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The Structure of Triphenylgermanium Hydroxide

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Abstract. $C_{18}H_{16}GeO$, $M_r = 320.9$, triclinic, $P\bar{1}$, $a = 15.408\ (6)$, $b = 19.974\ (7)$, $c = 23.264\ (11)$ Å, $\alpha = 107.78\ (4)$, $\beta = 103.54\ (4)$, $\gamma = 101.51\ (3)°$, $V = 6338\ (5)$ Å^3, $Z = 16$, $D_x = 1.34$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 19.1$ cm^{-1}, $F(000) = 2624$, $T = 293$ K, $R = 0.055$ for 6846 observed reflections. The eight independent molecules in the asymmetric unit form two independent O—H···O hydrogen-bonded tetramers with the O atoms in a flattened tetrahedral arrangement [hydrogen-bond distances in the range 2.609 (11) to 2.657 (11) Å]. The Ge atoms are tetrahedrally coordinated with mean Ge—O 1.791 (7) and Ge—C 1.931 (8) Å.

Introduction. The title compound was obtained from the slow hydrolysis of $PhB(OGePh_3)_2$ which had been previously prepared from the reaction of one equivalent of $PhB(OH)_2$ with two equivalents of Ph_3GeBr in ether. Recrystallization from petroleum ether (100–120° C) in aerobic conditions yielded colourless crystals of Ph_3GeOH.

Experimental. A colourless block crystal measuring 0.5 × 0.6 × 0.4 mm was mounted on a glass fibre with its long axis roughly parallel to the φ axis of the goniometer. Cell dimensions and crystal orientation matrix were determined on a CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation, from a least-squares refinement of the setting angles of 25 reflections in the range $30 < 2\theta < 32°$. Intensities of reflections with indices h −19 to 19, k 0 to 25, l −29 to 29 and with $2 < 2\theta < 43°$ measured; ω–2θ scans; ω-scan width $(0.8 + 0.35\tan\theta)°$. Intensities of three reflections were measured at 2 h intervals; these standards remained constant within experimental error throughout data collection. In all, we measured 14 490 reflections, 6846 with $I > 3\sigma(I)$ were labelled observed and used in structure solution and refinement. Space group $P\bar{1}$ deduced from cell reduction and refinement. Data were corrected for Lorentz and polarization effects. The structure was solved by direct methods. All non-H atoms were located from an E map and refined anisotropically. All phenyl groups were refined as planar hexagons (C—C 1.395, C—H 0.95 Å). The hydroxyl H atoms could not be unequivocally located; in the volume element between each pair of O atoms in each tetramer there was clear evidence for electron density between the O atoms – but not distinct well resolved maxima. We concluded that the pattern of four O—H···O hydrogen bonds in each tetramer was probably disordered. The final block-diagonal refinement cycles on F included 1153 variable parameters, $R = 0.055$, $wR = 0.071$, goodness of fit 1.39, $w = 1/[\sigma^2(F_o) + 0.0015(F_o)^2]$. Max. shift/e.s.d. < 0.02; density range in final difference map from −0.49 to 0.53 e Å^{-3} between O atoms. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on a Silicon Graphics 4D-380 computer using the *NRCVAX* suite of programs (Gabe, Le Page, Charland, Lee & White, 1989). Atomic coordinates and selected bond lengths, angles and dihedral angles are given in Tables 1 and 2, respectively.* Fig. 1 is a view of one tetramer prepared using *ORTEP*II (Johnson, 1976).

Discussion. A recent paper describes the structure of Ph_3SiOH at 208 K and reports that the Ge compound is isomorphous but gives no metrical data for

* Full details of molecular dimensions, calculated H-atom coordinates, anisotropic thermal parameters, mean-planes data, selected torsion angles and a list of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54985 (47 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0293]

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Table 1. *Positional and thermal parameters and their e.s.d.'s*

	x	y	z	B_{eq}*
Ge*A*	0.28796 (9)	0.55609 (7)	0.37920 (6)	5.40 (8)
Ge*B*	0.27623 (8)	0.27744 (7)	0.25176 (6)	4.41 (7)
Ge*C*	0.04338 (8)	0.35697 (7)	0.08954 (6)	4.33 (7)
Ge*D*	−0.06364 (9)	0.37119 (8)	0.29751 (6)	5.58 (9)
Ge*E*	0.77599 (8)	0.77987 (7)	0.23371 (6)	4.66 (7)
Ge*F*	0.77179 (9)	1.04706 (7)	0.37068 (6)	5.23 (8)
Ge*G*	0.41413 (8)	0.86165 (7)	0.28845 (6)	5.00 (8)
Ge*J*	0.52071 (8)	0.85023 (7)	0.08200 (6)	4.53 (8)
O*A*	0.2117 (6)	0.4663 (4)	0.3271 (4)	7.8 (6)
O*B*	0.2352 (5)	0.3538 (4)	0.2458 (4)	6.2 (5)
O*C*	0.0818 (5)	0.3529 (4)	0.1667 (3)	6.2 (5)
O*D*	0.0361 (5)	0.3916 (5)	0.2722 (4)	8.3 (7)
O*E*	0.7131 (5)	0.8435 (4)	0.2241 (3)	5.6 (5)
O*F*	0.7009 (5)	0.9584 (4)	0.3134 (4)	7.1 (5)
O*G*	0.5192 (5)	0.8884 (5)	0.2697 (4)	6.8 (6)
O*J*	0.5485 (5)	0.8444 (4)	0.1592 (3)	6.0 (5)
C11*A*	0.3204 (7)	0.5559 (5)	0.4639 (3)	5.9 (8)
C12*A*	0.2506 (5)	0.5288 (5)	0.4868 (4)	8.6 (11)
C13*A*	0.2738 (7)	0.5283 (5)	0.5483 (5)	9.7 (13)
C14*A*	0.3668 (8)	0.5550 (5)	0.5870 (3)	11.5 (14)
C15*A*	0.4366 (5)	0.5821 (5)	0.5641 (4)	11.7 (13)
C16*A*	0.4134 (6)	0.5826 (5)	0.5026 (5)	9.4 (11)
C21*A*	0.2199 (5)	0.6250 (4)	0.3701 (4)	5.3 (7)
C22*A*	0.2014 (6)	0.6697 (5)	0.4221 (3)	8.2 (10)
C23*A*	0.1508 (6)	0.7187 (5)	0.4145 (4)	10.7 (13)
C24*A*	0.1186 (6)	0.7231 (4)	0.3549 (5)	9.4 (12)
C25*A*	0.1370 (6)	0.6784 (5)	0.3029 (4)	9.8 (12)
C26*A*	0.1877 (6)	0.6294 (5)	0.3104 (3)	7.8 (10)
C31*A*	0.3945 (5)	0.5733 (5)	0.3501 (4)	5.8 (8)
C32*A*	0.4501 (6)	0.6456 (4)	0.3704 (4)	8.1 (10)
C33*A*	0.5264 (6)	0.6612 (4)	0.3491 (4)	8.8 (11)
C34*A*	0.5470 (5)	0.6043 (6)	0.3074 (4)	9.8 (12)
C35*A*	0.4914 (7)	0.5320 (5)	0.2870 (4)	11.3 (13)
C36*A*	0.4151 (6)	0.5164 (4)	0.3084 (4)	8.6 (11)
C11*B*	0.2987 (5)	0.2809 (5)	0.3386 (3)	5.7 (8)
C12*B*	0.3147 (6)	0.2201 (4)	0.3519 (4)	8.6 (11)
C13*B*	0.3337 (6)	0.2220 (5)	0.4141 (5)	12.1 (16)
C14*B*	0.3366 (6)	0.2847 (7)	0.4630 (3)	9.1 (12)
C15*B*	0.3206 (7)	0.3454 (5)	0.4496 (4)	11.4 (14)
C16*B*	0.3016 (6)	0.3435 (4)	0.3874 (4)	8.9 (12)
C21*B*	0.1815 (5)	0.1886 (3)	0.1897 (3)	4.0 (6)
C22*B*	0.0888 (6)	0.1782 (4)	0.1886 (4)	7.3 (9)
C23*B*	0.0203 (4)	0.1141 (5)	0.1452 (4)	9.6 (12)
C24*B*	0.0444 (5)	0.0605 (4)	0.1027 (4)	8.5 (10)
C25*B*	0.1371 (6)	0.0709 (4)	0.1037 (3)	8.0 (10)
C26*B*	0.2056 (4)	0.1350 (4)	0.1472 (4)	6.4 (8)
C31*B*	0.3884 (4)	0.2873 (4)	0.2289 (4)	5.3 (8)
C32*B*	0.4601 (6)	0.2630 (4)	0.2567 (4)	7.5 (10)
C33*B*	0.5407 (5)	0.2690 (5)	0.2389 (4)	10.3 (13)
C34*B*	0.5498 (5)	0.2992 (5)	0.1931 (4)	9.8 (11)
C35*B*	0.4781 (7)	0.3235 (5)	0.1653 (4)	11.6 (14)
C36*B*	0.3974 (6)	0.3176 (5)	0.1831 (4)	8.7 (11)
C11*C*	−0.0117 (5)	0.4368 (3)	0.0976 (4)	4.4 (7)
C12*C*	−0.0144 (5)	0.4796 (4)	0.1567 (3)	5.9 (8)
C13*C*	−0.0548 (6)	0.5369 (4)	0.1618 (3)	7.4 (9)
C14*C*	−0.0924 (5)	0.5514 (3)	0.1078 (4)	6.6 (9)
C15*C*	−0.0897 (5)	0.5086 (4)	0.0487 (3)	6.1 (9)
C16*C*	−0.0494 (5)	0.4513 (4)	0.0436 (3)	5.8 (8)
C21*C*	0.1547 (5)	0.3737 (4)	0.0653 (4)	4.6 (7)
C22*C*	0.2384 (6)	0.4223 (4)	0.1101 (3)	6.9 (9)
C23*C*	0.3183 (5)	0.4363 (4)	0.0920 (4)	8.9 (11)
C24*C*	0.3145 (5)	0.4016 (5)	0.0292 (5)	8.8 (12)
C25*C*	0.2307 (7)	0.3530 (5)	−0.0156 (3)	9.1 (12)
C26*C*	0.1509 (5)	0.3390 (4)	0.0024 (3)	6.5 (9)
C31*C*	−0.0469 (5)	0.2634 (3)	0.0326 (3)	5.3 (8)
C32*C*	−0.1367 (6)	0.2582 (4)	−0.0020 (4)	6.0 (8)
C33*C*	−0.1999 (4)	0.1894 (5)	−0.0406 (3)	7.2 (8)
C34*C*	−0.1732 (6)	0.1258 (3)	−0.0446 (4)	8.0 (10)
C35*C*	−0.0834 (6)	0.1310 (4)	−0.0100 (4)	10.6 (11)
C36*C*	−0.0202 (4)	0.1998 (5)	0.0286 (4)	8.2 (9)
C11*D*	−0.1445 (5)	0.2776 (4)	0.2374 (4)	6.0 (8)
C12*D*	−0.1506 (6)	0.2564 (5)	0.1733 (4)	9.1 (10)
C13*D*	−0.2105 (7)	0.1883 (5)	0.1298 (3)	10.8 (12)
C14*D*	−0.2645 (6)	0.1415 (4)	0.1504 (4)	9.2 (11)
C15*D*	−0.2584 (7)	0.1627 (5)	0.2145 (5)	14.0 (15)
C16*D*	−0.1984 (7)	0.2307 (5)	0.2580 (3)	11.7 (12)
C21*D*	−0.0186 (6)	0.3677 (5)	0.3810 (3)	6.1 (8)
C22*D*	0.0523 (7)	0.3356 (6)	0.3948 (4)	12.3 (16)
C23*D*	0.0833 (6)	0.3323 (6)	0.4549 (5)	13.5 (18)
C24*D*	0.0434 (7)	0.3611 (6)	0.5013 (3)	9.5 (12)
C25*D*	−0.0275 (7)	0.3932 (6)	0.4876 (4)	13.2 (17)
C26*D*	−0.0584 (6)	0.3965 (5)	0.4274 (4)	11.2 (14)

* B_{eq} is the mean of the principal axes of the thermal ellipsoid.

Table 1 (*cont.*)

	x	y	z	B_{eq}*
C31*D*	−0.1217 (6)	0.4492 (4)	0.2996 (4)	5.5 (8)
C32*D*	−0.2184 (6)	0.4338 (4)	0.2854 (4)	8.1 (10)
C33*D*	−0.2597 (5)	0.4905 (6)	0.2903 (4)	10.9 (13)
C34*D*	−0.2043 (8)	0.5628 (5)	0.3094 (4)	10.5 (13)
C35*D*	−0.1076 (7)	0.5782 (4)	0.3237 (4)	8.8 (11)
C36*D*	−0.0664 (4)	0.5214 (5)	0.3188 (4)	7.2 (9)
C11*E*	0.6942 (5)	0.6822 (3)	0.1805 (3)	5.2 (7)
C12*E*	0.6049 (6)	0.6721 (4)	0.1415 (4)	10.1 (11)
C13*E*	0.5471 (5)	0.6012 (5)	0.1032 (4)	10.7 (12)
C14*E*	0.5785 (6)	0.5405 (4)	0.1039 (4)	8.7 (10)
C15*E*	0.6677 (7)	0.5507 (4)	0.1429 (4)	9.0 (11)
C16*E*	0.7255 (5)	0.6215 (5)	0.1812 (4)	7.8 (10)
C21*E*	0.8088 (7)	0.7908 (5)	0.3216 (3)	6.3 (8)
C22*E*	0.7389 (5)	0.7838 (6)	0.3500 (5)	14.8 (16)
C23*E*	0.7610 (8)	0.7870 (7)	0.4126 (5)	21.0 (20)
C24*E*	0.8531 (9)	0.7972 (6)	0.4469 (3)	13.1 (15)
C25*E*	0.9231 (7)	0.8042 (6)	0.4185 (5)	16.6 (19)
C26*E*	0.9009 (6)	0.8010 (6)	0.3558 (5)	14.7 (18)
C31*E*	0.8849 (5)	0.7994 (6)	0.2053 (5)	6.8 (9)
C32*E*	0.9488 (7)	0.7591 (6)	0.2095 (6)	14.3 (19)
C33*E*	1.0253 (7)	0.7734 (7)	0.1880 (6)	17.7 (24)
C34*E*	1.0378 (6)	0.8279 (7)	0.1623 (5)	12.2 (16)
C35*E*	0.9738 (8)	0.8682 (6)	0.1582 (6)	18.7 (26)
C36*E*	0.8974 (7)	0.8539 (6)	0.1796 (6)	17.6 (25)
C11*F*	0.8048 (6)	1.0408 (5)	0.4538 (3)	6.1 (8)
C12*F*	0.8608 (7)	0.9962 (5)	0.4648 (4)	10.8 (13)
C13*F*	0.8890 (7)	0.9921 (5)	0.5248 (5)	13.1 (17)
C14*F*	0.8612 (7)	1.0326 (6)	0.5739 (4)	11.5 (14)
C15*F*	0.8051 (8)	1.0771 (6)	0.5630 (4)	12.9 (16)
C16*F*	0.7769 (6)	1.0812 (5)	0.5029 (5)	9.7 (12)
C21*F*	0.8794 (5)	1.0693 (5)	0.3442 (4)	5.1 (7)
C22*F*	0.8728 (6)	1.0342 (6)	0.2808 (4)	21.0 (21)
C23*F*	0.9499 (9)	1.0497 (8)	0.2603 (4)	37.2 (36)
C24*F*	1.0337 (6)	1.1004 (7)	0.3032 (6)	12.1 (15)
C25*F*	1.0404 (5)	1.1356 (6)	0.3666 (5)	12.5 (14)
C26*F*	0.9632 (7)	1.1200 (6)	0.3871 (3)	14.6 (16)
C31*F*	0.7004 (5)	1.1159 (4)	0.3709 (3)	5.3 (7)
C32*F*	0.7456 (4)	1.1913 (5)	0.3977 (4)	7.2 (9)
C33*F*	0.6935 (7)	1.2413 (3)	0.4020 (4)	9.1 (11)
C34*F*	0.5961 (7)	1.2158 (5)	0.3795 (4)	9.0 (11)
C35*F*	0.5510 (4)	1.1404 (5)	0.3527 (4)	7.8 (10)
C36*F*	0.6031 (6)	1.0905 (3)	0.3484 (3)	6.6 (9)
C11*G*	0.3475 (6)	0.9310 (4)	0.2786 (5)	5.9 (8)
C12*G*	0.3013 (8)	0.9570 (6)	0.3217 (4)	17.3 (24)
C13*G*	0.2489 (8)	1.0046 (6)	0.3127 (5)	19.3 (28)
C14*G*	0.2426 (7)	1.0261 (5)	0.2605 (6)	11.7 (16)
C15*G*	0.2888 (7)	1.0000 (5)	0.2174 (4)	9.2 (11)
C16*G*	0.3412 (6)	0.9525 (5)	0.2264 (4)	8.8 (11)
C21*G*	0.3425 (6)	0.7627 (4)	0.2290 (3)	5.6 (8)
C22*G*	0.2525 (6)	0.7483 (4)	0.1890 (4)	8.6 (9)
C23*G*	0.2015 (5)	0.6765 (5)	0.1482 (4)	9.5 (10)
C24*G*	0.2406 (7)	0.6190 (4)	0.1474 (4)	9.5 (12)
C25*G*	0.3306 (7)	0.6334 (4)	0.1874 (5)	10.9 (13)
C26*G*	0.3815 (5)	0.7052 (5)	0.2283 (4)	8.7 (10)
C31*G*	0.4549 (6)	0.8661 (5)	0.3744 (3)	5.8 (8)
C32*G*	0.3933 (5)	0.8293 (4)	0.3978 (4)	7.2 (9)
C33*G*	0.4205 (6)	0.8367 (5)	0.4616 (4)	8.1 (10)
C34*G*	0.5095 (7)	0.8808 (6)	0.5019 (3)	10.4 (12)
C35*G*	0.5711 (5)	0.9176 (6)	0.4785 (4)	16.7 (17)
C36*G*	0.5439 (6)	0.9102 (6)	0.4148 (4)	15.2 (16)
C11*J*	0.6343 (4)	0.8611 (5)	0.0587 (4)	4.7 (7)
C12*J*	0.6458 (5)	0.8052 (4)	0.0104 (4)	8.1 (10)
C13*J*	0.7277 (6)	0.8160 (5)	−0.0058 (4)	9.6 (12)
C14*J*	0.7983 (5)	0.8826 (6)	0.0264 (4)	9.0 (11)
C15*J*	0.7869 (5)	0.9384 (4)	0.0748 (4)	10.1 (11)
C16*J*	0.7049 (6)	0.9277 (4)	0.0909 (3)	7.2 (9)
C21*J*	0.4811 (5)	0.9378 (4)	0.0930 (4)	4.6 (7)
C22*J*	0.5103 (5)	0.9956 (5)	0.1518 (3)	8.6 (10)
C23*J*	0.4848 (6)	1.0598 (4)	0.1567 (4)	10.4 (12)
C24*J*	0.4302 (6)	1.0663 (4)	0.1028 (5)	8.1 (11)
C25*J*	0.4010 (6)	1.0085 (6)	0.0440 (4)	10.4 (12)
C26*J*	0.4265 (6)	0.9443 (4)	0.0391 (3)	9.5 (11)
C31*J*	0.4251 (4)	0.7601 (3)	0.0242 (3)	4.9 (7)
C32*J*	0.3897 (5)	0.7453 (4)	−0.0408 (4)	7.3 (9)
C33*J*	0.3211 (5)	0.6794 (5)	−0.0804 (3)	9.2 (10)
C34*J*	0.2880 (5)	0.6283 (4)	−0.0551 (4)	8.7 (10)
C35*J*	0.3234 (5)	0.6432 (4)	0.0098 (4)	7.3 (9)
C36*J*	0.3920 (5)	0.7090 (5)	0.0495 (3)	6.5 (9)

the Ph_3GeOH molecules (Puff, Braun & Reuter, 1991). Ph_3GeOH crystallizes with eight independent molecules in the asymmetric unit; these are arranged in two similar independent hydrogen-bonded tetra-

Table 2. *Selected bond lengths* (Å) *and angles* (°)

	y = *A*	*B*	*C*	*D*	*E*	*F*	*G*	*J*
Ge—O*y*	1.798 (8)	1.794 (7)	1.787 (7)	1.788 (7)	1.780 (7)	1.791 (7)	1.796 (7)	1.792 (7)
Ge—C11*y*	1.918 (8)	1.945 (8)	1.932 (8)	1.914 (6)	1.934 (6)	1.931 (9)	1.918 (10)	1.942 (8)
Ge—C21*y*	1.921 (9)	1.944 (5)	1.926 (8)	1.932 (8)	1.919 (7)	1.918 (9)	1.945 (6)	1.930 (8)
Ge—C31*y*	1.925 (9)	1.915 (8)	1.942 (6)	1.940 (10)	1.955 (10)	1.925 (9)	1.920 (7)	1.938 (5)
	y = *A*	*B*	*C*	*D*	*E*	*F*	*G*	*J*
O*y*—Ge—C11*y*	107.4 (4)	108.7 (4)	107.5 (4)	107.2 (4)	107.6 (3)	108.7 (4)	106.2 (4)	107.5 (4)
O*y*—Ge—C21*y*	106.7 (3)	107.1 (3)	104.7 (4)	106.8 (4)	107.8 (4)	105.0 (4)	108.5 (4)	104.9 (4)
O*y*—Ge—C31*y*	107.1 (4)	106.7 (4)	108.3 (3)	107.5 (4)	108.7 (4)	108.7 (3)	105.3 (4)	107.1 (4)
C11*y*—Ge—C21*y*	112.4 (4)	112.9 (3)	112.9 (4)	111.2 (3)	110.3 (4)	111.7 (4)	110.0 (3)	110.7 (4)
C11*y*—Ge—C31*y*	112.9 (4)	111.8 (4)	111.5 (3)	112.1 (3)	109.5 (4)	108.9 (4)	113.4 (4)	111.7 (3)
C21*y*—Ge—C31*y*	109.9 (4)	109.3 (3)	115.5 (3)	111.7 (4)	112.8 (4)	113.6 (4)	112.0 (4)	114.4 (3)

Hydrogen-bond contacts (Å)

O*A*···O*B*	2.609 (11)	O*A*···O*D*	2.604 (12)	O*B*···O*C*	2.626 (10)	O*C*···O*D*	2.642 (11)
O*E*···O*F*	2.657 (11)	O*E*···O*J*	2.636 (10)	O*F*···O*G*	2.657 (11)	O*G*···O*J*	2.628 (10)

O···O distances across the tetramer (Å)

O*A*···O*C*	3.548 (11)	O*B*···O*D*	3.446 (11)	O*E*···O*G*	3.580 (10)	O*F*···O*J*	3.536 (10)

Hydrogen-bond angles around the tetramer (°)

O*A*···O*B*···O*C*	85.3 (3)	O*B*···O*C*···O*D*	81.7 (3)	O*B*···O*A*···O*D*	82.8 (3)	O*A*···O*D*···O*C*	85.1 (3)
O*F*···O*E*···O*J*	83.8 (3)	O*E*···O*F*···O*G*	84.7 (3)	O*F*···O*G*···O*J*	84.0 (3)	O*E*···O*J*···O*G*	85.7 (3)

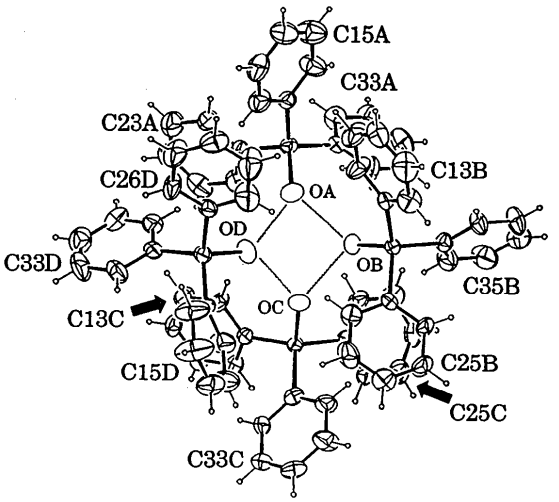

Fig. 1. A view of one of the Ph_3GeOH tetramers showing the numbering scheme; the other tetramer is similarly numbered with rings labelled *E*, *F*, *G* and *J*. The thermal ellipsoids shown for the Ge, O and C atoms are at the 25% probability level.

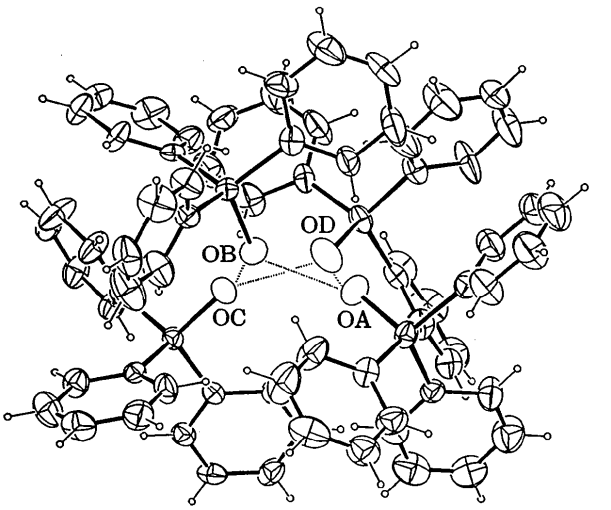

Fig. 2. A view of one of the Ph_3GeOH tetramers showing the flattened tetrahedral arrangement of the O atoms.

mers, one of which is shown in Fig. 1. The arrangement of phenyl rings in the Ph_3GeOH moieties corresponds to distorted propeller conformations, with the dihedral angles between the O—Ge—C and Ge—phenyl planes being in the range 1.1 (4) to 70.3 (4)°. The arrangement of the O atoms in these tetramers is best described as a flattened tetrahedron as depicted in Fig. 2. The angle between the planes through O*A*, O*B*, O*C* and O*A*, O*D*, O*C* is 126.5 (5)° [130.5 (4)° for the corresponding interplanar angle between O*E*, O*F*, O*G* and O*E*, O*J*, O*G*].

The Ge—O bond lengths in Ph_3GeOH are in the range 1.780 (7) to 1.798 (8) Å with a mean value of 1.791 (8) Å. Published Ge—O bond lengths span the range 1.718 (3) Å in $Ph_3GeOSiPh_3$ (Morosin & Harrah, 1981) to 1.86 (1) Å in $Ph_3GeOCOCF_3$ (Glidewell & Liles, 1983). The oxide derived from Ph_3GeOH (*i.e.* $Ph_3GeOGePh_3$) has a mean Ge—O bond length of 1.767 (2) Å (Glidewell & Liles, 1978*a*); essentially the same value [1.766 (4) Å] was found in $H_3GeOGeH_3$ determined by electron diffraction (Glidewell, Rankin, Robiette, Sheldrick, Beagley & Cradock, 1970).

The Ge—C distances in Ph_3GeOH are in the range 1.914 (6) to 1.955 (10) Å, mean value 1.931 (20) Å. For comparison, the Ge—C bond lengths in $Ph_3GeOGePh_3$ range from 1.934 (5) to 1.951 (5) Å with a mean value 1.942 (3) Å; in Ph_4Ge the single value of 1.957 (4) Å is reported (Karipides & Haller, 1972).

The structural determination of Ph_3GeOH is part of a series for the Group 14 Ph_3MOH molecules (M = C, Si, Ge, Sn, Pb). Crystals of Ph_3COH (Ferguson, Gallagher, Glidewell, Low & Scrimgeour, 1992) are trigonal, $R\bar{3}$, with 1.33 molecules in the asymmetric unit and consist of hydrogen-bonded pyramidal tetramers with one molecule on a three-fold axis and the other three lying around it. The Ph_3SiOH structure is isomorphous with Ph_3GeOH. Both Ph_3SnOH and Ph_3PbOH have structures consisting of zigzag chains of planar Ph_3M (M = Sn, Pb) groups joined by OH groups giving trigonal bipyramidal geometry at M (Glidewell & Liles, 1978*b*).

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Structure of *catena*-Poly[{(2,2′-bipyridyl)(diperchlorato)copper(II)}-μ-4,4′-bipyridyl]

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Abstract. $[Cu(C_{10}H_8N_2)(C_{10}H_8N_2)(ClO_4)_2]$, M_r = 574.5, orthorhombic, *Pbcn*, a = 12.413 (3), b = 14.645 (3), c = 12.287 (2) Å, V = 2233.6 (8) Å^3, Z = 4, D_x = 1.708 g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, μ = 13.12 cm^{-1}, $F(000)$ = 1164, room temperature, R = 0.050 and wR = 0.049 for 1498 observed reflections. The coordination around Cu^{II} is an elongated distorted octahedron. Two N atoms of 2,2′-bipyridyl and two N atoms from two 4,4′-bipyridyls form the equatorial coordination plane, and two perchlorate ions occupy the axial sites. The 4,4′-bipyridyl ligand bridges neighbouring Cu^{II} atoms to form polymeric chains along the c axis in the crystal. The rings of 4,4′-bipyridyl are coplanar and make a dihedral angle of 114° with the equatorial coordination plane; the 4,4′-bipyridyl may provide a pathway for magnetic superexchange interaction between the adjacent Cu^{II} atoms.

Introduction. In the last decade, a number of binuclear transition-metal complexes bridged by heterocyclic aromatic diamines have been investigated as models of metalloenzymes (Matsumoto, Ooi, Nakao, Mori & Nakahara, 1981; Richardson & Hatfield, 1975). In some imidazolate or pyrazine-bridged complexes, spin–spin exchange interactions, propagated by the bridging ligand between the adjacent metal ions, have been observed, and studies on the molecular and crystal structures of those complexes have shown the relationship between structure and magnetic property (Coronado, Drillon & Beltran, 1984; Xu, Chen, Xu, Cheng, Chen & Tang, 1991). Recently, a series of novel Cu^{II} complexes bridged by 4,4′-bipyridyl ligands have been synthesized, and studies on EPR spectra showed evidence of superexchange interaction propagated by the 4,4′-bipyridyl ligand in the title complex, but no interaction in the others (Chen, 1989). In order to investigate the structural reason for the differences in the magnetic properties, the crystal structures of these complexes have been determined by means of X-ray analysis.

Experimental. 2,2′-Bipyridyl (2 mmol) and 4,4′-bipyridyl (2 mmol) were dissolved in ethanol, and then the solution was mixed with an aqueous solu-

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