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## Key indicators

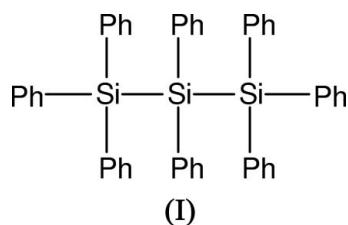
Single-crystal X-ray study  
 $T = 173$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.015$  Å  
 $R$  factor = 0.105  
 $wR$  factor = 0.310  
Data-to-parameter ratio = 17.3For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.A trigonal polymorph of diphenylbis-  
(triphenylsilyl)silane

A new polymorph of diphenylbis(triphenylsilyl)silane,  $\text{C}_{48}\text{H}_{40}\text{Si}_3$ , has been discovered. Until now, only an orthorhombic polymorph of the title compound was known. The newly found trigonal polymorph crystallizes in  $R\bar{3}$  with one molecule in the asymmetric unit.

Received 3 March 2006  
Accepted 6 March 2006

## Comment

Due to the need for well defined oligochlorosilanes as standards for  $^{29}\text{Si}$  NMR measurements, the usual synthetic pathway was used, first building the phenylated structure followed by chlorination using  $\text{HCl}/\text{AlCl}_3$  (Hengge & Kovar, 1977).



The structure of diphenylbis(triphenylsilyl)silane, (I), has already been determined at room temperature in the orthorhombic space group  $Pbca$  (Charissé *et al.*, 1993). We collected a data set at low temperature and obtained, surprisingly, the space group  $R\bar{3}$ . To verify that the crystal structure had not undergone a phase transition upon cooling, the same crystal was investigated again, at room temperature. We still obtained the space group  $R\bar{3}$ . Furthermore, we determined the structure at room temperature using a crystal which had not been cooled already, and again obtained the trigonal structure. In this way, a new polymorph of (I) has been discovered. A perspective view of the title compound is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 5.27 plus one update; *MOGUL* Version 1.1; Allen, 2002); only the Si—Si—Si angle is widened to  $120.61(12)^\circ$ . Since no coordinates of the orthorhombic polymorph are available, no comparison between the two structures can be made. In the trigonal polymorph, there are channels running along the  $c$  axis, which might contain solvent molecules. However, the difference Fourier map did not show any significant peaks, although *PLATON* (Spek, 2003) indicates that these channels represent a solvent-accessible volume.

## Experimental

If not otherwise mentioned, all operations were performed at room temperature.  $\text{Ph}_3\text{SiLi}$  was prepared by cleavage of  $\text{Si}_2\text{Ph}_6$  (10.7 g,

21 mmol) with Li (2.4 g, 346 mmol) in anhydrous tetrahydrofuran (THF; 100 ml) over 4 h with rapid stirring (Brook & Gilman, 1953). The reaction mixture was filtered through glass wool and added slowly (approximately 8 h) by use of a dropping funnel to  $\text{Ph}_2\text{SiCl}_2$  (4 ml, 19 mmol) dissolved in anhydrous THF (100 ml) constantly stirred (Gilman *et al.*, 1952). The mixture was stirred for an additional 12 h. The reaction mixture was filtered by suction. The brown filtrate was concentrated to yield a brown oil. This was dissolved in hot toluene (approximately 50 ml) and filtered hot. To the clear filtrate was added hexane (approximately 350 ml) and the mixture was stored at 254 K for 20 h, yielding (0.4 g, 1 mmol, 5%) of yellow needles (Charissé *et al.*, 1993). After replacing hexane with diethyl ether, additional colourless needles (1.1 g, 2 mmol, 10%) could be isolated. The first batch of yellow needles melted around 525 K [uncorrected; 575 K (Charissé *et al.*, 1993)]. IR  $\nu(\text{Si}-\text{Si})$ : 535  $\text{cm}^{-1}$ .  $^{29}\text{Si}$  NMR (79.5 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_2\text{HSiCl}$  as external standard):  $-19.6$  ( $\text{SiPh}_3$ ) and  $-42.9$  p.p.m. ( $\text{SiPh}_2$ ).

#### Crystal data

$\text{C}_{48}\text{H}_{40}\text{Si}_3$   
 $M_r = 701.07$   
 Trigonal,  $R\bar{3}$   
 $a = 26.947$  (2) Å  
 $c = 30.741$  (2) Å  
 $V = 19332$  (2) Å<sup>3</sup>  
 $Z = 18$   
 $D_x = 1.084$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 8951 reflections  
 $\theta = 2.4\text{--}23.4^\circ$   
 $\mu = 0.14$  mm<sup>-1</sup>  
 $T = 173$  (2) K  
 Needle, colourless  
 $0.31 \times 0.04 \times 0.03$  mm

#### Data collection

Stoe IPDS-II two-circle diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)  
 $T_{\min} = 0.948$ ,  $T_{\max} = 0.985$   
 59096 measured reflections

7936 independent reflections  
 2753 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.132$   
 $\theta_{\text{max}} = 25.6^\circ$   
 $h = -32 \rightarrow 32$   
 $k = -32 \rightarrow 32$   
 $l = -37 \rightarrow 36$

#### Refinement

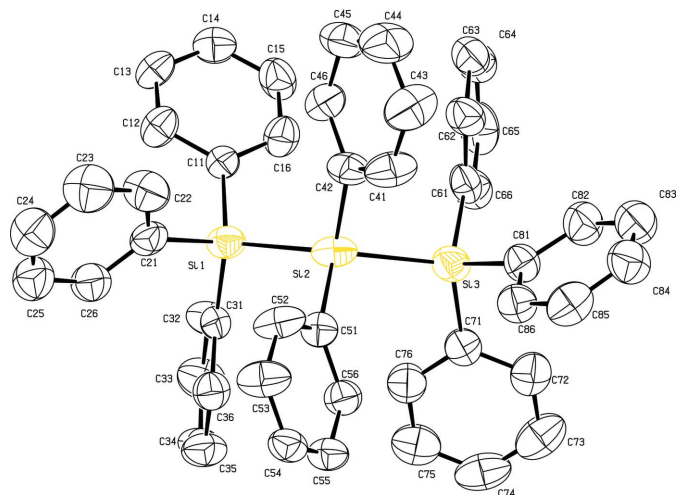
Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.105$   
 $wR(F^2) = 0.310$   
 $S = 0.90$   
 7936 reflections  
 460 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.1364P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.82$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.29$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Si1—Si2	2.443 (3)	Si2—Si3	2.493 (4)
Si1—Si2—Si3	120.61 (12)		



**Figure 1**

Perspective view of (I), showing the atom numbering and displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.

H atoms were located in a difference Fourier synthesis. They were refined with fixed individual displacement parameters [ $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ ] using a riding model, with  $\text{C}-\text{H} = 0.95$  Å. The high  $R_{\text{int}}$  value and the rather high final  $R$  values are due to the rather weak data.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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