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Acetates and Acetato Complexes.
IV.* The Crystal and Molecular Structure of Tin Tetraacetate

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Abstract

Crystals of $C_8H_{12}O_8Sn$ are monoclinic, space group $P2_1$, $a = 7.851$ (1), $b = 26.473$ (6), $c = 12.911$ (1) Å, $\beta = 106.47$ (1)°, $Z = 8$. Data were measured using a four-circle diffractometer, and the structure was solved by the heavy-atom method. $R = 0.050$ for 3258 observed reflections. There are four independent $Sn(O_2CMe)_4$ molecules each with nearly dodecahedral geometry, but with variable Sn—O distances, because of overcrowding (Sn—O mean 2·22, range 2·13 to 2·29 Å).

The structures and crystal data of the Group IV acetates have been the subject of conflicting reports (Kamenar, 1963; Kamenar & Bruvo, 1972). We have examined tin tetraacetate (I) in connection with the synthetic and spectroscopic studies reported in earlier parts of this series (Alcock, Tracy & Waddington, 1976*a*,*b*). The resulting structure shows the Sn atoms to have distorted eight coordination and explains conflicting NMR and IR data.

Experimental

Hygroscopic, white, lath-shaped crystals of (I) were prepared by the method of Alcock, Tracy & Waddington (1976*a*,*b*). The bounding faces were {230}, {101} and {001} with separations 0·12, 0·17, 0·79 mm respectively for the crystal studied. It was mounted in a capillary (in a dry box) to prevent decomposition.

Crystal data

$C_8H_{12}O_8Sn$ (I). $M_r = 354.7$, monoclinic, $P2_1$. $a = 7.851$ (1), $b = 26.473$ (6), $c = 12.911$ (1) Å, $\beta = 106.47$ (1)°, $U = 2573.2$ (8) Å^3, $Z = 8$ (assumed), $D_c = 1.839$ Mg m^{-3}, $F(000) = 1392$, Mo $K\alpha$ radiation, graphite monochromator, $\lambda = 0.71069$ Å, $\mu = 1.99$ mm^{-1}. No density measurement could be made.

Cell constants and standard deviations were determined by a least-squares method from the positions of 15 reflections with the standard programs of a Syntex $P2_1$ four-circle diffractometer. Data were collected with this instrument, in θ–2θ mode, range (2θ) 1·8 + ($\alpha_2 - \alpha_1$)°, with variable scan rates of 1–29° min^{-1}, depending on the intensity of a 2 s pre-scan. $2\theta_{\max}$ was 50°, and for the range 35–50° the pre-scan time was increased to 8 s, and those reflections with pre-scan intensities significantly below those expected for an observed reflection were not collected. 4255 reflections were collected, of which 3258 were considered observed [$I/\sigma(I) \geq 3.0$] and used in refinement. Three standards were examined after every 100 reflections; they showed slight decrease with time, which was compensated for by multiplying each F value by $(1 + \alpha T)$ where T was the elapsed collection time and α was chosen appropriately. Lorentz, polarization and absorption corrections (with *ABSCOR*, Alcock, 1970) were applied; the maximum and minimum transmission factors were 0·81 and 0·68.

The data set used for initial structure solution was collected on two separate crystals with a two-circle diffractometer (Alcock & Tracy, 1976). The apparent absences $0k0$, $k = 2n$ and $h0l$, $l = 2n$ suggested space group $P2_1/c$, while the conditions for $P2_1/a$ or $P2_1/n$ were also approximately satisfied. The peaks of a Patterson synthesis, however, were split. In particular, the expected $0,2y,\frac{1}{2}$ peak (with $V \simeq 0$) consisted of two separate maxima, clearly resolved in V. This can only be explained if the structure is pseudo-symmetric. An explanation of these Patterson peaks was found, with four independent Sn atoms in space group $P2_1$, with their positions close to but not satisfying a c glide condition. This can be seen from the y coordinates in Table 1, for which no shift of origin allows them to be paired precisely. The $h0l$ absences then arise accidentally. This structure was refined by Fourier and least-squares methods to R 0·14. All 16 acetate groups were located, but the positions of several were badly defined.

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* Part III: Alcock & Tracy (1976). The following corrections to the coordinates of that paper should be noted: C(241), $x = -0.024$; C(251), $x = 0.386$; C(252), $x = 0.301$.

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Table 1. *Atomic coordinates* ($\times 10^4$) *and temperature factors* ($Å^2$) ($\times 10^3$), *with standard deviations in parentheses*

Anisotropic temperature factors in the form:
$\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sn(1)	−169 (3)	0	1204 (2)	37·8 (13)	33·4 (14)	62·7 (14)	1·5 (10)	9·2 (11)	−3·3 (11)
Sn(2)	158 (3)	4805 (1)	3656 (2)	40 (1)	54 (2)	45 (1)	−9 (1)	−1 (1)	−5 (1)
Sn(3)	4786 (3)	2561 (1)	1330 (2)	34·6 (12)	47·2 (15)	44·8 (13)	1·8 (9)	3·9 (10)	1·7 (10)
Sn(4)	5160 (3)	7376·9 (4)	3800 (2)	42 (1)	40 (2)	50 (1)	−5 (1)	1 (1)	3 (1)

	x	y	z	U		x	y	z	U
O(211)	−1709 (15)	4392 (6)	2318 (13)	44 (5)	O(111)	−1348 (19)	636 (5)	1837 (12)	48 (4)
O(212)	1218 (14)	4281 (6)	2733 (11)	48 (4)	O(112)	−2052 (24)	−142 (4)	2224 (14)	64 (6)
C(211)	−350 (14)	4173 (4)	2192 (9)	96 (10)	C(111)	−2201 (14)	333 (4)	2259 (8)	31 (6)
C(212)	−612 (24)	3763 (7)	1359 (16)	63 (6)	C(112)	−3456 (30)	547 (7)	2831 (20)	64 (8)
O(221)	−508 (14)	4218 (7)	4628 (12)	50 (5)	O(121)	1098 (22)	658 (5)	684 (14)	57 (4)
O(222)	2236 (15)	4530 (5)	5106 (11)	52 (4)	O(122)	1748 (24)	−108 (4)	192 (14)	51 (5)
C(221)	1059 (13)	4234 (4)	5248 (8)	52 (4)	C(121)	1922 (16)	367 (5)	216 (9)	36 (6)
C(222)	1547 (23)	3885 (7)	6201 (13)	71 (6)	C(122)	3173 (40)	597 (8)	−339 (26)	77 (10)
O(231)	2648 (15)	5232 (5)	3616 (10)	51 (4)	O(131)	−2333 (17)	177 (4)	−307 (10)	46 (3)
O(232)	−5 (14)	5422 (6)	2505 (11)	60 (4)	O(132)	−1703 (18)	−611 (4)	276 (11)	54 (4)
C(231)	1654 (13)	5488 (4)	2857 (8)	57 (7)	C(131)	−2619 (13)	−293 (4)	−380 (8)	65 (11)
C(232)	2496 (22)	5893 (7)	2347 (17)	67 (6)	C(132)	−4122 (25)	−484 (6)	−1294 (15)	60 (7)
O(241)	−2372 (17)	5197 (6)	3528 (10)	58 (4)	O(141)	1954 (18)	71 (4)	2739 (10)	48 (4)
O(242)	0 (17)	5350 (7)	4917 (12)	80 (6)	O(142)	1209 (18)	−681 (4)	1973 (11)	53 (4)
C(241)	−1617 (17)	5414 (6)	4410 (10)	49 (10)	C(141)	2142 (14)	−403 (4)	2714 (8)	83 (11)
C(242)	−2697 (33)	5766 (17)	4882 (26)	152 (22)	C(142)	3545 (29)	−648 (7)	3611 (16)	77 (9)
O(311)	5798 (14)	3140 (7)	378 (13)	65 (6)	O(411)	4022 (30)	6713 (7)	4449 (20)	65 (8)
O(312)	2903 (15)	2970 (5)	−82 (11)	67 (4)	O(412)	3168 (26)	7505 (6)	4598 (15)	74 (5)
C(311)	4231 (14)	3200 (4)	−222 (9)	32 (5)	C(411)	3056 (21)	7035 (6)	4737 (12)	80 (10)
C(312)	3934 (25)	3564 (8)	−1147 (15)	73 (6)	C(412)	1709 (48)	6849 (10)	5271 (34)	83 (13)
O(321)	3689 (13)	3171 (7)	2196 (13)	63 (7)	O(421)	7152 (21)	7523 (5)	2984 (14)	56 (4)
O(322)	6563 (14)	2976 (5)	2756 (11)	66 (4)	O(422)	6162 (26)	6733 (6)	2934 (16)	59 (6)
C(321)	5232 (13)	3226 (4)	2831 (8)	19 (4)	C(421)	7148 (17)	7066 (5)	2701 (10)	94 (12)
C(322)	5498 (26)	3609 (8)	3718 (16)	53 (8)	C(422)	8376 (35)	6910 (8)	2055 (23)	80 (8)
O(331)	2324 (22)	2142 (8)	1174 (12)	81 (7)	O(431)	7590 (18)	7200 (4)	5112 (11)	52 (4)
O(332)	4669 (17)	2020 (6)	2598 (11)	59 (4)	O(432)	6535 (17)	7986 (4)	4956 (11)	48 (4)
C(331)	3077 (16)	1934 (5)	2068 (10)	56 (6)	C(431)	7634 (13)	7653 (4)	5431 (8)	55 (8)
C(332)	2025 (27)	1566 (10)	2523 (20)	92 (9)	C(432)	9065 (30)	7802 (7)	6426 (17)	82 (9)
O(341)	7544 (16)	2287 (5)	1330 (9)	55 (4)	O(441)	2809 (19)	7302 (5)	2425 (11)	57 (4)
O(342)	5089 (13)	1971 (7)	197 (12)	48 (5)	O(442)	3986 (21)	8068 (5)	2812 (13)	68 (5)
C(341)	6760 (13)	1992 (4)	575 (8)	67 (7)	C(441)	2853 (15)	7769 (5)	2233 (9)	56 (10)
C(342)	7868 (19)	1650 (6)	103 (13)	54 (5)	C(442)	1492 (33)	7980 (8)	1268 (18)	80 (10)

It was for this reason that a new, more accurate set of data was obtained. Refinement on four Sn atoms (anisotropic temperature factors) alone gave R 0·24, and successive Fourier syntheses revealed all the light atoms. The e.s.d.'s of these atoms showed that deviations from regular acetate groups should not be detectable, and they were therefore held rigid (C—C 1·50, C—O 1·264 Å, ∠O—C—O 118·3°). In the final refinement the Sn atoms only were given anisotropic temperature factors; weights $w = 0{\cdot}6/(\sin\theta/\lambda)$ were used. A test of absolute configuration was inconclusive, with no significant difference in R between the two hands. A final difference synthesis showed no significant features. The final R was 0·050.* The atoms are numbered O(mn1), O(mn2), C(mn1) central, C(mn2) terminal, for the nth acetate group attached to Sn(m) (Fig. 1). Final atomic parameters are in Table 1, bond lengths and angles in Table 2. For the atoms in

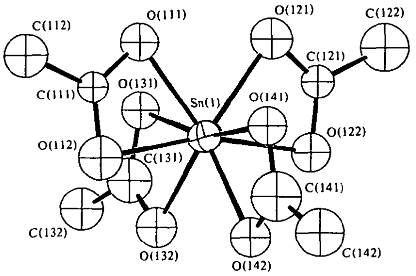

Fig. 1. The coordination of Sn(1) with the atomic numbering. The coordination of the other Sn atoms is superficially indistinguishable from this.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33884 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

the rigid groups, e.s.d.'s of fractional coordinates were calculated from the errors in the group parameters, taking into account the correlation between these; these e.s.d.'s were used to calculate the errors in Sn—O distances, which should therefore be valid to the normal level of approximation.

Table 2. *Bond lengths* (Å), *angles* (°) *and* O—O *contact distances* (<3·2 Å) *with e.s.d.'s in parentheses*

The letters *A* and *B* indicate whether the oxygen atoms occupy the *A* or *B* positions of the dodecahedron; opposite pairs of the latter are easily identifiable by the O—Sn—O angles of ~160° between them.

Sn(1)–O(111)	2·188 (15)	*A*	Sn(3)–O(311)	2·246 (18)	*B*
Sn(1)–O(112)	2·272 (21)	*B*	Sn(3)–O(312)	2·271 (12)	*A*
Sn(1)–O(121)	2·202 (16)	*A*	Sn(3)–O(321)	2·268 (18)	*B*
Sn(1)–O(122)	2·274 (20)	*B*	Sn(3)–O(322)	2·255 (12)	*A*
Sn(1)–O(131)	2·244 (11)	*B*	Sn(3)–O(331)	2·187 (18)	*A*
Sn(1)–O(132)	2·164 (11)	*A*	Sn(3)–O(332)	2·197 (15)	*B*
Sn(1)–O(141)	2·206 (12)	*B*	Sn(3)–O(341)	2·284 (13)	*A*
Sn(1)–O(142)	2·191 (12)	*A*	Sn(3)–O(342)	2·187 (17)	*B*
Sn(2)–O(211)	2·212 (14)	*A*	Sn(4)–O(411)	2·239 (23)	*A*
Sn(2)–O(212)	2·144 (15)	*B*	Sn(4)–O(412)	2·131 (23)	*B*
Sn(2)–O(221)	2·154 (17)	*B*	Sn(4)–O(421)	2·154 (19)	*B*
Sn(2)–O(222)	2·228 (12)	*A*	Sn(4)–O(422)	2·296 (19)	*A*
Sn(2)–O(231)	2·271 (13)	*A*	Sn(4)–O(431)	2·212 (12)	*B*
Sn(2)–O(232)	2·187 (15)	*B*	Sn(4)–O(432)	2·253 (12)	*A*
Sn(2)–O(241)	2·205 (14)	*A*	Sn(4)–O(441)	2·178 (13)	*B*
Sn(2)–O(242)	2·205 (18)	*B*	Sn(4)–O(442)	2·271 (14)	*A*

	Angle O—Sn—O	*d*(O—O)		Angle O—Sn—O	*d*(O—O)
O(111)–O(121)	77·3 (6)	2·74 (3)	O(211)–O(221)	83·2 (6)	2·90 (2)
O(111)–O(122)	136·9 (5)		O(211)–O(222)	131·3 (6)	
O(111)–O(131)	82·4 (5)	2·92 (2)	O(211)–O(231)	127·7 (6)	
O(111)–O(132)	123·6 (5)		O(211)–O(232)	86·6 (6)	3·02 (2)
O(111)–O(141)	83·8 (5)	2·93 (3)	O(211)–O(241)	77·9 (5)	2·78 (2)
O(111)–O(142)	132·2 (5)		O(211)–O(242)	137·4 (5)	
O(112)–O(121)	137·0 (5)		O(212)–O(221)	93·1 (6)	3·12 (2)
O(112)–O(122)	163·2 (4)		O(212)–O(222)	87·1 (5)	3·01 (2)
O(112)–O(131)	94·7 (5)		O(212)–O(231)	80·6 (5)	2·86 (2)
O(112)–O(132)	81·1 (5)	2·89 (2)	O(212)–O(232)	93·6 (5)	3·16 (2)
O(112)–O(141)	86·8 (6)	3·08 (2)	O(212)–O(241)	138·2 (4)	
O(112)–O(142)	85·4 (5)	3·03 (2)	O(212)–O(242)	161·0 (4)	
O(121)–O(131)	82·3 (5)	2·92 (2)	O(221)–O(231)	136·5 (4)	
O(121)–O(132)	130·6 (6)		O(221)–O(232)	163·3 (4)	
O(121)–O(141)	85·2 (5)	2·98 (2)	O(221)–O(241)	91·2 (5)	3·12 (2)
O(121)–O(142)	125·5 (6)		O(221)–O(242)	88·2 (7)	3·03 (3)
O(122)–O(131)	89·3 (5)	3·18 (2)	O(222)–O(231)	75·6 (5)	2·76 (2)
O(122)–O(132)	86·7 (5)	3·05 (2)	O(222)–O(232)	134·6 (5)	
O(122)–O(141)	94·1 (6)		O(222)–O(241)	130·3 (5)	
O(122)–O(142)	80·5 (5)	2·88 (2)	O(222)–O(242)	77·0 (5)	2·76 (2)
O(131) O(141)	163·0 (4)		O(231)–O(241)	121·8 (5)	
O(131)–O(142)	136·1 (4)		O(231)–O(242)	85·4 (5)	3·04 (2)
O(132)–O(141)	136·1 (4)		O(232)–O(241)	73·7 (5)	2·63 (2)
O(132)–O(142)	76·2 (4)	2·69 (2)	O(232)–O(242)	90·4 (6)	3·12 (2)
O(311)–O(321)	91·6 (7)		O(411)–O(421)	138·3 (7)	
O(311)–O(322)	83·2 (5)	2·99 (2)	O(411)–O(422)	80·1 (8)	2·92 (4)
O(311)–O(331)	137·4 (5)		O(411)–O(431)	84·1 (6)	2·98 (3)
O(311)–O(332)	161·6 (4)		O(411)–O(432)	118·7 (7)	
O(311)–O(341)	74·6 (5)	2·74 (2)	O(411)–O(441)	84·1 (7)	2·96 (3)
O(311)–O(342)	89·9 (6)	3·14 (3)	O(411)–O(442)	134·5 (7)	
O(312)–O(321)	78·7 (6)	2·88 (2)	O(412)–O(421)	160·5 (6)	
O(312)–O(322)	122·3 (5)		O(412)–O(422)	138·9 (7)	
O(312)–O(331)	79·1 (5)	2·84 (2)	O(412)–O(431)	104·8 (6)	
O(312)–O(332)	139·0 (5)		O(412)–O(432)	81·1 (6)	2·85 (2)
O(312)–O(341)	123·6 (5)		O(412) O(441)	80·8 (6)	2·79 (2)
O(312)–O(342)	88·5 (5)	3·12 (2)	O(412)–O(442)	84·6 (6)	2·97 (3)
O(321)–O(331)	87·5 (6)	3·08 (3)	O(421)–O(431)	79·9 (6)	2·80 (2)
O(321)–O(332)	89·7 (6)	3·15 (3)	O(421)–O(432)	85·2 (6)	2·99 (2)
O(321)–O(341)	135·9 (4)		O(421)–O(441)	100·6 (6)	
O(321)–O(342)	164·1 (4)		O(421)–O(442)	79·4 (6)	2·83 (2)
O(322)–O(331)	130·4 (6)		O(422)–O(431)	82·8 (6)	2·98 (2)
O(322)–O(332)	81·7 (5)		O(422)–O(432)	133·4 (6)	
O(322)–O(341)	77·7 (4)	2·85 (2)	O(422)–O(441)	82·1 (6)	2·94 (2)
O(322)–O(342)	136·9 (4)		O(422)–O(442)	117·7 (6)	
O(331)–O(341)	130·7 (6)		O(431)–O(441)	162·3 (4)	
O(331)–O(342)	80·7 (6)	2·84 (2)	O(431)–O(442)	136·5 (5)	
O(332)–O(341)	91·9 (5)		O(432)–O(441)	137·9 (4)	
O(332)–O(342)	93·9 (6)		O(432)–O(442)	80·7 (5)	2·93 (2)

Computing was with the XRAY 76 system (Stewart, 1976) on a Burroughs B6700 computer; scattering factors were taken from *International Tables for X-ray Crystallography* (1974) in the analytical form.

Results and discussion

The asymmetric unit contains four independent $Sn(OAc)_4$ molecules. Superficially, all are very similar, having the pseudo-dodecahedral coordination often found with four bidentate ligands; Fig. 1 shows the coordination of Sn(1) to illustrate this. On close examination, considerable irregularity is apparent. This can be seen in the Sn—O distances, which vary from 2·13 to 2·29 Å, far beyond experimental error. It might be expected that the atoms in the *A* positions of the dodecahedron (Hoard & Silverton, 1963), with four neighbours, would have shorter Sn—O distances than those in the *B* positions, with five neighbours. This is so for Sn(1), but the remaining molecules do not show this trend. However, almost all the acetate groups have one longer and one shorter Sn—O distance. The main exception is with Sn(3), where acetates (31) and (32) have two long bonds and (33) has two short bonds. The average Sn—O distances are shown in Table 3.

These Sn—O bonds are significantly longer and more variable than in the compound most closely comparable in geometry, $Sn(NO_3)_4$, (2·141–2·198, mean 2·163 Å; Garner, Sutton & Wallwork, 1967). Bidentate acetate has a significantly longer bite than nitrate (2·23, compared to 2·14 Å). It will therefore make the molecule more crowded, and we suggest that it is too overcrowded for regular coordination. To relieve this, some of the Sn—O distances become longer. However, because the overcrowding is fairly modest, different patterns of long Sn—O bonds are equally effective at relieving it, and each of the four Sn atoms shows a different arrangement. This correlates precisely with the spectroscopic evidence (Alcock, Tracy & Waddington, 1976*a*,*b*). There are doubled C—O frequencies, but with splitting less than that found for unidentate acetates; these correspond to the distorted bidentate groups. In solution, however, the ^1H NMR spectrum shows only one type of acetate group. It is thus clear that on the NMR time scale the acetate groups are

Table 3. *Average* Sn—O *distances* (Å)

Sn	Shorter Sn—O of each acetate	Longer Sn—O of each acetate	Overall
1	2·186	2·249	2·218
2	2·173	2·229	2·201
3	2·222	2·255	2·238
4	2·169	2·265	2·217
Average	2·188	2·250	2·219

equivalent, as would be expected, because interconversion would involve only slight positional shifts.

The crowding of the molecules can be viewed in an alternative way, considering the non-bonded O···O distances. The normal minimum for this is 2·75 to 2·8 Å, and in the acetate the minimum values for the four molecules are 2·69, 2·63, 2·74 and 2·79 Å, reflecting the overcrowding; in the nitrate, with shorter Sn—O, the minimum O···O is 2·72 Å.

The effect of further crowding can be seen in the structure of the Sn(OAc)$_5^-$ ion (Alcock & Tracy, 1976). For this to be eight-coordinate, it would need three bidentate and two unidentate acetate groups; compared to the tetraacetate, this would correspond to the replacement of one bidentate ligand of bite 2·23 Å with one of bite 2·8 Å (*i.e.* an unconstrained pair of donor atoms). This produces too much overcrowding; the effect is that one atom is extruded, and the coordination drops to seven with two bidentate and three unidentate ligands.

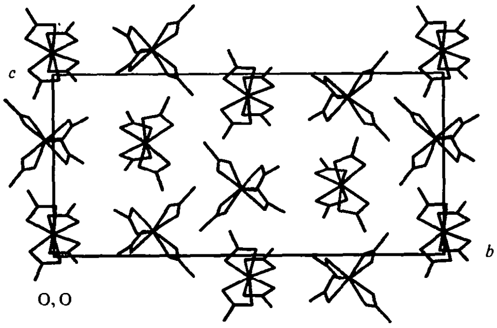

Fig. 2. Packing diagram, viewed down *a*.

Packing

Fig. 2 shows the packing. There are no very close interactions, and it is not possible to see just which interactions are responsible for the subtle difference in geometry between the four molecules, although these can certainly be attributed to packing effects. It also seems probable that it is the ability of the molecules to distort slightly in a variety of ways that causes the complex crystal structure with four different geometries, rather than the higher symmetry and more regular packing that would be usual.

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The Crystal and Molecular Structure of Triphenylgermanium Bromide

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Abstract

$(C_6H_5)_3GeBr$ is monoclinic, space group $P2_1/c$, a = 18·770 (4), b = 9·662 (3), c = 18·467 (4) Å, β = 106·84 (8)°, Z = 8. The structure was refined to R = 0·054 for 2304 counter reflections ($I > 3\sigma$). Eight discrete molecules are contained in the unit cell, comprising asymmetric units of two slightly distorted tetrahedral molecules, with slightly different orientations of the phenyl ligands. The principal geometrical parameters are: Ge—Br = 2·320 (3); Ge—C = 1·935 (10) Å; Br—Ge—C = 106·3 (4); C—Ge—C = 109·9 (4) to 114·5 (4)°.

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