Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Bis(triphenylsilyl)selenide

Iliana Medina, Joel T. Mague,* James P. Donahue and Mark J. Fink

Department of Chemistry, Tulane University, New Orleans, LA 70118, USA

Correspondence e-mail: joelt@tulane.edu

Received 12 July 2005
Accepted 21 July 2005
Online 23 July 2005

Bis(triphenylsilyl)selenide, $[(C_6H_5)_3Si]_2Se$ or $C_{36}H_{30}SeSi_2$, exists in the solid state as monomeric angular molecules. Si—Se—Si angles of 111.56 (3) and 109.45 (3)° are found in the two independent molecules.

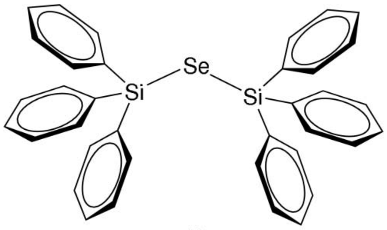
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.025
wR factor = 0.060
Data-to-parameter ratio = 18.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Silicon–chalcogen chemistry is dominated by the formation of cyclic and polycyclic compounds. Not many examples of acyclic compounds containing an Si—E—Si (E = S, Se and Te) unit are known (Lange & Herzog, 2002). The bis(triorganosilyl)chalcogenides have been widely used for the synthesis of binary and ternary metal chalcogenide nanoclusters (DeGroot *et al.*, 2003; Fenske *et al.*, 2000). For instance, the synthesis of the solid-state compound CdSe has been achieved under mild conditions by the reaction of cadmium(II) chloride or dimethylcadmium with $Se(SiMe_3)_2$ (Stuczynski *et al.*, 1989). Silylated chalcogenides also find applications in organic synthesis in the preparation of thiocarbonyl compounds, thiiranes, selenocarbonyl compounds and 1,3,5-triselenanes (Murai *et al.*, 1988). The solid-state structures of $(Ph_3Si)_2O$ (Glidewell & Liles, 1978) and $(Ph_3Si)_2S$ (Wojnowski *et al.*, 1985) represent contrasting extremes: in the siloxane, the Si—O—Si angle is 180° by symmetry, whereas in the silathiane, the Si—S—Si angle is 111.94 (8)°.



(I)

We now report the solid-state structure of the next heavier analog, $(Ph_3Si)_2Se$, (I). There are two independent molecules in the asymmetric unit, each having crystallographically imposed twofold rotation symmetry. The correctness of the choice of space group with its requirement of two independent molecules is supported by the significant difference (Δ/σ = 5) in the Si1—Se1 and Si2—Se2 distances and the absence of higher symmetry as determined by *PLATON* (Spek, 2005). A perspective view of the two independent molecules of (I) is shown in Fig. 1, with selected bond distances and angles contained in Table 1. Silaselenane (I) has a slightly smaller bond angle at the chalcogen atom [111.56 (3) and 109.45 (3)°] than observed for $(Ph_3Si)_2S$. The small bond angles at sulfur and selenium relative to oxygen in the series $(Ph_3Si)_2E$ (E = O, S and Se) are attributed to a lack of appreciable dative π bonding from either the sulfur or selenium to the silicon in conjunction with an enhanced degree of p-orbital character in the σ-bonding orbitals of sulfur and selenium as compared with oxygen.

Experimental

Bis(triphenylsilyl)selenide, $(Ph_3Si)_2Se$, was synthesized according to the procedure of Detty & Seidler (1982), which involves reaction of lithium triethylborohydride and elemental selenium in tetrahydrofuran (THF), followed by the addition of a slight excess of chlorotriphenylsilane. The product was extracted with benzene and filtered. $(Ph_3Si)_2Se$ was isolated as an air-sensitive colorless crystalline solid in 87% yield. X-ray quality crystals were obtained from a benzene–pentane (1:1) solution upon storage at 273 K.

Crystal data

$C_{36}H_{30}SeSi_2$
$M_r = 597.74$
Monoclinic, $C2$
$a = 18.159\ (2)$ Å
$b = 7.8122\ (9)$ Å
$c = 21.777\ (3)$ Å
$\beta = 106.724\ (2)^\circ$
$V = 2958.6\ (6)$ Å^3
$Z = 4$

$D_x = 1.342$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5506 reflections
$\theta = 2.3$–28.3°
$\mu = 1.37$ mm^{-1}
$T = 100\ (2)$ K
Plate, colorless
$0.35 \times 0.22 \times 0.11$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{min} = 0.713$, $T_{max} = 0.867$
12735 measured reflections

6362 independent reflections
6027 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 27.5^\circ$
$h = -23 \rightarrow 23$
$k = -10 \rightarrow 10$
$l = -28 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.060$
$S = 1.02$
6362 reflections
353 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0074P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.60$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Absolute structure: Flack (1983), 2714 Friedel pairs
Flack parameter: 0.016 (5)

Table 1
Selected geometric parameters (Å, °).

Se1—Si1	2.2976 (6)	Se2—Si2	2.2947 (6)
Si1—C1	1.867 (2)	Si2—C31	1.869 (2)
Si1—C7	1.870 (2)	Si2—C25	1.869 (2)
Si1—C13	1.872 (2)	Si2—C19	1.874 (2)
Si1^{i}—Se1—Si1	111.56 (3)	Si2^{ii}—Se2—Si2	109.45 (3)
C1—Si1—C7	109.58 (10)	C31—Si2—C25	109.79 (10)
C1—Si1—C13	110.28 (9)	C31—Si2—C19	110.24 (9)
C7—Si1—C13	109.79 (9)	C25—Si2—C19	109.38 (9)
C1—Si1—Se1	113.26 (7)	C31—Si2—Se2	113.25 (7)
C7—Si1—Se1	112.71 (6)	C25—Si2—Se2	112.36 (7)
C13—Si1—Se1	100.91 (7)	C19—Si2—Se2	101.52 (7)

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

The H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.95 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$.

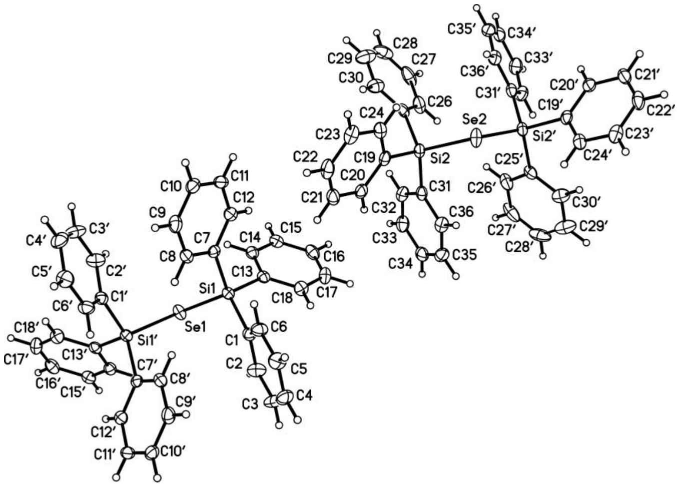


Figure 1
A perspective view of the two independent molecules of (I). Displacement ellipsoids are drawn at the 50% probability level. Primed atoms in the molecule containing Se1 are related to the unprimed atoms by the symmetry operation $(-x, y, 1 - z)$, while in the molecule containing Se2, the relevant symmetry operation is $(-x + 1, y, 2 - z)$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

We thank the Chemistry Department of Tulane University for support of the X-ray laboratory and the Louisiana Educational Quality Support Fund administered by the Louisiana Board of Regents for purchase of the CCD diffractometer under grant LEQSF (2002–03)-ENH-TR-67. MJF thanks NASA (NCC3-946) for support through the Tulane Institute for Macromolecular Engineering and Science, NASA (NCC3-97) through the Ohio Aerospace Institute, and the Louisiana Board of Regents (LaSPACE C164092).

References

Bruker (2000). *SMART* (Version 5.625) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2004). *SAINT-Plus*. Version 7.03. Bruker AXS Inc., Madison, Wisconsin, USA.
DeGroot, M. W., Taylor, N. J. & Corrigan, J. F. (2003). *J. Am. Chem. Soc.* **125**, 864–865.
Detty, M. R. & Seidler, M. D. (1982). *J. Org. Chem.* **47**, 1354–1356.
Fenske, D., Langetepe, T., Kappes, M. M., Hampe, O. & Weis, P. (2000). *Angew. Chem. Int. Ed.* **39**, 1857–1860.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Glidewell, C. & Liles, D. C. (1978). *Acta Cryst.* B**34**, 124–128.
Lange, H. & Herzog, U. J. (2002). *J. Organomet. Chem.* **660**, 36–42.
Murai, S., Ryu, I., Ogawa, A., Sonoda, N., Segi, M., Nakajima, T. & Suga, S. (1988). *J. Am. Chem. Soc.* **110**, 1976–1978.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2002). *SADABS*. Version 2.05. University of Göttingen, Germany.
Spek, A. L. (2005). *PLATON*. Utrecht University, The Netherlands.
Stuczynski, S. M., Brennan, J. G. & Steigerwald, M. L. (1989). *Inorg. Chem.* **28**, 4431–4432.
Wojnowski, W., Peters, K., Peters, E. M. & Von Schnering, H. G. (1985). *Z. Anorg. Allg. Chem.* **525**, 121–126.

o2688 Medina *et al.* • $C_{36}H_{30}SeSi_2$ *Acta Cryst.* (2005). E**61**, o2687–o2688