

TRIMETHYLPHOSPHINECOBALT(I) COMPLEXES: CRYSTAL AND MOLECULAR STRUCTURE OF $[\text{Co}(\text{CO})_2(\text{PMe}_3)_3]\text{BPh}_4$

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Summary

Crystals of dicarbonyltris(trimethylphosphine)cobalt(I) tetraphenylborate belong to the monoclinic space group $P2_1/c$ with a 10.135(4), b 12.630(4), c 29.35(1) Å, β 106.11(3)° and $Z = 4$. The structure was refined on 2696 nonzero Cu- K_α reflections to $R = 0.043$. The unit-cell contains discrete BPh_4^- anions and $[\text{Co}(\text{CO})_2(\text{PMe}_3)_3]^+$ cations. The coordination geometry of the cation lies roughly halfway between a trigonal bipyramid (with equatorial CO ligands) and a square pyramid (with *trans* basal CO ligands), defining an intermediate configuration along the interconversion pathway in the Berry process. The Co–P distance (2.246 Å) to the unique PMe_3 ligand (apical in the square pyramid or equatorial in the trigonal bipyramid) is longer than the other two (2.209 and 2.211 Å). The Co–CO bond lengths are 1.740 and 1.766 Å. The $\nu(\text{CO})$ infrared bands indicate that the same geometry is retained in CH_2Cl_2 and acetone.

Introduction

The trimethylphosphinecobalt(I) complexes $\text{CoX}(\text{PMe}_3)_3$ ($X = \text{halide}$) and $[\text{Co}(\text{PMe}_3)_4]\text{BPh}_4$ display interesting reactivity toward π -bonding ligands, forming cationic Co^I species $[\text{Co}(\text{PMe}_3)_3\text{L}_x]^+$ with ligands L such as alkenes, alkynes and dienes [1]. The same starting materials also react with CO, but most of the known mixed species $[\text{Co}(\text{CO})_x(\text{PR}_3)_{5-x}]^+$ have been obtained by other synthetic routes [2]. Stoichiometry depends on the PR_3 ligand used and no complete series of mixed species ($1 \leq x \leq 4$) has apparently been reported so far for any given PR_3 ligand.

Structural characterization of carbonyl complexes has generally been based on infrared or NMR spectroscopy. However, these results leave room for both specula-

TABLE 2

DIHEDRAL ANGLES ($^{\circ}$) FOR $[\text{Co}(\text{CO})_2(\text{PMe}_3)_3]^+$ COMPARED WITH THOSE CALCULATED FOR IDEAL TBP AND SP GEOMETRIES

Dihedral angles	Ideal TBP	Ideal SP ^a	Exper.
P(2)P(3)C(2)/P(2)C(2)C(1)	78.5	61.5	68.7
P(2)P(3)C(2)/P(2)C(1)P(3)	78.5	103.1	93.2
P(2)C(1)P(3)/P(2)C(2)C(1)	78.5	61.5	70.2
P(1)C(2)C(1)/P(1)P(3)C(2)	78.5	61.5	69.3
P(1)C(1)P(3)/P(1)P(3)C(2)	78.5	103.1	93.0
P(1)C(1)P(3)/P(1)C(2)C(1)	78.5	61.5	70.0
P(2)P(3)C(2)/P(1)P(3)C(2)	126.9	103.1	113.7
P(2)C(1)P(3)/P(1)C(1)P(3)	126.9	103.1	112.5
P(2)C(2)C(1)/P(1)C(2)C(1)	126.9	180.0	156.5

^a Ideal SP: P(3) apical, P(3)-Co-P and P(3)-Co-C 105° .

this procedure is found to favor, but only slightly, a square pyramid with an angle of 105.0° (rms deviation 5.5°) over the trigonal bipyramid (rms deviation 8.0°).

$[\text{Co}(\text{CO})_2(\text{PMe}_3)_2(\text{N}_3)]$ [8] and two $[\text{NiX}_2(\text{CO})(\text{PMe}_3)_2]$ compounds ($X = \text{Cl}$ [9] and I [10]) are the only other mixed CO/PMe₃ complexes of first row d^8 atoms for which X-ray results are available. All three are virtually undistorted TBP with axial PMe₃ ligands. When the present $[\text{Co}(\text{CO})_2(\text{PMe}_3)_3]^+$ ion is regarded as a distorted TBP, its Co-P_{ax} bonds are found to be longer than those observed for the N₃⁻ complex (mean 2.188(3) Å). The Co-CO distances (1.740(6) and 1.766(5) Å) show a similar tendency (1.739(6) and 1.749(5) Å [8]). This may be ascribed to the greater steric effect of the equatorial PMe₃ ligand compared with N₃⁻. Although the Ni-P_{ax} distances found in the $[\text{NiX}_2(\text{CO})(\text{PMe}_3)_2]$ compounds are similar to those observed here for $[\text{Co}(\text{CO})_2(\text{PMe}_3)_3]^+$, the Ni-CO bonds (1.730(2) Å) are shorter than our Co-CO bonds, in spite of the fact that Co^I should produce better back-bonding than Ni^{II}. However, except for CO, these compounds have different types of ligands in the equatorial plane. In the Ni^{II} complexes, the metal back-bonding capability is used to stabilize only one CO ligand and it is probably helped in this respect by some halide-to-metal π -donation. In the Co compound, back-bonding is shared by two equatorial CO ligands and the third ligand, being a phosphine, is unable to π -donate to the metal.

The coordinated CO molecules are linearly bonded as usual (Table 1) and the C-O distances (1.138(7) Å) are normal. The Co-P bond (2.246(2) Å) to the unique PMe₃ ligand (which would be equatorial in a TBP and apical in a SP) is longer than the other two (mean 2.210(2) Å) in agreement with theoretical predictions for d^8 systems [5]. The P-C distances in the coordinated PMe₃ ligands (mean 1.811(7) Å) are similar to those found in other Co^I complexes [1,8]. The P-CH₃ bonds in phosphines P(1) and P(2) are staggered with respect to the underlying Co-ligand bonds in the $\text{Co}(\text{CO})_2\text{P}(3)$ plane. The C-P-C angles for phosphines P(1) and P(2) (mean 103.3(4) Å) are greater than those of phosphine P(3) (mean 100.9(4) $^{\circ}$), showing the strain imposed on the latter by steric hindrance. Details on the geometry of the BPh₄⁻ ion, which shows no unusual features, are provided in the supplementary material.

Besides confirming the five-coordination of cobalt, the present X-ray work yielded a OC-Co-CO angle ($137.9(3)^{\circ}$) equal to the 2θ value inferred from the

infrared results in solution. This suggests that the structure observed in the solid is retained in solution.

There are no available crystallographic studies on other $[\text{Co}(\text{CO})_2(\text{PR}_3)_3]^+$ systems with non-chelating phosphines, which could provide insight into the steric effect of R. Several $[\text{Co}(\text{CO})_3(\text{PPh}_3)\text{L}]$ compounds (with $\text{L} = \text{C}_2\text{F}_5$ [11], GePh_3 [12] and $\text{SnR}^1\text{R}^2\text{R}^3$ [13]), as well as the recently reported $[\text{Co}(\text{CO})_2(\text{PMe}_3)_2(\text{N}_3)]$ compound [8], all display almost perfect TBP structures. From spectroscopic results, DuBois and Meek [3] showed that bis(diphenylphosphinoethyl)phenylphosphine and bis(diphenylphosphinopropyl)phenylphosphine both form TBP $[\text{Co}(\text{CO})_2(\text{P}-\text{P})]^+$ cations, with two equatorial CO ligands for the latter phosphine, but one equatorial and one axial for the former. However, their crystal structures were not determined. More X-ray diffraction results are available for isoelectronic Fe^0 systems. A series of $[\text{Fe}(\text{CO})_3(\text{PR}_3)_2]$ [14,15], $[\text{Fe}(\text{CO})_2(\text{PR}_3)_2\text{L}]$ (with $\text{L} =$ small ligand like SO_2 , CS , N_2 , PhNN) [16–18] and $[\text{Fe}(\text{CO})_4\text{PR}_3]$ compounds [14,19,20] form TBP molecules with equatorial CO and axial PR_3 ligands. The preference of CO for equatorial sites is therefore supported by the bulk of these results. However, displacement of CO to axial sites is one of the mechanisms whereby steric hindrance can be relieved, as shown by the structures of two related $[\text{Fe}(\text{CO})_2(\text{PR}_3)_2(\text{SO}_2)]$ complexes: the compound with $\text{R} =$ phenoxide shows the normal axial PR_3 -equatorial CO distribution, whereas for the bulkier substituent $\text{R} = o$ -methylphenoxide, the opposite distribution is found [18]. A series of $\text{Ni}(\text{CN})_2(\text{PR}_3)_3$ complexes also show this ligand distribution [21]. A more common mechanism to reduce steric hindrance is the one observed here for $[\text{Co}(\text{CO})_2(\text{PMe}_3)_3]^+$ and for the isoelectronic $[\text{Fe}(\text{CO})_2(\text{P}(\text{OMe})_3)_3]$ compound [17]. In both cases, the CO ligands remain equatorial in the distorted TBP. Steric repulsion between the equatorial PR_3 ligand and those in axial positions displaces the latter in the opposite direction, so that the tilted $\text{M}-\text{P}_{\text{ax}}$ bonds project roughly on the bisector of the $\text{OC}-\text{M}-\text{CO}$ bonds. Also, the equatorial $\text{M}-\text{CO}$ bonds have been displaced in opposite directions. The extent of distortion from ideal TBP geometry can be measured from the $\text{P}_{\text{ax}}-\text{M}-\text{P}_{\text{ax}}$ and the $\text{OC}-\text{M}-\text{CO}$ angles, which are $162.32(7)$ and $137.9(3)^\circ$, respectively, in the Co^I cation. The distortions are large and so distributed around the metal centre that a SP gives a slightly better description of the structure than the TBP. Distortion is smaller in the Fe^0 compound, as can be appreciated from the corresponding angles of $168.3(1)^\circ$ and $130.0(5)^\circ$, respectively. This time, the structure remains on the TBP side. This is in good agreement with the respective sizes of these PR_3 ligands as measured from their cone angles (PMe_3 118° , $\text{P}(\text{OMe})_3$ 107°) [22]. The increase of the $\text{OC}-\text{M}-\text{CO}$ angle above 120° could be considered as resulting from steric hindrance between the equatorial CO ligands and the tilted axial PR_3 molecules. An alternate explanation would be the presence of a low-energy interconversion between axial and equatorial ligands by the Berry process (with $\text{Co}-\text{P}(3)$ as pivot) [6], allowing stabilization of the molecule at some step along the pathway, depending on the type of ligand. However, other effects may also have to be taken into account, since in $\text{NiBr}_2(\text{PMe}_3)_3$ [23] and $\text{NiI}_2[\text{P}(\text{OMe}_3)_3]_3$ [24], where the $\text{P}_{\text{ax}}-\text{Ni}-\text{P}_{\text{ax}}$ angles range from 167.3 to 174.3° , the $\text{X}-\text{Ni}-\text{X}$ angles are found to be in the 112.1 – 117.6° range.

In summary, the $[\text{Co}(\text{CO})_2(\text{PMe}_3)_3]^+$ cation displays in CD_2Cl_2 and acetone solution, and in the solid BPh_4^- salt, a geometry roughly halfway between the

regular trigonal bipyramid and the square pyramid. It might thus correspond to an intermediate configuration along the pathway of the Berry process [6], whereby smooth interconversion of the axial and equatorial ligands is responsible for the fluxionality of these molecules.

Experimental

Preparation

All the reactions were performed under an atmosphere of dry argon. The solvents were freshly distilled and deoxygenated prior to use. PMe_3 [25] and $\text{CoBr}(\text{PMe}_3)_3$ [2] were prepared as reported in the literature.

$[\text{Co}(\text{CO})_2(\text{PMe}_3)_3]\text{BPh}_4$. A solution of $\text{CoBr}(\text{PMe}_3)_3$ (1 g; 2.7 mmol) in 10 ml of acetonitrile is allowed to react with CO in excess. The violet solution turns brown. Addition of NaBPh_4 (0.92 g; 2.7 mmol) in methanol (30 ml) precipitates yellow crystals (yield, 90%), which are filtered off and dried under CO. Recrystallization in methanol at -20°C gives yellow single crystals suitable for X-ray work. Anal. Found: C, 62.80; H, 6.90. $\text{C}_{35}\text{H}_{47}\text{BCoO}_2\text{P}_3$ calc.: C, 63.46; H, 7.15%.

Physical measurements

Infrared spectra were recorded as Nujol mulls between KBr plates or as dichloromethane or acetone solutions, with a Perkin-Elmer 983 spectrophotometer. $^{31}\text{P}\{^1\text{H}\}$ NMR spectra were recorded on a Bruker 250 MHz spectrometer, operated at 101.202 MHz, using a deuterium lock and 85% H_3PO_4 in D_2O as standard. Elemental microanalyses were performed by the Laboratoire de Microanalyse du CNRS, Lyon.

Crystal data. $\text{C}_{35}\text{H}_{47}\text{BCoO}_2\text{P}_3$; M_w 662.43; monoclinic, space group $P2_1/c$, a 10.135(4), b 12.630(4), c 29.35(1) Å, β 106.11(9)°, V 3609.4 Å³, $Z = 4$, D_{calc} 1.219 g cm⁻³, $\lambda(\text{Cu-K}\alpha)$ 1.54178 Å (graphite monochromator), $\mu(\text{Cu-K}\alpha)$ 53.91 cm⁻¹, $F(000) = 1400$. T 298 K.

Crystal structure determination

The specimen used had the following dimensions between the indicated pairs of faces: 0.045 mm (001–00 $\bar{1}$) \times 0.11 mm ($1\bar{1}\bar{2}$ – $\bar{1}12$) \times 0.21 mm ($11\bar{2}$ – $\bar{1}12$). Space group, cell dimensions and intensity data were obtained as described previously [26]. A total of 6863 independent reflections (hkl , $h\bar{k}l$, $2\theta \leq 140^\circ$) were collected with a CAD4 diffractometer. The fluctuations of the standard reflections remained within $\pm 2\%$ during the experiment. A set of 2696 reflections significantly above background ($I > 3\sigma(I)$) was retained for structure determination. These data were corrected for the Lorentz effect, polarization and absorption (Gaussian integration, grid $10 \times 10 \times 10$, transmission range 0.27–0.65).

The positions of all non-hydrogen atoms except the CO ligands were determined by MULTAN [27]. The CO molecules were then found from a difference Fourier (ΔF) map. The structure was refined on $|F_o|$ by full-matrix least-squares procedures in the early stages. Anisotropic refinement of Co and P atoms, and isotropic refinement of the remaining non-hydrogen atoms converged to $R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.087$ and $R_w = [\Sigma w(|F_o| - |F_c|)^2 / \Sigma w |F_o|^2]^{1/2} = 0.099$. Refinement was continued by block-diagonal least squares. The phenyl hydrogens were fixed at idealized positions with isotropic B values of 5.5 Å². Most of the methyl

TABLE 3

REFINED FRACTIONAL COORDINATES ($\times 10^4$, Co, P $\times 10^5$) AND EQUIVALENT TEMPERATURE FACTORS ($\times 10^3$)

Atom	x	y	z	U_{eq}
Co	52523(8)	24408(6)	13715(3)	40
P(1)	45775(14)	8821(11)	10494(6)	54
P(2)	65490(15)	37569(11)	17437(5)	48
P(3)	31907(14)	32334(11)	12149(5)	44
O(1)	5829(6)	1358(4)	2268(2)	135
O(2)	6558(4)	2771(3)	619(1)	73
C(1)	5609(6)	1796(4)	1916(2)	79
C(2)	6052(5)	2640(4)	915(2)	47
C(11)	5965(6)	-71(4)	1195(3)	95
C(12)	4045(7)	819(5)	409(2)	95
C(13)	3204(6)	223(4)	1224(2)	72
C(14)	8291(6)	3319(5)	1994(2)	94
C(15)	6739(5)	4903(4)	1401(2)	57
C(16)	6104(7)	4303(5)	2254(2)	79
C(17)	3109(6)	4659(4)	1156(2)	77
C(18)	2320(7)	3012(6)	1671(2)	95
C(19)	1856(5)	2871(4)	682(2)	55
C(31)	-1553(4)	2567(3)	-1115(2)	34
C(32)	-2660(5)	2536(4)	-1519(2)	42
C(33)	-4021(5)	2591(4)	-1496(2)	55
C(34)	-4315(5)	2672(4)	-1073(2)	55
C(35)	-3243(6)	2667(4)	-663(2)	57
C(36)	-1895(5)	2624(4)	-685(2)	46
C(41)	1109(4)	3204(4)	-735(2)	31
C(42)	723(5)	4173(4)	-587(2)	43
C(43)	1760(5)	4876(4)	-309(2)	55
C(44)	3015(5)	4617(4)	-153(2)	50
C(45)	3452(5)	3657(4)	-283(2)	46
C(46)	2503(5)	2965(4)	-569(2)	38
C(51)	387(4)	1176(3)	-1017(2)	33
C(52)	224(5)	403(4)	-1367(2)	45
C(53)	451(5)	-677(4)	-1263(2)	59
C(54)	818(5)	-1007(4)	-804(2)	58
C(55)	968(5)	-289(4)	-448(2)	58
C(56)	751(5)	780(4)	-555(2)	48
C(61)	314(5)	2828(4)	-1630(2)	36
C(62)	-379(5)	3661(4)	-1899(2)	46
C(63)	-59(6)	4044(4)	-2300(2)	58
C(64)	994(6)	3591(5)	-2443(2)	68
C(65)	1722(5)	2772(4)	-2184(2)	61
C(66)	1386(5)	2392(4)	-1789(2)	49
B	44(5)	2429(4)	-1129(2)	33

hydrogens of the PMe_3 ligands were visible on the ΔF map. They were fixed at idealized positions (C-H 0.95 Å, sp^3 hybridization, B 7.0 Å²) giving the best fit with those observed in the ΔF map. The hydrogen parameters were not refined, but the coordinates were recalculated after each least-squares cycles. All non-hydrogen atoms were refined anisotropically. The final residuals were $R = 0.043$ and $R_w = 0.046$. The goodness-of-fit ratio was 1.31 for 380 parameters varied. The general background on the final ΔF map was below $\pm 0.22 \text{ e \AA}^{-3}$.

The refined coordinates are listed in Table 3. Lists of temperature factors, calculated hydrogen coordinates and structure factors have been deposited *. The scattering curves and the contributions of Co and P to anomalous dispersion were from standard sources [28].

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