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Synthesis of η^3-phosphaallyl-cobalt and -nickel complexes. Crystal structure of [η^3-1-(2,4,6-tri-t-butylphenyl)-1-phosphaallyl]tricarbonylcobalt

François Mercier, Catherine Hugel-Le Goff, Louis Ricard and François Mathey

Laboratoire de Chimie du Phosphore et des Métaux de Transition DCPH, Ecole Polytechnique, 91128 Palaiseau Cedex (France)

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Abstract

The first (η^3-1-phosphaallyl)tricarbonylcobalt complex has been obtained by reaction of a (supermesityl)(vinyl)halophosphine with $Co_2(CO)_8$ at room temperature, and its structure established by X-ray diffraction study. The reaction of (mesityl)(propenyl)phosphine with nickelocene and allyl iodide gives the corresponding CpNi(I)(R_2PH) complex, which, upon treatment with DABCO, gives a complex in which the phosphaallyl group is η^3-ligated to CpNi and η^1-P-ligated to CpNi(I).

Introduction

η^3-Allyl-cobalt and -nickel complexes play some role in homogeneous catalysis. For example, (η^3-C_3H_5)Co[$P(OMe)_3$]$_3$ is a useful catalyst precursor for the homogeneous hydrogenation of arenes at room temperature [1] and (η^3-C_3H_5)NiLn is used for the oligomerization of alkenes [2]. On the other hand, we have recently described a series of new η^3-phosphaallyl complexes derived from iron [3], molybdenum [4] and tungsten [4]. We have also demonstrated the existence of a $\eta^1 \rightleftharpoons \eta^3$-phosphaallyl equilibrium [5] which results in transient creation of vacant sites on the metal. The possible use of η^3-phosphaallyl complexes in homogeneous catalysis is thus attractive. With this in mind we decided to undertake the synthesis of η^3-phosphaallyl-cobalt and -nickel complexes.

Results and discussion

Since Appel had previously demonstrated that complexes such as **1** could be obtained by using the bulky 2,4,6-tri-t-butylphenyl (supermesityl) group as the substituent R [6], we decided first to investigate the reaction of (supermesityl)-(vinyl)halophosphine **2** [7] with cobalt carbonyl. The reaction did, indeed, give the

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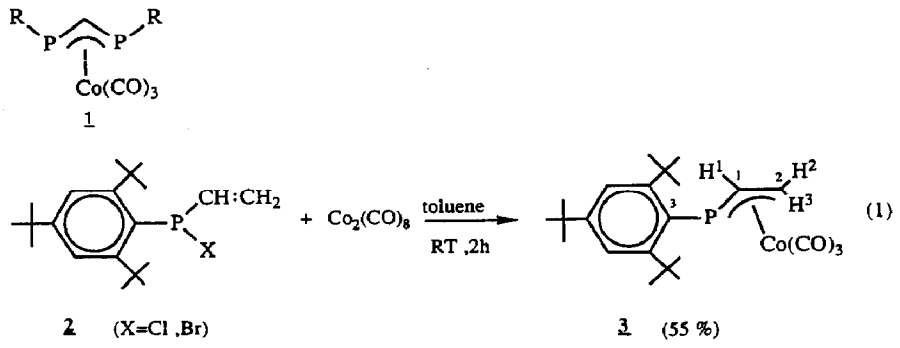
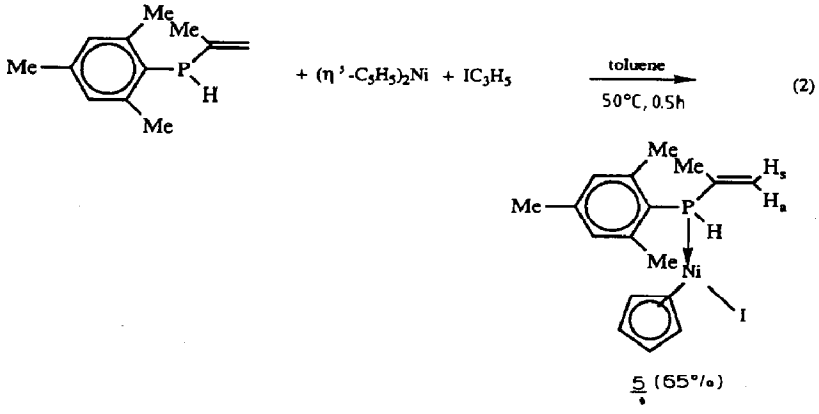
expected (η^3-phosphaallyl) cobalt complex **3** in fair yield (eq. 1).



Interestingly, only one isomer of **3** was detected during all our experiments, in contrast to what was observed with the η^3-phosphaallyl complexes of iron, molybdenum or tungsten [3,4].

The geometry of the η^3-phosphaallylcobalt skeleton, as established by an X-ray crystal structure study of **3** (Fig. 1), is closely similar to that of the η^3-1,3-diphosphaallylcobalt complex described by Appel et al. [6]. There are two significant differences, however. Rather surprisingly on steric grounds, the P–C–P angle observed by Appel et al. (101.8°) is smaller than the P–C–CH_2 angle in our compound (112.1°). On the other hand, the 1-phosphaallyl ligand appears to be more electron-releasing than the 1,3-diphosphaallyl ligand, as expected. Indeed, the degree of back-bonding from the cobalt atom to the carbonyl groups is higher in the first case as indicated by the Co–CO (apical) bond lengths (1.787(6) Å in **3** vs 1.84(1) Å in Appel's complex [6]. Otherwise, the structure of **3** is very similar to the *anti* structure of the previously described molybdenum complex [4], with the ipso carbon of the P-substituent (C3) lying in the PC1C2 plane and outside the PC1C2 angle. On the other hand, the two most noteworthy spectroscopic features of **3** are the very large ^1J(P–C1) coupling constant (63.3 Hz) and the appearance of the ^{31}P resonance at rather low field (+56.5 ppm) for a phosphorus analogue of a π-complex [8].

Encouraged by this first positive result with cobalt, and since Appel and coworkers had also succeeded in preparing (η^3-1,3-diphosphaallyl)nickel complexes



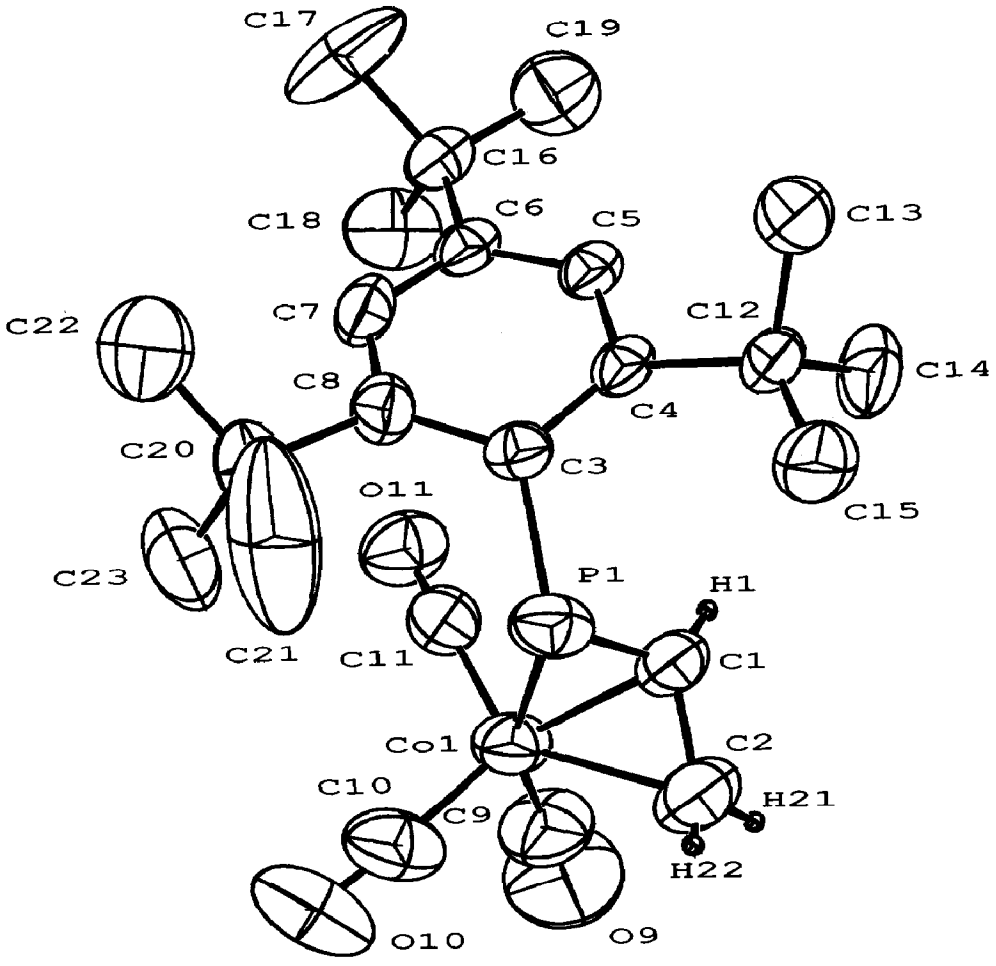
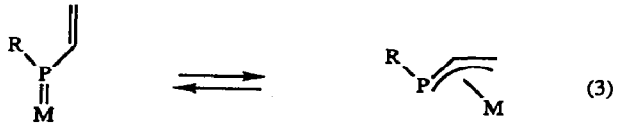


Fig. 1. ORTEP drawing of one molecule of **3**. Vibrational ellipsoids are scale to enclose 50% of the electron density. Hydrogen atoms are omitted except for H1C1.

[9], we then decided to attempt the synthesis of a η^3-1-phosphaallyl complex of nickel. The overall approach was more sophisticated, however. In an initial step we prepared the (cyclopentadienyl)(iodo)nickel complex of a bulky secondary vinylphosphine [10] (eq. 2) using a well-established method of synthesis of tertiary phosphine complexes [11]

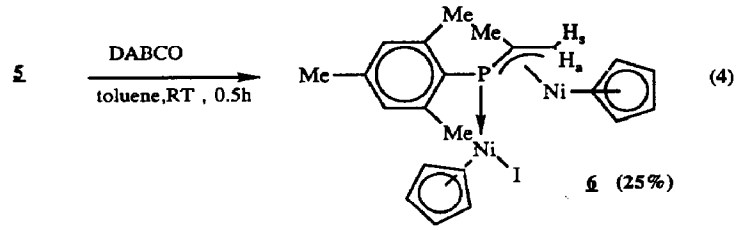
Our idea was then to use the known interconversion between 3-electron η^1- and η^3-phosphaallyl complexes [5] (eq. 3)



(3)

We thus studied the reaction of complex **5** with a series of basic tertiary amines in an attempt to generate a P=Ni double bond [12]. We obtained an (η^3-phosphaallyl)nickel complex (**6**) directly on treatment of **5** with 1,4-diazabi-

cyclo[2.2.2]octane (DABCO) (eq. 4)



(4)

The formula of **6** was unambiguously established by among other methods, mass spectral analysis (molecular peak at 564 for ^{58}Ni) and ^1H NMR spectroscopy (two C_5H_5 signals are present, and the CH_2 protons resonate at high fields). The reaction leading to **6** probably involves the formation of a P = Ni double bond followed by a $\eta^1 \rightarrow \eta^3$ isomerization according to eq. 3, and the displacement of a CpNi(I) complexing group from a CpNi(I)L complex by the resulting η^3-complex. The use of a bulkier substituent at phosphorus would probably inhibit this displacement reaction.

Experimental

All reactions were performed under argon or nitrogen. NMR spectra were recorded on multinuclear WP 80 SY and AC 200 SY Bruker spectrometers operating at 80.13 and 200.13 (^1H), 20.15 and 50.32 (^{13}C), and 32.44 (^{31}P) MHz; chemical shifts are in ppm downfield from internal TMS (^1H or ^{13}C) or external 85% H_3PO_4 (^{31}P), and coupling constants are in hertz. Mass spectra were recorded on a Shimadzu GC-MS QP 1000 instrument at 70 eV under electronic impact. Infrared spectra were obtained with a Perkin-Elmer model 297 spectrometer. Elemental analyses were performed by the Service Central de Microanalyse du CNRS, France.

General data

Chromatographic separations were carried out on silica gel columns (70–230 mesh, Merck).

$Co_2(CO)_8$, IC_3H_5, $(\eta^5\text{-}C_5H_5)_2Ni$ and 1,4-diazabicyclo[2.2.2]octane were commercial samples used without further purification.

Syntheses

[η^3-1-(Tri-2,4,6-t-butylphenyl)-1-phosphaallyl]tricarbonylcobalt, 3. A mixture of equimolecular amounts of **2** (1.8 g, 5×10^{-3} mol) and $Co_2(CO)_8$ (1.7 g) in 15 ml of toluene was stirred for 2 hours at room temperature. The solution was filtered, and then evaporated to leave an oil, which was chromatographed on a short column. After elution with a mixture of hexane/CH_2Cl_2 (90/10) and recrystallization in MeOH, we obtained 1.2 g (55% yield) of complex **3** as orange-brown crystals; m.p. 94°C. NMR (C_6D_6): ^1H: δ 1.26 (s, 9H; *p*-t-Bu), 1.61 (s, 18H; *o*-t-Bu), 2.28 (d × d, ^3J(H–H) 12.7 Hz, ^3J(H–P) 28.35 Hz, 1H, CH_2), 3.04 (dxd, ^3J(H–H) 7.32 Hz, ^3J(H–P) 35.90 Hz, 1H, CH_2), 5.20 (m, ^2J(H–P) ≃ 7.0 Hz, 1H, CH), 7.31 (s, 2H; *meta* H); $^{13}C\text{-}\{^1H\}$: δ 31.27 (s, *p*-CH_3); 34.78 (br; *o*-CH_3 and *p*-CCH_3); 39.42 (s, *o*-CCH_3); 50.25 [d, ^2J(C–P) 37.9 Hz, CH_2]; 92.45 [d, ^1J(C–P) 63.3 Hz; CH]; 123.16 [s, *meta* CH); 136 (d, J(C–P) 68 Hz; C_3]; 149.73 (s, *para* C); 156.49 (s, *ortho* C);

Table 1

Positional parameters and their estimated standard deviations

Atom	x	y	z	B (Å^2)
Co1	0.12863(2)	0.44195(5)	0.45746(6)	4.92(2)
Co2	0.62712(2)	0.05579(4)	0.15991(6)	4.39(1)
P1	0.06513(5)	0.49993(8)	0.3071(1)	4.57(3)
P2	0.56376(4)	0.00044(8)	0.2533(1)	4.20(3)
O9	0.1833(2)	0.4058(4)	0.6911(4)	12.7(2)
O10	0.1107(2)	0.2606(3)	0.3731(4)	9.1(1)
O11	0.2117(1)	0.5250(3)	0.3796(3)	6.5(1)
O19	0.6113(2)	0.2367(2)	0.2312(4)	8.7(1)
O20	0.6783(2)	0.0909(4)	−0.0291(4)	10.1(1)
O21	0.7108(1)	−0.0261(3)	0.3138(3)	6.03(9)
C1	0.0794(2)	0.5429(3)	0.4489(4)	4.9(1)
C2	0.0689(2)	0.4818(4)	0.5237(5)	7.2(2)
C3	0.0917(2)	0.5838(3)	0.2229(4)	3.29(9)
C4	0.0889(2)	0.6752(3)	0.2455(3)	3.15(9)
C5	0.1259(2)	0.7291(3)	0.2215(3)	3.37(9)
C6	0.1634(2)	0.6996(3)	0.1689(3)	3.5(1)
C7	0.1582(2)	0.6137(3)	0.1257(4)	4.1(1)
C8	0.1226(2)	0.5557(3)	0.1465(4)	4.0(1)
C9	0.1610(3)	0.4206(4)	0.6005(5)	7.8(2)
C10	0.1168(2)	0.3313(4)	0.4062(5)	6.5(1)
C11	0.1788(2)	0.4929(3)	0.4069(4)	5.1(1)
C12	0.0445(2)	0.7241(3)	0.2806(4)	3.8(1)
C13	0.0247(2)	0.7882(3)	0.1799(4)	5.5(1)
C14	0.0616(2)	0.7795(4)	0.3924(4)	5.9(1)
C15	0.0006(2)	0.6680(4)	0.2946(5)	5.7(1)
C16	0.2064(2)	0.7572(3)	0.1519(4)	4.2(1)
C17	0.2086(2)	0.7630(5)	0.0247(5)	9.3(2)
C18	0.2536(2)	0.7195(5)	0.2223(6)	7.3(2)
C19	0.2022(2)	0.8526(4)	0.1944(5)	6.9(2)
C20	0.1180(2)	0.4660(3)	0.0787(4)	5.1(1)
C21	0.0676(3)	0.4256(5)	0.0652(8)	13.8(2)
C22	0.1278(4)	0.4818(5)	−0.0420(5)	13.1(3)
C23	0.1566(3)	0.3985(4)	0.1374(6)	7.8(2)
C31	0.5778(2)	−0.0451(3)	0.1257(4)	4.4(1)
C32	0.5672(2)	0.0135(4)	0.0300(5)	6.5(1)
C33	0.5906(2)	−0.0809(3)	0.3649(4)	3.24(9)
C34	0.6222(2)	−0.0510(3)	0.4699(4)	3.9(1)
C35	0.6575(2)	−0.1081(3)	0.5260(4)	4.0(1)
C36	0.6628(2)	−0.1941(3)	0.4911(3)	3.36(9)
C37	0.6254(1)	−0.2258(3)	0.4052(3)	3.22(9)
C38	0.5878(2)	−0.1728(3)	0.3433(3)	3.11(9)
C39	0.6161(2)	0.1660(3)	0.2013(5)	5.8(1)
C40	0.6581(2)	0.0764(4)	0.0456(5)	6.4(1)
C41	0.6773(2)	0.0043(3)	0.2554(4)	4.5(1)
C42	0.6185(2)	0.0406(3)	0.5298(4)	5.0(1)
C43	0.6621(3)	0.0986(4)	0.5184(6)	8.3(2)
C44	0.5712(3)	0.0913(4)	0.4847(6)	9.4(2)
C45	0.6210(3)	0.0235(4)	0.6593(5)	9.5(2)
C46	0.7061(2)	−0.2514(3)	0.5488(4)	4.5(1)
C47	0.7082(2)	−0.2557(5)	0.6803(5)	8.4(2)
C48	0.7026(2)	−0.3456(4)	0.5018(5)	7.5(2)

Table 1 (*continued*)

Positional parameters and their estimated standard deviations

Atom	x	y	z	B (Å^2)
C49	0.7536(2)	−0.2106(5)	0.5257(5)	7.9(2)
C50	0.5440(2)	−0.2234(3)	0.2690(3)	3.7(1)
C51	0.4998(2)	−0.1677(3)	0.2096(5)	5.4(1)
C52	0.5609(2)	−0.2831(4)	0.1766(4)	5.9(1)
C53	0.5241(2)	−0.2845(3)	0.3542(4)	5.5(1)

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $\frac{4}{3}[a^2B(1,1)+b^2B(2,2)+c^2B(3,3)+ab(\cos\gamma)B(1,2)+ac(\cos\beta)B(1,3)+bc(\cos\alpha)B(2,3)]$

203.43 (br, CO); ^{31}P-{^{1}H}: δ 56.55 ppm; IR: ν(C=O) 2070 (w), 2050 (m), 2015 (w), 1998 (m) (decalin); mass spectrum (EI, 70 eV): m/z (relative intensity) 446 (M^+, 2); 390 ($M^+ - 2CO$; 25), 362 ($M^+ - 3CO$, 100). Anal. Calcd. for $C_{23}H_{32}CoO_3P$: C, 61.94; H, 7.23; found: C, 62.05; H, 7.45.

(η^5-Cyclopentadienyl)(mesityl-isopropenylphosphine)iodonickel, 5. To a solution of 1.5 g (7.8 mmol) of mesityl-isopropenylphosphine in 20 ml of toluene were added 1.5 g (7.8 mmol) of (η^5-C_5H_5)$_2$Ni and 0.9 ml (7.8 mmol) of IC_3H_5. The mixture was kept for 0.5 h at 50 °C, its colour turning to dark red. After filtration and evaporation, the residual oil was extracted with 10 ml of toluene and 10 ml of hexane and the combined extracts were filtered, then evaporated to give 2.26 g (65%) of the dark red solid **5**. Some of the product crystallized out from toluene at 0 °C. **5**: dark red crystals, m.p. 128 °C; ^{1}H NMR (C_6D_6): δ 1.54 (d, ^3J(H–P) = 10.5 Hz, CH_3), 2.00 (s, p-CH_3), 2.33 (s, o-CH_3), 5.06 (s, Cp), 5.47 (d, ^3J(H–P) = 42.7 Hz, Hs), 6.15 (d, ^3J(H–P) = 20.4 Hz, Ha), 6.58 (d, ^4J(H–P) = 2.2 Hz, m-H), 6.62 (d, ^1J(H–P) = 373.1 Hz, P–H); ^{13}C NMR (C_6D_6): δ 20.94 (s, p-CH_3), 22.23 (d, ^2J(C–P) = 6.3 Hz, CH_3), 22.75 (d, ^3J(C–P) = 8.7 Hz, o-CH_3), 93.87 (s, Cp), 125–141 (m, mesityl), 132.10 (d, ^1J(C–P) = 17.1 Hz, P–C(CH_3)=CH_2), 140.13 (s, P–C(CH_3)=CH_2); ^{31}P NMR (C_6D_6): δ −14.27, ^1J(P–H) = 373.0 Hz; MS (EI, 70 eV, ^{58}Ni): m/z (relative intensity) 442 (M, 2), 192 (M − NiICp, 100). Anal. Found: C, 46.48; H, 5.03. $C_{17}H_{22}INiP$ calc.: C, 46.09; H, 5.00%.

(η^1-,η^3-1-Mesityl-2-methyl-1-phosphaallyl)(iodo)(di-η^5-cyclopentadienyl)dinickel, 6. 1,4-Diazabicyclo[2.2.2]octane (500 mg, 4.5 mmol) was added to a solution of 2 g (4.5 mmol) of **5** in 10 ml of toluene. The solution was stirred for 0.5 h at room temperature, then filtered and evaporated. The residue was rapidly chromatographed with toluene as eluent to give 650 mg (25%) of the dark red oil **6**: ^{1}H NMR (CD_2Cl_2): δ 1.75 (d, ^3J(H–P) = 10.2 Hz, H_a), 2.16 and 2.29 (s, o-CH_3), 2.55 (d, ^3J(H–P) = 10.2 Hz, CH_3), 2.61 (s, p-CH_3), 3.21 (d, ^3J(H–P) = 23.7 Hz, H_s), 5.15 (s, Cp), 5.49 (s, Cp), 6.62 and 6.70 (s, m-H); ^{13}C NMR (C_6D_6): δ 20.95 (s, p-CH_3), 23.54 (d, ^2J(C–P) = 14.3 Hz, CH_3), 24.42 and 24.71 (s, o-CH_3), 47.93 (s, P–C(CH_3)=CH_2), 92.90 (s, Cp), 94.73 (s, Cp), 99.48 (d, ^1J(C–P) = 13.3 Hz, P–C(CH_3)=CH_2), 125–142 (m, mesityl); ^{31}P NMR (C_6D_6): δ 30.6; MS (EI, 70 eV, ^{58}Ni): m/z (relative intensity) 564 (M, 100), 372 (M − CpI, 46%).

X-Ray structure determination for 3

Crystals of **3**, $C_{23}H_{32}CoPO_3$, were obtained by slow evaporation of a methanol solution of the compound. Data were collected at 18 ± 1° on an Enraf Nonius

Table 2

Bond distances in angstroms with esd's

Atom 1	Atom 2	Distance	Atom 1	Atom 2	Distance
Co1	P1	2.396(1)	C7	C8	1.379(6)
Co1	C1	2.037(4)	C8	C20	1.565(5)
Co1	C2	2.114(6)	C12	C13	1.542(5)
Co1	C9	1.767(6)	C12	C14	1.548(6)
Co1	C10	1.787(6)	C12	C15	1.520(6)
Co1	C11	1.792(6)	C16	C17	1.509(6)
Co2	P2	2.400(1)	C16	C18	1.513(6)
Co2	C31	2.036(4)	C16	C19	1.538(7)
Co2	C32	2.116(4)	C20	C21	1.505(8)
Co2	C39	1.780(6)	C20	C22	1.513(8)
Co2	C40	1.760(6)	C20	C23	1.539(7)
Co2	C41	1.782(5)	C31	C32	1.414(6)
P1	C1	1.755(5)	C33	C34	1.434(5)
P1	C3	1.849(4)	C33	C38	1.412(5)
P2	C31	1.761(5)	C34	C35	1.371(5)
P2	C33	1.842(4)	C34	C42	1.565(5)
O9	C9	1.140(6)	C35	C36	1.381(5)
O10	C10	1.138(6)	C36	C37	1.379(5)
O11	C11	1.135(6)	C36	C46	1.527(5)
O19	C39	1.141(6)	C37	C38	1.398(5)
O20	C40	1.151(6)	C38	C50	1.549(5)
O21	C41	1.135(5)	C42	C43	1.521(7)
C1	C2	1.421(7)	C42	C44	1.521(8)
C3	C4	1.413(5)	C42	C45	1.529(7)
C3	C8	1.424(5)	C46	C47	1.533(6)
C4	C5	1.383(5)	C46	C48	1.523(7)
C4	C12	1.560(5)	C46	C49	1.528(7)
C5	C6	1.385(5)	C50	C51	1.536(6)
C6	C7	1.392(5)	C50	C52	1.552(6)
C6	C16	1.523(5)	C50	C53	1.543(6)

CAD4 diffractometer. The crystal structure was solved and refined by use of the Enraf Nonius-supplied SDP package. The compound crystallizes in space group $P2_1/c$, $a = 27.742(2)$ Å, $b = 15.128(1)$ Å, $c = 11.729(1)$ Å, $\beta = 101.5(1)$, $U = 4823(1)$ Å^3; $Z = 8$; $d_{calc} = 1.229$ g/cm^3; Mo-K_α radiation ($\lambda = 0.71073$ Å) graphite monochromator; $\mu = 7.9$ cm^{-1}; $F(000) = 1888$. A total of 6058 unique reflexions were recorded in the range $2° \leqslant 2\theta \leqslant 50°$, and 2213 were considered as unobserved ($F^2 < 3\sigma(F^2)$), leaving 3845 for solution and refinement. The structure was solved by Patterson methods. Except for those in the t-butyl groups, the hydrogen atoms were refined isotropically in the final stages of least-squares; anisotropic temperature factors were used for all other atoms. A non-Poisson weighting scheme was applied, with a p factor of 0.08. The final R factors were $R = 0.039$, $R_1 = 0.066$, G.O.F. = 1.47

Final atom coordinates are listed in Table 1, bond lengths in Table 2, and bond angles in Table 3. Lists of thermal parameters and structure factors are available from the authors.

Table 3

Bond angles in degrees with esd's

Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle
P1	Co1	C1	45.8(1)	C4	C12	C13	106.1(3)
P1	Co1	C2	71.4(2)	C4	C12	C14	110.9(3)
P1	Co1	C9	157.7(2)	C4	C12	C15	117.1(3)
P1	Co1	C10	92.1(2)	C13	C12	C14	108.0(4)
P1	Co1	C11	96.0(1)	C13	C12	C15	105.2(4)
C1	Co1	C2	40.0(2)	C14	C12	C15	109.0(4)
C1	Co1	C9	113.1(2)	C6	C16	C17	110.5(4)
C1	Co1	C10	127.5(2)	C6	C16	C18	109.2(4)
C1	Co1	C11	102.6(2)	C6	C16	C19	112.5(3)
C2	Co1	C9	87.0(3)	C17	C16	C18	111.1(4)
C2	Co1	C10	107.5(3)	C17	C16	C19	106.7(4)
C2	Co1	C11	137.8(2)	C18	C16	C19	106.8(4)
C9	Co1	C10	100.0(3)	C8	C20	C21	112.7(4)
C9	Co1	C11	96.4(3)	C8	C20	C22	109.1(4)
C10	Co1	C11	113.3(2)	C8	C20	C23	111.7(4)
P2	Co2	C31	45.9(1)	C21	C20	C22	107.5(6)
P2	Co2	C32	71.5(1)	C21	C20	C23	109.1(6)
P2	Co2	C39	91.2(2)	C22	C20	C23	106.5(5)
P2	Co2	C40	157.5(2)	P2	C31	C32	112.6(4)
P2	Co2	C41	96.4(1)	P2	C33	C34	119.4(3)
C31	Co2	C32	39.8(2)	P2	C33	C38	122.0(3)
C31	Co2	C39	127.6(2)	C34	C33	C38	117.9(3)
C31	Co2	C40	113.2(2)	C33	C34	C35	117.9(3)
C31	Co2	C41	101.9(2)	C33	C34	C42	125.5(4)
C32	Co2	C39	109.1(2)	C35	C34	C42	116.5(3)
C32	Co2	C40	86.4(2)	C34	C35	C36	124.0(4)
C32	Co2	C41	136.3(2)	C35	C36	C37	115.8(3)
C39	Co2	C40	100.3(2)	C35	C36	C46	121.7(3)
C39	Co2	C41	113.1(2)	C37	C36	C46	122.5(4)
C40	Co2	C41	96.7(2)	C36	C37	C38	123.6(3)
CO1	P1	Cl	56.2(1)	C33	C38	C37	117.5(3)
CO1	P1	C3	109.3(1)	C33	C38	C50	126.7(3)
C1	P1	C3	102.3(2)	C37	C38	C50	115.4(3)
Co2	P2	C31	56.1(1)	Co2	C39	O19	176.6(4)
Co2	P2	C33	109.2(1)	Co2	C40	O20	179.2(6)
C31	P2	C33	102.5(2)	Co2	C41	O21	176.7(4)
P1	C1	C2	112.1(5)	C34	C42	C43	110.3(4)
P1	C3	C4	122.0(3)	C34	C42	C44	114.8(4)
P1	C3	C8	119.0(3)	C34	C42	C45	107.6(4)
C4	C3	C8	118.2(3)	C43	C42	C44	108.9(5)
C3	C4	C5	117.9(3)	C43	C42	C45	107.8(5)
C3	C4	C12	126.0(3)	C44	C42	C45	107.2(5)
C5	C4	C12	115.6(3)	C36	C46	C47	109.8(4)
C4	C5	C6	123.7(4)	C36	C46	C48	112.5(4)
C5	C6	C7	115.5(3)	C36	C46	C49	108.8(3)
C5	C6	C16	123.5(4)	C47	C46	C48	108.1(4)
C7	C6	C16	120.9(4)	C47	C46	C49	109.5(4)
C6	C7	C8	123.8(4)	C48	C46	C49	108.0(4)
C3	C8	C7	117.6(3)	C38	C50	C51	116.7(3)
C3	C8	C20	125.5(4)	C38	C50	C52	111.6(3)
C7	C8	C20	116.8(4)	C38	C50	C53	105.9(3)
Co1	C9	O9	177.6(7)	C51	C50	C52	109.1(3)
Co1	C10	O10	177.8(5)	C51	C50	C53	105.4(4)
Co1	C11	O11	177.1(4)	C52	C50	C53	107.5(4)

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