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Preparation and Structure of Bis(trifluoroacetato)dicarbonyl bis(triethylphosphine)molybdenum(II)

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Abstract

The $Mo(CO)_2(PEt_3)_2(CF_3COO)_2$ complex, obtained by prolonged reaction of trifluoroacetic acid with $Mo(CO)_3(PEt_3)_3$ in dichloromethane, was characterized by spectroscopic methods and by X-ray crystal structure determination. The crystals belong to the monoclinic space group *Cc*, with $a = 20.037$, $b = 10.879$, $c = 15.176$ Å, $\beta = 129.28°$ and $Z = 4$. The structure was refined on 2135 observed reflections to $R = 0.047$. The Mo(II) atom is 7-coordinated and has a capped trigonal prism geometry, the capping position being occupied by a monodentate CF_3CO_2 ligand. The quadrangular plane contains one CO, one PEt_3 and one bidentate CF_3CO_2 ligand. The remaining CO and PEt_3 molecules are found on the opposite edge.

Introduction

The importance of carboxylate ligands in transition metal chemistry is well known, and numerous strategies have been devised for introducing this ligand into metal complexes [1]. As part of our continuing interest for mixed CO–phosphine–carboxylate compounds of Mo and W, we previously reported on the preparation and X-ray structure determination of $Mo(CO)_2(PMe_3)_3(CF_3CO_2)_2$ [2] and $Mo_2(\mu\text{-}P(O)(OCH_3)_2)_2(CO)_4[P(OCH_3)_3]_2(CF_3COO)_2$ [3]. We now describe the preparation of $Mo(CO)_2(PEt_3)_2(CF_3COO)_2$. The presence of only two phosphine ligands instead of three [2] suggested a different binding type for the carboxylate ligand, which prompted us to undertake a crystallographic study on this compound.

Experimental

Preparation of $Mo(CO)_2(PEt_3)_2(CF_3COO)_2$

1.6 g (2.98 mmol) of $Mo(CO)_3(PEt_3)_3$ was stirred in 25 ml of CH_2Cl_2. To the colorless solution was added an excess (2 ml, 22.8 mmol) of CF_3COOH, whereupon the reaction solution immediately turned blue–green. The solvent was removed under vacuum, and the residue was dissolved in 25 ml of CH_2Cl_2 and stirred for 24 h under nitrogen. Filtration gave an orange solution from which the solvent was removed under vacuum. The residue was dissolved in an ethanol/CH_2Cl_2 mixture. Cooling to −20 °C yielded orange crystals of the title compound (12% yield based on $Mo(CO)_3(PEt_3)_3$). *Anal.* Calc. for $C_{18}H_{30}F_6MoO_6P_2$: H, 4.92, C, 35.19; P, 10.08; F, 18.56. Found: H, 4.94; C, 35.35; P, 9.20; F, 19.20%. ^1H NMR (CD_2Cl_2, 300 K, *vs.* Me_4Si (δ 0)): δ 2.159, $J = 7.7$ Hz; δ 2.119, $J = 7.7$ Hz; δ 1.22 t, $J = 7.6$ Hz; δ 1.15 t, $J = 7.6$ Hz; $^{31}P\{^1H\}$ NMR (CD_2Cl_2, 300 K, *vs.* 85% H_3PO_4 (δ 0)): δ 55.38. IR wavenumbers (cm^{-1}), KBr: 1945 m, 1855 s, 1700 m, 1610 w.

Crystal Data

$C_{18}H_{30}F_6MoP_2O_6$, formula weight = 614.31, monoclinic, *Cc*, $a = 20.037(12)$, $b = 10.879(3)$, $c = 15.176(5)$ Å, $\beta = 129.28(4)°$, $V = 2560.7$ Å^3, $Z = 4$, $D_c = 1.593$ g cm^{-3}, λ(Cu K$\bar{\alpha}$) = 1.54178 Å (graphite monochromator), μ(Cu Kα) = 61.65 cm^{-1}, $T = 298$ K.

Crystallographic Measurements and Structure Determination

The crystal, obtained as described above, was mounted in a Lindemann capillary filled with nitrogen. The specimen used had the following dimensions (mm) between the indicated pairs of faces: $0.55(11\bar{1}-\bar{1}\bar{1}1) \times 0.57(001-00\bar{1}) \times 0.36(1\bar{1}\bar{1}-\bar{1}11)$.

A set of precession and cone axis photographs showed monoclinic Laue symmetry, and the systematic absences (hkl, $h + k \neq 2n$; $h0l$, $l \neq 2n$)

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were consistent with space groups $C2/c$ (centric) and Cc (non-centric). The intensity data were collected with an Enraf-Nonius CAD-4 diffractometer, according to the procedure described earlier [4]. The intensities of three standard reflections showed random fluctuation within ±2.9% during data collection. A total of 2432 independent hkl and $hk\bar{l}$ Cu Kα reflections with $h + k = 2n$ were collected within a sphere limited by $2\theta = 140°$. On the basis of the criterion $I > 5\ \sigma(I)$, a set of 2135 independent 'observed' reflections was retained to solve and refine the structure. The data were corrected for the Lorentz effect, polarization and absorption (Gaussian integration, grid $8 \times 8 \times 8$, transmission range: 0.058–0.254).

The structure was solved by the heavy-atom method, but pseudo-symmetry introduced serious complications. In the centric $C2/c$ unit cell, the four Mo atoms would occupy a special position. The Patterson map indicated that it would lie on the crystallographic two-fold axis (equipoint 4e) at $y \sim 0.30$. A difference Fourier (ΔF) map phased on Mo revealed the positions of the CO ligand and the P atom. However, the map failed to show unambiguous positions for the remaining atoms. Ethyl groups attached to P could be assembled, but each appeared as two superimposed images. A similar pair of images could be recognized for the CCO_2 fragment of the carboxylate. In order to check for possible disorder, we introduced in the phasing model these pairs of images (with 50% occupancy, as suggested by peak heights) for the ethyl groups, or the CCO_2 fragment, or both at the same time. Calculation of ΔF maps in all cases did not improve the definition of the atoms not yet introduced in the phasing model. Furthermore, in no case was it possible to find reasonable positions for the carboxylate F atoms. Since it was unlikely that the structure be so severely disordered, resolution in the non-centric space group was undertaken.

In the Cc unit cell, the Mo atom occupies a general position. The y coordinate deduced from the Patterson map is the same as above, whereas the x and z coordinates can be chosen arbitrarily to fix the origin. For convenience, the Mo position used for the $C2/c$ cell was retained. The CO molecules and P atoms, which existed as symmetry-equivalent pairs in $C2/c$, are now symmetry independent and have to be described individually. The starting model consisted of the $Mo(CO)_2P_2$ fragment, together with an ethyl group corresponding to one of the images mentioned above. At this point, the Mo and P atoms were refined isotropically by the usual least-squares procedure (on $|F_o|$, full matrix), and a new ΔF map was calculated. Although this map still contained pairs of superimposed images for the ethyl and carboxylate regions, one of the images had become stronger. An extra ethyl group, which could safely be assigned to the stronger image, was added to the phasing model. A few cycles of isotropic refinement of Mo and P were run, followed by a ΔF map calculation. By repeating this procedure, the six ethyl groups and the two CCO_2 fragments were progressively defined. They showed no evidence for disorder. However, disorder remained for the carboxylate F atoms. At this stage, all non-hydrogen atoms were isotropically refined. The subsequent ΔF map showed six peaks at suitable distances from the terminal C atom of each carboxylate, and their positions were consistent with two orientations related by a $\sim$60° rotation of the $-CF_3$ group about the C–C bond. These twelve 'half-fluorine' atoms were introduced into the structure with occupancy factors of 0.50. Isotropic refinement of all non-hydrogen atoms converged to $R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.101$ and $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2} = 0.124$. The methylene hydrogen atoms were placed at the calculated positions (C–H = 0.95 Å, sp^2 hybridization, $B = 6.5$ Å^2). Their parameters were not refined, but their coordinates were recalculated after each least-squares cycle. The methyl hydrogens could not be found in the ΔF map. The structure was then refined anisotropically (block-diagonal least squares). The final residuals were $R = 0.047$ and $R_w = 0.063$, whereas the goodness-of-fit ratio was 2.46 for 351 parameters varied.

At the end of the refinement, a few extra cycles were run in which the occupancy factors for the disordered F atoms were allowed to refine simultaneously with the other parameters. The refined values were uniformly distributed in the 0.41–0.59 range ($\sigma = 0.10$), and there was no trend for the higher values to belong to a given orientation of the $-CF_3$ group. Therefore, it was concluded that both orientations are equally populated, and occupancy factors of 0.50 were retained.

The refined coordinates are listed in Table I (see also 'Supplementary Material'). The scattering curves were from standard sources [5]. The contributions of Mo and P to anomalous dispersion [6] were taken into account. The programs used are listed elsewhere [7].

Results and Discussion

Reacting CF_3COOH with $Mo(CO)_3(PR_3)_3$ (where $R = CH_3$) was found [2] to lead to the new 7-coordinated complex $Mo(CO)_2(PMe_3)_3(CF_3COO)_2$, which contains two monodentate trifluoroacetate ligands. When $R = -OCH_3$, a cleavage of the $O-CH_3$ linkage was observed and the new heptacoordinate dinuclear complex $Mo_2[\mu\text{-}P(O)(OCH_3)_2]_2(CO)_4[P(OCH_3)_3]_2(CF_3COO)_2$ was obtained [3]. The protonation of $Mo(CO)_3(PEt_3)_3$ gives complex **1** (Scheme 1), formulated by analogy with the chemistry of $Mo(CO)_3L_3$ (where $L = P(CH_3)_3$ or $P(OCH_3)_3$) [8].

$$Mo(CO)_3(PEt_3)_3 + CF_3COOH \longrightarrow [HMo(CO)_3(PEt_3)_3]^+CF_3COO^-$$

$$\mathbf{1}$$

$$\Big\downarrow CF_3COOH$$

$$[Mo(CO)_2(PEt_3)_3(CF_3COO)]CF_3COO + CO + H_2$$

$$\mathbf{2}$$

$$-PEt_3 \Big\downarrow$$

$$Mo(CO)_2(PEt_3)_2(CF_3COO)_2$$

$$\mathbf{3}$$

Scheme 1.

TABLE I. Refined Coordinates (×10^4)

Atom	*x*	*y*	*z*
Mo	0	2921(1)	2500
P1	878(2)	1899(2)	2083(3)
P3	−954(2)	2078(3)	2849(3)
F1^a	−2183(10)	6100(15)	641(14)
F2^a	−1114(9)	6874(10)	1986(10)
F3^a	−1279(7)	6729(10)	549(8)
F4^a	−1794(9)	6423(14)	1517(14)
F5^a	−871(9)	7029(10)	1474(14)
F6^a	−1923(8)	6260(13)	98(10)
F7^a	2603(13)	5861(20)	4690(24)
F8^a	1604(10)	6452(12)	3221(13)
F9^a	1670(9)	6711(10)	4564(11)
F10^a	2256(6)	5971(9)	3796(8)
F11^a	1314(7)	6788(10)	3629(9)
F12^a	2342(8)	6193(12)	5154(10)
O1	−1180(6)	1137(11)	464(7)
O2	1226(5)	1139(9)	4518(6)
O3	−549(7)	4589(8)	2532(11)
O4	−947(7)	4289(9)	908(8)
O5	944(7)	4342(10)	2887(12)
O6	1520(8)	4261(13)	4729(9)
C1	−748(7)	1789(15)	1225(9)
C2	783(9)	1797(15)	3757(12)
C3	−958(9)	4932(12)	1535(13)
C4	−1383(7)	6157(12)	1164(9)
C5	1384(8)	4726(11)	3878(13)
C6	1809(7)	5920(11)	4106(10)
C7	959(11)	2755(14)	1106(12)
C8	60(15)	2909(19)	−73(16)
C9	2028(14)	1745(45)	3304(18)
C10	2644(14)	1975(43)	3019(21)
C11	649(11)	378(12)	1458(14)
C12	785(22)	−596(15)	2313(29)
C13	−681(12)	2550(23)	4198(16)
C14	−245(13)	3770(17)	4534(15)
C15	−2091(12)	2461(31)	1733(17)
C16	−2670(17)	1537(44)	1812(30)
C17	−951(13)	374(15)	2820(20)
C18	−1551(14)	−178(18)	3014(22)

^{a}Occupancy factor: 0.50.

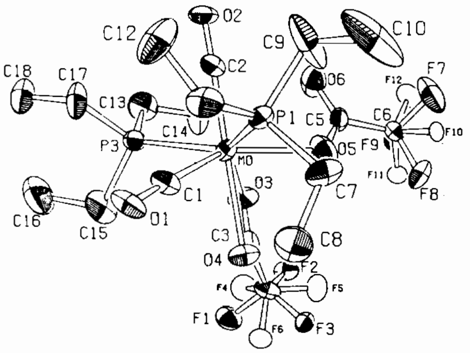

Fig. 1. View of the $Mo(CO)_2(PEt_3)_2(CF_3CO_2)_2$ molecule. Ellipsoids correspond to 50% probability. Hydrogens are omitted for simplicity. The open F atoms correspond to the alternate orientation of the disordered CF_3 groups.

Further attack by CF_3COOH, followed by the loss of a PEt_3 ligand, would then give **3**. Complex **3**, $Mo(CO)_2(PEt_3)_2(CF_3COO)_2$, is formulated on the basis of the following X-ray diffraction study.

The crystals of **3** contain monomeric molecules in which Mo is seven-coordinated (Fig. 1). The coordination sphere is best described in terms of a capped trigonal prism. The capping position is occupied by the O5 atom of a monodentate $CF_3CO_2^-$ ligand. The quadrangular plane contains one CO ligand (C2), one PEt_3 molecule (P1) and a $CF_3CO_2^-$ ion acting as a bidentate ligand via O3 and O4. The remaining edge contains one CO (C1) and one PEt_3 (P3) molecule.

The IR spectrum shows absorptions due to a monodentate CF_3CO_2 group at 1700 cm^{-1}, and to a bidentate CF_3CO_2 group at 1610 cm^{-1}. The ^1H NMR spectrum shows a pair of overlapping A_2B_3 multiplets in 2:3 ratio. Finally, the ^{31}P NMR spectrum consists of one resonance at δ 55.38 indicating one type of phosphine environment.

Interatomic distances and bond angles are listed in Table II. The most significant feature of this molecule is the presence of two differently coordinated CF_3CO_2 ligands. One of them is bonded in a unidentate manner: O5 is found at 2.215(15) Å from

TABLE II. Interatomic Distances and Bond Angles

Distances (Å)			
Mo–P1	2.478(4)	C17–C18	1.53(5)
Mo–P3	2.461(5)	O3–C3	1.24(2)
Mo–C1	1.958(13)	O4–C3	1.19(2)
Mo–C2	1.951(15)	C3–C4	1.49(2)
Mo–O3	2.139(12)	O5–C5	1.24(2)
Mo–O4	2.418(9)	O6–C5	1.25(2)
Mo–O5	2.215(15)	C5–C6	1.47(2)
C1–O1	1.149(16)	C4–F1	1.26(3)
C2–O2	1.156(17)	C4–F2	1.27(2)
P1–C7	1.84(2)	C4–F3	1.24(2)
P1–C9	1.84(3)	C4–F4	1.27(3)
P1–C11	1.82(1)	C4–F5	1.25(2)
P3–C13	1.83(2)	C4–F6	1.26(2)
P3–C15	1.83(3)	C6–F7	1.24(4)
P3–C17	1.85(2)	C6–F8	1.27(2)
C7–C8	1.55(3)	C6–F9	1.24(2)
C9–C10	1.57(5)	C6–F10	1.25(2)
C11–C12	1.56(4)	C6–F11	1.22(2)
C13–C14	1.49(3)	C6–F12	1.27(2)
C15–C16	1.60(6)		
Angles (deg)			
P1–Mo–P3	131.4(1)	Mo–P1–C11	123.8(6)
P1–Mo–C1	71.9(4)	C7–P1–C9	99.5(13)
P1–Mo–C2	73.0(5)	C7–P1–C11	99.3(9)
P1–Mo–O3	147.7(4)	C9–P1–C11	100.7(13)
P1–Mo–O4	104.8(3)	Mo–P3–C13	114.5(8)
P1–Mo–O5	75.9(4)	Mo–P3–C15	113.4(9)
P3–Mo–C1	76.4(5)	Mo–P3–C17	110.7(8)
P3–Mo–C2	79.0(5)	C13–P3–C15	106.7(12)
P3–Mo–O3	80.2(4)	C13–P3–C17	107.7(11)
P3–Mo–O4	102.7(3)	C15–P3–C17	103.1(12)
P3–Mo–O5	148.5(4)	P1–C7–C8	110.7(14)
C1–Mo–C2	102.2(7)	P1–C9–C10	114.4(25)
C1–Mo–O3	118.2(6)	P1–C11–C12	109.4(17)
C1–Mo–O4	77.0(5)	P3–C13–C14	109.2(17)
C1–Mo–O5	133.8(6)	P3–C15–C16	109.0(22)
C2–Mo–O3	128.1(6)	P3–C17–C18	111.9(18)
C2–Mo–O4	117.8(6)	Mo–C1–O1	178.4(13)
C2–Mo–O5	98.9(6)	Mo–C2–O2	177.0(15)
O3–Mo–O4	53.9(5)	Mo–O3–C3	100.4(11)
O3–Mo–O5	76.5(5)	Mo–O4–C3	87.9(10)
O4–Mo–O5	80.4(5)	Mo–O5–C5	112.9(12)
Mo–P1–C7	114.7(6)	O3–C3–O4	117.8(16)
Mo–P1–C9	115.1(12)	O3–C3–C4	120.7(15)
O4–C3–C4	121.3(15)	F4–C4–F6	105.2(15)
O5–C5–O6	129.9(17)	F5–C4–F6	104.4(15)
O5–C5–C6	116.4(15)	C5–C6–F7	114.4(18)
O6–C5–C6	113.6(15)	C5–C6–F8	113.8(14)
C3–C4–F1	112.9(15)	C5–C6–F9	116.2(14)
C3–C4–F2	113.2(14)	F7–C6–F8	100.9(19)
C3–C4–F3	113.9(13)	F7–C6–F9	106.1(18)
F1–C4–F2	103.2(15)	F8–C6–F9	103.8(15)
F1–C4–F3	107.6(15)	C5–C6–F10	113.4(13)
F2–C4–F3	105.2(15)	C5–C6–F11	114.3(13)
C3–C4–F4	114.5(14)	C5–C6–F12	113.9(13)
C3–C4–F5	113.6(14)	F10–C6–F11	104.5(13)
C3–C4–F6	112.8(14)	F10–C6–F12	103.1(13)
F4–C4–F5	105.4(15)	F11–C6–F12	106.6(14)

Mo, whereas the long Mo–O6 distance of 3.130(13) Å indicates that this second oxygen is free. Similar Mo–oxygen distances were observed for other $CF_3CO_2^-$ complexes [2, 9]. For the second carboxylate, the Mo–O3 distance (2.139(12) Å) is shorter than above, and O4 lying at 2.418(9) Å from Mo completes an unbalanced chelate ring. Besides these three oxygens, the coordination sphere includes two CO and two PEt_3 ligands. The Mo–C and Mo–P distances (mean values 1.955(14) and 2.470(5) Å, respectively) compare well with those found in similar complexes [2, 10–13].

In the two CF_3CO_2 ligands, the $-CF_3$ groups are found in two disordered orientations ~60° apart about the C–C bond (Fig. 1). However, the geometry in both orientations remains reasonable (Table II). In the unit cell, these disordered groups are in van der Waals contact with ethyl groups of nearby molecules (Fig. 2). Therefore, some of the ethyl carbon atoms are likely to respond to this disorder by occupying slightly different positions for the two $-CF_3$ orientations, and the rather large thermal motion for some C atoms indicates that this probably takes place. As a consequence, large standard deviations are found for the bond lengths and angles in the phosphine. The following mean values are found for the distances and angles: P–C, 1.83(2) Å; $C-CH_3$, 1.55(4) Å; $P-C-CH_3$, 110.8(19)°; C–P–C, 102.8(12)° and Mo–P–C, 115.4(8)°. Within the limits of accuracy of the present work, the geometry of the PEt_3 molecules compares well with the literature [11, 13]. The C–O distance (mean 1.153(17) Å) in the carbon monoxide ligands is also normal, and coordination takes place linearly as expected (mean Mo–C–O = 177.7(14)°). The large standard deviations do not permit to detect any significant difference between the various C–O distances in the CF_3CO_2 ligands.

Discussion

The problems raised by the choice of an ideal polyhedron to describe the structure of seven-coordinate molecules has been discussed previously [2, 14, 15]. Data are provided in Table III to help in making such a decision. They are based on a procedure involving optimization of both the relative orientation of the actual and the ideal polyhedra, and the adjustable angles (not fixed by symmetry) in the ideal polyhedron. The R' value (Table III) corresponds to the r.m.s. residual, after optimization, for the angles between the bond directions in the actual and the ideal polyhedra, respectively. The smallest R' value is found for the capped trigonal prism, which is therefore preferred to the capped octahedron and the pentagonal bipyramid.

Table III also includes similar data for the seven-coordinate $Mo(CO)_2(PMe_3)_3(CF_3CO_2)_2$ complex,

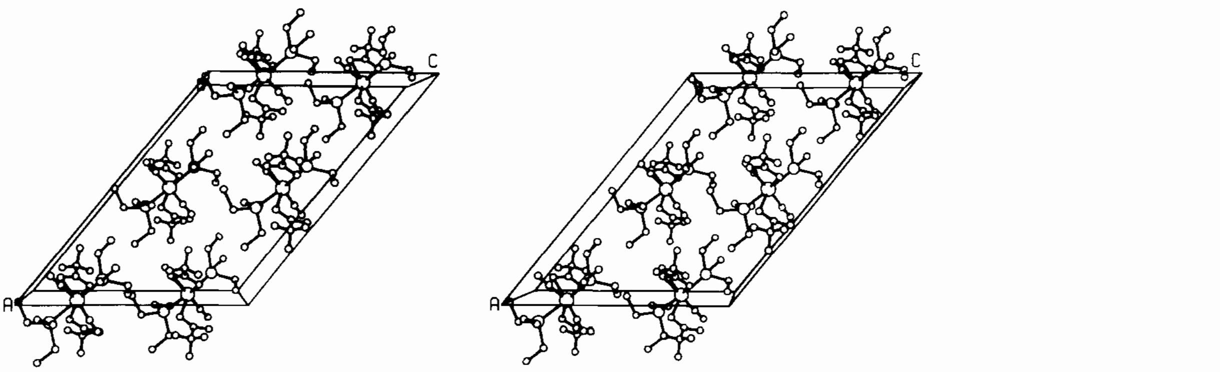

Fig. 2. Stereoview of the unit cell of $Mo(CO)_2(PEt_3)_2(CF_3CO_2)_2$. Atoms are represented as spheres of arbitrary sizes: large for Mo, medium for P, small for other non-hydrogen atoms. Only one orientation of the disordered $-CF_3$ groups is represented.

TABLE III. Fit of Ideal Polyhedra with Observed Geometry

	$[Mo(CO)_2)(PMe_3)_3(CF_3CO_2)_2]$ (Ref. 5)	$[Mo(CO)_2(PEt_3)_2(CF_3CO_2)_2]$ (This work)
Capped trigonal prism (C_{2v})		
R'^{a} (Å) (deg)	0.079, 4.5	0.153, 8.7
$\phi_B{}^c$	83.7	83.5
θ_B	52.1	63.0
ϕ_E	145.3	142.0
Capping site	O^b	O5
Quadrangular face	2 PMe_3, O^b, CO	C1, P1, O3, O4
Opposite edge	PMe_3, CO	P3, C2
Capped octahedron (C_{3v})		
R' (Å) (deg)	0.117, 6.7	0.203, 11.7
ϕ_B	72.7	78.5
ϕ_E	125.8	128.0
Capping site	CO	P3
Capped face	3 PMe_3	C1, C2, O3
Uncapped face	CO, 2 O^b	P1, O4, O5
Pentagonal bipyramid (C_{5v})		
R' (Å) (deg)	0.224, 12.9	0.270, 15.5
Axial	PMe_3, CO	P1, O5
Equatorial	2 PMe_3, 2 O^b, CO	P3, C1, C2, O3, O4

[a] R' (in degrees) is the mean value for the angles between corresponding bond directions in the actual and ideal polyhedra, respectively [$= 2 \sin^{-1} (R(\text{Å})/2)$]. [b] Oxygen from monodentate CF_3CO_2 ligand. [c] Angle symbols as in ref. 11.

which was also shown to possess a capped trigonal prismatic structure [2]. The latter complex contains three PMe_3 molecules and only unidentate CF_3CO_2 ligands. The diagrams of Fig. 3 show the ligand distribution on the three types of apices for both compounds. The capping position is occupied in both cases by the unidentate carboxylate oxygen O5, in agreement with the prediction that this site should be used by a good π-donor [15]. One CO and one PR_3 ligand are found on the opposite edge for the two molecules. As to the quadrangular face, three of the

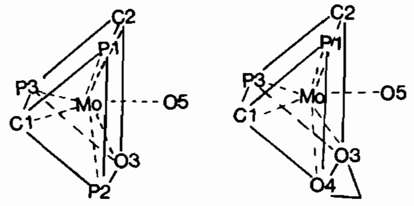

Fig. 3. Line drawing of the capped trigonal prism around Mo for $Mo(CO)_2(PMe_3)_3(CF_3CO_2)$ (left) and $Mo(CO)_2(PEt_3)_2(CF_3CO_2)_2$ (right).

four apices are similarly occupied, whereas one Mo–P bond in the $Mo(CO)_2(PMe_3)_3(CF_3CO_2)_2$ compound is replaced here by the weaker Mo–O4 bond with the chelating carboxylate. The ϕ_B and ϕ_E angles are also found to be quite similar for the two complexes (84° and 145° *vs.* 83° and 142°). The difference found for θ_B (52° *vs.* 63°) reflects the fact that the bite of the carboxylate ligand imposes a small angle of 53.9(5)° between two of the bonds in the quadrangular face. Therefore, the present $Mo(CO)_2(PEt_3)_2(CF_3CO_2)_2$ complex can be regarded as derived from a $Mo(CO)_2(PR_3)_3(CF_3CO_2)_2$ species, by dissociation of a phosphine in the quadrangular face and closure of the carboxylate ring to fill the vacant position in the coordination sphere. Because of the greater cone angle of PEt_3 compared with PMe_3 (132° *vs.* 118°) [16], the presence for PEt_3 could introduce greater steric hindrance, which could make phosphine dissociation and subsequent ring closure easier. However, the Mo–O4 bond is much longer than Mo–O3 and opening of the chelate ring leading to a six-coordinate intermediate should be facile.

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Supplementary Material

The supplementary material includes list of temperature factors, fixed hydrogen coordinates, atom-to-plane distances, and structure factor amplitudes. It is available upon request from author A.L.B.

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