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Structures of [1,4-Bis(diphenylphosphino)butane]tetracarbonylchromium(0) (1) and [1,4-Bis(diphenylphosphino)butane]tetracarbonyltungsten(0) (2)

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Abstract. (1): $C_{32}H_{28}CrO_4P_2$, $M_r = 590.3$, monoclinic, $P2_1/n$, $a = 11.996$ (5), $b = 15.298$ (6), $c = 16.508$ (9) Å, $\beta = 105.40$ (3)°, $V = 2921$ (1) Å^3, $Z = 4$, $D_x = 1.34$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, μ(Mo $K\alpha$) = 0.52 mm^{-1}, $F(000) = 1224$, $T = 298$ K, final $R = 0.038$ for 3007 observed reflections. (2): $C_{32}H_{28}O_4P_2W$, $M_r = 722.4$, monoclinic, $P2_1/n$, $a = 12.055$ (4), $b = 15.352$ (5), $c = 16.544$ (4) Å, $\beta = 104.68$ (2)°, $V = 2962$ (2) Å^3, $Z = 4$, $D_x = 1.62$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, μ(Mo $K\alpha$) = 4.12 mm^{-1}, $F(000) = 1424$, $T = 298$ K, final $R = 0.028$ for 4394 observed reflections. The molecular structures of both title compounds, which have a distorted octahedron with a seven-membered chelating ring, are similar except for the difference of the M—CO and M—P bond lengths. The three homologues $M(CO)_4[Ph_2P(CH_2)_4PPh_2)]$, M = Cr, Mo and W, have similar molecular structures, and are isomorphous with each other.

Introduction. Crystal structures of [bis(diphenylphosphino)propane]tetracarbonylmolybdenum(0), $Mo(CO)_4(dppp)$, and [bis(diphenylphosphino)butane]-tetracarbonylmolybdenum(0), $Mo(CO)_4(dppb)$, have been published recently and were studied to investigate the ring effect on the structure of bidentate diphosphine derivatives of molybdenum(0) carbonyl (Ueng & Hwang, 1991*a*). In order to obtain further information about the structures of the diphosphine derivatives of VI*B* metal carbonyls containing a seven-membered chelating ring and to continue further structural study on the derivatives of the metal carbonyls (Ueng & Hwang, 1991*b*; Ueng & Lee, 1991; Ueng & Leu, 1991), the structure determinations of the title compounds were undertaken.

Experimental. The title compounds were prepared by refluxing stoichiometric amounts of $M(CO)_6$ [M = Cr for (1), W for (2)] and $Ph_2P(CH_2)_4PPh_2$ in purified *n*-decane under nitrogen for *ca* 6 h. The reaction medium for the thermolytic substitution reaction is *n*-decane instead of ethyldiglycol, dimethylglycol or diglyme that have been previously reported (Dietsche, 1966; Grim, Briggs, Barth, Tolman & Jesson, 1974). (1): $Cr(CO)_4[Ph_2P(CH_2)_4PPh_2]$, crystal 0.20 × 0.40 × 0.50 mm. CAD-4 diffractometer. Unit cell: 24 reflections, 2θ range 18.44 to 25.78°. $\theta/2\theta$ scan with scan parameter: $(0.70 + 0.35\tan\theta)$°, $2\theta_{\max} = 49.8$°, ranges of h, k, l: −14 to 13, 0 to 18, 0 to 19, respectively. Three standard reflections monitored every 2 h: variation on $I < 3$%. 5117 unique reflections ($R_{\rm int} = 0.012$), 3007 observed with $I > 2.0\sigma(I)$. Absorption correction was made with experimental ψ rotation (minimum and maximum transmission factors: 0.9002 and 0.9996). Structure solved by heavy-atom method. H atoms of phenyl groups calculated after isotropic refinement, other H atoms found in difference Fourier map. The final least-squares cycle was calculated with 67 atoms, 353 parameters and 3007 reflections, non-H atoms anisotropic and H atoms isotropic, quantity minimized $\sum w(KF_o - F_c)^2$, with $R(F) = 0.038$, $wR = 0.028$, $S = 1.57$. Weighting scheme from counting statistics. $(\Delta/\sigma)_{\max} = 0.044$. Peaks in final ΔF map 0.220 to −0.360 e Å^{-3}. Secondary-extinction coefficient 0.28 (2) (length in μm). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computing programs: NRCC *SDP* VAX package (Gabe & Lee, 1981); *ORTEP* from Enraf–Nonius (1979) *Structure Determination Package*. (2): $W(CO)_4[Ph_2P(CH_2)_4PPh_2]$, crystal 0.50 × 0.60 × 0.65 mm. CAD-4 diffractometer. Unit cell: 25 reflections, 2θ range 21.00 to 24.66°. $\theta/2\theta$ scan with scan parameter: $(0.75 + 0.35\tan\theta)$,° $2\theta_{\max} = 49.8$°, ranges of h, k, l: −14 to 13, 0 to 18, 0 to 19, respectively. Three standard reflections monitored every 2 h: variation on $I < 2$%. 5206 unique reflections ($R_{\rm int} = 0.008$), 4394 observed with $I > 2.0\sigma(I)$. Absorption correction was made with experimental ψ rotation (minimum and maximum transmission factors: 0.7840 and 0.9903). Structure solved by heavy-atom method. H atoms of phenyl groups calculated after isotropic refinement, other H atoms found in difference Fourier map. The final least-squares cycle was calculated with 67 atoms, 353 parameters and 4394 reflections, quantity minimized

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$\sum w(KF_o - F_c)^2$, with $R(F) = 0.028$, $wR = 0.028$, $S = 3.54$. Weighting scheme from counting statistics. $(\Delta/\sigma)_{\max} = 0.008$. Peaks in final ΔF map 0.760 to -0.890 e Å^{-3}. Secondary-extinction coefficient 6.3 (2) (length in μm). Other details as in (1).

Discussion. Atomic positional parameters and equivalent isotropic temperature factors and selected bond lengths and angles for the title compounds are listed in Tables 1 and 2 respectively.* The two structures are isomorphous with each other and with the Mo complex (Ueng & Hwang, 1991*a*). The molecular structures are shown in Fig. 1.

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors, with estimated standard deviations in parentheses*

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq} (Å^2)
Cr(CO)$_4$(dppb)				
Cr	0.57799 (6)	0.23300 (4)	0.88075 (4)	2.61 (3)
P(1)	0.44386 (9)	0.22878 (7)	0.96692 (6)	2.59 (5)
P(2)	0.7186 (1)	0.31333 (7)	0.98225 (7)	2.68 (5)
C(1)	0.6562 (4)	0.1271 (3)	0.9213 (3)	3.3 (2)
C(2)	0.4980 (4)	0.3342 (3)	0.8345 (3)	3.4 (2)
C(3)	0.4784 (4)	0.1665 (3)	0.7994 (3)	3.4 (2)
C(4)	0.6694 (4)	0.2373 (3)	0.8070 (2)	3.6 (2)
C(5)	0.3936 (4)	0.3354 (3)	0.9971 (2)	3.1 (2)
C(6)	0.4691 (4)	0.3821 (2)	1.0738 (3)	3.0 (2)
C(7)	0.5879 (4)	0.4133 (3)	1.0684 (3)	3.4 (2)
C(8)	0.6828 (4)	0.3427 (3)	1.0803 (2)	2.8 (2)
O(1)	0.7044 (3)	0.0629 (2)	0.9385 (2)	5.4 (2)
O(2)	0.4466 (3)	0.3947 (2)	0.8044 (2)	5.4 (2)
O(3)	0.4214 (3)	0.1259 (2)	0.7455 (2)	4.8 (2)
O(4)	0.7233 (3)	0.2360 (2)	0.7585 (2)	5.5 (2)
C(1*A*)	0.3063 (3)	0.1818 (3)	0.9057 (2)	2.9 (2)
C(2*A*)	0.2305 (4)	0.2318 (3)	0.8437 (3)	3.6 (2)
C(3*A*)	0.1315 (4)	0.1942 (3)	0.7937 (3)	4.3 (3)
C(4*A*)	0.1054 (4)	0.1076 (3)	0.8031 (3)	4.4 (3)
C(5*A*)	0.1798 (4)	0.0585 (3)	0.8633 (3)	4.5 (3)
C(6*A*)	0.2800 (4)	0.0954 (3)	0.9148 (3)	3.7 (2)
C(1*B*)	0.4668 (3)	0.1687 (2)	1.0668 (2)	2.7 (3)
C(2*B*)	0.3824 (4)	0.1709 (3)	1.1107 (3)	3.6 (2)
C(3*B*)	0.4000 (4)	0.1280 (3)	1.1868 (3)	4.2 (3)
C(4*B*)	0.4993 (5)	0.0814 (3)	1.2195 (3)	4.7 (3)
C(5*B*)	0.5838 (4)	0.0786 (3)	1.1768 (3)	4.6 (3)
C(6*B*)	0.5674 (4)	0.1220 (3)	1.1005 (3)	3.4 (2)
C(1*C*)	0.8551 (3)	0.2531 (3)	1.0177 (2)	2.8 (2)
C(2*C*)	0.8841 (4)	0.2060 (3)	1.0924 (3)	3.5 (2)
C(3*C*)	0.9829 (4)	0.1543 (3)	1.1126 (3)	4.2 (3)
C(4*C*)	1.0522 (4)	0.1489 (3)	1.0590 (3)	4.4 (3)
C(5*C*)	1.0251 (4)	0.1961 (3)	0.9860 (3)	4.9 (3)
C(6*C*)	0.9278 (4)	0.2480 (3)	0.9648 (3)	3.8 (2)
C(1*D*)	0.7702 (4)	0.4191 (3)	0.9526 (3)	3.3 (2)
C(2*D*)	0.8351 (4)	0.4749 (3)	1.0131 (3)	4.1 (2)
C(3*D*)	0.8736 (4)	0.5540 (3)	0.9909 (3)	5.1 (3)
C(4*D*)	0.8496 (4)	0.5791 (3)	0.9086 (3)	5.2 (3)
C(5*D*)	0.7861 (5)	0.5245 (3)	0.8469 (3)	5.1 (3)
C(6*D*)	0.7458 (4)	0.4452 (3)	0.8687 (3)	4.2 (3)
W(CO)$_4$(dppb)				
W	0.42319 (2)	0.23207 (2)	0.12588 (1)	2.50 (1)
P(1)	0.5621 (1)	0.2271 (1)	0.03399 (8)	2.67 (6)
P(2)	0.2795 (1)	0.3155 (1)	0.01790 (9)	2.75 (7)
C(1)	0.3369 (6)	0.1221 (4)	0.0839 (4)	3.6 (3)
C(2)	0.5074 (6)	0.3400 (4)	0.1737 (3)	3.9 (3)
C(3)	0.5303 (5)	0.1609 (4)	0.2109 (4)	3.2 (3)
C(4)	0.3283 (5)	0.2380 (4)	0.2058 (3)	3.5 (3)
C(5)	0.6112 (5)	0.3326 (3)	0.0014 (3)	3.1 (3)
C(6)	0.5338 (5)	0.3800 (4)	−0.0718 (3)	3.2 (3)
C(7)	0.4183 (5)	0.4115 (4)	−0.0628 (4)	3.6 (3)
C(8)	0.3217 (5)	0.3420 (4)	−0.0774 (3)	3.0 (3)
O(1)	0.2834 (4)	0.0592 (3)	0.0651 (3)	5.7 (3)
O(2)	0.5580 (5)	0.4021 (3)	0.1991 (3)	6.5 (3)
O(3)	0.5906 (4)	0.1200 (3)	0.2620 (3)	4.9 (2)
O(4)	0.2743 (4)	0.2393 (3)	0.2544 (3)	5.9 (3)
C(1*A*)	0.6989 (5)	0.1823 (4)	0.0947 (3)	2.8 (3)
C(2*A*)	0.7691 (5)	0.2300 (4)	0.1560 (3)	3.7 (3)
C(3*A*)	0.8685 (6)	0.1940 (5)	0.2061 (4)	4.7 (4)
C(4*A*)	0.8947 (6)	0.1096 (5)	0.1966 (4)	4.9 (4)
C(5*A*)	0.8259 (6)	0.0605 (4)	0.1357 (4)	4.9 (4)
C(6*A*)	0.7269 (5)	0.0958 (4)	0.0851 (4)	3.8 (3)
C(1*B*)	0.5369 (5)	0.1671 (3)	−0.0644 (3)	2.6 (3)
C(2*B*)	0.6156 (5)	0.1695 (4)	−0.1113 (4)	3.6 (3)
C(3*B*)	0.5952 (6)	0.1253 (4)	−0.1871 (4)	4.5 (4)
C(4*B*)	0.4961 (7)	0.0793 (4)	−0.2163 (4)	4.9 (4)
C(5*B*)	0.4161 (6)	0.0759 (4)	−0.1711 (4)	5.0 (4)
C(6*B*)	0.4376 (6)	0.1209 (4)	−0.0947 (4)	3.9 (3)
C(1*C*)	0.1460 (4)	0.2558 (3)	−0.0174 (3)	2.8 (3)
C(2*C*)	0.1175 (5)	0.2088 (4)	−0.0913 (4)	3.8 (3)
C(3*C*)	0.0208 (6)	0.1575 (4)	−0.1108 (4)	4.3 (3)
C(4*C*)	−0.0491 (6)	0.1517 (4)	−0.0578 (4)	4.8 (4)
C(5*C*)	−0.0249 (6)	0.1990 (5)	0.0144 (4)	4.9 (4)
C(6*C*)	0.0731 (5)	0.2493 (4)	0.0352 (4)	3.9 (3)
C(1*D*)	0.2272 (5)	0.4210 (4)	0.0455 (4)	3.3 (3)
C(2*D*)	0.1642 (5)	0.4762 (4)	−0.0150 (4)	4.1 (3)
C(3*D*)	0.1247 (6)	0.5541 (4)	0.0065 (5)	5.1 (4)
C(4*D*)	0.1464 (6)	0.5805 (4)	0.0878 (5)	5.2 (4)
C(5*D*)	0.2072 (7)	0.5274 (4)	0.1490 (4)	5.1 (4)
C(6*D*)	0.2479 (6)	0.4475 (4)	0.1289 (4)	4.5 (3)

Table 2. *Selected bond lengths* (Å) *and angles* (°) *for* M(CO)$_4$(dppb) [M = Cr (1), W (2), Mo (3)]

	(1)	(2)	(3)*
M—P	2.415 (2)	2.533 (2)	2.550 (1)
	2.379 (2)	2.503 (2)	2.515 (1)
M—C	1.843 (4)	1.957 (6)	1.976 (4)
	1.847 (4)	1.980 (6)	1.982 (4)
	1.875 (4)	1.998 (7)	2.009 (4)
	1.903 (4)	2.012 (7)	2.041 (4)
*M*C—O	1.156 (5)	1.156 (8)	1.153 (5)
	1.151 (5)	1.152 (7)	1.141 (5)
	1.149 (5)	1.154 (8)	1.146 (5)
	1.136 (5)	1.160 (8)	1.139 (5)
P—C	1.853 (4)	1.851 (6)	1.852 (4)
	1.838 (4)	1.822 (5)	1.830 (4)
P—C(phenyl)	1.837 (4)	1.833 (6)	1.840 (4)
	1.844 (4)	1.827 (5)	1.834 (4)
	1.845 (4)	1.838 (6)	1.844 (4)
	1.835 (4)	1.815 (6)	1.825 (4)
P—*M*—P	93.29 (5)	91.75 (5)	91.65 (4)
cis-C—*M*—C	85.6 (2)	86.2 (2)	86.1 (2)
trans-C—*M*—C	176.6 (2)	176.9 (2)	176.8 (2)

* Ueng & Hwang (1991*a*).

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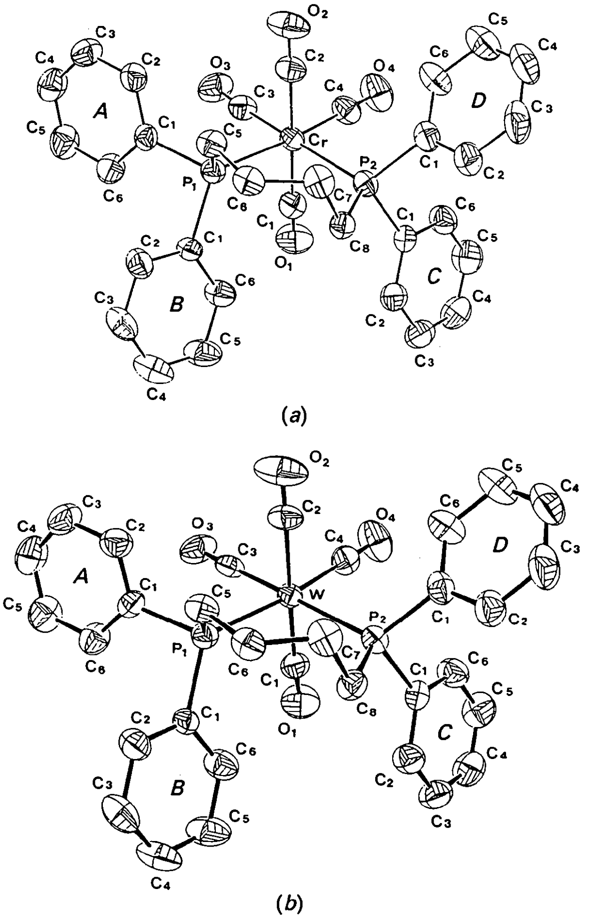
* Lists of structure factors, anisotropic thermal parameters and H-atom parameters, complete bond lengths and angles, and dihedral angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54779 (49 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0545]

The coordination around the metal is a distorted octahedron. The metal atom is bonded to four carbonyls and a bidentate ligand having two P atoms as donors for both compounds, the equatorial plane being formed by the two *cis* CO groups and two P atoms. There is a pseudo twofold axis through *M* and the middle point of the C(6)—C(7) bond for both title compounds. The deviations of the metal atom from this equatorial plane are −0.006 (2) and −0.009 (3) Å for compounds (1) and (2), respectively. Distortion of compound (1) and compound (2) is also revealed by the dihedral angles among the three mutually perpendicular planes of the octahedron [88.7 (1), 95.5 (1) and 89.58 (7)° for (1); 84.2 (2), 92.1 (2) and 90.6 (1)° for (2)]. The dihedral angles between the equatorial least-squares plane and the phenyl groups are comparable [79.9 (1) (*A*), 66.0 (1) (*B*), 70.2 (1) (*C*) and 51.2 (1)° (*D*) for (1); 77.7 (2), 66.0 (2), 69.8 (2) and 50.3 (2)° for (2)]. The four C atoms of the chelating rings for both compounds (1) and (2) are located at the same side of the least-squares equatorial plane with distances from the plane being 1.436 (5) [C(5)], 1.370 (5) [C(6)], 1.358 (5) [C(7)] and 0.013 (5) Å [C(8)] for compound (1) and 1.425 (7), 1.385 (7), 1.397 (7) and 0.032 (6) Å for compound (2).



(*a*)

(*b*)

Fig. 1. Molecular structures of (*a*) $Cr(CO)_4(dppb)$ (1) and (*b*) $W(CO)_4(dppb)$ (2).

The mutually *cis* CO groups (*trans* to the bidentate phosphine ligand) have average *M*—C distances of 1.845 (4) and 1.969 (6) Å while the mutually *trans* CO ligands exhibit average bonding distances of 1.889 (4) and 2.005 (7) Å for compounds (1) and (2), respectively. The two *trans* CO groups have longer *M*—C distances than those of the two *cis* carbonyls. This feature is expected on the basis of the directional nature of π bonding in substituted octahedral metal carbonyls, since P is known to be a poorer π acceptor than CO (Cotton, Darensbourg, Klein & Kolthammer, 1982). The shorter Cr—CO lengths compared with the W—CO lengths may be attributed to the smaller atomic radius for the Cr atom.

The two *M*—P lengths are significantly different [2.415 (2) and 2.379 (2) Å for (1); 2.533 (1) and 2.503 (1) Å for(2)]. The inequality, which is also found in the Mo analogue, may be due to the conformational difference about P(1) and P(2): the two P—C—C bond angles in the seven-membered ring [118.1 (3) and 113.3 (3)° for (1); 118.4 (4) and 112.5 (4)° for (2)] and the two angles P(1)—*M*—C(2) and P(2)—*M*—C(2) [85.0 (1) and 92.9 (1)° for (1); 85.2 (2) and 92.8 (2)° for (2)].

The bond angles P—*M*—P are 93.29 (5) and 91.75 (4)° and *cis* C—*M*—C are 85.6 (2) and 86.2 (2)° for compounds (1) and (2), respectively. The larger P—Cr—P angle compared with P—W—P is also a result of the small size of the Cr atom. The *cis* C—*M*—C angles are complementary with the P—*M*—P angles. The ranges of P—C bond lengths and of C—C bond lengths in the phenyl groups for both title compounds are reasonable. Neither compound has any intermolecular contact of structural significance.

Among the three homologous complexes of Cr, W and Mo, the *M*—P and the *M*—C bond lengths of the Cr complex are smaller and the bite angle, P—*M*—P, is larger than those of the W and Mo complexes, whereas the Mo and W homologues have similar bond lengths and angles.

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Synthesis and Structure of Dihydroxo(η^3-2,2′-iminodiethoxo)germanium(IV)

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Abstract. $[Ge(OH)_2\{HN(CH_2CH_2O)_2\}]$ was prepared from germanium dioxide and 2,2′-iminodiethanol in refluxed H_2O and identified with IR, ^1H NMR, ^{13}C NMR, elemental analysis, and X-ray single-crystal structure determination. $C_4H_{11}GeNO_4$, $M_r = 209.6$, monoclinic, $P2_1/n$, $a = 5.161$ (2), $b = 12.251$ (4), $c = 11.266$ (3) Å, $\beta = 94.87$ (2)°, $V = 709.8$ (3) Å^3, $Z = 4$, $D_x = 1.96$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7093 Å, $\mu = 4.21$ mm^{-1}, $F(000) = 424$, $T = 298$ K, $R = 0.039$ for 1096 observed reflections. The title compound has a trigonal bipyramidal structure with the N atom of the tridentate ligand and one of the hydroxo groups in the axial position. The two Ge—O bond lengths for 2,2′-iminodiethoxide are similar, but are significantly different for the two hydroxo groups.

Introduction. Germanium complexes are attracting particular interest because of their specific biological activity (Brutkiewicz & Suzuki, 1987). It is also important to examine the expansion of the valence shell of the metal in these compounds beyond the limits permitted by the filled-octet rule. Reports about the structures of germanium complexes reveal that the coordination numbers of the metal are diverse (Breliere, Carre, Corriu & Royo, 1988; Day, Holmes, Sau & Holmes, 1982; Gurkova, Gusev, Sharapov, Alekseev, Gar & Chromova, 1984; Lazraq, Couret, Declercq, Dubourg, Escudie & Riviere-Bandet, 1990; Mizuta, Yoshida & Miyoshi, 1989). Among them, Gurkova *et al.* (1984) reported that the derivative for the triethanolamine has a trigonal bipyramidal structure. The main purpose of this paper is to try to synthesize the title compound from GeO_2 and diethanolamine and to determine the crystal structure.

Experimental. The title compound was prepared as follows: germanium dioxide (0.52 g, 5.0 mmol) was added to an aqueous solution (100 cm^3) of diethanolamine (2,2′-iminodiethanol) (1.05 g, 10.0 mmol) and the mixture refluxed for 2 h. After removal of the solvent, the crude product was recrystallized from methanol. The final products were colorless needle crystals, m.p. 458 K (dec.); yield 0.902 g (86%) (elemental analysis found: C, 22.54; H, 5.21; N, 6.56; Ge, 34.60%, $C_4H_{11}GeNO_4$ requires: C, 22.90; H, 5.24; N, 6.68; Ge, 34.61%); ν_{max} at 934 (*s*) and 821 (*s*) cm^{-1} (Ge—OC), 685 (*s*) cm^{-1} (Ge—N); δ_H (D_2O) 2.95 (4H, *t*, $2NCH_2$) and 3.77 (4H, *t*, $2OCH_2$); δ_C (D_2O) 51.8 ($2NCH_2$) and 60.8 ($2OCH_2$). A crystal, prepared from methanol by evaporation, had dimensions 0.30 × 0.30 × 0.50 mm. CAD-4 diffractometer. Unit cell: 25 reflections, 2θ range 18.64 to 26.42°. $\theta/2\theta$ mode with θ scan width = $(0.80 + 0.35\tan\theta)$°. Mo $K\alpha$ radiation. Three standard reflections were monitored every 1 h: variation in $I < 2.0\%$. 1383 reflections measured ($2.0 < 2\theta < 50.0°$, h, k, l: -6 to 6, 0 to 14, 0 to 13, respectively), giving 1244 unique reflections and 1096 observed with $I > 2.0\sigma(I)$. Absorption correction was made according to experimental ψ rotation (Enraf-Nonius, 1979) (maximum/minimum transmission factors = 0.644/0.982). Structure solved by heavy-atom method. Positions of H atoms were found in a difference Fourier map and were included in the structure factor calculation but not refined. The last least-squares cycle was calculated with 21 atoms, 92 parameters with anisotropic temperature factors for non-H atoms, and 1096 reflections. $(\Delta/\sigma)_{max} = 0.001$. Weighting scheme, $w = 1/\sigma^2(F_o)$, with $\sigma(F_o)$ from counting statistics. Quantity minimized $\sum w(KF_o - F_c)^2$. Final R, wR and S were 0.039, 0.046 and 2.96, respectively. Peaks in the final ΔF map were 1.10 to

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