049 (8)	0.0138 (9)	0.0151 (9)
F46	0.0692 (14)	0.0311 (9)	0.0734 (14)	−0.0123 (9)	0.0190 (11)	−0.0125 (9)

Geometric parameters (Å, º)

Si1—C11^{i}	1.873 (3)	Si2—C41^{ii}	1.876 (3)
Si1—C11	1.873 (3)	Si2—C41	1.876 (3)
Si1—C31^{i}	1.877 (3)	Si2—C21	1.879 (3)
Si1—C31	1.877 (3)	Si2—C21^{ii}	1.879 (3)
C11—C16	1.392 (4)	C21—C22	1.392 (4)
C11—C12	1.406 (4)	C21—C26	1.411 (4)
C12—C13	1.392 (4)	C22—C23	1.393 (4)
C12—H12	0.9500	C22—H22	0.9500
C13—C14	1.367 (4)	C23—C24	1.383 (4)
C13—C17	1.503 (4)	C23—C27	1.496 (4)
C14—C15	1.382 (4)	C24—C25	1.375 (4)
C14—H14	0.9500	C24—H24	0.9500
C15—C16	1.400 (4)	C25—C26	1.391 (4)
C15—C18	1.493 (4)	C25—C28	1.505 (4)
C16—H16	0.9500	C26—H26	0.9500
C17—F13	1.325 (3)	C27—F23	1.318 (4)
C17—F11	1.332 (4)	C27—F21	1.330 (3)
C17—F12	1.348 (4)	C27—F22	1.351 (4)
C18—F15	1.321 (4)	C28—F26	1.332 (4)
C18—F16	1.340 (4)	C28—F25	1.338 (4)
C18—F14	1.351 (4)	C28—F24	1.341 (4)
C31—C36	1.392 (4)	C41—C42	1.394 (4)
C31—C32	1.397 (4)	C41—C46	1.405 (4)
C32—C33	1.393 (4)	C42—C43	1.393 (4)
C32—H32	0.9500	C42—H42	0.9500
C33—C34	1.381 (4)	C43—C44	1.388 (4)
C33—C37	1.505 (4)	C43—C47	1.493 (4)
C34—C35	1.388 (4)	C44—C45	1.383 (4)
C34—H34	0.9500	C44—H44	0.9500
C35—C36	1.390 (4)	C45—C46	1.385 (4)
C35—C38	1.494 (4)	C45—C48	1.503 (4)
C36—H36	0.9500	C46—H46	0.9500
C37—F33	1.328 (4)	C47—F41	1.329 (4)
C37—F31	1.333 (3)	C47—F42	1.340 (4)
C37—F32	1.342 (4)	C47—F43	1.344 (3)
C38—F34	1.322 (4)	C48—F45	1.328 (4)
C38—F36	1.327 (4)	C48—F44	1.335 (3)
C38—F35	1.340 (4)	C48—F46	1.338 (4)
F12···F14^{iii}	2.847 (3)	F21···F44^{v}	2.861 (3)
F13···F31^{iv}	2.919 (3)	F31···F41	2.901 (3)
F16···F42	2.997 (3)	F33···F26	2.969 (3)
F16···F43	2.848 (3)	F33···F41	2.852 (3)
C11^{i}—Si1—C11	106.27 (16)	C41^{ii}—Si2—C41	105.22 (16)
C11^{i}—Si1—C31^{i}	110.63 (11)	C41^{ii}—Si2—C21	111.80 (11)

C11—Si1—C31^{i}	110.97 (11)	C41—Si2—C21	110.87 (11)
C11^{i}—Si1—C31	110.97 (11)	C41^{ii}—Si2—C21^{ii}	110.87 (11)
C11—Si1—C31	110.63 (11)	C41—Si2—C21^{ii}	111.80 (11)
C31^{i}—Si1—C31	107.40 (16)	C21—Si2—C21^{ii}	106.39 (16)
C16—C11—C12	118.4 (2)	C22—C21—C26	117.7 (2)
C16—C11—Si1	123.5 (2)	C22—C21—Si2	122.9 (2)
C12—C11—Si1	118.1 (2)	C26—C21—Si2	119.28 (19)
C13—C12—C11	120.5 (3)	C21—C22—C23	121.0 (2)
C13—C12—H12	119.7	C21—C22—H22	119.5
C11—C12—H12	119.7	C23—C22—H22	119.5
C14—C13—C12	120.4 (3)	C24—C23—C22	120.3 (2)
C14—C13—C17	120.6 (3)	C24—C23—C27	118.9 (2)
C12—C13—C17	119.0 (3)	C22—C23—C27	120.7 (2)
C13—C14—C15	120.2 (3)	C25—C24—C23	119.9 (3)
C13—C14—H14	119.9	C25—C24—H24	120.0
C15—C14—H14	119.9	C23—C24—H24	120.0
C14—C15—C16	120.2 (3)	C24—C25—C26	120.3 (3)
C14—C15—C18	120.6 (3)	C24—C25—C28	119.4 (3)
C16—C15—C18	119.2 (3)	C26—C25—C28	120.2 (3)
C11—C16—C15	120.3 (3)	C25—C26—C21	120.8 (2)
C11—C16—H16	119.8	C25—C26—H26	119.6
C15—C16—H16	119.8	C21—C26—H26	119.6
F13—C17—F11	107.8 (3)	F23—C27—F21	108.1 (3)
F13—C17—F12	106.7 (3)	F23—C27—F22	105.5 (2)
F11—C17—F12	105.3 (3)	F21—C27—F22	105.2 (3)
F13—C17—C13	112.9 (3)	F23—C27—C23	113.3 (3)
F11—C17—C13	112.8 (2)	F21—C27—C23	113.3 (2)
F12—C17—C13	110.8 (3)	F22—C27—C23	110.8 (2)
F15—C18—F16	106.7 (3)	F26—C28—F25	106.9 (3)
F15—C18—F14	107.6 (3)	F26—C28—F24	106.7 (2)
F16—C18—F14	104.8 (3)	F25—C28—F24	106.5 (3)
F15—C18—C15	112.6 (3)	F26—C28—C25	113.0 (2)
F16—C18—C15	112.7 (3)	F25—C28—C25	112.1 (2)
F14—C18—C15	112.1 (3)	F24—C28—C25	111.3 (3)
C36—C31—C32	118.2 (2)	C42—C41—C46	117.8 (2)
C36—C31—Si1	122.45 (19)	C42—C41—Si2	123.4 (2)
C32—C31—Si1	119.36 (19)	C46—C41—Si2	118.7 (2)
C33—C32—C31	120.7 (2)	C43—C42—C41	120.8 (2)
C33—C32—H32	119.6	C43—C42—H42	119.6
C31—C32—H32	119.6	C41—C42—H42	119.6
C34—C33—C32	120.6 (2)	C44—C43—C42	120.6 (3)
C34—C33—C37	120.7 (2)	C44—C43—C47	119.1 (3)
C32—C33—C37	118.7 (2)	C42—C43—C47	120.2 (3)
C33—C34—C35	119.2 (2)	C45—C44—C43	119.2 (2)
C33—C34—H34	120.4	C45—C44—H44	120.4
C35—C34—H34	120.4	C43—C44—H44	120.4
C34—C35—C36	120.5 (2)	C44—C45—C46	120.6 (2)
C34—C35—C38	119.3 (2)	C44—C45—C48	119.5 (2)

C36—C35—C38	120.2 (2)	C46—C45—C48	119.9 (2)
C35—C36—C31	120.9 (2)	C45—C46—C41	121.1 (2)
C35—C36—H36	119.6	C45—C46—H46	119.5
C31—C36—H36	119.6	C41—C46—H46	119.5
F33—C37—F31	107.1 (2)	F41—C47—F42	106.0 (3)
F33—C37—F32	107.3 (2)	F41—C47—F43	106.4 (3)
F31—C37—F32	105.4 (2)	F42—C47—F43	107.7 (2)
F33—C37—C33	112.7 (3)	F41—C47—C43	112.9 (3)
F31—C37—C33	112.4 (2)	F42—C47—C43	111.6 (3)
F32—C37—C33	111.5 (2)	F43—C47—C43	111.9 (2)
F34—C38—F36	108.0 (3)	F45—C48—F44	106.8 (3)
F34—C38—F35	105.7 (3)	F45—C48—F46	106.0 (2)
F36—C38—F35	105.4 (3)	F44—C48—F46	106.8 (2)
F34—C38—C35	112.2 (3)	F45—C48—C45	112.8 (2)
F36—C38—C35	113.3 (2)	F44—C48—C45	112.6 (2)
F35—C38—C35	111.6 (3)	F46—C48—C45	111.3 (2)

C11^{i}—Si1—C11—C16	−130.9 (3)	C41^{ii}—Si2—C21—C22	−4.0 (3)
C31^{i}—Si1—C11—C16	108.8 (2)	C41—Si2—C21—C22	113.0 (2)
C31—Si1—C11—C16	−10.4 (3)	C21^{ii}—Si2—C21—C22	−125.2 (2)
C11^{i}—Si1—C11—C12	48.89 (18)	C41^{ii}—Si2—C21—C26	172.4 (2)
C31^{i}—Si1—C11—C12	−71.4 (2)	C41—Si2—C21—C26	−70.5 (2)
C31—Si1—C11—C12	169.4 (2)	C21^{ii}—Si2—C21—C26	51.23 (18)
C16—C11—C12—C13	−0.1 (4)	C26—C21—C22—C23	0.7 (4)
Si1—C11—C12—C13	−179.9 (2)	Si2—C21—C22—C23	177.2 (2)
C11—C12—C13—C14	−0.5 (4)	C21—C22—C23—C24	0.6 (4)
C11—C12—C13—C17	176.4 (3)	C21—C22—C23—C27	−175.5 (3)
C12—C13—C14—C15	0.9 (4)	C22—C23—C24—C25	−0.8 (4)
C17—C13—C14—C15	−176.0 (3)	C27—C23—C24—C25	175.3 (3)
C13—C14—C15—C16	−0.6 (4)	C23—C24—C25—C26	−0.3 (4)
C13—C14—C15—C18	178.3 (3)	C23—C24—C25—C28	−175.8 (3)
C12—C11—C16—C15	0.3 (4)	C24—C25—C26—C21	1.6 (4)
Si1—C11—C16—C15	−179.9 (2)	C28—C25—C26—C21	177.1 (3)
C14—C15—C16—C11	0.0 (4)	C22—C21—C26—C25	−1.8 (4)
C18—C15—C16—C11	−179.0 (3)	Si2—C21—C26—C25	−178.4 (2)
C14—C13—C17—F13	−27.5 (4)	C24—C23—C27—F23	47.1 (4)
C12—C13—C17—F13	155.6 (3)	C22—C23—C27—F23	−136.7 (3)
C14—C13—C17—F11	−150.0 (3)	C24—C23—C27—F21	170.7 (3)
C12—C13—C17—F11	33.1 (4)	C22—C23—C27—F21	−13.1 (4)
C14—C13—C17—F12	92.2 (3)	C24—C23—C27—F22	−71.3 (3)
C12—C13—C17—F12	−84.7 (3)	C22—C23—C27—F22	104.9 (3)
C14—C15—C18—F15	−95.9 (4)	C24—C25—C28—F26	−159.3 (3)
C16—C15—C18—F15	83.0 (3)	C26—C25—C28—F26	25.2 (4)
C14—C15—C18—F16	143.4 (3)	C24—C25—C28—F25	−38.4 (4)
C16—C15—C18—F16	−37.7 (4)	C26—C25—C28—F25	146.1 (3)
C14—C15—C18—F14	25.5 (4)	C24—C25—C28—F24	80.7 (3)
C16—C15—C18—F14	−155.6 (3)	C26—C25—C28—F24	−94.7 (3)
C11^{i}—Si1—C31—C36	18.9 (2)	C41^{ii}—Si2—C41—C42	132.2 (2)

C11—Si1—C31—C36	−98.8 (2)	C21—Si2—C41—C42	11.1 (3)
C31^{i}—Si1—C31—C36	140.0 (2)	C21^{ii}—Si2—C41—C42	−107.4 (2)
C11^{i}—Si1—C31—C32	−160.32 (19)	C41^{ii}—Si2—C41—C46	−44.69 (17)
C11—Si1—C31—C32	82.0 (2)	C21—Si2—C41—C46	−165.72 (19)
C31^{i}—Si1—C31—C32	−39.28 (17)	C21^{ii}—Si2—C41—C46	75.7 (2)
C36—C31—C32—C33	−0.1 (4)	C46—C41—C42—C43	−0.5 (4)
Si1—C31—C32—C33	179.19 (19)	Si2—C41—C42—C43	−177.4 (2)
C31—C32—C33—C34	0.1 (4)	C41—C42—C43—C44	0.4 (4)
C31—C32—C33—C37	−177.5 (2)	C41—C42—C43—C47	178.4 (2)
C32—C33—C34—C35	0.3 (4)	C42—C43—C44—C45	0.3 (4)
C37—C33—C34—C35	177.8 (2)	C47—C43—C44—C45	−177.8 (3)
C33—C34—C35—C36	−0.7 (4)	C43—C44—C45—C46	−0.7 (4)
C33—C34—C35—C38	−179.0 (3)	C43—C44—C45—C48	177.1 (3)
C34—C35—C36—C31	0.8 (4)	C44—C45—C46—C41	0.6 (4)
C38—C35—C36—C31	179.0 (3)	C48—C45—C46—C41	−177.3 (2)
C32—C31—C36—C35	−0.3 (4)	C42—C41—C46—C45	0.1 (4)
Si1—C31—C36—C35	−179.6 (2)	Si2—C41—C46—C45	177.1 (2)
C34—C33—C37—F33	20.9 (4)	C44—C43—C47—F41	−161.8 (3)
C32—C33—C37—F33	−161.5 (2)	C42—C43—C47—F41	20.1 (4)
C34—C33—C37—F31	142.1 (3)	C44—C43—C47—F42	79.0 (3)
C32—C33—C37—F31	−40.3 (4)	C42—C43—C47—F42	−99.1 (3)
C34—C33—C37—F32	−99.8 (3)	C44—C43—C47—F43	−41.7 (4)
C32—C33—C37—F32	77.8 (3)	C42—C43—C47—F43	140.2 (3)
C34—C35—C38—F34	72.9 (4)	C44—C45—C48—F45	154.3 (3)
C36—C35—C38—F34	−105.4 (3)	C46—C45—C48—F45	−27.9 (4)
C34—C35—C38—F36	−164.4 (3)	C44—C45—C48—F44	33.2 (4)
C36—C35—C38—F36	17.3 (4)	C46—C45—C48—F44	−148.9 (3)
C34—C35—C38—F35	−45.5 (4)	C44—C45—C48—F46	−86.7 (3)
C36—C35—C38—F35	136.2 (3)	C46—C45—C48—F46	91.2 (3)

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