

found on successive Fourier syntheses. Anisotropic temperature factors were used for all non-H atoms. H atoms were given fixed isotropic temperature factors, $U = 0.07 \text{ \AA}^2$. All were visible on difference Fourier syntheses, but proved slightly unstable on refinement. A weak C-H constraint [0.94 (2) Å] was applied to H atoms attached to C(14), C(17) and C(18), those nearest the metal atom and likely to be most affected by diffraction ripples. These atoms then refined to chemically satisfactory positions. Methyl groups were treated as rigid CH₃ units, with their initial orientation taken from the strongest H-atom peaks on a difference Fourier synthesis. Final refinement was on F by cascaded least-squares methods refining 283 parameters. Largest positive and negative peaks on a final difference Fourier synthesis were of height 0.7 and -0.6 e \AA^{-3} . A weighting scheme of the form $w = 1/(\sigma^2 F + gF^2)$ with $g = 0.00013$ was used and shown to be satisfactory by a weight analysis. Final $R = 0.046$, $wR = 0.039$. Maximum shift/e.s.d. in final cycle 0.4. Computing was with *SHELXTL* on a Data General DG30. Scattering factors in the analytical form and anomalous-dispersion factors taken from *International Tables for X-ray Crystallography* (1974). Final atomic coordinates are given in Table 1, and selected bond lengths and angles in Table 2.*

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and full bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51540 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. Figs. 1(a) and 1(b) show views of the complex parallel and perpendicular to the Rh coordination plane respectively. The molecular configuration is more obvious from Fig. 1(b). The unit cell is a racemate of this form and its enantiomer. Thus only one of the two possible configurational isomers of the coordinated amide, with *R re* relative configuration is observed. This corresponds to the stereoisomer which would be disfavoured on the directed hydrogenation pathway and it is notable that the amide remains uncoordinated. The torsion angle which defines steric interactions around the coordinated olefin is $\text{H}_3\text{C}-\text{C}-\text{C}_\alpha-\text{CO}_2\text{Me}$. In the crystal this is $-54.4 (5)^\circ$ but inspection of molecular models indicates that chelate coordination of the amide carbonyl group would alter this to around 0° and entail severe eclipsing interactions between the CH₃ and ester group (*cf.* 2b).

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The Structure of $[\text{Co}_4(\text{CO})_8(\mu_4\text{-PPh})_2\{\text{P}(\text{OEt})_3\}_2]$. Further Evidence for Thermodynamic Product Control in Ligand Substitution Reactions Involving $\text{Co}_4(\text{CO})_{10}(\mu_4\text{-PPh})_2$

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Abstract. Di- μ -carbonyl-hexacarbonyl-bis(μ_4 -diphenylphosphindene)-bis(triethoxyphosphine)tetracobalt(I)-

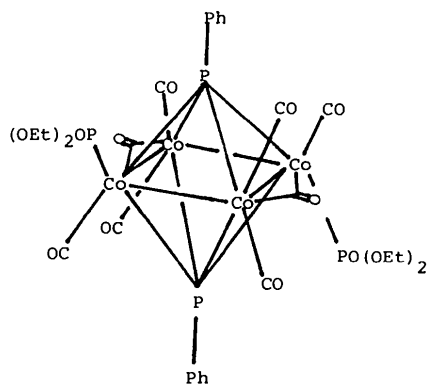
(4Co-Co), $[\text{Co}_4(\text{CO})_8(\text{C}_6\text{H}_5\text{P})_2\{\text{P}(\text{C}_2\text{H}_5\text{O})_3\}_2]$, $M_r = 1008.29$, monoclinic, $P2_1/n$, $a = 10.348 (1)$, $b = 17.186 (2)$, $c = 12.311 (1) \text{ \AA}$, $\beta = 106.07 (1)^\circ$, $V = 2103.7 (4) \text{ \AA}^3$, $Z = 2$, $D_x = 1.592 \text{ g cm}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71073 \text{ \AA}$, $\mu = 17.56 \text{ cm}^{-1}$, $F(000) = 1024$, $T = 293 \text{ K}$, $R = 0.0349$ for 3209 independent reflections.

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The Co atoms are in a rectangular array with capping μ_4 -phenylphosphinidene groups while the two $\text{P}(\text{OEt})_3$ ligands are arranged in a 1,3-*trans* configuration with respect to inversion through the tetracobalt plane. The phenyl groups of the phosphinidene caps are coplanar and their planes approximately bisect the μ_2 -carbonyl-bridged Co—Co bonds.

Introduction. Homogeneous catalysis of organic reactions by ligand-tethered clusters has received much attention (Whyman, 1980; Pittman & Ryan, 1978). For example, the bicapped tetracobalt cluster $[\text{Co}_4(\text{CO})_{10}(\mu_4\text{-PPh})_2]$ and its phosphine-substituted derivatives have been extensively studied in olefin hydrogenation and hydroformylation reactions (Ryan, Pittman & O'Connor, 1977; Pittman, Wilemon, Wilson & Ryan, 1980; Pittman, Richmond, Wilemon & Absi-Halibi, 1984). Our interest in this tetracobalt cluster stems from the varied ligand regio- and stereochemistry attendant upon substitution (Richmond, Korp & Kochi, 1985; Richmond & Kochi, 1987a). Carbonyl fluxionality and steric effects within the tetracobalt cluster have been shown to afford thermodynamically equilibrated products in ligand substitution reactions involving $[\text{P}(\text{OMe})_3]$ (Richmond & Kochi, 1986, 1987b). To test the generality of thermodynamic product control in determining the course of ligand substitution in this genre of cluster we have initiated substitution studies involving phosphines and phosphites of varying steric and electronic properties. The single-crystal X-ray determination of $[\text{Co}_4(\text{CO})_8(\mu_4\text{PPh})_2\{\text{P}(\text{OEt})_3\}_2]$, the product from the reaction of $[\text{Co}_4(\text{CO})_{10}(\mu_4\text{-PPh})_2]$ and $[\text{P}(\text{OEt})_3]$ (2 eq.), was undertaken to establish the disposition of the ancillary $[\text{P}(\text{OEt})_3]$ groups about the cobalt cluster.



Experimental. The title compound was prepared from the reaction of 0.2 g of $[\text{Co}_4(\text{CO})_{10}(\mu_4\text{-PPh})_2]$ (0.27 mmol) and 0.1 ml of $[\text{P}(\text{OEt})_3]$ (0.6 mmol) in benzene. The reaction was stirred at 323 K for 24 h at which time TLC examination indicated ~85% conversion to the bis-substituted cluster. Purification by chromatography using silica gel and a 7:3 (*v/v*) mixture

of petroleum ether and benzene afforded 0.15 g (55.1%) of red $[\text{Co}_4(\text{CO})_8(\mu_4\text{-PPh})_2\{\text{P}(\text{OEt})_3\}_2]$. Crystals suitable for X-ray diffraction analysis were grown by slowly evaporating the CH_2Cl_2 from a CH_2Cl_2 /heptane solution containing the bis-phosphite cluster. Crystal of dimensions 0.23 \times 0.28 \times 0.43 mm; Nicolet R3m/ μ update of P2₁ diffractometer; data collected in ω -scan mode ($3 \leq 2\theta \leq 50^\circ$), scan rate 4 to 29.3° min⁻¹, graphite-monochromated Mo K α radiation, lattice parameters from a least-squares refinement of 25 reflections ($47.92 \leq 2\theta \leq 49.91^\circ$), angles measured by a centering routine; Laue symmetry and statistics consistent with space group P2₁/*n*; monitored reflections ($\bar{6}22$ and 294) show no statistical changes in intensities; 3706 independent reflections measured ($-12 \leq h \leq 11$, $0 \leq k \leq 20$, $0 \leq l \leq 14$), $3209 \geq 3\sigma(I)$; Lorentz-polarization corrections, ψ -scan-based empirical absorption correction (transmission factors 0.923 to 0.635); structure solved by direct methods, block-cascade least-squares refinement; H atoms were located in difference maps and refined with isotropic thermal parameters except for five H atoms associated with side chain C(5)C(6) which were allowed to ride at fixed distances from the C atoms with fixed isotropic thermal parameter; final $R = 0.0349$, $wR = 0.040$ for 305 parameters and 3209 reflections ($R = 0.0420$, $wR = 0.0407$ for all data), $S = 1.513$, $(\Delta/\sigma)_{\text{max}} = 0.034$, $(\Delta/\sigma)_{\text{av}} = 0.008$; largest peaks in final difference map +0.44 and -0.62 e Å⁻³; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0.00024F_o^2]^{-1}$; extinction correction $F^* = F_c/[1.0 + 0.002gF_c^2/\sin(2\theta)]^{0.25}$ applied, g refined to 0.00020 (6). All computer programs supplied by Nicolet (Nicolet Instrument Corporation, 1986) for Desktop 30 Micro-eclipse and Nova 4/C configuration; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Table 1 lists atomic positional parameters for $[\text{Co}_4(\text{CO})_8(\mu_4\text{-PPh})_2\{\text{P}(\text{OEt})_3\}_2]$ while Table 2 presents bond distances and valence angles.† Fig. 1 is a drawing of the title compound.

Discussion. The structure of $[\text{Co}_4(\text{CO})_8(\mu_4\text{-PPh})_2\{\text{P}(\text{OEt})_3\}_2]$ consists of four Co atoms in a rectangular array whose shorter sides are bridged by an asymmetric μ_2 -carbonyl group. The two carbonyl-bridged Co—Co bonds of 2.523 (1) Å (av.) are 0.185 Å shorter than the non-carbonyl-bridged Co—Co bonds of 2.708 (1) Å. The capping of the cobalt rectangle by a pair of μ_4 -phenylphosphinidene groups gives rise to an overall octahedral Co_4P_2 core. The four Co atoms and

† Lists of H-atom coordinates, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51621 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($\times 10^4$) and isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)

	x	y	z	U_{eq}^*
Co(1)	6092 (1)	5065 (1)	1459 (1)	29 (1)
Co(2)	5902 (1)	4078 (1)	-94 (1)	30 (1)
P(1)	7720 (1)	5865 (1)	2205 (1)	35 (1)
O(1)	7754 (2)	6600 (1)	1438 (2)	46 (1)
C(1)	8811 (5)	7183 (3)	1689 (4)	64 (2)
C(2)	8417 (5)	7829 (3)	891 (4)	69 (2)
O(2)	7724 (2)	6224 (1)	3408 (2)	46 (1)
C(3)	6636 (4)	6735 (2)	3486 (4)	57 (2)
C(4)	6587 (5)	6741 (3)	4690 (4)	84 (2)
O(3)	9203 (2)	5535 (2)	2508 (2)	54 (1)
C(5)	10154 (7)	5432 (7)	3455 (6)	250 (7)
C(6)	11286 (7)	5267 (5)	3797 (6)	165 (5)
C(7)	5598 (4)	4924 (2)	2717 (3)	44 (1)
O(7)	5276 (4)	4859 (2)	3524 (2)	77 (1)
C(8)	7106 (3)	4144 (2)	1481 (3)	36 (1)
O(8)	7943 (2)	3758 (1)	2047 (2)	51 (1)
C(9)	7067 (4)	3909 (2)	-892 (3)	48 (1)
O(9)	7753 (3)	3764 (2)	-1442 (3)	84 (1)
C(10)	5308 (3)	3102 (2)	-61 (3)	44 (1)
O(10)	4974 (3)	2475 (2)	-91 (3)	76 (1)
P(2)	4132 (1)	4622 (1)	342 (1)	28 (1)
C(11)	2960 (3)	4101 (2)	934 (2)	33 (1)
C(12)	1611 (3)	4279 (2)	620 (3)	43 (1)
C(13)	741 (4)	3890 (3)	1106 (3)	57 (1)
C(14)	1217 (4)	3323 (2)	1902 (3)	58 (2)
C(15)	2558 (4)	3136 (2)	2200 (3)	52 (1)
C(16)	3427 (3)	3524 (2)	1720 (3)	40 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

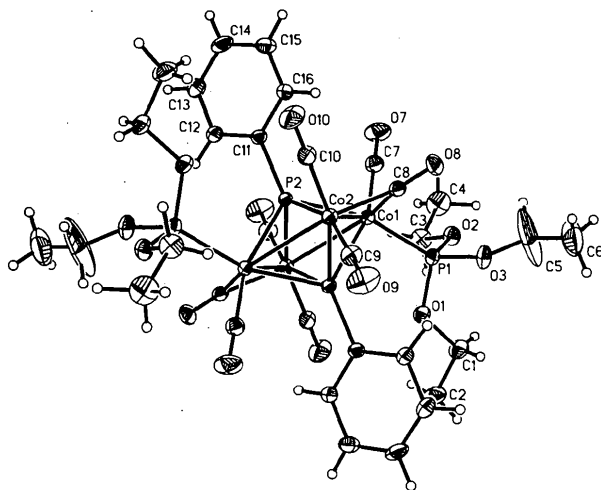


Fig. 1. Drawing of the title compound with thermal ellipsoids drawn at the 30% probability level.

the two bridging carbonyl groups are planar (0.005 Å r.m.s.d.) with the P(2) atoms lying 1.271 (4) Å out of the plane.

The plane of the phenyl ring makes an angle of 85.4 (5)° with the Co-atom plane and is oriented with the phenyl plane bisecting the shorter carbonyl-bridged Co-Co bonds. The two phenyl groups are coplanar. The Co-P(μ₄) bonds of 2.245 (9) Å (av.) agree well with those observed in the parent cluster [Co₄(CO)₁₀-

Table 2. Bond lengths (Å) and bond angles (°)

Co(1)—Co(2)	2.523 (1)	Co(1)—P(1)	2.171 (1)
Co(1)—C(7)	1.776 (4)	Co(1)—C(8)	1.896 (3)
Co(1)—P(2)	2.245 (1)	Co(1)—Co(2A)	2.708 (1)
Co(1)—P(2A)	2.231 (1)	Co(2)—C(8)	1.996 (3)
Co(2)—C(9)	1.777 (4)	Co(2)—C(10)	1.791 (3)
Co(2)—P(2)	2.249 (1)	Co(2)—Co(1A)	2.708 (1)
Co(2)—P(2A)	2.254 (1)	P(1)—O(1)	1.584 (3)
P(1)—O(2)	1.603 (3)	P(1)—O(3)	1.580 (2)
O(1)—C(1)	1.452 (5)	C(1)—C(2)	1.464 (6)
O(2)—C(3)	1.452 (5)	C(3)—C(4)	1.498 (7)
O(3)—C(5)	1.313 (7)	C(5)—C(6)	1.164 (10)
C(7)—O(7)	1.137 (5)	C(8)—O(8)	1.158 (4)
C(9)—O(9)	1.136 (6)	C(10)—O(10)	1.128 (4)
P(2)—C(11)	1.815 (3)	P(2)—Co(1A)	2.231 (1)
P(2)—Co(2A)	2.254 (1)	P(2)—P(2A)	2.541 (2)
C(11)—C(12)	1.376 (4)	C(11)—C(16)	1.376 (4)
C(12)—C(13)	1.383 (6)	C(12)—C(14)	1.372 (6)
C(14)—C(15)	1.372 (6)	C(15)—C(16)	1.377 (6)
Co(2)—Co(1)—P(1)	129.4 (1)	Co(2)—Co(1)—C(7)	126.0 (1)
P(1)—Co(1)—C(7)	95.4 (1)	Co(2)—Co(1)—C(8)	51.4 (1)
P(1)—Co(1)—C(8)	99.3 (1)	C(7)—Co(1)—C(8)	99.6 (2)
Co(2)—Co(1)—P(2)	55.9 (1)	P(1)—Co(1)—P(2)	159.6 (1)
C(7)—Co(1)—P(2)	93.4 (1)	C(8)—Co(1)—P(2)	97.4 (1)
Co(2)—Co(1)—Co(2A)	90.5 (1)	P(1)—Co(1)—Co(2A)	106.7 (1)
C(7)—Co(1)—Co(2A)	105.2 (1)	C(8)—Co(1)—Co(2A)	141.9 (1)
P(2)—Co(1)—Co(2A)	53.1 (1)	Co(2)—Co(1)—P(2A)	56.2 (1)
P(1)—Co(1)—P(2A)	97.0 (1)	C(7)—Co(1)—P(2A)	157.4 (1)
C(8)—Co(1)—P(2A)	96.9 (1)	P(2)—Co(1)—P(2A)	69.2 (1)
Co(2A)—Co(1)—P(2A)	53.1 (1)	Co(1)—Co(2)—C(8)	47.9 (1)
Co(1)—Co(2)—C(9)	127.3 (1)	C(8)—Co(2)—C(9)	102.1 (1)
Co(1)—Co(2)—C(10)	124.6 (1)	C(8)—Co(2)—C(10)	99.1 (1)
C(9)—Co(2)—C(10)	98.6 (2)	Co(1)—Co(2)—P(2)	55.8 (1)
C(8)—Co(2)—F(2)	94.3 (1)	C(9)—Co(2)—P(2)	157.1 (1)
C(10)—Co(2)—P(2)	94.4 (1)	Co(1)—Co(2)—Co(1A)	89.5 (1)
C(8)—Co(2)—Co(1A)	137.4 (1)	C(9)—Co(2)—Co(1A)	105.2 (1)
C(10)—Co(2)—Co(1A)	108.4 (1)	P(2)—Co(2)—Co(1A)	52.5 (1)
Co(1)—Co(2)—P(2A)	55.3 (1)	C(8)—Co(2)—P(2A)	93.3 (1)
C(9)—Co(2)—P(2A)	94.3 (1)	C(10)—Co(2)—P(2A)	159.9 (1)
P(2)—Co(2)—P(2A)	68.7 (1)	Co(1A)—Co(2)—P(2A)	52.8 (1)
Co(1)—P(1)—O(1)	113.3 (1)	Co(1)—P(1)—O(2)	115.9 (1)
O(1)—P(1)—O(2)	104.4 (1)	Co(1)—P(1)—O(3)	117.5 (1)
O(1)—P(1)—O(3)	104.3 (1)	O(2)—P(1)—O(3)	99.5 (1)
P(1)—O(1)—C(1)	124.3 (2)	O(1)—C(1)—C(2)	109.1 (3)
P(1)—O(2)—C(3)	119.4 (2)	O(2)—C(3)—C(4)	108.0 (3)
P(1)—O(3)—C(5)	134.3 (4)	O(3)—C(5)—C(6)	141.8 (8)
Co(1)—C(7)—O(7)	177.9 (3)	Co(1)—C(8)—Co(2)	80.8 (1)
Co(1)—C(8)—O(8)	143.7 (3)	Co(2)—C(8)—O(8)	135.5 (3)
Co(2)—C(9)—O(9)	175.4 (3)	Co(2)—C(10)—O(10)	175.6 (4)
Co(1)—P(2)—Co(2)	68.3 (1)	Co(1)—P(2)—C(11)	120.8 (1)
Co(1)—P(2)—C(11)	124.7 (1)	Co(1)—P(2)—Co(1A)	110.8 (1)
Co(2)—P(2)—Co(1A)	74.4 (1)	C(11)—P(2)—Co(1A)	128.4 (1)
Co(1)—P(2)—Co(2A)	74.0 (1)	Co(2)—P(2)—Co(2A)	111.3 (1)
C(11)—P(2)—Co(2A)	123.8 (1)	Co(1A)—P(2)—Co(2A)	68.5 (1)
Co(1)—P(2)—P(2A)	55.1 (1)	Co(2)—P(2)—P(2A)	55.7 (1)
C(11)—P(2)—P(2A)	175.8 (1)	Co(1A)—P(2)—P(2A)	55.7 (1)
Co(2A)—P(2)—P(2A)	55.6 (1)	P(2)—C(11)—C(12)	121.1 (2)
P(2)—C(11)—C(16)	119.6 (2)	C(12)—C(11)—C(16)	119.3 (3)
C(11)—C(12)—C(13)	120.1 (3)	C(12)—C(13)—C(14)	120.3 (3)
C(13)—C(14)—C(15)	119.6 (4)	C(14)—C(15)—C(16)	120.2 (3)
C(11)—C(16)—C(15)	120.4 (3)		

(μ₄-PPh)₂] (Ryan & Dahl, 1975; Ryan, Pittman, O'Connor & Dahl, 1980).

The two [P(OEt)₃] ligands are oriented in a 1,3-*trans* configuration with respect to inversion through the midpoint of the tetracobalt plane. A Co(1)—P(1) distance of 2.171 (1) Å is unexceptional and requires no further comment. The regio- and stereochemical disposition of the [P(OEt)₃] groups about the cluster core is similar to that reported for [Co₄(CO)₈(μ₄-

$\text{PPh}_2\{\text{P(OMe)}_3\}_2$ (Richmond & Kochi, 1986). Finally, no significant steric interactions are observed within the cluster framework and the similarities between the two phosphite clusters suggest that thermodynamic product control directs the incoming $[\text{P(OEt)}_3]$ groups to their respective locations.

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Structure of Tris(cyclopentadienyl)uranium Bromide

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Abstract. $[\text{U}(\text{C}_5\text{H}_5)_3]\text{Br}$, $M_r = 513.23$, monoclinic, $P2_1/n$, $a = 14.138$ (4), $b = 8.233$ (2), $c = 24.743$ (4) Å, $\beta = 90.90$ (4)°, $V = 2879$ (2) Å³, $Z = 8$, $D_x = 2.367$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 134.180$ cm⁻¹, $F(000) = 1856$, $T = 295$ (1) K, $R = 0.023$ for 2642 observed reflections. The three cyclopentadienyl rings are η^5 -covalently bonded to the uranium atom to form a distorted tetrahedron with the bromine atom. Although geometrically equivalent, $[\text{U}(\text{C}_5\text{H}_5)_3]\text{Br}$ is not isostructural with $[\text{U}(\text{C}_5\text{H}_5)_3]\text{Cl}$.

Introduction. As part of our investigation on the bonding and coordination geometry in organoactinide complexes, we recently reported the structure of tris(indenyl)uranium bromide, $[\text{U}(\text{C}_9\text{H}_7)_3]\text{Br}$ (Spirlet, Rebizant & Goffart, 1987). It was found to be isostructural with the corresponding chloride derivative, $[\text{U}(\text{C}_9\text{H}_7)_3]\text{Cl}$ (Burns & Laubereau, 1971). Undertaking the crystal structure analysis of the title complex, we expected to verify its isomorphism with $[\text{U}(\text{C}_5\text{H}_5)_3]\text{Cl}$ (Wong, Yen & Lee, 1965).

Experimental. $[\text{U}(\text{C}_5\text{H}_5)_3]\text{Br}$ was prepared from uranium bromide and potassium cyclopentadienide in

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