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9-(Chlorodimethylsilyl)fluorene

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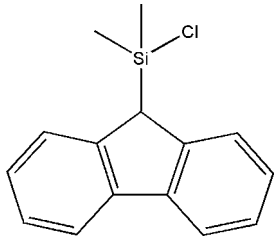
Key indicators: single-crystal X-ray study; T = 213 K; mean σ(C–C) = 0.005 Å; R factor = 0.063; wR factor = 0.152; data-to-parameter ratio = 15.3.

The title compound, $C_{15}H_{15}ClSi$, crystallizes with two molecules in the asymmetric unit. The conformations of the molecules are slightly different. The coordinaton around the Si atom is distorted tetrahedral.

Related literature

For related literature, see: Kirillov *et al.* (2003, 2006); Okuda *et al.* (1995).



Experimental

Crystal data

$C_{15}H_{15}ClSi$
M_r = 258.81
Monoclinic, $P2_1/n$
a = 17.371 (9) Å
b = 9.423 (5) Å
c = 17.511 (9) Å
β = 110.065 (7)°
V = 2692 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 213 (2) K
0.3 × 0.2 × 0.2 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.903, T_{max} = 0.934
12768 measured reflections
4753 independent reflections
4122 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.152
S = 1.14
4753 reflections
311 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2588).

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supplementary materials

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9-(Chlorodimethylsilyl)fluorene

L.-L. Guan, X. Chen, H.-B. Tong and H.-F. Li

Comment

The introduction of fluorenyl ligands in the coordination chemistry of early transition elements has opened up a rich domain of investigations (Kirillov *et al.*, 2006)·Organometallic complexes that contain linked cyclopentadienyl-amido ligands have attracted considerable attention. Some of them have been used in polymerization catalysis (Kirillov *et al.*,2003, 2006). Herein we report the crystal structure of compound (I).

The molecular structure of compound (I) shows in Fig.1,There are two independent molecules in the asymmetric unit. The conformations of the two molecules are slightly different·The coordinatons aroud Si atoms are distorted tetrahedron. The Si1,Si2 atoms are outside the planes of fluorene with distance 1.700Å and 1.693 Å, respectively·There is intermolecular interaction between C19H19 of fluorenyl ring and Cl2 with distance 2.945 Å·In the absence of classical hydrogen bonds, the crystal packing is stabilized by intermolecular interaction.

Experimental

All experiments were performed under an argon atmosphere using Schlenk apparatus. Solvents were dried and distilled from CaH_2 (methylene chloride), K—Na (hexane) and Na (Et_2O). The title complex was synthesized according to literature methods (Okuda *et al.*, 1995). To a solution of fluorene (14.99 g, 90 mmol) in diethyl ether (90 ml) was added at 273 K a solution of n-butyllithium (31.2 ml, 90 mmol, 2.88 *M* in hexane) within 1 hr. After stirring for 3 h at room temperature, the resulting orange solution was transferred to an addition funnel and added dropwise at 273 K within 1 hr to a solution of dichlorodimethylsilane (12 ml) in diethyl ether (200 ml). The resulting suspension was stirred for 30 min at room temperature, and the solvent and excess dichlorodimethylsilane was removed under vacuum. After precipitating lithium chloride with methylene chloride and filtering, followed by removal of the solvent, 18.6 g (80%) of a white solid was obtained. A solution of the white solid (0.52 g, 2.1 mmol) in hexane (10 ml) was concentrated carefully under vacuum, yielding colourless crystals of the title compound.

Refinement

All H atoms were positioned geometrically (C—H = 0.93–0.97 A°) and refined as riding, with U_{iso}(H) = 1.5Ueq(C).

Figures

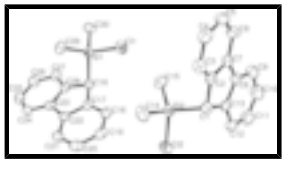


Fig. 1. The structure of (I), showing the atom-labelling scheme and displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted for clarity.

9-(Chlorodimethylsilyl)fluorene

Crystal data

$C_{15}H_{15}ClSi$	F_{000} = 1088
M_r = 258.81	D_x = 1.277 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 17.371 (9) Å	Cell parameters from 3020 reflections
b = 9.423 (5) Å	θ = 2.5–24.3º
c = 17.511 (9) Å	μ = 0.35 mm^{-1}
β = 110.065 (7)º	T = 213 (2) K
V = 2692 (3) Å^3	Block, colorless
Z = 8	0.3 × 0.2 × 0.2 mm

Data collection

Siemens SMART CCD area-detector diffractometer	4753 independent reflections
Radiation source: fine-focus sealed tube	4122 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.036
T = 213(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 1.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −20→20
T_{min} = 0.903, T_{max} = 0.934	k = −11→8
12768 measured reflections	l = −20→20

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.063	H-atom parameters constrained
$wR(F^2)$ = 0.152	$w = 1/[\sigma^2(F_o^2) + (0.0668P)^2 + 1.6658P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.14	$(\Delta/\sigma)_{max} < 0.001$
4753 reflections	$\Delta\rho_{max}$ = 0.38 e Å^{-3}
311 parameters	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Cl1	0.12013 (6)	0.47289 (10)	0.27171 (6)	0.0518 (3)
Cl2	0.26359 (6)	0.26572 (9)	0.62829 (6)	0.0474 (3)
Si1	0.09767 (5)	0.32326 (10)	0.18047 (5)	0.0320 (2)
Si2	0.17080 (5)	0.41633 (9)	0.60134 (5)	0.0318 (2)
C1	0.20671 (19)	0.5596 (3)	0.68047 (18)	0.0299 (7)
H1	0.2157	0.5230	0.7352	0.036*
C2	0.14297 (19)	0.6765 (3)	0.65773 (17)	0.0288 (7)
C3	0.0633 (2)	0.6761 (4)	0.6565 (2)	0.0366 (8)
H3	0.0420	0.5967	0.6737	0.044*
C4	0.0154 (2)	0.7956 (4)	0.6293 (2)	0.0402 (8)
H4	−0.0387	0.7956	0.6279	0.048*
C5	0.0466 (2)	0.9147 (4)	0.6044 (2)	0.0411 (8)
H5	0.0134	0.9936	0.5857	0.049*
C6	0.1270 (2)	0.9171 (4)	0.6071 (2)	0.0376 (8)
H6	0.1484	0.9977	0.5911	0.045*
C7	0.17534 (18)	0.7977 (3)	0.63405 (18)	0.0285 (7)
C8	0.26155 (19)	0.7714 (3)	0.64487 (18)	0.0298 (7)
C9	0.3199 (2)	0.8587 (4)	0.6323 (2)	0.0421 (8)
H9	0.3066	0.9501	0.6120	0.051*
C10	0.3989 (2)	0.8067 (4)	0.6506 (2)	0.0510 (10)
H10	0.4389	0.8635	0.6419	0.061*
C11	0.4188 (2)	0.6712 (4)	0.6816 (2)	0.0479 (10)
H11	0.4723	0.6387	0.6941	0.058*
C12	0.3608 (2)	0.5839 (4)	0.6942 (2)	0.0380 (8)
H12	0.3745	0.4929	0.7148	0.046*
C13	0.28135 (18)	0.6342 (3)	0.67555 (18)	0.0296 (7)
C14	0.0793 (2)	0.3221 (4)	0.6050 (2)	0.0504 (10)
H14A	0.0346	0.3877	0.5942	0.076*
H14B	0.0907	0.2812	0.6580	0.076*
H14C	0.0650	0.2483	0.5648	0.076*
C15	0.1567 (2)	0.4895 (4)	0.5002 (2)	0.0458 (9)
H15A	0.1377	0.4161	0.4601	0.069*
H15B	0.2079	0.5260	0.4992	0.069*
H15C	0.1170	0.5647	0.4883	0.069*
C16	0.18105 (18)	0.1852 (3)	0.21391 (19)	0.0300 (7)
H16	0.2350	0.2263	0.2215	0.036*
C17	0.18047 (18)	0.1071 (3)	0.28879 (18)	0.0300 (7)
C18	0.20171 (19)	0.1534 (4)	0.36798 (19)	0.0351 (8)

H18	0.2207	0.2454	0.3820	0.042*
C19	0.1943 (2)	0.0611 (4)	0.4261 (2)	0.0416 (9)
H19	0.2086	0.0914	0.4797	0.050*
C20	0.1661 (2)	−0.0760 (4)	0.4061 (2)	0.0446 (9)
H20	0.1614	−0.1363	0.4464	0.054*
C21	0.1448 (2)	−0.1240 (4)	0.3274 (2)	0.0371 (8)
H21	0.1256	−0.2160	0.3139	0.045*
C22	0.15257 (18)	−0.0322 (3)	0.26859 (18)	0.0291 (7)
C23	0.13895 (18)	−0.0551 (3)	0.18232 (18)	0.0290 (7)
C24	0.1135 (2)	−0.1740 (4)	0.13442 (19)	0.0357 (8)
H24	0.0997	−0.2566	0.1558	0.043*
C25	0.1090 (2)	−0.1685 (4)	0.0546 (2)	0.0431 (9)
H25	0.0914	−0.2476	0.0214	0.052*
C26	0.1307 (2)	−0.0460 (4)	0.0232 (2)	0.0433 (9)
H26	0.1280	−0.0443	−0.0308	0.052*
C27	0.1561 (2)	0.0732 (4)	0.0705 (2)	0.0379 (8)
H27	0.1704	0.1551	0.0490	0.046*
C28	0.15978 (18)	0.0693 (3)	0.15032 (18)	0.0288 (7)
C29	−0.00303 (19)	0.2408 (4)	0.1666 (2)	0.0381 (8)
H29A	−0.0452	0.3115	0.1488	0.057*
H29B	−0.0025	0.2012	0.2172	0.057*
H29C	−0.0135	0.1671	0.1264	0.057*
C30	0.0978 (2)	0.4220 (4)	0.0885 (2)	0.0395 (8)
H30A	0.0832	0.3587	0.0427	0.059*
H30B	0.1514	0.4601	0.0975	0.059*
H30C	0.0587	0.4980	0.0776	0.059*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0634 (6)	0.0378 (5)	0.0489 (6)	0.0006 (5)	0.0125 (5)	−0.0093 (4)
Cl2	0.0487 (5)	0.0335 (5)	0.0552 (6)	0.0103 (4)	0.0114 (4)	−0.0041 (4)
Si1	0.0329 (5)	0.0295 (5)	0.0320 (5)	−0.0021 (4)	0.0089 (4)	0.0000 (4)
Si2	0.0342 (5)	0.0277 (5)	0.0320 (5)	0.0007 (4)	0.0094 (4)	−0.0008 (4)
C1	0.0342 (17)	0.0309 (17)	0.0265 (16)	0.0004 (14)	0.0129 (14)	−0.0001 (13)
C2	0.0329 (16)	0.0306 (17)	0.0243 (16)	0.0009 (14)	0.0115 (13)	−0.0050 (13)
C3	0.0384 (18)	0.0360 (19)	0.0384 (19)	−0.0025 (16)	0.0172 (15)	−0.0020 (15)
C4	0.0310 (17)	0.051 (2)	0.0398 (19)	0.0053 (17)	0.0137 (15)	−0.0051 (17)
C5	0.043 (2)	0.040 (2)	0.0396 (19)	0.0131 (17)	0.0126 (16)	−0.0002 (16)
C6	0.043 (2)	0.0333 (19)	0.0383 (19)	0.0048 (16)	0.0164 (16)	0.0026 (15)
C7	0.0319 (16)	0.0295 (17)	0.0255 (16)	0.0003 (14)	0.0115 (13)	−0.0050 (13)
C8	0.0312 (16)	0.0317 (18)	0.0281 (16)	0.0002 (14)	0.0123 (13)	−0.0030 (13)
C9	0.041 (2)	0.038 (2)	0.050 (2)	−0.0017 (17)	0.0184 (17)	0.0007 (17)
C10	0.042 (2)	0.051 (2)	0.064 (3)	−0.0120 (19)	0.0246 (19)	−0.007 (2)
C11	0.0282 (18)	0.056 (3)	0.058 (2)	0.0025 (18)	0.0123 (17)	−0.0050 (19)
C12	0.0344 (18)	0.040 (2)	0.0365 (18)	0.0059 (16)	0.0075 (15)	−0.0039 (15)
C13	0.0286 (16)	0.0341 (18)	0.0249 (16)	0.0021 (14)	0.0076 (13)	−0.0043 (13)
C14	0.052 (2)	0.038 (2)	0.062 (3)	−0.0106 (18)	0.020 (2)	−0.0043 (18)

C15	0.048 (2)	0.050 (2)	0.0349 (19)	−0.0006 (18)	0.0077 (17)	−0.0011 (16)
C16	0.0260 (15)	0.0323 (18)	0.0338 (17)	−0.0038 (14)	0.0130 (14)	−0.0036 (14)
C17	0.0247 (15)	0.0360 (18)	0.0300 (17)	0.0029 (14)	0.0104 (13)	−0.0019 (14)
C18	0.0326 (17)	0.0382 (19)	0.0319 (17)	0.0033 (15)	0.0079 (14)	−0.0052 (15)
C19	0.043 (2)	0.052 (2)	0.0282 (17)	0.0133 (18)	0.0091 (15)	−0.0023 (16)
C20	0.056 (2)	0.046 (2)	0.0344 (19)	0.0118 (19)	0.0193 (17)	0.0090 (16)
C21	0.0430 (19)	0.0322 (18)	0.0378 (19)	0.0038 (16)	0.0159 (16)	0.0011 (15)
C22	0.0268 (15)	0.0300 (17)	0.0290 (16)	0.0052 (13)	0.0078 (13)	−0.0007 (13)
C23	0.0266 (16)	0.0328 (18)	0.0292 (16)	0.0060 (14)	0.0115 (13)	0.0024 (13)
C24	0.0414 (19)	0.0315 (18)	0.0341 (18)	−0.0018 (15)	0.0128 (15)	−0.0011 (14)
C25	0.051 (2)	0.039 (2)	0.0349 (19)	−0.0023 (17)	0.0095 (17)	−0.0105 (16)
C26	0.050 (2)	0.053 (2)	0.0263 (18)	0.0032 (18)	0.0132 (16)	−0.0030 (16)
C27	0.0427 (19)	0.038 (2)	0.0367 (19)	−0.0016 (16)	0.0187 (16)	0.0007 (15)
C28	0.0248 (15)	0.0331 (18)	0.0292 (16)	0.0009 (14)	0.0101 (13)	−0.0024 (13)
C29	0.0302 (17)	0.047 (2)	0.0369 (19)	−0.0048 (16)	0.0117 (15)	−0.0031 (16)
C30	0.047 (2)	0.0349 (19)	0.0377 (19)	0.0010 (16)	0.0167 (16)	0.0089 (15)

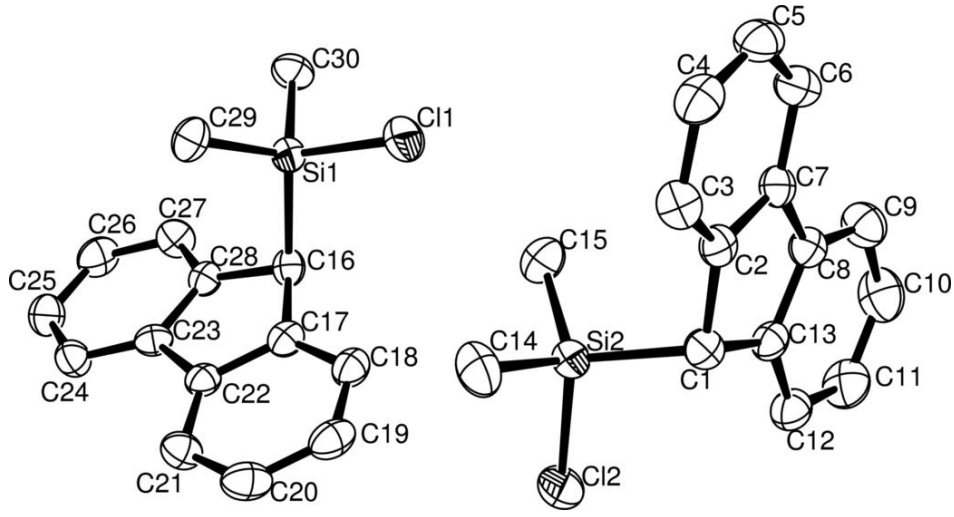
Geometric parameters (Å, °)

Cl1—Si1	2.0653 (15)	C14—H14C	0.9600
Cl2—Si2	2.0768 (15)	C15—H15A	0.9600
Si1—C29	1.852 (3)	C15—H15B	0.9600
Si1—C30	1.861 (3)	C15—H15C	0.9600
Si1—C16	1.885 (3)	C16—C17	1.506 (4)
Si2—C15	1.838 (4)	C16—C28	1.512 (4)
Si2—C14	1.840 (4)	C16—H16	0.9800
Si2—C1	1.881 (3)	C17—C18	1.378 (4)
C1—C13	1.503 (4)	C17—C22	1.402 (4)
C1—C2	1.515 (4)	C18—C19	1.378 (5)
C1—H1	0.9800	C18—H18	0.9300
C2—C3	1.376 (4)	C19—C20	1.383 (5)
C2—C7	1.397 (4)	C19—H19	0.9300
C3—C4	1.384 (5)	C20—C21	1.376 (5)
C3—H3	0.9300	C20—H20	0.9300
C4—C5	1.381 (5)	C21—C22	1.387 (4)
C4—H4	0.9300	C21—H21	0.9300
C5—C6	1.381 (5)	C22—C23	1.463 (4)
C5—H5	0.9300	C23—C24	1.378 (4)
C6—C7	1.387 (4)	C23—C28	1.400 (4)
C6—H6	0.9300	C24—C25	1.375 (5)
C7—C8	1.465 (4)	C24—H24	0.9300
C8—C9	1.381 (5)	C25—C26	1.385 (5)
C8—C13	1.397 (4)	C25—H25	0.9300
C9—C10	1.386 (5)	C26—C27	1.376 (5)
C9—H9	0.9300	C26—H26	0.9300
C10—C11	1.384 (5)	C27—C28	1.377 (4)
C10—H10	0.9300	C27—H27	0.9300
C11—C12	1.376 (5)	C29—H29A	0.9600
C11—H11	0.9300	C29—H29B	0.9600

C12—C13	1.388 (4)	C29—H29C	0.9600
C12—H12	0.9300	C30—H30A	0.9600
C14—H14A	0.9600	C30—H30B	0.9600
C14—H14B	0.9600	C30—H30C	0.9600
C29—Si1—C30	112.15 (16)	Si2—C15—H15A	109.5
C29—Si1—C16	109.99 (15)	Si2—C15—H15B	109.5
C30—Si1—C16	112.65 (15)	H15A—C15—H15B	109.5
C29—Si1—Cl1	107.90 (12)	Si2—C15—H15C	109.5
C30—Si1—Cl1	105.82 (13)	H15A—C15—H15C	109.5
C16—Si1—Cl1	108.06 (11)	H15B—C15—H15C	109.5
C15—Si2—C14	112.55 (18)	C17—C16—C28	102.4 (3)
C15—Si2—C1	109.99 (16)	C17—C16—Si1	111.9 (2)
C14—Si2—C1	113.10 (16)	C28—C16—Si1	107.6 (2)
C15—Si2—Cl2	108.19 (13)	C17—C16—H16	111.5
C14—Si2—Cl2	105.93 (14)	C28—C16—H16	111.5
C1—Si2—Cl2	106.71 (11)	Si1—C16—H16	111.5
C13—C1—C2	102.2 (2)	C18—C17—C22	120.0 (3)
C13—C1—Si2	111.6 (2)	C18—C17—C16	129.9 (3)
C2—C1—Si2	107.5 (2)	C22—C17—C16	110.0 (3)
C13—C1—H1	111.7	C19—C18—C17	118.8 (3)
C2—C1—H1	111.7	C19—C18—H18	120.6
Si2—C1—H1	111.7	C17—C18—H18	120.6
C3—C2—C7	120.1 (3)	C18—C19—C20	121.2 (3)
C3—C2—C1	130.0 (3)	C18—C19—H19	119.4
C7—C2—C1	109.9 (3)	C20—C19—H19	119.4
C2—C3—C4	119.1 (3)	C21—C20—C19	120.7 (3)
C2—C3—H3	120.4	C21—C20—H20	119.7
C4—C3—H3	120.4	C19—C20—H20	119.7
C5—C4—C3	120.9 (3)	C20—C21—C22	118.5 (3)
C5—C4—H4	119.5	C20—C21—H21	120.7
C3—C4—H4	119.5	C22—C21—H21	120.7
C6—C5—C4	120.3 (3)	C21—C22—C17	120.7 (3)
C6—C5—H5	119.8	C21—C22—C23	130.7 (3)
C4—C5—H5	119.8	C17—C22—C23	108.6 (3)
C5—C6—C7	119.0 (3)	C24—C23—C28	120.7 (3)
C5—C6—H6	120.5	C24—C23—C22	130.8 (3)
C7—C6—H6	120.5	C28—C23—C22	108.5 (3)
C6—C7—C2	120.4 (3)	C25—C24—C23	118.9 (3)
C6—C7—C8	130.9 (3)	C25—C24—H24	120.5
C2—C7—C8	108.7 (3)	C23—C24—H24	120.5
C9—C8—C13	121.0 (3)	C24—C25—C26	120.4 (3)
C9—C8—C7	130.8 (3)	C24—C25—H25	119.8
C13—C8—C7	108.2 (3)	C26—C25—H25	119.8
C8—C9—C10	118.4 (3)	C27—C26—C25	121.1 (3)
C8—C9—H9	120.8	C27—C26—H26	119.4
C10—C9—H9	120.8	C25—C26—H26	119.4
C11—C10—C9	120.7 (4)	C26—C27—C28	118.9 (3)
C11—C10—H10	119.6	C26—C27—H27	120.6
C9—C10—H10	119.6	C28—C27—H27	120.6

C12—C11—C10	121.0 (3)	C27—C28—C23	120.0 (3)
C12—C11—H11	119.5	C27—C28—C16	130.0 (3)
C10—C11—H11	119.5	C23—C28—C16	110.0 (3)
C11—C12—C13	118.8 (3)	Si1—C29—H29A	109.5
C11—C12—H12	120.6	Si1—C29—H29B	109.5
C13—C12—H12	120.6	H29A—C29—H29B	109.5
C12—C13—C8	120.0 (3)	Si1—C29—H29C	109.5
C12—C13—C1	129.4 (3)	H29A—C29—H29C	109.5
C8—C13—C1	110.6 (3)	H29B—C29—H29C	109.5
Si2—C14—H14A	109.5	Si1—C30—H30A	109.5
Si2—C14—H14B	109.5	Si1—C30—H30B	109.5
H14A—C14—H14B	109.5	H30A—C30—H30B	109.5
Si2—C14—H14C	109.5	Si1—C30—H30C	109.5
H14A—C14—H14C	109.5	H30A—C30—H30C	109.5
H14B—C14—H14C	109.5	H30B—C30—H30C	109.5

Fig. 1


C30
C29
Cl1
Si1
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C20
C5
C4
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C9
C15
C2
C8
C10
C14
Si2
C1
C13
C11
C12
Cl2