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COBALT METALLOCYCLES

XIII *. PREPARATION AND X-RAY CRYSTALLOGRAPHY OF COBALTACYCLOPENTADIENE AND DINUCLEAR COBALT COMPLEXES **

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Summary

(η-Cyclopentadienyl)(triphenylphosphine)cobaltacyclopentadienes having an electron withdrawing substituent on the cyclopentadienyl ring, (η-C_5H_4R)-(PPh_3)($\overline{\mathrm{CoCH{=}CH{=}CH}}$) (**1b**: R = COOMe; **1c**: R = COMe), were prepared in reasonable yields by treatment of a solution of (η-C_5H_4R)$(PPh_3)_2$Co with acetylene. A non-substituted cyclopentadienyl analog (**1a**: R = H) was also isolated in low yield according to a similar procedure. Novel dinuclear complexes were also formed as by-products and the structure of (η-C_5H_4R)Co($\overline{\mathrm{PPh_2C_6H_4)(\mu\text{-}CMe}}$)Co($\eta$-$C_5H_4R$) (**2b**: R = COOMe), having a μ_2,η^3-benzyl moiety, was determined by an X-ray crystallographic analysis. The X-ray analyses of **1a** and **1b** were also carried out. Crystals of **1a** are monoclinic, space group *Pa*, *a* 8.529(3), *b* 16.010(6), *c* 8.028(4) Å, β 100.31(3)°, $Z = 2$; crystals of **1b** are monoclinic, space group $P2_1/a$, *a* 8.327(2), *b* 36.468(7), *c* 8.021(1) Å, β 98.75(2)°, $Z = 4$; and crystals of **2b** are monoclinic, space group $P2_1/c$, *a* 10.681(2), *b* 30.722(7), *c* 8.912(1) Å, β 93.55(1)°, $Z = 4$. They have been refined to $R = 0.034$, 0.047 and 0.050, respectively.

Introduction

We have shown in earlier reports of this series that cobaltacyclopentadiene complexes having the formula (η-C_5H_5)(PPh_3)($\overline{\mathrm{CoCR{=}CR{-}CR{=}CR}}$) can be obtained readily by the reaction of η-cyclopentadienylbis(triphenylphosphine)cobalt with a variety of acetylenes [2,3]. Although many metallacyclopentadiene complexes have been hitherto known [1], none of them have an unsubstituted metalloring

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* For part XII see Ref. 1.

** Dedicated to Prof. S. Otsuka.

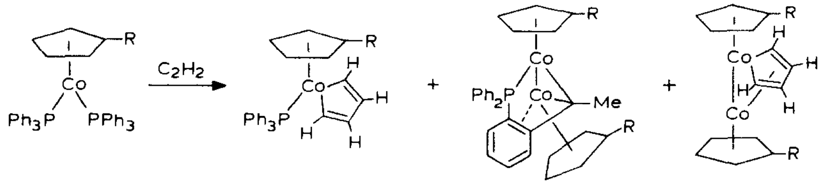
except some dinuclear complexes of molybdenum [5], iron [1,6,7] and cobalt [8] in which the diene moiety of the ring coordinates to an other metal. Accordingly, it is of interest to attempt the preparation of a mononuclear non-substituted cobaltacyclopentadiene by the reaction of η-cyclopentadienylbis(triphenylphosphine)cobalt with acetylene itself.

Now, we wish to report successful isolation and the X-ray crystallographic analyses of such cobaltacyclopentadienes, together with structure determination of a novel dinuclear cobalt complex isolated as a by-product.

Results and discussion

Our earlier attempt to prepare non-substituted cobaltacyclopentadiene using a similar procedure to that employed for substituted ones, which involves the reaction of η-cyclopentadienylbis(triphenylphosphine)cobalt with acetylenes at room temperature, was unsuccessful. We assumed that the difficulty of isolation of the non-substituted cobaltacyclopentadiene mostly arose from ready dissociation of triphenylphosphine ligand leading to decomposition of the complex. We have already shown that the ease of dissociation of the phosphine ligand from $(\eta\text{-}C_5H_5)(PPh_3)(\overline{CoCR{=}CR{-}CR{=}C}R)$ can be affected by the nature of substituents (R) of the cobaltacyclopentadiene ring i.e. electron withdrawing substituents result in a stronger cobalt to phosphine bond [9]. Expecting a similar trend, we introduced electron withdrawing substituents to the cyclopentadienyl ligand. The reaction condition was further improved by employing a large excess amount of triphenylphosphine in the reaction medium.

According to the reported procedure for η-cyclopentadienylbis(triphenylphosphine)cobalt [3], the dark red solution of (η-methoxycarbonylcyclopentadienyl)-bis(triphenylphosphine)cobalt was prepared from chlorotris(triphenylphosphine)cobalt and sodium methoxycarbonylcyclopentadienide [10]. Without isolation of the complex, acetylene was bubbled through the solution at room temperature. The red color turned brown immediately. From the solution orange brown crystals of the expected cobaltacyclopentadiene complex **1b** (4%) were isolated together with dark brown crystals of unknown compound **2b** (11%) and dark green crystals of known dinuclear cobaltacyclopentadiene complex **3b** [8] (2%) (eq. 1).



(1a-1c) (2a-2c) (3a-3c)

(R = H, COOMe, COMe)

In accordance with the structure, the ^1H NMR spectrum of **1** showed an $AA'XX'$ pattern of the cyclopentadienyl protons at δ 4.66 and 5.21 ppm as two triplets, and an $AA'MM'X$ pattern of the cobaltacyclopentadiene ring protons coupled with the phosphorus nucleus at δ 5.92 and 7.93 ppm as two multiplets, except for the resonances of methyl and phenyl protons. We assigned the signal at δ 5.92 to β and the one at δ 7.93 ppm to α protons, based on the chemical shifts of related complexes [1].

The ^{1}H NMR, IR spectra, and especially the elemental analysis of **2b** suggested the molecule was composed of two methoxycarbonylcyclopentadienylcobalt moieties, one triphenylphosphine molecule, and one acetylene molecule. In the ^{1}H NMR spectrum of **2b**, methyl protons (3H) appeared as a doublet (ca. 1 Hz) at δ 3.20 and methoxy protons (6H) as a singlet at δ 3.68 ppm which resulted from accidental degeneracy of resonances of two different methoxy groups. As the other signals showed complex multiplets and did not give enough information about the structure, the X-ray crystallographic analysis was carried out to elucidate the structure. A perspective view of the molecule is given in Fig. 1, together with the numbering scheme of the atoms. Selective bond lengths and angles are shown in Table 1. One of the important features of this structure is the existence of a bridging carbene carbon which is bonded to a methyl group and C(32), an *ortho* position of a phenyl group of triphenylphosphine ligand. One edge of a phenyl group, C(31) and C(32), also coordinates to one cobalt atom Co(1), thus making a $\mu_2\eta^3$-benzyl type bond. A μ-diphenylmethylene complex of a formula $(\eta\text{-}C_5H_5)_2(CO)_4Mo_2(\mu\text{-}CPh_2)$ [11] has been shown to have similar bonding by X-ray crystallography. The formation of **2b** may be explained by the following scheme (eq. 2): Formation of a μ-vinylidene-dicobalt complex from a reaction of a mononuclear vinylidenecobalt complex with a cyclopentadienylcobalt moiety and a subsequent addition of a C–H bond of one of the phenyls of coordinated triphenylphosphine to the vinylidene moiety through activation by a cobalt. Related vinylidenerhodium complexes, $(i\text{-}Pr_3P)(\eta\text{-}C_5H_5)\text{-}Rh(C{=}CH_2)$ [12] and $(CO)_2(\eta\text{-}C_9H_7)_2Rh_2(\mu\text{-}C{=}CH_2)$ [13], are known.

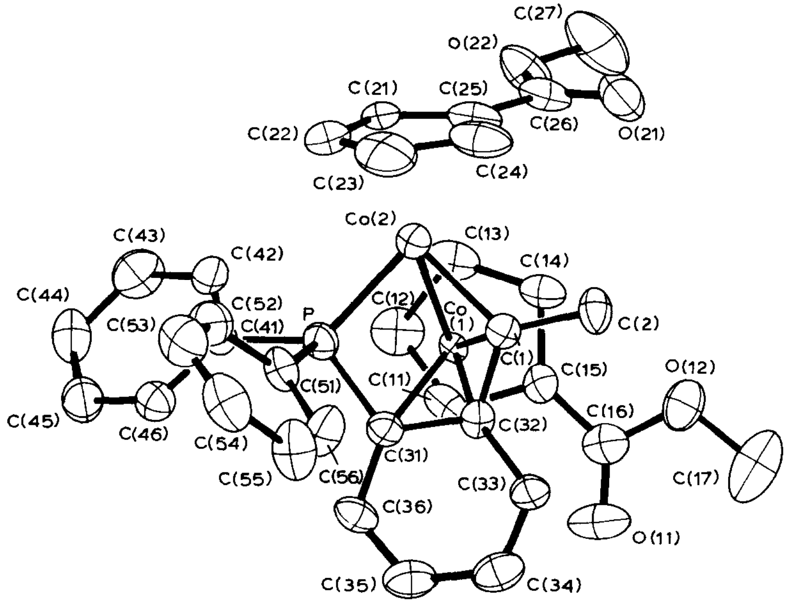


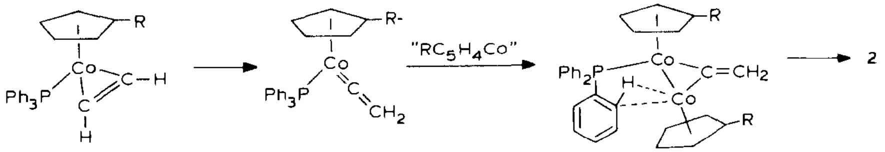
Fig. 1. Molecular diagram and atom labeling scheme for **2b**.

TABLE 1

SELECTED BOND DISTANCES (Å) AND ANGLES (°)[a] FOR **2b**

Co(1)-Co(2)	2.491(1)	C(1)-C(2)	1.509(8)
Co(1)-C(1)	1.971(5)	C(31)-C(32)	1.446(7)
Co(1)-C(31)	2.087(5)	C(31)-C(36)	1.445(8)
Co(1)-C(32)	2.032(5)	C(32)-C(33)	1.430(7)
Co(1)-C(11)	2.077(6)	C(33)-C(34)	1.368(9)
Co(1)-C(12)	2.095(8)	C(34)-C(35)	1.423(9)
Co(1)-C(13)	2.060(8)	C(35)-C(36)	1.357(8)
Co(1)-C(14)	2.068(6)	C(11)-C(12)	1.405(8)
Co(1)-C(15)	2.077(5)	C(11)-C(15)	1.438(8)
Co(2)-P	2.104(2)	C(12)-C(13)	1.420(8)
Co(2)-C(1)	1.918(5)	C(13)-C(14)	1.417(8)
Co(2)-C(21)	2.117(6)	C(14)-C(15)	1.430(8)
Co(2)-C(22)	2.092(7)	C(21)-C(22)	1.414(9)
Co(2)-C(23)	2.093(6)	C(21)-C(25)	1.426(8)
Co(2)-C(24)	2.103(6)	C(22)-C(23)	1.420(10)
Co(2)-C(25)	2.112(6)	C(23)-C(24)	1.409(9)
		C(24)-C(25)	1.415(8)
Co(1)-Co(2)-P	72.3(1)	P-C(31)-C(32)	110.7(4)
Co(1)-Co(2)-C(1)	51.1(2)	P-C(31)-C(36)	128.0(4)
P-Co(2)-C(1)	86.5(2)	C(32)-C(31)-C(36)	119.3(4)
Co(2)-Co(1)-C(1)	49.3(1)	C(1)-C(32)-C(31)	113.7(4)
Co(1)-C(1)-Co(2)	79.6(2)	C(1)-C(32)-C(33)	128.1(5)
		C(31)-C(32)-C(33)	117.8(5)

[a] Numbers in parentheses are estimated standard deviations



To minimize the formation of **2b**, the reaction of (η-methoxycarbonylcyclopentadienyl)bis(triphenylphosphine)cobalt with acetylene was carried out in the presence of excess acetylene. Thus, the solution of the cobalt complex was cooled with dry ice-acetone bath and acetylene was bubbled several minutes to allow sufficient dissolution and then the temperature was raised gradually until room temperature. This procedure increased the yield of **1b** (24%), and decreased that of **2b** (2%) as expected. A similar series of η-acetylcyclopentadienylcobalt complexes (**1c**, **2c** and **3c**) were also prepared by the reaction of (η-acetylcyclopentadienyl)bis(triphenylphosphine)cobalt with acetylene in reasonable yields.

According to the same procedure, even the unsubstituted cyclopentadienylcobalt analogue (η-cyclopentadienyl)(triphenylphosphine)cobaltacyclopentadiene(**1a**) could be isolated although the yield was low (1%). A dinuclear μ_2,η^3-benzyl complex (**2a**) was also formed (8%).

The complexes **1a**–**1c** are stable in air in the solid state. Analytical and ^{1}H NMR spectra data of the present complexes are summarized in Table 2.

The X-ray crystallographic structure analyses of **1a** and **1b** were carried out in

TABLE 2

PHYSICAL AND ANALYTICAL DATA

Compound	Yield [a] (%)	M.p. (°C)	Analyses (Found (calcd.) (%))		^{1}H NMR, δ(ppm) [e,f]		
			C	H	C_5H_4R	CoCH=CH-CH=CH	Me
1a	1	116–118 [b]	73.95 (73.97)	5.46 (5.52)	4.60	6.04 8.08	
1b	24	116–118 [b]	70.54 (70.16)	5.32 (5.28)	4.66 5.21	5.92 7.93	3.64
1c	24	117–119 [b]	72.95 (72.50)	5.54 (5.45)	4.63 5.34	5.97 7.92	1.97
2a	8	213–214 [b]	67.08 (67.18)	5.09 (5.07)	4.11 4.73		3.39
2b	2	153–155	62.64 (62.59)	4.77 (4.79)	3.8–5.4		3 20 (3H) 3.68 (6H)
2c	7	216–218	65 99 (65.82)	5.07 (5.04)	3.9–5.4		1.64, 1.81 3.27
3a	1	120 [b,c]					
3b [g]	3	68–70 [d]	51.94 (51.95)	4.34 (4.36)	~ 5 (6H) [h] ~ 5.5 (4H)	8.25(t, J4 Hz)	3.85 3.90
3c		oil			~ 5 (6H) [h] ~ 5.5 (4H)	8.15(t, J4 Hz)	2.32 2.38

[a] Yields were calculated on the basis of $(Ph_3P)_3CoCl$ employed. [b] Measured in a nitrogen filled capillary tube. [c] Ref. 8: 121–122 °C. [d] Ref. 8: 68.7–70 °C. [e] The spectra of **1** and **3** were taken in CD_2Cl_2 and those of **2** in $CDCl_3$. [f] Phenyl protons are ommitted. [g] The NMR spectrum is consistent with that reported [5]. [h] Two protons of cobaltacyclopentadiene are included.

order to know exact features of the structures and to compare with the known structure of (η-cyclopentadienyl)(triphenylphosphine)tetrakis(pentafluorophenyl)-cobaltacyclopentadiene (**1d**) [14]. Perspective views of the molecules, together with numbering of the atoms are given in Figs. 2 and 3. Both molecules take approximate piano stool configurations. In **1b**, the methoxycarbonyl substituent on the cyclopentadienyl ring is directed to the opposite side of the phosphine ligand, which may arise from steric interaction between them. Selected bond lengths and angles are shown in Table 3. The Co–P distances of **1a** (2.144(1) A) and **1b** (2.148(2) Å) are shorter than that of **1d** (2.234(3) Å), and Co–C(metallocycle) distances of **1a** (1.943(6) and 1.947(6) Å) and **1b** (1.928(6) and 1.933(7) Å) are also shorter than those of **1d** (1.995(1) and 1.993(11) Å). It is interesting to note that Co–C(metallocycle) distances are markedly affected by the steric requirement of substituents on the cobaltacyclopentadiene ring. The Co–C(metallocycle) bond lengths in the present study are in fair agreement with the value (1.934 A) calculated by ab initio molecular orbital method [2]. The observed slightly shorter bond lengths of Co–C(metallocycle) in **1b** than those of **1a** may suggest the electron-withdrawing substituent on the cyclopentadienyl ring affects stronger bonding interaction between the cobalt and carbon. Aromaticity of several metallacyclopentadiene rings has been discussed by Thorn and Hoffmann using extended Hückel method and occupation of electron in $\pi_{b_2}^*$ orbital of the C_4H_4 fragment was taken as a qualitative indication of delocalization. For a cobaltacyclopentadiene, $C_5H_5Co(PH_3)(C_4H_4)$, 0.10 electron occupation in the orbital was deduced from the fragment analysis, suggesting very small d_π–p_π interaction. The argument is con-

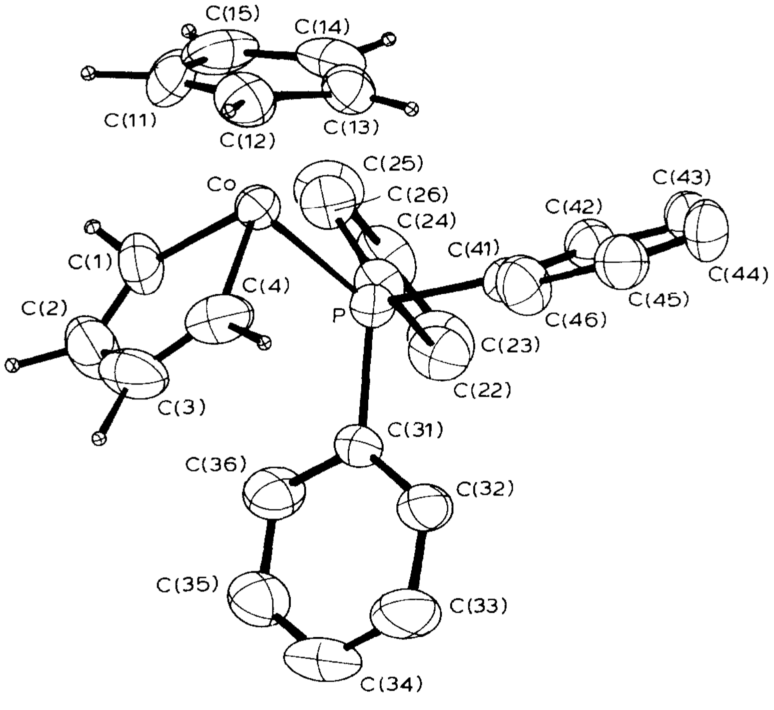


Fig. 2. Molecular diagram and atom labeling scheme for **1a**.

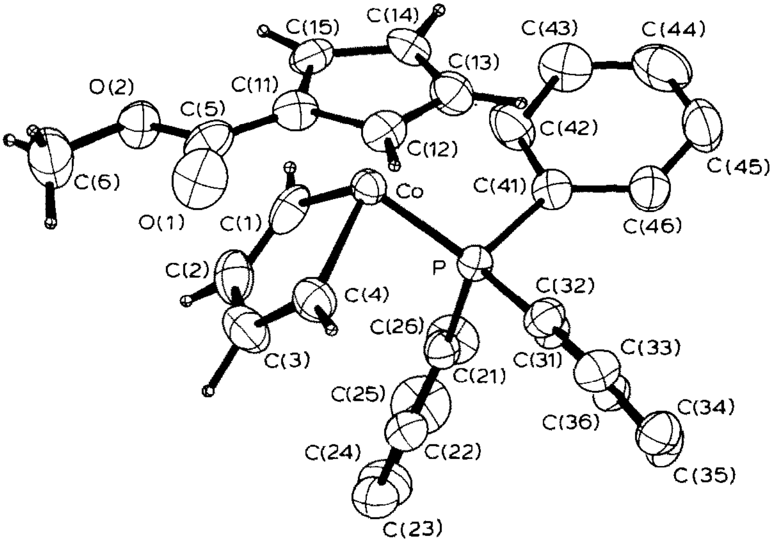


Fig. 3. Molecular diagram and atom labeling scheme for **1b**.

sistent with the observed bond length alternation in the present complexes since the difference between the formal double and single bonds will be smaller if the charge flow from the metal to C_4H_4 is effective. The double bond (av.) and the single bond lengths are 1.345 and 1.447 Å for **1a** and 1.337 and 1.442 Å for **1b** which are

TABLE 3

SELECTED BOND DISTANCES (Å) AND ANGLES (°)[a] FOR **1a** AND **1b**

	1a	**1b**
Co–P	2.144(1)	2.148(2)
Co–C(1)	1.943(6)	1.928(6)
Co–C(4)	1.947(6)	1.933(7)
Co–C(11)	2.085(6)	2.056(6)
Co–C(12)	2.122(5)	2.116(6)
Co–C(13)	2.136(6)	2.137(7)
Co–C(14)	2.125(6)	2.137(6)
Co–C(15)	2.104(7)	2.090(6)
C(1)–C(2)	1.338(9)	1.333(10)
C(2)–C(3)	1.447(10)	1.442(10)
C(3)–C(4)	1.351(10)	1.341(9)
C(11)–C(12)	1.386(8)	1.435(8)
C(12)–C(13)	1.412(8)	1.404(9)
C(13)–C(14)	1.410(8)	1.424(8)
C(14)–C(15)	1.411(9)	1.413(9)
C(11)–C(15)	1.424(9)	1.414(8)
P–Co–C(1)	90.6(2)	89.2(2)
P–Co–C(4)	91.2(2)	89.5(2)
C(1)–Co–C(4)	83.2(3)	82.6(3)

[a] Numbers in parentheses are estimated standard deviations.

comparable to 1.32 ± 0.05 and 1.44 Å for cyclopentadiene [15]. Thus the C_4 moieties of cobaltacyclopentadienes are considered as a butadiene fragment and planar with deviation from the best planes of 0.001, −0.002, 0.003, and −0.001 Å for **1a**, and of 0.001, −0.004, 0.003, and −0.001 Å for **1b**. The cobalt atoms are displaced by 0.104 Å for **1a** and 0.147 Å for **1b** from the planes in the direction of the cyclopentadienyl ring, respectively. Similar displacement (0.203 Å) was observed in **1d**.

Experimental

Equipment. Melting points were determined by using a Mitamura capillary melting point apparatus and were uncorrected. The ^1H NMR spectra were recorded on a Varian HA 100 spectrometer using tetramethylsilane as an internal standard. IR spectra were obtained by using a Shimadzu IR-27G spectrometer.

Reagents. For column chromatography, Sumitomo activatated alumina KCG-30 was used and deactivated by water depending on the purpose.

Reaction of (η-methoxycarbonylcyclopentadienyl)bis(triphenylphosphine)cobalt with acetylene

(1) According to the method described by Rausch [10], sodium methoxycarbonylcyclopentadienide was prepared from sodium cyclopentadienide (5 mmol in 5 ml of THF) and dimethyl carbonate (1 ml). The cyclopentadienide was added under nitrogen atmosphere to the flask (100 ml) containing chlorotris(triphenylphosphine)cobalt (3.52 g, 4 mmol) dispersed in benzene (20 ml). The flask was stoppered and shaked vigorously. After 10 min, the resulting dark red solution including (η-methoxycarbonylcyclopentadienyl)bis(triphenyphosphine)cobalt was filtered through a short column of alumina (30 × 30 mm) to exclude excess amounts of the cyclopentadienide. Acetylene was bubbled through the filtrate at room temperature for a few min. After concentration of the resulting dark brown solution, the residue was chromatographed on alumina (deactivated by addition of H_2O 5% by weight, 25 × 150 mm). The first very pale yellow eluate, which included triphenylphosphine, from elution with a 1/1 mixture of hexane/benzene was discarded. The second pale yellow eluate from a 1/2 mixture of hexane/benzene was evaporated to almost dryness. The residue was dissolved in hexane and kept in a refrigerator overnight to give orange brown crystals of **1b** (0.085 g, 4%). The third dark brown eluate from benzene was evaporated to almost dryness and the residue was dissolved in hexane and kept in a refrigerator to give black crystals of **2b** (0.145 g, 11%). From the fourth green eluate, known complex **3b** (0.017 g, 2%) was isolated as dark green crystals.

(2) Similarly, the solution of (η-methoxycarbonylcyclopentadenyl)bis(triphenylphosphine)cobalt in toluene (50 ml) was prepared from sodium methoxycarbonylcyclopentadienide (6 mmol) and chlorotris(triphenylphosphine)cobalt (6.6 g, 7.5 mmol). The solution was cooled by a dry ice-methanol bath and acetylene was bubbled for a few minutes. The bath was removed and the solution was allowed to warm to room temperature under stirring. A similar procedure to that described above afforded **1b** (0.90 g, 24%), **2b** (0.052 g, 2%), and **3b** (0.051 g, 3%).

According to similar procedures as (2) above, unsubstituted cyclopentadienylcobalt and acetylcyclopentadienylcobalt analogs were obtained.

X-ray study

The crystal and refinement data of **1a**, **1b**, and **2b** are summarized in Table 4. X-Ray measurements were carried out with a Rigaku automatic four-circle diffractometer using a graphite monochromater.

Intensities were measured using ω-scans ($2\theta < 30°$) or ω–2θ scans ($30° < 2\theta < 55°$) with 5-*s* stationary background counts at the lower and upper limits of each scan. A constant scan speed of 0.06 ° s^{-1} was used.

Lorentz and polarization corrections were applied. Corrections for absorption were not made for every complex.

The structures were solved using three-dimensional Patterson and Fourier techniques and refined by block-diagonal least squares. The cobalt and phosphorus atoms in **1a** and **1b**, and two cobalt atoms in **2b** were located from each Patterson map. For each complex a subsequent difference-Fourier synthesis revealed the positions of all the non-hydrogen atoms. The computer programs used are listed elsewhere [16]. Anomalous-dispersion effects for Co were included in the calculation using Cromer's values of $\Delta f'$ and $\Delta f''$ [17]. The atomic scattering factors were from International Tables [18]. The hydrogen atoms in each complex should be located from a difference-Fourier synthesis and included in the refinement with isotropic thermal parameters. Tables of observed and calculated structure factors, hydrogen atom coordinates, and thermal parameters are deposited as supplementary materials. The final non-hydrogen atomic coordinates of the compounds **1a**, **1b**, and **2b** are listed in Table 5.

(Continued on p. 262)

TABLE 4

CRYSTAL DATA

	1a	**1b**	**2b**
Formula	$C_{27}H_{24}PCo$	$C_{29}H_{26}O_2PCo$	$C_{34}H_{31}O_4PCo$
M	438.4	496.4	601.4
Crystal size (mm)	0.33×0.20×0.10	0.4×0.10×0.20	0.48×0.27×0.15
Space group	*Pa*	$P2_1/a$	$P2_1/c$
Crystal system	monoclinic	monoclinic	monoclinic
a (Å)	8.529(3)	8.327(2)	10.681(2)
b (Å)	16.010(6)	36.468(7)	30.722(7)
c (Å)	8.028(4)	8.021(1)	8.912(1)
β (°)	100.31(3)	98.75(2)	93.55(1)
U (Å^3)	1078.5	2407.5	2918.8
Z	2	4	4
D_c (g cm^{-3})	1.350	1.370	1.369
F(000)	456	1032	1236
Radiation (λ (Å))	Mo-K_α (0.7107)	Mo-K_α	Mo-K_α
μ (cm^{-1})	8.7	8.0	12.2
No. of unique reflections	2242	2810	4107
$[(\sin\theta)/\lambda]_{max}$	0.65	0.65	0.65
R	0.034	0.047	0.050
R'	0.037	0.057	0.053
Maximum residual electron density (Å^{-3})	0.41	0.42	0.54

TABLE 5

ATOMIC COORDINATES FOR **1a**, **1b**, AND **2b** [a,b]

Atom	*x*	*y*	*z*
Co	2500(0)	1569(0)	5000(0)
P	1365(1)	2526(1)	3340(2)
C(1)	3258(7)	1033(3)	3123(7)
C(2)	4739(8)	1253(5)	2977(8)
C(3)	5462(7)	1876(5)	4179(9)
C(4)	4535(7)	2150(4)	5265(7)
C(11)	2881(8)	551(4)	6642(8)
C(12)	2954(7)	1269(4)	7617(7)
C(13)	1473(7)	1685(4)	7229(7)
C(14)	473(7)	1197(4)	6017(7)
C(15)	1331(9)	489(4)	5635(8)
C(21)	−155(5)	2169(3)	1575(6)
C(22)	−798(6)	2713(4)	268(7)
C(23)	−1909(6)	2418(4)	−1087(7)
C(24)	−2402(6)	1585(4)	−1151(7)
C(25)	−1777(7)	1043(4)	138(8)
C(26)	−649(6)	1332(4)	1504(7)
C(31)	2576(5)	3210(3)	2259(6)
C(32)	2934(7)	4039(4)	2717(8)
C(33)	3962(9)	4501(4)	1912(10)
C(34)	4632(8)	4153(4)	634(8)
C(35)	4262(8)	3339(4)	140(7)
C(36)	3252(7)	2871(3)	927(6)
C(41)	303(6)	3267(3)	4501(6)
C(42)	−1322(6)	3438(4)	4049(7)
C(43)	−2055(7)	3974(4)	5062(9)
C(44)	−1175(8)	4350(4)	6481(9)
C(45)	447(8)	4177(4)	6953(8)
C(46)	1173(7)	3639(3)	5965(7)
Co	3612(1)	4032(0)	3863(1)
P	4689(2)	3596(0)	5456(2)
O(1)	2253(6)	5047(1)	3704(6)
O(2)	561(5)	4689(1)	1959(6)
C(1)	1597(7)	3762(2)	3504(8)
C(2)	555(8)	3858(2)	4534(9)
C(3)	1164(8)	4125(2)	5800(9)
C(4)	2697(8)	4231(2)	5745(8)
C(5)	2020(8)	4777(2)	2813(8)
C(6)	−802(8)	4911(2)	2291(10)
C(11)	3275(7)	4514(2)	2527(7)
C(12)	4899(7)	4515(2)	3434(8)
C(13)	5710(7)	4213(2)	2859(8)
C(14)	4642(7)	4027(2)	1579(7)
C(15)	3151(7)	4219(2)	1372(7)
C(21)	3385(6)	3301(2)	6538(7)
C(22)	2779(7)	3440(2)	7930(8)
C(23)	1717(8)	3240(2)	8742(8)
C(24)	1280(9)	2891(2)	8182(9)
C(25)	1983(9)	2743(2)	6839(10)
C(26)	2955(8)	2949(2)	6025(8)
C(31)	6244(6)	3728(2)	7231(7)
C(32)	6792(7)	4092(2)	7362(7)

TABLE 5 (continued)

Atom	x	y	z
C(33)	7895(8)	4201(2)	8735(8)
C(34)	8472(7)	3953(2)	9994(8)
C(35)	7938(7)	3593(2)	9873(8)
C(36)	6824(7)	3481(2)	8500(8)
C(41)	5722(7)	3269(1)	4233(7)
C(42)	4838(7)	3128(2)	2746(8)
C(43)	5535(8)	2885(2)	1746(8)
C(44)	7129(9)	2777(2)	2204(9)
C(45)	8032(8)	2913(2)	3657(9)
C(46)	7331(7)	3157(2)	4675(8)
Co(1)	1901(1)	985(0)	−1552(1)
Co(2)	1019(1)	1718(0)	−2186(1)
P	−447(1)	1329(0)	−1454(1)
O(11)	3997(4)	217(2)	923(5)
O(12)	5108(4)	802(2)	286(5)
O(21)	4312(4)	2183(2)	−3090(5)
O(22)	3621(4)	1779(2)	−5082(5)
C(1)	1964(5)	1533(2)	−398(5)
C(2)	3133(5)	1768(2)	195(7)
C(11)	2373(5)	337(2)	−1861(7)
C(12)	1892(6)	510(2)	−3238(7)
C(13)	2649(5)	871(2)	−3591(6)
C(14)	3628(5)	916(2)	−2453(6)
C(15)	3465(5)	583(2)	−1361(6)
C(16)	4186(5)	504(2)	47(7)
C(17)	5863(8)	752(3)	1646(9)
C(21)	1076(6)	1908(2)	−4463(6)
C(22)	−14(6)	2072(2)	−3840(8)
C(23)	349(6)	2348(2)	−2613(8)
C(24)	1670(6)	2357(2)	−2469(7)
C(25)	2123(5)	2087(2)	−3604(6)
C(26)	3453(6)	2028(2)	−3860(7)
C(27)	4895(8)	1710(3)	−5436(10)
C(31)	396(4)	979(2)	−165(5)
C(32)	1486(5)	1198(2)	518(5)
C(33)	2028(5)	1028(2)	1904(6)
C(34)	1593(6)	651(2)	2502(6)
C(35)	561(6)	426(2)	1776(6)
C(36)	−29(5)	580(2)	491(6)
C(41)	−1421(5)	994(2)	−2761(6)
C(42)	−1082(5)	939(2)	−4236(2)
C(43)	−1775(6)	669(2)	−5216(7)
C(44)	−2810(6)	453(2)	−4736(8)
C(45)	−3163(5)	508(2)	−3280(7)
C(46)	−2479(5)	782(2)	−2300(6)
C(51)	−1607(5)	1616(2)	−385(6)
C(52)	−2617(6)	1830(2)	−1125(7)
C(53)	−3452(6)	2071(2)	−315(8)
C(54)	−3275(6)	2099(2)	1224(8)
C(55)	−2288(6)	1891(2)	1963(8)
C(56)	−1447(5)	1650(2)	1157(7)

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[a] Positional parameters are multiplied by 10^4. [b] Estimated standard deviations in parentheses.

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