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Bis[2-(triphenylsilyl)phenyl] Disulfide

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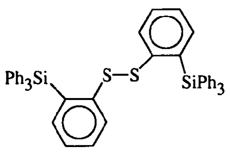
Abstract

The title compound, $C_{48}H_{38}S_2Si_2$, was obtained by the oxidation of the corresponding thiol in the presence of copper(I) chloride; the molecular structure is reported.

Comment

Bis[2-(triphenylsilyl)phenyl] disulfide, (I), was obtained fortuitously from the reaction under basic conditions of the corresponding thiol (TPSTH) with copper(I) chloride. The TPST unit has been studied (Ahmet *et al.*, 1995) as an example of a sterically hindered thiolate ligand to transition metals. The molecular geometry of the title compound suggests that steric interactions between the two halves of the molecule do not play an important role in governing bond distances, bond angles or even the torsion angle across the S1—S2 bond.



(I)

A database survey of C—S—S—C fragments (Allen *et al.*, 1987) found that S—S bond distances are bimodally distributed; for torsion angles in the ranges 75–105 and 0–20°, S—S bond-distance means were found to be 2.031 (15) and 2.070 (22) Å, respectively. The corresponding values in (I) are 84.1 (1)° and 2.037 (1) Å, placing (I) in the upper quartile of Allen's first set. The C—S and C—Si distances are in the upper ranges of Allen's survey. The title compound contains no intramolecular non-bonded distances (hydrogen excluded) between individual TPST half molecules shorter than 3.5 Å, except for C52—S1, C53—S1 and C12—S2; it therefore seems unlikely that steric factors constrain this molecule. The dispositions of the triphenylsilyl fragments in this molecule and in metal complexes have been discussed elsewhere (Ahmet *et al.*, 1995).

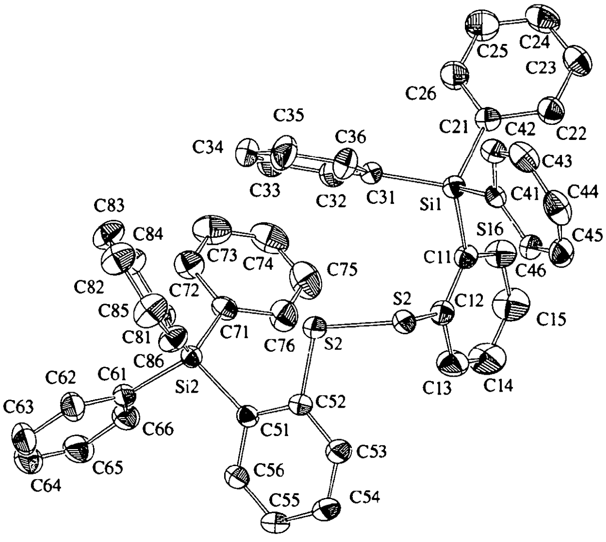


Fig. 1. The structure of the title compound; displacement ellipsoids are drawn at the 30% probability level and H atoms have been omitted for clarity (Zsolnai, 1994).

Experimental

2-(Triphenylsilyl)benzenethiol (TPSTH) was synthesized according to the method of Zheng (1993). A mixture of 0.01 g (0.1 mmol) CuCl, 0.15 g (0.14 mmol) TPSTH and 0.04 g (0.6 mmol) EtONa in 15 ml of dimethoxyethane and 10 ml of ethanol was heated under reflux for 3 h. Triphenylphosphine (0.05 g, 0.2 mmol) was added to the yellow solution which was then refluxed for a further 2 h. The solution was concentrated *in vacuo*, 10 ml acetone was added and a white solid formed which was filtered off, washed twice with 10 ml ether and airdried giving a yield of 0.13 g. Recrystallization from dichloromethane/ether gave colourless cube-shaped crystals suitable for X-ray analysis.

Crystal data

$C_{48}H_{38}S_2Si_2$
$M_r = 735.08$
Triclinic
$P\bar{1}$
$a = 9.276\,(6)$ Å
$b = 9.879\,(6)$ Å
$c = 22.499\,(13)$ Å
$\alpha = 92.54\,(4)°$
$\beta = 95.08\,(4)°$
$\gamma = 102.67\,(4)°$
$V = 1999.4\,(2)$ Å^3
$Z = 2$
$D_x = 1.221$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 20$–$21°$
$\mu = 0.226$ mm^{-1}
$T = 291\,(1)$ K
Cube
$0.5 \times 0.5 \times 0.5$ mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (Fair, 1990)
$T_{\min} = 0.840$, $T_{\max} = 0.893$
7488 measured reflections
7009 independent reflections
5142 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.025$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -26 \rightarrow 26$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0375$
$wR(F^2) = 0.1172$
$S = 1.010$
7009 reflections
621 parameters
H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2 + 0.6217P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.004$
$\Delta\rho_{\max} = 0.303$ e Å^{-3}
$\Delta\rho_{\min} = -0.314$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

S1—C12	1.783 (3)	Si1—C11	1.882 (3)
S1—S2	2.0374 (14)	Si2—C71	1.872 (3)
S2—C52	1.797 (3)	Si2—C81	1.874 (3)
Si1—C31	1.869 (2)	Si2—C61	1.883 (3)
Si1—C41	1.875 (2)	Si2—C51	1.888 (2)
Si1—C21	1.879 (3)		
C12—S1—S2	105.93 (9)	C21—Si1—C11	109.73 (11)
C52—S2—S1	106.77 (9)	C71—Si2—C81	114.87 (11)
C31—Si1—C41	113.00 (11)	C71—Si2—C61	106.81 (11)
C31—Si1—C21	109.14 (11)	C81—Si2—C61	107.49 (11)
C41—Si1—C21	106.39 (11)	C71—Si2—C51	110.39 (11)
C31—Si1—C11	109.52 (11)	C81—Si2—C51	108.85 (11)
C41—Si1—C11	109.00 (11)	C61—Si2—C51	108.19 (11)
C12—S1—S2—C52	84.1 (1)		

Table 2. *Contact distances (Å)*

S1···C52	3.081 (3)	S2···C12	3.054 (3)
S1···C53	3.266 (3)		

Intensity data were corrected for Lorentz and polarization factors and for absorption using empirical scan data.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1994). Software used to prepare material for publication: *SHELXL*93.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: KA1224). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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