Crystal structures and Hirshfeld surface analyses of bis(4,5-dihydrofuran-2-yl)dimethylsilane and (4,5-dihydrofuran-2-yl)(methyl)diphenylsilane

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The title compounds, C_{10}H_{16}O_{2}Si (1) and C_{17}H_{18}OSi (2), are classified as dihydrofurylsilanes, which show great potential as building blocks for various functionalized silanes. They both crystallize in the space group P\bar{1} in the triclinic crystal system. Analyses of the Hirshfeld surfaces show packing-determining interactions for both compounds, resulting in a polymeric chain along the [011] for silane 1 and a layered-interconnected structure along the b-axis direction for silane 2.

1. Chemical context

Dihydrofurylsilanes are interesting starting materials for tailor-made silicon compounds. First presented in the 1980s by Lukevics (Lukevics et al., 1985), they turned out to be versatile building blocks for multiple silicon compound classes. Tetra-substituted silicon compounds are obtainable as a result of the excellent nature of the dihydrofuryl (DHF) substituent as a leaving group in various nucleophilic substitutions at the silicon atom. Si—C(DHF) bond cleavages under substitution of the dihydrofuryl group was observed for the reactions with C-nucleophiles (e.g. organolithium compounds) (Gevorgyan et al., 1992), H-nucleophiles (e.g. LiAlH₄, NaH, NaBH₄) (Gevorgyan et al., 1989, 1990), O-nucleophiles (e.g. t-butanol) and N-nucleophiles [e.g. Li[N(Et)₂]] (Lukevics et al., 1997). By means of this efficient pathway, a noteworthy approach to pentacoordinated organyl silatranes has been made (Gevorgyan et al., 1997), as well as for (α-aminomethyl)silanes (Labrecque et al., 1994). Along with their easy preparation and hydrolytical and chromatographical stability (Gevorgyan et al., 1997), dihydrofurylsilanes offer the potential to be useful reagents as protecting groups for the synthesis of aminomethylsilazanes (Colquhoun et al., 2011; Colquhoun & Strohmann, 2012).

Herein, we report the structures of two further dihydrofurylsilanes, bis(4,5-dihydrofuran-2-yl)dimethylsilane (1) and...
(4,5-dihydrofuran-2-yl)(methyl)diphenylsilane (2) and their structural analysis, supplemented by a Hirshfeld surface analysis.

2. Structural commentary

The molecular structure of 1 is given in Fig. 1 and selected bond lengths and angles are given in Table 1. Compound 1 shows $C_2$ molecular symmetry. The lengths of the Si—C(DHF) bonds are similar but slightly longer than the lengths of the Si—C(Me) bonds. However, all bonds have characteristic dimensions (Allen et al., 1987). Furthermore, the length of the C=C double bond corresponds well with literature values (Allen et al., 1987) and is clearly shortened in comparison to the C—C single bonds in the dihydrofuranyl substituent. The silicon atom is tetrahedrally surrounded by its substituents, however slightly distorted as evident from the slight deviations from the ideal angle of 109.47°. These deviations are congruent with a former publication on dihydrofurylsilanes (Krupp et al., 2020). Both DHF planes display planarity while the C1—C4/O1 ring has an r.m.s. deviation of 0.0197 Å from an ideal least-squares plane with atom C4 showing the largest deviation of $-0.0269$ (2) Å. The C5—C8/O2 ring deviates more strongly from an ideal least-square plane with an r.m.s. deviation of 0.0608 Å, with the C8 atom deviating the most by 0.0838 (2) Å. The angle between the normals of the least-squares planes through the DHF rings is 78.943 (15)°.

The molecular structure of 2 is given in Fig. 2 and selected bond lengths and angles are given in Table 2. The length of the Si—C(DHF) bond is in the range of the lengths of the Si—C(Ph) bonds, which are again slightly longer than the Si—C(Me) bond. The bond lengths of the dihydrofuran ring are consistent with those of structure 1. Again, a slightly distorted tetrahedral environment at the silicon atom is observed. The DHF ring is less planar than the phenyl rings, with an r.m.s. deviation from the least-squares plane of 0.0426 Å with the C4 atom having the largest deviation of $-0.0582$ (7) Å. The phenyl rings show r.m.s. deviations of 0.0066 and 0.0047 Å. The angle between the normals of the least-squares planes of the DHF ring and the C5–C10 phenyl ring is 87.68 (4)° and the angle between the normals of the least-squares planes of the phenyl rings is 60.03 (4)°.

3. Supramolecular features

The crystal packing of compound 1 is defined by C10—H10A···C2′ van der Waals interactions as can be seen in Fig. 3. The interactions show relatively large distances [C10···C2′ = 3.6208 (5) Å, H10A···C2′ = 2.752 (10) Å] and C10—

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### Table 1

| Bond Type | Length (Å) | Angle (°) |
|-----------|------------|-----------|
| Si1—C1    | 1.8742 (3) | C1—Si1—C5 108.189 (12) |
| Si1—C5    | 1.8693 (3) | C1—Si1—C9 106.801 (14) |
| Si1—C9    | 1.8631 (3) | C1—Si1—C10 109.191 (13) |
| Si1—C10   | 1.8579 (3) | C5—Si1—C9 110.770 (15) |
|           |            | C5—Si1—C10 108.128 (13) |
| C1—C2     | 1.3370 (4) | C9—Si1—C10 113.628 (16) |
| C3—C4     | 1.5331 (5) |                       |
| C5—C6     | 1.3409 (4) |                       |
| C7—C8     | 1.5298 (5) |                       |

### Table 2

| Bond Type | Length (Å) | Angle (°) |
|-----------|------------|-----------|
| Si1—C1    | 1.8742 (10)| C1—Si1—C5 108.44 (4) |
| Si1—C5    | 1.8721 (9) | C1—Si1—C11 105.51 (4) |
| Si1—C11   | 1.8713 (10)| C1—Si1—C17 109.26 (5) |
| Si1—C17   | 1.8591 (11)| C5—Si1—C11 113.08 (4) |
|           |            | C5—Si1—C17 110.49 (5) |
| C1—C2     | 1.3356 (14)| C11—Si1—C17 109.88 (59) |
| C3—C4     | 1.5416 (17)|                       |

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Figure 1
The molecular structure of compound 1 with displacement ellipsoids drawn at the 50% probability level.

Figure 2
The molecular structure of compound 2 with displacement ellipsoids drawn at the 50% probability level.
H10A⋯C2i = 148.4 (8)°; symmetry code: (i) −x + 1, −y + 2, −z. As a result of the interactions between carbon atom C2 and hydrogen atom H10A, a polymeric chain structure along the [011] direction is formed (Fig. 4). The interactions can be displayed by a Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) generated by CrystalExplorer21 (Spackman et al., 2021), here indicated by the red spots (Fig. 5). The Hirshfeld surface mapped over \( d_{\text{norm}} \) is in the range from −0.0783 to 1.0981 a.u. The contributions of the different types of intermolecular interactions for 1 are shown in the two-dimensional fingerprint plots (McKinnon et al., 2007) in Fig. 6. The contribution of the H⋯H interactions, with a value of 76.4%, has the largest share of the crystal packing of 1. The O⋯H/ H⋯O interactions have a smaller share with a 15% contribution and the C⋯H/H⋯C interactions with a 8.6% contribution. Both heteronuclear interactions appear as spikes.

The structure of compound 2 is more strongly defined by C—H⋯O hydrogen bonds (Fig. 7, Table 3). Two different layers are formed along the b-axis direction and interconnected by hydrogen bonds between the O1 atom and the H17C atom. An additional interaction in each of the layers is observed by C—H⋯O hydrogen bonds between the O1 atom and
and the H15 atom. The C17—H17/C_C1/C_O1ii hydrogen bond can be described by the $R_2^2(10)$ graph-set motif and the C15—H15...O1i hydrogen bond by the $C_1^1(7)$ graph-set motif (Etter et al., 1990). Both C—H...O hydrogen bonds can be identified as weak interactions according to Desiraju & Steiner (2001). A Hirshfeld analysis, carried out analogously as for structure 1, was used to further study the crystal packing. In Fig. 8, the nearest contacts are shown in red. The Hirshfeld surface mapped over $d_{	ext{norm}}$ is in the range from $-0.0717$ to $1.0768$ a.u. By analysis of the two-dimensional fingerprint plots (Fig. 9), again, the biggest contribution to the crystal packing can be assigned to H...H interactions (66%). Although the closest contacts were identified as C—H...O hydrogen bonds, O...H/H...O interactions contribute only 6.4% to the crystal packing, while C...H/H...C interactions have a larger share of 27%.

4. Database survey

A search of the Cambridge Crystallographic Database (WebCSD, November 2021; Groom et al., 2016) for 2-(4,5-dihydrofuryl)silanes revealed solely the structures of tris(4,5-dihydrofuran-2-yl)methylsilane and tris(4,5-dihydrofuran-2-yl)phenylsilane published by our group previously (Krupp et al., 2020). A more extended search for 3-(4,5-dihydrofuryl)silane gave some structures with substituted dihydrofuran rings, such as [4-(4-fluorophenyl)-5-(4-nitrophenyl)-4,5-dihydrofuran-3-yl](trimethyl)silane (JIVLIM; Li & Zhang, 2018), rac-5-phenyl-4-(t-butyldiphenylsilil)-2,3-dihydrofuran-2-carboxylic acid ethyl ester (PUXCAM; Evans et al., 2001) and (1'S,2'R)-5-methyl-4-(t-butyldiphenylsilil)-2,3-dihydrofuran-2-carboxylic acid (1'-phenylethyl)amide (PUXCEQ; Evans et al., 2001). Contrary to the here and previously presented 2-(4,5-dihydrofuryl)silanes (Krupp et al., 2020), the published 3-(4,5-dihydrofuryl)silanes do not show an elongated Si—C(DHF) bond in comparison to the other substituents at the silicon atom. This can be attributed to the changed connection on the DHF ring. The slightly distorted tetrahedral silicon atom can be observed in all structures as well as the shortened C—C double bond in the DHF ring.

5. Synthesis and crystallization

Bis(4,5-dihydrofuran-2-yl)dimethylsilane (1) as already described by Lukevics and co-workers (Lukevics et al., 1985)
was synthesized by adding 1BuLi (1.9 M in pentane, 8.16 mL, 15.5 mmol, 2.0 eq.) to a solution of 2,3-dihydrofuran (1.09 g, 15.5 mmol, 2.0 eq.) in diethyl ether at 243 K and subsequent stirring for an hour. Dichloromethylsilane (1.00 g, 7.75 mmol, 1.0 eq.) was added at 243 K and warmed to room temperature under stirring for 2 h. All solids were filtered off inerly and all volatile components were removed in vacuo. After cleaning by Kugelrohr distillation (temperature: 373 K, pressure: 2.0 mbar), bis(4,5-dihydrofuran-2-yl)dimethylsilane (1) (1.50 g, 7.65 mmol, 99%) was obtained as a colorless oil. By crystallization from diethyl ether at 193 K, colorless blocks were obtained.

$^3$H NMR (400.25 MHz, C$_6$D$_6$): δ = 0.39 [s, 6H; Si(CH$_3$)$_2$], 2.22 [dt, $^3$J$_{HH} = 2.57$ Hz, $^3$J$_{HH} = 9.78$ Hz, 4H; Si(CCH$_2$)$_2$], 4.07 [t, $^3$J$_{HH} = 9.78$ Hz, 4H; Si(COCH$_2$)$_2$], 5.31 [t, $^3$J$_{HH} = 2.57$ Hz, 2H; Si(CCH$_3$)] ppm.

$^{[1]}$H/$^{13}$C NMR (100.6 MHz, C$_6$D$_6$): δ: $^1$H = 3.8 [2C; (SiCH$_3$)$_2$], 31.6 [2C; Si(CCH$_2$)$_2$], 71.8 [2C; Si(COCH$_2$)$_2$], 113.4 [2C; Si(COCH$_2$)$_2$], 160.0 [2C; Si(CO)] ppm.

$^{[1]}$H/$^{29}$Si NMR (79.52 MHz, C$_6$D$_6$): δ = −22.29 [1Si; Si(DHF)] ppm.

GC/EI-MS: $t_r = 3.94$ min [353 K (1 min) − 40 K min$^{-1}$ − 543 K (5.5 min)]; m/z (%): 196 (94) [M$^+$], 181 (2) [(M−Me$^+$)], 167 (14) [(M−CHO)+], 153 (40) [(M−C$_2$H$_3$O$^+$)], 97 (100) [Si(DHF)$^+$].

(4,5-Dihydrofuran-2-yl)(methyl)diphenylsilane (2), already described by Tsai and co-workers (Tsai et al., 1992) by cyclization of a haloacylsilane, was synthesized analogously to 1. 1BuLi (1.90 M in pentane, 21.5 mmol, 11.3 mL, 1.00 eq.) was slowly added dropwise to a solution of 2,3-dihydrofuran (1.51 g, 21.5 mmol, 1.00 eq.) in diethyl ether (80 mL) at 243 K and the reaction solution was stirred for 1 h at this temperature. Methylidiphenylchlorosilane (5.00 g, 21.5 mmol, 1.00 eq.) was then added at 243 K and the reaction solution was stirred at room temperature overnight. All solids were separated by inert filtration and the solvent was removed in vacuo. The residue was purified by Kugelrohr distillation (temperature: 453 K, pressure: 2.0 × 10$^{-1}$ mbar) and the product 2 (5.11 g, 19.2 mmol, 89%) was obtained as a colorless liquid. By crystallization from pentane at 193 K, colorless platelets were obtained.

$^3$H NMR (400.25 MHz, C$_6$D$_6$): δ = 0.69 [s, 3H; SiCH$_3$], 2.24 (dt, $^3$J$_{HH} = 2.57$ Hz, $^3$J$_{HH} = 9.66$ Hz, 2H; SiCCH$_2$)$_2$, 4.07 (t, $^3$J$_{HH} = 9.66$ Hz, 2H; SiCOCH$_2$), 5.20 (t, $^3$J$_{HH} = 2.57$ Hz, 1H; SiCCH), 7.18−7.21 (m, 6H; CH$_{ortho,para}$), 7.71−7.73 (m, 4H; CH$_{meta}$) ppm.

$^{[1]}$H/$^{13}$C NMR (100.65 MHz, C$_6$D$_6$): δ: $^1$H = −3.9 [1C; SiCH$_3$], 31.3 [1C; SiCCH$_2$], 71.1 [1C; SiCCH$_2$], 115.5 [1C; SiCCH], 128.5 (2C, C$_{ortho}$), 130.2 (1C, $C_{para}$), 135.8 (2C, C$_{meta}$), 135.8 (1C, C$_{ipso}$), 160.2 (1C, SiCO) ppm.

$^{[1]}$H/$^{29}$Si NMR (79.52 MHz, C$_6$D$_6$): δ = −19.51 [s, 18Si; Si(DHF)] ppm.

GC/EI-MS: $t_r = 5.97$ min [353 K (1 min) − 40 K min$^{-1}$ − 543 K (5.5 min)]; m/z (%): 266 (100) [M$^+$], 251 (33)
[(M – Me)], 238 (27) [(M – C₂H₄)], 222 (20) [(M – C₂H₄O)], 197 (75) [(M – DHF)], 105 (52) [(SiPh)], 77 (6) [(Ph)].

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. H atoms were positioned geometrically (C—H = 0.95–1.00 Å) and were refined using a riding model, with \(U_{iso}(H) = 1.2U_{eq}(C)\) for CH₂ and CH hydrogen atoms and \(U_{iso}(H) = 1.5U_{eq}(C)\) for CH₃ hydrogen atoms. Hydrogen atoms H2 and H6 for compound 1 and H2, H15 and H17 for compound 2 were refined independently.

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Crystal structures and Hirshfeld surface analyses of bis(4,5-dihydrofuran-2-yl)dimethylsilane and (4,5-dihydrofuran-2-yl)(methyl)diphenylsilane

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Computing details

For both structures, data collection: APEX2 (Bruker, 2018); cell refinement: SAINT (Bruker, 2016); data reduction: SAINT (Bruker, 2016); program(s) used to solve structure: SHELXS (Sheldrick, 2008); program(s) used to refine structure: SHELXL2014/7 (Sheldrick, 2015); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009), CrystalExplorer21 (Spackman et al., 2021), publCIF (Westrip, 2010), Mercury (Macrae et al., 2020).

Bis(4,5-dihydrofuran-2-yl)dimethylsilane (1)

Crystal data

C_{10}H_{16}O_{2}Si

Mr = 196.32

Triclinic, P\(\overline{1}\)

\(a = 8.2422 \text{ (3) Å} \)

\(b = 8.3075 \text{ (4) Å} \)

\(c = 8.2940 \text{ (4) Å} \)

\(a = 94.149 \text{ (2)°} \)

\(\beta = 103.012 \text{ (1)°} \)

\(\gamma = 104.909 \text{ (1)°} \)

\(V = 529.55 \text{ (4) Å}^3 \)

\(Z = 2 \)

\(F(000) = 212 \)

\(D_\text{x} = 1.231 \text{ Mg m}^{-3} \)

Mo Ka radiation, \(\lambda = 0.71073 \text{ Å} \)

Cell parameters from 8689 reflections

\(\theta = 2.5–20.9° \)

\(\mu = 0.19 \text{ mm}^{-1} \)

\(T = 100 \text{ K} \)

Block, colourless

0.72 × 0.66 × 0.59 mm

Data collection

Bruker D8 Venture diffractometer

Radiation source: microfocus sealed X-ray tube,

Incoatec μs

Mirror optics monochromator

Detector resolution: 7.9 pixels mm\(^{-1} \)

\(\omega and \phi \text{ scans} \)

Absorption correction: multi-scan (SADABS; Krause et al., 2015)

Refinement

Refinement on \(F^2 \)

Least-squares matrix: full

\(R[F^2 > 2\sigma(F^2)] = 0.026 \)

\(wR(F^2) = 0.089 \)

\(S = 1.06 \)

11412 reflections

182 parameters

0 restraints

Primary atom site location: iterative

Hydrogen site location: difference Fourier map

H\_atoms\_treated\_by\_a\_mixture\_of\_independent\_and\_constrained\_refinement
supporting information

\[ w = \frac{1}{\sigma^2(F_o^2) + (0.0535P)^2 + 0.0207P} \]
where \( P = (F_o^2 + 2F_c^2)/3 \)
\( (\Delta/\sigma)_{\text{max}} = 0.002 \)
\( \Delta \rho_{\text{max}} = 0.68 \text{ e Å}^{-3} \)
\( \Delta \rho_{\text{min}} = -0.32 \text{ e Å}^{-3} \)

**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     | \( U_{iso}^{/}/U_{eq} \) |
|-----|-------|-------|-------|---------------------------|
| Si1 | 0.64185 (2) | 0.82439 (2) | 0.20897 (2) | 0.01354 (2) |
| O1  | 0.28272 (3) | 0.68623 (3) | 0.09023 (4) | 0.02227 (4) |
| O2  | 0.70145 (4) | 0.71875 (3) | 0.52219 (3) | 0.02213 (4) |
| C1  | 0.41042 (3) | 0.81964 (3) | 0.19462 (3) | 0.01504 (3) |
| C2  | 0.34535 (4) | 0.92843 (4) | 0.26763 (4) | 0.01740 (4) |
| C3  | 0.14995 (4) | 0.87390 (4) | 0.21112 (4) | 0.02057 (4) |
| H3A | 0.0906 (12) | 0.8525 (12) | 0.3024 (12) | 0.032 (2)* |
| H3B | 0.1063 (12) | 0.9549 (11) | 0.1543 (11) | 0.032 (2)* |
| C4  | 0.11591 (4) | 0.70688 (5) | 0.10008 (5) | 0.02242 (5) |
| H4A | 0.0538 (11) | 0.6100 (11) | 0.1453 (11) | 0.032 (2)* |
| H4B | 0.0486 (12) | 0.6963 (12) | −0.0129 (12) | 0.037 (2)* |
| C5  | 0.70144 (3) | 0.67768 (3) | 0.35736 (3) | 0.01419 (3) |
| C6  | 0.73403 (4) | 0.52967 (3) | 0.32841 (3) | 0.01609 (4) |
| C7  | 0.75301 (4) | 0.44890 (4) | 0.48628 (4) | 0.01889 (4) |
| H7A | 0.8533 (11) | 0.4108 (11) | 0.5151 (11) | 0.0284 (18)* |
| H7B | 0.6498 (10) | 0.3515 (11) | 0.4771 (10) | 0.0251 (17)* |
| C8  | 0.75865 (6) | 0.59310 (5) | 0.61494 (4) | 0.02397 (6) |
| H8A | 0.6831 (12) | 0.5602 (11) | 0.6876 (12) | 0.033 (2)* |
| H8B | 0.8762 (14) | 0.6488 (14) | 0.6853 (14) | 0.048 (3)* |
| C9  | 0.77560 (4) | 1.04490 (4) | 0.28985 (5) | 0.02162 (5) |
| H9A | 0.7566 (12) | 1.0847 (12) | 0.3964 (12) | 0.037 (2)* |
| H9B | 0.9008 (14) | 1.0571 (13) | 0.3185 (14) | 0.048 (3)* |
| H9C | 0.7555 (14) | 1.1226 (14) | 0.2124 (13) | 0.047 (3)* |
| C10 | 0.66177 (4) | 0.74698 (4) | 0.00048 (4) | 0.01893 (4) |
| H10A| 0.6108 (13) | 0.8064 (13) | −0.0859 (13) | 0.040 (2)* |
| H10B| 0.6006 (14) | 0.6296 (14) | −0.0355 (13) | 0.045 (3)* |
| H10C| 0.7765 (13) | 0.7669 (12) | −0.0041 (12) | 0.036 (2)* |
| H6  | 0.7325 (10) | 0.4798 (10) | 0.2272 (10) | 0.0225 (16)* |
| H2  | 0.4137 (12) | 1.0273 (11) | 0.3403 (11) | 0.033 (2)* |

**Atomic displacement parameters (Å²)**

|      | \( U_{11} \) | \( U_{22} \) | \( U_{33} \) | \( U_{12} \) | \( U_{13} \) | \( U_{23} \) |
|------|---------------|---------------|---------------|---------------|---------------|---------------|
| Si1  | 0.01424 (3)   | 0.01281 (3)   | 0.01491 (3)   | 0.00527 (2)   | 0.00450 (2)   | 0.00244 (2)   |
| O1   | 0.01651 (7)   | 0.02070 (9)   | 0.02720 (10)  | 0.00574 (6)   | 0.00287 (7)   | −0.00545 (7)  |
| O2   | 0.03579 (12)  | 0.02135 (9)   | 0.01466 (7)   | 0.01572 (9)   | 0.00835 (7)   | 0.00268 (6)   |

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| Atom | U1   | U2   | U3   | U4   | U5   | U6   |
|------|------|------|------|------|------|------|
| C1   | 0.01497 (7) | 0.01501 (7) | 0.01637 (8) | 0.00611 (6) | 0.00417 (6) | 0.00253 (6) |
| C2   | 0.01676 (8) | 0.01709 (8) | 0.01982 (9) | 0.00723 (7) | 0.00523 (7) | 0.00100 (7) |
| C3   | 0.01692 (9) | 0.02220 (11) | 0.02551 (12) | 0.00930 (8) | 0.00669 (8) | 0.00381 (9) |
| C4   | 0.01543 (9) | 0.02642 (13) | 0.02320 (11) | 0.00544 (8) | 0.00243 (8) | −0.00117 (9) |
| C5   | 0.01579 (7) | 0.01407 (7) | 0.01381 (7) | 0.00590 (6) | 0.00406 (6) | 0.00171 (5) |
| C6   | 0.01916 (9) | 0.01436 (8) | 0.01617 (8) | 0.00689 (6) | 0.00502 (7) | 0.00142 (6) |
| C7   | 0.02225 (10) | 0.01595 (8) | 0.02068 (10) | 0.00752 (7) | 0.00644 (8) | 0.00561 (7) |
| C8   | 0.03680 (17) | 0.02108 (11) | 0.01494 (9) | 0.01061 (11) | 0.00498 (9) | 0.00432 (8) |
| C9   | 0.02043 (10) | 0.01447 (9) | 0.02932 (13) | 0.00399 (7) | 0.00669 (9) | 0.00107 (8) |
| C10  | 0.02063 (10) | 0.02226 (10) | 0.01611 (9) | 0.00769 (8) | 0.00686 (7) | 0.00360 (7) |

**Geometric parameters (Å, °)**

| Bond                        | Length (Å) | Angle (°) |
|-----------------------------|------------|-----------|
| Si1—C1                      | 1.8742 (3) |           |
| Si1—C5                      | 1.8693 (3) |           |
| Si1—C9                      | 1.8631 (3) |           |
| Si1—C10                     | 1.8579 (3) |           |
| O1—C1                       | 1.3915 (4) |           |
| O1—C4                       | 1.4479 (4) |           |
| C2—C3                       | 1.5075 (4) |           |
| C2—H2                       | 0.945 (9)  |           |
| C3—H3A                      | 0.992 (9)  |           |
| C3—H3B                      | 0.948 (9)  |           |
| C4—H4A                      | 0.984 (9)  |           |
| C5—Si1—C1                  | 108.189 (12)|          |
| C9—Si1—C1                  | 106.801 (14)|          |
| C9—Si1—C5                  | 110.770 (15)|          |
| C10—Si1—C1                 | 109.191 (13)|          |
| C10—Si1—C5                 | 108.128 (13)|          |
| C10—Si1—C9                 | 113.628 (16)|          |
| C1—O1—C4                   | 107.60 (2) |           |
| C5—O2—C8                   | 107.15 (2) |           |
| C1—C1—Si1                  | 117.093 (19)|          |
| C2—C1—Si1                  | 129.94 (2) |           |
| C2—C1—O1                   | 112.95 (2) |           |
| C1—C2—C3                   | 110.20 (3) |           |
| C1—C2—H2                   | 124.1 (5)  |           |
| C3—C2—H2                   | 125.7 (5)  |           |
| C2—C3—H3A                  | 114.7 (5)  |           |
| C2—C3—H3B                  | 111.8 (5)  |           |
| C2—C3—H3A                  | 108.8 (5)  |           |

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C4—C3—H3B 113.8 (5)  H9A—C9—H9B 102.7 (8)
O1—C4—C3  107.47 (3)  H9A—C9—H9C 108.8 (8)
O1—C4—H4A 108.9 (5)  H9B—C9—H9C 106.8 (9)
O1—C4—H4B 106.6 (6)  Si1—C10—H10A 111.1 (6)
C3—C4—H4A 112.4 (5)  Si1—C10—H10B 112.9 (6)
C3—C4—H4B 116.7 (6)  Si1—C10—H10C 112.3 (6)
H4A—C4—H4B 104.5 (8)  H10A—C10—H10B 105.1 (8)
O2—C5—Si1 116.686 (18)  H10A—C10—H10C 104.8 (8)
C6—C5—Si1 130.12 (2)  H10B—C10—H10C 110.1 (8)

Si1—C1—C2—C3 177.42 (2)  C5—O2—C8—C7 −13.01 (4)
Si1—C5—C6—C7 −172.51 (2)  C5—C6—C7—C8 −10.06 (3)
O1—C1—C2—C3 −1.01 (4)  C6—C7—C8—O2 13.68 (4)
O1—C1—C2—C3 −1.01 (4)  C5—O2—C5—Si1 −177.44 (2)
O2—C5—C6—C7 2.56 (4)  C8—O2—C5—Si1 52.26 (3)
C1—Si1—C5—O2 −64.48 (2)  C8—O2—C5—C6 6.78 (4)
C1—Si1—C5—C6 110.44 (3)  C9—Si1—C1—O1 160.08 (2)
C1—O1—C4—C3 4.15 (4)  C9—Si1—C1—C2 −18.30 (3)
C1—C2—C3—C4 3.38 (4)  C9—Si1—C5—O2 52.26 (3)
C2—C3—C4—O1 −4.46 (4)  C9—Si1—C5—C6 −132.81 (3)
C4—O1—C1—Si1 179.30 (2)  C10—Si1—C1—O1 36.82 (3)
C4—O1—C1—C2 −2.04 (4)  C10—Si1—C1—C2 −141.56 (3)
C5—Si1—C1—O1 −80.64 (2)  C10—Si1—C5—O2 177.38 (2)
C5—Si1—C1—C2 100.98 (3)  C10—Si1—C5—C6 −7.70 (3)

(4,5-Dihydrofuran-2-yl)(methyl)diphenylsilane (2)

Crystal data

C17H18OSi  Z = 2
Mr = 266.40  F(000) = 284
Triclinic, P1  D_x = 1.227 Mg m^{-3}
a = 8.7737 (4) Å  Mo Kα radiation, λ = 0.71073 Å
b = 9.1715 (4) Å  Cell parameters from 8775 reflections
\( c = 9.8130 (4) \, \text{Å} \)  \( \theta = 2.5–36.3° \)
\( \alpha = 102.219 (2)° \)  \( \mu = 0.15 \, \text{mm}^{-1} \)
\( \beta = 90.613 (2)° \)  \( T = 100 \, \text{K} \)
\( \gamma = 110.280 (2)° \)  Plate, colourless
\( V = 720.85 \) (6) Å³  \( 0.51 \times 0.19 \times 0.07 \, \text{mm} \)

Data collection

Bruker D8 Venture  \( T_{\text{min}} = 0.713, \ T_{\text{max}} = 0.747 \)
diffractometer  \( T_{\text{min}} = 0.713, \ T_{\text{max}} = 0.747 \)
Radiation source: microfocus sealed X-ray tube,
INCOATEC microfocus sealed tube, Lys 3.0  \( R_{\text{int}} = 0.030 \)
Multilayer optics monochromator  \( \theta_{\text{max}} = 34.0°, \ \theta_{\text{min}} = 2.8° \)
Detector resolution: 10.4167 pixels mm⁻¹  \( h = -11 \rightarrow 13 \)
φ and ω scans  \( k = -14 \rightarrow 14 \)
Absorption correction: multi-scan  \( l = -15 \rightarrow 15 \)
(SADABS; Krause et al., 2015)
Refinement

Refinement on $F^2$
Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.105$
$S = 1.06$

5826 reflections
244 parameters
0 restraints

Primary atom site location: iterative
Hydrogen site location: difference Fourier map

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 0.2951P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta \sigma)_{\text{max}} < 0.001$
$\Delta \rho_{\text{max}} = 0.48 \text{ e Å}^{-3}$
$\Delta \rho_{\text{min}} = -0.27 \text{ e Å}^{-3}$

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 (Å$^2$)

|      | x        | y        | z        | $U_{eq}/U_{eq}$ |
|------|----------|----------|----------|-----------------|
| Si1  | 0.69854  | 0.37535  | 0.34297  | 0.01325 (7)      |
| O1   | 0.92417  | 0.22237  | 0.28540  | 0.02194 (16)     |
| C1   | 0.86366  | 0.32897  | 0.24356  | 0.01447 (16)     |
| C2   | 0.93945  | 0.38813  | 0.13924  | 0.01886 (18)     |
| C3   | 1.07424  | 0.32450  | 0.10108  | 0.0250 (2)       |
| H3A  | 1.182    | 0.410    | 0.1241   | 0.035 (4)*       |
| H3B  | 1.068    | 0.282    | 0.0052   | 0.037 (4)*       |
| C4   | 1.04574  | 0.20014  | 0.19082  | 0.0241 (2)       |
| H4A  | 1.003    | 0.091    | 0.1329   | 0.037 (4)*       |
| H4B  | 1.143    | 0.213    | 0.2498   | 0.034 (4)*       |
| C5   | 0.50711  | 0.19477  | 0.29840  | 0.01461 (16)     |
| C6   | 0.44747  | 0.11776  | 0.15886  | 0.01701 (17)     |
| H6   | 0.4976   | 0.1611   | 0.0857   | 0.021 (3)*       |
| C7   | 0.31274  | −0.02389 | 0.12562  | 0.01983 (19)     |
| H7   | 0.276    | −0.0716  | 0.0290   | 0.030 (4)*       |
| C8   | 0.23486  | −0.09184 | 0.23187  | 0.0221 (2)       |
| H8   | 0.1407   | −0.1954  | 0.2088   | 0.028 (4)*       |
| C9   | 0.28606  | −0.01617 | 0.37054  | 0.0235 (2)       |
| H9   | 0.232    | −0.063   | 0.4496   | 0.034 (4)*       |
| C10  | 0.42448  | 0.12571  | 0.40346  | 0.01958 (18)     |
| H10  | 0.4595   | 0.1741   | 0.5007   | 0.025 (4)*       |
| C11  | 0.68003  | 0.55198  | 0.28740  | 0.01557 (16)     |
| C12  | 0.54668  | 0.54371  | 0.20270  | 0.01901 (18)     |
| H12  | 0.4588   | 0.4466   | 0.1690   | 0.021 (3)*       |
| C13  | 0.53731  | 0.67991  | 0.16669  | 0.0236 (2)       |
| H13  | 0.447    | 0.6702   | 0.1106   | 0.029 (4)*       |
| C14  | 0.66215  | 0.82626  | 0.21337  | 0.0239 (2)       |
| H14  | 0.6560   | 0.9203   | 0.1908   | 0.029 (4)*       |
| C15  | 0.79733  | 0.83698  | 0.29579  | 0.0234 (2)       |
| C16  | 0.80543  | 0.70145  | 0.33290  | 0.02067 (19)     |
H16 0.902 (2) 0.7112 (19) 0.3922 (17) 0.029 (4)*
C17 0.76169 (14) 0.42855 (14) 0.53383 (11) 0.02152 (19)
H17A 0.779 (2) 0.341 (2) 0.5607 (19) 0.042 (5)*
H17B 0.678 (2) 0.459 (2) 0.5845 (19) 0.041 (5)*
H15 0.886 (2) 0.9381 (19) 0.3294 (16) 0.028 (4)*
H17C 0.863 (2) 0.524 (2) 0.5574 (19) 0.045 (5)*
H2 0.9161 (19) 0.4673 (19) 0.1014 (16) 0.028 (4)*

Atomic displacement parameters (Å²)

|       | U¹¹   | U¹²   | U¹³   | U²²   | U²³   | U³³   |
|-------|-------|-------|-------|-------|-------|-------|
| Si1   | 0.01095 (11) | 0.01317 (12) | 0.01416 (12) | 0.00328 (9) | 0.00118 (8) | 0.00181 (9) |
| O1    | 0.0202 (3) | 0.0199 (3) | 0.0313 (4) | 0.0113 (3) | 0.0083 (3) | 0.0103 (3) |
| C1    | 0.0112 (3) | 0.0134 (4) | 0.0177 (4) | 0.0040 (3) | 0.0002 (3) | 0.0020 (3) |
| C2    | 0.0156 (4) | 0.0227 (5) | 0.0198 (4) | 0.0084 (3) | 0.0040 (3) | 0.0054 (4) |
| C3    | 0.0176 (4) | 0.0309 (6) | 0.0272 (5) | 0.0104 (4) | 0.0078 (4) | 0.0051 (4) |
| C4    | 0.0194 (4) | 0.0225 (5) | 0.0318 (6) | 0.0116 (4) | 0.0051 (4) | 0.0019 (4) |
| C5    | 0.0114 (3) | 0.0150 (4) | 0.0170 (4) | 0.0042 (3) | 0.0020 (3) | 0.0036 (3) |
| C6    | 0.0144 (4) | 0.0176 (4) | 0.0169 (4) | 0.0044 (3) | 0.0023 (3) | 0.0015 (3) |
| C7    | 0.0150 (4) | 0.0186 (4) | 0.0217 (4) | 0.0044 (3) | 0.0004 (3) | 0.0014 (3) |
| C8    | 0.0158 (4) | 0.0165 (4) | 0.0307 (5) | 0.0030 (3) | 0.0023 (4) | 0.0036 (4) |
| C9    | 0.0198 (4) | 0.0210 (5) | 0.0270 (5) | 0.0019 (4) | 0.0052 (4) | 0.0088 (4) |
| C10   | 0.0171 (4) | 0.0197 (4) | 0.0192 (4) | 0.0026 (3) | 0.0025 (3) | 0.0056 (4) |
| C11   | 0.0138 (4) | 0.0149 (4) | 0.0178 (4) | 0.0055 (3) | 0.0038 (3) | 0.0024 (3) |
| C12   | 0.0176 (4) | 0.0185 (4) | 0.0214 (4) | 0.0071 (3) | 0.0017 (3) | 0.0046 (3) |
| C13   | 0.0252 (5) | 0.0249 (5) | 0.0249 (5) | 0.0127 (4) | 0.0020 (4) | 0.0084 (4) |
| C14   | 0.0303 (5) | 0.0206 (5) | 0.0265 (5) | 0.0133 (4) | 0.0083 (4) | 0.0100 (4) |
| C15   | 0.0243 (5) | 0.0159 (4) | 0.0292 (5) | 0.0058 (4) | 0.0070 (4) | 0.0055 (4) |
| C16   | 0.0169 (4) | 0.0169 (4) | 0.0265 (5) | 0.0043 (3) | 0.0019 (4) | 0.0042 (4) |
| C17   | 0.0205 (4) | 0.0237 (5) | 0.0161 (4) | 0.0040 (4) | 0.0001 (3) | 0.0025 (4) |

Geometric parameters (Å, °)

|       | Si—C1   | 1.8743 (10) | C8—H8   | 0.998 (16) |
|-------|---------|-------------|---------|------------|
| Si—C5 | 1.8720 (9) | C8—C9   | 1.3854 (17) |
| Si—C11| 1.8715 (10)| C9—H9   | 1.023 (16) |
| Si—C17| 1.8591 (11)| C9—C10  | 1.3944 (15) |
| O1—C1 | 1.3897 (12)| C10—H10 | 0.960 (15) |
| O1—C4 | 1.4603 (13)| C11—C12 | 1.3988 (14) |
| C1—C2 | 1.3356 (14)| C11—C16 | 1.4050 (14) |
| C2—C3 | 1.5082 (15)| C12—H12 | 0.944 (15) |
| C2—H2 | 0.961 (16)| C12—C13 | 1.3962 (15) |
| C3—H3A| 0.981 (17)| C13—H13 | 0.928 (16) |
| C3—H3B| 0.933 (18)| C13—C14 | 1.3861 (17) |
| C3—C4 | 1.5398 (18)| C14—H14 | 0.953 (16) |
| C4—H4A| 0.982 (17)| C14—C15 | 1.3909 (17) |
| C4—H4B| 0.987 (17)| C15—C16 | 1.3911 (15) |
| C5—C6 | 1.4023 (14)| C15—H15 | 0.969 (16) |

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| Bond          | Distance (Å) | Bond          | Distance (Å) | Angle (°) |
|--------------|--------------|--------------|--------------|-----------|
| C5—C10       | 1.4014 (13)  | C16—H16      | 0.990 (16)   |           |
| C6—H6        | 0.934 (15)   | C17—H17A     | 0.950 (18)   |           |
| C6—C7        | 1.3926 (14)  | C17—H17B     | 0.975 (19)   |           |
| C7—H7        | 0.956 (16)   | C17—H17C     | 0.992 (19)   |           |
| C7—C8        | 1.3919 (16)  |              |              |           |
| C5—Si1—C1    |              | C7—C8—H8     | 120.4 (9)    |           |
| C11—Si1—C1   |              | C9—C8—H8     | 119.76 (10)  |           |
| C11—Si1—C5   | 113.08 (4)   | C9—C8—H8     | 119.8 (9)    |           |
| C17—Si1—C1   | 109.26 (5)   | C8—C9—H9     | 120.5 (9)    |           |
| C17—Si1—C5   | 110.48 (5)   | C8—C9—C10    | 120.10 (10)  |           |
| C17—Si1—C11  | 109.89 (5)   | C10—C9—H9    | 119.4 (9)    |           |
| C1—O1—C4     | 107.48 (8)   | C5—C10—H10   | 121.0 (9)    |           |
| O1—C1—Si1    | 117.13 (7)   | C9—C10—C5    | 121.24 (10)  |           |
| C2—C1—Si1    | 129.71 (8)   | C9—C10—H10   | 117.7 (9)    |           |
| C2—C1—O1     | 113.10 (9)   | C12—C11—Si1  | 123.40 (7)   |           |
| C1—C2—C3     | 110.25 (9)   | C12—C11—C16  | 117.71 (9)   |           |
| C1—C2—H2     | 123.6 (9)    | C16—C11—Si1  | 118.89 (8)   |           |
| C3—C2—H2     | 126.0 (9)    | C11—C12—H12  | 121.5 (9)    |           |
| C2—C3—H3A    | 111.2 (10)   | C13—C12—C11  | 121.09 (10)  |           |
| C2—C3—H3B    | 112.3 (11)   | C13—C12—H12  | 117.4 (9)    |           |
| C2—C3—C4     | 101.51 (9)   | C12—C13—H13  | 118.8 (10)   |           |
| H3A—C3—H3B   | 105.5 (14)   | C14—C13—C12  | 120.16 (10)  |           |
| C4—C3—H3A    | 112.9 (10)   | C14—C13—H13  | 121.0 (10)   |           |
| C4—C3—H3B    | 113.7 (11)   | C13—C14—H14  | 120.8 (10)   |           |
| O1—C4—C3     | 106.70 (8)   | C13—C14—C15  | 119.80 (10)  |           |
| O1—C4—H4A    | 108.8 (10)   | C15—C14—H14  | 119.4 (10)   |           |
| O1—C4—H4B    | 107.0 (10)   | C14—C15—C16  | 119.92 (10)  |           |
| C3—C4—H4A    | 111.5 (10)   | C14—C15—H15  | 121.1 (9)    |           |
| C3—C4—H4B    | 114.2 (10)   | C16—C15—H15  | 119.0 (9)    |           |
| H4A—C4—H4B   | 108.4 (14)   | C11—C16—H16  | 119.7 (9)    |           |
| C6—C5—Si1    | 121.29 (7)   | C15—C16—C11  | 121.32 (10)  |           |
| C10—C5—Si1   | 120.94 (7)   | C15—C16—H16  | 118.9 (9)    |           |
| C10—C5—C6    | 117.64 (9)   | Si1—C17—H17A | 109.9 (11)   |           |
| C5—C6—H6     | 120.3 (9)    | Si1—C17—H17B | 108.7 (11)   |           |
| C7—C6—C5     | 121.26 (9)   | Si1—C17—H17C | 110.5 (11)   |           |
| C7—C6—H6     | 118.4 (9)    | H17A—C17—H17B| 112.2 (15)   |           |
| C6—C7—H7     | 118.2 (10)   | H17A—C17—H17C| 109.4 (15)   |           |
| C8—C7—C6     | 119.98 (10)  | H17B—C17—H17C| 106.1 (15)   |           |
| C8—C7—H7     | 121.8 (10)   |              |              |           |
| Si1—C1—C2—C3 | 175.12 (8)   | C6—C7—C8—C9  | 1.46 (16)    |           |
| Si1—C5—C6—C7 | 174.69 (8)   | C7—C8—C9—C10 | −1.42 (17)   |           |
| Si1—C5—C10—C9| −174.66 (9)  | C8—C9—C10—C5 | 0.07 (17)    |           |
| Si1—C11—C12—C13| 178.63 (8)| C10—C5—C6—C7 | −1.15 (15)   |           |
| Si1—C11—C16—C15| −179.42 (8)| C11—Si1—C1—O1| 167.77 (7)   |           |
| O1—C1—C2—C3  | −2.00 (12)   | C11—Si1—C1—C2| −9.26 (11)   |           |
| C1—Si1—C5—C6 | −51.23 (9)   | C11—Si1—C5—C6| 65.41 (9)    |           |
C1—Si1—C5—C10 124.48 (8)  C11—Si1—C5—C10 −118.88 (9)
C1—Si1—C11—C12 109.11 (9)  C11—C12—C13—C14 0.82 (17)
C1—Si1—C11—C16 −71.17 (9)  C12—C11—C16—C15 0.31 (15)
C1—O1—C4—C3 8.95 (11)  C12—C13—C14—C15 −1.03 (17)
C1—C2—C3—C4 7.17 (12)  C13—C14—C15—C16 0.74 (17)
C2—C3—C4—O1 −9.52 (11)  C14—C15—C16—C11 −1.09 (15)
C4—O1—C1—Si1 177.92 (7)  C17—Si1—C1—C2 −127.35 (10)
C4—O1—C1—C2 −4.57 (12)  C17—Si1—C1—O1 49.68 (8)
C5—Si1—C1—O1 −70.80 (8)  C17—Si1—C5—C6 −170.95 (8)
C5—Si1—C1—C2 112.17 (10)  C17—Si1—C5—C10 4.76 (10)
C5—Si1—C11—C12 −9.26 (10)  C17—Si1—C11—C12 −133.23 (9)
C5—Si1—C11—C16 170.46 (8)  C17—Si1—C11—C16 46.49 (9)
C5—C6—C7—C8 −0.15 (16)  C17—Si1—C5—C10 4.76 (10)
C6—C5—C10—C9 1.20 (15)

Hydrogen-bond geometry (Å, °)

\[
\begin{array}{ccccc}
D—H···A & D—H & H···A & D···A & D—H···A \\
C15—H15···O1\textsuperscript{i} & 0.969 (16) & 2.640 (16) & 3.3422 (13) & 129.6 (12) \\
C17—H17C···O1\textsuperscript{ii} & 0.992 (19) & 2.584 (19) & 3.5168 (14) & 156.5 (15) \\
\end{array}
\]

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

Selected geometric parameters for compound 1 (Å, °)

\[
\begin{array}{ccccc}
 & & & & \\
Si1—C1 & 1.8742 (3) & C1—Si1—C5 & 108.189 (12) \\
Si1—C5 & 1.8693 (3) & C1—Si1—C9 & 106.801 (14) \\
Si1—C9 & 1.8631 (3) & C1—Si1—C10 & 109.191 (13) \\
Si1—C10 & 1.8579 (3) & C5—Si1—C9 & 110.770 (15) \\
C1—C2 & 1.3370 (4) & C5—Si1—C10 & 108.128 (13) \\
C3—C4 & 1.5331 (5) & C9—Si1—C10 & 113.628 (16) \\
C5—C6 & 1.3409 (4) & & & \\
C7—C8 & 1.5298 (5) & & & \\
\end{array}
\]

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