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Single- and Mixed-Metal Complexes with *cyclo*-Triphosphorus and 1,1,1-Tris((diphenylphosphino)methyl)ethane, triphos. Synthesis and Structural Characterization of [(triphos)Co(η^3-P_3)] and [(triphos)Co(μ-(η^3-P_3)){$Cr_2(CO)_{10}$}]

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Cobalt(II) hexahydrate tetrafluoroborate in the presence of the tripod ligand 1,1,1-tris((diphenylphosphino)methyl)ethane, "triphos", reacts with white phosphorus to give the sandwich complex [(triphos)Co(η^3-P_3)], where the novel *c*-P_3 unit acts as an η^3 ligand. A complete X-ray structure determination, carried out from counter diffraction data, has shown that the cobalt atom is coordinated by the three phosphorus atoms of the triphos ligand and by the *cyclo*-triphosphorus group. The [(triphos)Co(η^3-P_3)] complex crystallizes in the space group *R*3 with the following cell dimensions: a = 10.574 (5) Å and γ = 109.5 (1)°. Full-matrix least-squares refinement led to a conventional *R* index of 0.029 for the 1365 observations. The [(triphos)Co(η^3-P_3)] complex forms with $Cr(CO)_6$ bi- and trinuclear metal complexes of formulas [(triphos)Co(μ-(η^3-P_3))$Cr(CO)_5$] and [(triphos)Co(μ-(η^3-P_3)){$Cr_2(CO)_{10}$}], respectively. The X-ray structure of the latter compound has shown that two phosphorus atoms of the *cyclo*-triphosphorus group are linked to the two chromium atoms of two $Cr(CO)_5$ fragments. The crystals of the [(triphos)Co(μ-(η^3-P_3)){$Cr_2(CO)_{10}$}] complex are monoclinic, *C*2/*c* space group, with the following cell dimensions: a = 36.48 (2) Å, b = 13.45 (1) Å, c = 26.59 (1) Å, and β = 105.0 (1)°. The structure was refined by full-matrix least-squares methods to a final *R* index of 0.074 for the 3691 observations. All the compounds have been characterized by the usual chemical and physical techniques.

Introduction

Few reactions of white phosphorus with transition-metal compounds have been sofar reported.[1–3] With the use of such reactions, some phosphorus containing compounds are obtained, whose stereochemistry has been generally assigned on the basis of only indirect information.

In the reaction of white phosphorus with cobalt(II) tetrafluoroborate hexahydrate and the tripodlike poly(tertiary phosphines), as 1,1,1-tris((diphenylphosphino)methyl)ethane ($CH_3C(CH_2PPh_2)_3$), triphos, and tris(2-(diphenylphosphino)ethyl)amine ($N(CH_2CH_2PPh_2)_3$, np$_3$), metal complexes of formula [LCo(η^3-P_3)] (L = np$_3$, triphos) have been obtained, containing one *cyclo*-triphosphorus unit linked to the metal as a η^3-P_3 ligand.[4,5]

The [(triphos)Co(η^3-P_3)] complex has been found capable of reacting with electron-acceptor groups. Indeed, by the reaction of this complex with triphos and cobalt(II) or nickel(II) tetrafluoroborate, triple-decker dinuclear metal complexes of formula $[(\text{triphos})M(\mu\text{-}(\eta^3\text{-}P_3))M(\text{triphos})]^{2+}$ have been isolated. Triple-decker sandwich complexes of this kind can be obtained also by direct reaction of the metal salts and the ligand triphos with white phosphorus.[6] In these complexes the *cyclo*-triphosphorus unit is linked to the two metal atoms acting as a 3π-donor ligand.

For the obtainment of further information about donor properties of [(triphos)Co(η^3-P_3)], this complex was reacted with hexacarbonyl chromium; as a result bi- and trinuclear carbonyl derivatives of formula [(triphos)Co(μ(η^3-P_3)){$Cr(CO)_5$}$_n$] (n = 1, 2) have been isolated.

The complete X-ray structural determinations for the compounds [(triphos)Co(η^3-P_3)] and [(triphos)Co(μ-(η^3-P_3)){$Cr_2(CO)_{10}$}] are reported here. Preliminary reports have been previously published.[4,7]

Experimental Section

Reagents. All solvents were reagent grade quality and were used without further purification. The ligand triphos was prepared by the method previously described.[8]

Preparation of the complexes. All the reactions were carried out under dry nitrogen.

[(triphos)Co(η^3-P_3)]. A solution of P_4 (1.2 mmol) in 20 mL of THF was added, with stirring, at room temperature, to the solution obtained by mixing triphos in THF (1 mmol in 20 mL) and $Co(H_2O)_6(BF_4)_2$ in ethanol (1 mmol in 15 mL). The resultant solution was heated (at 50 °C), and after ca. 15 min, yellow crystals were precipitated. They were filtered, washed with absolute ethanol and petroleum ether, and finally dried in vacuo. Anal. Calcd for $C_{41}H_{39}CoP_6$: C, 63.41; H, 5.06; Co, 7.58; P, 23.93. Found: C, 63.56; H, 5.28; Co, 7.30; P, 24.92.

[(triphos)Co(μ-(η^3-P_3))$Cr(CO)_5$]·THF. A solution of [(triphos)Co(η^3-P_3)] (0.5 mmol) in THF (50 mL) was added under nitrogen to 0.50 mmol of solid $Cr(CO)_6$ in a quartz vessel. The mixture was refluxed under UV light for 4 h. Butyl ether (20 mL) was added to the filtrate, and after concentration at 50 °C, crystals were precipitated. They were filtered and washed with THF/butyl ether and petroleum ether. Anal. Calcd for $C_{50}H_{47}CoCrO_6P_6$: C, 57.70; H, 4.55; Co, 5.66; Cr, 5.00. Found: C, 57.98; H, 5.11; Co, 5.62; Cr, 4.50.

[(triphos)Co(μ-(η^3-P_3)){$Cr_2(CO)_{10}$}]. The compound was prepared by the method used to prepare the above derivative by using a large excess of $Cr(CO)_6$. At the end of the reaction, the unreacted $Cr(CO)_6$ was removed by sublimation. Anal. Calcd for $C_{51}H_{39}CoCr_2O_{10}P_6$: C, 52.78; H, 3.39; Co, 5.08; Cr, 8.96. Found: C, 51.70; H, 4.01; Co, 4.75; Cr, 8.75.

Physical Measurements. Infrared spectra and magnetic susceptibilities were recorded by previously described methods.[9]

Collection and Reduction of X-ray Intensity Data. Unit cell parameters for each complex were determined from a least-squares refinement of the setting angles of 24 and 20 reflections carefully centered on a Philips PW 1100 diffractometer for [(triphos)Co(η^3-P_3)] (I) and [(triphos)Co(μ-(η^3-P_3)){$Cr_2(CO)_{10}$}] (II), respectively. Details of the crystal data and data collection for the two compounds are given in Table I. After correction for background, the standard deviation $\sigma(I)$ of the intensity was calculated as described elsewhere,[10] by using values of the instability factor *K* of 0.05 and 0.04 for complexes I and II, respectively.

The data were corrected for Lorentz–polarization effects, but no absorption correction was applied, the linear absorption coefficient

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Table I. Crystal Data Collection Details

	$[(\text{triphos})\text{Co}(\eta^3\text{-P}_3)]$	$[(\text{triphos})\text{Co}(\mu\text{-}(\eta^3\text{-P}_3))\{\text{Cr}_2(\text{CO})_{10}\}]$
formula	$C_{41}H_{39}CoP_6$	$C_{51}H_{39}CoCr_2O_{10}P_6$
mol wt	776.5	1160.6
a, Å	10.574 (5)	36.48 (2)
b, Å		13.45 (1)
c, Å		26.59 (1)
α, deg	109.5 (1)	90
β, deg		105.0 (1)
γ, deg		90
V, Å^3	910.2	12599.6
d_{calcd}, g cm^{-3}	1.416	1.223
d_{obsd}(by flot), g cm^{-3}	1.42	1.21
Z	1	8
space group	$R3$	$C2/c$
abs coeff (Mo Kα), cm^{-1}	7.83	7.85
color	yellow	orange-brown
habit	rhombododecahedron	irregular prism
dimens, mm	0.12 × 0.12 × 0.10	0.27 × 0.55 × 0.15
diffractometer	Philips PW 1100	Philips PW 1100
λ(Mo Kα), Å	0.7107	0.7107
monochromator	flat graphite crystal	flat graphite crystal
takeoff angle, deg	3.5	3.5
method	ω–2θ scan	ω–2θ scan
scan speed, deg/s	0.06	0.08
scan range	$(A + B \tan\theta)^\circ$ with $A = 0.7$ and $B = 0.69$	0.8°
bkgd time	half scan time	half scan time
stds	3 every 2 h	3 every 2 h
2θ limits, deg	5–55	5–40
final no. of variables	144	254
unique data set, $I \geq 3\sigma(I)$	1365	3691
error in observn of unit wt, e	1.95	2.53
R	0.029	0.074
R_w	0.040	0.098

Table II. Atomic Parameters (×10^4) for the Structure of $[(\text{triphos})\text{Co}(\eta^3\text{-P}_3)]$ [a]

atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Co	0	0	0	217 (2)	217	217	95 (2)	95	95
P(1)	2 (1)	1225 (1)	2124 (1)	254 (4)	270 (4)	218 (4)	127 (3)	109 (3)	113 (3)
P(2)	−1060 (1)	−1978 (1)	−2470 (1)	351 (5)	328 (5)	267 (4)	135 (4)	142 (4)	73 (4)
C(1)	4676 (6)	4676	4676	321 (17)	321	321	−4 (21)	−4	−4
C(2)	3199 (5)	3199	3199	251 (15)	251	251	80 (18)	80	80
C(3)	2033 (4)	2536 (4)	3722 (4)	294 (17)	359 (18)	242 (15)	126 (14)	75 (13)	92 (14)
C(4)	−849 (4)	2505 (4)	2372 (4)	350 (17)	291 (16)	349 (17)	176 (14)	182 (15)	144 (14)
C(5)	−1866 (4)	2454 (4)	1104 (4)	328 (18)	368 (19)	408 (21)	154 (16)	173 (17)	212 (17)
C(6)	−2591 (5)	3341 (5)	1295 (6)	399 (21)	460 (23)	630 (28)	236 (19)	224 (20)	322 (22)
C(7)	−2273 (6)	4278 (6)	2753 (7)	577 (27)	483 (24)	879 (37)	367 (22)	487 (28)	364 (25)
C(8)	−1273 (7)	4333 (6)	4013 (7)	766 (33)	546 (27)	664 (31)	426 (27)	472 (28)	249 (25)
C(9)	−569 (6)	3439 (6)	3829 (5)	678 (30)	550 (26)	463 (24)	407 (25)	350 (23)	243 (21)
C(10)	−986 (4)	52 (4)	2797 (4)	382 (18)	314 (17)	307 (16)	188 (15)	217 (15)	163 (14)
C(11)	−2586 (5)	−764 (5)	1934 (5)	353 (19)	437 (21)	449 (21)	161 (16)	184 (17)	272 (18)
C(12)	−3479 (6)	−1747 (6)	2277 (6)	465 (24)	476 (25)	642 (29)	144 (20)	334 (23)	272 (22)
C(13)	−2750 (7)	−1926 (6)	3503 (7)	801 (35)	463 (25)	681 (32)	283 (23)	546 (30)	362 (24)
C(14)	−1155 (7)	−1137 (6)	4369 (6)	812 (35)	564 (27)	457 (24)	368 (26)	374 (25)	362 (22)
C(15)	−273 (5)	−122 (5)	4041 (5)	475 (22)	459 (22)	323 (18)	229 (18)	233 (17)	221 (17)

[a] The form of the thermal ellipsoid is $\exp(-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + \ldots))$. F (relative scale factor) = 4028.

μ being 7.83 and 7.85 cm^{-1} for complexes I and II, respectively. An empirical estimation of the effects of absorption was made by considering a dozen reflections whose intensities were measured as the crystals were rotated about their azimuthal angle ϕ; the largest changes in intensities are ±6% and ±8% for I and II, respectively. Atomic scattering factors for Co, Cr, P, O, and C were those calculated by Cromer and Waber[11] while those for hydrogen were those calculated by Stewart.[12]

The effects of anomalous dispersion of the cobalt, chromium, and phosphorus atoms were included in the calculation of F_c, the values of $\Delta f'$ and $\Delta f''$ being those calculated by Cromer.[13]

Solution and Refinement of the Structures. $[(\text{triphos})\text{Co}(\eta^3\text{-P}_3)]$. The structure was solved by heavy-atom techniques. All nonhydrogen atoms were located on subsequent Fourier maps. Least-squares refinements were carried out,[14] the function minimized being $\sum w[(F_o) - (F_c)]^2$. The weights were taken as $1/\sigma^2(F_o)$. A few cycles of full-matrix least-squares refinements using isotropic thermal parameters led to the R value of 0.064. Anisotropic temperature factors were then introduced, and R was reduced to 0.040. Hydrogen atoms were introduced in calculated positions (C–H = 0.95 Å) but not refined. Because of the polarity of the space group, two possible enantiomeric structures must be considered: the model initially assumed yielded R and R_w of 0.029 and 0.040 and the enantiomorphic form had an R factor of 0.030 and 0.041, indicating that the original choice was correct. The Hamilton test[15] together with an analysis

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(14) Programs: ORTEP for molecular drawings; MULTAN for direct methods. The main routines of the X-RAY-72 system for data reduction, Fourier synthesis, and least-squares refinement were obtained from different sources and locally implemented on a C.I.I. 10070 computer.

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Table III. Atomic Parameters ($\times 10^4$) for the Structure of $[(triphos)Co(\mu\text{-}(\eta^3\text{-}P_3))\{Cr_2(CO)_{10}\}]$[a]

atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Co	1461 (1)	4510 (1)	1144 (1)	523 (12)	617 (13)	410 (11)	−15 (10)	133 (9)	78 (10)
Cr(1)	1221 (1)	5638 (2)	−584 (1)	717 (17)	755 (17)	432 (13)	−13 (14)	178 (12)	41 (13)
Cr(2)	2429 (1)	6666 (2)	1103 (1)	686 (16)	607 (16)	499 (14)	−121 (13)	179 (12)	15 (12)
P(1)	1752 (1)	3095 (3)	1107 (1)	622 (27)	590 (27)	502 (22)	−6 (22)	121 (20)	84 (20)
P(2)	1604 (1)	4594 (3)	1999 (1)	587 (26)	758 (29)	439 (22)	−13 (22)	163 (19)	99 (21)
P(3)	923 (1)	3730 (3)	1136 (1)	590 (26)	766 (30)	505 (23)	−75 (23)	138 (20)	78 (21)
P(4)	1284 (1)	6161 (3)	929 (1)	728 (28)	656 (28)	525 (24)	89 (23)	192 (21)	85 (21)
P(5)	1403 (1)	5221 (3)	339 (1)	649 (26)	648 (28)	397 (21)	−41 (22)	125 (19)	63 (20)
P(6)	1856 (1)	5691 (3)	970 (1)	608 (26)	581 (26)	452 (22)	−35 (21)	143 (19)	37 (20)

[a] The form of the thermal ellipsoid is $\exp(-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + \ldots))$. F (relative scale factor) = 68 923.

Table IV. Atomic Parameters ($\times 10^4$) for the Structure of $[(triphos)Co(\mu\text{-}(\eta^3\text{-}P_3))\{Cr_2(CO)_{10}\}]$

atom	x	y	z	U, Å^2
O(6)	814 (3)	3736 (9)	−999 (4)	986 (34)
O(7)	494 (4)	6504 (9)	−438 (5)	1221 (42)
O(8)	1596 (3)	7625 (9)	−309 (4)	990 (34)
O(9)	1938 (3)	4751 (8)	−750 (4)	1079 (37)
O(10)	988 (3)	6402 (8)	−1668 (4)	927 (33)
O(11)	2474 (3)	7049 (7)	2235 (4)	813 (30)
O(12)	3029 (3)	5076 (9)	1427 (4)	1052 (36)
O(13)	2456 (3)	6266 (8)	−4 (5)	1049 (36)
O(14)	1908 (3)	8413 (9)	812 (4)	1050 (36)
O(15)	3091 (3)	8054 (8)	1282 (4)	978 (34)
C(1)	1389 (4)	1621 (12)	2268 (6)	918 (49)
C(2)	1412 (4)	2558 (11)	1922 (5)	712 (44)
C(3)	1765 (4)	2398 (10)	1710 (5)	662 (39)
C(4)	1436 (4)	3475 (10)	2266 (5)	670 (41)
C(5)	1043 (4)	2533 (10)	1477 (5)	693 (41)
C(6)	967 (4)	4463 (12)	−816 (6)	770 (45)
C(7)	770 (5)	6163 (12)	−484 (6)	887 (50)
C(8)	1463 (4)	6851 (12)	−393 (5)	755 (45)
C(9)	1667 (5)	5107 (12)	−683 (6)	853 (49)
C(10)	1091 (4)	6087 (11)	−1241 (6)	792 (45)
C(11)	2443 (4)	6871 (10)	1801 (5)	652 (40)
C(12)	2781 (4)	5646 (12)	1301 (6)	786 (47)
C(13)	2429 (4)	6432 (12)	413 (6)	831 (49)
C(14)	2111 (4)	7745 (12)	908 (6)	837 (48)
C(15)	2828 (5)	7513 (12)	1193 (6)	827 (48)

of the standard deviations of the refined parameters supported this hypothesis. A final difference Fourier map did not show particular features. Final positional and thermal parameters are listed in Table II.

$[(triphos)Co(\mu\text{-}(\eta^3\text{-}P_3))\{Cr_2(CO)_{10}\}]$. The structure was solved by direct methods using MULTAN program.[14] The initial E map yielded the positions of Co and Cr atoms. Successive three-dimensional Fourier synthesis showed the positions of all nonhydrogen atoms. Full-matrix least-squares refinement was then undertaken. The phenyl ring carbon atoms were refined as rigid groups of D_{6h} symmetry with d(C–C) = 1.390 Å. Two cycles of isotropic refinement were followed by two cycles using anisotropic thermal parameters for Co, Cr, and P atoms. The hydrogen atoms were introduced in their geometrical calculated positions but not refined. Convergence was reached, the final R and R_w being respectively 0.074 and 0.098. A three-dimensional electron density difference synthesis showed no particular features. Final positional and thermal parameters are listed in Tables III–VI.

Results and Discussion

The complex $[(triphos)Co(\eta^3\text{-}P_3)]$ was obtained from the reaction of $Co(H_2O)_6(BF_4)_2$ and triphos with white phosphorus in THF at ca. 50 °C:

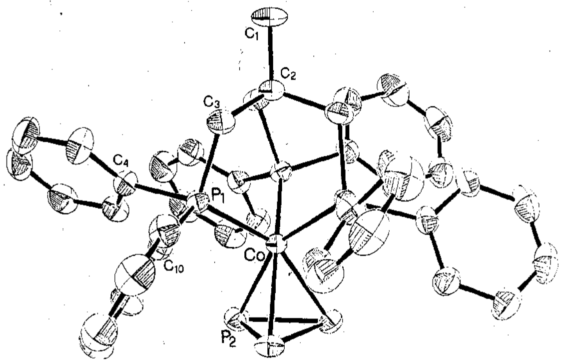

Figure 1. Perspective view of the complex molecule $[(triphos)Co(\eta^3\text{-}P_3)]$ (ORTEP drawing with 50% probability ellipsoids).

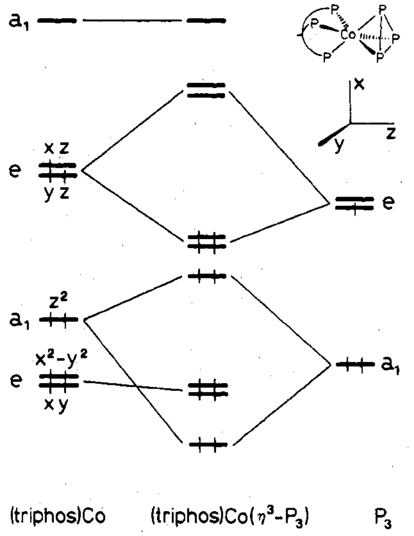

Figure 2. Energy level diagram for the complex $[(triphos)Co(\eta^3\text{-}P_3)]$.

$$[Co(H_2O)_6]^{2+} + \text{triphos} + P_4 \xrightarrow[50\ {}^\circ C]{THF} [(triphos)Co(\eta^3\text{-}P_3)]$$

The diamagnetic complex is air stable in the solid state and soluble in tetrahydrofuran, methylene chloride, and benzene.

Table V. Final Least-Squares Parameters for $[(triphos)Co(\mu\text{-}(\eta^3\text{-}P_3))\{Cr_2(CO)_{10}\}]$ Groups[a]

group	x'	y'	z'	θ	ϕ	ψ
1	0.1462 (2)	0.1639 (5)	0.0229 (3)	1.235 (10)	−2.172 (7)	−1.928 (8)
2	0.2542 (2)	0.2982 (5)	0.0955 (3)	1.008 (6)	2.900 (7)	0.217 (8)
3	0.1310 (2)	0.6369 (6)	0.2478 (2)	0.584 (7)	−0.788 (8)	1.762 (8)
4	0.2418 (2)	0.4638 (4)	0.2663 (2)	1.459 (6)	−0.623 (5)	−3.032 (8)
5	0.0364 (2)	0.3244 (6)	0.0115 (3)	1.926 (6)	−1.327 (7)	1.720 (5)
6	0.0334 (2)	0.4661 (6)	0.1608 (2)	2.761 (6)	−1.793 (8)	−1.353 (6)

[a] x', y', z' (fractional coordinates) define the origin of the working system of the group. The Goldstein definition is used for Euler angles θ, ϕ, and ψ (rad): Goldstein, H. "Classical Mechanics"; Addison-Wesley: Reading, Mass., 1959.

Table VI. Thermal and Derived Positional Parameters ($\times 10^4$) of Group Atoms

atom	ring	x	y	z	U, Å^2
C(1)	1	1549 (3)	2193 (7)	591 (4)	664 (40)
C(2)	1	1699 (3)	1235 (7)	630 (4)	988 (53)
C(3)	1	1597 (3)	589 (7)	208 (4)	1208 (62)
C(4)	1	1346 (3)	900 (7)	−253 (4)	1105 (59)
C(5)	1	1196 (3)	1858 (7)	−293 (4)	924 (51)
C(6)	1	1298 (3)	2504 (7)	129 (4)	680 (41)
C(1)	2	2240 (2)	3057 (7)	1049 (4)	629 (40)
C(2)	2	2303 (2)	3513 (7)	609 (4)	783 (47)
C(3)	2	2654 (2)	3425 (7)	499 (4)	950 (54)
C(4)	2	2943 (2)	2882 (7)	829 (4)	1006 (57)
C(5)	2	2881 (2)	2426 (7)	1270 (4)	978 (56)
C(6)	2	2530 (2)	2513 (7)	1380 (4)	720 (43)
C(1)	3	1421 (3)	5655 (7)	2296 (3)	652 (43)
C(2)	3	1540 (3)	6600 (7)	2194 (3)	817 (50)
C(3)	3	1410 (3)	7433 (7)	2406 (3)	1019 (56)
C(4)	3	1161 (3)	7320 (7)	2720 (3)	1115 (60)
C(5)	3	1042 (3)	6375 (7)	2822 (3)	1019 (59)
C(6)	3	1173 (3)	5542 (7)	2610 (3)	855 (49)
C(1)	4	2101 (2)	4660 (7)	2393 (3)	587 (39)
C(2)	4	2403 (2)	4509 (7)	2172 (3)	596 (39)
C(3)	4	2773 (2)	4484 (7)	2486 (3)	712 (43)
C(4)	4	2842 (2)	4609 (7)	3022 (3)	705 (44)
C(5)	4	2540 (2)	4760 (7)	3244 (3)	773 (46)
C(6)	4	2170 (2)	4785 (7)	2929 (3)	642 (40)
C(1)	5	565 (3)	3411 (9)	537 (3)	688 (42)
C(2)	5	448 (3)	2442 (9)	397 (3)	960 (53)
C(3)	5	213 (3)	2247 (9)	−96 (3)	1175 (67)
C(4)	5	96 (3)	3021 (9)	−449 (3)	1017 (53)
C(5)	5	214 (3)	3990 (9)	−309 (3)	847 (47)
C(6)	5	448 (3)	4185 (9)	184 (3)	735 (43)
C(1)	6	576 (2)	4295 (8)	1440 (3)	688 (43)
C(2)	6	564 (2)	5322 (8)	1500 (3)	811 (48)
C(3)	6	282 (2)	5748 (8)	1696 (3)	1019 (54)
C(4)	6	12 (2)	5148 (8)	1832 (3)	1018 (53)
C(5)	6	24 (2)	4122 (8)	1772 (3)	1241 (66)
C(6)	6	306 (2)	3695 (8)	1576 (3)	1038 (56)

Table VII. [(triphos)Co(η^3-P_3)] Bond Lengths (Å) and Angles (Deg) with Estimated Standard Deviations

Co–P(1)	2.186 (1)	C(6)–C(7)	1.373 (9)
Co–P(2)	2.301 (1)	C(7)–C(8)	1.366 (10)
P(2)–P(2′)	2.141 (2)	C(8)–C(9)	1.389 (11)
P(1)–C(3)	1.850 (3)	C(9)–C(4)	1.387 (7)
P(1)–C(4)	1.856 (5)	C(10)–C(11)	1.383 (5)
P(1)–C(10)	1.833 (5)	C(11)–C(12)	1.389 (9)
C(1)–C(2)	1.561 (3)	C(12)–C(13)	1.380 (10)
C(2)–C(3)	1.566 (7)	C(13)–C(14)	1.379 (8)
C(4)–C(5)	1.381 (6)	C(14)–C(15)	1.392 (9)
C(5)–C(6)	1.402 (8)	C(15)–C(10)	1.387 (7)

P(1)–Co–P(1′)	93.55 (4)	P(1)–C(4)–C(5)	120.8 (3)
P(1)–Co–P(2)	154.97 (5)	P(1)–C(4)–C(9)	120.1 (4)
P(1)–Co–P(2′)	105.31 (5)	C(5)–C(4)–C(9)	118.9 (5)
P(1)–Co–P(2″)	101.40 (4)	C(4)–C(5)–C(6)	120.3 (4)
P(2)–Co–P(2′)	55.43 (5)	C(5)–C(6)–C(7)	119.7 (5)
Co–P(1)–C(3)	108.6 (2)	C(6)–C(7)–C(8)	120.4 (7)
Co–P(1)–C(4)	124.1 (2)	C(7)–C(8)–C(9)	120.2 (6)
Co–P(1)–C(10)	117.4 (1)	C(8)–C(9)–C(4)	120.5 (5)
C(3)–P(1)–C(4)	102.3 (2)	P(1)–C(10)–C(11)	115.7 (4)
C(3)–P(1)–C(10)	106.1 (2)	P(1)–C(10)–C(15)	125.9 (3)
C(4)–P(1)–C(10)	96.1 (2)	C(11)–C(10)–C(15)	118.3 (5)
Co–P(2)–P(2′)	62.3 (4)	C(10)–C(11)–C(12)	121.8 (5)
P(2′)–P(2)–P(2″)	60.0	C(11)–C(12)–C(13)	119.1 (5)
C(1)–C(2)–C(3)	107.1 (2)	C(12)–C(13)–C(14)	120.1 (6)
C(3)–C(2)–C(3′)	111.7 (3)	C(13)–C(14)–C(15)	120.3 (6)
C(2)–C(3)–P(1)	114.0 (3)	C(14)–C(15)–C(10)	120.4 (4)

The solutions slowly decompose in air.

The crystal structure consists of individual molecules of [(triphos)Co(η^3-P_3)]. The coordination geometry is shown in Figure 1. Selected interatomic distances and angles are reported in Table VII. The cobalt atom is bonded to the three phosphorus atoms of a triangular P_3 unit and to the three phosphorus atoms of the triphos ligand, the two ligands being in a staggered position with a dihedral angle of 55°. From a valence-bond electron bookkeeping viewpoint the structure of the [(triphos)Co(η^3-P_3)] complex can be simply described as resulting from the replacement of a phosphorus atom in the P_4 molecule with an electronically equivalent Co(triphos) moiety. The P–P bond length within the η^3-P_3 system of 2.141 (2) Å, matching that reported for the [(np$_3$)Co(η^3-P_3)] complex,[5] is significantly shorter than the P–P lengths in P_4 vapor (2.21 Å).[16] Such decrease in bond length can be explained on the basis of electron-pair repulsion arguments in analogous way as for the (CO)$_3$Co(η^3-As$_3$) compound:[17] the more electronegative Co(triphos) group is considered to act as an "electron sink" which removes electron density from the P_3 group, thus decreasing the electron-pair repulsions between the three phosphorus atoms.

The Co–P(η^3-P_3) distance (2.301 (1) Å) is significantly longer than the Co–P(triphos) (2.186 (1) Å) distance.

The electronic structure of [(triphos)Co(η^3-P_3)] may be approached from a fragment analysis[6,18] by partitioning the molecule into the (triphos)Co and the P_3 fragments. Figure 2 shows the qualitative energy level diagram for the complex (C_{3v} symmetry). The MO orbitals of the (triphos)Co fragment (left side) can be considered to arise mainly from the contributions of the metal d orbitals and from the phosphorus (triphos) lone pairs. The latter 3 MO's which are the lowest in energy have been omitted in the figure. The metal orbitals have nonzero overlap with the orbitals of the P_3 group normal to the plane of the ring (right side). If the latter are considered to provide 3 electrons, 18 electrons can now be accommodated in the nine lowest MO's. This approach is also in agreement with the capability of the *cyclo*-triphosphorus group to link two metal atoms in double sandwich complexes acting as a 3π system. In this framework the [(triphos)Co(η^3-P_3)] complex can be considered as a sandwich complex electronically analogous to the cyclopentadienyl(triphenylcyclopropenyl)-nickel complex.[19]

On the other hand three lone pairs of electrons are available on the three phosphorus atoms of the P_3 group, so that such phosphorus atoms can act as bases. With this aim the [(triphos)Co(η^3-P_3)] complex was allowed to react with hexacarbonylchromium complexes, where a CO group could be replaced by another monodentate ligand. Indeed, the substitution products of chromium hexacarbonyl with [(triphos)Co(η^3-P_3)] complex were easily prepared according to

$$[(\mathrm{triphos})\mathrm{Co}(\eta^3\text{-}\mathrm{P}_3)] + n\mathrm{Cr(CO)}_6 \xrightarrow{h\nu} [(\mathrm{triphos})\mathrm{Co}(\mu\text{-}(\eta^3\text{-}\mathrm{P}_3))\{\mathrm{Cr(CO)}_5\}_n] \quad n = 1, 2$$

Even when a large excess of Cr(CO)$_6$ was used, only the trinuclear [(triphos)Co(μ-(η^3-P_3)){Cr$_2$(CO)$_{10}$}] was isolated.

The two chromium carbonyl derivatives are air stable and very soluble in benzene, THF, and methylene chloride. The IR spectra of the two complexes are similar in CHCl$_3$ solution with bands in the CO region at 2058 (w), 1945 (vw), 1945 (s), and 1917 (m) cm^{-1}. The first, third, and fourth bands are assigned to the three infrared active CO stretching fundamental of a DM(CO)$_5$ derivative: A_1^1, E, and A_1^2, respectively.[20] The splitting of the band at 1945 cm^{-1} observed in

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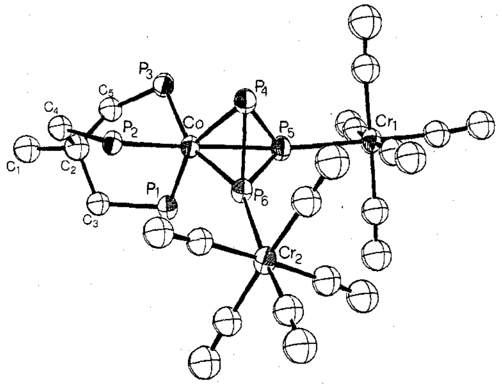

Figure 3. Skeleton of the complex molecule $[(\text{triphos})Co(\mu\text{-}(\eta^3\text{-}P_3))\{Cr_2(CO)_{10}\}]$ (ORTEP drawing with 30% probability ellipsoids).

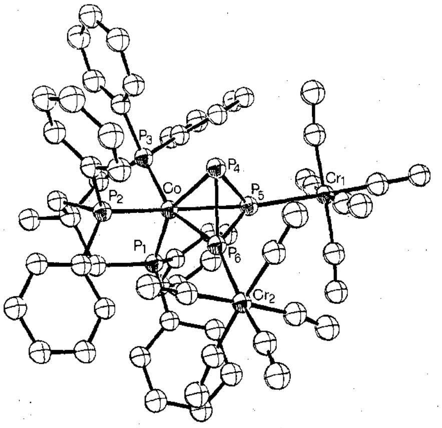

Figure 4. Perspective view of the complex molecule $[(\text{triphos})Co(\mu\text{-}(\eta^3\text{-}P_3))\{Cr_2(CO)_{10}\}]$ (ORTEP drawing with 30% probability ellipsoids).

the solid-state (Nujol mull) spectrum of $[(\text{triphos})Co(\mu\text{-}(\eta^3\text{-}P_3))Cr(CO)_5]$ is in agreement with this assignment. The very weak band at 1990 cm^{-1} may be attributed to the formally infrared inactive B_1 mode which becomes IR active due to the ligand-induced molecular distortion.

The X-ray analysis of the $[(\text{triphos})Co(\mu\text{-}(\eta^3\text{-}P_3))\{Cr_2(CO)_{10}\}]$ has shown that two phosphorus atoms of the *cyclo*-triphosphorus group are linked to the two chromium atoms of two $Cr(CO)_5$ fragments. Figures 3 and 4 show perspective views of the molecule. Bond lengths and angles are given in Table VIII.

The $(\text{triphos})Co(\eta^3\text{-}P_3)$ moiety is essentially unchanged with respect to the mononuclear donor complex, the small differences in bond lengths being insignificant. The chromium atoms are out of the plane containing the η^3-P_3 unit, the angles between the P–Cr linkages and the η^3-P_3 plane being 114.7 and 109.7°, respectively. The Co–P–Cr bond angles are 165.3 (2) and 157.1 (2)°, this difference being probably due to packing effects. The Cr–P–P bond angles range from 123.2 to 138.0°. The Cr–P bond distances, averaging 2.425 (4) Å, are the same as those in other phosphine–chromium complexes, e.g., in *cis*-(methoxycarbene)(triphenylphosphine)tetracarbonylchromium (2.42 (1) Å)[21] and in pentacarbonyl(triphenylphosphine)chromium (2.422 (1) Å).[22] The values of both cis and trans Cr–C bond distances agree well with other reported Cr–C linkages.[22] On the other hand, the Cr–C distances for CO in cis positions to the phosphorus (1.86 (2) Å) are significantly longer than the Cr–C distances trans to the phosphorus (1.80 (2) Å). This difference can be explained by assuming that the CO trans to phosphorus competes with the same phosphorus to form a partial multiple Cr–C bond.

Table VIII. $[(\text{triphos})Co(\mu\text{-}(\eta^3\text{-}P_3))\{Cr_2(CO)_{10}\}]$ Selected Bond Lengths (Å) and Angles (Deg) with Estimated Standard Deviations

Co–P(1)	2.195 (4)	P(1)–C(1,1)	1.84 (3)
Co–P(2)	2.198 (4)	P(1)–C(1,2)	1.83 (4)
Co–P(3)	2.219 (4)	P(2)–C(4)	1.84 (1)
Co–P(4)	2.340 (4)	P(2)–C(1,3)	1.84 (4)
Co–P(5)	2.303 (4)	P(2)–C(1,4)	1.84 (3)
Co–P(6)	2.270 (4)	P(3)–C(5)	1.84 (1)
P(4)–P(5)	2.145 (6)	P(3)–C(1,5)	1.83 (3)
P(4)–P(6)	2.154 (6)	P(3)–C(1,6)	1.83 (4)
P(5)–P(6)	2.124 (4)	O(6)–C(6)	1.17 (2)
Cr(1)–P(5)	2.435 (4)	O(7)–C(7)	1.14 (2)
Cr(1)–C(6)	1.85 (2)	O(8)–C(8)	1.15 (2)
Cr(1)–C(7)	1.87 (2)	O(9)–C(9)	1.15 (2)
Cr(1)–C(8)	1.86 (2)	O(10)–C(10)	1.18 (2)
Cr(1)–C(9)	1.86 (2)	O(11)–C(11)	1.15 (2)
Cr(1)–C(10)	1.79 (2)	O(12)–C(12)	1.16 (2)
Cr(2)–P(6)	2.416 (4)	O(13)–C(13)	1.16 (2)
Cr(2)–C(11)	1.86 (1)	O(14)–C(14)	1.15 (2)
Cr(2)–C(12)	1.86 (2)	O(15)–C(15)	1.18 (2)
Cr(2)–C(13)	1.86 (2)	C(1)–C(2)	1.57 (2)
Cr(2)–C(14)	1.84 (2)	C(2)–C(3)	1.55 (2)
Cr(2)–C(15)	1.81 (2)	C(2)–C(4)	1.52 (2)
P(1)–C(3)	1.85 (1)	C(2)–C(5)	1.55 (2)

P(1)–Co–P(2)	95.9 (1)	P(6)–Cr(2)–C(15)	174.0 (6)
P(1)–Co–P(3)	91.5 (2)	Co–P(4)–P(5)	61.6 (1)
P(1)–Co–P(4)	156.6 (2)	Co–P(4)–P(6)	60.5 (1)
P(1)–Co–P(5)	104.3 (1)	P(5)–P(4)–P(6)	59.2 (2)
P(1)–Co–P(6)	104.9 (2)	Co–P(5)–P(4)	63.3 (1)
P(2)–Co–P(3)	90.4 (1)	Co–P(5)–P(6)	61.5 (1)
P(2)–Co–P(4)	100.4 (1)	P(4)–P(5)–P(6)	60.6 (2)
P(2)–Co–P(5)	151.5 (1)	Co–P(5)–Cr(1)	165.3 (2)
P(2)–Co–P(6)	100.4 (1)	P(4)–P(5)–Cr(1)	123.2 (2)
P(3)–Co–P(4)	105.0 (2)	P(6)–P(5)–Cr(1)	132.9 (2)
P(3)–Co–P(5)	108.6 (1)	Co–P(6)–P(4)	63.8 (2)
P(3)–Co–P(6)	159.1 (2)	Co–P(6)–P(5)	63.1 (1)
P(4)–Co–P(5)	55.0 (1)	P(4)–P(6)–P(5)	60.2 (2)
P(4)–Co–P(6)	55.7 (1)	Co–P(6)–Cr(2)	157.1 (2)
P(5)–Co–P(6)	55.3 (1)	P(4)–P(6)–Cr(2)	129.6 (2)
P(5)–Cr(1)–C(10)	173.6 (5)	P(5)–P(6)–Cr(2)	138.0 (2)
P(5)–Cr(1)–C(6)	96.9 (5)	P(6)–Cr(2)–C(11)	91.4 (4)
P(5)–Cr(1)–C(7)	87.9 (5)	P(6)–Cr(2)–C(12)	98.5 (5)
P(5)–Cr(1)–C(8)	86.6 (4)	P(6)–Cr(2)–C(13)	89.2 (5)
P(5)–Cr(1)–C(9)	92.0 (5)	P(6)–Cr(2)–C(14)	85.9 (5)
P(5)–Cr(1)–C(10)	173.6 (5)	P(6)–Cr(2)–C(15)	174.0 (6)
C(6)–Cr(1)–C(7)	89.3 (7)	C(11)–Cr(2)–C(12)	89.3 (6)
C(6)–Cr(1)–C(8)	176.3 (6)	C(11)–Cr(2)–C(13)	178.0 (7)
C(6)–Cr(1)–C(9)	90.3 (7)	C(11)–Cr(2)–C(14)	90.9 (6)
C(6)–Cr(1)–C(10)	88.8 (7)	C(11)–Cr(2)–C(15)	87.8 (6)
C(7)–Cr(1)–C(8)	90.1 (7)	C(12)–Cr(2)–C(13)	88.7 (7)
C(7)–Cr(1)–C(9)	179.6 (9)	C(12)–Cr(2)–C(14)	175.5 (7)
C(7)–Cr(1)–C(10)	89.5 (7)	C(12)–Cr(2)–C(15)	87.4 (7)
C(8)–Cr(1)–C(9)	90.3 (7)	C(13)–Cr(2)–C(14)	91.0 (7)
C(8)–Cr(1)–C(10)	87.6 (6)	C(13)–Cr(2)–C(15)	91.8 (7)
C(9)–Cr(1)–C(10)	90.6 (7)	C(14)–Cr(2)–C(15)	88.1 (7)

The equality of the Cr–P bond length in the present complex with the Cr–P bond length in the $Cr(PPh_3)(CO)_5$ complex and the closeness of the CO stretching frequencies E mode[22] (1945 and 1942 cm^{-1} for the two complexes, respectively) suggest that the donor–acceptor properties of the phosphorus atoms of the *cyclo*-triphosphorus group are similar to those of the phosphorus atoms of the triphenylphosphine. In this framework the bond between the cobalt and the η^3-P_3 unit can be also viewed in terms of localized electron pair σ bonds between the metal and the three phosphorus atoms of the *cyclo*-triphosphorus unit.

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Registry No. (triphos)Co(η^3-P_3), 66745-30-0; (triphos)Co(μ-(η^3-P_3))($Cr_2(CO)_{10}$), 69102-14-3; (triphos)Co(μ-(η^3-P_3))$Cr(CO)_5$, 69102-15-4; P_4, 12185-10-3; $Cr(CO)_6$, 13007-92-6; $Co(H_2O)_6(BF_4)_2$, 37041-75-1.

Supplementary Material Available: Listings of structure factor amplitudes for [(triphos)Co(η^3-P_3)] and [(triphos)Co(μ-(η^3-P_3))-{$Cr_2(CO)_{10}$}] (27 pages). Ordering information is given on any current masthead page.

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Structural Studies on Polynuclear Osmium Carbonyl Hydrides. 14.[1,2] X-ray Diffraction Study of $(\mu\text{-}H)_4Os_4(CO)_{11}(CNMe)$, Including the Direct Location of All Four Bridging Hydride Ligands

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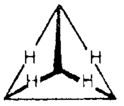
The complex $(\mu\text{-}H)_4Os_4(CO)_{11}(CNMe)$ crystallizes in the monoclinic space group $C2/c$ with a = 8.4605 (16) Å, b = 16.484 (4) Å, c = 15.620 (3) Å, β = 97.73 (2)°, V = 2158.6 (8) Å^3, and ρ(calcd) = 3.43 g cm^{-3} for mol wt 1114.0 and Z = 4. Diffraction data were collected with a Syntex $P2_1$ automated four-circle diffractometer using Mo Kα radiation and a θ–2θ scan technique. The structure was solved via Patterson, Fourier, and least-squares refinement techniques; convergence was reached with R_F = 3.5% and R_{wF} = 3.0% for all 1412 independent data (none rejected). The molecule lies on a site of C_2 symmetry, and there is disorder of one CO ligand and the CNMe ligand. The $(\mu\text{-}H)_4Os_4$ core approximates to D_{2d} symmetry with the hydrido-bridged Os–Os vectors averaging 2.963 Å (range 2.956 (1)–2.971 (1) Å) and the nonbridged Os–Os bond lengths being 2.822 (1) Å. The hydride ligands occupy the exterior bisecting positions about the appropriate tetrahedral edges. The Os–Os–CO angles and the nature of the disorder in the crystal are discussed in detail.

Introduction

A variety of tetranuclear ruthenium carbonyl complexes derived from $(\mu\text{-}H)_4Ru_4(CO)_{12}$ have been examined crystallographically. The species $(\mu\text{-}H)_4Ru_4(CO)_{12}$,[3] $(\mu\text{-}H)_4Ru_4(CO)_{10}(PPh_3)_2$,[3] and $(\mu\text{-}H)_4Ru_4(CO)_{11}[P(OMe)_3]$[4] are based on a $(\mu\text{-}H)_4M_4$ core of D_{2d} symmetry (I), whereas the species $(\mu\text{-}H)_4Ru_4(CO)_{10}$(diphos)[5] and $(\mu\text{-}H)_4Ru_4(CO)_{10}$($\mu$-diphos)[6] have a $(\mu\text{-}H)_4M_4$ core of C_s symmetry (II).



I

II

The situation with tetranuclear osmium carbonyl hydrides is rather less well developed although X-ray structural studies of $[NMe_4^+][(\mu\text{-}H)_3Os_4(CO)_{12}^-]$,[7] $[(PPh_3)_2N^+]_2[(\mu\text{-}H)_2Os_4(CO)_{12}^{2-}]$,[8] and $(\mu\text{-}H)_3(\mu\text{-}I)Os_4(CO)_{12}$[9] have appeared. However, in none of these studies have the bridging hydride ligands been located directly.

We now report the results of single-crystal X-ray structural analysis of the complex $(\mu\text{-}H)_4Os_4(CO)_{11}(CNMe)$, which was prepared by hydrogenation of $(\mu\text{-}H)Os_3(CO)_{10}(CNHMe)$ by Shapley and co-workers.[10] Despite a disorder problem (vide infra) the molecular structure of this new complex has been elucidated, and the hydride ligands have been located directly and their positions optimized by least-squares refinement.

Table I. Experimental Data for the Diffraction Study on $(\mu\text{-}H)_4Os_4(CO)_{11}(CNMe)$

(A) Crystal Parameters at 25 °C[a]

cryst class: monoclinic
space group: $C2/c$
a = 8.4605 (16) Å
b = 16.484 (4) Å
c = 15.620 (3) Å
β = 97.73 (2)°
V = 2158.6 (8) Å^3
Z = 4
mol wt 1114.0
ρ(calcd) = 3.43 g cm^{-3}

(B) Measurement of Intensity Data

diffractometer: Syntex $P2_1$
radiation: Mo Kα (λ 0.710 730 Å); pyrolytic graphite monochromator
rflctns measd: $\pm h, +k, \pm l$ (2 forms); 2θ = 4.5–45°
scan type: coupled 2θ(counter)–θ(cryst) at 2.0°/min in 2θ
scan width: $[2\theta(K\alpha_1) - 1.0]° \rightarrow [2\theta(K\alpha_2) + 1.1]°$
bkgd measmt: stationary crystal, stationary counter at each end of scan range; each measured for one-fourth the scan time
rflctns collected: 2816 total, yielding 1412 symmetry-independent data (R_{av} = 2.7% for 1404 averaged pairs of reflections)
abs coeff: μ = 249 cm^{-1}

(C) Data for Absorption Corrections

hkl	2θ, deg	I_{max}/I_{min}[b]
$\pm(3\bar{1}\bar{1})$	14.68	1.23
$\pm(40\bar{2})$	20.87	1.22
$\pm(5\bar{1}1)$	25.05	1.21
$\pm(60\bar{2})$	30.60	1.21
$\pm(7\bar{3}\bar{3})$	35.13	1.20

[a] Unit cell parameters were derived from a least-squares fit to the setting angles of the unresolved Mo K$\bar{\alpha}$ components of 24 reflections of the forms {0,4,10}, {196}, {22$\bar{9}$}, {513}, {51$\bar{5}$}, and {48$\bar{4}$}–all with 2θ between 25 and 30°. [b] Average for the two Friedel-related reflections.

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