

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ ) for (2)

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{eq}$
Se	0.04443 (3)	0.21728 (2)	0.05502 (2)	0.05538 (11)
O	0.0492 (2)	0.16221 (11)	0.22408 (12)	0.0621 (5)
N	0.2559 (2)	0.14000 (12)	0.28273 (13)	0.0463 (4)
C1	0.1760 (2)	0.28094 (13)	0.11507 (15)	0.0442 (5)
C2	0.2232 (2)	0.25693 (13)	0.19475 (14)	0.0429 (5)
C3	0.3160 (3)	0.3049 (2)	0.2381 (2)	0.0553 (6)
C4	0.3620 (3)	0.3760 (2)	0.2032 (2)	0.0666 (7)
C5	0.3163 (3)	0.3993 (2)	0.1248 (2)	0.0644 (7)
C6	0.2254 (3)	0.35232 (14)	0.0807 (2)	0.0542 (6)
C7	0.1687 (2)	0.18249 (14)	0.23479 (14)	0.0438 (5)
C8	0.2294 (2)	0.07190 (13)	0.33304 (14)	0.0450 (5)
C9	0.0989 (3)	0.0437 (2)	0.3504 (2)	0.0558 (6)
C10	0.0834 (3)	-0.0219 (2)	0.4035 (2)	0.0696 (8)
C11	0.1936 (4)	-0.0598 (2)	0.4384 (2)	0.0739 (8)
C12	0.3228 (3)	-0.0320 (2)	0.4211 (2)	0.0733 (8)
C13	0.3404 (3)	0.0333 (2)	0.3687 (2)	0.0600 (6)
C14	0.0722 (5)	0.2586 (3)	-0.0579 (2)	0.0875 (11)

Table 4. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (2)

Se—C1	1.920 (2)	N—C7	1.343 (3)
Se—C14	1.935 (3)	N—C8	1.412 (3)
O—C7	1.228 (3)		
C1—Se—C14	99.55 (13)	O—C7—C2	120.7 (2)
C7—N—C8	128.8 (2)	N—C7—C2	116.9 (2)
O—C7—N	122.4 (2)		
C14—Se—C1—C6	19.8 (2)	C1—C2—C7—O	-34.2 (3)
C14—Se—C1—C2	-160.9 (2)	C3—C2—C7—N	-37.0 (3)
C8—N—C7—O	-5.4 (4)	C1—C2—C7—N	146.2 (2)
C8—N—C7—C2	174.2 (2)	C7—N—C8—C13	172.5 (2)
C3—C2—C7—O	142.6 (2)	C7—N—C8—C9	-9.6 (4)

Of the 11 506 reflections measured for compound (1) (9535 of which are unique), 3088 reflections with either large negative  $F^2$  values or showing evidence of overlap with adjacent reflections were omitted from the refinement. Of these 8418 reflections, 7182 were unique and used in the refinement.

For both compounds, program(s) used to solve structures: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, least-squares-planes data and torsion angles have been deposited with the IUCr (Reference: TA1069). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Tetrabenzylgermanium

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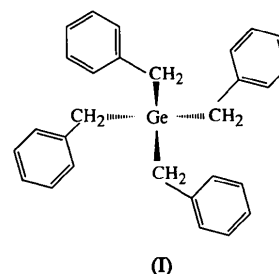
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## Abstract

Tetrabenzylgermanium,  $[\text{Ge}(\text{C}_7\text{H}_7)_4]$ , crystallizes with approximate  $\bar{4}$  ( $S_4$ ) molecular symmetry and with almost perfect staggering about the Ge—C bonds. The Ge—C—C angles are significantly larger than the tetrahedral value, with a mean value of  $115 (1)^\circ$ .

## Comment

Few tribenzyl or tetrabenzyl derivatives of the elements have been structurally characterized. For germanium, the sole examples appear to be  $\text{O}[(\text{PhCH}_2)_3\text{Ge}]_2$  (Glidewell & Liles, 1979) and  $\text{S}[(\text{PhCH}_2)_3\text{Ge}]_2$  (Glidewell & Liles, 1982). In the oxo compound, the  $(\text{PhCH}_2)_3\text{Ge}$  fragments have precise threefold rotation symmetry, with the molecule as a whole having  $\bar{3}$  ( $S_6$ ) symmetry, while in the thio compound, although the whole molecule has only twofold rotation symmetry, the  $(\text{PhCH}_2)_3\text{Ge}$  fragments again have approximate threefold local symmetry. In both cases, the benzyl groups are arranged so as to give almost perfect staggering about the Ge—C bonds, but the core conformations of the two compounds are entirely different (see Fig. 2 of Glidewell & Liles, 1982). These observations lead naturally to the question of the conformational behaviour of the corresponding homoleptic tetrabenzyl compound and we report here the structure of tetrabenzylgermanium,  $(\text{PhCH}_2)_4\text{Ge}$ , (I).



The title compound crystallizes in space group  $P2_1/n$ , with a single molecule in the asymmetric unit. Although no crystallographic symmetry is imposed by the space group, the molecules nonetheless exhibit near  $\bar{4}$  ( $S_4$ ) symmetry (Table 2 and Fig. 1). The lack of crystallographic symmetry in (I) may be contrasted with both

(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>Ge (space group  $P\bar{4}2_1c$ ; Chieh, 1971; Karipides & Haller, 1972) and (C<sub>6</sub>F<sub>5</sub>)<sub>4</sub>Ge (space group  $P4_1/a$ ; Karipides, Forman, Thomas & Reed, 1974), the molecules of which have exact  $\bar{4}$  symmetry. Each benzyl group has its C<sub>n1</sub>—C<sub>n7</sub> ( $n = 1-4$ ) bond essentially *trans* to the Ge—C<sub>m7</sub> bond ( $m = 1-4$ ;  $m = 3, 4, 2, 1$  when  $n = 1, 2, 3, 4$ , respectively; see Table 2 and Fig. 1), corresponding to almost perfect staggering about the Ge—C<sub>n7</sub> bonds. Similarly, the absolute values of the torsion angles Ge—C<sub>n7</sub>—C<sub>n1</sub>—C<sub>n2</sub> are very similar. Hence, if any one benzyl group were notionally removed, the conformation of the remaining (PhCH<sub>2</sub>)<sub>3</sub>Ge fragment would have approximate threefold rotational symmetry.

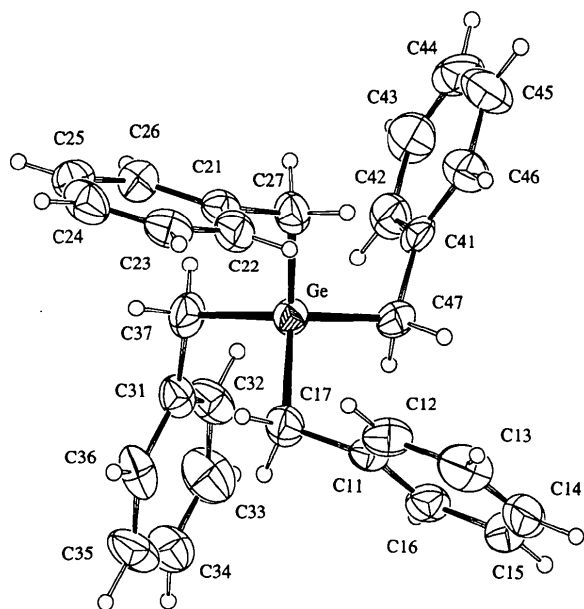


Fig. 1. A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

The bond lengths within the molecule are unexpected; the mean Ge—C distance is 1.957 (11) Å. It is noteworthy that the Ge—C<sub>n7</sub>—C<sub>n1</sub> angles ( $n = 1-4$ ) are all significantly greater than the tetrahedral value, precisely as was observed in O(PhCH<sub>2</sub>)<sub>3</sub>Ge<sub>2</sub> (Glidewell & Liles, 1979); the metrical precision of the structure determination for S[(PhCH<sub>2</sub>)<sub>3</sub>Ge]<sub>2</sub> (Glidewell & Liles, 1982) does not allow such a comparison to be made. Large M—C—C angles have also been observed in both O[(PhCH<sub>2</sub>)<sub>3</sub>Ti]<sub>2</sub> (Stoekli-Evans, 1974) and O[(PhCH<sub>2</sub>)<sub>3</sub>Si]<sub>2</sub> (Glidewell & Liles, 1981). A preliminary report of the structure of tetrabenzyltin, (PhCH<sub>2</sub>)<sub>4</sub>Sn (Davies, Jarvis & Kilbourn, 1971), appears to show no such effect, but the reported precision is low and there are no coordinates in the Cambridge Structural Database (Allen & Kennard, 1993). The highly asymmetric structure reported for tetrabenzyltitanium, (PhCH<sub>2</sub>)<sub>4</sub>Ti (Bassi, Allegra, Scordamaglia & Chioccola, 1971; Davies, Jarvis & Kilbourn, 1971), may be in-

dicative of the presence of two mono- $\eta$  and two di- $\eta$  benzyl groups. The structures of tetrabenzylzirconium, (PhCH<sub>2</sub>)<sub>4</sub>Zr (Davies, Jarvis, Kilbourn & Pioli, 1971), and tetrabenzylhafnium, (PhCH<sub>2</sub>)<sub>4</sub>Hf (Davies, Jarvis & Kilbourn, 1971), may in turn be indicative of the presence of one mono- $\eta$  and three di- $\eta$  benzyl ligands.

## Experimental

A sample of (I) was prepared from germanium(IV) chloride and benzylmagnesium bromide (Glockling & Hooton, 1962). Crystals suitable for single-crystal X-ray diffraction analysis were grown from diethyl ether solution.

### Crystal data

[Ge(C<sub>7</sub>H<sub>7</sub>)<sub>4</sub>]  
 $M_r = 437.09$   
 Monoclinic  
 $P2_1/n$   
 $a = 10.0809$  (9) Å  
 $b = 12.1757$  (9) Å  
 $c = 19.1154$  (11) Å  
 $\beta = 91.443$  (9)°  
 $V = 2345.5$  (3) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.238$  Mg m<sup>-3</sup>  
 $D_m$  not measured

### Data collection

Enraf-Nonius CAD-4 diffractometer  
 $\theta/2\theta$  scans  
 Absorption correction:  $\psi$  scans at 4° steps (North, Phillips & Mathews, 1968)  
 $T_{\min} = 0.6968$ ,  $T_{\max} = 0.9976$   
 4331 measured reflections  
 4076 independent reflections

### Refinement

Refinement on  $F^2$   
 $R(F) = 0.0564$   
 $wR(F^2) = 0.1171$   
 $S = 0.828$   
 4076 reflections  
 262 parameters  
 H atoms riding (SHELXL93 defaults; C—H = 0.93–0.97 Å)

Mo  $K\alpha$  radiation  
 $\lambda = 0.7107$  Å  
 Cell parameters from 25 reflections  
 $\theta = 9.22-13.14^\circ$   
 $\mu = 1.316$  mm<sup>-1</sup>  
 $T = 294$  (1) K  
 Plate  
 $0.36 \times 0.32 \times 0.15$  mm  
 Colorless

1671 observed reflections [ $I > 2\sigma(I)$ ]  
 $R_{\text{int}} = 0.045$   
 $\theta_{\text{max}} = 24.83^\circ$   
 $h = -11 \rightarrow 11$   
 $k = 0 \rightarrow 14$   
 $l = 0 \rightarrow 22$   
 3 standard reflections  
 frequency: 120 min  
 intensity decay: 2.4% (allowed for by scaling)

$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.098$   
 $\Delta\rho_{\text{max}} = 0.432$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.300$  e Å<sup>-3</sup>  
 Extinction correction: none  
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{\text{eq}}$
Ge	0.12820 (7)	0.15634 (5)	0.82328 (3)	0.0535 (2)
C11	-0.0009 (6)	0.1376 (6)	0.6834 (3)	0.057 (2)

C12	-0.0379 (7)	0.2284 (6)	0.6471 (4)	0.075 (2)
C13	-0.0166 (8)	0.2357 (7)	0.5755 (5)	0.098 (3)
C14	0.0427 (9)	0.1513 (10)	0.5406 (4)	0.105 (3)
C15	0.0800 (8)	0.0618 (8)	0.5771 (5)	0.091 (3)
C16	0.0583 (7)	0.0536 (6)	0.6478 (4)	0.071 (2)
C17	-0.0250 (6)	0.1252 (5)	0.7601 (3)	0.067 (2)
C21	0.0351 (7)	0.3763 (5)	0.8548 (4)	0.057 (2)
C22	-0.0418 (7)	0.4332 (5)	0.8057 (4)	0.070 (2)
C23	-0.1540 (7)	0.4894 (5)	0.8247 (4)	0.071 (2)
C24	-0.1927 (7)	0.4897 (6)	0.8929 (5)	0.084 (2)
C25	-0.1174 (7)	0.4343 (6)	0.9410 (4)	0.073 (2)
C26	-0.0056 (7)	0.3793 (5)	0.9230 (3)	0.064 (2)
C27	0.1531 (6)	0.3141 (5)	0.8333 (3)	0.065 (2)
C31	0.0565 (8)	-0.0277 (6)	0.9098 (3)	0.062 (2)
C32	0.1535 (8)	-0.1059 (7)	0.9181 (4)	0.082 (2)
C33	0.1331 (10)	-0.2151 (8)	0.9114 (5)	0.106 (3)
C34	0.0121 (12)	-0.2510 (7)	0.8964 (4)	0.101 (3)
C35	-0.0906 (9)	-0.1810 (8)	0.8884 (4)	0.099 (3)
C36	-0.0661 (8)	-0.0672 (7)	0.8949 (4)	0.083 (2)
C37	0.0887 (7)	0.0913 (5)	0.9140 (3)	0.076 (2)
C41	0.4154 (7)	0.1263 (5)	0.8197 (3)	0.056 (2)
C42	0.4730 (7)	0.0664 (6)	0.8743 (4)	0.078 (2)
C43	0.5905 (8)	0.0983 (9)	0.9053 (5)	0.106 (3)
C44	0.6533 (9)	0.1877 (10)	0.8848 (6)	0.120 (5)
C45	0.5991 (10)	0.2494 (8)	0.8305 (6)	0.118 (4)
C46	0.4808 (7)	0.2188 (6)	0.7987 (4)	0.078 (2)
C47	0.2874 (6)	0.0907 (5)	0.7852 (3)	0.065 (2)

 Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Ge—C17	1.974 (5)	C11—C17	1.500 (7)
Ge—C27	1.946 (6)	C21—C27	1.478 (7)
Ge—C37	1.956 (6)	C31—C37	1.486 (8)
Ge—C47	1.950 (6)	C41—C47	1.498 (8)
C17—Ge—C27	110.3 (3)	C37—Ge—C47	110.6 (3)
C17—Ge—C37	106.9 (3)	Ge—C17—C11	115.8 (4)
C17—Ge—C47	109.4 (2)	Ge—C27—C21	115.5 (4)
C27—Ge—C37	110.0 (3)	Ge—C37—C31	113.3 (4)
C27—Ge—C47	109.6 (3)	Ge—C47—C41	115.1 (4)
C12—C11—C17—Ge	-99.5 (6)	C32—C31—C37—Ge	91.4 (7)
C37—Ge—C17—C11	-162.6 (5)	C27—Ge—C37—C31	174.9 (5)
C22—C21—C27—Ge	-100.3 (6)	C42—C41—C47—Ge	93.5 (6)
C47—Ge—C27—C21	174.0 (5)	C17—Ge—C47—C41	168.2 (5)

Examination of the structure with *PLATON* (Spek, 1995a) showed that there were no solvent-accessible voids in the crystal lattice.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992). Cell refinement: *CELDIM* and *SET4* (Enraf–Nonius, 1992). Data reduction: *DATRD2* in *NRCVAX94* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *NRCVAX94* and *SHELXL93* (Sheldrick, 1993). Molecular graphics: *NRCVAX94*, *ORTEPII* (Johnson, 1976) as implemented in *PLATON*, *PLUTON* (Spek 1995b). Software used to prepare material for publication: *NRCVAX94*, *SHELXL93*, *WordPerfect* (macro *PREPCIF*).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: AB1377). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Chloro[( $\alpha$ -dimethylamino[3]ferrocenophan-2-yl)diphenylphosphino]gold(I) Benzene Solvate

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## Abstract

The title complex, chloro{[1,1'-(1-dimethylamino-1,3-propanediyl)-2-ferrocenyl]diphenylphosphino-*P*}gold(I) 1.5-benzene solvate,  $[\text{AuCl}(\text{C}_{27}\text{H}_{28}\text{FeNP})].1.5\text{C}_6\text{H}_6$ , is the first gold(I) complex comprising a ferrocenophane ligand bearing phosphorus and nitrogen coordination sites. The Au atom is preferentially bound to the soft