SYNTHESIS, CHARACTERIZATION AND CRYSTAL STRUCTURE OF DICARBONYL(*syn*-η³-2,4-PENTADIENYL)TRIPHENYLPHOSPHINECOBALT

GENE-HSIANG LEE, SHIE-MING PENG*,

Department of Chemistry, National Taiwan University, Taipei (Taiwan)

MING-YUAN LIAO and RAI-SHUNG LIU*

Department of Chemistry, National Tsinghua University, Hsinchu (Taiwan)

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Summary

The reaction of $NaCo(CO)_4$ with *trans*-1-bromopenta-2,4-diene in tetrahydrofuran at $-78°C$ affords (syn-η³-2,4-pentadienyl)Co(CO)₃ (1) in moderate yield. Addition of triphenylphosphine to **1** in ether at $0°C$ gives (syn-η³-2,4-pentadienyl)Co(CO)₂[P(C₆H₅)₃] (**2a**). In solution, **2a** equilibrates with its *anti* isomer (**2b**) at room temperature. The crystal structure of **2a** has been determined by X-ray diffraction analysis. Compound **2a** crystallizes in the triclinic space group $P\bar{1}$ with lattice parameters: a 10.326(2), b 10.763(4), c 11.452(2) Å, α 105.99(2), β 100.10(1), γ 108.05(2)°, V 1114.98 Å³, Mol. wt. 539.69, D (calcd.) 1.324 g/cm³ and $Z = 2$. The structure was solved and refined to R and R_w values of 0.0357 and 0.0408, respectively for 4046 reflections.

Introduction

There is a growing interest in the chemistry of transition-metal acyclic pentadienyl complexes [1]. Pentadienyl alkaline-metal salts C_5H_7M (M = Na, K) [2] have served as a major source for the synthesis of these complexes. The allyl halide is a convenient reagent for the preparation of a variety of metal allyls because it undergoes oxidative addition or nucleophilic substitution with many metal complexes [3]. In principle the 1-halopenta-2,4-diene should be a convenient material for synthesis of novel classes of transition-metal pentadienyl complexes; nevertheless, examples are rare [4].

Acyclic pentadienylcobalt complexes have been previously obtained from the reaction of cobalt(II) chloride with potassium 2,4-dimethylpentadienide [5,6]. In this paper we will show that with the use of *trans*-1-bromopenta-2,4-diene, two novel pentadienylcobalt complexes (syn-η³-2,4-pentadienyl)Co(CO)₃ (**1**) and (syn-η³-2,4-

pentadienyl)Co(CO)$_2$[P(C$_6$H$_5$)$_3$] (**2a**) can be prepared. The structure of **2a** has been determined by X-ray diffraction studies. In solution, **2a** equilibrates with its *anti* isomer (**2b**) at room temperature.

Results and discussion

When a tetrahydrofuran solution of *trans*-1-bromopenta-2,4-diene was stirred with excess NaCo(CO)$_4$ at $-78°$C for 6 h, followed by repeated purification using vacuum distillation, a dark red oil of (*syn*-η^3-2,4-pentadienyl)Co(CO)$_3$ (**1**) was obtained in moderate yield (52%). Excess NaCo(CO)$_4$ was required for the preparation, otherwise a coupling reaction of **1** with *trans*-1-bromopenta-2,4-diene would have occurred to give 1,3,7,9-decatetraene. The formula of **1** was confirmed by its mass spectrum, which showed the highest mass ion of 210, and the fragment ions after successive loss of CO. The pentadienyl ligand is linked to the cobalt center in a *syn*-η^3 configuration as shown by its ^{1}H NMR resonances (400 MHz, C$_6$D$_6$): δ 1.90 (d, H$_a$), 2.49 (d, H$_b$), 3.29 (t, H$_d$), 4.38 (td, H$_c$), 4.97 (dd, H$_f$), 5.09 (dd, H$_g$), 5.70 (dt, H$_e$). The assignment of *syn*-configuration to **1** was based on the coupling constants $J_{ac} = J_{cd} = 10.2$ Hz, $J_{bc} = 6.2$ Hz, which indicated the arrangement of two *anti*-protons H$_a$ and H$_d$, and a *syn*-positioned H$_b$. Moreover, the IR spectrum in pentane shows a weak ν(C=C) band at 1619 cm^{-1}, and the bands of ν(C=O) at 2062vs, 1999sh, 1996vs cm^{-1}. These spectroscopic data suggest the structure of **1** as represented:
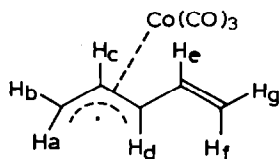
(**1**)

Stirring a tetrahydrofuran solution of **1** with excess triphenylphosphine in a 1/1 molar ratio at 0°C for 6 h gave a red precipitate of (*syn*-η^3-pentadienyl)Co(CO)$_2$[P(C$_6$H$_5$)$_3$] (**2a**). Further crystallization from a saturated tetrahydrofuran solution at $-25°$C afforded a good yield (65%) of dark red crystals. C and H elemental analysis confirmed the formula. The molecular structure of **2a** has been further characterized by X-ray diffraction studies. The unit-cell contains two discrete (*syn*-η^3-C$_5$H$_7$)Co(CO)$_2$(PPh$_3$) molecules. The molecular structure of the complex with atomic labelling and pertinent bond parameters is depicted in Fig. 1. The positional and thermal parameters of all the atoms are given in Table 1. The bond lengths and angles are presented in Table 2.

The coordination geometry about the cobalt atom approximates a trigonal bipyramid with C(4), C(5) and C(6) atoms occupying the three equatorial positions and P, C(8) atoms residing in the axial positions *. All the L(axial)–Co–L(equa-

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* The allyl group is treated as a bidentate ligand. The geometry of this complex could also be described as a tetrahedron with C(4), C(5), P(atom) and the centroid of the allylic C(a) [(C(6) + C(7) + C(8))/3] occupying the four coordination sites. The L–Co–L angles are 94.9, 98.0, 124.6, 115.3, 115.2, 108.3° for P–Co–C(4), P–Co–C(5), P–Co–C(a), C$_4$–Co–C(5), C(4)–Co–C(a), C(5)–Co–C(a), respectively.
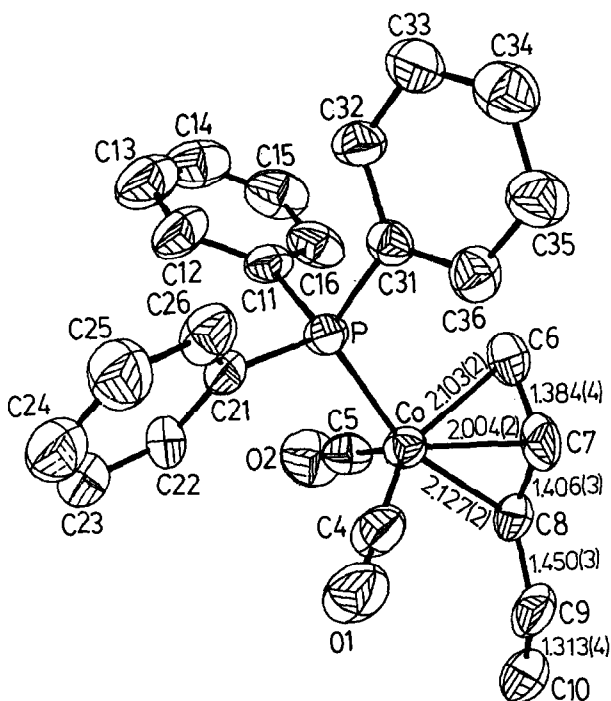
Fig. 1. ORTEP drawing for (syn-η^3-pentadienyl)Co(CO)$_2$P(C$_6$H$_5$)$_3$. Atoms are represented by a thermal vibration sphere drawn to encompass 50% of electron density.

torial) angles are between 92.42(7) and 98.04(8)°. The exception is the C(6)–Co–C(8) (allyl) angle which is 68.7(1)° due to the small bite angle of the allyl group. The structure of **2a** is similar to that of (η^3-allyl)Co(CO)$_2$(PPh$_3$) [7] but differs from that of ($anti$-η^3-2,4-dimethylpentadienyl)Co(PMe$_3$)$_3$ [6] which is described as a square pyramid. The reason is attributed to the three bulky trimethylphosphine ligands.

The most interesting features of the structure are the configuration and geometry of the pentadienyl ligand. The complex has a W-shaped η^3-pentadienyl group; i.e. a syn-configuration. The pentadienyl moiety is not planar, the dihedral angle between the vinylic plane (C(8), C(9), C(10)) and the allylic plane (C(6), C(7), C(8)) is 19.8°. Carbon atoms C(6), C(7), C(8) and C(9) of the pentadienyl ligand, however do form a least-squares plane with a deviation of about 0.04 Å, while the C(10) atom, which lies out of the plane, is 0.24 Å away from the cobalt atom. Similar observations of η^3-pentadienyl ligands have been found in syn, syn-bis(η^3-pentadienyl)bis(trimethylphosphine)iron [8] (twist angle 16.7°, the terminal vinylic atom lies 0.26 Å out of the plane), (syn-η^3-pentadienyl)Mn(dmpe)$_2$ [9] (twist angle 22°, the terminal vinylic atom lies 0.35 Å out of the plane), (syn-η^3-pentadienyl)Mo(CO)$_2$Cp [10] (twist angle 21.4°, the terminal vinylic atom lies 0.29 Å out of the plane). The C–C bond distances within the allylic moiety are 1.384(4) Å for C(6)–C(7), 1.406(3) Å for C(7)–C(8). The lengths of the C(8)–C(9) bond and the C(9)–C(10) bond are 1.450(3) and 1.313(4) Å, respectively. The pentadienyl ligand has a staggered orientation with respect to the P–C(4)–C(5) faces; i.e. the mouth of the allylic

TABLE 1

POSITIONAL PARAMETERS WITH ESTIMATED STANDARD DEVIATION FOR NON-HY-DROGEN ATOMS IN $(syn\text{-}\eta^3\text{-}2,4\text{-PENTADIENYL})Co(CO)_2P(C_6H_5)_3$

Atom	x	y	z	B_{iso}
Co	0.27948(3)	0.18380(3)	0.41619(3)	3.20(1)
P	0.36354(6)	0.10720(6)	0.26093(5)	3.01(3)
C(11)	0.2860(2)	−0.0817(2)	0.1752(2)	3.5(1)
C(12)	0.2524(3)	−0.1381(3)	0.0455(3)	5.1(2)
C(13)	0.1994(4)	−0.2816(4)	−0.0147(3)	6.6(2)
C(14)	0.1810(4)	−0.3687(3)	0.0520(4)	6.9(3)
C(15)	0.2133(3)	−0.3147(3)	0.1810(4)	6.7(2)
C(16)	0.2646(3)	−0.1707(3)	0.2436(3)	5.0(2)
C(21)	0.3352(3)	0.1775(2)	0.1350(2)	3.6(1)
C(22)	0.1984(3)	0.1673(3)	0.0833(2)	4.9(2)
C(23)	0.1717(3)	0.2224(4)	−0.0095(3)	6.4(2)
C(24)	0.2807(4)	0.2891(4)	−0.0506(3)	7.3(2)
C(25)	0.4162(4)	0.3006(4)	−0.0008(3)	7.1(2)
C(26)	0.4439(3)	0.2446(3)	0.0927(2)	5.0(1)
C(31)	0.5543(2)	0.1439(2)	0.2999(2)	3.3(1)
C(32)	0.6109(3)	0.0553(3)	0.2333(2)	4.0(1)
C(33)	0.7557(3)	0.0883(3)	0.2668(3)	5.2(2)
C(34)	0.8455(3)	0.2091(3)	0.3651(3)	5.3(2)
C(35)	0.7900(3)	0.2977(3)	0.4310(3)	4.6(1)
C(36)	0.6447(3)	0.2647(3)	0.3988(2)	3.9(1)
C(4)	0.2819(3)	0.3346(3)	0.3830(2)	4.6(1)
C(5)	0.1114(3)	0.0458(3)	0.3621(2)	4.2(1)
C(6)	0.4025(3)	0.1235(3)	0.5397(3)	5.5(2)
C(7)	0.4002(3)	0.2540(3)	0.5958(2)	4.4(1)
C(8)	0.2685(3)	0.2614(3)	0.6047(2)	4.2(1)
C(9)	0.2458(3)	0.3908(3)	0.6474(2)	5.2(2)
C(10)	0.1340(4)	0.4028(4)	0.6813(3)	7.2(2)
O(1)	0.2782(3)	0.4305(2)	0.3600(2)	7.8(2)
O(2)	0.0026(2)	−0.0426(2)	0.3241(2)	6.3(1)
H(61)	0.492(2)	0.120(2)	0.524(2)	4.8(6)
H(62)	0.336(3)	0.044(3)	0.546(2)	5.9(6)
H(7)	0.480(3)	0.334(3)	0.619(2)	5.5(6)
H(8)	0.203(2)	0.188(2)	0.613(2)	3.4(5)
H(9)	0.323(3)	0.464(3)	0.651(3)	6.7(7)
H(101)	0.128(3)	0.492(3)	0.704(3)	7.0(7)
H(102)	0.059(3)	0.325(3)	0.681(3)	8.5(9)

moiety faces the axial PPh_3 ligand and the vinylic moiety lies further down the axial position which minimizes repulsion of the ligands.

Compound 2a undergoes facile isomerism with its *anti* isomer (2b) via $\pi\text{-}\sigma\text{-}\pi$ mechanism at room temperature. When the crystals of 2a were dissolved in toluene-d_8 at −20°C, in the initial 20 min, the ^1H NMR spectrum showed only the resonances of the *syn* isomer 2a. The *syn*-pentadienyl group of 2a was characterized by the observed coupling constants $J_{ac} = J_{cd} = 10.0$ Hz and $J_{bc} = 6.2$ Hz. When the NMR solution was allowed to stand, the resonances of 2b appeared and slowly grew in intensity until a final 5/1 ratio of 2a/2b was observed after 3 h. The ^1H NMR spectrum of the *anti* isomer 2b has a pattern closely analogous to that of its *syn*

TABLE 2

BOND LENGTHS (Å) AND BOND ANGLES (°) WITH ESTIMATED STANDARD DEVIATIONS FOR (syn-η^3-2,4-PENTADIENYL)Co(CO)$_2$P(C$_6$H$_5$)$_3$

Co–P	2.1843(7)	Co–C(4)	1.760(2)	Co–C(5)	1.766(2)
Co–C(6)	2.103(2)	Co–C(7)	2.004(2)	Co–C(8)	2.127(2)
P–C(11)	1.831(2)	P–C(21)	1.824(2)	P–C(31)	1.830(2)
C(11)–C(12)	1.373(3)	C(11)–C(16)	1.387(3)	C(12)–C(13)	1.384(4)
C(13)–C(14)	1.354(5)	C(14)–C(15)	1.366(5)	C(15)–C(16)	1.393(4)
C(21)–C(22)	1.390(3)	C(21)–C(26)	1.378(3)	C(22)–C(23)	1.380(3)
C(23)–C(24)	1.369(5)	C(24)–C(25)	1.368(4)	C(25)–C(26)	1.396(3)
C(31)–C(32)	1.391(3)	C(31)–C(36)	1.381(3)	C(32)–C(33)	1.380(3)
C(33)–C(34)	1.377(4)	C(34)–C(35)	1.379(4)	C(35)–C(36)	1.385(3)
C(4)–O(1)	1.143(3)	C(5)–O(2)	1.138(3)	C(6)–C(7)	1.384(4)
C(6)–H(61)	0.979(3)	C(6)–H(62)	0.946(3)	C(7)–C(8)	1.406(3)
C(7)–H(7)	0.920(3)	C(8)–C(9)	1.450(3)	C(8)–H(8)	0.908(3)
C(9)–C(10)	1.313(4)	C(9)–H(9)	0.917(3)	C(10)–H(101)	0.950(3)
C(10)–H(102)	0.946(4)				

P–Co–C(4)	94.43(8)	P–Co–C(5)	98.04(8)
P–Co–C(6)	92.42(7)	P–Co–C(7)	119.72(8)
P–Co–C(8)	159.24(7)	C(4)–Co–C(5)	115.3(1)
C(4)–Co–C(6)	139.5(1)	C(4)–Co–C(7)	105.2(1)
C(4)–Co–C(8)	94.8(1)	C(5)–Co–C(6)	103.1(1)
C(5)–Co–C(7)	121.4(1)	C(5)–Co–C(8)	94.8(1)
C(6)–Co–C(7)	39.3(1)	C(6)–Co–C(8)	68.7(1)
C(7)–Co–C(8)	39.7(1)	Co–P–C(11)	115.93(8)
Co–P–C(21)	113.35(7)	Co–P–C(31)	117.23(7)
C(11)–P–C(21)	102.8(1)	C(11)–P–C(31)	102.4(1)
C(21)–P–C(31)	103.2(1)	P–C(11)–C(12)	122.1(2)
P–C(11)–C(16)	119.0(1)	C(12)–C(11)–C(16)	118.9(2)
C(11)–C(12)–C(13)	120.0(3)	C(12)–C(13)–C(14)	121.3(3)
C(13)–C(14)–C(15)	119.6(2)	C(14)–C(15)–C(16)	120.2(3)
C(11)–C(16)–C(15)	120.0(2)	P–C(21)–C(22)	118.2(1)
P–C(21)–C(26)	122.9(1)	C(22)–C(21)–C(26)	118.9(2)
C(21)–C(22)–C(23)	120.6(2)	C(22)–C(23)–C(24)	120.0(2)
C(23)–C(24)–C(25)	120.3(2)	C(24)–C(25)–C(26)	120.0(2)
C(21)–C(26)–C(25)	120.2(2)	P–C(31)–C(32)	122.4(1)
P–C(31)–C(36)	118.4(1)	C(32)–C(31)–C(36)	119.2(2)
C(31)–C(32)–C(33)	119.9(2)	C(32)–C(33)–C(34)	120.7(2)
C(33)–C(34)–C(35)	119.6(2)	C(34)–C(35)–C(36)	120.0(2)
C(31)–C(36)–C(35)	120.5(2)	Co–C(4)–O(1)	177.5(2)
Co–C(5)–O(2)	178.1(2)	Co–C(6)–C(7)	66.5(1)
Co–C(6)–H(61)	116.7(2)	Co–C(6)–H(62)	104.0(2)
C(7)–C(6)–H(61)	117.2(3)	C(7)–C(6)–H(62)	119.7(3)
H(61)–C(6)–H(62)	119.2(3)	Co–C(7)–C(6)	74.2(1)
Co–C(7)–C(8)	74.9(1)	Co–C(7)–H(7)	115.5(2)
C(6)–C(7)–C(8)	117.6(2)	C(6)–C(7)–H(7)	121.3(3)
C(8)–C(7)–H(7)	120.8(3)	Co–C(8)–C(7)	65.5(1)
Co–C(8)–C(9)	119.4(1)	Co–C(8)–H(8)	104.5(2)
C(7)–C(8)–C(9)	124.1(2)	C(7)–C(8)–H(8)	118.7(3)
C(9)–C(8)–H(8)	113.4(2)	C(8)–C(9)–C(10)	125.2(2)
C(8)–C(9)–H(9)	109.8(3)	C(10)–C(9)–H(9)	125.0(3)
C(9)–C(10)–H(101)	118.1(3)	C(9)–C(10)–H(102)	121.5(3)

isomer excepting the variations in the coupling constants $J_{bc} \cong J_{cd} \cong 6$ and $J_{ac} = 11$ Hz which are characteristic of an *anti*-η^3-pentadienyl group.

(2a)   (2b)
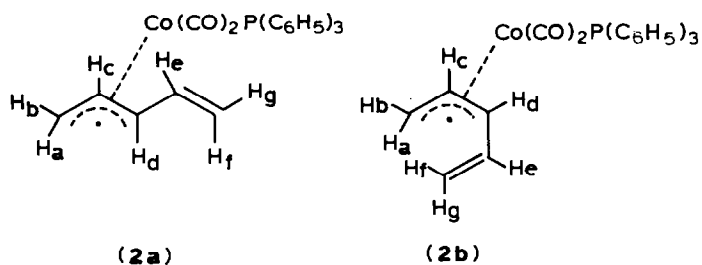
Bleeke and coworkers [6] have recently described the synthesis and molecular structure of (*anti*-η^3-2,4-dimethylpentadienyl)Co(PMe$_3$)$_3$. Comparing this structure with that of **2a**, pronounced variations in the pentadienyl configuration are evident. The U-shaped *anti* isomer forms the solid state structure and is also the major species in solution. These results reflect the configurational flexibility of the cobalt-bound η^3-pentadienyl ligand, which can be selectively adopted as a *syn* or *anti* ligand by modification of the metal coordination sphere. Although, the origins of the structural flexibility of the η^3-pentadienyl ligand remain unclear, such unusual features are certainly interesting in fundamental aspects.

Experimental

All reactions were carried out under dry nitrogen using Schlenk-tube techniques. *trans*-1-Bromopenta-2,4-diene was synthesized by treatment of penta-1,4-diene-3-ol with the acid HBr at 0°C [11]. NaCo(CO)$_4$ was prepared by stirring a 50 ml tetrahydrofuran solution of Co$_2$(CO)$_8$ (1.5 g, 4.4 mmol) with 40 g of sodium amalgam (2.4% sodium content) for 16 h. NMR spectra were recorded on either a JEOL FX-100 or a Bruker AM-400 spectrometer. Microanalyses were performed by the microanalytic laboratory at the National Taiwan University, Taipei.

Preparation of (syn-η^3-C$_5$H$_7$)Co(CO)$_3$ (1)

A tetrahydrofuran (10 ml) solution containing *trans*-1-bromopenta-2,4-diene (0.93 g, 6.3 mmol) was added dropwise to a rapidly stirring solution of NaCo(CO)$_4$ (1.71 g, 8.8 mmol) in 50 ml tetrahydrofuran solution at $-78°$C, and stirred for 6 h. After the insoluble solids were filtered out at $-78°$C, the solvent was evaporated under reduced pressure at 0°C to leave a red residue. Repeated purification of the residue by vacuum distillation (5×10^{-3} Torr) at 23°C into a 0°C cold trap several times, afforded the red oil of **1** (0.70 g, 3.3 mmol). Mass spectrum, m/e: 210 (M^+), 182 (M^+-CO), 154($M^+ - 2$CO), 126 ($M^+ - 3$CO), 67 ($M^+ - C_5H_7 - 3$CO). IR spectrum (in pentane) ν(CO): 2062vs, 1999sh, 1996vs cm^{-1}; ν(C=C) 1619 cm^{-1}. ^{1}H NMR spectrum (400 MHz, benzene-d_6): 1.70 (d, 1H, H$_a$), 2.49 (d, 1H, H$_b$), 3.29 (t, 1H, H$_d$), 4.38 (td, 1H, H$_c$), 4.97 (dd, 1H, H$_g$), 5.09 (dd, 1H, H$_f$), 5.70 (dt, 1H, H$_e$), J_{ac} 10.2, J_{bc} 6.2, J_{cd} 10.2, J_{de} 10.2, J_{eg} 10.2, J_{ef} 17.0, J_{fg} 1.0 Hz.

Synthesis of (syn-η^3-C$_5$H$_7$)Co(CO)$_3$P(C$_6$H$_5$)$_3$

P(C$_6$H$_5$)$_3$ (0.4 g, 1.53 mmol) was stirred with (*syn*-η^3-C$_5$H$_7$)Co(CO)$_3$ (0.3 g, 1.43 mmol) in 20 ml tetrahydrofuran at 0°C for 6 h. The solvent was removed under

reduced pressure to leave a red precipitate which was further crystallized in a saturated tetrahydrofuran solution at $-25°C$ to give red crystals of (syn-η^3-C_5H_7)Co(CO)$_2$P(C$_6$H$_5$)$_3$ (0.36 g, 0.86 mmol). Anal. Found: C, 67.21; H, 5.00. C$_{25}$H$_{22}$CoO$_2$ calcd.: C, 67.57; H, 4.99%. IR spectrum (in pentane) ν(CO): 1996vs and 1949vs; ν(C=C) 1610w cm^{-1}. ^{1}H NMR (400 MHz, C$_6$D$_6$): syn isomer 1.90 (dd, 1H, H$_a$), 2.23 (dd, 1H, H$_b$), 3.42 (t, 1H, H$_d$), 4.59 (td, 1H, H$_c$), 5.03 (dd, 1H, H$_g$), 5.28 (dd, 1H, H$_f$), 6.05 (dt, 1H, H$_e$), J(P–H$_a$) 1.2, J(P–H$_b$) 9.0, J_{ac} 10.0, J_{bc} 6.2, J_{cd} 10.0, J_{de} 10.0, J_{ef} 16.9, J_{eg} 10.0, J_{fg} 1.0 Hz; $anti$ isomer 2.71 (d, 1H, H$_a$), 2.57 (dd, 1H, H$_b$), 4.29 (dd, 1H, H$_d$), 4.40 (ddd, 1H, H$_c$), 4.85 (dd, 1H, H$_g$), 5.01 (dd, 1H, H$_f$), 5.43 (dt, 1H, H$_e$), J(P–H$_b$) 5.5, J_{ac} 11.0, J_{bc} 5.8, J_{cd} 6.0, J_{de} 10.0, J_{ef} 16.8, J_{eg} 10.0, J_{fg} 1.0 Hz.

Crystal data for (syn-η^3-2,4-pentadienyl)Co(CO)$_2$P(C$_6$H$_5$)$_3$

C$_{25}$H$_{22}$CoO$_2$, mol. wt. 444.36, triclinic, space group $P\bar{1}$, a 10.326(2), b 10.763(4), c 11.452(2) Å, α 105.99(2), β 100.10(1), γ 108.05(2)°, V 114.98 Å^3 $Z = 2$, D_c 1.324 g/cm^3, λ(Mo-K_α) 0.7093, μ(Mo-K_α) 8.54 cm^{-1}, $F(000) = 459.93$.

Collection and reduction of X-ray data

A brick-red block-shaped crystal of dimensions $0.40 \times 0.45 \times 0.53$ mm was coated with paraffin, sealed in a capillary tube, and used for data collection. Diffraction data were collected on a CAD4 diffractometer using graphite-mono-chromated Mo-K_α radiation. Unit cell dimensions were determined from a least-squares refinement of 25 carefully centered reflections ($23.2° < 2\theta < 29.2°$). Intensity data within $2\theta \le 60°$ were collected at various scan speeds of 20/3 to 20/23 °/s by using the $\omega - 2\theta$ scan techniques, with a scan range calculated according to the expression $0.75 + 0.35 \tan\theta$. Stationary background measurements were taken before and after each scan for a period of time equal to a quarter of the scan time. The three intensity monitors were checked every 2 h and were fluctuated randomly within 5% over the entire collection. After correction for background, the intensities were reduced to F and $\sigma(F)$ according to counting statistics, and the weights were assigned as $\omega = 1/\sigma^2(F) + 0.0001F^2$. Of the 6484 total reflections, 4046 having $I > 3\sigma(I)$, were considered observed. Intensity data were corrected for Lorentz-polarization effects and elliptical absorption.

Solution and refinement of the structure

All calculations were carried out on a PDP-11/23 computer, using the NRCC SDP PDP-11 package [12], MULTAN, and the ORTEP (Enraf–Nonius) structure determination package [13]. Atomic scattering factors were obtained from International Tables for X-Ray Crystallography [14]. The structure was solved by a direct method using MULTAN with 455 highest E's, 100 smallest E's and 9506 σ-two phase relationships. All the non-hydrogen atoms were located using the E map. The final least-squares cycle with anisotropic thermal parameters for all non-hydrogen atoms and isotropic thermal parameters for hydrogen atoms, gave R and R_w factors respectively of 3.57 and 4.08%. The positional and isotropic, thermal parameters of all the atoms are given in Table 1. Tables of anisotropic thermal parameters and structure factors can be obtained from authors.

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