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Key indicators

Single-crystal X-ray study

$T = 294$ K

Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å

R factor = 0.050

w R factor = 0.108

Data-to-parameter ratio = 23.4

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

1,5-Di-*tert*-butyl-3,3,7,7,10,10-hexaphenyl-2,4,6,8,9,11-hexaoxa-3,7,10-trisila-1,5-digermabicyclo[3.3.3]undecane

In the title complex, $[\text{Ge}_2(\text{C}_4\text{H}_9)_2(\text{C}_{12}\text{H}_{10}\text{O}_2\text{Si})_3]$, the Si atoms are coordinated by two O atoms and two phenyl groups and the Ge atoms by three O atoms and a tertiary butyl group.

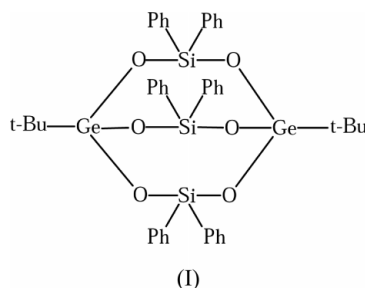
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Comment

This structure determination is part of a series of structures of germanium complexes. Introductory material was presented in recent papers (Akkurt *et al.*, 2003; Öztürk *et al.*, 2003). Here we present the structure of the title compound, (I).



The Ge—C [average 1.9455 (14) Å] and Ge—O [average 1.7608 (8) Å] bond lengths are in the expected ranges (Akkurt *et al.*, 2003; Öztürk *et al.*, 2003; Allen *et al.*, 1987). The Si—C bond lengths range from 1.858 (4) to 1.873 (3) Å and the Si—O bond lengths range from 1.626 (2) to 1.634 (2) Å. The C—Si—C angles range from 109.41 (14) to 112.19 (15)° and the O—Si—O angles range from 110.17 (12) to 111.95 (11)°. These values can be compared with those found in 4,4,8,8-tetraethyl-2,2,6,6-tetraphenyl-1,3,5,7,2,6,4,8-tetroxadisiladigermocane, where the Ge—O and Si—O bond lengths are 1.755 (5) and 1.610 (6) Å, respectively, and the O—Si—O and O—Ge—O bond angle are 112.7 (3) and 104.8 (3)°, respectively (Akkurt *et al.*, 1994).

The geometries of the six phenyl rings are very similar; the average C—C bond length is 1.379 (1) Å and the rings are all planar, with a maximum deviation of 0.022 (5) Å for atom C2 (Nardelli, 1995). The dihedral angle between the phenyl rings on atom Si1 is 66.1 (1)°, on atom Si2 59.9 (1)° and on atom Si3 69.5 (1)°. The dihedral angles between neighbouring phenyl rings range from 59.93 (13) to 69.49 (13)°.

Experimental

A solution of ^tBuGeCl₃ (4.4 g, 18.4 mmol) in toluene (100 ml) was added dropwise to a solution of triethylamine and Ph₂Si(OH)₂ (6 g, 27.6 mmol), ratio 1:1, in an acetone–toluene mixture (250 ml). This mixture was stirred at room temperature for 6 h. After cooling, the precipitate was filtered off and the solvent evaporated. The resulting

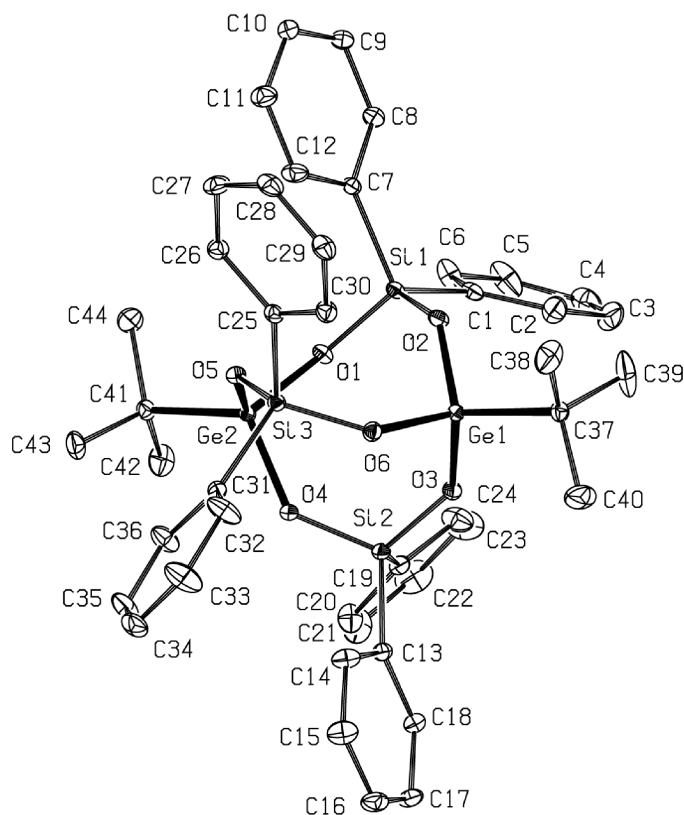


Figure 1
View of the molecule of (I), with displacement ellipsoids drawn at the 10% probability level. The H atoms have been omitted for clarity.

oily product was crystallized using a chloroform–acetone mixture (m.p.: 508 K). Analysis calculated for $C_{44}Ge_2H_{48}O_6Si_3$: C 58.52, H 5.26, O 10.63, Si 9.33%; found: C 58.47, H 5.33, O 10.61, Si 9.75%.

Crystal data

$[Ge_2(C_4H_9)_2(C_{12}H_{10}O_2Si)_3]$
 $M_r = 902.31$
 Triclinic, $P\bar{1}$
 $a = 10.789$ (5) Å
 $b = 11.212$ (5) Å
 $c = 19.071$ (5) Å
 $\alpha = 96.831$ (5)°
 $\beta = 105.521$ (5)°
 $\gamma = 90.594$ (5)°
 $V = 2204.9$ (15) Å³

$Z = 2$
 $D_x = 1.359$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 226 reflections
 $\theta = 6.0$ – 20.0 °
 $\mu = 1.49$ mm⁻¹
 $T = 294$ (2) K
 Irregular, colorless
 $0.22 \times 0.18 \times 0.12$ mm

Data collection

Nonius KappaCCD diffractometer ω scan
 Absorption correction: refined from ΔF (cubic fit to $\sin\theta/\lambda$, 24 parameters; Parkin *et al.*, 1995)
 $T_{min} = 0.735$, $T_{max} = 0.841$
 49 113 measured reflections
 11 629 independent reflections
 6529 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.076$
 $\theta_{max} = 29.0$ °
 $h = -14 \rightarrow 14$
 $k = -15 \rightarrow 15$
 $l = -26 \rightarrow 26$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.109$
 $S = 0.95$
 11629 reflections
 496 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.7808P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.97$ e Å⁻³
 $\Delta\rho_{min} = -0.45$ e Å⁻³

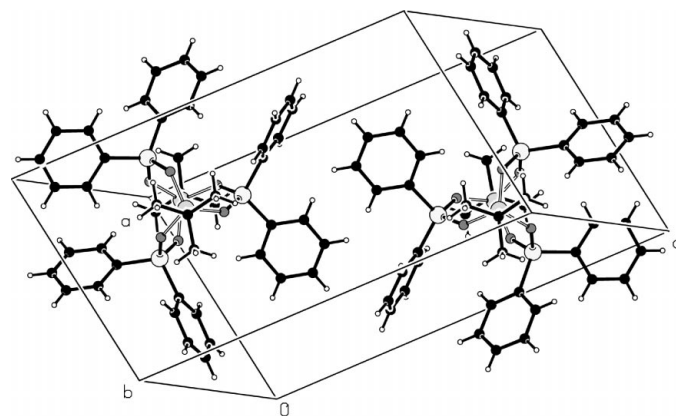


Figure 2
A view of the unit-cell contents of (I).

Table 1

Selected geometric parameters (Å, °).

Ge1—O2	1.759 (2)	Si1—C1	1.865 (4)
Ge1—O3	1.756 (2)	Si1—C7	1.858 (4)
Ge1—O6	1.762 (2)	Si2—O3	1.634 (3)
Ge1—C37	1.945 (4)	Si2—O4	1.641 (2)
Ge2—O1	1.757 (2)	Si2—C13	1.873 (4)
Ge2—O4	1.761 (2)	Si2—C19	1.840 (4)
Ge2—O5	1.770 (2)	Si3—O5	1.632 (2)
Ge2—C41	1.946 (4)	Si3—O6	1.634 (2)
Si1—O1	1.626 (2)	Si3—C25	1.864 (4)
Si1—O2	1.634 (2)	Si3—C31	1.872 (3)
O2—Ge1—O3	107.36 (10)	O1—Si1—O2	110.17 (12)
O2—Ge1—O6	108.17 (10)	C1—Si1—C7	112.19 (15)
O3—Ge1—O6	105.75 (10)	O3—Si2—O4	110.89 (12)
O1—Ge2—O4	107.13 (10)	C13—Si2—C19	111.80 (16)
O1—Ge2—O5	107.83 (10)	O5—Si3—O6	111.95 (11)
O4—Ge2—O5	103.97 (10)	C25—Si3—C31	109.41 (14)
C37—Ge1—O6—Si3	143.6 (2)	O4—Si2—O3—Ge1	23.3 (2)
C41—Ge2—O5—Si3	143.75 (18)	O3—Si2—O4—Ge2	45.1 (2)
O2—Si1—O1—Ge2	35.9 (2)	O6—Si3—O5—Ge2	37.0 (2)
O1—Si1—O2—Ge1	29.9 (2)	O5—Si3—O6—Ge1	26.9 (3)

The rotational orientations of the methyl H atoms of the *tert*-butyl groups were refined by the circular Fourier methods available in *SHELXL97* (Sheldrick, 1997), with C—H bond lengths of 0.96 Å and $U_{iso}(H) = 1.5U_{eq}$ (parent C atom). Other H atoms were included using a riding model, with C—H bond lengths of 0.93 Å and $U_{iso}(H) = 1.2U_{eq}$ (parent C atom).

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *EVALCCD* (Duisenberg, 1998); data reduction: *EVALCCD*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

References

- Akkurt, M., Kök, T. R., Faleschine, P., Randaccio, L., Puff, H. & Schuh, W. (1994). *J. Organomet. Chem.* **470**, 59–66.
 Akkurt, M., Öztürk, S., Kök, T. R. & Fun, H.-K. (2003). *Acta Cryst.* **E59**, m664–m665.
 Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

- Duisenberg, A. J. M. (1998). PhD thesis, University of Utrecht, The Netherlands.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Nonius (2002). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Öztürk, S., Akkurt, M., Kök, T. R. & Fun, H.-K. (2003). *Acta Cryst.* **E59**, m1018–m1019.
- Parkin, S., Moezzi, B. & Hope, H. (1995). *J. Appl. Cryst.* **28**, 53–56.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.