

Syntheses and crystal structures of 2-methyl-1,1,2,3,3-pentaphenyl-2-silapropane and 2-methyl-1,1,3,3-tetraphenyl-2-silapropan-2-ol

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The sterically hindered silicon compound 2-methyl-1,1,2,3,3-pentaphenyl-2-silapropane, C₃₃H₃₀Si (**I**), was prepared *via* the reaction of two equivalents of diphenylmethyl lithium (benzhydryllithium) and dichloromethylphenylsilane. This bisbenzhydryl-substituted silicon compound was then reacted with trifluoromethanesulfonic acid, followed by hydrolysis with water to give the silanol 2-methyl-1,1,3,3-tetraphenyl-2-silapropan-2-ol, C₂₇H₂₆OSi (**II**). Key geometric features for **I** are the Si—C bond lengths that range from 1.867 (2) to 1.914 (2) Å and a τ_4 descriptor for fourfold coordination around the Si atom of 0.97 (indicating a nearly perfect tetrahedron). Key geometric features for compound **II** include Si—C bond lengths that range from 1.835 (4) to 1.905 (3) Å, a Si—O bond length of 1.665 (3) Å, and a τ_4 descriptor for fourfold coordination around the Si atom of 0.96. In compound **II**, there is an intramolecular C—H···O hydrogen bond present. In the crystal of **I**, molecules are linked by two pairs of C—H··· π interactions, forming dimers that are linked into ribbons propagating along the *b*-axis direction. In the crystal of **II**, molecules are linked by C—H··· π and O—H··· π interactions that result in the formation of ribbons that run along the *a*-axis direction.

1. Chemical context

The benzhydryl substituent and its derivatives occur in many medicinal compounds, for example: diphenhydramine, modafinil and meclizine (Fig. 1). The addition of the benzhydryl group to a drug significantly increases its lipophilicity and the two aromatic rings add electron density and bulk. There is an active field looking at the switching of silicon for carbon to discover new medicinal compounds and there have been several recent publications and reviews in the area (Franz & Wilson, 2013; Geyer *et al.*, 2015; Ramesh & Reddy, 2018; Tacke & Doerrich, 2016). It seemed to us that another option is to replace the sulfoxide group with a silanol, in which the silicon has a size that is similar to sulfur and the alcohol will occupy the space of the sulfoxide oxygen. The conversion of a

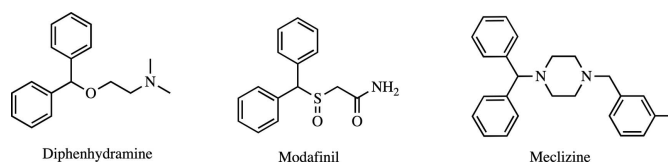
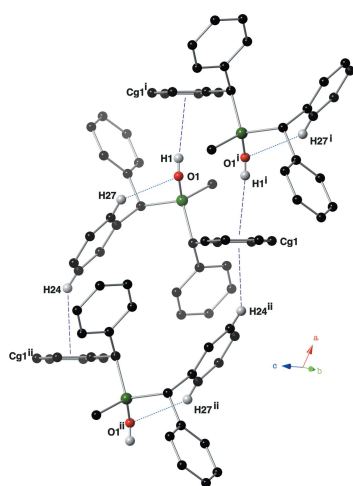
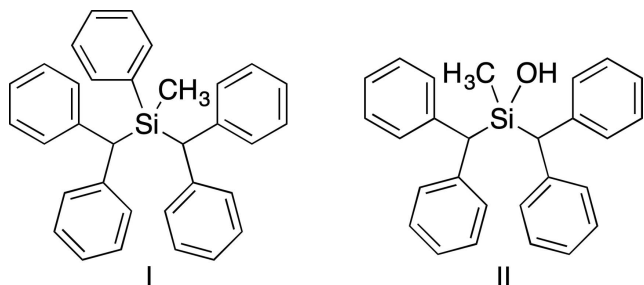


Figure 1
The benzhydryl group in some representative medicinal compounds.

phenylsilane to a silanol by the reaction with trifluoromethanesulfonic acid followed by hydrolysis has been used previously (Kira *et al.*, 2007; Shainyan *et al.*, 2017), and worked well for the introduction of the silanol in compound **I**, silanol 2-methyl-1,1,3,3-tetraphenyl-2-silapropan-2-ol.



The steric bulk of the benzhydryl group has been used to advantage in several silyl reagents. It has been reported that the benzhydryldimethylsilyl group is readily synthesized and undergoes facile oxidation with hydrogen peroxide to form alcohols (Peng & Woerpel, 2001). Yoshida and coworkers have started with a tris(diphenylmethyl)silane and further substituted aromatic rings using electrophilic aromatic substitution to produce the sterically demanding TEDAMS group (Terao *et al.*, 2010). Unno *et al.* (2006) have addressed the influence of bulky silyl groups on the ability of silanols to hydrogen bond, and found that (*i*-Pr₃Si)₃SiOH exists as a monomer while (*t*-BuMe₂Si)₃SiOH is a hydrogen-bonded dimer. Our observation that compound **II** is monomeric indicates that the silicon atom is very hindered by the presence of the two benzhydryl groups.

2. Structural commentary

The molecular structure of compound **I** is shown in Fig. 2. The Si–C bond lengths range from 1.867 (2) to 1.914 (2) Å, with the Si–C1 bond to the methyl group being the shortest. The τ_4 descriptor for fourfold coordination around Si1 is 0.97, indicating a nearly perfect tetrahedral geometry around this silicon atom (where 0 = square planar, 0.85 = trigonal pyramidal, and 1 = tetrahedral; Yang *et al.*, 2007). The Si1–C1 bond and aromatic ring (C4–C9) are nearly co-planar with a C1–Si1–C4–C5 torsion angle of 12.2 (2)°. The orientation of the benzhydryl group bonded to C2 is such that when the molecule is viewed down the C2–Si1 bond the methyl group (C1) is *anti* to H2 (torsion angle C1–Si1–C2–H2 is 169°), with the aromatic rings *gauche*. For the benzhydryl group containing C3, the hydrogen atom H3 is *gauche* to the methyl group (C1) with a C1–Si1–C3–H3 torsion angle of 69°, with the aromatic ring (C22–C27) occupying the *anti* position.

The molecular structure of compound **II** is shown in Fig. 3. The Si–C bond lengths range from 1.835 (4) to 1.905 (3) Å, with an Si–O bond length of 1.665 (3) Å. The τ_4 descriptor for fourfold coordination around Si1 is 0.96, again indicating an almost perfect tetrahedral geometry around this silicon atom. The orientation of the C2 benzhydryl group is such that

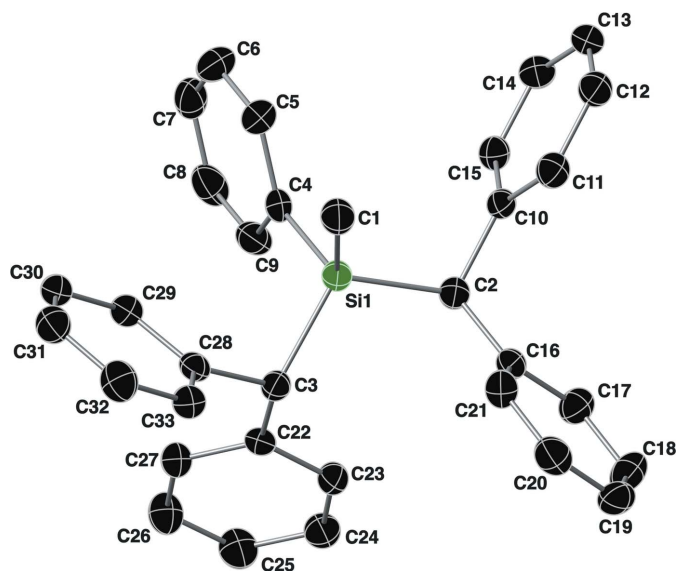


Figure 2
The molecular structure of compound **I**, with the atom-labeling scheme. Displacement ellipsoids are drawn at the 40% probability level. For clarity, the hydrogen atoms have been omitted.

the hydrogen atom H2 is *anti* to the methyl group (C1) with a C1–Si1–C2–H2 torsion angle of -165° . For the benzhydryl group containing C3, the hydrogen atom H3 is again *gauche* to the methyl group (C1) with a C1–Si1–C3–H3 torsion angle of 55° , and the aromatic ring C22–C27 occupies the *anti* position. An intramolecular C–H...O hydrogen bond is present between H27 and O1 with an H...O distance of 2.55 Å (Table 2).

3. Supramolecular features

In the crystal of **I**, molecules are linked by two pairs of intermolecular C–H... π interactions involving inversion-

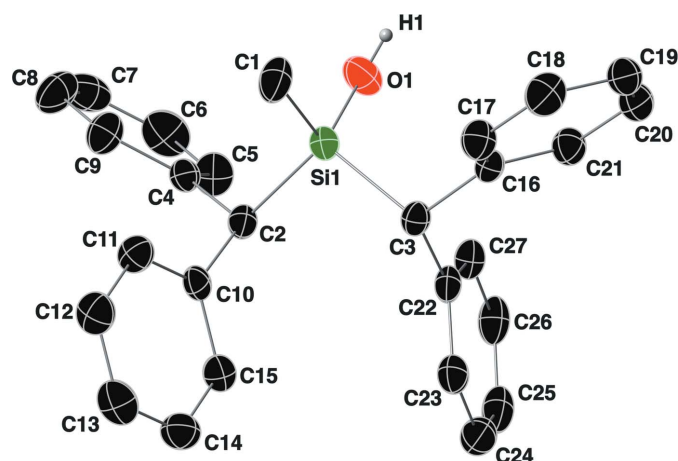


Figure 3
The molecular structure of compound **II**, with the atom-labeling scheme. Displacement ellipsoids are drawn at the 40% probability level. For clarity, the C-bound hydrogen atoms have been omitted.

Table 1

 Hydrogen-bond geometry (Å, °) for **I**.

Cg2 and Cg4 are the centroids of the C10–C15 and C22–C27 rings, respectively.

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|-------|-------------|-------------|---------------|
| C20–H20 \cdots Cg4 ⁱ | 0.95 | 2.94 | 3.716 (2) | 140 |
| C24–H24 \cdots Cg2 ⁱⁱ | 0.95 | 2.75 | 3.696 (2) | 175 |

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

Table 2

 Hydrogen-bond geometry (Å, °) for **II**.

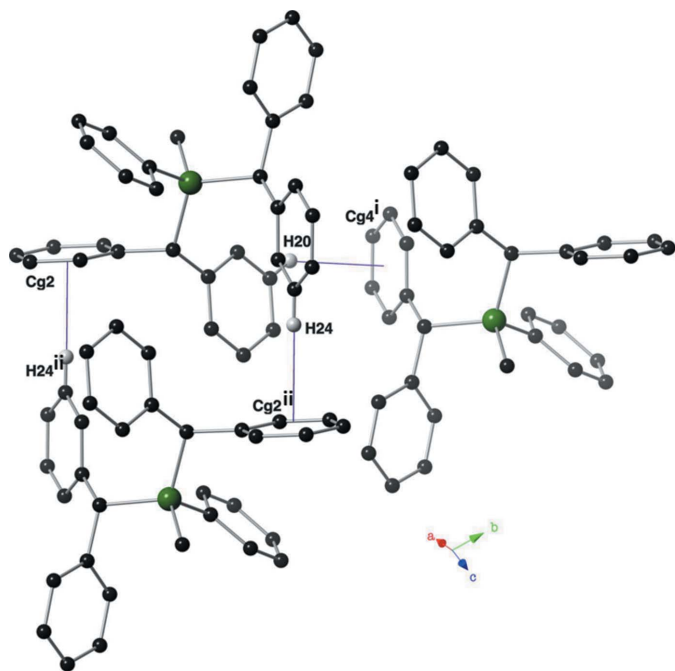
Cg1 is the centroid of the C4–C9 ring.

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|----------|-------------|-------------|---------------|
| C27–H27 \cdots O1 | 0.93 | 2.55 | 3.237 (4) | 131 |
| O1–H1 \cdots Cg1 ⁱ | 0.75 (8) | 2.70 (7) | 3.416 (3) | 162 (7) |
| C24–H24 \cdots Cg1 ⁱⁱ | 0.93 | 2.86 | 3.582 (4) | 135 |

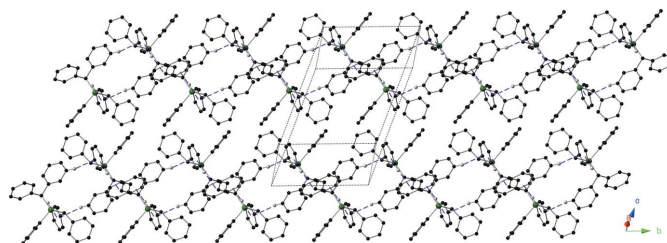
 Symmetry codes: (i) $-x + 1, -y + 1, -z$; (ii) $-x, -y + 1, -z$.

related compounds (Fig. 4 and Table 1). The result of these interactions is the formation of dimers that are linked to form ribbons along the b -axis direction (Fig. 5).

In the crystal of **II**, inversion-related molecules are linked by a pair of O–H \cdots π interactions, forming dimers (Table 2, Fig. 6). Similar interactions between aryl groups and OH groups in silanols have been reported previously (Al-Juaid *et al.*, 1992). In the crystal of **II**, the dimers are linked by a pair of C–H \cdots π interactions (Table 2), so forming ribbons that propagate along the a -axis direction (Fig. 7).


Figure 4

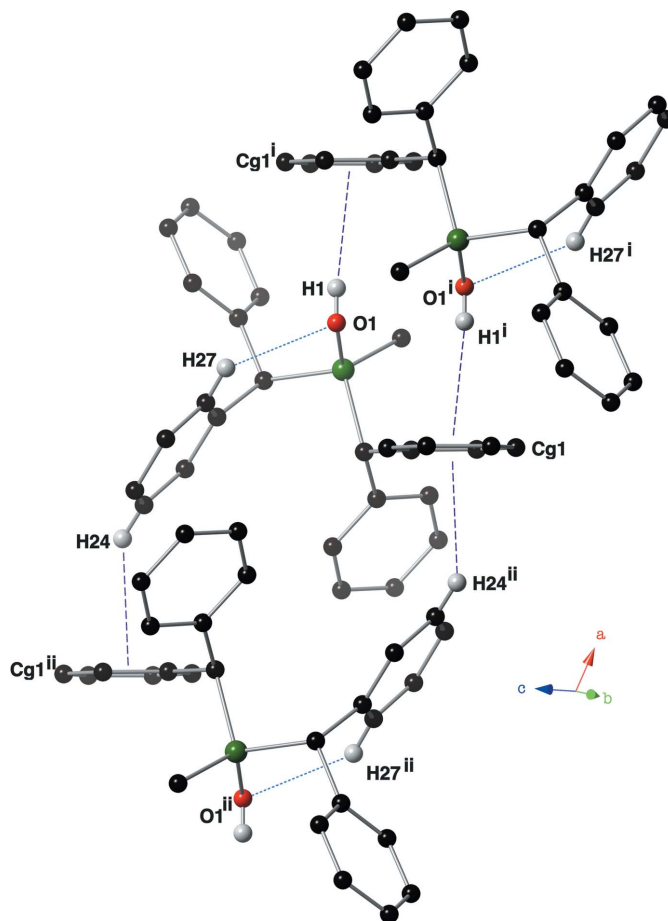
Intermolecular C–H \cdots π interactions present in the crystal of compound **I**; see Table 1 for details. Only hydrogen atoms H24 and H20 are shown for clarity, and the C–H \cdots π interactions are depicted as purple lines. Symmetry codes: (i) $-x + 2, -y, -z$; (ii) $-x + 2, -y + 1, -z$.


Figure 5

The crystal packing of compound **I**, viewed along the a -axis, showing the supramolecular ribbons formed by intermolecular C–H \cdots π interactions (Table 1; shown as dashed purple lines). Only hydrogen atoms H20 and H24 are shown for clarity.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.40, May 2019; Groom *et al.*, 2016) gave only one hit for a structure in which a silicon atom is bonded to two benzhydryl groups, *viz.* bis(diethylamino)bis(diphenylmethyl)silane (CSD refcode YEPTUI; Huppmann, *et al.*, 1994). In


Figure 6

Intramolecular hydrogen bond (blue dotted lines) and intermolecular C–H \cdots π and O–H \cdots π interactions (Table 2; purple dashed lines) present in the crystal of compound **II**. For clarity, only hydrogen atoms H1, H24 and H27 have been included. Symmetry codes: (i) $-x + 1, -y + 1, -z$; (ii) $-x, -y + 1, -z$.

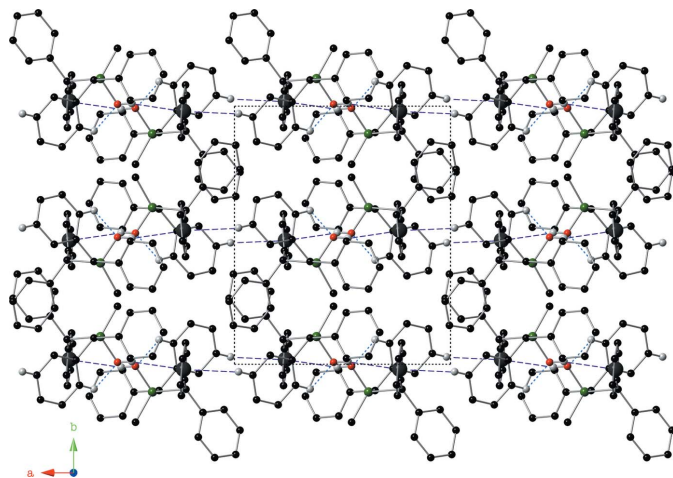


Figure 7

The crystal packing of compound **II**, viewed along the *c*-axis, showing the supramolecular ribbons formed by O—H··· π and C—H··· π interactions (Table 2). For clarity, only hydrogen atoms H1, H24 and H27 have been included.

this compound, the silicon atom is also bonded to two diethylamino groups. There are four other structures in the CSD with a silicon atom bonded to one benzhydryl group and a different alkyl group (this count excludes organometallic compounds). These compounds include, *tert*-butyl 1'-acetyl-4-[(diphenylmethyl)(dimethyl)silyl]-5'-fluoro-2'-oxo-1',2'-dihydrospiro[cyclopentane-1,3'-indole]-2-carboxylate (SOSZIL; Ball-Jones *et al.*, 2014), (3*S*,4*R*,5*S*)-4-[dimethyl-(diphenylmethyl)silyl]-5-[[dimethyl(phenyl)silyl]methyl]-3-methyl-tetrahydrofuran-2-one (XICWUB; Peng & Woerpel, 2001), diphenyl(trimethylsilyl)methane (MOQWY; Hill & Hitchcock, 2002) and diphenyl[*t*-butyl(dimethyl)silyl]methane (MOQWEU; Hill & Hitchcock, 2002). This search revealed zero structures in the CSD that contained a silanol group where the silicon atom is bonded to a benzhydryl group. However, the related structures (triphenylmethyl)silanetriol acetone solvate (GAWVUW; Kim, *et al.*, 2005) and (triphenylmethyl)silanetriol tetrahydrofuran solvate (BAVQOF; Yoo, *et al.*, 2001) are both silanetriols that bear a trityl group (—CPh₃) coordinated to the central silicon atom.

5. Synthesis and crystallization

Synthesis of 2-methyl-1,1,2,3,3-pentaphenyl-2-silapropane (I): Diphenylmethane (1.68 g, 10 mmol) was added to an oven-dried, argon-flushed 100 ml Schlenk flask along with a magnetic stirbar. Anhydrous tetrahydrofuran (10 ml) was then added to the flask to dissolve the solid and the solution was cooled to 273 K. After the solution had cooled for 10 min, *n*-butyllithium (6.25 ml, 1.6 M in hexanes, 10 mmol) was added and the solution was stirred for 1 h. The reaction mixture was then cooled further to 195 K and dichloromethylphenylsilane was added (0.955 g, 5 mmol). After warming to room temperature and stirring for 12 h, the solution was poured into hexanes (20 ml) and the organic layer was washed with water (20 ml), dilute hydrochloric acid (3 N, 10 ml), water (10 ml)

and finally brine (10 ml). The hexanes solution was dried over sodium sulfate, filtered and concentrated *in vacuo*. The product was purified by dissolving it in 20 ml hexane, cooling to 195 K and isolating the white crystals by filtration. The crystals were then washed with pentane and dried *in vacuo* (2.1 g, 93% yield). Colorless block-like crystals suitable for analysis by X-ray diffraction were grown by recrystallization of compound **I** (0.1 g) from hexanes (2 ml) with heating (0.08 g isolated yield). FT-IR (ν , cm⁻¹): 3057, 3019, 2869, 1597, 1493, 696; ¹H NMR (400 MHz, chloroform-*d*) δ 0.39 (*s*, 3H), 3.87 (*s*, 2H), 6.8–7.4 (*m*, 25H); ¹³C NMR (101 MHz, chloroform-*d*) δ -4.77, 42.70, 125.28, 125.67, 127.37, 128.14, 128.49, 129.21, 129.46, 129.81, 134.62, 135.99, 142.02, 142.14; ²⁹Si NMR (79 MHz, chloroform-*d*) δ -3.12.

Synthesis of 2-methyl-1,1,3,3-tetraphenyl-2-silapropan-2-ol (II):

Bis(diphenylmethyl)methylphenylsilane (0.455 g, 1.0 mmol) was added to an oven-dried, argon-flushed 50 ml Schlenk flask along with a stirbar. Anhydrous toluene (5 ml) was added to dissolve the solid and the solution was cooled to 273 K. Trifluoromethanesulfonic acid was weighed in a vial (150 mg, 1 mmol) and then added to the Schlenk flask using a Pasteur pipette, at which point the solution went from colorless to a bright yellow. The solution was stirred for 2 h at room temperature after which time the solution went from cloudy to clear. At this point a mixture of water (40 mg, 2.2 mmol) and triethylamine (200 mg, 2.0 mmol) in ether (2 ml) was prepared and added to the rapidly stirring solution of the triflate in toluene, which caused the yellow solution to immediately turn colorless. After stirring for 1 h, the mixture was poured into hexanes (20 ml) and the organic layer was washed with water (20 ml), dilute hydrochloric acid (3 N, 10 ml), water (10 ml) and finally brine (10 ml). The hexanes solution was dried over sodium sulfate, filtered and the solvent was removed *in vacuo*. The crude product was then dissolved in 5 ml hexane and cooled to 195 K. The white crystals were isolated by vacuum filtration, washed with pentane and dried *in vacuo* (319 mg, 81% yield). Colourless block-like crystals suitable for analysis by X-ray diffraction were grown by recrystallization of compound **II** (0.4 g) from hexanes (5 ml) with heating (0.3 g isolated yield, m.p. (uncorrected) 375.8–376.2 K). FT-IR (ν , cm⁻¹): 3591, 3057, 3024, 1597, 1491, 696; ¹H NMR (400 MHz, chloroform-*d*) δ 0.15 (*s*, 3H), 3.49 (*s*, 2H), 7.22 (*m*, 20H). ¹³C NMR (101 MHz, chloroform-*d*) δ -2.34, 44.44, 125.64, 125.68, 128.62, 129.06, 129.28, 141.20, 141.43. ²⁹Si NMR (79 MHz, chloroform-*d*) δ 7.98.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. For both compounds, the hydrogen atoms bonded to carbon atoms were placed in calculated positions and refined as riding: C—H = 0.95–1.00 Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C-methyl})$ and $1.2U_{\text{eq}}(\text{C})$ for other H atoms in compound **I**, and C—H = 0.93–0.98 Å with $1.2U_{\text{eq}}(\text{C})$ in compound **II**. The hydrogen atom bonded to O1 (H1) in compound **II** was located in an electron-density difference map and freely refined.

Table 3
Experimental details.

| | I | II |
|--|--|--|
| Crystal data | | |
| Chemical formula | C ₃₃ H ₃₀ Si | C ₂₇ H ₂₆ OSi |
| <i>M_r</i> | 454.66 | 394.57 |
| Crystal system, space group | Triclinic, <i>P</i> $\bar{1}$ | Monoclinic, <i>P</i> ₂ / <i>c</i> |
| Temperature (K) | 173 | 173 |
| <i>a</i> , <i>b</i> , <i>c</i> (Å) | 10.3879 (7), 10.5037 (7), 13.6350 (9) | 11.8576 (5), 13.2995 (6), 14.3948 (6) |
| α , β , γ (°) | 68.8212 (7), 70.6364 (7), 84.7947 (8) | 90, 110.363 (3), 90 |
| <i>V</i> (Å ³) | 1308.06 (15) | 2128.20 (16) |
| <i>Z</i> | 2 | 4 |
| Radiation type | Mo <i>K</i> α | Cu <i>K</i> α |
| μ (mm ⁻¹) | 0.11 | 1.08 |
| Crystal size (mm) | 0.21 × 0.21 × 0.17 | 0.10 × 0.09 × 0.08 |
| Data collection | | |
| Diffractometer | Bruker APEXII CCD | Bruker APEXII CCD |
| Absorption correction | Multi-scan (<i>SADABS</i> ; Bruker, 2013) | Multi-scan (<i>SADABS</i> ; Bruker, 2013) |
| <i>T</i> _{min} , <i>T</i> _{max} | 0.695, 0.745 | 0.624, 0.754 |
| No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections | 16773, 4803, 3734 | 11901, 4113, 2414 |
| <i>R</i> _{int} | 0.036 | 0.096 |
| (sin θ /λ) _{max} (Å ⁻¹) | 0.603 | 0.617 |
| Refinement | | |
| <i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i> | 0.045, 0.120, 1.08 | 0.062, 0.171, 0.97 |
| No. of reflections | 4803 | 4113 |
| No. of parameters | 308 | 267 |
| H-atom treatment | H-atom parameters constrained | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³) | 0.34, -0.22 | 0.29, -0.36 |

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS* (Sheldrick, 2008), *SHELXL* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009; Bourhis *et al.*, 2015) and *CrystalMaker* (Palmer, 2007).

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supporting information

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Syntheses and crystal structures of 2-methyl-1,1,2,3,3-pentaphenyl-2-silapropane and 2-methyl-1,1,3,3-tetraphenyl-2-silapropan-2-ol

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Computing details

For both structures, data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: *SHELXS* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009; Bourhis *et al.*, 2015); software used to prepare material for publication: *CrystalMaker* (Palmer, 2007).

2-Methyl-1,1,2,3,3-pentaphenyl-2-silapropane (I)

Crystal data

| | |
|----------------------------------|---|
| $C_{33}H_{30}Si$ | $Z = 2$ |
| $M_r = 454.66$ | $F(000) = 484$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.154 \text{ Mg m}^{-3}$ |
| $a = 10.3879 (7) \text{ \AA}$ | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| $b = 10.5037 (7) \text{ \AA}$ | Cell parameters from 6620 reflections |
| $c = 13.6350 (9) \text{ \AA}$ | $\theta = 2.2\text{--}25.4^\circ$ |
| $\alpha = 68.8212 (7)^\circ$ | $\mu = 0.11 \text{ mm}^{-1}$ |
| $\beta = 70.6364 (7)^\circ$ | $T = 173 \text{ K}$ |
| $\gamma = 84.7947 (8)^\circ$ | Block, colourless |
| $V = 1308.06 (15) \text{ \AA}^3$ | $0.21 \times 0.21 \times 0.17 \text{ mm}$ |

Data collection

| | |
|--|--|
| Bruker APEXII CCD diffractometer | 4803 independent reflections |
| φ and ω scans | 3734 reflections with $I > 2\sigma(I)$ |
| Absorption correction: multi-scan (SADABS; Bruker, 2013) | $R_{\text{int}} = 0.036$ |
| $T_{\text{min}} = 0.695$, $T_{\text{max}} = 0.745$ | $\theta_{\text{max}} = 25.4^\circ$, $\theta_{\text{min}} = 1.7^\circ$ |
| 16773 measured reflections | $h = -12 \rightarrow 12$ |
| | $k = -12 \rightarrow 12$ |
| | $l = -16 \rightarrow 16$ |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.045$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.120$ | H-atom parameters constrained |
| $S = 1.08$ | |
| 4803 reflections | |
| 308 parameters | |
| 0 restraints | |

$$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 0.3536P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.34 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|---------------|----------------------------------|
| Si1 | 0.75666 (5) | 0.17147 (5) | 0.27134 (4) | 0.02998 (15) |
| C1 | 0.58208 (18) | 0.0942 (2) | 0.31735 (16) | 0.0399 (5) |
| H1A | 0.580675 | 0.040999 | 0.271655 | 0.060* |
| H1B | 0.516044 | 0.166936 | 0.309113 | 0.060* |
| H1C | 0.557892 | 0.034234 | 0.395435 | 0.060* |
| C2 | 0.78889 (17) | 0.32685 (18) | 0.13751 (14) | 0.0293 (4) |
| H2 | 0.875549 | 0.371155 | 0.128137 | 0.035* |
| C3 | 0.88432 (17) | 0.03163 (18) | 0.25388 (14) | 0.0290 (4) |
| H3 | 0.879419 | 0.013883 | 0.187902 | 0.035* |
| C4 | 0.77121 (19) | 0.23587 (18) | 0.37890 (15) | 0.0337 (4) |
| C5 | 0.6560 (2) | 0.2462 (2) | 0.46416 (16) | 0.0436 (5) |
| H5 | 0.569129 | 0.220352 | 0.467114 | 0.052* |
| C6 | 0.6643 (3) | 0.2927 (2) | 0.54426 (18) | 0.0549 (6) |
| H6 | 0.583678 | 0.298764 | 0.601188 | 0.066* |
| C7 | 0.7882 (3) | 0.3304 (2) | 0.54221 (19) | 0.0539 (6) |
| H7 | 0.793896 | 0.361141 | 0.598221 | 0.065* |
| C8 | 0.9045 (2) | 0.3235 (2) | 0.45856 (19) | 0.0492 (6) |
| H8 | 0.990631 | 0.350498 | 0.456297 | 0.059* |
| C9 | 0.8961 (2) | 0.2771 (2) | 0.37750 (17) | 0.0401 (5) |
| H9 | 0.976882 | 0.273302 | 0.319904 | 0.048* |
| C10 | 0.67921 (17) | 0.43090 (18) | 0.15447 (14) | 0.0294 (4) |
| C11 | 0.55994 (19) | 0.4316 (2) | 0.12873 (16) | 0.0361 (4) |
| H11 | 0.546285 | 0.365865 | 0.099890 | 0.043* |
| C12 | 0.46085 (19) | 0.5266 (2) | 0.14451 (16) | 0.0415 (5) |
| H12 | 0.380019 | 0.525025 | 0.126874 | 0.050* |
| C13 | 0.4791 (2) | 0.6233 (2) | 0.18562 (16) | 0.0416 (5) |
| H13 | 0.411325 | 0.688460 | 0.196376 | 0.050* |
| C14 | 0.5969 (2) | 0.6242 (2) | 0.21100 (16) | 0.0403 (5) |
| H14 | 0.610168 | 0.690778 | 0.239220 | 0.048* |
| C15 | 0.69603 (19) | 0.52935 (19) | 0.19580 (15) | 0.0348 (4) |
| H15 | 0.776475 | 0.531510 | 0.213801 | 0.042* |
| C16 | 0.81390 (17) | 0.30119 (18) | 0.02966 (15) | 0.0302 (4) |
| C17 | 0.89389 (19) | 0.3951 (2) | -0.06919 (16) | 0.0395 (5) |
| H17 | 0.929517 | 0.474662 | -0.068264 | 0.047* |
| C18 | 0.9227 (2) | 0.3751 (2) | -0.16910 (17) | 0.0481 (5) |
| H18 | 0.977671 | 0.440845 | -0.235724 | 0.058* |

| | | | | |
|-----|--------------|---------------|---------------|------------|
| C19 | 0.8724 (2) | 0.2603 (2) | -0.17273 (17) | 0.0458 (5) |
| H19 | 0.894110 | 0.245561 | -0.241102 | 0.055* |
| C20 | 0.7903 (2) | 0.1675 (2) | -0.07596 (18) | 0.0454 (5) |
| H20 | 0.753497 | 0.089032 | -0.077590 | 0.054* |
| C21 | 0.7611 (2) | 0.1883 (2) | 0.02404 (16) | 0.0387 (5) |
| H21 | 0.703758 | 0.123685 | 0.090102 | 0.046* |
| C22 | 1.03391 (17) | 0.06948 (17) | 0.22632 (14) | 0.0288 (4) |
| C23 | 1.10054 (19) | 0.16811 (19) | 0.12395 (15) | 0.0365 (5) |
| H23 | 1.051484 | 0.211263 | 0.073556 | 0.044* |
| C24 | 1.2365 (2) | 0.2044 (2) | 0.09431 (17) | 0.0438 (5) |
| H24 | 1.279369 | 0.272864 | 0.024624 | 0.053* |
| C25 | 1.3101 (2) | 0.1413 (2) | 0.16568 (18) | 0.0446 (5) |
| H25 | 1.403699 | 0.165709 | 0.145408 | 0.054* |
| C26 | 1.2463 (2) | 0.0427 (2) | 0.26653 (17) | 0.0421 (5) |
| H26 | 1.296597 | -0.001657 | 0.315750 | 0.050* |
| C27 | 1.10940 (19) | 0.00747 (19) | 0.29711 (15) | 0.0341 (4) |
| H27 | 1.066750 | -0.060027 | 0.367444 | 0.041* |
| C28 | 0.82851 (17) | -0.09864 (18) | 0.35234 (15) | 0.0300 (4) |
| C29 | 0.80721 (19) | -0.10980 (19) | 0.46160 (15) | 0.0347 (4) |
| H29 | 0.833986 | -0.035807 | 0.475796 | 0.042* |
| C30 | 0.74772 (19) | -0.2268 (2) | 0.54981 (16) | 0.0388 (5) |
| H30 | 0.734851 | -0.232753 | 0.623661 | 0.047* |
| C31 | 0.7071 (2) | -0.3347 (2) | 0.53092 (17) | 0.0432 (5) |
| H31 | 0.664258 | -0.414051 | 0.591663 | 0.052* |
| C32 | 0.7288 (2) | -0.3271 (2) | 0.42349 (18) | 0.0452 (5) |
| H32 | 0.702693 | -0.401861 | 0.409890 | 0.054* |
| C33 | 0.78895 (19) | -0.20994 (19) | 0.33547 (16) | 0.0365 (4) |
| H33 | 0.803491 | -0.205565 | 0.261749 | 0.044* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|--------------|--------------|
| Si1 | 0.0258 (3) | 0.0328 (3) | 0.0277 (3) | 0.0011 (2) | -0.0077 (2) | -0.0073 (2) |
| C1 | 0.0296 (10) | 0.0442 (11) | 0.0373 (11) | -0.0004 (9) | -0.0088 (9) | -0.0059 (9) |
| C2 | 0.0258 (9) | 0.0315 (10) | 0.0295 (10) | -0.0010 (7) | -0.0094 (8) | -0.0086 (8) |
| C3 | 0.0280 (9) | 0.0321 (10) | 0.0258 (9) | 0.0014 (7) | -0.0088 (7) | -0.0092 (8) |
| C4 | 0.0377 (10) | 0.0305 (10) | 0.0282 (10) | 0.0086 (8) | -0.0126 (8) | -0.0050 (8) |
| C5 | 0.0459 (12) | 0.0458 (12) | 0.0319 (11) | 0.0053 (10) | -0.0075 (9) | -0.0111 (9) |
| C6 | 0.0677 (16) | 0.0552 (14) | 0.0333 (12) | 0.0104 (12) | -0.0060 (11) | -0.0173 (11) |
| C7 | 0.0861 (19) | 0.0466 (13) | 0.0412 (13) | 0.0236 (12) | -0.0339 (13) | -0.0222 (11) |
| C8 | 0.0592 (14) | 0.0445 (12) | 0.0609 (15) | 0.0170 (11) | -0.0375 (12) | -0.0249 (11) |
| C9 | 0.0383 (11) | 0.0410 (11) | 0.0459 (12) | 0.0115 (9) | -0.0176 (9) | -0.0196 (10) |
| C10 | 0.0282 (9) | 0.0312 (9) | 0.0241 (9) | -0.0005 (7) | -0.0076 (7) | -0.0048 (8) |
| C11 | 0.0328 (10) | 0.0414 (11) | 0.0353 (11) | 0.0007 (8) | -0.0139 (8) | -0.0122 (9) |
| C12 | 0.0290 (10) | 0.0557 (13) | 0.0368 (11) | 0.0079 (9) | -0.0134 (9) | -0.0120 (10) |
| C13 | 0.0374 (11) | 0.0489 (12) | 0.0340 (11) | 0.0151 (9) | -0.0098 (9) | -0.0141 (10) |
| C14 | 0.0447 (12) | 0.0412 (11) | 0.0372 (11) | 0.0068 (9) | -0.0121 (9) | -0.0184 (9) |
| C15 | 0.0340 (10) | 0.0409 (11) | 0.0305 (10) | 0.0027 (8) | -0.0136 (8) | -0.0113 (9) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C16 | 0.0256 (9) | 0.0351 (10) | 0.0304 (10) | 0.0053 (8) | -0.0119 (8) | -0.0106 (8) |
| C17 | 0.0360 (11) | 0.0473 (12) | 0.0336 (11) | -0.0033 (9) | -0.0106 (9) | -0.0121 (9) |
| C18 | 0.0414 (12) | 0.0656 (15) | 0.0312 (11) | -0.0012 (11) | -0.0091 (9) | -0.0118 (10) |
| C19 | 0.0427 (12) | 0.0668 (15) | 0.0368 (12) | 0.0137 (11) | -0.0168 (10) | -0.0274 (11) |
| C20 | 0.0543 (13) | 0.0442 (12) | 0.0526 (14) | 0.0126 (10) | -0.0287 (11) | -0.0263 (11) |
| C21 | 0.0429 (11) | 0.0366 (11) | 0.0365 (11) | 0.0002 (9) | -0.0163 (9) | -0.0094 (9) |
| C22 | 0.0272 (9) | 0.0292 (9) | 0.0303 (10) | 0.0055 (7) | -0.0085 (8) | -0.0126 (8) |
| C23 | 0.0324 (10) | 0.0384 (11) | 0.0302 (10) | 0.0056 (8) | -0.0082 (8) | -0.0052 (9) |
| C24 | 0.0336 (11) | 0.0433 (12) | 0.0379 (12) | -0.0025 (9) | -0.0027 (9) | -0.0028 (9) |
| C25 | 0.0310 (11) | 0.0460 (12) | 0.0513 (13) | -0.0058 (9) | -0.0122 (10) | -0.0101 (10) |
| C26 | 0.0371 (11) | 0.0444 (12) | 0.0457 (12) | 0.0000 (9) | -0.0211 (9) | -0.0096 (10) |
| C27 | 0.0350 (10) | 0.0329 (10) | 0.0317 (10) | -0.0004 (8) | -0.0112 (8) | -0.0075 (8) |
| C28 | 0.0239 (9) | 0.0294 (9) | 0.0328 (10) | 0.0044 (7) | -0.0077 (8) | -0.0086 (8) |
| C29 | 0.0339 (10) | 0.0343 (10) | 0.0336 (10) | 0.0046 (8) | -0.0097 (8) | -0.0112 (8) |
| C30 | 0.0337 (10) | 0.0417 (11) | 0.0306 (10) | 0.0069 (9) | -0.0050 (8) | -0.0069 (9) |
| C31 | 0.0373 (11) | 0.0367 (11) | 0.0401 (12) | -0.0019 (9) | -0.0065 (9) | -0.0007 (9) |
| C32 | 0.0477 (12) | 0.0345 (11) | 0.0498 (13) | -0.0036 (9) | -0.0161 (10) | -0.0090 (10) |
| C33 | 0.0354 (10) | 0.0367 (11) | 0.0337 (11) | 0.0015 (8) | -0.0090 (8) | -0.0104 (9) |

Geometric parameters (Å, °)

| | | | |
|---------|-------------|---------|-----------|
| Si1—C1 | 1.8669 (18) | C15—H15 | 0.9500 |
| Si1—C2 | 1.9099 (18) | C16—C17 | 1.388 (3) |
| Si1—C3 | 1.9138 (18) | C16—C21 | 1.387 (3) |
| Si1—C4 | 1.8744 (19) | C17—H17 | 0.9500 |
| C1—H1A | 0.9800 | C17—C18 | 1.384 (3) |
| C1—H1B | 0.9800 | C18—H18 | 0.9500 |
| C1—H1C | 0.9800 | C18—C19 | 1.380 (3) |
| C2—H2 | 1.0000 | C19—H19 | 0.9500 |
| C2—C10 | 1.526 (2) | C19—C20 | 1.376 (3) |
| C2—C16 | 1.524 (2) | C20—H20 | 0.9500 |
| C3—H3 | 1.0000 | C20—C21 | 1.388 (3) |
| C3—C22 | 1.527 (2) | C21—H21 | 0.9500 |
| C3—C28 | 1.520 (2) | C22—C23 | 1.395 (2) |
| C4—C5 | 1.393 (3) | C22—C27 | 1.388 (3) |
| C4—C9 | 1.396 (3) | C23—H23 | 0.9500 |
| C5—H5 | 0.9500 | C23—C24 | 1.382 (3) |
| C5—C6 | 1.376 (3) | C24—H24 | 0.9500 |
| C6—H6 | 0.9500 | C24—C25 | 1.381 (3) |
| C6—C7 | 1.369 (3) | C25—H25 | 0.9500 |
| C7—H7 | 0.9500 | C25—C26 | 1.376 (3) |
| C7—C8 | 1.377 (3) | C26—H26 | 0.9500 |
| C8—H8 | 0.9500 | C26—C27 | 1.388 (3) |
| C8—C9 | 1.389 (3) | C27—H27 | 0.9500 |
| C9—H9 | 0.9500 | C28—C29 | 1.394 (3) |
| C10—C11 | 1.394 (2) | C28—C33 | 1.390 (3) |
| C10—C15 | 1.392 (3) | C29—H29 | 0.9500 |
| C11—H11 | 0.9500 | C29—C30 | 1.384 (3) |

| | | | |
|------------|-------------|-------------|-------------|
| C11—C12 | 1.387 (3) | C30—H30 | 0.9500 |
| C12—H12 | 0.9500 | C30—C31 | 1.378 (3) |
| C12—C13 | 1.377 (3) | C31—H31 | 0.9500 |
| C13—H13 | 0.9500 | C31—C32 | 1.379 (3) |
| C13—C14 | 1.380 (3) | C32—H32 | 0.9500 |
| C14—H14 | 0.9500 | C32—C33 | 1.386 (3) |
| C14—C15 | 1.385 (3) | C33—H33 | 0.9500 |
| | | | |
| C1—Si1—C2 | 111.34 (8) | C10—C15—H15 | 119.6 |
| C1—Si1—C3 | 107.30 (9) | C14—C15—C10 | 120.79 (18) |
| C1—Si1—C4 | 109.22 (9) | C14—C15—H15 | 119.6 |
| C2—Si1—C3 | 111.98 (8) | C17—C16—C2 | 118.87 (16) |
| C4—Si1—C2 | 106.03 (8) | C21—C16—C2 | 123.72 (16) |
| C4—Si1—C3 | 110.98 (8) | C21—C16—C17 | 117.42 (17) |
| Si1—C1—H1A | 109.5 | C16—C17—H17 | 119.4 |
| Si1—C1—H1B | 109.5 | C18—C17—C16 | 121.26 (19) |
| Si1—C1—H1C | 109.5 | C18—C17—H17 | 119.4 |
| H1A—C1—H1B | 109.5 | C17—C18—H18 | 119.7 |
| H1A—C1—H1C | 109.5 | C19—C18—C17 | 120.5 (2) |
| H1B—C1—H1C | 109.5 | C19—C18—H18 | 119.7 |
| Si1—C2—H2 | 105.2 | C18—C19—H19 | 120.5 |
| C10—C2—Si1 | 109.56 (11) | C20—C19—C18 | 119.09 (19) |
| C10—C2—H2 | 105.2 | C20—C19—H19 | 120.5 |
| C16—C2—Si1 | 117.65 (12) | C19—C20—H20 | 119.9 |
| C16—C2—H2 | 105.2 | C19—C20—C21 | 120.20 (19) |
| C16—C2—C10 | 112.90 (14) | C21—C20—H20 | 119.9 |
| Si1—C3—H3 | 105.4 | C16—C21—C20 | 121.47 (18) |
| C22—C3—Si1 | 116.07 (12) | C16—C21—H21 | 119.3 |
| C22—C3—H3 | 105.4 | C20—C21—H21 | 119.3 |
| C28—C3—Si1 | 107.51 (11) | C23—C22—C3 | 119.13 (16) |
| C28—C3—H3 | 105.4 | C27—C22—C3 | 123.19 (16) |
| C28—C3—C22 | 116.09 (14) | C27—C22—C23 | 117.65 (16) |
| C5—C4—Si1 | 120.97 (15) | C22—C23—H23 | 119.3 |
| C5—C4—C9 | 116.66 (18) | C24—C23—C22 | 121.36 (18) |
| C9—C4—Si1 | 122.36 (15) | C24—C23—H23 | 119.3 |
| C4—C5—H5 | 119.0 | C23—C24—H24 | 119.9 |
| C6—C5—C4 | 122.0 (2) | C25—C24—C23 | 120.21 (18) |
| C6—C5—H5 | 119.0 | C25—C24—H24 | 119.9 |
| C5—C6—H6 | 119.9 | C24—C25—H25 | 120.4 |
| C7—C6—C5 | 120.3 (2) | C26—C25—C24 | 119.18 (18) |
| C7—C6—H6 | 119.9 | C26—C25—H25 | 120.4 |
| C6—C7—H7 | 120.2 | C25—C26—H26 | 119.6 |
| C6—C7—C8 | 119.7 (2) | C25—C26—C27 | 120.72 (19) |
| C8—C7—H7 | 120.2 | C27—C26—H26 | 119.6 |
| C7—C8—H8 | 120.0 | C22—C27—H27 | 119.6 |
| C7—C8—C9 | 120.0 (2) | C26—C27—C22 | 120.87 (17) |
| C9—C8—H8 | 120.0 | C26—C27—H27 | 119.6 |
| C4—C9—H9 | 119.3 | C29—C28—C3 | 122.69 (16) |

| | | | |
|-------------|-------------|-------------|-------------|
| C8—C9—C4 | 121.4 (2) | C33—C28—C3 | 119.78 (16) |
| C8—C9—H9 | 119.3 | C33—C28—C29 | 117.43 (17) |
| C11—C10—C2 | 121.61 (16) | C28—C29—H29 | 119.4 |
| C15—C10—C2 | 120.62 (16) | C30—C29—C28 | 121.16 (18) |
| C15—C10—C11 | 117.78 (17) | C30—C29—H29 | 119.4 |
| C10—C11—H11 | 119.4 | C29—C30—H30 | 119.9 |
| C12—C11—C10 | 121.14 (18) | C31—C30—C29 | 120.25 (19) |
| C12—C11—H11 | 119.4 | C31—C30—H30 | 119.9 |
| C11—C12—H12 | 119.8 | C30—C31—H31 | 120.1 |
| C13—C12—C11 | 120.32 (18) | C30—C31—C32 | 119.74 (18) |
| C13—C12—H12 | 119.8 | C32—C31—H31 | 120.1 |
| C12—C13—H13 | 120.4 | C31—C32—H32 | 120.1 |
| C12—C13—C14 | 119.21 (18) | C31—C32—C33 | 119.76 (19) |
| C14—C13—H13 | 120.4 | C33—C32—H32 | 120.1 |
| C13—C14—H14 | 119.6 | C28—C33—H33 | 119.2 |
| C13—C14—C15 | 120.76 (19) | C32—C33—C28 | 121.64 (19) |
| C15—C14—H14 | 119.6 | C32—C33—H33 | 119.2 |

Hydrogen-bond geometry (\AA , $^\circ$)

Cg2 and Cg4 are the centroids of the C10–C15 and C22–C27 rings, respectively.

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|------------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C20—H20 \cdots Cg4 ⁱ | 0.95 | 2.94 | 3.716 (2) | 140 |
| C24—H24 \cdots Cg2 ⁱⁱ | 0.95 | 2.75 | 3.696 (2) | 175 |

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

2-Methyl-1,1,3,3-tetraphenyl-2-silapropan-2-ol (II)*Crystal data*

$\text{C}_{27}\text{H}_{26}\text{OSi}$

$M_r = 394.57$

Monoclinic, $P2_1/c$

$a = 11.8576$ (5) \AA

$b = 13.2995$ (6) \AA

$c = 14.3948$ (6) \AA

$\beta = 110.363$ (3) $^\circ$

$V = 2128.20$ (16) \AA^3

$Z = 4$

$F(000) = 840$

$D_x = 1.231$ Mg m^{-3}

Cu $K\alpha$ radiation, $\lambda = 1.54178$ \AA

Cell parameters from 1512 reflections

$\theta = 4.0\text{--}71.6^\circ$

$\mu = 1.08$ mm^{-1}

$T = 173$ K

Block, colourless

$0.10 \times 0.09 \times 0.08$ mm

Data collection

Bruker APEXII CCD
diffractometer

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2013)

$T_{\min} = 0.624$, $T_{\max} = 0.754$

11901 measured reflections

4113 independent reflections

2414 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.096$

$\theta_{\max} = 72.1^\circ$, $\theta_{\min} = 4.0^\circ$

$h = -14 \rightarrow 14$

$k = -14 \rightarrow 16$

$l = -17 \rightarrow 16$

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.062$ $wR(F^2) = 0.171$ $S = 0.97$

4113 reflections

267 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0793P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.29 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.35 \text{ e } \text{\AA}^{-3}$ *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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|-------------|----------------------------------|
| Si1 | 0.37978 (7) | 0.60730 (7) | 0.11066 (6) | 0.0322 (2) |
| O1 | 0.4586 (2) | 0.5078 (2) | 0.0982 (2) | 0.0478 (7) |
| H1 | 0.525 (6) | 0.511 (5) | 0.125 (5) | 0.14 (3)* |
| C1 | 0.4582 (3) | 0.7247 (3) | 0.1046 (3) | 0.0496 (10) |
| H1A | 0.538510 | 0.722512 | 0.152061 | 0.074* |
| H1B | 0.415753 | 0.780473 | 0.119230 | 0.074* |
| H1C | 0.461428 | 0.732630 | 0.039250 | 0.074* |
| C2 | 0.2251 (3) | 0.5965 (3) | 0.0102 (2) | 0.0311 (7) |
| H2 | 0.186321 | 0.541154 | 0.032661 | 0.037* |
| C3 | 0.3493 (3) | 0.6018 (3) | 0.2320 (2) | 0.0305 (7) |
| H3 | 0.304983 | 0.663543 | 0.233789 | 0.037* |
| C4 | 0.2310 (3) | 0.5599 (3) | -0.0879 (2) | 0.0314 (7) |
| C5 | 0.2221 (3) | 0.4579 (3) | -0.1059 (3) | 0.0465 (9) |
| H5 | 0.212646 | 0.415105 | -0.058001 | 0.056* |
| C6 | 0.2269 (4) | 0.4173 (3) | -0.1928 (3) | 0.0597 (12) |
| H6 | 0.220922 | 0.348044 | -0.202343 | 0.072* |
| C7 | 0.2403 (4) | 0.4783 (4) | -0.2651 (3) | 0.0594 (12) |
| H7 | 0.242726 | 0.451111 | -0.323899 | 0.071* |
| C8 | 0.2500 (4) | 0.5796 (4) | -0.2490 (3) | 0.0558 (11) |
| H8 | 0.259536 | 0.621704 | -0.297355 | 0.067* |
| C9 | 0.2457 (3) | 0.6210 (3) | -0.1611 (3) | 0.0446 (9) |
| H9 | 0.252840 | 0.690203 | -0.151519 | 0.054* |
| C10 | 0.1461 (3) | 0.6868 (3) | 0.0079 (2) | 0.0318 (7) |
| C11 | 0.1675 (3) | 0.7822 (3) | -0.0220 (3) | 0.0402 (8) |
| H11 | 0.233780 | 0.792719 | -0.041198 | 0.048* |
| C12 | 0.0917 (3) | 0.8616 (3) | -0.0236 (3) | 0.0478 (9) |
| H12 | 0.106084 | 0.924134 | -0.046117 | 0.057* |
| C13 | -0.0053 (3) | 0.8494 (3) | 0.0078 (3) | 0.0495 (10) |

| | | | | |
|-----|-------------|------------|------------|-------------|
| H13 | -0.055857 | 0.903091 | 0.006978 | 0.059* |
| C14 | -0.0253 (3) | 0.7557 (3) | 0.0404 (3) | 0.0463 (9) |
| H14 | -0.089227 | 0.746580 | 0.062864 | 0.056* |
| C15 | 0.0485 (3) | 0.6755 (3) | 0.0402 (2) | 0.0379 (8) |
| H15 | 0.032870 | 0.612895 | 0.061775 | 0.046* |
| C16 | 0.4654 (3) | 0.6075 (3) | 0.3226 (2) | 0.0330 (7) |
| C17 | 0.5172 (3) | 0.7002 (3) | 0.3555 (3) | 0.0417 (9) |
| H17 | 0.482228 | 0.758048 | 0.321262 | 0.050* |
| C18 | 0.6206 (3) | 0.7080 (3) | 0.4391 (3) | 0.0517 (10) |
| H18 | 0.655291 | 0.770730 | 0.459002 | 0.062* |
| C19 | 0.6718 (3) | 0.6238 (3) | 0.4923 (3) | 0.0483 (10) |
| H19 | 0.739662 | 0.629258 | 0.549267 | 0.058* |
| C20 | 0.6213 (3) | 0.5311 (3) | 0.4603 (3) | 0.0485 (10) |
| H20 | 0.656040 | 0.473577 | 0.495283 | 0.058* |
| C21 | 0.5185 (3) | 0.5229 (3) | 0.3757 (3) | 0.0402 (8) |
| H21 | 0.485335 | 0.459927 | 0.354838 | 0.048* |
| C22 | 0.2661 (3) | 0.5165 (3) | 0.2345 (2) | 0.0330 (7) |
| C23 | 0.1654 (3) | 0.5350 (3) | 0.2610 (2) | 0.0401 (8) |
| H23 | 0.151976 | 0.599365 | 0.280198 | 0.048* |
| C24 | 0.0848 (3) | 0.4581 (3) | 0.2589 (3) | 0.0503 (10) |
| H24 | 0.018734 | 0.471259 | 0.277708 | 0.060* |
| C25 | 0.1022 (3) | 0.3627 (3) | 0.2294 (3) | 0.0507 (11) |
| H25 | 0.047234 | 0.311931 | 0.226694 | 0.061* |
| C26 | 0.2021 (4) | 0.3430 (3) | 0.2038 (3) | 0.0459 (9) |
| H26 | 0.214929 | 0.278564 | 0.184382 | 0.055* |
| C27 | 0.2826 (3) | 0.4188 (3) | 0.2069 (2) | 0.0401 (8) |
| H27 | 0.349762 | 0.404301 | 0.190084 | 0.048* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| Si1 | 0.0319 (4) | 0.0348 (5) | 0.0275 (4) | 0.0005 (4) | 0.0074 (3) | 0.0024 (4) |
| O1 | 0.0406 (14) | 0.0544 (18) | 0.0440 (15) | 0.0134 (13) | 0.0092 (12) | -0.0039 (13) |
| C1 | 0.056 (2) | 0.052 (3) | 0.0361 (19) | -0.0172 (19) | 0.0088 (17) | 0.0081 (19) |
| C2 | 0.0329 (15) | 0.0312 (18) | 0.0273 (15) | -0.0035 (14) | 0.0080 (12) | 0.0031 (15) |
| C3 | 0.0369 (15) | 0.0296 (17) | 0.0250 (15) | 0.0011 (14) | 0.0109 (13) | -0.0004 (14) |
| C4 | 0.0295 (14) | 0.0357 (19) | 0.0259 (15) | 0.0000 (14) | 0.0057 (12) | -0.0012 (15) |
| C5 | 0.057 (2) | 0.038 (2) | 0.044 (2) | 0.0052 (18) | 0.0170 (18) | -0.0050 (18) |
| C6 | 0.064 (3) | 0.048 (3) | 0.065 (3) | 0.000 (2) | 0.020 (2) | -0.020 (2) |
| C7 | 0.051 (2) | 0.075 (3) | 0.052 (2) | -0.009 (2) | 0.018 (2) | -0.030 (3) |
| C8 | 0.061 (2) | 0.074 (3) | 0.036 (2) | -0.016 (2) | 0.0224 (18) | -0.008 (2) |
| C9 | 0.056 (2) | 0.043 (2) | 0.0346 (18) | -0.0080 (18) | 0.0161 (16) | -0.0049 (18) |
| C10 | 0.0341 (15) | 0.0340 (19) | 0.0224 (15) | 0.0024 (14) | 0.0035 (12) | 0.0004 (14) |
| C11 | 0.0462 (18) | 0.034 (2) | 0.043 (2) | 0.0005 (16) | 0.0191 (16) | 0.0010 (17) |
| C12 | 0.057 (2) | 0.033 (2) | 0.053 (2) | 0.0039 (17) | 0.0201 (19) | 0.0022 (19) |
| C13 | 0.053 (2) | 0.040 (2) | 0.052 (2) | 0.0144 (18) | 0.0137 (19) | -0.0072 (19) |
| C14 | 0.0429 (19) | 0.051 (3) | 0.047 (2) | 0.0037 (17) | 0.0176 (17) | -0.0020 (19) |
| C15 | 0.0401 (17) | 0.036 (2) | 0.0361 (18) | 0.0022 (15) | 0.0110 (14) | 0.0023 (16) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C16 | 0.0344 (15) | 0.042 (2) | 0.0239 (14) | 0.0003 (15) | 0.0117 (12) | 0.0022 (15) |
| C17 | 0.0471 (19) | 0.039 (2) | 0.0362 (18) | -0.0033 (16) | 0.0111 (15) | -0.0015 (17) |
| C18 | 0.052 (2) | 0.060 (3) | 0.038 (2) | -0.018 (2) | 0.0100 (17) | -0.009 (2) |
| C19 | 0.0381 (17) | 0.073 (3) | 0.0287 (17) | -0.0061 (19) | 0.0054 (14) | 0.001 (2) |
| C20 | 0.0407 (18) | 0.063 (3) | 0.0373 (19) | 0.0030 (19) | 0.0075 (16) | 0.012 (2) |
| C21 | 0.0386 (17) | 0.043 (2) | 0.0353 (18) | -0.0004 (16) | 0.0085 (15) | 0.0046 (17) |
| C22 | 0.0366 (16) | 0.0341 (19) | 0.0239 (15) | -0.0030 (14) | 0.0051 (13) | 0.0058 (15) |
| C23 | 0.0424 (18) | 0.045 (2) | 0.0290 (17) | 0.0017 (16) | 0.0072 (14) | 0.0034 (16) |
| C24 | 0.0377 (18) | 0.070 (3) | 0.039 (2) | -0.0073 (19) | 0.0075 (16) | 0.009 (2) |
| C25 | 0.052 (2) | 0.052 (3) | 0.0356 (19) | -0.0222 (19) | -0.0008 (17) | 0.0107 (18) |
| C26 | 0.063 (2) | 0.033 (2) | 0.0337 (18) | -0.0076 (18) | 0.0068 (17) | 0.0041 (17) |
| C27 | 0.0480 (19) | 0.037 (2) | 0.0341 (18) | 0.0006 (16) | 0.0128 (15) | 0.0073 (16) |

Geometric parameters (Å, °)

| | | | |
|-----------|-------------|-------------|-----------|
| Si1—O1 | 1.666 (3) | C12—H12 | 0.9300 |
| Si1—C1 | 1.835 (4) | C12—C13 | 1.384 (5) |
| Si1—C2 | 1.905 (3) | C13—H13 | 0.9300 |
| Si1—C3 | 1.904 (3) | C13—C14 | 1.380 (5) |
| O1—H1 | 0.75 (6) | C14—H14 | 0.9300 |
| C1—H1A | 0.9600 | C14—C15 | 1.380 (5) |
| C1—H1B | 0.9600 | C15—H15 | 0.9300 |
| C1—H1C | 0.9600 | C16—C17 | 1.385 (5) |
| C2—H2 | 0.9800 | C16—C21 | 1.383 (5) |
| C2—C4 | 1.518 (4) | C17—H17 | 0.9300 |
| C2—C10 | 1.517 (4) | C17—C18 | 1.391 (5) |
| C3—H3 | 0.9800 | C18—H18 | 0.9300 |
| C3—C16 | 1.533 (4) | C18—C19 | 1.374 (5) |
| C3—C22 | 1.513 (4) | C19—H19 | 0.9300 |
| C4—C5 | 1.378 (5) | C19—C20 | 1.378 (6) |
| C4—C9 | 1.389 (5) | C20—H20 | 0.9300 |
| C5—H5 | 0.9300 | C20—C21 | 1.396 (5) |
| C5—C6 | 1.382 (5) | C21—H21 | 0.9300 |
| C6—H6 | 0.9300 | C22—C23 | 1.396 (5) |
| C6—C7 | 1.372 (6) | C22—C27 | 1.392 (5) |
| C7—H7 | 0.9300 | C23—H23 | 0.9300 |
| C7—C8 | 1.366 (6) | C23—C24 | 1.394 (5) |
| C8—H8 | 0.9300 | C24—H24 | 0.9300 |
| C8—C9 | 1.397 (5) | C24—C25 | 1.376 (6) |
| C9—H9 | 0.9300 | C25—H25 | 0.9300 |
| C10—C11 | 1.391 (5) | C25—C26 | 1.383 (6) |
| C10—C15 | 1.397 (4) | C26—H26 | 0.9300 |
| C11—H11 | 0.9300 | C26—C27 | 1.378 (5) |
| C11—C12 | 1.381 (5) | C27—H27 | 0.9300 |
| O1—Si1—C1 | 111.00 (17) | C11—C12—H12 | 119.5 |
| O1—Si1—C2 | 106.65 (15) | C11—C12—C13 | 121.0 (4) |
| O1—Si1—C3 | 111.15 (15) | C13—C12—H12 | 119.5 |

| | | | |
|-------------|-------------|-------------|-----------|
| C1—Si1—C2 | 113.46 (16) | C12—C13—H13 | 120.7 |
| C1—Si1—C3 | 109.71 (16) | C14—C13—C12 | 118.5 (4) |
| C3—Si1—C2 | 104.69 (14) | C14—C13—H13 | 120.7 |
| Si1—O1—H1 | 116 (5) | C13—C14—H14 | 119.6 |
| Si1—C1—H1A | 109.5 | C15—C14—C13 | 120.8 (4) |
| Si1—C1—H1B | 109.5 | C15—C14—H14 | 119.6 |
| Si1—C1—H1C | 109.5 | C10—C15—H15 | 119.4 |
| H1A—C1—H1B | 109.5 | C14—C15—C10 | 121.1 (3) |
| H1A—C1—H1C | 109.5 | C14—C15—H15 | 119.4 |
| H1B—C1—H1C | 109.5 | C17—C16—C3 | 119.7 (3) |
| Si1—C2—H2 | 104.2 | C21—C16—C3 | 122.1 (3) |
| C4—C2—Si1 | 112.7 (2) | C21—C16—C17 | 118.2 (3) |
| C4—C2—H2 | 104.2 | C16—C17—H17 | 119.5 |
| C10—C2—Si1 | 112.4 (2) | C16—C17—C18 | 121.0 (4) |
| C10—C2—H2 | 104.2 | C18—C17—H17 | 119.5 |
| C10—C2—C4 | 117.5 (3) | C17—C18—H18 | 119.8 |
| Si1—C3—H3 | 105.5 | C19—C18—C17 | 120.5 (4) |
| C16—C3—Si1 | 112.2 (2) | C19—C18—H18 | 119.8 |
| C16—C3—H3 | 105.5 | C18—C19—H19 | 120.5 |
| C22—C3—Si1 | 112.6 (2) | C18—C19—C20 | 119.1 (3) |
| C22—C3—H3 | 105.5 | C20—C19—H19 | 120.5 |
| C22—C3—C16 | 114.6 (3) | C19—C20—H20 | 119.7 |
| C5—C4—C2 | 117.7 (3) | C19—C20—C21 | 120.5 (4) |
| C5—C4—C9 | 117.1 (3) | C21—C20—H20 | 119.7 |
| C9—C4—C2 | 125.2 (3) | C16—C21—C20 | 120.7 (4) |
| C4—C5—H5 | 119.0 | C16—C21—H21 | 119.7 |
| C4—C5—C6 | 122.0 (4) | C20—C21—H21 | 119.7 |
| C6—C5—H5 | 119.0 | C23—C22—C3 | 120.1 (3) |
| C5—C6—H6 | 119.7 | C27—C22—C3 | 122.4 (3) |
| C7—C6—C5 | 120.5 (4) | C27—C22—C23 | 117.5 (3) |
| C7—C6—H6 | 119.7 | C22—C23—H23 | 119.7 |
| C6—C7—H7 | 120.6 | C24—C23—C22 | 120.7 (4) |
| C8—C7—C6 | 118.7 (4) | C24—C23—H23 | 119.7 |
| C8—C7—H7 | 120.6 | C23—C24—H24 | 119.7 |
| C7—C8—H8 | 119.5 | C25—C24—C23 | 120.5 (4) |
| C7—C8—C9 | 121.0 (4) | C25—C24—H24 | 119.7 |
| C9—C8—H8 | 119.5 | C24—C25—H25 | 120.3 |
| C4—C9—C8 | 120.7 (4) | C24—C25—C26 | 119.5 (4) |
| C4—C9—H9 | 119.7 | C26—C25—H25 | 120.3 |
| C8—C9—H9 | 119.7 | C25—C26—H26 | 120.0 |
| C11—C10—C2 | 123.5 (3) | C27—C26—C25 | 120.0 (4) |
| C11—C10—C15 | 117.5 (3) | C27—C26—H26 | 120.0 |
| C15—C10—C2 | 119.0 (3) | C22—C27—H27 | 119.1 |
| C10—C11—H11 | 119.5 | C26—C27—C22 | 121.9 (4) |
| C12—C11—C10 | 121.0 (3) | C26—C27—H27 | 119.1 |
| C12—C11—H11 | 119.5 | | |

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the C4–C9 ring.

| <i>D</i> —H··· <i>A</i> | <i>D</i> —H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> —H··· <i>A</i> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| C27—H27···O1 | 0.93 | 2.55 | 3.237 (4) | 131 |
| O1—H1···Cg1 ⁱ | 0.75 (8) | 2.70 (7) | 3.416 (3) | 162 (7) |
| C24—H24···Cg1 ⁱⁱ | 0.93 | 2.86 | 3.582 (4) | 135 |

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x, -y+1, -z$.