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Crystal and Molecular Structure of (Acetylene)bis(trimethylphosphine)(tetracarbonyl)dicobalt(0)

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Received December 27, 1977

The structure of (acetylene)bis(trimethylphosphine)(tetracarbonyl)dicobalt(0), $(C_2H_2)Co_2(P(CH_3)_3)_2(CO)_4$, has been determined crystallographically at −50 °C. The dinuclear complex possesses a C_{2v} perfect symmetry. Each cobalt atom is bonded to two carbon atoms of the carbonyl groups to two carbon atoms of the acetylene group and to a phosphorus atom of the phosphine ligand in a square-pyramidal type arrangement. Furthermore the two cobalt atoms are linked by a metal–metal bond of 2.464 (1) Å. The compound crystallizes in space group $C_{2v}{}^{19}$-$Fdd2$ of the orthorhombic system with 16 half-formulas in a cell of dimensions $a = 20.846$ (8) Å, $b = 26.656$ (8) Å, and $c = 6.668$ (1) Å. The 93 structural data were refined by full-matrix least-squares methods to a conventional R index of 0.024 based on those 1085 reflections having $F_o{}^2 > 3\sigma(F_o{}^2)$.

Introduction

It is well-known that the reaction of alkynes with dicobalt octacarbonyl gives $(RC{\equiv}CR)Co_2(CO)_6$ complexes at room temperature.[1] The molecular structures of such compounds are well established for $R = C_6H_5$[2] and $R = (CH_3)_3C$.[3] These structures are deduced from that of dicobalt octacarbonyl replacing the two bridging carbonyl groups by the alkyne. Although the action of group 5 ligands is quite well studied and is known to give rise to $(RC{\equiv}CR)Co_2(CO)_{6-n}L_n$ compounds,[1,4] with n ranging from 1 to 4, the only structural information for these compounds was deduced from IR and NMR data. We present here the synthesis and crystal structure of $HC{\equiv}CHCo_2(CO)_4(P(CH_3)_3)_2$ which we have undertaken in order to check structural hypothesis given elsewhere[1,4] and to assess the influence of the phosphine ligand on the molecular structure, in particular on the metal–metal bond.

Experimental Section

Infrared spectra were recorded in a solution of hexadecane on a Perkin-Elmer 225 spectrometer and proton NMR data were recorded on a Varian A60A.

All reactions were carried out under nitrogen atmosphere. Dicobalt octacarbonyl was purchased from Pressure Chemical Co.; $HC{\equiv}CHCo_2(CO)_6$ and $P(CH_3)_3$ were prepared following literature methods.[5,6]

Synthesis of $HC{\equiv}CHCo_2(CO)_4(P(CH_3)_3)_2$. A mixture of 1 g of $HC{\equiv}CHCo_2(CO)_6$ (3.2 mol) and 0.8 cm^3 (8.4 mol) of phosphine $P(CH_3)_3$ in 40 cm^3 of benzene was refluxed for 2 h at room temperature. The reaction solution was then cooled and filtered; the benzene was evaporated under vacuum. Recrystallization of the crude product from methanol gives 0.8 g (yield 60%) of long red needles of $HC{\equiv}CHCo_2(CO)_4(P(CH_3)_3)_2$.

Table I

Physical and Crystallographic Data

Formula: $(C_2H_2)Co_2(CO)_4$-$(P(CH_3)_3)_2$
Crystal system: orthorhombic
$a = 20.846$ (8) Å
$b = 26.656$ (8) Å
$c = 6.668$ (1) Å
$\rho_{\text{exptl}} = 1.42$ g cm^{-3}
$\rho_X = 1.463$ g cm^{-3}
Mol wt: 408.1
Space group: $Fdd2(C_{2v}{}^{19}$, No. 43)
$V = 3\,705$ Å^3
$Z = 16$ (half of the formula)
Abs factor: μ(Mo Kα) 20.38 cm^{-1}

Data Collection

Temperature: −50 °C
Radiation: molybdenum $\lambda(K\alpha_1)$ 0.709 26 Å
Monochromatization: graphite
Crystal-detector distance: 273 mm
Detector window: height = 4 mm, width = (3.2 + 0.75 tan θ) mm
Takeoff angle: 4°
Scan mode: $\omega/2\theta$
Maximum Bragg angle: 27°
Scan angle: $(0.7 + 0.35 \tan\theta)°$
Reflections for intensity controls: $\bar{4}80$, $\bar{8}20$, and $\bar{2}22$
Periodicity: every 2 h

Conditions for Refinement

Reflections for the refinement of the cell dimensions: 25
Recorded independent reflections: 2274 $(-h,+k,+l)$
Utilized reflections: 1085 with $F^2 > 3\sigma(F_o{}^2)$
Refined parameters: 93
Reliability factors: $R = \Sigma|k|F_o| - |F_c||/\Sigma k|F_o|$
$R_w = [\Sigma w(k|F_o| - |F_c|)^2/\Sigma wk^2F_o{}^2]^{1/2}$

Preliminary X-ray Study. Examination of crystals of the title compound by precession methods using Mo Kα radiation showed that the compound belongs to the orthorhombic system. Systematic absences hkl ($h + k$ odd), hkl ($k + l$ odd), $0kl$ ($k + l \neq 4n$), and $h0l$ ($h + l \neq 4n$) lead to the $Fdd2$ ($C_{2v}{}^{19}$, No. 43) space group. Cell constants at −50 °C and corresponding standard deviations, listed in Table I, were derived from a least-squares refinement of the settings

0020-1669/78/1317-1973$01.00/0

Table II. Atomic Parameters for $(HC{\equiv}CH)Co_2(CO)_4(P(CH_3)_3)_2$[a]

Atom	x	y	z	U_{11} or B, Å^2	U_{22}, Å^2	U_{33}, Å^2	U_{12}, Å^2	U_{13}, Å^2	U_{23}, Å^2
Co	0.05102 (2)	0.02332 (1)	0	0.0394 (2)	0.0347 (2)	0.0494 (2)	−0.0017 (2)	−0.0037 (2)	−0.0004 (2)
P	0.13421 (4)	0.04876 (3)	−0.1703 (2)	0.0391 (4)	0.0499 (5)	0.0657 (7)	−0.0083 (4)	−0.0024 (5)	−0.0003 (5)
C(1)	0.0320 (2)	0.0814 (1)	0.1179 (6)	0.0482 (19)	0.0531 (21)	0.0533 (26)	−0.0012 (16)	−0.0075 (19)	−0.0037 (20)
C(2)	0.0933 (2)	−0.0161 (1)	0.1709 (8)	0.0636 (24)	0.0479 (22)	0.0727 (29)	−0.0048 (18)	−0.0105 (24)	−0.0048 (22)
C(3)	0.0161 (1)	−0.0215 (1)	−0.2057 (6)	0.0393 (17)	0.0449 (18)	0.0473 (26)	−0.0069 (13)	0.0033 (16)	−0.0061 (18)
C(4)	0.1894 (2)	−0.0001 (1)	−0.2490 (9)	0.0502 (22)	0.0908 (27)	0.088 (3)	0.0112 (20)	0.0101 (29)	−0.005 (3)
C(5)	0.1162 (2)	0.0804 (2)	−0.4050 (8)	0.0693 (27)	0.089 (3)	0.089 (4)	−0.0169 (24)	0.0057 (27)	0.037 (3)
C(6)	0.1865 (2)	0.0924 (2)	−0.0414 (9)	0.0623 (25)	0.092 (3)	0.127 (5)	−0.0347 (23)	0.000 (3)	−0.031 (4)
O(1)	0.0193 (1)	0.1196 (1)	0.1872 (5)	0.0877 (20)	0.0545 (16)	0.0948 (26)	0.0142 (15)	−0.0061 (19)	−0.0248 (17)
O(2)	0.1205 (2)	−0.0418 (1)	0.2792 (6)	0.1237 (26)	0.0479 (18)	0.073 (3)	0.0096 (18)	−0.0520 (26)	0.0214 (21)
HC(3)	0.0357 (19)	−0.0398 (16)	−0.295 (9)	5					

[a] The form of the anisotropic thermal ellipsoid is $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$.

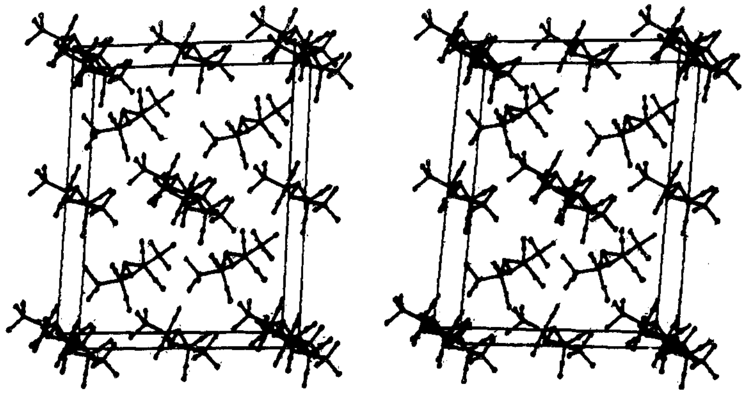

Figure 1. Stereoview of a unit cell of $(HC{\equiv}CH)Co_2(CO)_4(P(CH_3)_3)_2$. The vibration ellipsoids are drawn at an arbitrary level.

of 25 reflections automatically centered on a CAD 4 Enraf-Nonius computer-controlled diffractometer.

Based on a calculated volume of 3705 Å^3 and 16 half-formula units in the cell, the calculated density of 1.463 g cm^{-3} is in satisfactory agreement with the density of 1.42 g cm^{-3} measured by flotation in a zinc chloride aqueous solution.

X-ray Data Collection. The crystal selected for data collection was a parallelepiped elongated along the c axis having boundary faces {100}, {010}, and {001}, the dimensions 0.37 × 0.34 × 0.048 mm; it was mounted with [001] approximately along the spindle axis. Data collection was carried out at −50 °C following the scheme which appears in Table I and fully described elsewhere,[7] using a CAD 4 Enraf-Nonius four-circle diffractometer equipped with a scintillation counter and pulse height analyser tuned to accept 90% of the Mo Kα peak.

A rapid prescan allows the elimination of hkl reflections for which the intensities registered were less than ten counts above background. The final scan speed for all other reflections was calculated such as either the $\sigma(I)/I$ is equal to 0.4 or the maximal time scan is 90 s.

Structure Solution and Refinement. The structure was solved by standard Patterson and Fourier methods and refined by full-matrix least-squares techniques.[8] The quantity minimized is $\sum w(|F_o| - |F_c|)^2$ where $|F_o|$ and $|F_c|$ are the observed and calculated structure amplitudes and where the weights w are taken as $4F_o^2/\sigma^2(F_o^2)$. The agreement indices R and R_w are defined in Table I. The values of the atomic scattering factors and the anomalous terms used for the cobalt and phosphorus atoms were taken from normal sources.[9]

The positions of the Co and P atoms were determined from the Patterson function and all the atoms but the hydrogens were located in a subsequent Fourier map. R and R_w were 0.12 and 0.15, respectively, after two cycles of least-squares refinements of the atomic parameters for all atoms located involving isotropic thermal parameters. The anisotropic thermal parameters were then used for all atoms in a subsequent least-squares refinement; R and R_w were then 0.043 and 0.052, respectively. At this stage, a difference Fourier map reveals clearly all hydrogen atoms; their contribution with isotropic thermal parameters on a next least-squares refinement drops R and R_w to 0.024 and 0.023.

In Table II, we present the atomic parameters together with their standard deviations as derived from the inverse matrix. The root-mean-square amplitudes of vibration for those atoms refined anisotropically appear in Table III.

Table III. Hydrogen Atom Positions as Observed on a Difference Fourier Map

Atom	x	y	z
HAC(1)	0.2267	0.0131	−0.3269
HBC(1)	0.1668	−0.0263	−0.3372
HCC(1)	0.2071	−0.0195	−0.1289
HAC(2)	0.1572	0.0920	−0.4777
HBC(2)	0.0897	0.1123	−0.3838
HCC(2)	0.0919	0.0588	−0.5022
HAC(3)	0.2224	0.1029	−0.1309
HBC(3)	0.2029	0.0778	0.0839
HCC(3)	0.1606	0.1241	−0.0065

Discussion of the Structure in Solution from IR and NMR Data

The compound $HC{\equiv}CHCo_2(CO)_4(P(CH_3)_3)_2$ exhibits three infrared-active bands in the ν(CO) stretching region from which it can be concluded that the complex has a C_{2v} symmetry as has been shown to be the case for other $RC{\equiv}CRCo_2(CO)_4(PA_3)_2$ complexes.[4] The proton NMR spectra shows one triplet for the HC≡CH region ($\delta = -4.83$ ppm; $J_{P-H} = 3.5$ Hz) due to the coupling with the two phosphorus atoms and one triplet ($\delta = -1.28$ ppm; $J_{P-H} = 8.4$ Hz) for $P(CH_3)_3$ as the X part of $X_9AA'X'_9$ spin system.

From these observations we conclude that the two phosphine ligands are trans with respect to the metal–metal bond.

Description and Discussion of the Structure in the Solid State

The crystal structure consists of the packing of eight discrete dinuclear units per unit cell (Figure 1). A perspective view of the molecule with the labeling scheme used elsewhere is shown in Figure 2.

The molecular geometry of $Co_2(C_2H_2)(P(CH_3)_3)_2(CO)_4$ is in perfect agreement with the results deduced from IR and NMR data as just discussed. The molecule has perfect C_{2v} symmetry; indeed the dinuclear unit is built from a mononuclear unit by the binary crystallographic symmetry.

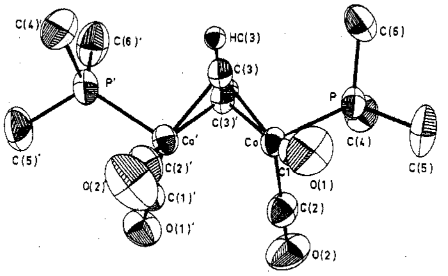

Figure 2. Perspective view showing the labeling scheme. Hydrogen atoms have been omitted. The vibration ellipsoids are drawn at the 50% probability level.

Table IV. Bond Lengths (Å) for $(HC{\equiv}CH)Co_2(CO)_4(P(CH_3)_3)_2$ and $(t\text{-}BuC{\equiv}C\text{-}t\text{-}Bu)Co_2CO_6$ Complexes

	$(HC{\equiv}CH)Co_2$-$(CO)_4(P(CH_3)_3)_2$	$(t\text{-}BuC{\equiv}C\text{-}t\text{-}Bu)Co_2$-$(CO)_6$[a]
Co-Co	2.464 (1)	2.463 (1)
Co-C(1)	1.780 (3)	1.816 (4)
Co-C(2)	1.784 (4)	1.804 (9)
Co-C(3)	1.958 (3)	2.003 (4)
Co-C'(3)	1.959 (3)	1.994 (4)
C(3)-C'(3)	1.327 (6)	1.335 (6)
C(1)-O(1)	1.150 (3)	Av 1.150
C(2)-O(2)	1.144 (4)	
Co-P	2.180 (1)	
C(3)-H	0.87 (2)	
P-C(4)	1.814 (4)	
P-C(5)	1.816 (4)	
P-C(6)	1.811 (4)	

[a] Taken from ref 3.

Each cobalt atom is bonded to carbon atoms C(1) and C(2) of the carbonyl groups, carbon atoms C(3) and C(3′) of the acetylene group, and to a phosphorus atom of the phosphine. The metal is displaced from the mean basal plane (equation: $-0.7679x - 0.3279y + 0.55032z + 0.7603 = 0$) toward the P atom by 0.26 Å; this displacement compares well with that (0.27 and 0.33 Å) in $(\mu\text{-}(SCH_3)Fe(CO)_2P(CH_3)_3)_2$.[10] Furthermore, the two cobalt atoms are linked by a metal–metal bond equal to 2.464 (1) Å.

It is of interest to compare the molecular structure of the present compound with that observed for $Co_2(CO)_6\text{-}t\text{-}BuC{\equiv}C\text{-}t\text{-}Bu$ [3] (Tables IV and V). Indeed, it appears that the substitution of two CO groups for two phosphine ligands does not affect the metal–metal bond. A significant shortening (10σ) of the cobalt–carbonyl distances is observed in the present complex: 1.782 Å instead of 1.810 Å. Such a shortening is well-known for mononuclear complexes and has been extensively discussed;[11] it occurs when a carbonyl group is replaced by a poorer π-acceptor ligand such as a phosphine. Is such a phenomenon quite general for dinuclear compounds too? Not enough data in the literature seem yet available to answer this question but the shortening observed in the present complex is comparable with that found in an analogous dinuclear compound of cobalt(0) with a diphosphine ligand $(tolan)(dpm)Co_2(CO)_4$,[12] as the mean Co–C distance for the pseudoequatorial CO groups is equal to 1.77 Å.

There is no report of analogous dinuclear complexes of cobalt(0) with phosphine ligands which would allow us to compare our Co–P bond distance of 2.180 (1) Å; however, the present value, although significantly shorter (10σ) is similar to the average cobalt–phosphorus distance of 2.213 Å in the $(tolan)(dpm)Co_2(CO)_4$ complex involving a diphosphine ligand.

Another difference between the present compound and the $Co_2(CO)_6t\text{-}BuC{\equiv}C\text{-}t\text{-}Bu$ complex may be pointed out. It occurs for the interatomic angles around the cobalt atom: the carbonyl carbon–cobalt–carbonyl carbon angle is much larger in the phosphine complex than the corresponding basal one in the carbonyl compound, 109.9 (2)° instead of 103.6 (2)°, while the phosphorus–cobalt–acetylene carbon is lower than the corresponding carbonyl carbon–cobalt–*t*-Bu acetylene carbon, 102.2 (1)° instead of 103.5 (2)° (Table V). These variations would be an effect of steric hindrance due to the *tert*-butyl groups in the case of the carbonyl complex.

Table V. Bond Angles (deg) for $(HC{\equiv}CH)Co_2(CO)_4(P(CH_3)_3)_2$ and $(t\text{-}BuC{\equiv}C\text{-}t\text{-}Bu)Co_2(CO)_6$

	$(HC{\equiv}CH)Co_2$-$(CO)_4$-$(P(CH_3)_3)_2$	$(t\text{-}BuC{\equiv}C\text{-}t\text{-}Bu)Co_2$-$(CO)_6$[a]
Co-Co-P	145.5 (3)	152.4 (2) (Co-Co-C)
Co-Co-C(1)	104.2 (1)	98.2 (2)
Co-Co-C(2)	97.4 (1)	99.6 (2)
C(3)-Co-P	96.9 (1)	103.5 (2) (C(3)-Co-C)
C'(3)-Co-P	102.2 (1)	101.9 (2) (C'(3)-Co-C)
C(1)-Co-C(3)	99.9 (1)	103.4 (2)
C(1)-Co-C'(3)	139.1 (1)	140.6 (2)
C(3)-Co-C(2)	141.7 (2)	142.5 (2)
C'(3)-Co-C(2)	105.8 (1)	106.6 (2)
C(1)-Co-P	97.9 (1)	98.4 (2)
C(2)-Co-P	97.0 (1)	97.7 (2)
C(1)-Co-C(2)	109.9 (2)	103.6 (2)
Co-C(3)-Co	77.9 (1)	76.1 (1)
C(3)-Co-C'(3)	39.6 (2)	39.0 (2)
C(3)-C'(3)-Co	70.2 (2)	70.8 (3)
Co-C-O (av)	178.4	178.6
C(3)-C'(3)-H	138	144.5 (4) (C-C-C) 144.8 (4)
C(4)-P-C(6)	102.5 (2)	
C(5)-P-C(6)	103.6 (2)	
C(4)-P-C(5)	102.3 (3)	

[a] Taken from ref 3.

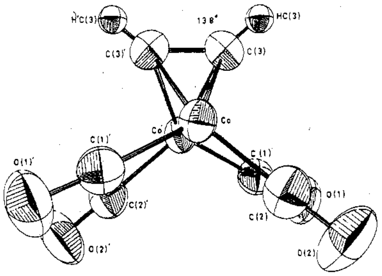

Figure 3. Perspective view of the dinuclear unit showing the bending of the H–C–C group. The phosphine ligands have been omitted for clarity.

Figure 3 shows the bending of the acetylene group around the carbon–carbon bond. In metal complexes involving the RC≡CR group, the bending around the carbon–carbon bond is generally related to the extent of both acetylene to metal σ-orbital donation and back-donation from metal π orbitals to the acetylene π^* orbital.[13] The bending increases with the excess of π back-donation from metal to acetylene. In the present complex the C–C–H angle of 138.0° is smaller than in $Co_2(CO)_6\text{-}t\text{-}BuC{\equiv}C\text{-}t\text{-}Bu$ (144.5 and 144.8°) and for other complexes compiled in ref 12. The strengthening of the RC≡CR bending in the present complex might be due to an electronic effect because of a poorer π-acceptor ligand in the coordination sphere of the metal or due to the fact that the R group is an H atom instead of bulky groups such as *tert*-butyl or phenyl groups in all other complexes.

Registry No. $(C_2H_2)Co_2(P(CH_3)_3)_2(CO)_4$, 66324-34-3; $HC{\equiv}CHCo_2(CO)_6$, 12264-05-0.

Supplementary Material Available: A listing of structure factor amplitudes (8 pages). Ordering information is given on any current masthead page.

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1,3-Butadiene Coordination to Polynuclear Metal Clusters. Crystal and Molecular Structures of $Os_3(CO)_{10}(s\text{-}cis\text{-}C_4H_6)$ and $Os_3(CO)_{10}(s\text{-}trans\text{-}C_4H_6)$

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Received January 19, 1978

The complexes $Os_3(CO)_{10}$(*s-cis*-C_4H_6) and $Os_3(CO)_{10}$(*s-trans*-C_4H_6) have been examined crystallographically. Crystals of $Os_3(CO)_{10}$(*s-cis*-C_4H_6) belong to the centrosymmetric monoclinic space group $P2_1/n$ with $a = 8.051$ (2) Å, $b = 14.778$ (3) Å, $c = 15.356$ (2) Å, $\beta = 94.60$ (1)°, $V = 1821.1$ Å^3, and d(calcd) = 3.30 g cm^{-3} for $Z = 4$. The complex $Os_3(CO)_{10}$(*s-trans*-C_4H_6) crystallizes in the centrosymmetric monoclinic space group $C2/c$ with $a = 30.638$ (6) Å, $b = 9.770$ (2) Å, $c = 13.285$ (3) Å, $\beta = 112.82$ (2)°, $V = 3659.7$ Å^3, and d(calcd) = 3.30 g cm^{-3} for $Z = 8$. The structures were determined and refined with 2418 independent reflections for $Os_3(CO)_{10}$(*s-cis*-C_4H_6) and 2473 for $Os_3(CO)_{10}$(*s-trans*-C_4H_6). Refinement of the $Os_3(CO)_{10}$(*s-cis*-C_4H_6) structure converged with $R_F = 0.036$ and $R_{wF} = 0.041$, while refinement of $Os_3(CO)_{10}$(*s-trans*-C_4H_6) converged wth $R_F = 0.036$ and $R_{wF} = 0.044$. The diene ligand of $Os_3(CO)_{10}$(*s-cis*-C_4H_6) is bonded to a single metal of the cluster at axial and equatorial positions. The $Os_3(CO)_{10}$(*s-trans*-C_4H_6) molecule has approximate C_2 symmetry with the diene ligand bridging one Os–Os bond of the cluster and bonding at equatorial sites of adjacent metal centers.

Introduction

Many of the models for adsorption of small substrate molecules on metallic surfaces parallel bonding features found first in molecular complexes. Polymetallic cluster complexes serve as useful models for metal surface adsorption where the concerted action of more than one metal atom center may be responsible for substrate activation.[1] Though the bonding and fluxional properties of polymetallic complexes containing unsaturated, cyclic hydrocarbons have preoccupied organometallic chemists for some time,[2] investigations on the coordination and dynamic behavior of clusters containing simple conjugated dienes have been few.[3] 1,3-Butadiene has been reported to bond to metal surfaces in both s-cis and s-trans conformational forms.[4] Coordination of the s-cis conformer in molecular complexes is well established since only a single metal center is required. *s-trans*-Butadiene coordination is far more unusual since the planarity of the ligand requires two metal centers. The recent use of $H_2Os_3(CO)_{10}$ as a precursor to $Os_3(CO)_{10}$(diene) complexes under mild conditions by the groups of Shapley[5] and Lewis[6] has opened to investigation this interesting series of conjugated diene adducts of the triangular $Os_3(CO)_{10}$ unit. Accounts of the dynamic properties of these complexes have appeared.[5,6] In this report we present the results of structural investigations on $Os_3(CO)_{10}$(*s-cis*-C_4H_6) and $Os_3(CO)_{10}$(*s-trans*-C_4H_6).

Experimental Section

Crystalline samples of $Os_3(CO)_{10}$(*s-cis*-C_4H_6) and $Os_3(CO)_{10}$(*s-trans*-C_4H_6) were generously provided by Professor John Shapley of the University of Illinois. A preliminary account of this work appeared previously.[5]

Data Collection and Reduction for $Os_3(CO)_{10}$(*s-cis*-C_4H_6). Preliminary photographs taken on crystals of the complex indicated monoclinic symmetry and an extinction pattern consistent with space group $P2_1/n$. A crystal of dimensions 0.316 × 0.213 × 0.180 mm was mounted and centered on a Syntex $P\bar{1}$ automated diffractometer equipped with a graphite-crystal monochromator. The settings of 15 reflections with 2θ values greater than 20° (Mo Kα radiation) were used to calculate the cell constants given in Table I. A complete set of intensity data were collected within the angular range $2° \leq 2\theta \leq 50°$. Symmetrical θ–2θ scans were used within the angular range ±0.7° on either side of the Mo Kα_1–Mo Kα_2 doublet at a scan rate of 4°/min. Four standard reflections measured after every 96 reflections showed normal variations in intensity during data collection. A total of 3380 reflections were measured. Data were corrected for Lorentz, polarization, and absorption effects.

Table I

Crystal Data for $Os_3(CO)_{10}$(*s-cis*-C_4H_6)	
$a = 8.051$ (2) Å	Mol wt 904.80
$b = 14.778$ (3) Å	d(calcd) = 3.30 g cm^{-3}
$c = 15.356$ (2) Å	$\mu = 222.1$ cm^{-1}
$\beta = 94.60$ (1)°	Transmission coeff:
$V = 1821.1$ Å^3	max, 0.190; min, 0.094
Space group $P2_1/n$	Facial planes:
$Z = 4$	$\{011\}, \{01\bar{1}\}, \{100\}$
Crystal Data for $Os_3(CO)_{10}$(*s-trans*-C_4H_6)	
$a = 30.638$ (6) Å	Mol wt 904.80
$b = 9.770$ (2) Å	d(calcd) = 3.30 g cm^{-3}
$c = 13.285$ (3) Å	$\mu = 221.1$ cm^{-1}
$\beta = 112.82$ (2)°	Transmission coeff:
$V = 3659.7$ Å^3	max, 0.241; min, 0.083
Space group $C2/c$	Facial planes:
$Z = 8$	$\{100\}, \{\bar{3}11\}, \{31\bar{1}\}$

Solution and Refinement of $Os_3(CO)_{10}$(*s-cis*-C_4H_6). The positions of the three Os atoms were determined from a three-dimensional Patterson map. A difference Fourier map calculated with phases obtained by refinement of the Os atoms revealed the positions of all other nonhydrogen atoms of the molecule. Three cycles of isotropic

0020-1669/78/1317-1976$01.00/0