Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^4$)

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

	x	y	z	U_{eq}
C(1)	0.6025 (15)	−0.3967 (12)	1.1101 (12)	74 (14)
P(10)	0.4737 (4)	−0.3446 (3)	1.1448 (3)	60 (3)
C(11)	0.4789 (14)	−0.2158 (10)	1.1340 (10)	56 (11)
C(12)	0.3868 (17)	−0.1627 (14)	1.1606 (12)	79 (15)
C(13)	0.4011 (26)	−0.0640 (16)	1.1606 (14)	107 (21)
C(14)	0.4957 (32)	−0.0196 (16)	1.1334 (17)	119 (25)
C(15)	0.5844 (26)	−0.0747 (18)	1.1065 (19)	125 (25)
C(16)	0.5784 (17)	−0.1732 (13)	1.1073 (13)	80 (15)
C(21)	0.4882 (15)	−0.3692 (11)	1.2718 (10)	61 (12)
C(22)	0.6053 (17)	−0.3920 (14)	1.3336 (12)	86 (16)
C(23)	0.6214 (21)	−0.4012 (16)	1.4330 (16)	106 (21)
C(24)	0.5202 (26)	−0.3824 (14)	1.4691 (13)	101 (20)
C(25)	0.4012 (22)	−0.3632 (15)	1.4098 (15)	102 (20)
C(26)	0.3798 (17)	−0.3599 (14)	1.3057 (11)	85 (16)
C(31)	0.3293 (15)	−0.3917 (11)	1.0676 (11)	60 (12)
C(32)	0.2822 (16)	−0.4800 (13)	1.0867 (12)	73 (14)
C(33)	0.1767 (18)	−0.5211 (14)	1.0180 (16)	87 (17)
C(34)	0.1215 (17)	−0.4750 (18)	0.9313 (15)	94 (19)
C(35)	0.1650 (20)	−0.3888 (17)	0.9110 (14)	103 (20)
C(36)	0.2709 (17)	−0.3481 (13)	0.9782 (13)	81 (16)

cation, Table 2 gives selected bond distances and angles.*

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

P(10)—C(1)	1.77 (2)	P(10)—C(21)	1.79 (1)
P(10)—C(11)	1.81 (1)	P(10)—C(31)	1.76 (2)
C(1)—P(10)—C(11)	109.9 (8)	C(12)—C(11)—P(10)	118.0 (12)
C(1)—P(10)—C(21)	110.5 (8)	C(16)—C(11)—P(10)	119.9 (13)
C(1)—P(10)—C(31)	108.0 (7)	C(22)—C(21)—P(10)	119.1 (13)
C(11)—P(10)—C(21)	106.3 (7)	C(26)—C(21)—P(10)	119.0 (12)
C(11)—P(10)—C(31)	111.3 (7)	C(32)—C(31)—P(10)	121.6 (13)
C(21)—P(10)—C(31)	110.8 (8)	C(36)—C(31)—P(10)	119.6 (13)

Related literature. There are no reported examples of X-ray molecular structures of tetrasubstituted phosphonium salts of TaF_6^-. The closest example is 18-crown-6 bis(methyltriphenylphosphonium) bis(hexafluorophosphate) clathrate.

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Structure of $[PrCl_3(EO4)]_2$

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Abstract. Di-μ-chloro-bis[dichloro(tetraethylene glycol)praseodymium(III)], $M_r = 882.99$, triclinic, $P\bar{1}$, $a = 8.564$ (5), $b = 9.439$ (9), $c = 9.566$ (5) Å, $\alpha = 86.01$ (9), $\beta = 72.30$ (6), $\gamma = 78.09$ (9)°, $V = 720.8$ Å^3, $Z = 1$, D_m not determined, $D_x = 2.03$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 39.3$ cm^{-1}, $F(000) = 432$, $T = 293$ K, final $R = 0.037$ for 2114 observed [$F_o \geq 5\sigma(F_o)$] reflections. The dimer resides around a crystallographic center of inversion with asymmetric [2.986 (2), 2.910 (2) Å] chloro bridges. Two terminal chloride ions and the pentadentate tetraethylene glycol ligand (EO4) complete the 9-coordinate tricapped trigonal-prismatic Pr coordination sphere. There is one intramolecular hydrogen bond and one

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intermolecular hydrogen bond which creates polymers of dimers propagating along **b**. The complex is isostructural with its La analog [Rogers, Etzenhouser, Murdoch & Reyes (1991). *Inorg. Chem.* **30**, 1445–1455].

Experimental. The title complex was unintentionally isolated during a reaction designed to complex an EO4 ligand functionalized with cinnamic acid to $PrCl_3.6H_2O$. The reaction mixture contained $PrCl_3.6H_2O$, EO4, cinnamic acid, and thionyl chloride in approximate 1:1:2:2 stoichiometry. Clear parallelepipeds grown from acetonitrile:methanol (3:1). D_m not determined. Crystal 0.08 × 0.13 × 0.23 mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$, ω–2θ scans. Cell constants from setting angles of 25 reflections ($\theta >$ 20°). Corrections for Lorentz–polarization effect and for absorption (empirical, ψ scans), range of relative transmission factors 66/100%. $\theta_{\max} = 25°$; h 0 to 10, k −11 to 11, l −11 to 11. Standard reflections (400; 050; 006) measured every 3600 s of data-collection time, variation ±2%. 2537 reflections measured (2384 unique, $R_{\rm int} = 0.0048$), 2114 independent observed reflections [$F_o \geq 5\sigma(F_o)$]. Structure solved *via* utilization of fractional coordinates from isostructural La complex (Rogers, Etzenhouser, Murdoch & Reyes, 1991). Glycol H atoms placed 0.95 Å from the bonded C atom with a fixed isotropic thermal parameter $B = 5.5$ Å^2 and allowed to ride with the C—H distance fixed; alcoholic H atoms located from a difference Fourier map and placed in fixed positions with $B = 5.5$ Å^2. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV); structure refined with *SHELX76* (Sheldrick, 1976). $\sum w(|F_o| - |F_c|)^2$ minimized, weights $= [\sigma(F_o)^2 + 0.00033F_o^2]^{-1}$, 154 parameters varied. $R = 0.037$, $wR = 0.050$, $S = 1.2$. Δ/σ in final least-squares refinement cycle < 0.01, $\Delta\rho < 1.3$ e Å^{-3} near Pr position in final difference map. Fractional coordinates and $B_{\rm eq}$ values are given in Table 1,* selected distances and angles in Table 2, and an *ORTEP* illustration (Johnson, 1976) of the atom labeling and intramolecular hydrogen bonding in Fig. 1.

Table 1. *Final fractional coordinates for* [PrCl$_3$(EO4)]$_2$

$$B_{\rm eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (ab\cos\gamma)\beta_{12} + (ac\cos\beta)\beta_{13} + (bc\cos\alpha)\beta_{23}].$$

	x	y	z	$B_{\rm eq}$ (Å^2)
Pr	0.00723 (6)	0.29726 (5)	0.66427 (5)	1.19
Cl(1)	0.0672 (3)	0.5995 (2)	0.6067 (2)	1.67
Cl(2)	−0.3032 (3)	0.4796 (2)	0.7866 (2)	1.76
Cl(3)	0.1861 (3)	0.0137 (2)	0.6706 (2)	1.94
O(1)	−0.1684 (8)	0.1428 (7)	0.5938 (7)	2.22
O(2)	−0.1840 (8)	0.1502 (7)	0.8728 (6)	1.91
O(3)	0.0162 (8)	0.3080 (7)	0.9387 (6)	1.87
O(4)	0.2863 (7)	0.3173 (7)	0.7124 (7)	2.04
O(5)	0.2639 (7)	0.2774 (7)	0.4439 (6)	1.76
C(1)	−0.242 (1)	0.037 (1)	0.692 (1)	2.46
C(2)	−0.315 (1)	0.107 (1)	0.840 (1)	2.50
C(3)	−0.232 (1)	0.203 (1)	1.0210 (8)	1.84
C(4)	−0.072 (1)	0.217 (1)	1.0495 (9)	2.09
C(5)	0.169 (1)	0.315 (1)	0.967 (1)	2.11
C(6)	0.265 (1)	0.396 (1)	0.844 (1)	2.14
C(7)	0.420 (1)	0.350 (1)	0.589 (1)	2.17
C(8)	0.426 (1)	0.258 (1)	0.467 (1)	2.12

Table 2. *Selected distances* (Å) *and angles* (°)

Pr—Cl(1)	2.986 (2)	Pr—Cl(1)$^{\rm i}$	2.910 (2)
Pr—Cl(2)	2.810 (2)	Pr—Cl(3)	2.803 (2)
Pr—O(1)	2.550 (6)	Pr—O(2)	2.675 (6)
Pr—O(3)	2.659 (6)	Pr—O(4)	2.613 (6)
Pr—O(5)	2.525 (6)		
O—C$_{\rm avg}$	1.44 (2)	C—C$_{\rm avg}$	1.495 (5)
Cl(1)—Pr—Cl(1)$^{\rm i}$	71.49 (8)	Pr—Cl(1)—Pr$^{\rm i}$	108.51 (8)
C—O—C$_{\rm avg}$	111 (2)	O—C—C$_{\rm avg}$	107 (2)

Symmetry code: (i) $-x$, $1-y$, $1-z$.

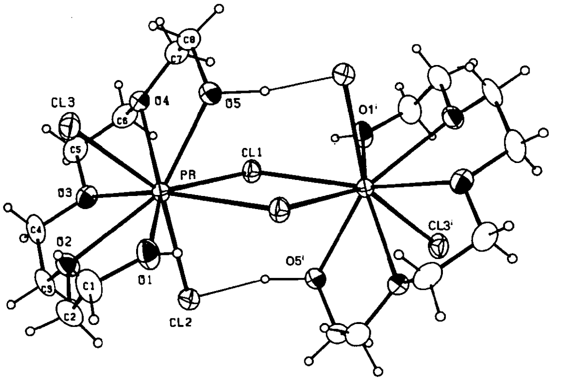

Fig. 1. *ORTEP* illustration of [PrCl$_3$(EO4)]$_2$ with 50% probability ellipsoids for thermal motion. The H atoms have been arbitrarily reduced. The intramolecular hydrogen bonds are indicated by lines. Symmetry code: (i) $-x$, $1-y$, $1-z$.

Related literature. The average r.m.s. distance between atomic parameters in the title complex and its La analog is 0.038 (Rogers, Etzenhouser, Murdoch & Reyes, 1991). The differences observed in bonding parameters are those expected by the 0.037 Å difference in effective ionic radii of nine-coordinate La^{3+} and Pr^{3+} (Shannon, 1976). The differences in average bonding parameters for donor type (alcoholic *vs* etheric oxygen or chloride ion) and geometric site (capping *vs* prismatic) range from

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, least-squares-planes results, bond distances and angles, and hydrogen-bonding contact geometries have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54803 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD0074]

0.022 Å [M—Cl(1)($-x$, $1-y$, $1-z$)] to 0.040 Å (M—O etheric, capping) and average 0.03 Å. Similar average distances in the related complex $[PrCl(OH_2)_3(EO4)]Cl_2.H_2O$ (Rogers, Etzenhouser, Murdoch & Reyes, 1991) are an average 0.07 Å shorter as a consequence of the reduced number of tight ion pairs compared to the title complex.

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Structure of Bis(N,N-diethyldithiocarbamato)gold(III) Hexafluoroantimonate

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Abstract. $[Au\{S_2CN(C_2H_5)_2\}_2]SbF_6$, $M_r = 1458.55$, triclinic, $P\bar{1}$, $a = 14.533\,(3)$, $b = 18.004\,(5)$, $c = 8.538\,(2)$ Å, $\alpha = 100.37\,(4)$, $\beta = 105.92\,(5)$, $\gamma = 74.69\,(3)^\circ$, $V = 2058.06$ Å^3, $Z = 4$, $D_x = 2.353$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\mu = 88.7$ cm^{-1}, $F(000) = 1368$, $T = 296\,(1)$ K, $R = 0.048$ and $wR = 0.056$ for 4323 reflections with $I > 3\sigma(I)$. Each Au atom is four coordinated in a plane. The Au—S bond distances fall in the range 2.318–2.330 Å.

Experimental. The title compound was prepared by reaction of Ph_2PAuCl, $NaS_2CNEt_2.3H_2O$ and $NaSbF_6$ in EtOH solution and a crystal suitable for X-ray analysis was grown by evaporation of the reaction solution in air.

Diffraction intensities were collected from a prismatic pale-yellow crystal 0.30 × 0.30 × 0.60 mm in the θ–2θ scan mode on a Rigaku AFC5R diffractometer using graphite-monochromatized Mo $K\alpha$ radiation. Cell constants were obtained from least-squares refinement of 20 reflections (θ from 8 to 10°). A total of 7399 reflections were collected in the range $3 < 2\theta < 50^\circ$ ($0 < h < 15$, $-21 < k < 21$, $-10 < l < 10$). The intensities were monitored by three representative reflections. The data were corrected for the fluctuation of the monitored reflections (between 1.000 and 0.981), the Lp factor, and empirical absorption (between 1.112 and 0.905), but no extinction correction was made. 4323 unique reflections with $I > 3\sigma(I)$ were used for structure solution and refinement. Calculations were performed on a VAX 11/785 computer with the *SDP* program package (Frenz, 1978). All metal atoms were located from the E map. A Fourier map phased by the metal atoms contained most of the remaining non-H atoms. H atoms were not included in the calculation. 433 variables were used in the refinement. $R_{\rm int} = 0.067$. Full-matrix least-squares refinement with anisotropic thermal parameters for all non-H atoms led to convergence with $R = 0.048$ and $wR = 0.056$. Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w^{-1} = 1 + \{[(F_o)^2 + 2\sigma(F_o)|F_o|]^{1/2} - |F_o|\}^2 + (0.01F_o)^2$. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (Cromer & Waber, 1974). The goodness of fit (S) is 2.01. The largest Δ/σ value in the final cycle is 0.40 while the extreme peaks in the difference electron density map are 1.95 and -1.67 e Å^{-3}. Final positional parameters given in Table 1, and atomic distances and bond angles are list in Table 2.† The atomic labelling scheme and the structure of the cation are shown in Fig. 1, and the cell packing is shown in Fig. 2.

Related literature. Crystal structures of related compounds have been studied: $[Au(S_2CNBu_2)_2]$-$[AuBr_2]$ (Beurskens, Blaauw, Cras & Steggerda, 1968), $[Au(S_2CNBu_2)_2]Br$ (Beurskens, Cras & van der Linden, 1970), $[3,3'\text{-}Au(1,2\text{-}C_2B_9H_{11})_2][Au\text{-}(S_2CNEt_2)_2]$ (Colquhoun, Greenhough & Wallbridge, 1977).

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† Tables of anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54810 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0529]

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