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Tetrameric triphenylsilanol, $(Ph_3SiOH)_4$, and the adduct $(Ph_3SiOH)_2$–dimethyl sulfoxide, both at 120 K, and the adduct $(Ph_3SiOH)_4$–1,4-dioxan at 150 K: interplay of O—H···O and C—H···π(arene) interactions

Katharine F. Bowes,[a] Christopher Glidewell[a]* and John N. Low[b]†

[a]School of Chemistry, University of St Andrews, St Andrews, Fife KY16 9ST, Scotland, and [b]Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland
Correspondence e-mail: cg@st-andrews.ac.uk

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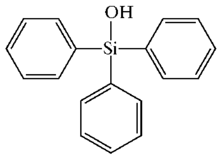
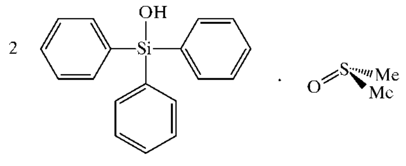
The structure of tetrameric triphenylsilanol, $C_{18}H_{16}OSi$, (I), has been re-investigated at 120 (2) K. The hydroxyl H atoms were readily located and one of the arene rings is disordered over two closely positioned sets of sites. The molecules are linked into cyclic tetramers, having approximate $\overline{4}$ (S_4) symmetry, *via* O—H···O hydrogen bonds [H···O 1.81–1.85 Å, O···O 2.634 (3)–2.693 (3) Å and O—H···O 156–166°]. At ambient temperature, there are indications of multiple disorder of the phenyl-ring sites. In bis(triphenylsilanol) dimethyl sulfoxide solvate, $2C_{18}H_{16}OSi{\cdot}C_2H_6OS$, (II), the dimethyl sulfoxide component is disordered across a twofold rotation axis in $C2/c$, and the molecular components are linked by a single O—H···O hydrogen bond [H···O 1.85 Å, O···O 2.732 (2) Å and O—H···O 172°] into three-molecule aggregates, which are themselves linked into a single three-dimensional framework by two C—H···π(arene) interactions. In tetrakis(triphenylsilanol) 1,4-dioxan solvate, $4C_{18}H_{16}OSi{\cdot}C_4H_8O_2$, (III), the 1,4-dioxan component lies across an inversion centre in space group $P\overline{1}$ and centrosymmetric five-molecule aggregates are linked by paired C—H···π(arene) interactions to form molecular ladders.

Comment

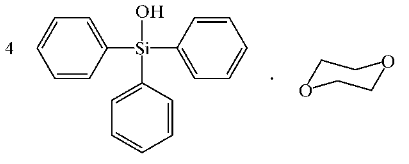
We recently reported the structure analysis at 120 (2) K of *catena*-poly[[triphenyltin(IV)]-μ-hydroxo-$\kappa^2O{:}O$], $(Ph_3SnOH)_n$, and, by use of a low-temperature data set collected on a CCD diffractometer, the location of the hydroxyl H atom was readily achieved (Glidewell *et al.*, 2002). By contrast, use of an ambient-temperature data set, collected using a four-circle diffractometer, did not allow location of the hydroxyl H atom (Glidewell & Liles, 1978). We also noted (Glidewell *et al.*, 2002) that the use of low-temperature CCD data sets should render possible the location of the hydroxyl H atoms in other Ph_3MOH compounds, in particular, those in Ph_3SiOH, (I). This compound crystallizes in space group $P\overline{1}$ with $Z = 16$, *i.e.* with $Z' = 8$ (Puff *et al.*, 1991), and the molecules are arranged to form two independent cyclic tetramers, each having approximate $\overline{4}$ (S_4) symmetry. The short intermolecular O···O distances indicate the presence of O—H···O hydrogen bonds linking the molecules, but, using data collected at 198 K on a four-circle diffractometer, it was not possible to locate any of the eight hydroxyl H atoms (Puff *et al.*, 1991). Furthermore, scrutiny of the original structure report reveals anomalously high U_{eq} values for several of the C atoms, two of which, moreover, were only refined isotropically. These facts, and the rather high residual densities, suggest some difficulties with the refinement model. This may, in turn, account for the rather high R value of 0.079 for only 12 146 reflections, with 9079 labelled 'observed' and used in the refinement, giving a reflections:parameters (n/p) ratio of only 6.35. The overall precision of the structure determination is thus rather modest [σ(Si—O) 0.005 Å, σ(Si—C) 0.007 Å and σ(C—C) 0.007–0.026 Å (mean 0.015 Å)].



(I)



(II)



(III)

We have therefore re-investigated (I), both at 120 (2) K and at ambient temperature. At both temperatures, the unit-cell dimensions, the space group and the atomic coordinates indicate the same phase as originally investigated by Puff *et al.* (1991). The same phase was obtained by crystallization from a wide range of solvents, including toluene (as used in the original study); a crystal grown from chloroform solution gave marginally the best data set, and it is the refinement based on these data that is reported here. Data collection at 120 (2) K resulted in a much larger data set than previously employed, with an n/p ratio of 17.2. The hydroxyl H atoms were readily located from difference maps, but it was necessary to model one of the phenyl rings using two sets of sites, each with 50% occupancy. The overall supramolecular structure matches that reported earlier, based solely on O—H···O hydrogen bonds; there are neither C—H···π(arene) interactions nor aromatic π–π-stacking interactions in the crystal structure of (I).

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† Postal address: School of Engineering, University of Dundee, Dundee DD1 4HN, Scotland.

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The principal structural features of (I) (Fig. 1), in which the C atoms are labelled as C*pqr*, where the index *p* (= 1 to 8) defines the molecule, *q* (= 1 to 3) defines the ring within a specified molecule, and *r* (= 1 to 6) defines the atom within a specified ring, are identical to those reported earlier, but the key structural parameters (Table 1) are more precise [σ(Si—O) 0.002 Å, σ(Si—C) 0.003 Å and σ(C—C) 0.004–0.005 Å]. The hydrogen bonds (Table 2), in which all of the hydroxyl H are fully ordered at 120 (2) K, are characterized by O···O distances which are short for simple neutral *R*OH species, and by O—H···O angles which are close to 160°. For comparison, the O···O distances in the tetrahedral tetramer $(Ph_3COH)_4$ are 2.859 (5) and 2.854 (5) Å at 113 K, and 2.905 (4) and 2.901 (4) Å at 293 K (Serrano-González *et al.*, 1999). The hydroxyl H atoms in $(Ph_3COH)_4$ could not be located from ambient-temperature X-ray data (Ferguson *et al.*, 1992); these H atoms are, in fact, mobile over a number of sites at ambient temperature (Aliev *et al.*, 1998), and neutron diffraction at 100 K was required to locate these H sites unambiguously (Serrano-González *et al.*, 1999).

Using data collected at 298 (2) K from a crystal grown from toluene solution, so mimicking the original crystal preparation, we then investigated the ambient-temperature structure of (I). The overall tetrameric aggregation is unchanged and there were reasonably good indications, from difference maps, for at least some of the hydroxyl H atoms. However, not only was the diffraction intensity rather poor, with only 27.6% of the reflections labelled 'observed' compared with over 60% at 120 (2) K, but there were indications of possible disorder in no fewer than eight of the 24 independent phenyl rings. Accordingly, refinement was not pursued. After completing this study, we learned of a recent unpublished determination at 123 K (Nieger, 2001), deposited in the Cambridge Structural Database (CSD; Allen & Kennard, 1993) as a private communication (CSD reference code JIPTIL01). The findings of this study appear to be identical with those reported here,

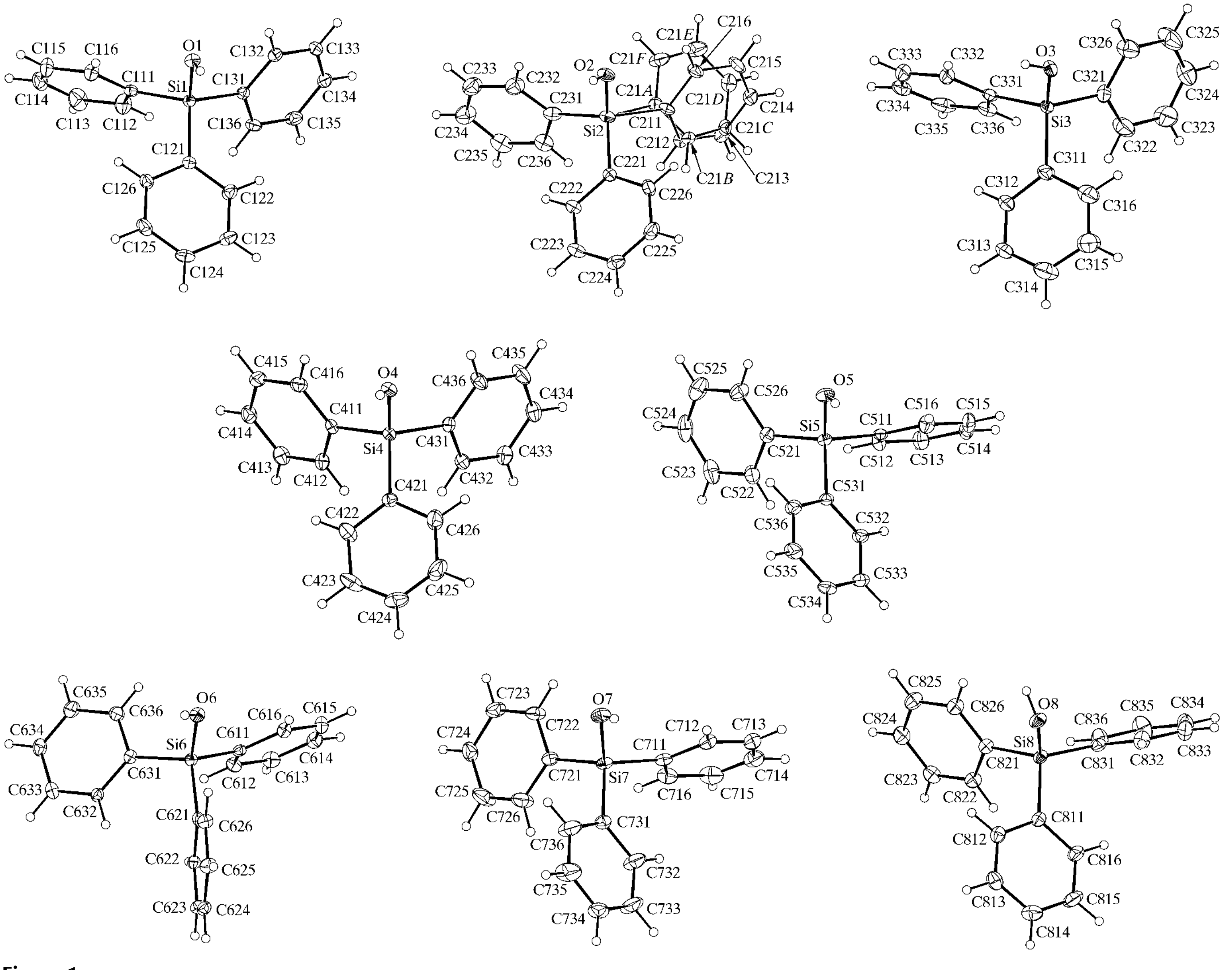

Figure 1

Views of the eight independent molecules in (I), showing the atom-labelling schemes. Displacement ellipsoids are drawn at the 30% probability level in all cases and H atoms are shown as small spheres of arbitrary radii.

although it is perhaps unfortunate that there are no anisotropic displacement parameters stored in the CSD files.

Crystallization of triphenylsilanol from dimethyl sulfoxide (DMSO), on the other hand, leads to a 2:1 solvate, $(Ph_3SiOH)_2 \cdot DMSO$, (II) (Fig. 3). In (II), the silanol molecule lies in a general position in space group *C*2/*c*, and the DMSO molecule is disordered across a twofold rotation axis, such that it could be satisfactorily modelled using a single oxygen site on the twofold axis, a single carbon site in a general position and two distinct sulfur sites of 0.25 occupancy, corresponding to two confacial pyramidal orientations having common oxygen and carbon sites on a shared face. Within the silanol molecule in (II), the Si—O distance (Table 3) is slightly shorter than those in (I), but the Si—C distances in (I) and (II) span comparable ranges, with virtually identical mean values.

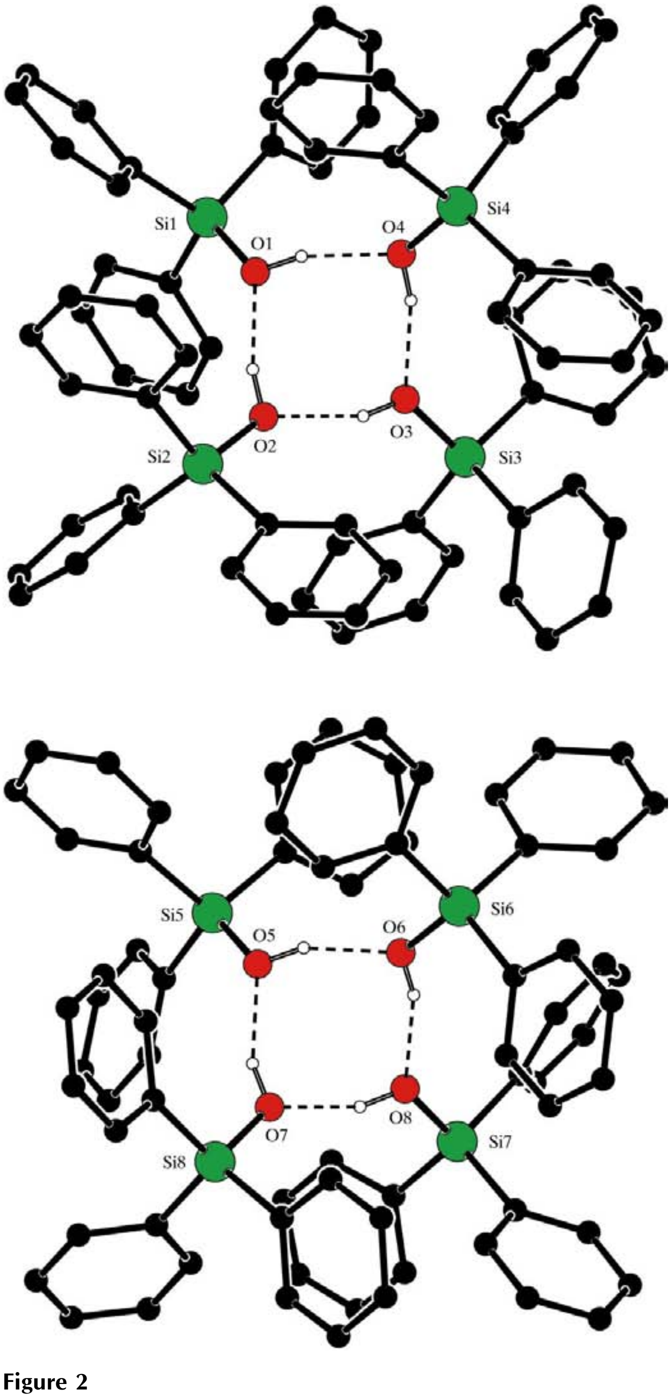

Figure 2
The two independent cyclic tetramers of (I). For the sake of clarity, only one of the disordered rings in molecule 2 is shown.

Whereas the intermolecular aggregation in (I) produces finite oligomers, compound (II) forms a continuous framework. Silanol atom O1 acts as a hydrogen-bond donor to DMSO atom O2 (Table 4), so generating a three-molecule aggregate lying across a rotation axis (Fig. 4). There are four of these aggregates in each unit cell and they are linked into a single three-dimensional framework by means of two distinct C—H$\cdots\pi$(arene) hydrogen bonds (Table 4). Atom C4 in the silanol molecule at (*x*, *y*, z) acts as a hydrogen-bond donor to the centroid, *Cg*3, of the C31–C36 ring of the silanol molecule at $(x - \frac{1}{2}, y - \frac{1}{2}, z)$, so generating by translation a chain running parallel to the [110] direction (Fig. 5), while the action of a twofold rotation axis generates a similar chain parallel to $[1\bar{1}0]$. These two chains are linked *via* O—H$\cdots$O hydrogen

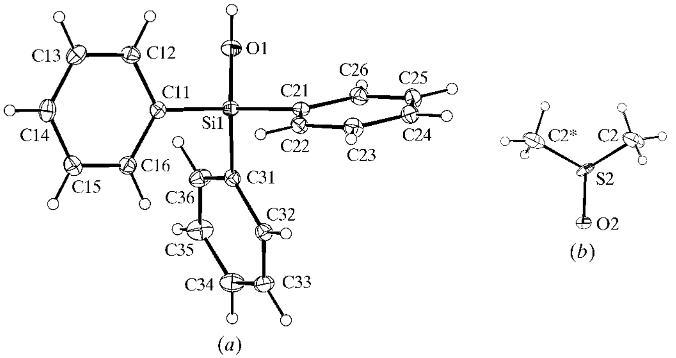

Figure 3
The molecular components of (II), showing the atom-labelling scheme; (*a*) the silanol component and (*b*) the disordered DMSO, where the atom marked with an asterisk (*) is at the symmetry position $(-x, y, \frac{1}{2} - z)$. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

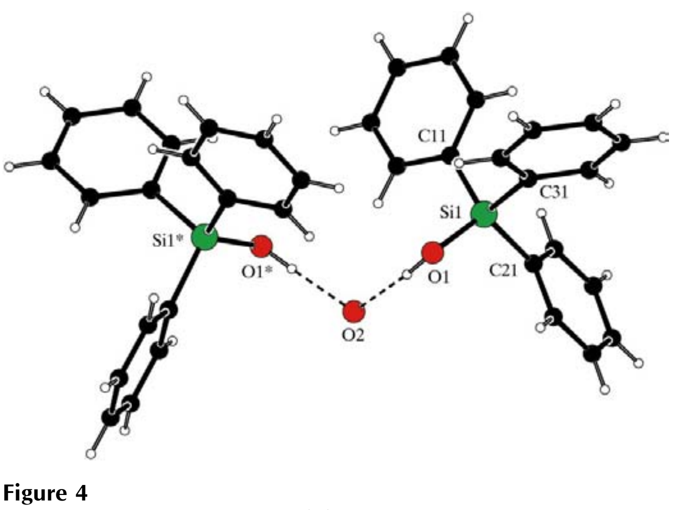

Figure 4
Part of the crystal structure of (II), showing the formation of a three-molecule aggregate by means of O—H$\cdots$O hydrogen bonds. For the sake of clarity, only the O atom of the DMSO molecule is shown. Atoms marked with an asterisk (*) are at the symmetry position $(-x, y, \frac{1}{2} - z)$.

bonds to the DMSO, and propagation of the two interactions generates an (001) sheet lying in the domain $0 < z < \frac{1}{2}$. Adjacent sheets are linked by a second C—H$\cdots\pi$(arene) interaction.

Atom C23 in the silanol molecule at (x, y, z) lies in the $0 < z < \frac{1}{2}$ sheet. This atom acts as a hydrogen-bond donor to the centroid, *Cg*1, of the C11–C16 ring in the silanol molecule at ($-x$, $1 - y$, $1 - z$) (Fig. 6), which is part of the sheet in the domain $\frac{1}{2} < z < 1$. The action of the twofold axes similarly links the $0 < z < \frac{1}{2}$ sheet to its neighbour in the domain $-\frac{1}{2} < z < 0$, and hence all the sheets are linked into a single framework by the action of this centrosymmetric motif.

Crystallization of triphenylsilanol from 1,4-dioxan yields a 4:1 adduct, $(Ph_3SiOH)_4{\cdot}C_4H_8O_2$, (III). The structure of this adduct has been reported using data collected at 293 K (Bourne, Johnson *et al.*, 1991), and the supramolecular structure was discussed in terms of a finite centrosymmetric five-molecule aggregate. We have now re-investigated this compound at 150 (2) K, using a rather larger data set (6905 reflections, as opposed to 4435). The same phase clearly exists at the two temperatures, but careful examination of the structure refined from the 150 K data shows that the centrosymmetric aggregates (Fig. 7) are in fact linked by two C—H$\cdots\pi$(arene) hydrogen bonds (Table 6), which co-operate to form molecular ladders along the [100] direction. Atoms C134 and C214 in the two silanol molecules at (x, y, z) act as hydrogen-bond donors to, respectively, the rings C111–C116 and C121–C126 in the type 1 silanol molecule at ($x - 1$, y, z) (Table 6 and Fig. 8). The Si—O distances in (III) (Table 5) are intermediate between those in (I) and (II), while the Si—C distances are not significantly different from those in (I) and (II).

It is striking that crystallization of triphenylsilanol yields specific stoichiometric solvates when crystallized from DMSO or dioxan, but, when crystallized from acetone, which is expected to be at least as good an acceptor of hydrogen bonds as dioxan, the solvent-free phase (I) is formed. Likewise, it has been reported (Bourne, Nassimbeni *et al.*, 1991) that crystallization of triphenylsilanol from mixtures of ethanol with any of methanol, propanol or water selectively yields a 4:1 ethanol solvate, $(Ph_3SiOH)_4{\cdot}C_2H_6O$. Clearly, the crystallization conditions which lead to the formation of phases other than (I) are highly specific. It is also notable that no solvent-free

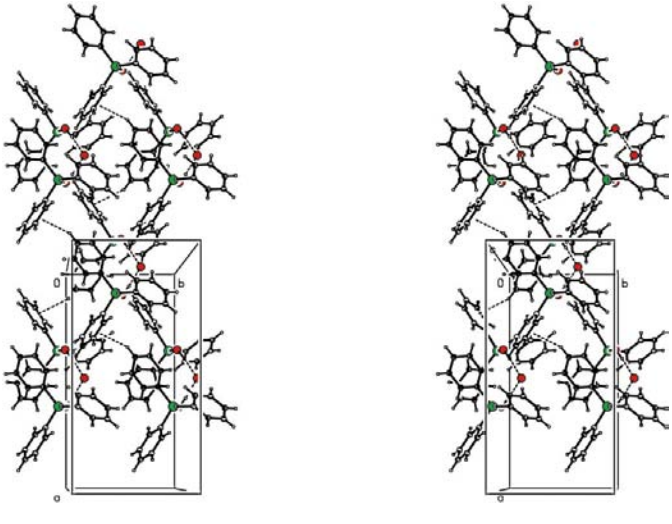

Figure 5
A stereoview of part of the crystal structure of (II), showing the formation of the [110] and [1$\bar{1}$0] chains which comprise the (001) sheet built from O—H$\cdots$O and C—H$\cdots\pi$(arene) interactions. For the sake of clarity, only the O atom of the DMSO molecule is shown.

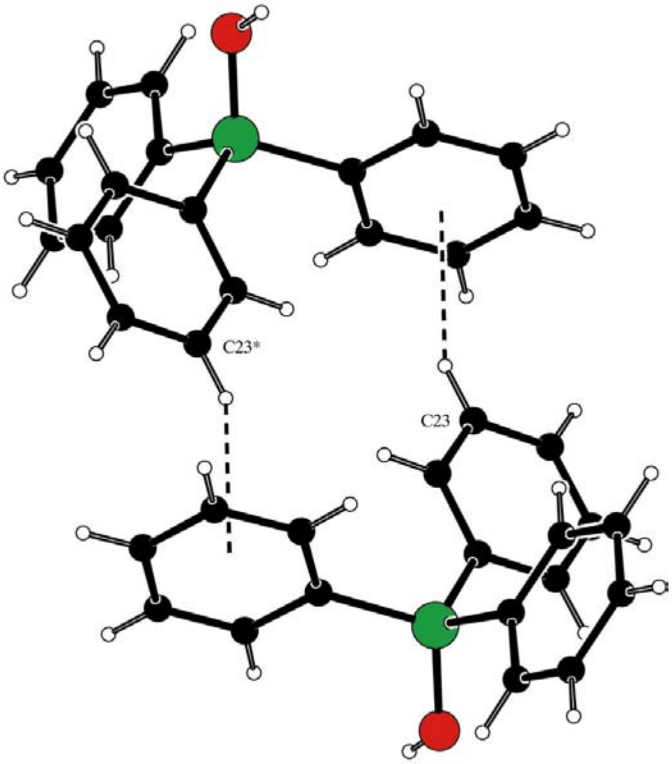

Figure 6
Part of the crystal structure of (II), showing the formation of the centrosymmetric C—H$\cdots\pi$(arene) motif linking the (001) sheets. The atom marked with an asterisk (*) is at the symmetry position ($-x$, $1 - y$, $1 - z$).

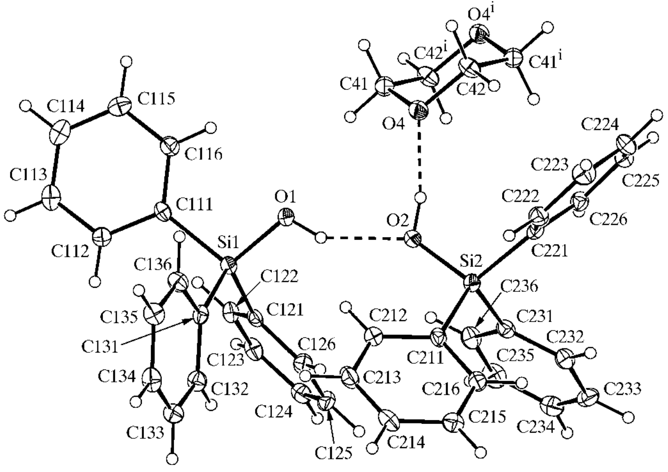

Figure 7
The molecular components of (III), showing the atom-labelling scheme [symmetry code: (i) $1 - x$, $1 - y$, $1 - z$]. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

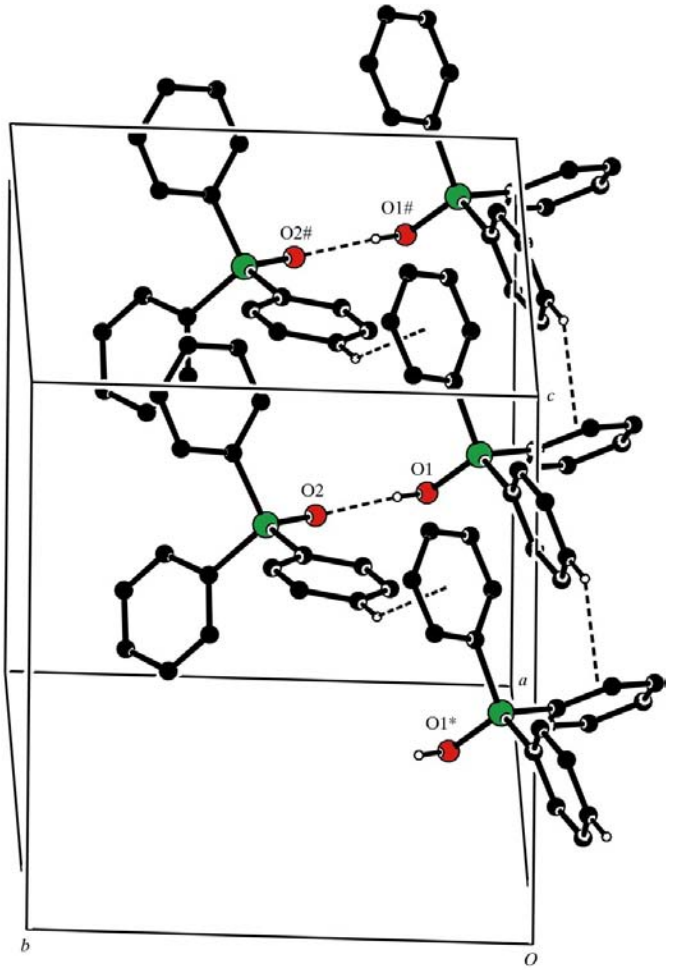

Figure 8
Part of the crystal structure of (III), showing the formation of a [100] chain by C—H···π(arene) interactions. Atoms marked with an asterisk (*) or hash (#) are at the symmetry positions $(x - 1, y, z)$ and $(1 + x, y, z)$, respectively.

polymorph of triphenylsilanol other than (I) has yet been observed, suggesting that this phase may be particularly stable, despite the absence from the structure of both C—H···π(arene) and π–π-stacking interactions.

In summary, the hydroxyl H atoms in $(Ph_3SiOH)_4$ have been located at 120 (2) K, as for $(Ph_3SnOH)_n$ (Glidewell *et al.*, 2002), and the ring disorder is straightforwardly handled. At 198 K (Puff *et al.*, 1991), the ring disorder appears to be more extensive, and at ambient temperature, the extensive disorder and the poor diffraction intensity effectively preclude a straightforward refinement. In the solvates (II) and (III), finite aggregates generated by O—H···O hydrogen bonds are further linked by C—H···π(arene) interactions.

Experimental

A sample of triphenylsilanol was purchased from Aldrich. Crystals of (I), (II) and (III) suitable for single-crystal X-ray diffraction were grown by slow evaporation of solutions in the appropriate solvents. The same phase, (I), was obtained from solutions in acetone, chlorobenzene, chloroform, cyclohexane, dichloromethane, ethyl acetate, ethylbenzene, toluene and *p*-xylene.

Compound (I)

Crystal data

$C_{18}H_{16}OSi$
$M_r = 276.40$
Triclinic, $P\overline{1}$
$a = 15.0514$ (2) Å
$b = 19.5456$ (2) Å
$c = 23.0921$ (6) Å
$\alpha = 108.0455$ (5)°
$\beta = 102.7869$ (6)°
$\gamma = 101.3081$ (8)°
$V = 6037.06$ (19) Å^3

$Z = 16$
$D_x = 1.216$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 279 reflections
$\theta = 2.9$–27.4°
$\mu = 0.15$ mm^{-1}
$T = 120$ (2) K
Plate, colourless
0.40 × 0.26 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO-SMN*; Otwinowski & Minor, 1997)
$T_{min} = 0.915$, $T_{max} = 0.993$
25 408 measured reflections

25 279 independent reflections
15 492 reflections with $I > 2\sigma(I)$
$R_{int} = 0.084$
$\theta_{max} = 27.4°$
$h = -19 \rightarrow 19$
$k = -25 \rightarrow 25$
$l = -28 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.174$
$S = 1.02$
25 279 reflections
1471 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0913P)^2 + 0.8422P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.04$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}

Table 1
Selected interatomic distances (Å) for (I).

Si1—O1	1.6452 (18)	Si5—O5	1.6397 (19)
Si2—O2	1.646 (2)	Si6—O6	1.6446 (19)
Si3—O3	1.6398 (19)	Si7—O7	1.645 (2)
Si4—O4	1.6435 (19)	Si8—O8	1.6441 (19)

Table 2
Hydrogen-bonding geometry (Å, °) for (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O4	0.88	1.81	2.666 (3)	163
O2—H2···O1	0.88	1.83	2.686 (3)	165
O3—H3···O2	0.88	1.86	2.700 (3)	159
O4—H4···O3	0.88	1.82	2.680 (3)	166
O5—H5···O6	0.88	1.84	2.671 (3)	157
O6—H6···O7	0.89	1.81	2.657 (3)	160
O7—H7···O8	0.88	1.81	2.635 (3)	156
O8—H8···O5	0.88	1.81	2.644 (3)	156

Compound (II)

Crystal data

$2C_{18}H_{16}OSi \cdot C_2H_6OS$
$M_r = 630.94$
Monoclinic, $C2/c$
$a = 16.8842$ (3) Å
$b = 8.5391$ (2) Å
$c = 23.7053$ (6) Å
$\beta = 96.1310$ (10)°
$V = 3398.18$ (13) Å^3
$Z = 4$

$D_x = 1.233$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3808 reflections
$\theta = 3.1$–27.5°
$\mu = 0.20$ mm^{-1}
$T = 120$ (2) K
Block, colourless
0.35 × 0.25 × 0.15 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO-SMN*; Otwinowski & Minor, 1997)
$T_{\min} = 0.910$, $T_{\max} = 0.969$
21 226 measured reflections
3808 independent reflections
2792 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.066$
$\theta_{\max} = 27.5°$
$h = -21 \rightarrow 21$
$k = -11 \rightarrow 11$
$l = -30 \rightarrow 30$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.109$
$S = 1.00$
3808 reflections
207 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 2.3569P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}

Table 3
Selected interatomic distance (Å) for (II).

Si1—O1	1.6300 (12)

Table 4
Hydrogen-bonding geometry (Å, °) for (II).

*Cg*1 and *Cg*3 are the centroids of aryl rings C11–C16 and C31–C36, respectively.

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
O1—H1$\cdots$O2	0.89	1.85	2.7320 (15)	172
C14—H14$\cdots$*Cg*3$^{\rm i}$	0.95	2.88	3.757 (2)	154
C23—H23$\cdots$*Cg*1$^{\rm ii}$	0.95	2.90	3.767 (2)	152

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

Compound (III)

Crystal data

$4C_{18}H_{16}OSi\cdot C_4H_8O_2$
$M_r = 1193.7$
Triclinic, $P\overline{1}$
$a = 9.3033$ (3) Å
$b = 11.6929$ (5) Å
$c = 14.7154$ (6) Å
$\alpha = 87.730$ (3)°
$\beta = 80.906$ (3)°
$\gamma = 87.8040$ (17)°
$V = 1578.58$ (11) Å^3
$Z = 1$
$D_x = 1.256$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6905 reflections
$\theta = 3.1$–$27.5°$
$\mu = 0.15$ mm^{-1}
$T = 150$ (2) K
Block, colourless
0.25 × 0.15 × 0.10 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO-SMN*; Otwinowski & Minor, 1997)
$T_{\min} = 0.937$, $T_{\max} = 0.985$
23 329 measured reflections
6905 independent reflections
3185 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.136$
$\theta_{\max} = 27.5°$
$h = -12 \rightarrow 12$
$k = -15 \rightarrow 15$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.135$
$S = 0.92$
6905 reflections
388 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.039P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.41$ e Å^{-3}

Table 5
Selected interatomic distances (Å) for (III).

Si1—O1	1.636 (2)	Si2—O2	1.642 (2)

Table 6
Hydrogen-bonding geometry (Å, °) for (III).

*Cg*2 and *Cg*3 are the centroids of aryl rings C111–C116 and C121–C126, respectively.

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
O1—H1$\cdots$O2	0.88	1.96	2.760 (3)	151
O2—H2$\cdots$O4	0.88	1.83	2.692 (3)	168
C134—H134$\cdots$*Cg*2$^{\rm i}$	0.95	2.99	3.727 (4)	135
C214—H214$\cdots$*Cg*3$^{\rm i}$	0.95	2.82	3.606 (4)	141

Symmetry code: (i) $x - 1, y, z$.

Compound (I) is triclinic; space group $P\overline{1}$ was selected and confirmed by the subsequent analysis. At 120 (2) K, all H atoms were located from difference maps and treated as riding atoms, with C—H = 0.95 Å; hydroxyl H atoms were treated as riding, using *AFIX* 3 (*SHELXL*97; Sheldrick, 1997) and with O—H = 0.88 Å, and the mean of the eight values was derived directly from the difference maps. One ring is disordered at 120 (2) K over two sets of sites, denoted C21*r* (*r* = 1 to 6) and C21*N* (*N* = *A* to *F*), occupying closely similar volumes of space. The rings were modelled as rigid hexagons and their site-occupancy factors, when constrained to sum to 1, refined to 0.52 (4) and 0.48 (4); hence, the occupancies were thereafter fixed at 0.50 for both orientations. Compound (II) crystallized in the monoclinic system. The systematic absences permitted $C2/c$ and Cc as possible space groups; $C2/c$ was selected and confirmed by the analysis. The position of the hydroxyl H atom was obtained from a difference map, and all H atoms in the silanol component were treated as riding atoms, with C—H = 0.95 Å and O—H = 0.89 Å. The disordered DMSO molecule was modelled using the *PART* instruction in *SHELXL*97. The unique C atom was treated as two parts, with occupancies of 0.5 on a fixed common site, and the S atom was positioned in two symmetry-related sites. This was performed to permit positioning of the H atoms of the methyl groups, which were treated as riding atoms, with C—H = 0.98 Å. The partial C atoms were given common anisotropic displacement parameters, as were the S atoms. Compound (III) is triclinic; space group $P\overline{1}$ was selected and confirmed by the subsequent analysis. The H atoms were treated as riding, with C—H = 0.95 (aromatic) or 0.99 Å (CH_2), and O—H = 0.88 Å.

For all compounds, data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2002); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1558). Services for accessing these data are described at the back of the journal.

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