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Iodo(triphenyl)silane

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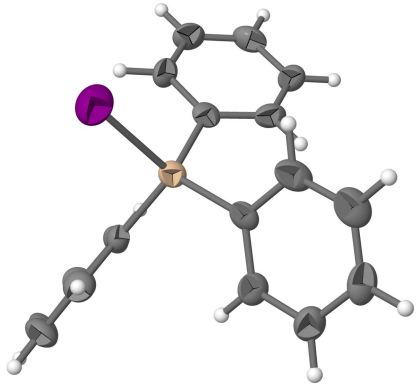
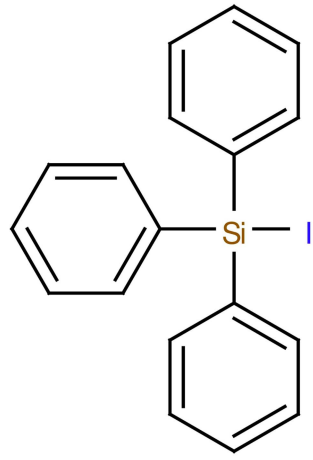
The molecular structure of the title compound, $C_{18}H_{15}ISi$, which crystallizes in the space group $C2/c$, does not exhibit any unusual features. Two weak C—H···π interactions may help to consolidate the packing. The present structure is not isostructural with the known Ph_3SiX (X = F, Cl or Br) compounds.

3D view



Chemical scheme



Structure description

The chemistry and properties of Si—B compounds are of great importance as they are already widely adopted in organic syntheses. Despite the long existence and common usage of these compounds, their reactivity has not yet been thoroughly investigated. In a preliminary study, we investigated the chemical behaviour of supersilylated trieles (Wiberg *et al.*, 1998), which had been synthesized from appropriate triel halides and the donor-unsupported silanide Na[Si^{t}Bu$_3$] (Lerner, 2005). It has been shown that the halides (^{t}Bu$_3$Si)BX_2 and (^{t}Bu$_3$Si)$_2$BX (X = F, Cl and Br) are more labile than the corresponding hydrogen derivatives (Wiberg *et al.*, 2001). In this paper, we report the structure of one of the products from the reaction of BI_3 with donor-unsupported K[SiPh$_3$] (Alig *et al.*, 2019).

The molecular structure of the title compound (Fig. 1) does not exhibit any unusual features. The Si—I bond has a length of 2.478 (2) Å, which compares well with the value of 2.48 (3) Å retrieved from the Cambridge Structural Database (Version 5.40 of November 2018 plus one update; Groom *et al.*, 2016) for a comparable fragment. The crystal packing features two C—H···π interactions, with H···Cg distances less than 3 Å, *viz.* C25—H25···Cg(C11–C16)^{i} = 2.77 Å and C15—H15···Cg(C1–C6)^{ii} = 2.98 Å [symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$].

The crystal structures of fluoro(triphenyl)silane (Brendler *et al.*, 2012), chloro(triphenyl)silane (Lobkovskii *et al.*, 1981) and bromo(triphenyl)silane (Steinert *et al.*, 2008; Brendler *et al.*, 2012) have already been determined. Whereas Ph$_3$SiCl and Ph$_3$SiBr display isomorphous structures (space group $P2_1/c$ with Z = 8), Ph$_3$SiF (space group $P2_1/c$ with Z = 4) and the title compound (space group $C2/c$ with Z = 8) crystallize in a different way.

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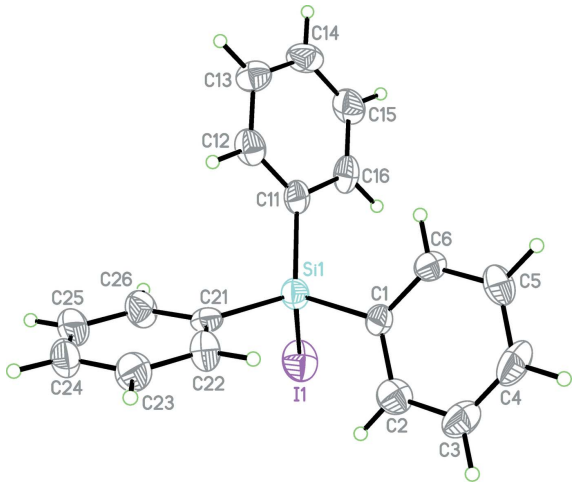

Figure 1
A perspective view of the title compound. Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn with an arbitrary radius.

Synthesis and crystallization

Neat BI_3 (39 mg, 0.10 mmol) was added to $K[SiPh_3]$ (28 mg, 13.87 mmol) suspended in benzene (1 ml) at room temperature. Insoluble material was removed by filtration. The liquor was stored at room temperature for a period of approximately one month, yielding colourless blocks of the title compound (Fig. 2).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The H atoms were refined using a riding model, with C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

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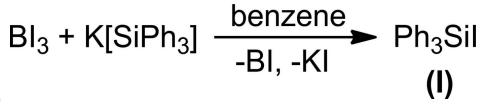

Figure 2
Reaction scheme for the synthesis of the title compound.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{15}ISi$
M_r	386.29
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	173
a, *b*, *c* (Å)	16.0488 (8), 11.2984 (6), 19.648 (1)
β (°)	112.379 (4)
V (Å^3)	3294.4 (3)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	2.01
Crystal size (mm)	0.23 × 0.14 × 0.13
Data collection	
Diffractometer	Stoe IPDS II two-circle
Absorption correction	Multi-scan (*X-AREA*; Stoe & Cie, 2001)
T_{min}, T_{max}	0.411, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12164, 2901, 2338
R_{int}	0.052
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.073, 0.153, 1.34
No. of reflections	2901
No. of parameters	181
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.02, −0.80

Computer programs: *X-AREA* (Stoe & Cie, 2001), *SHELXS* and *XP* in *SHELXTL-Plus* (Sheldrick, 2008), *SHELXL2018* (Sheldrick, 2015) and *publCIF* (Westrip, 2010).

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full crystallographic data

IUCrData (2019). **4**, x190959 [https://doi.org/10.1107/S2414314619009593]

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Iodo(triphenyl)silane

Crystal data

$C_{18}H_{15}ISi$
M_r = 386.29
Monoclinic, $C2/c$
a = 16.0488 (8) Å
b = 11.2984 (6) Å
c = 19.648 (1) Å
β = 112.379 (4)°
V = 3294.4 (3) Å^3
Z = 8
$F(000)$ = 1520
D_x = 1.558 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 12164 reflections
θ = 3.4–26.2°
μ = 2.01 mm^{-1}
T = 173 K
Block, colourless
0.23 × 0.14 × 0.13 mm

Data collection

Stoe IPDS II two-circle diffractometer
Radiation source: Genix 3D IμS microfocus X-ray source
ω scans
Absorption correction: multi-scan (X-AREA; Stoe & Cie, 2001)
T_{min} = 0.411, T_{max} = 1.000
12164 measured reflections
2901 independent reflections
2338 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 25.0°, θ_{min} = 3.5°
h = −19→19
k = −13→13
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.153
S = 1.34
2901 reflections
181 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0192P)^2 + 60.7486P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.02 e Å^{-3}
$\Delta\rho_{min}$ = −0.80 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.48451 (4)	0.29151 (7)	0.36275 (4)	0.0545 (3)
Si1	0.37819 (15)	0.2688 (2)	0.42609 (12)	0.0318 (5)
C1	0.3086 (5)	0.4064 (7)	0.4073 (4)	0.0286 (18)
C2	0.3497 (6)	0.5180 (8)	0.4247 (5)	0.040 (2)
H2	0.413607	0.523733	0.445319	0.048*
C3	0.2981 (7)	0.6198 (8)	0.4121 (5)	0.046 (2)
H3	0.326338	0.694979	0.424663	0.055*
C4	0.2059 (7)	0.6116 (8)	0.3814 (6)	0.051 (3)
H4	0.170471	0.681529	0.372985	0.062*
C5	0.1639 (6)	0.5031 (9)	0.3626 (5)	0.044 (2)
H5	0.100048	0.498101	0.340368	0.053*
C6	0.2161 (6)	0.4014 (8)	0.3766 (5)	0.0342 (19)
H6	0.187274	0.326477	0.364695	0.041*
C11	0.3062 (5)	0.1356 (7)	0.3867 (5)	0.0319 (19)
C12	0.2905 (6)	0.0529 (8)	0.4309 (5)	0.038 (2)
H12	0.319523	0.061021	0.482753	0.046*
C13	0.2340 (6)	−0.0425 (8)	0.4028 (5)	0.038 (2)
H13	0.223722	−0.098452	0.434838	0.045*
C14	0.1922 (6)	−0.0554 (8)	0.3269 (5)	0.044 (2)
H14	0.153113	−0.120504	0.306742	0.052*
C15	0.2075 (6)	0.0254 (8)	0.2818 (5)	0.044 (2)
H15	0.179228	0.016171	0.229906	0.053*
C16	0.2637 (6)	0.1206 (8)	0.3105 (5)	0.040 (2)
H16	0.273743	0.176448	0.278260	0.048*
C21	0.4477 (5)	0.2505 (7)	0.5266 (5)	0.0320 (19)
C22	0.4370 (6)	0.3288 (8)	0.5773 (5)	0.039 (2)
H22	0.395100	0.392077	0.560743	0.047*
C23	0.4872 (7)	0.3149 (9)	0.6516 (5)	0.046 (2)
H23	0.480875	0.369976	0.685812	0.055*
C24	0.5469 (6)	0.2206 (9)	0.6765 (5)	0.045 (2)
H24	0.579917	0.209497	0.727698	0.054*
C25	0.5576 (6)	0.1433 (9)	0.6260 (5)	0.047 (2)
H25	0.599434	0.079949	0.642366	0.056*
C26	0.5082 (6)	0.1578 (8)	0.5524 (5)	0.044 (2)
H26	0.515407	0.103099	0.518381	0.052*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0438 (4)	0.0704 (5)	0.0570 (4)	0.0112 (4)	0.0277 (3)	0.0091 (4)
Si1	0.0317 (12)	0.0303 (12)	0.0301 (12)	0.0037 (10)	0.0080 (10)	−0.0003 (10)
C1	0.032 (4)	0.029 (4)	0.025 (4)	0.002 (3)	0.010 (3)	0.004 (3)
C2	0.036 (5)	0.044 (5)	0.043 (5)	0.000 (4)	0.018 (4)	0.000 (4)
C3	0.056 (6)	0.027 (5)	0.058 (6)	0.003 (4)	0.025 (5)	−0.001 (4)
C4	0.063 (7)	0.030 (5)	0.069 (7)	0.018 (5)	0.034 (6)	0.013 (5)

C5	0.024 (4)	0.050 (6)	0.056 (6)	0.012 (4)	0.013 (4)	0.003 (5)
C6	0.037 (5)	0.028 (4)	0.035 (5)	−0.004 (4)	0.012 (4)	0.001 (4)
C11	0.029 (4)	0.030 (4)	0.036 (5)	0.012 (4)	0.011 (4)	0.002 (4)
C12	0.041 (5)	0.040 (5)	0.028 (5)	0.005 (4)	0.007 (4)	−0.008 (4)
C13	0.045 (5)	0.038 (5)	0.036 (5)	−0.006 (4)	0.021 (4)	−0.003 (4)
C14	0.040 (5)	0.033 (5)	0.055 (6)	−0.007 (4)	0.015 (5)	−0.012 (5)
C15	0.047 (5)	0.046 (6)	0.030 (5)	−0.002 (4)	0.003 (4)	−0.010 (4)
C16	0.048 (5)	0.042 (5)	0.026 (4)	0.011 (4)	0.010 (4)	0.008 (4)
C21	0.032 (4)	0.023 (4)	0.039 (5)	−0.006 (3)	0.011 (4)	−0.002 (3)
C22	0.036 (5)	0.045 (5)	0.034 (5)	0.011 (4)	0.010 (4)	0.006 (4)
C23	0.056 (6)	0.045 (6)	0.032 (5)	−0.004 (5)	0.011 (4)	0.000 (4)
C24	0.038 (5)	0.055 (6)	0.034 (5)	−0.001 (5)	0.005 (4)	0.013 (5)
C25	0.031 (5)	0.045 (6)	0.053 (6)	0.008 (4)	0.003 (4)	0.006 (5)
C26	0.036 (5)	0.037 (5)	0.048 (6)	0.011 (4)	0.006 (4)	−0.003 (4)

Geometric parameters (Å, º)

I1—Si1	2.478 (2)	C13—C14	1.389 (13)
Si1—C1	1.867 (8)	C13—H13	0.9500
Si1—C21	1.873 (9)	C14—C15	1.360 (13)
Si1—C11	1.876 (9)	C14—H14	0.9500
C1—C6	1.374 (11)	C15—C16	1.379 (13)
C1—C2	1.405 (12)	C15—H15	0.9500
C2—C3	1.384 (12)	C16—H16	0.9500
C2—H2	0.9500	C21—C26	1.386 (12)
C3—C4	1.373 (14)	C21—C22	1.391 (12)
C3—H3	0.9500	C22—C23	1.382 (12)
C4—C5	1.380 (14)	C22—H22	0.9500
C4—H4	0.9500	C23—C24	1.391 (13)
C5—C6	1.387 (12)	C23—H23	0.9500
C5—H5	0.9500	C24—C25	1.381 (14)
C6—H6	0.9500	C24—H24	0.9500
C11—C12	1.363 (12)	C25—C26	1.368 (13)
C11—C16	1.399 (12)	C25—H25	0.9500
C12—C13	1.382 (12)	C26—H26	0.9500
C12—H12	0.9500		
C1—Si1—C21	111.8 (4)	C12—C13—C14	119.1 (8)
C1—Si1—C11	111.0 (4)	C12—C13—H13	120.5
C21—Si1—C11	111.9 (4)	C14—C13—H13	120.5
C1—Si1—I1	106.8 (3)	C15—C14—C13	119.8 (8)
C21—Si1—I1	107.0 (3)	C15—C14—H14	120.1
C11—Si1—I1	108.0 (3)	C13—C14—H14	120.1
C6—C1—C2	118.2 (8)	C14—C15—C16	120.6 (8)
C6—C1—Si1	121.2 (6)	C14—C15—H15	119.7
C2—C1—Si1	120.6 (6)	C16—C15—H15	119.7
C3—C2—C1	120.6 (8)	C15—C16—C11	120.6 (8)
C3—C2—H2	119.7	C15—C16—H16	119.7

C1—C2—H2	119.7	C11—C16—H16	119.7
C4—C3—C2	119.7 (9)	C26—C21—C22	118.6 (8)
C4—C3—H3	120.2	C26—C21—Si1	121.5 (7)
C2—C3—H3	120.2	C22—C21—Si1	119.9 (6)
C3—C4—C5	120.8 (9)	C23—C22—C21	120.2 (8)
C3—C4—H4	119.6	C23—C22—H22	119.9
C5—C4—H4	119.6	C21—C22—H22	119.9
C4—C5—C6	119.1 (8)	C22—C23—C24	120.4 (9)
C4—C5—H5	120.4	C22—C23—H23	119.8
C6—C5—H5	120.4	C24—C23—H23	119.8
C1—C6—C5	121.6 (8)	C25—C24—C23	119.2 (8)
C1—C6—H6	119.2	C25—C24—H24	120.4
C5—C6—H6	119.2	C23—C24—H24	120.4
C12—C11—C16	117.8 (8)	C26—C25—C24	120.2 (9)
C12—C11—Si1	121.4 (6)	C26—C25—H25	119.9
C16—C11—Si1	120.8 (7)	C24—C25—H25	119.9
C11—C12—C13	122.2 (8)	C25—C26—C21	121.4 (9)
C11—C12—H12	118.9	C25—C26—H26	119.3
C13—C12—H12	118.9	C21—C26—H26	119.3

C21—Si1—C1—C6	−118.2 (7)	Si1—C11—C12—C13	176.6 (7)
C11—Si1—C1—C6	7.5 (8)	C11—C12—C13—C14	0.7 (14)
I1—Si1—C1—C6	125.0 (7)	C12—C13—C14—C15	0.0 (14)
C21—Si1—C1—C2	61.3 (8)	C13—C14—C15—C16	−0.4 (14)
C11—Si1—C1—C2	−173.0 (7)	C14—C15—C16—C11	0.1 (14)
I1—Si1—C1—C2	−55.5 (7)	C12—C11—C16—C15	0.5 (13)
C6—C1—C2—C3	0.9 (13)	Si1—C11—C16—C15	−177.0 (7)
Si1—C1—C2—C3	−178.7 (7)	C1—Si1—C21—C26	−176.4 (7)
C1—C2—C3—C4	−0.9 (14)	C11—Si1—C21—C26	58.4 (8)
C2—C3—C4—C5	−0.3 (16)	I1—Si1—C21—C26	−59.8 (7)
C3—C4—C5—C6	1.4 (15)	C1—Si1—C21—C22	5.8 (8)
C2—C1—C6—C5	0.3 (13)	C11—Si1—C21—C22	−119.4 (7)
Si1—C1—C6—C5	179.8 (7)	I1—Si1—C21—C22	122.4 (7)
C4—C5—C6—C1	−1.4 (14)	C26—C21—C22—C23	1.2 (13)
C1—Si1—C11—C12	−109.8 (7)	Si1—C21—C22—C23	179.1 (7)
C21—Si1—C11—C12	15.9 (8)	C21—C22—C23—C24	−1.8 (14)
I1—Si1—C11—C12	133.5 (6)	C22—C23—C24—C25	2.1 (14)
C1—Si1—C11—C16	67.7 (8)	C23—C24—C25—C26	−1.7 (15)
C21—Si1—C11—C16	−166.6 (7)	C24—C25—C26—C21	1.2 (15)
I1—Si1—C11—C16	−49.1 (7)	C22—C21—C26—C25	−0.9 (14)
C16—C11—C12—C13	−0.9 (13)	Si1—C21—C26—C25	−178.7 (7)