

been observed in the symmetric diaquahydrogen ion $\text{H}_2\text{O}\cdot\text{H}\cdot\text{OH}_2^+$ (Selenius & Delaplane, 1978; Brown, Noe-Spirlet, Busing & Levy, 1977). Unlike those of homoconjugated dicarboxylate anions in Speakman's (1972) type *A* salts, the two CO distances in the acetate ligands are identical: hydrogen bonding and coordination to the metal atom have similar effects on the C—O bond lengths. The geometry of the carboxylate group is more comparable to that of the acetate acidium ion where the CO groups are equally bonded to two H atoms (Jönsson & Olovsson, 1968).

The planes of the acetate groups are nearly perpendicular to the CoN(1)N(2) planes [93.6 (8)° for cation (*A*) and 93.0 (8)° for cation (*B*)] and almost bisect the N(1)CoN(2) outer ring angle. The dihedral angles between the acetate planes and the planes defined by CoN(1)O(1) are 47.0 (6)° [cation (*A*)] and 46.7 (6)° [cation (*B*)]. As can be readily seen the orientation of the plane of the acetate ligand is such that the carbonyl O(2) atom comes into a hydrogen-bonding contact with atoms H(1) and H(7): the O atoms involved in the very short OHO bonds also participate in interligand hydrogen bonds with the amino groups of the ethylenediamine ring. This behavior is anomalous with respect to usual type *A* symmetric hydrogen bonds. This situation has only been encountered in the case of potassium hydrogen *meso*-tartrate (Currie, Speakman, Kanters & Kroon, 1975).

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The Crystal Structure of μ_4 -Antimonio-tetrakis[tricarbonyl(triphenylphosphine)cobalt] Tetraphenylborate–Dichloromethane, $[\{\text{Co}(\text{CO})_3\text{PPh}_3\}_4\text{Sb}][\text{BPh}_4]\cdot\text{CH}_2\text{Cl}_2$

BY R. E. COBBLEDICK AND F. W. B. EINSTEIN

Department of Chemistry, Simon Fraser University, Burnaby, British Columbia, Canada V5A 1S6

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Abstract

$[\{\text{Co}(\text{CO})_3\text{PPh}_3\}_4\text{Sb}][\text{BPh}_4]\cdot\text{CH}_2\text{Cl}_2$ is monoclinic, space group $P2_1/c$, with $a = 16.695$ (6), $b = 38.026$ (14), $c = 15.861$ (6) Å, $\beta = 90.64$ (2)°, $Z = 4$. The structure was refined to an R of 0.060 for 3305 counter reflexions. It consists of discrete $[\{\text{Co}(\text{CO})_3\text{PPh}_3\}_4\text{Sb}]^+$ cations, $[\text{BPh}_4]^-$ anions and

CH_2Cl_2 solvent of crystallization. In the cation there is tetrahedral geometry about the Sb atom with Sb—Co = 2.593 (3)–2.602 (2) Å (mean Sb—Co = 2.596 Å, mean Co—Sb—Co = 109.5°). The coordination at the Co atoms is trigonal bipyramidal with the CO groups in equatorial positions and the P atoms *trans* to Sb with mean Co—C = 1.74 Å (range 1.71–1.78) and mean Co—P = 2.217 Å (range 2.212–2.226 Å).

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Introduction

The reaction of SbX_3 ($X = \text{Cl}$ or Br) with $[\text{Co}(\text{CO})_3\text{PPh}_3]_2$ gives salts of the $\{[\text{Co}(\text{CO})_3\text{PPh}_3]_4\text{Sb}\}^+$ cation (Cullen, Patmore & Sams, 1973). The structure of the $[\text{BPh}_4]^-$ salt has been determined in order to confirm the characterization of the cation.

Experimental

Dark-red crystals were obtained from CH_2Cl_2 . Weissenberg and precession photographs ($\text{Cu K}\alpha$) were used to obtain approximate cell dimensions and the systematic absences $h0l: l = 2n + 1, 0k0: k = 2n + 1$ indicated space group $P2_1/c$. A roughly equidimensional crystal (0.30 mm) was used for the measurement of accurate cell dimensions and for the collection of intensities. The cell constants (Table 1) were obtained by least-squares refinement of the setting angles of 12 reflexions centred on a Picker FACS-I computer-controlled diffractometer [$\lambda(\text{Mo K}\alpha_1) = 0.70926 \text{ \AA}$]. Intensities were collected by the θ - 2θ scan method with graphite-monochromated $\text{Mo K}\alpha$ radiation. A scan rate of 2° min^{-1} in 2θ with a base-width of 1.3° (increased to allow for spectral dispersion) was employed and background counts of 10 s were taken on each side of the reflexions. Two standard reflexions were measured regularly to monitor intensity variation. 4098 unique reflexions with $2\theta \leq 30^\circ$ were measured; of these, 3305 with $I > 2.3\sigma(I)$ were considered observed, where $\sigma(I) = [S + (t_s/t_b)^2 B + (0.03I)^2]^{1/2}$, S is the total scan count, and t_s and t_b are the scan and background count times respectively. Intensities were corrected for Lorentz and polarization effects but not for absorption.

Structure determination and refinement

A Patterson function indicated the positions of the Sb and Co atoms. All the non-hydrogen atoms were located from subsequent Fourier and difference maps. Full-matrix least-squares refinement was employed with isotropic thermal parameters for all atoms initially and with anisotropic parameters for the Co, Sb and O atoms in the final cycles. As a result of storage limita-

tion, the molecule was divided into two fragments which were allowed to refine on alternate cycles. Calculated positions for the H atoms were determined. H atoms were assigned isotropic temperature factors equal to those of the C atoms to which they were bonded. Further refinement with the H atoms in fixed positions reduced R to 0.060 ($R = \sum ||F_o| - |F_c|| / \sum |F_o|$). The function minimized was $\sum w(|F_o| - |F_c|)^2$ where the weights w were derived from counting statistics ($w = 1/\sigma^2 F_o$). No significant variation in $\sum w(|F_o| - |F_c|)^2$ as a function of $\sin \theta/\lambda$, $|F_o|$ or various parity groups was observed. The maximum shift in the atomic parameters on the final cycle was 0.16σ .

Scattering factors (including anomalous-dispersion corrections for Sb, Co and P) were taken from *International Tables for X-ray Crystallography* (1974); for H, those of Stewart, Davidson & Simpson (1965) were used. The programs have been described elsewhere (Einstein & Jones, 1972). The final atomic coordinates and isotropic temperature parameters are given in Table 2.*

Results and discussion

Bond lengths and angles are given in Table 3 and the molecular configuration is illustrated in Fig. 1. The structure consists of discrete $\{[\text{Co}(\text{CO})_3\text{PPh}_3]_4\text{Sb}\}^+$ cations, $[\text{BPh}_4]^-$ anions and CH_2Cl_2 solvent of crystallization. Coordination geometry about the Sb atom in the cation is tetrahedral; the Sb-Co distances range from 2.593 (3) to 2.602 (2) \AA with a mean of 2.596 \AA

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34525 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Crystal data

$\{[\text{Co}(\text{CO})_3\text{PPh}_3]_4\text{Sb}\}[\text{BPh}_4] \cdot \text{CH}_2\text{Cl}_2$, FW 2147.01, monoclinic, space group $P2_1/c$

$a = 16.695$ (6) \AA
 $b = 38.026$ (14)
 $c = 15.861$ (6)
 $\beta = 90.64$ (2) $^\circ$
 $V = 10068.94$ \AA^3

$Z = 4$
 $D_m = 1.40$ Mg m^{-3} (by flotation)
 $D_c = 1.416$
 $\lambda(\text{Mo K}\alpha_1) = 0.70926$ \AA
 $\mu(\text{Mo K}\alpha) = 1.117$ mm^{-1}

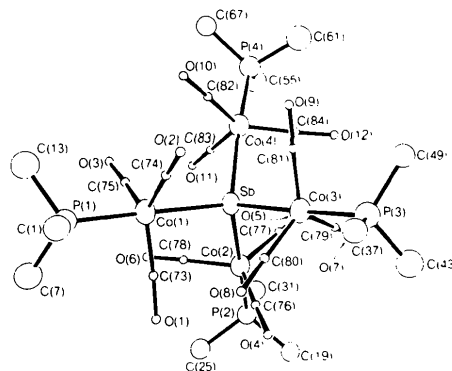


Fig. 1. Perspective view of the $\{[\text{Co}(\text{CO})_3\text{PPh}_3]_4\text{Sb}\}^+$ cation in $\{[\text{Co}(\text{CO})_3\text{PPh}_3]_4\text{Sb}\}[\text{BPh}_4] \cdot \text{CH}_2\text{Cl}_2$ drawn by ORTEP (Johnson, 1965). For clarity the phenyl groups have been omitted.

Table 2. Final fractional coordinates ($\times 10^5$ for Sb and Co and $\times 10^4$ for other atoms) and isotropic thermal parameters with e.s.d.'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso} (Å ²)		<i>x</i>	<i>y</i>	<i>z</i>	U_{iso} (Å ²)
Sb	26968 (6)	10623 (3)	24690 (7)	—	C(46)	−1310 (16)	1965 (7)	3453 (14)	122 (9)
Co(1)	39686 (12)	10664 (7)	34233 (14)	—	C(47)	−545 (17)	2030 (7)	3719 (15)	139 (10)
Co(2)	26677 (13)	16173 (6)	15197 (14)	—	C(48)	−106 (12)	1739 (7)	3952 (12)	93 (7)
Co(3)	14312 (12)	10608 (6)	34115 (13)	—	C(49)	−252 (8)	647 (4)	4005 (11)	38 (5)
Co(4)	26877 (12)	5056 (6)	15238 (13)	—	C(50)	−359 (10)	542 (5)	3181 (12)	66 (6)
O(1)	3763 (7)	1825 (4)	3596 (7)	—	C(51)	−732 (11)	216 (6)	3035 (12)	84 (7)
O(2)	3168 (6)	598 (3)	4583 (8)	—	C(52)	−1001 (11)	22 (5)	3691 (14)	83 (7)
O(3)	4898 (7)	759 (3)	2060 (8)	—	C(53)	−955 (11)	132 (5)	4492 (12)	77 (6)
O(4)	2022 (7)	2012 (3)	2913 (7)	—	C(54)	−545 (10)	445 (5)	4660 (11)	67 (6)
O(5)	1612 (7)	1260 (3)	338 (8)	—	C(55)	2623 (10)	167 (4)	−452 (10)	58 (6)
O(6)	4409 (8)	1559 (3)	1323 (7)	—	C(56)	2239 (10)	468 (5)	−681 (12)	84 (7)
O(7)	520 (7)	1362 (3)	2005 (8)	—	C(57)	2292 (11)	564 (6)	−1536 (13)	111 (8)
O(8)	2253 (6)	1538 (3)	4570 (7)	—	C(58)	2758 (12)	361 (6)	−2080 (12)	103 (8)
O(9)	1610 (6)	307 (3)	3594 (7)	—	C(59)	3131 (12)	66 (6)	−1815 (14)	105 (8)
O(10)	3433 (6)	104 (3)	2911 (6)	—	C(60)	3048 (11)	−34 (5)	−991 (13)	78 (6)
O(11)	3735 (6)	864 (3)	363 (6)	—	C(61)	1642 (10)	−193 (5)	807 (11)	66 (6)
O(12)	972 (6)	587 (3)	1383 (7)	—	C(62)	1187 (14)	−268 (6)	130 (14)	116 (8)
P(1)	5047 (3)	1079 (1)	4250 (3)	41 (2)	C(63)	397 (15)	−440 (6)	306 (16)	140 (9)
P(2)	2649 (3)	2091 (1)	710 (3)	40 (2)	C(64)	220 (13)	−513 (6)	1077 (16)	117 (9)
P(3)	322 (3)	1047 (1)	4177 (3)	47 (2)	C(65)	692 (13)	−447 (6)	1758 (14)	113 (8)
P(4)	2585 (3)	43 (1)	683 (3)	47 (2)	C(66)	1400 (10)	−280 (5)	1600 (11)	89 (7)
B	7639 (11)	3266 (5)	2470 (12)	40 (6)	C(67)	3364 (10)	−290 (5)	775 (9)	43 (5)
C(1)	4828 (9)	1055 (4)	5338 (9)	43 (5)	C(68)	3228 (10)	−637 (5)	697 (10)	58 (6)
C(2)	5266 (10)	850 (5)	5914 (12)	63 (6)	C(69)	3820 (12)	−891 (5)	732 (10)	72 (6)
C(3)	5069 (12)	841 (5)	6773 (13)	88 (7)	C(70)	4600 (12)	−762 (6)	812 (11)	79 (7)
C(4)	4385 (13)	1011 (6)	7039 (12)	90 (7)	C(71)	4767 (11)	−417 (6)	891 (11)	73 (6)
C(5)	3931 (11)	1205 (5)	6522 (13)	82 (7)	C(72)	4131 (12)	−173 (5)	876 (10)	69 (6)
C(6)	4175 (11)	1239 (5)	5666 (13)	84 (7)	C(73)	3819 (10)	1521 (6)	3517 (10)	44 (7)
C(7)	5653 (9)	1466 (4)	4142 (11)	48 (5)	C(74)	3486 (11)	775 (5)	4096 (12)	58 (7)
C(8)	6116 (10)	1613 (5)	4797 (10)	57 (6)	C(75)	4490 (11)	891 (5)	2590 (12)	52 (7)
C(9)	6544 (9)	1915 (5)	4717 (11)	52 (6)	C(76)	2294 (10)	1846 (5)	2355 (13)	49 (7)
C(10)	6551 (10)	2086 (5)	3990 (12)	62 (6)	C(77)	2029 (11)	1394 (5)	819 (12)	50 (7)
C(11)	6124 (11)	1959 (5)	3302 (11)	72 (6)	C(78)	3722 (13)	1578 (5)	1398 (10)	51 (7)
C(12)	5716 (10)	1650 (5)	3374 (11)	61 (6)	C(79)	904 (11)	1242 (5)	2549 (12)	51 (7)
C(13)	5708 (10)	705 (5)	4085 (10)	53 (6)	C(80)	1943 (11)	1351 (6)	4112 (12)	59 (8)
C(14)	6518 (12)	756 (5)	3894 (11)	74 (6)	C(81)	1543 (10)	610 (6)	3524 (11)	48 (7)
C(15)	7036 (11)	464 (6)	3866 (11)	77 (6)	C(82)	3110 (11)	265 (5)	2368 (13)	57 (7)
C(16)	6737 (12)	137 (5)	3993 (11)	75 (7)	C(83)	3305 (11)	716 (5)	832 (12)	55 (7)
C(17)	5964 (13)	86 (5)	4096 (11)	79 (7)	C(84)	1668 (13)	564 (5)	1454 (11)	55 (7)
C(18)	5428 (11)	365 (6)	4174 (10)	73 (6)	C(85)	7583 (9)	3623 (5)	1952 (11)	55 (6)
C(19)	1816 (8)	2384 (4)	893 (9)	35 (5)	C(86)	7756 (10)	3680 (5)	1113 (13)	78 (6)
C(20)	1890 (9)	2743 (4)	824 (9)	43 (5)	C(87)	7736 (10)	3995 (6)	671 (11)	75 (6)
C(21)	1238 (11)	2965 (5)	972 (10)	69 (6)	C(88)	7520 (11)	4284 (5)	1078 (14)	83 (7)
C(22)	528 (12)	2819 (6)	1200 (11)	82 (7)	C(89)	7282 (12)	4260 (6)	1925 (14)	101 (8)
C(23)	440 (11)	2459 (5)	1297 (10)	69 (6)	C(90)	7332 (11)	3926 (6)	2317 (12)	92 (7)
C(24)	1073 (10)	2244 (5)	1102 (10)	60 (6)	C(91)	7678 (9)	2921 (4)	1896 (10)	55 (6)
C(25)	3511 (9)	2362 (4)	847 (11)	41 (5)	C(92)	7125 (10)	2654 (5)	1887 (11)	69 (6)
C(26)	3927 (11)	2494 (5)	179 (11)	72 (6)	C(93)	7198 (12)	2351 (6)	1369 (14)	102 (8)
C(27)	4642 (11)	2705 (5)	269 (12)	85 (7)	C(94)	7815 (14)	2325 (6)	801 (13)	101 (8)
C(28)	4864 (10)	2761 (5)	1114 (13)	78 (7)	C(95)	8349 (11)	2584 (6)	762 (12)	81 (7)
C(29)	4450 (12)	2641 (5)	1812 (12)	75 (6)	C(96)	8309 (11)	2871 (5)	1296 (12)	76 (6)
C(30)	3800 (11)	2439 (5)	1638 (12)	68 (6)	C(97)	6858 (10)	3225 (5)	3086 (11)	58 (6)
C(31)	2604 (9)	1997 (4)	−408 (9)	43 (5)	C(98)	6852 (12)	3097 (5)	3877 (13)	85 (7)
C(32)	2084 (10)	2174 (5)	−963 (12)	74 (6)	C(99)	6173 (14)	3065 (5)	4372 (12)	95 (7)
C(33)	2049 (11)	2085 (5)	−1809 (13)	80 (7)	C(100)	5464 (12)	3152 (5)	4034 (13)	88 (7)
C(34)	2530 (12)	1813 (6)	−2088 (11)	80 (7)	C(101)	5395 (11)	3280 (5)	3243 (13)	76 (6)
C(35)	3035 (10)	1630 (5)	−1585 (13)	72 (6)	C(102)	6077 (12)	3322 (5)	2780 (11)	78 (7)
C(36)	3064 (9)	1724 (5)	−736 (11)	56 (6)	C(103)	8399 (12)	3271 (6)	3116 (12)	79 (7)
C(37)	488 (10)	1077 (4)	5304 (9)	42 (5)	C(104)	8618 (14)	3550 (6)	3579 (15)	122 (9)
C(38)	1179 (9)	965 (4)	5679 (11)	45 (5)	C(105)	9321 (17)	3572 (7)	4172 (16)	153 (11)
C(39)	1304 (10)	990 (5)	6534 (12)	70 (6)	C(106)	9768 (14)	3293 (8)	4206 (15)	125 (9)
C(40)	702 (12)	1136 (5)	7041 (11)	77 (7)	C(107)	9689 (17)	3029 (8)	3674 (19)	162 (11)
C(41)	−4 (11)	1240 (5)	6697 (12)	74 (6)	C(108)	8998 (17)	3013 (7)	3145 (15)	139 (9)
C(42)	−105 (10)	1212 (5)	5806 (12)	71 (6)	C(109)	7990 (26)	1357 (12)	936 (26)	332 (21)
C(43)	−350 (11)	1404 (5)	3943 (10)	57 (6)	Cl(1)	6942 (9)	1245 (4)	1450 (9)	400 (8)
C(44)	−1119 (14)	1353 (6)	3652 (12)	98 (7)	Cl(2)	8342 (9)	974 (4)	1421 (9)	403 (8)
C(45)	−1632 (13)	1646 (7)	3467 (13)	115 (8)					

Table 3. Selected interatomic distances (Å) and angles (°)

Sb—Co(1)	2.594 (2)	C(73)—O(1)	1.17 (3)
Sb—Co(2)	2.593 (3)	C(74)—O(2)	1.16 (2)
Sb—Co(3)	2.602 (2)	C(75)—O(3)	1.20 (2)
Sb—Co(4)	2.594 (3)	C(76)—O(4)	1.18 (2)
Mean	2.596	C(77)—O(5)	1.15 (2)
		C(78)—O(6)	1.16 (2)
Co(1)—P(1)	2.216 (5)	C(79)—O(7)	1.16 (2)
Co(2)—P(2)	2.213 (5)	C(80)—O(8)	1.14 (2)
Co(3)—P(3)	2.226 (5)	C(81)—O(9)	1.16 (3)
Co(4)—P(4)	2.212 (5)	C(82)—O(10)	1.18 (2)
Mean	2.217	C(83)—O(11)	1.18 (2)
		C(84)—O(12)	1.17 (2)
Co(1)—C(73)	1.75 (2)	Mean	1.17
Co(1)—C(74)	1.74 (2)		
Co(1)—C(75)	1.73 (2)	B—C(85)	1.59 (3)
Co(2)—C(76)	1.71 (2)	B—C(91)	1.60 (3)
Co(2)—C(77)	1.75 (2)	B—C(97)	1.64 (3)
Co(2)—C(78)	1.78 (2)	B—C(103)	1.62 (3)
Co(3)—C(79)	1.76 (2)	Mean	1.61
Co(3)—C(80)	1.78 (2)		
Co(3)—C(81)	1.73 (2)	C(109)—Cl(1)	1.98 (5)
Co(4)—C(82)	1.76 (2)	C(109)—Cl(2)	1.75 (5)
Co(4)—C(83)	1.71 (2)		
Co(4)—C(84)	1.72 (2)	Mean	1.38
Mean	1.74	C—C(phenyl)	
P(1)—C(1)	1.77 (2)		
P(1)—C(7)	1.80 (2)		
P(1)—C(13)	1.82 (2)		
P(2)—C(19)	1.81 (2)		
P(2)—C(25)	1.78 (2)		
P(2)—C(31)	1.81 (2)		
P(3)—C(37)	1.81 (2)		
P(3)—C(43)	1.80 (2)		
P(3)—C(49)	1.82 (2)		
P(4)—C(55)	1.86 (2)		
P(4)—C(61)	1.83 (2)		
P(4)—C(67)	1.82 (2)		
Mean	1.81		
Co(1)—Sb—Co(2)	110.11 (8)	Sb—Co(1)—C(73)	86.5 (5)
Co(1)—Sb—Co(3)	109.23 (8)	Sb—Co(1)—C(74)	88.4 (6)
Co(1)—Sb—Co(4)	109.98 (8)	Sb—Co(1)—C(75)	88.1 (6)
Co(2)—Sb—Co(3)	108.99 (8)	Sb—Co(2)—C(76)	88.2 (7)
Co(2)—Sb—Co(4)	109.20 (8)	Sb—Co(2)—C(77)	89.0 (6)
Co(3)—Sb—Co(4)	109.31 (8)	Sb—Co(2)—C(78)	89.0 (6)
Mean	109.47	Sb—Co(3)—C(79)	87.4 (6)
		Sb—Co(3)—C(80)	88.3 (6)
Sb—Co(1)—P(1)	178.9 (3)	Sb—Co(3)—C(81)	88.5 (6)
Sb—Co(2)—P(2)	179.8 (2)	Sb—Co(4)—C(82)	89.2 (7)
Sb—Co(3)—P(3)	177.7 (2)	Sb—Co(4)—C(83)	89.3 (6)
Sb—Co(4)—P(4)	175.5 (2)	Sb—Co(4)—C(84)	86.1 (6)
Mean	178.0	Mean	88.2
P(1)—Co(1)—C(73)	92.5 (6)	C(73)—Co(1)—C(74)	120.5 (9)
P(1)—Co(1)—C(74)	91.7 (6)	C(73)—Co(1)—C(75)	121.4 (8)
P(1)—Co(1)—C(75)	92.7 (6)	C(74)—Co(1)—C(75)	117.6 (9)
P(2)—Co(2)—C(76)	91.9 (7)	C(76)—Co(2)—C(77)	120.9 (9)
P(2)—Co(2)—C(77)	91.2 (6)	C(76)—Co(2)—C(78)	119.8 (8)
P(2)—Co(2)—C(78)	90.8 (6)	C(77)—Co(2)—C(78)	119.1 (8)
P(3)—Co(3)—C(79)	91.3 (6)	C(79)—Co(3)—C(80)	118.4 (9)
P(3)—Co(3)—C(80)	94.0 (6)	C(79)—Co(3)—C(81)	121.3 (8)
P(3)—Co(3)—C(81)	90.5 (6)	C(80)—Co(3)—C(81)	119.9 (9)
P(4)—Co(4)—C(82)	94.2 (7)	C(82)—Co(4)—C(83)	119.4 (9)
P(4)—Co(4)—C(83)	91.7 (7)	C(82)—Co(4)—C(84)	120.2 (8)
P(4)—Co(4)—C(84)	89.6 (6)	C(83)—Co(4)—C(84)	120.0 (9)
Mean	91.8	Mean	119.9

and a mean Co—Sb—Co angle of 109.5°. Thus, the Sb—Co distances are longer than the 2.480 (1) Å in dicarbonyl(nitrosyl)(triphenylantimonio)cobalt(0) (Gilli, Sacerdoti & Domiano, 1974) but close to the average

Table 3 (cont.)

Co(1)—C(73)—O(1)	176 (1)	Co(1)—P(1)—C(1)	113.6 (5)
Co(1)—C(74)—O(2)	176 (2)	Co(1)—P(1)—C(7)	114.6 (6)
Co(1)—C(75)—O(3)	175 (2)	Co(1)—P(1)—C(13)	112.8 (6)
Co(2)—C(76)—O(4)	178 (2)	Co(2)—P(2)—C(19)	114.5 (5)
Co(2)—C(77)—O(5)	177 (2)	Co(2)—P(2)—C(25)	113.2 (6)
Co(2)—C(78)—O(6)	179 (2)	Co(2)—P(2)—C(31)	114.0 (6)
Co(3)—C(79)—O(7)	176 (2)	Co(3)—P(3)—C(37)	114.7 (6)
Co(3)—C(80)—C(8)	178 (2)	Co(3)—P(3)—C(43)	113.0 (6)
Co(3)—C(81)—O(9)	179 (2)	Co(3)—P(3)—C(49)	112.2 (5)
Co(4)—C(82)—O(10)	176 (2)	Co(4)—P(4)—C(55)	112.3 (6)
Co(4)—C(83)—O(11)	179 (2)	Co(4)—P(4)—C(61)	112.8 (6)
Co(4)—C(84)—O(12)	176 (2)	Co(4)—P(4)—C(67)	117.0 (5)
Mean	177	Mean	113.7
C(85)—B—C(91)	114 (1)	Cl(1)—C(109)—Cl(2)	86 (2)
C(85)—B—C(97)	110 (1)	Mean P—C—C(phenyl)	121
C(85)—B—C(103)	111 (2)	Mean C—P—C(phenyl)	104.9
C(91)—B—C(97)	107 (1)	Mean C—C—C(phenyl)	120
C(91)—B—C(103)	109 (1)	Mean B—C—C	124
C(97)—B—C(103)	104 (2)		
Mean	109		

of 2.614 (2) Å found in $\text{Co}_4(\text{CO})_{12}\text{Sb}_4$ (Foust & Dahl, 1970). Around each Co atom there is trigonal-bipyramidal geometry with three CO groups in the equatorial plane and the Sb and P atoms in the axial positions. Mean bond distances are Co—P = 2.217 (6), Co—C(carbonyl) = 1.74 (2) and C—O = 1.17 (2) Å. The ranges of the angles are Sb—Co—P = 175.5 (2)–179.8 (2) (mean = 178.0), Sb—Co—C = 86.1 (6)–89.3 (6) (mean = 88.2), P—Co—C = 89.6 (6)–94.2 (7) (mean = 91.8) and C—Co—C = 117.6 (9)–121.4 (8)° (mean = 119.9°). The carbonyl C atoms are slightly displaced towards the Sb atom; all the Sb—Co—C angles are acute while all the P—Co—C angles are obtuse. There is no significant deviation of the CO groups from linearity; the Co—C—O angles vary from 175 (2) to 179 (2)° with a mean of 177°.

Distances and angles in the $[\text{BPh}_4]$ anion are normal with a mean B—C distance of 1.61 Å and a mean C—B—C angle of 109°.

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