Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71083 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1024]

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Acta Cryst. (1993). C49, 1456–1458

Structure of [1,2-Bis(diphenylphosphino)ethane]chloro(η^5-cyclopentadienyl)ruthenium(II)

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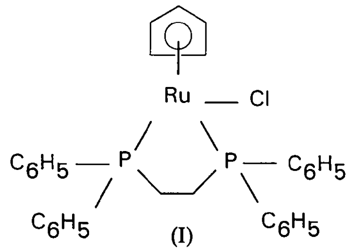
Abstract

The asymmetric unit is comprised of a molecule of (η^5-Cp)RuCl(dppe), where dppe is 1,2-bis(diphenylphosphino)ethane, and a co-crystallized molecule of trichloromethane-*d* solvent. Bonds to the P atoms of the chelating dppe ligand have an average length of 2.279 (2) Å and the Ru—Cl bond measures 2.452 (2) Å. The Ru atom is located at a distance of 1.851 Å from the centroid defined by the five C atoms of the Cp ring, which is planar and coordinates in an η^5 manner.

Comment

The chemistry of Ru complexes containing cyclopentadienyl ligands is extensive. A large number of bis(phosphine)(η^5-C_5H_5)Ru^{II} halide complexes have been prepared and the structures of (η^5-C_5H_5)RuClL_2 (L = PMe_3, PPh_3) have been described (Bruce, Wong, Skelton & White, 1981). The ancillary chloride and phosphine ligands in these complexes can be readily replaced to give a variety of neutral and cationic derivatives, including (η^5-Cp)Ru^{II} complexes containing chelating phosphines (Davies, McNally & Smallridge, 1990). The preparations of two complexes containing the chelating phosphines 1,2-bis(cyclohexylphosphino)methane (dcpm) and 2-methyl-1,2-bis(triphenylphosphino)ethane (prophos) have recently been described and their structures determined (Joslin, Mague & Roundhill, 1991; Morandini, Consiglio, Straub, Ciani & Sironi, 1983).

The title compound (I) was prepared according to the literature procedure (Ashby, Bruce, Tomkins & Wallis, 1979) and isolated fortuitously as well formed red crystals following crystallization from $CDCl_3$. The asymmetric unit consists of one complete molecule of (η^5-Cp)RuCl(dppe) and a co-crystallized molecule of $CDCl_3$ solvent. The Ru atom possesses an approximately octahedral coordination geometry and forms bonds to the two P atoms of the chelating dppe group, a chloride ligand and the cyclopentadienyl ligand. The bond to Cl(1) measures 2.452 (2) Å and bonds to the P atoms measure 2.275 (2) and 2.282 (2) Å for Ru(1)—P(1) and Ru(1)—P(2), respectively.



(I)

Within the cyclopentadienyl ligand we find the five C atoms to be essentially coplanar with a maximum out-of-plane displacement of 0.0123 Å (mean deviation of 0.0079 Å) and C—C bonds in the range 1.372 (8) to 1.421 (9) Å. The Ru atom is 1.851 Å from the centroid defined by the five C atoms of the Cp ring and is displaced slightly (approximately 0.08 Å) towards the C(54) and C(55) atoms which are *trans* to Cl(1).

Structural features of this complex resemble the chiral complex (η^5-Cp)RuCl(prophos), where prophos is a chiral derivative of dppe containing a methyl group on the ethylene bridge (Morandini, Consiglio, Straub, Ciani & Sironi, 1983). Bond lengths and angles within the coordination sphere of the Ru atom are also in good agreement with those reported for another (η^5-Cp)Ru^{II} chloride complex containing dcpm. A principal difference between the two lies in the bite angle of the latter which, owing to the fact that it contains one atom less in the carbon bridge linking the two phosphine moieties, is remarkably acute [P—Ru—P = 69.57 (8)° (Joslin, Mague & Roundhill, 1991)].

Significantly, the nature of the ancillary *R* group bonded to P of the phosphine ligand appears to have only a minor influence on the coordination geometry about the transition-metal center. A comparison of the structures of complexes containing chelating phosphines with those containing monodentate phosphines such as (η^5-Cp)RuCl(PR_3)$_2$ (*R* = Ph, Me) indicates that the P—Ru—P angle in those complexes containing chelating phosphines is acute, whereas in complexes containing either PMe$_3$ or PPh$_3$ the P—Ru—P angles are obtuse (Bruce, Wong, Skelton & White, 1981).

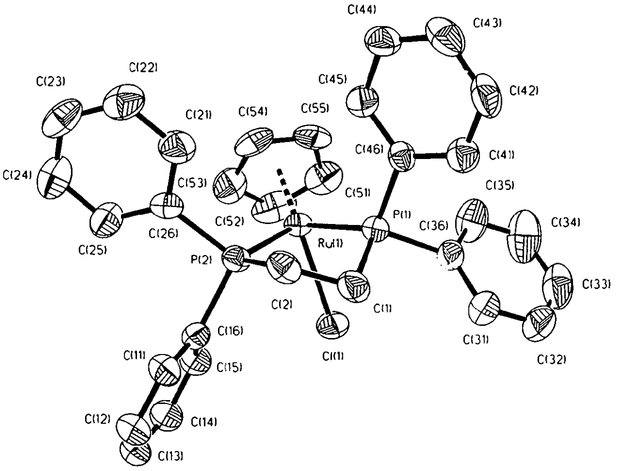


Fig. 1. A perspective view of the structure illustrating the atomic numbering scheme. H atoms have been omitted for clarity and the solvent molecule is not shown.

Experimental

Crystal data

[RuCl($C_{26}H_{24}P_2$)(C_5H_5)].-CDCl$_3$
M_r = 719.4
D_x = 1.522 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Monoclinic
$P2_1/c$
a = 15.582 (7) Å
b = 10.816 (5) Å
c = 19.701 (10) Å
β = 108.99 (4)°
V = 3140 (3) Å^3
Z = 4
Cell parameters from 47 reflections
θ = 7.5-15°
μ = 0.955 mm^{-1}
T = 295 K
Irregular
0.80 × 0.80 × 0.70 mm
Red

Data collection

Siemens *R3m/V* diffractometer
ω scans
Absorption correction: empirical
$T_{\min}$ = 0.287, $T_{\max}$ = 0.418
4315 measured reflections
4136 independent reflections
4136 observed reflections
$R_{\rm int}$ = 0.033
$\theta_{\max}$ = 22.5°
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 11$
$l = -21 \rightarrow 20$
3 standard reflections monitored every 97 reflections
intensity variation: <2%

Refinement

Refinement on F
Final R = 4.82
wR = 7.50
S = 0.79
4135 reflections
304 parameters
H-atom parameters not refined
$w = [\sigma^2(F) + 0.0064F^2]^{-1}$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = −0.62 e Å^{-3}
$\Delta\rho_{\min}$ = 0.51 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{\rm eq}$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U_{\rm eq}$
Ru(1)	0.2179 (1)	0.4286 (1)	0.6757 (1)	0.034 (1)
Cl(1)	0.1706 (1)	0.2152 (1)	0.6863 (1)	0.051 (1)
Cl(2)	0.2478 (2)	0.5097 (3)	0.0581 (1)	0.138 (1)
Cl(3)	0.3440 (2)	0.5938 (3)	0.1990 (2)	0.151 (2)
Cl(4)	0.4231 (2)	0.4134 (2)	0.1324 (2)	0.123 (1)
P(1)	0.2613 (1)	0.4227 (1)	0.7977 (1)	0.036 (1)
P(2)	0.0876 (1)	0.5106 (1)	0.6857 (1)	0.036 (1)
C(1)	0.1589 (3)	0.4235 (5)	0.8243 (3)	0.049 (2)
C(2)	0.0990 (3)	0.5276 (5)	0.7820 (2)	0.045 (2)
C(3)	0.3241 (4)	0.4683 (6)	0.1409 (3)	0.071 (3)
C(11)	−0.0963 (2)	0.4605 (3)	0.6655 (2)	0.051 (2)
C(12)	−0.1779	0.3974	0.6350	0.065 (2)
C(13)	−0.1834	0.3024	0.5859	0.069 (2)
C(14)	−0.1072	0.2706	0.5672	0.064 (2)
C(15)	−0.0256	0.3337	0.5976	0.053 (2)
C(16)	−0.0202	0.4286	0.6468	0.042 (2)
C(21)	0.1051 (2)	0.7692 (3)	0.6843 (2)	0.057 (2)
C(22)	0.0861	0.8870	0.6543	0.060 (2)
C(23)	0.0187	0.9029	0.5880	0.061 (3)
C(24)	−0.0299	0.8010	0.5518	0.068 (2)
C(25)	−0.0109	0.6832	0.5817	0.055 (2)
C(26)	0.0566	0.6673	0.6480	0.040 (2)
C(31)	0.3031 (2)	0.2097 (3)	0.8881 (2)	0.071 (2)
C(32)	0.3591	0.1107	0.9200	0.093 (3)
C(33)	0.4409	0.0927	0.9069	0.085 (3)
C(34)	0.4667	0.1736	0.8619	0.092 (3)
C(35)	0.4107	0.2726	0.8300	0.069 (3)
C(36)	0.3289	0.2907	0.8431	0.046 (2)
C(41)	0.3693 (2)	0.5332 (2)	0.9257 (1)	0.050 (2)

C(42)	0.4199	0.6288	0.9674	0.061 (2)
C(43)	0.4296	0.7406	0.9353	0.061 (2)
C(44)	0.3886	0.7568	0.8616	0.052 (2)
C(45)	0.3380	0.6612	0.8199	0.047 (2)
C(46)	0.3283	0.5494	0.8519	0.036 (2)
C(51)	0.3329 (4)	0.3984 (6)	0.6342 (3)	0.065 (3)
C(52)	0.2521 (5)	0.3872 (6)	0.5767 (3)	0.075 (3)
C(53)	0.2063 (4)	0.5005 (6)	0.5673 (3)	0.064 (2)
C(54)	0.2579 (4)	0.5805 (5)	0.6213 (3)	0.058 (2)
C(55)	0.3349 (4)	0.5200 (5)	0.6619 (3)	0.058 (2)

Table 2. *Geometric parameters* (Å, °)

Ru(1)—Cl(1)	2.452 (2)	Ru(1)—P(1)	2.275 (2)
Ru(1)—P(2)	2.282 (2)	Ru(1)—C(51)	2.223 (8)
Ru(1)—C(52)	2.227 (8)	Ru(1)—C(53)	2.224 (6)
Ru(1)—C(54)	2.161 (6)	Ru(1)—C(55)	2.167 (6)
Cl(2)—C(3)	1.737 (6)	Cl(3)—C(3)	1.737 (7)
Cl(4)—C(3)	1.711 (8)	P(1)—C(1)	1.834 (6)
P(1)—C(36)	1.827 (4)	P(1)—C(46)	1.839 (3)
P(2)—C(2)	1.856 (5)	P(2)—C(16)	1.832 (3)
P(2)—C(26)	1.850 (3)	C(1)—C(2)	1.525 (7)
C(51)—C(52)	1.398 (9)	C(51)—C(55)	1.421 (9)
C(52)—C(53)	1.399 (10)	C(53)—C(54)	1.404 (8)
C(54)—C(55)	1.372 (8)		
Cl(1)—Ru(1)—P(1)	83.0 (1)	Cl(1)—Ru(1)—P(2)	93.3 (1)
P(1)—Ru(1)—P(2)	83.5 (1)	Cl(1)—Ru(1)—C(51)	101.2 (2)
P(1)—Ru(1)—C(51)	112.7 (2)	P(2)—Ru(1)—C(51)	159.3 (2)
Cl(1)—Ru(1)—C(52)	92.9 (2)	P(1)—Ru(1)—C(52)	147.7 (2)
P(2)—Ru(1)—C(52)	128.8 (2)	C(51)—Ru(1)—C(52)	36.6 (2)
Cl(1)—Ru(1)—C(53)	118.6 (2)	P(1)—Ru(1)—C(53)	158.0 (2)
P(2)—Ru(1)—C(53)	98.6 (2)	C(51)—Ru(1)—C(53)	61.4 (2)
C(52)—Ru(1)—C(53)	36.6 (2)	Cl(1)—Ru(1)—C(54)	154.3 (2)
P(1)—Ru(1)—C(54)	120.7 (1)	P(2)—Ru(1)—C(54)	98.9 (2)
C(51)—Ru(1)—C(54)	62.1 (2)	C(52)—Ru(1)—C(54)	61.9 (2)
C(53)—Ru(1)—C(54)	37.3 (2)	Cl(1)—Ru(1)—C(55)	136.8 (2)
P(1)—Ru(1)—C(55)	99.9 (2)	P(2)—Ru(1)—C(55)	129.9 (2)
C(51)—Ru(1)—C(55)	37.7 (2)	C(52)—Ru(1)—C(55)	62.1 (3)
C(53)—Ru(1)—C(55)	61.9 (2)	C(54)—Ru(1)—C(55)	37.0 (2)
Ru(1)—P(1)—C(1)	108.3 (2)	Ru(1)—P(1)—C(36)	117.7 (1)
C(1)—P(1)—C(36)	106.2 (2)	Ru(1)—P(1)—C(46)	120.7 (1)
C(1)—P(1)—C(46)	102.3 (2)	C(36)—P(1)—C(46)	99.8 (1)
Ru(1)—P(2)—C(2)	109.6 (2)	Ru(1)—P(2)—C(16)	119.7 (1)
C(2)—P(2)—C(16)	104.2 (2)	Ru(1)—P(2)—C(26)	116.5 (1)
C(2)—P(2)—C(26)	104.2 (2)	C(16)—P(2)—C(26)	101.0 (1)
P(1)—C(1)—C(2)	106.4 (4)	P(2)—C(2)—C(1)	108.8 (4)
Cl(2)—C(3)—Cl(3)	109.8 (4)	Cl(2)—C(3)—Cl(4)	111.1 (4)
Cl(3)—C(3)—Cl(4)	111.3 (4)	P(2)—C(16)—C(11)	120.8 (1)
P(2)—C(16)—C(15)	119.2 (1)	P(2)—C(26)—C(21)	119.4 (1)
P(2)—C(26)—C(25)	120.5 (1)	P(1)—C(36)—C(31)	124.3 (1)
P(1)—C(36)—C(35)	115.7 (1)	P(1)—C(46)—C(41)	119.6 (1)
P(1)—C(46)—C(45)	120.4 (1)	Ru(1)—C(51)—C(52)	71.9 (5)
Ru(1)—C(51)—C(55)	69.0 (4)	C(52)—C(51)—C(55)	107.0 (5)
Ru(1)—C(52)—C(51)	71.5 (4)	Ru(1)—C(52)—C(53)	71.5 (4)
C(51)—C(52)—C(53)	108.5 (5)	Ru(1)—C(53)—C(52)	71.8 (4)
Ru(1)—C(53)—C(54)	68.9 (3)	C(52)—C(53)—C(54)	107.3 (5)
Ru(1)—C(54)—C(53)	73.8 (4)	Ru(1)—C(54)—C(55)	71.8 (4)
C(53)—C(54)—C(55)	108.9 (5)	Ru(1)—C(55)—C(51)	73.3 (4)
Ru(1)—C(55)—C(54)	71.3 (4)	C(51)—C(55)—C(54)	108.2 (5)

Phenyl rings were refined as rigid groups using H atoms placed in idealized postions with fixed isotropic $U = 0.08$ Å^2. The $\bar{1}11$ reflection suffered from extinction and was therefore omitted during the refinement process. All structure calculations were performed with the *SHELXTL-Plus* system (Sheldrick, 1990).

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Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71065 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1036]

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A New Polymorph of Tetraruthenium Tridecacarbonyl Dihydride

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Abstract

The tetranuclear ruthenium carbonyl hydride cluster, di-μ-carbonyl-undecacarbonyl-di-μ-hydrido-*tetrahedro*-tetraruthenium(6 *Ru*—*Ru*), crystallizes in a monoclinic cell with two crystallographically independent $H_2Ru_4(CO)_{13}$ molecules, each with a tetrahedral array of Ru atoms as also previously found in two earlier polymorphs. Two of the Ru—Ru vectors [Ru(2)—Ru(3) 2.790 (1), Ru(2′)—Ru(3′) 2.778 (1) Å and Ru(3)—Ru(4) 2.782 (2), Ru(3′)—Ru(4′) 2.817 (1) Å] are unsymmetrically bridged by carbonyl ligands. The fourth Ru atom, Ru(1), caps the basal plane of Ru(2), Ru(3) and Ru(4) and is unique in that it carries three terminal carbonyl ligands, only.

Comment

This compound is the third reported polymorph of $H_2Ru_4(CO)_{13}$. A monoclinic form was reported by

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