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Synthesis and Properties of the Transition Metal Complexes of a Tritertiary Stibine, 1,1,1-Tris((diphenylstibino)methyl)ethane. Structure of *fac*-[Mo(CO)$_3${MeC(CH$_2$SbPh$_2$)$_3$}]

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The tritertiary stibine MeC(CH$_2$SbPh$_2$)$_3$ (tts) reacted with [M(CO)$_6$] (M = Cr, Mo, W) in ethanol in the presence of NaBH$_4$ to form *fac*-[M(CO)$_3$(tts)], which were characterized by analysis, IR spectroscopy, and ^{1}H and ^{13}C{^{1}H} NMR spectroscopy. The crystal structure of the Mo derivative has been determined by single-crystal X-ray diffraction and shown to be the *fac* isomer (Sb−Mo = 2.7263(5)−2.7462(5) Å). The complexes [Pd(tts)Cl$_2$] and [Pt(tts)Cl$_2$] in which the tts coordinates as a bidentate ligand, and *fac*-[Rh(tts)Cl$_3$] and [Cu(tts)(MeCN)]PF$_6$ in which it is tridentate, were also prepared and characterized spectroscopically. These complexes are the first examples reported containing a tritertiary stibine ligand.

Introduction

Many polydentate phosphine and arsine ligands have been prepared, and their coordination chemistry has been studied in considerable detail.[1−3] Several of the phosphine ligands are commercially available. In marked contrast the transition metal compounds of stibine ligands mostly contain monodentate SbR$_3$ ligands.[4] Bidentate ditertiary stibines have received limited study, and only two complexes have been structurally characterized: [Co{*o*-C$_6$H$_4$(SbMe$_2$)$_2$}$_2$Cl$_2$]$_2$[CoCl$_4$][5] and [Pd$_2$(Ph$_2$SbCH$_2$SbPh$_2$)$_2$Ph$_2$Cl$_2$].[6] Here we report the synthesis and characterization of the first complexes containing a tritertiary stibine ligand, 1,1,1-tris(diphenylstibinomethyl)ethane, MeC(CH$_2$SbPh$_2$)$_3$.

Results and Discussion

The ligand MeC(CH$_2$SbPh$_2$)$_3$ (tts) was first prepared by Ellermann and Veit,[7] who obtained it along with tetraphenyldistibane (Ph$_4$Sb$_2$) by reaction of NaSbPh$_2$ with MeC(CH$_2$Br)$_3$ in liquid ammonia and subsequently converted it into MeC(CH$_2$SbCl$_2$)$_3$ and the tristibane MeC(CH$_2$Sb)$_3$. No complexes of tts have been reported. We obtained tts as a white air-stable powder in poor yields (12−15%) by Ellermann's route, the poor yields in part resulting from the fractional crystallizations necessary to separate tts from Ph$_4$Sb$_2$. The ^{1}H and ^{13}C{^{1}H} NMR spectra (Experimental Section) confirm the structure of the ligand, and the FAB mass spectrum (3-NOBA matrix) exhibits a very weak parent ion multiplet (m/z = 896, C$_{41}$H$_{39}$^{121}Sb$_2$^{123}Sb$^+$) and stronger peaks due to cleavage of C$_6$H$_6$ (m/z = 818, C$_{35}$H$_{33}$^{121}Sb$_2$^{123}Sb$^+$) and SbPh$_2$ (m/z = 621, C$_{29}$H$_{29}$^{121}Sb$_2$$^+$) groups from the molecular ion.

***fac*-[M(CO)$_3$(tts)] (M= Cr, Mo, W).** The reaction of tts with Mo(CO)$_6$ in refluxing ethanol containing NaBH$_4$ resulted in high yields of white [Mo(CO)$_3$(tts)]. The pale yellow chromium and white tungsten analogues were obtained similarly, and under the conditions used even with different M(CO)$_6$:tts ratios, the tricarbonyl complexes were the only significant products, neither the IR nor ^{1}H NMR spectra showing evidence for the presence of other complexes. [Mo(CO)$_3$(tts)] was also produced although in an impure form, by reaction of [Mo(CO)$_3$(MeCN)$_3$] with the ligand. The reactions of MeC(CH$_2$PPh$_2$)$_3$ with M(CO)$_6$ in the presence of NaBH$_4$ were reported by Chatt *et al.*,[8] but interestingly in this case the tetracarbonyls *cis*-[M(CO)$_4${MeC(CH$_2$PPh$_2$)$_3$}] in which the triphosphine is bound as a bidentate were produced. *fac*-[M(CO)$_3${MeC(CH$_2$PPh$_2$)$_3$}][9,10] were obtained either by displacement of the arene from [M(π-arene)(CO)$_3$] or by direct thermal reaction with M(CO)$_6$, and complexes of the related arsine MeC(CH$_2$AsMe$_2$)$_3$ have been described.[11] The IR spectra of [M(CO)$_3$(tts)] (Table 1)[12,13] contain two carbonyl stretches, and the ^{1}H NMR spectra show single δ(CH$_2$) resonances consistent with a *fac* geometry; this was confirmed by the X-ray crystal structure determination of the molybdenum complex. The carbonyl stretching frequencies of the tts complexes may be compared with those of related complexes (Table 1). The ν(CO) frequencies are generally higher in the stibine complexes than those of the corresponding phosphine, consistent with the lower σ-donor power of the stibine. The ^{13}C{^{1}H} NMR spectra

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Table 1. IR Spectroscopic Data on $[M(CO)_3L_3]$ Complexes

complex	ν(CO)/cm$^{-1\ a}$	ν(CO)/cm^{-1}
$[Cr(CO)_3(tts)]$	1925, 1844	1922, 1838^b
$[Mo(CO)_3(tts)]$	1942, 1854	1934, 1850^b
$[W(CO)_3(tts)]$	1934, 1844	1928, 1838^b
$[Cr(CO)_3\{MeC(CH_2PPh_2)_3\}]^c$	1930, 1834	
$[Mo(CO)_3\{MeC(CH_2PPh_2)_3\}]^c$	1930, 1834	
$[W(CO)_3\{MeC(CH_2PPh_2)_3\}]^c$	1905, 1830	
$[Cr(CO)_3\{MeC(CH_2AsMe_2)_3\}]^d$		1923, 1825^e
$[Mo(CO)_3\{MeC(CH_2AsMe_2)_3\}]^d$		1935, 1835^e
$[W(CO)_3\{MeC(CH_2AsMe_2)_3\}]^d$		1930, 1835^e
$[Mo(CO)_3(SbEt_3)_3]^f$		1994, 1859^e
$[Mo(CO)_3(SbPh_3)_3]^g$		1972, 1875^e

[a] 1,2-Dichloroethane solution. [b] KBr disk. [c] Data from ref 9. [d] Data from ref 11. [e] Nujol mulls. [f] Data from ref 12. [g] Data from ref 13.

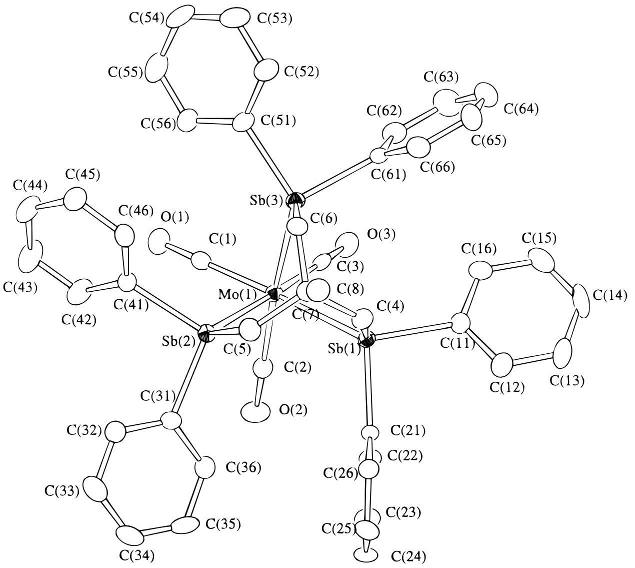

Figure 1. Molecular structure of $[Mo(CO)_3\{MeC(CH_2SbPh_2)_3\}]$ showing the atom-labeling scheme. H atoms have been omitted for clarity, and the thermal ellipsoids are drawn at the 50% probability level.

revealed single carbonyl resonances at δ 235.0 (Cr), 222.2 (Mo), and 212.3 (W), which show the expected shift[14,15] to low frequency as group 6 is descended.

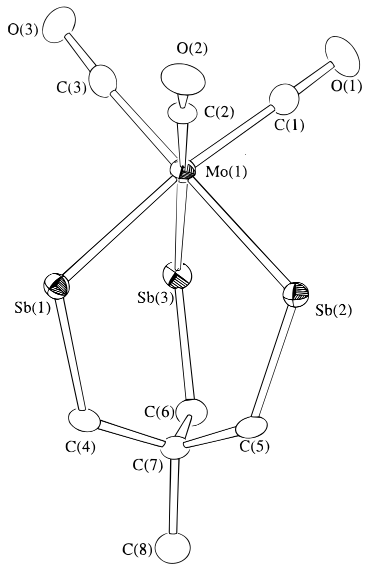

Figure 2. Molecular structure of $[Mo(CO)_3\{MeC(CH_2SbPh_2)_3\}]$ with phenyl C atoms and H atoms omitted. Thermal ellipsoids are drawn at the 50% level.

Table 2. Selected Bond Distances (Å) and Angles (deg)

Sb(1)−Mo(1)	2.7341(5)	Sb(3)−C(61)	2.148(5)
Sb(1)−C(4)	2.174(4)	Mo(1)−C(1)	1.979(5)
Sb(1)−C(11)	2.146(4)	Mo(1)−C(2)	1.965(5)
Sb(1)−C(21)	2.132(5)	Mo(1)−C(3)	1.971(5)
Sb(2)−Mo(1)	2.7263(5)	O(1)−C(1)	1.152(5)
Sb(2)−C(5)	2.163(5)	O(2)−C(2)	1.162(5)
Sb(2)−C(31)	2.137(5)	O(3)−C(3)	1.160(5)
Sb(2)−C(41)	2.145(5)	C(4)−C(7)	1.548(6)
Sb(3)−Mo(1)	2.7462(5)	C(5)−C(7)	1.547(6)
Sb(3)−C(6)	2.168(5)	C(6)−C(7)	1.546(6)
Sb(3)−C(51)	2.138(5)	C(7)−C(8)	1.543(6)
C−C(phenyl) 1.358(8)−1.399(7)			
Mo(1)−Sb(1)−C(4)	113.8(1)	Sb(2)−Mo(1)−Sb(3)	82.91(1)
Mo(1)−Sb(1)−C(11)	128.4(1)	Sb(2)−Mo(1)−C(1)	89.3(1)
Mo(1)−Sb(1)−C(21)	112.8(1)	Sb(2)−Mo(1)−C(2)	91.5(1)
C(4)−Sb(1)−C(11)	97.1(2)	Sb(2)−Mo(1)−C(3)	178.4(1)
C(4)−Sb(1)−C(21)	103.8(2)	Sb(3)−Mo(1)−C(1)	96.9(1)
C(11)−Sb(1)−C(21)	97.3(2)	Sb(3)−Mo(1)−C(2)	170.9(1)
Mo(1)−Sb(2)−C(5)	114.9(1)	Sb(3)−Mo(1)−C(3)	96.1(1)
Mo(1)−Sb(2)−C(31)	125.2(1)	C(1)−Mo(1)−C(2)	90.2(2)
Mo(1)−Sb(2)−C(41)	112.9(1)	C(1)−Mo(1)−C(3)	89.6(2)
C(5)−Sb(2)−C(31)	97.5(2)	C(2)−Mo(1)−C(3)	89.7(2)
C(5)−Sb(2)−C(41)	102.7(2)	Mo(1)−C(1)−O(1)	179.0(4)
C(31)−Sb(2)−C(41)	100.3(2)	Mo(1)−C(2)−O(2)	179.0(4)
Mo(1)−Sb(3)−C(6)	113.3(1)	Mo(1)−C(3)−O(3)	176.2(4)
Mo(1)−Sb(3)−C(51)	124.1(1)	Sb(1)−C(4)−C(7)	121.9(3)
Mo(1)−Sb(3)−C(61)	117.9(1)	Sb(2)−C(5)−C(7)	120.3(3)
C(6)−Sb(3)−C(51)	102.3(2)	Sb(3)−C(6)−C(7)	121.4(3)
C(6)−Sb(3)−C(61)	100.8(2)	C(4)−C(7)−C(5)	112.9(4)
C(51)−Sb(3)−C(61)	94.6(2)	C(4)−C(7)−C(6)	112.3(4)
Sb(1)−Mo(1)−Sb(2)	83.88(2)	C(4)−C(7)−C(8)	106.4(4)
Sb(1)−Mo(1)−Sb(3)	80.20(1)	C(5)−C(7)−C(6)	112.7(4)
Sb(1)−Mo(1)−C(1)	172.9(1)	C(5)−C(7)−C(8)	105.9(4)
Sb(1)−Mo(1)−C(2)	92.2(1)	C(6)−C(7)−C(8)	106.0(4)
Sb(1)−Mo(1)−C(3)	97.1(1)		

Structure of *fac*-$[Mo(CO)_3(tts)]$. The structure consists of discrete molecules with the expected *fac* stereochemistry which are shown in Figures 1 and 2. Complexes of the phosphine ligand corresponding to tts have been reported many times, and in particular *fac*-$[Mo(CO)_3\{MeC(CH_2PPh_2)_3\}]$ is known.[16] A number of closely related phosphine complexes with phenyl replaced by other aryl/alkyl groups have also been reported. The arsenic analogue of tts has been characterized by X-ray analysis in a binuclear cobalt complex[17] $[Co_2H_3\{MeC(CH_2AsPh_2)_3\}_2]^+$ and the corresponding phosphorus compound is also known. The geometry of the present compound is detailed in Table 2 and provides the first characterization of this ligand. The Sb−Mo distances (2.7263(5)−2.7462(5) Å) may be compared with $[Mo(CO)_3\{SbPh_2(SPh)\}_3]$ (2.74 Å (av)).[18] The remaining distances seem unexceptional, and Mo−C and C−O agree well with the phosphine analogue[16] (1.969(3) and 1.155(3) Å, respectively). Inspection of Figure 2 and Table 2 shows the approximate (noncrystallographic) 3-fold symmetry along Mo(1)···C(7). There is a small twist of Sb(n) ($n = 1-3$) relative to C($n + 3$) with a mean

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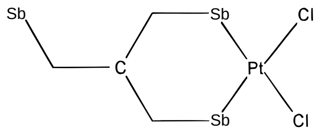
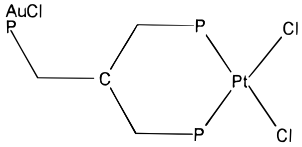
torsion angle Mo(1)−Sb(*n*)−C(*n* + 3)−C(7) of 15°. A similar twist (35.6°) was noted in the phosphine analogue.[16]

Metal Halide Complexes. No complexes were formed by reaction of anhydrous cobalt(II) bromide or nickel(II) iodide with tts in *n*-butanol−CH_2Cl_2. Nor did a mixture of $CoBr_2$, $NaBPh_4$, and tts in nitromethane with free access of dry air show any tendency to form a cobalt(III) complex, a route succesful in forming complexes of alkyl-substituted distibines.[5]

The reaction of $[M'(MeCN)_2Cl_2]$ (M′ = Pd, Pt) with tts in CH_2Cl_2 yielded orange $[Pd(tts)Cl_2]$ and pale yellow $[Pt(tts)Cl_2]$. The UV−visible spectra of these two complexes are typical of planar d^8 species, for example $[M'\{Ph_2Sb(CH_2)_3SbPh_2\}Cl_2]$,[19] indicating that the tts is bound as a bidentate ligand (I). This was confirmed by the ^1H NMR spectra which showed a sharp singlet overlaying a complex multiplet in the $\delta(CH_2)$ region assignable to the pendant arm and ring methylene protons, respectively. The ^1H NMR spectra did not change on cooling the samples to *ca* −50 °C, showing no evidence for exchange processes in this temperature range. For $[Pt(tts)Cl_2]$ the $\delta(^{195}Pt)$ resonance of −4617 may be compared with a value of −4556 in $[Pt\{Ph_2Sb(CH_2)_3SbPh_2\}Cl_2]$.[20] The coordination of tts to these two metals is analogous to the behavior of $MeC(CH_2PPh_2)_3$ and $MeC(CH_2AsPh_2)_3$ (L_3), which also function as bidentate chelates.[21] For the latter complexes, it proved possible to coordinate the "free" $-PPh_2$ or $-AsPh_2$ groups to a second metal. For example reaction with $[Au(SMe_2)Cl]$ produced bimetallic complexes $[M'(L_3)AuCl_3]$ (II); however, $[M'(tts)Cl_2]$ did not react with $[Au(SMe_2)Cl]$ in CH_2Cl_2 at ambient temperatures, presumably due to the weaker donor power of the $-SbPh_2$ group.



(I)



(II)

Rhodium trichloride and tts in ethanol readily generated the orange $[Rh(tts)Cl_3]$, assigned a *fac* geometry, since only a single $\delta(CH_2)$ resonance was present in the ^1H NMR spectrum, showing the three methylene groups are equivalent.

The reaction of $[Cu(MeCN)_4]PF_6$ with tts in MeCN solution formed white $[Cu(tts)(MeCN)]PF_6$, the MeCN being retained on recrystallization from cold CH_2Cl_2, indicating it is coordinated to the copper, producing tetrahedral (Sb_3N) coordination. The MeCN ligand was identified by the ν(CN) stretch at 2273 cm^{-1} in the IR spectrum, and by a singlet at $\delta = 1.8$ in the ^1H NMR spectrum, in addition to the usual ligand resonances. An attempt to prepare an Au(I) complex of tts by reaction of the ligand with [Au(tetrahydrothiophen)Cl] failed with production of a metallic mirror.

Conclusions

The first complexes of a tritertiary stibine ligand have been prepared and examples of tri- and bidentate coordination characterized.

Experimental Section

Physical Measurements. ^1H NMR spectra were recorded from $CDCl_3$ or CD_2Cl_2 solutions on a Bruker AC300 spectrometer. $^{13}C\{^1H\}$ NMR spectra were obtained from $CDCl_3$ solutions on Bruker AC300 and AM360 spectrometers, in the cases of the carbonyl complexes with 2 s pulse delays and in the presence of $Cr(acac)_3$. $^{195}Pt\{^1H\}$ NMR spectra were also obtained on a Bruker AM360 (at 77.7 MHz) and are referenced to $[PtCl_6]^{2-}$ in water. UV−visible spectra were obtained from $CHCl_3$ or CH_2Cl_2 solutions on a Perkin-Elmer Lambda 19, and IR spectra as KBr disks, Nujol mulls, or in 1,2-dichloroethane solutions on a Perkin Elmer 983G. C, H analyses were from the Microanalytical Laboratory of Imperial College, London.

1,1,1-Tris((diphenylstibino)methyl)ethane, $MeC(CH_2SbPh_2)_3$ (tts). Sodium metal (2.0 g, 0.08 mol) in liquid ammonia (250 cm^3) was treated with Ph_3Sb (14.0 g, 0.04 mol). The reaction was stirred for 3 h to produce a deep red solution. Dry NH_4Cl (2.0 g, 0.037 mol) was added in small portions to destroy NaPh. $MeC(CH_2Br)_3$[22] (4.0 g, 0.012 mol) in dry THF (150 cm^3) was added dropwise from a pressure-equalized dropping funnel. The ammonia was allowed to boil off, and the reaction was stirred overnight. Dichloromethane (150 cm^3) was added, and the solution turned dark green. Any solid material was removed by filtration under vacuum to leave a yellow solution. The volume of solvent was reduced to 50 cm^3 at reduced pressure, and ethanol (100 cm^3) was added. The solution was left to crystallize in the freezer overnight. The products Ph_4Sb_2 and $MeC(CH_2SbPh_2)_3$ were separated by fractional crystallization from ethanol−dichloromethane (1.4 g, 12% yield). Anal. Calcd for $C_{41}H_{39}Sb_3$: C, 54.9; H, 4.4. Found: C, 54.9; H, 4.4. ^1H NMR in $CDCl_3$: $\delta(CH_3)$ 1.2 (s) [3H], $\delta(CH_2)$ 2.3 (s) [6H], δ(Ph) 7.1−7.4 (m) [30H] (lit.[7] ^1H NMR: 1.2, 2.3, 7.2 ppm). $^{13}C\{^1H\}$ NMR in $CDCl_3$: $\delta(CH_3)$ 34.9, $\delta(CH_2)$ 43.5, δ(*C*Me) 39.6, δ(Ph) 128.9, 134.1, 136.2, 137.8 ppm.

$[Pd\{MeC(CH_2SbPh_2)_3\}Cl_2]$. $[PdCl_2(MeCN)_2]$ (0.026 g, 0.10 mmol) was added to a solution of $MeC(CH_2SbPh_2)_3$ (0.10 g, 0.11 mmol) in dichloromethane (20 cm^3). The solution was stirred for 1 h and then concentrated in volume at reduced pressure (2 cm^3). Diethyl ether (20 cm^3) was slowly added to afford an orange precipitate. The solid was filtered off and dried *in vacuo.* Three recrystallizations from CH_2Cl_2/Et_2O gave a deep yellow powder. Yield: 0.076 g (71%). Anal. Calcd for $C_{41}H_{39}Cl_2PdSb_3$: C, 45.8; H, 3.6. Found: C, 46.3; H, 3.7. ^1H NMR in $CDCl_3$: $\delta(CH_3)$ 0.95 (s), $\delta(CH_2)$ 2.0−2.5 (m), δ(Ph) 7.1−7.4 (m). UV-vis (CH_2Cl_2) [$E_{max}/10^3$ cm^{-1} (ϵ_{mol}/dm^3 cm^{-1} mol^{-1})] 33.7 (8000), 27.8 (5200). ν(Pd-Cl) (Nujol mull): 317, 295 cm^{-1}.

$[Pt\{MeC(CH_2SbPh_2)_3\}Cl_2]$ was prepared analogously using $[PtCl_2(MeCN)_2]$ in 68% yield. Anal. Calcd for $C_{41}H_{39}Cl_2PtSb_3$: C, 42.3; H, 3.4. Found: C, 42.3; H, 3.5. ^1H NMR in $CDCl_3$: $\delta(CH_3)$ 0.95, $\delta(CH_2)$ 2.0−2.5 (m), δ(Ph) 7.1−7.4 (m). ^{195}Pt NMR (CH_2Cl_2): δ −4617. UV−vis (CH_2Cl_2): 32.7 (6500), 25.5 (sh). ν(Pt−Cl) (Nujol mull): 310, 295 cm^{-1}.

$[Rh\{MeC(CH_2SbPh_2)_3\}Cl_3]$. $RhCl_3\cdot 3H_2O$ (0.029 g, 0.11 mmol) in ethanol (15 cm^3) was added dropwise to a solution of $MeC(CH_2SbPh_2)_3$ (0.105 g, 0.11 mmol) in CH_2Cl_2 (10 cm^3). The solution was stirred for 2 h, and then the volume of solvent

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was reduced to 2 cm^3 at reduced pressure. The orange solid that precipitated was filtered off, recrystallized twice from $CH_2Cl_2-Et_2O$, and dried *in vacuo*. Yield: 0.089 g (73%). Anal. Calcd for $C_{41}H_{39}Cl_3RhSb_3$: C, 44.5; H, 3.5. Found: C, 45.2; H, 4.1. ^{1}H NMR in $CDCl_3$: $\delta(CH_3)$ 1.1 (s), $\delta(CH_2)$ 2.20 (s), δ(Ph) 7.1−7.4 (m). UV−vis (CH_2Cl_2): 32.2 (3100), 26.7 (3400). ν(Rh−Cl) (Nujol mull): 334, 298 cm^{-1}.

[Cu(MeCN){MeC(CH_2SbPh_2)$_3$}]PF_6. [Cu(MeCN)$_4$]PF_6 (0.056 g, 0.15 mmol) dissolved in MeCN (15 cm^3) was treated with a solution of MeC(CH_2SbPh_2)$_3$ (0.134 g, 0.15 mmol) in MeCN (10 cm^3). The solution was stirred for 1 h and then concentrated (2 cm^3). Diethyl ether (20 cm^3) was added to precipitate a white solid. This was filtered off and recrystallized from CH_2Cl_2-Et_2O before drying *in vacuo*. Yield: 0.101 g (59%). Anal. Calcd for $C_{43}H_{42}CuF_6NPSb_3$: C, 45.0; H, 3.7. Found: C, 44.7; H, 3.4. IR spectrum (Nujol): 838, 557 cm^{-1} ([PF_6]$^-$); 2273 cm^{-1} (MeCN). ^{1}H NMR in $CDCl_3$: $\delta(CH_3)$ 1.1 (s), $\delta(CH_3CN)$ 1.8 (s), $\delta(CH_2)$ 2.20 (s), δ(Ph) 7.1−7.4 (m).

[Mo(CO)$_3${MeC(CH_2SbPh_2)$_3$}]. Mo(CO)$_6$ (0.03 g, 0.11 mmol) was added to a suspension of MeC(CH_2SbPh_2)$_3$ (0.190 g, 0.21 mmol) and $NaBH_4$ (0.009 g, 0.24 mmol) in ethanol (30 cm^3). The mixture was heated to reflux for 5 h and then allowed to cool with stirring overnight. The material that crystallized was filtered off, washed with H_2O (3 × 30 cm^3) and ethanol (30 cm^3), and air dried. The white solid was recrystallized from CH_2Cl_2−MeOH and dried *in vacuo*. Yield: 0.08 g (64%). Anal. Calcd for $C_{44}H_{39}MoO_3Sb_3$: C, 49.1; H, 3.6. Found: C, 48.8; H, 3.4. ^{1}H NMR in $CDCl_3$: $\delta(CH_3)$ 1.2 (s) [3H], $\delta(CH_2)$ 2.25 (s) [6H], δ(Ph) 7.0−7.5 (m) [30H]. $^{13}C\{^1H\}$ NMR in $CDCl_3$: $\delta(CH_3)$ 34.8, $\delta(CH_2)$ 43.5, δ(*C*Me) 39.6, δ(Ph) 128.9−137.8, δ(CO) 222.2.

[Cr(CO)$_3${MeC(CH_2SbPh_2)$_3$}] was prepared analogously using Cr(CO)$_6$ (50% yield). Anal. Calcd for $C_{44}H_{39}CrO_3Sb_3$: C, 51.1; H, 3.8. Found: C, 51.2; H, 3.8. ^{1}H NMR in $CDCl_3$: $\delta(CH_3)$ 1.2 (s) [3H], $\delta(CH_2)$ 2.25 (s) [6H], δ(Ph) 7.0−7.5 (m) [30H]. $^{13}C\{^1H\}$ NMR in $CDCl_3$: $\delta(CH_3)$ 34.8, $\delta(CH_2)$ 43.5, δ(*C*Me) 39.6, δ(Ph) 128.9−137.8, δ(CO) 235.0.

[W(CO)$_3${MeC(CH_2SbPh_2)$_3$}] was prepared analogously using W(CO)$_6$ (60% yield). Anal. Calcd for $C_{44}H_{39}O_3Sb_3W$: C, 45.4; H, 3.4. Found: C, 45.5; H, 3.4. ^{1}H NMR in $CDCl_3$: $\delta(CH_3)$ 1.2 (s) [3H], $\delta(CH_2)$ 2.25 (s) [6H], δ(Ph) 7.0−7.5 (m) [30H]. $^{13}C\{^1H\}$ NMR in $CDCl_3$: $\delta(CH_3)$ 34.8, $\delta(CH_2)$ 43.5, δ(*C*Me) 39.6, δ(Ph) 128.9−137.8, δ(CO) 212.3.

Crystal Structure of [Mo(CO)$_3${MeC(CH_2SbPh_2)$_3$}]. Air-stable colorless crystals were obtained from dichloromethane−pentane mixtures by liquid diffusion and mounted on a Rigaku AFC7S diffractometer fitted with Mo Kα radiation and a graphite monochromator. The crystals were examined at 150 K and mounted with the oil method on a glass fiber. Intensities were corrected for the Lorentz and polarization factors in the usual manner, a small amount (0.13%) of decay was allowed for, and an empirical ψ-scan absorption correction applied. The heavy atoms were located using SHELXS-86[23] and developed using structure factor and electron-density calculations. Hydrogen atoms appeared in later maps, and all were introduced in calculated positions (d(C−H) = 0.97 Å). Full-matrix least-squares refinement (on F) converged to R = 0.023.[24] Crystallographic details are given in Table 3.

Table 3. Crystallographic Data

mol formula	$C_{44}H_{39}MoO_3Sb_3$
mol wt	1076.98
cryst syst	orthorhombic
space group	*Pbca* (No. 61)
a, Å	20.358(2)
b, Å	20.488(2)
c, Å	19.164(1)
V, Å^3	7994(1)
T, K	150
d(calcd), g cm^{-3}	1.790
Z	8
F(000), e	4176
cryst size, mm	0.45 × 0.35 × 0.20
type of data collcn	$2\theta/\omega$
tot. no. of observns	7869
tot. no. of unique observns	7707
abs corr	ψ-scan (3 reflns)
transm factors	0.65 (min), 1.00 (max)
no. of data used in refinement	4955 ($I > 3\sigma(I)$)
no. of params	460
weighting scheme (w^{-1})	$\sigma^2(Fo)$
λ, Å (Mo Kα)	0.710 69
μ, cm^{-1}	23.5
max 2θ, deg	50.0
hkl limits	0−24, 0−24, 0−22
S	1.45
max shift/esd	0.07
max, min electron dens (e Å^{-3})	+0.39, −0.42
R[a]	0.023
R_w	0.023

[a] $R = \sum||F_o| - |F_c||/\sum|F_o|$; $R_w = [\sum w(F_o - F_c)^2/\sum wF_o^2]^{1/2}$.

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Supporting Information Available: Listings of complete atomic coordinates, anisotropic thermal parameters, and complete bond lengths and angles (7 pages). Ordering information is given on any current masthead page.

OM950823A

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(23) Sheldrick, G. M. SHELXS86, *Program for the Solution of Crystal Structures* University of Göttingen, FRG, 1986.

(24) teXsan: Crystal Structure Analysis Package, Molecular Structure Corp., The Woodlands, TX, 1985, 1992.