Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement factors* ($Å^2 \times 10^4$)

$$U_{eq} = \tfrac{1}{3}[U_{33} + \tfrac{4}{3}(U_{11} + U_{22} - U_{12})].$$

	x	y	z	U_{eq}
Li(1)	9797 (4)	1917 (4)	845 (5)	202
Li(2)	9840 (4)	1917 (4)	4141 (5)	197
Se	9854 (1)	1978 (1)	7495 (1)	118
O(1)	1104 (2)	2156 (2)	7504 (2)	185
O(2)	−45 (2)	3196 (1)	7512 (2)	171
O(3)	9189 (2)	1282 (2)	8928 (2)	178
O(4)	9223 (2)	1297 (2)	6046 (2)	179

Table 2. *Some interatomic distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Li(1)—O(1)	1·931 (5)	O(1)—O(2)	3·164 (3)	108·4 (2)
O(2)	1·968 (6)	O(3)	3·347 (2)	117·2 (2)
O(3)	1·990 (5)	O(3′)	3·131 (3)	106·6 (2)
O(3′)	1·975 (6)	O(2)—O(3)	3·139 (3)	104·9 (2)
		O(3′)	3·225 (3)	109·7 (2)
		O(3)—O(3′)	3·245 (4)	109·9 (2)
Li(2)—O(1)	1·946 (5)	O(1)—O(2)	3·218 (3)	112·1 (2)
O(2)	1·932 (6)	O(4)	3·334 (2)	116·3 (2)
O(4)	1·979 (5)	O(4′)	3·163 (3)	108·9 (2)
O(4′)	1·942 (6)	O(2)—O(4)	3·117 (3)	105·7 (2)
		O(4′)	3·047 (3)	103·7 (2)
		O(4)—O(4′)	3·201 (4)	109·4 (2)
Se—O(1)	1·636 (2)	O(1)—O(2)	2·650 (3)	108·2 (1)
O(2)	1·636 (2)	O(3)	2·673 (3)	109·3 (1)
O(3)	1·642 (1)	O(4)	2·654 (3)	108·5 (1)
O(4)	1·636 (1)	O(2)—O(3)	2·680 (3)	109·7 (1)
		O(4)	2·692 (3)	110·8 (1)
		O(3)—O(4)	2·692 (2)	110·4 (1)

Related literature. The crystals of the title compound correspond to Li_2SeO_4 as described by Pistorius (1967) based on a powder diffraction diagram. They are representatives of the phenakite-type structure (*cf.* Wyckoff, 1965). Recently Zemann (1986) pointed out a very short interpolyhedral O(2)—O(2) distance of 2·75 Å in phenakite, Be_2SiO_4. The analogous distance in Li_2SeO_4 is 3·128 (3) Å. The reasons are the average cation—oxygen distances in the XO_4 tetrahedra: Be—O (1·645 and 1·646 Å) is shorter than Li—O (1·966 and 1·950 Å), and Si—O (1·632 Å) is shorter than Se—O (1·638 Å).

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Structure of Tetrakis(trimethylphosphine)cobalt(I) Tetraphenylborate

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Abstract. $[Co\{P(CH_3)_3\}_4]B(C_6H_5)_4$, $M_r = 682{\cdot}48$, monoclinic, $P2_1/c$, $a = 16{\cdot}190\,(9)$, $b = 12{\cdot}422\,(4)$, $c = 20{\cdot}900\,(10)$ Å, $\beta = 111{\cdot}87\,(5)°$, $V = 3900{\cdot}7$ Å^3, $Z = 4$, $D_x = 1{\cdot}162$ Mg m^{-3}, $\lambda(\text{Mo } K\bar{\alpha}) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}63$ mm^{-1}, $F(000) = 1456$, $T = 296$ K, final $R = 0{\cdot}037$ for 2167 unique observed reflections. The unit cell contains BPh_4^- anions and $[Co(PMe_3)_4]^+$ cations. Co has a distorted tetrahedral coordination (P—Co—P = 101·1–125·4°, $\sigma = 0{\cdot}1°$), and the Co—P distances range from 2·193 (2) to 2·252 (2) Å.

Experimental. Compound prepared by mixing 1·4 g (4·1 mmol) $NaBPh_4$ in methanol (15 mL) with 1·5 g (4·1 mmol) $CoBr(PMe_3)_3$ in 15 mL methanol. Color changed from blue to brown, and a brownish green solid precipitated. Blue–green crystals obtained by recrystallization from methanol at 243 K. Elongated prism, 0·30 × 0·35 × 0·54 mm. Space group $P2_1/c$ determined from precession and cone-axis photographs, unambiguously defined from systematic absences ($h0l$, $l \neq 2n$; $0k0$, $k \neq 2n$). Enraf–Nonius CAD-4 diffractometer, accurate cell dimensions from 25 centered reflections ($11° \leq \theta \leq 16°$), detailed pro-

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Table 1. *Refined coordinates* (10^4, Co × 10^5), U_{eq} ($Å^2 \times 10^3$)

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

	x	y	z	U_{eq}
Co	21950 (5)	25304 (8)	11577 (4)	656 (3)
P(1)	1211 (1)	2527 (2)	84 (1)	708 (7)
P(2)	3393 (1)	3221 (1)	1008 (1)	613 (7)
P(3)	2548 (1)	832 (2)	1391 (1)	848 (10)
P(4)	1642 (1)	3622 (1)	1761 (1)	782 (9)
C(1)	1546 (4)	2077 (5)	−609 (3)	85 (3)
C(2)	701 (5)	3813 (6)	−272 (3)	112 (4)
C(3)	234 (4)	1688 (7)	−66 (4)	124 (5)
C(4)	3937 (3)	2428 (5)	544 (3)	72 (3)
C(5)	3248 (4)	4481 (5)	565 (3)	89 (4)
C(6)	4309 (4)	3495 (6)	1810 (3)	99 (4)
C(7)	1802 (5)	36 (7)	1649 (4)	136 (5)
C(8)	2672 (6)	41 (6)	735 (4)	128 (5)
C(9)	3554 (5)	554 (8)	2094 (4)	138 (5)
C(10)	1836 (5)	5007 (6)	1690 (4)	122 (5)
C(11)	452 (5)	3580 (6)	1550 (4)	117 (5)
C(12)	2060 (5)	3451 (7)	2650 (4)	133 (5)
C(41)	7083 (3)	3754 (4)	1115 (2)	47 (2)
C(42)	6216 (4)	4163 (4)	954 (3)	63 (3)
C(43)	6028 (4)	5230 (5)	996 (3)	73 (3)
C(44)	6719 (4)	5966 (5)	1202 (3)	78 (3)
C(45)	7564 (4)	5611 (5)	1367 (3)	77 (3)
C(46)	7734 (3)	4545 (5)	1313 (3)	61 (3)
C(51)	6582 (3)	1672 (4)	1115 (2)	44 (2)
C(52)	6267 (3)	747 (5)	729 (3)	59 (3)
C(53)	5769 (3)	−51 (4)	910 (3)	66 (3)
C(54)	5569 (3)	83 (5)	1486 (3)	65 (3)
C(55)	5872 (3)	984 (5)	1883 (3)	64 (3)
C(56)	6358 (3)	1751 (4)	1697 (3)	55 (3)
C(61)	7134 (3)	2540 (5)	145 (2)	51 (2)
C(62)	6311 (3)	2504 (5)	−381 (2)	58 (2)
C(63)	6190 (4)	2617 (5)	−1066 (3)	79 (3)
C(64)	6894 (5)	2773 (5)	−1253 (3)	87 (4)
C(65)	7731 (4)	2805 (5)	−754 (3)	78 (3)
C(66)	7845 (4)	2708 (4)	−75 (3)	62 (3)
C(71)	8247 (3)	2060 (4)	1472 (3)	57 (3)
C(72)	8677 (3)	2402 (5)	2140 (3)	71 (3)
C(73)	9464 (4)	1930 (6)	2575 (3)	107 (4)
C(74)	9833 (4)	1106 (6)	2371 (4)	111 (4)
C(75)	9413 (4)	738 (5)	1726 (4)	96 (4)
C(76)	8645 (3)	1199 (5)	1277 (3)	68 (3)
B	7267 (4)	2504 (6)	959 (3)	53 (3)

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

Co—P(1)	2·216 (2)	P(2)—C(5)	1·789 (7)
Co—P(2)	2·247 (2)	P(2)—C(6)	1·809 (7)
Co—P(3)	2·193 (2)	P(3)—C(7)	1·792 (9)
Co—P(4)	2·252 (2)	P(3)—C(8)	1·758 (8)
P(1)—C(1)	1·814 (7)	P(3)—C(9)	1·774 (8)
P(1)—C(2)	1·825 (8)	P(4)—C(10)	1·765 (8)
P(1)—C(3)	1·821 (8)	P(4)—C(11)	1·810 (8)
P(2)—C(4)	1·823 (6)	P(4)—C(12)	1·737 (8)
P(1)—Co—P(2)	101·1 (1)	P(2)—Co—P(3)	103·4 (1)
P(1)—Co—P(3)	104·7 (1)	P(2)—Co—P(4)	113·1 (1)
P(1)—Co—P(4)	106·4 (1)	P(3)—Co—P(4)	125·4 (1)
Co—P(1)—C(1)	119·6 (2)	Co—P(2)—C(4)	118·7 (2)
Co—P(1)—C(2)	117·2 (3)	Co—P(2)—C(5)	116·9 (2)
Co—P(1)—C(3)	115·2 (3)	Co—P(2)—C(6)	113·3 (2)
C(1)—P(1)—C(2)	99·7 (3)	C(4)—P(2)—C(5)	101·2 (3)
C(1)—P(1)—C(3)	100·7 (3)	C(4)—P(2)—C(6)	101·7 (3)
C(2)—P(1)—C(3)	101·5 (4)	C(5)—P(2)—C(6)	102·7 (3)
Co—P(3)—C(7)	116·7 (3)	Co—P(4)—C(10)	114·8 (3)
Co—P(3)—C(8)	117·4 (3)	Co—P(4)—C(11)	117·1 (3)
Co—P(3)—C(9)	117·0 (3)	Co—P(4)—C(12)	116·6 (3)
C(7)—P(3)—C(8)	102·2 (4)	C(10)—P(4)—C(11)	102·4 (4)
C(7)—P(3)—C(9)	99·0 (4)	C(10)—P(4)—C(12)	101·4 (4)
C(8)—P(3)—C(9)	101·6 (4)	C(11)—P(4)—C(12)	102·3 (4)

cedure for data collection described elsewhere (Bélanger-Gariépy & Beauchamp, 1980), graphite-monochromatized Mo $K\bar{\alpha}$ radiation, $2\theta_{max} = 45°$, $0 \leq h \leq 17$, $0 \leq k \leq 13$, $-21 \leq l \leq 20$; orientation checked every 100 measurements, intensity of three standards checked every hour, max. fluctuation 2·3%, 5378 independent reflections measured, 2167 with $I > 3{\cdot}0\sigma(I)$. Data corrected for Lp and absorption, transmission range: 0·64–0·76. Co position determined from Patterson map, other atoms from ΔF map; structure refined on F isotropically by full-matrix least squares, then anisotropically by block-diagonal least squares. At least one H located on ΔF map for each methyl group, used to calculate ideal coordinates for each set of methyl H (C—H 0·95 Å, isotropic $B = 7$ Å^2). Ring H fixed at idealized positions ($B = 5{\cdot}5$ Å^2). H repositioned after each least-squares cycle. Final $R = 0{\cdot}037$, $wR = 0{\cdot}041$ (weights based on counting statistics), $S = 1{\cdot}35$, (shift/σ), mean = 0·02, max. = 0·06. Residual electron density on final ΔF map: general background from −0·18 to 0·17 e Å^{-3}, peaks 0·20 to 0·39 e Å^{-3} near Co or P atoms. Scattering factors from Cromer & Mann (1968), except for H, from Stewart, Davidson & Simpson (1965). Anomalous dispersion f' and f'' terms for Co and P from Cromer & Liberman (1970). Programs used listed elsewhere (Authier-Martin & Beauchamp, 1977). Refined coordinates in Table 1.*

Related literature. In contrast to $Co(PMe_3)_2(BPh_4)$ (De Carvalho, Dartiguenave, Dartiguenave & Beauchamp, 1984), the BPh_4^- ion is not π-bonded to the metal, but

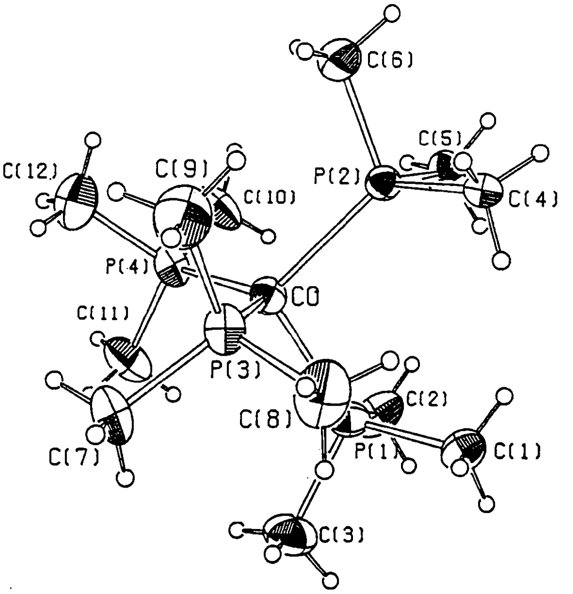

Fig. 1. *ORTEP* drawing of the $[Co(PMe_3)_4]^+$ cation. The ellipsoids correspond to 50% probability. H atoms are represented as spheres of arbitrary size.

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* Lists of observed and calculated structure factor amplitudes, H atom fixed coordinates, anisotropic thermal parameters, distances and angles in the BPh_4 ions and details on least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51350 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

plays its usual role of counter-ion. Its geometry is normal (see supplementary material). This Co^I compound is isomorphous with $[Ni(PMe_3)_4]BPh_4$ (Gleizes, Dartiguenave, Dartiguenave, Galy & Klein, 1977), but the tetrahedral environment of the metal [P—Co—P = 101·1 (1)–125·4 (1)°, Table 2 and Fig. 1] is more distorted than in the Ni^{II} complex [P—Ni—P = 104·6(1)–119·9(1)°]. Similar distortions are also present in the related four-coordinate Co^I molecules $CoCl(PMe_3)_3$ [Cl—Co—P = 113·8 (2)°, P—Co—P = 104·8 (3)° (Jones, Stuart, Atwood & Hunter, 1983)] and $CoI(PMe_3)_3$ [I—Co—P = 119·26 (6)–109·77 (4)°, P—Co—P = 105·24 (4)–106·84 (7)° (Bandy, Green & Kirchner, 1985)], and they probably result from the phosphine methyl groups being interlocked to minimize steric hindrances about the metal. The bond lengths in the $[Co(PMe_3)_4]^+$ ion [av. 2·227 Å] remain roughly the same as in the iodo [av. 2·235 Å] and chloro [2·240 Å] compounds.

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Structure of a Nickel(II) Complex of 18-Crown-6 at 163 K

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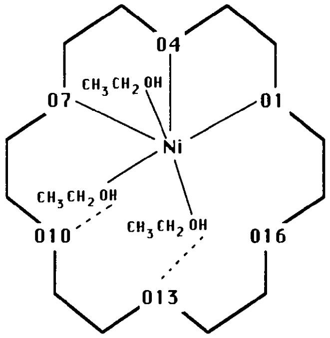
Abstract. Tris(ethanol)(1,4,7,10,13,16-hexaoxacyclooctadecane)nickel(II) bis(hexafluorophosphate), $[Ni(C_2H_6O)_3(C_{12}H_{24}O_6)](PF_6)_2$, $M_r = 751·16$, monoclinic, $P2_1/c$, $a = 8·976$ (2), $b = 16·533$ (4), $c = 22·230$ (6) Å, $\beta = 109·37$ (3)°, $V = 3112·2$ (14) Å^3, $Z = 4$, $D_x = 1·603$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 8·483$ cm^{-1}, $F(000) = 1552$, $R = 0·0404$ for 4161 reflections ($F \geq 6\sigma_F$). The Ni^{2+} ion is coordinated to three adjacent O atoms of the crown (O1, O4, O7) and to the O atoms of three ethanol solvate molecules, the O atoms forming a distorted octahedron. The range of Ni—O distances is 1·991 (2)–2·114 (3) Å. Two ethanol molecules are hydrogen bonded to crown O atoms and the third is hydrogen bonded to an F atom of one PF_6^- ion. The crown has approximate mirror symmetry. The O1—O4—O7 and O10—O13—O16 planes have a dihedral angle of 106·2 (2)°. The average crown C—O bond lengths are 1·448 (2) Å for Ni-bound O atoms and 1·426 (4) Å for non-Ni-bound O atoms. The average C—O bond length in the ethanol molecules is 1·448 (7) Å. All C—C bonds have lengths in the range 1·489–1·505 Å.

Experimental. The title compound was prepared by the reaction of 18-crown-6 with $Ni(EtOH)_6(PF_6)_2$ in ethanol and ethyl orthoformate (EOF). $Ni(EtOH)_6(PF_6)_2$ was prepared by the reaction of $NiCl_2.6H_2O$ with $AgPF_6$ in ethanol and EOF. A pale-green crystal was mounted in a capillary tube with grease using a Schlenk apparatus to exclude moisture. The grease prevented an accurate measurement of crystal size; the maximum dimension was 0·5 mm. A summary of data collection and structural refinement is given in Table 1.



13 atoms obtained from *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) were

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