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cis-Dichlorobis(1-butenyldiphenylphosphine)-ruthenium(II)

ALEXANDER J. BLAKE,* THOMAS EASTON AND
T. ANTHONY STEPHENSON

*Department of Chemistry, The University of Edinburgh,
West Mains Road, Edinburgh EH9 3JJ, Scotland*

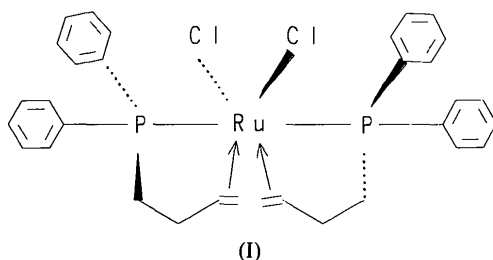
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Abstract

The isomer found in the crystal has the Cl ligands mutually *cis*, the P donors mutually *trans*, and the distorted octahedral coordination is completed by two mutually *cis* π bonds from the terminal $-\text{CH}=\text{CH}_2$ groups.

Comment

The title compound (I) was prepared by heating $\text{RuCl}_2(\text{PPh}_3)_3$ with $\text{PPh}_2\text{CH}_2\text{CH}_2\text{CH}=\text{CH}_2$ in petroleum spirit (333–353 K boiling fraction) and crystals were obtained from $\text{CH}_2\text{Cl}_2/\text{CH}_3\text{OH}$. The structure determination was undertaken to identify which isomer was present and to establish the details of its geometry.



Phosphines which contain substituents carrying potential donor atoms are an important class of polyfunctional ligands; for example $\text{P}(\text{CH}_2\text{CH}_2\text{CN})_3$ or the corresponding chalcogenides have potential PN_3 or EN_3 ($E = \text{O}, \text{S}$ or Se) donor sets (Blake & McQuillan, 1984). The phosphine here is potentially bifunctional with the possibility of coordination *via* the P atom and another group. While there is a multitude of Ru complexes containing phosphine ligands and many examples involving alkenes {e.g. $[(\text{CH}_3\text{CN})(\text{CO})\text{Cl}_2(\text{cycloocta-1,5-diene})\text{Ru}^{\text{II}}]$ (Gould, Jones, Robertson & Stephenson, 1977); $[\text{Cl}_2(\text{norbornadiene})(\text{PhNH}_2)_2\text{Ru}^{\text{II}}]$ (Gaughan, Ibers & Manoli, 1974)}, those containing both are less common {e.g. $[(\text{CO})\text{Cl}_2(\text{CH}_2=\text{CH}_2)(\text{PMe}_2\text{Ph})_2\text{Ru}^{\text{II}}]$ (Barnard, Brown, Daniels, Ibers & Mawby, 1978)}.

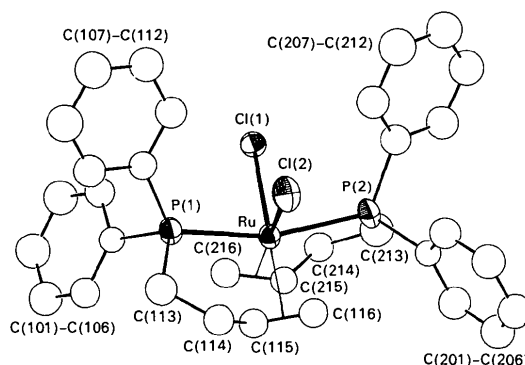


Fig. 1. View of the molecule showing the labelling of the non-H atoms. Thermal ellipsoids are drawn at the 30% probability level; H atoms have been omitted for clarity.

The title compound is unusual in that the phosphine and alkene functions are combined in one ligand.

Fig. 1 was produced using the interactive version of *ORTEP* (Johnson, 1976) included in the *GX* crystallographic program system (Mallinson & Muir, 1985); molecular geometry calculations utilized *CALC* (Gould & Taylor, 1985).

Experimental

Crystal data

$[\text{RuCl}_2(\text{C}_{16}\text{H}_{17}\text{P})_2]$

$M_r = 652.5$

Monoclinic

$P2_1$

$a = 9.1814$ (25) Å

$b = 14.841$ (6) Å

$c = 11.598$ (4) Å

$\beta = 113.301$ (20)°

Cell parameters from 17 reflections

$\theta = 5.5\text{--}21.5^\circ$

$V = 1452$ Å³

$Z = 2$

$D_x = 1.493$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

$\mu = 0.80$ mm⁻¹

$T = 298$ K

Block

$0.36 \times 0.24 \times 0.20$ mm

Yellow

Data collection

Stoe Stadi-2 two-circle diffractometer

ω scans

Absorption correction:

none

2726 measured reflections

2431 observed reflections

$[F > 2.0\sigma(F)]$

$R_{\text{int}} = 0.012$

$\theta_{\text{max}} = 25^\circ$

$h = -10 \rightarrow 10$

$k = 0 \rightarrow 17$

$l = 0 \rightarrow 13$

2 standard reflections

monitored every fourth layer

intensity variation:

$\leq \pm 2\%$

Refinement

Refinement on F

Final $R = 0.0583$

$wR = 0.0704$

$S = 1.290$

2431 reflections

138 parameters

H-atom positions calculated

$w = 1/[\sigma^2(F) + 0.001F^2]$

$(\Delta/\sigma)_{\text{max}} = 0.06$

$\Delta\rho_{\text{max}} = 0.80$ e Å⁻³

$\Delta\rho_{\text{min}} = -1.19$ e Å⁻³

Atomic scattering factors

inlaid except for Ru (Cromer & Mann, 1968)

Table 1. Atomic coordinates and isotropic/equivalent thermal parameters (\AA^2)

Ru, Cl and P atoms were refined with anisotropic thermal parameters;

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	U_{iso}/U_{eq}
Ru	0.12020 (8)	0.2500†	0.18872 (6)	0.0330 (4)
Cl(1)	0.1314 (4)	0.41392 (21)	0.1585 (3)	0.0549 (19)
Cl(2)	-0.0738 (3)	0.2583 (3)	0.28477 (22)	0.0404 (14)
P(1)	-0.1210 (3)	0.2464 (3)	-0.00130 (21)	0.0403 (13)
P(2)	0.3211 (3)	0.28946 (21)	0.39224 (24)	0.0382 (15)
C(101)	-0.1089 (10)	0.2675 (3)	-0.1546 (7)	0.0380 (24)
C(102)	-0.0961 (10)	0.1993 (3)	-0.2327 (7)	0.057 (3)
C(103)	-0.0837 (10)	0.2211 (3)	-0.3455 (7)	0.063 (4)
C(104)	-0.0841 (10)	0.3112 (3)	-0.3801 (7)	0.063 (4)
C(105)	-0.0969 (10)	0.3794 (3)	-0.3019 (7)	0.064 (3)
C(106)	-0.1093 (10)	0.3576 (3)	-0.1891 (7)	0.055 (3)
C(107)	-0.2876 (9)	0.3207 (5)	-0.0167 (7)	0.052 (3)
C(108)	-0.4392 (9)	0.2936 (5)	-0.0978 (7)	0.059 (3)
C(109)	-0.5688 (9)	0.3500 (5)	-0.1189 (7)	0.068 (4)
C(110)	-0.5469 (9)	0.4334 (5)	-0.0590 (7)	0.072 (4)
C(111)	-0.3954 (9)	0.4605 (5)	0.0220 (7)	0.078 (4)
C(112)	-0.2657 (9)	0.4041 (5)	0.0432 (7)	0.060 (3)
C(113)	-0.1966 (17)	0.1324 (9)	-0.0115 (13)	0.063 (3)
C(114)	-0.0986 (11)	0.0783 (8)	0.1041 (8)	0.047 (3)
C(115)	0.0717 (14)	0.1041 (8)	0.1504 (11)	0.041 (3)
C(116)	0.1697 (10)	0.1159 (7)	0.2751 (9)	0.048 (3)
C(201)	0.3727 (8)	0.2058 (5)	0.5200 (7)	0.0372 (23)
C(202)	0.2678 (8)	0.1961 (5)	0.5794 (7)	0.051 (3)
C(203)	0.2914 (8)	0.1277 (5)	0.6675 (7)	0.066 (4)
C(204)	0.4197 (8)	0.0691 (5)	0.6963 (7)	0.067 (4)
C(205)	0.5246 (8)	0.0788 (5)	0.6369 (7)	0.062 (3)
C(206)	0.5010 (8)	0.1472 (5)	0.5488 (7)	0.049 (3)
C(207)	0.2985 (7)	0.3898 (5)	0.4797 (7)	0.044 (3)
C(208)	0.1549 (7)	0.4348 (5)	0.4508 (7)	0.054 (3)
C(209)	0.1460 (7)	0.5071 (5)	0.5245 (7)	0.066 (4)
C(210)	0.2806 (7)	0.5346 (5)	0.6270 (7)	0.075 (4)
C(211)	0.4242 (7)	0.4897 (5)	0.6559 (7)	0.075 (4)
C(212)	0.4332 (7)	0.4173 (5)	0.5823 (7)	0.058 (3)
C(213)	0.5038 (15)	0.3132 (9)	0.3684 (12)	0.056 (3)
C(214)	0.4700 (11)	0.3069 (8)	0.2291 (8)	0.050 (3)
C(215)	0.3501 (12)	0.2351 (8)	0.1649 (10)	0.046 (3)
C(216)	0.2241 (11)	0.2445 (5)	0.0463 (7)	0.0452 (22)

† Coordinate fixed to define origin for noncentrosymmetric structure.

Table 2. Geometric parameters (\AA , $^\circ$)

Ru—Cl(1)	2.466 (3)	C(113)—C(114)	1.515 (18)
Ru—Cl(2)	2.450 (3)	C(114)—C(115)	1.489 (16)
Ru—P(1)	2.430 (3)	C(115)—C(116)	1.380 (16)
Ru—P(2)	2.423 (3)	P(2)—C(201)	1.846 (8)
Ru—M(1)*	2.096 (13)	P(2)—C(207)	1.859 (8)
Ru—M(2)	2.114 (10)	P(2)—C(213)	1.840 (14)
P(1)—C(101)	1.851 (8)	C(213)—C(214)	1.522 (17)
P(1)—C(107)	1.837 (8)	C(214)—C(215)	1.502 (16)
P(1)—C(113)	1.814 (15)	C(215)—C(216)	1.411 (14)
Cl(1)—Ru—Cl(2)	95.38 (11)	P(1)—C(101)—C(102)	123.6 (6)
Cl(1)—Ru—P(1)	88.12 (11)	P(1)—C(101)—C(106)	116.4 (6)
Cl(1)—Ru—P(2)	80.88 (10)	P(1)—C(107)—C(108)	117.6 (6)
Cl(1)—Ru—M(1)	177.2 (4)	P(1)—C(107)—C(112)	122.3 (6)
Cl(1)—Ru—M(2)	85.8 (3)	P(1)—C(113)—C(114)	111.0 (9)
Cl(2)—Ru—P(1)	81.24 (11)	C(113)—C(114)—C(115)	110.8 (10)
Cl(2)—Ru—P(2)	87.43 (10)	C(114)—C(115)—C(116)	124.6 (11)
Cl(2)—Ru—M(1)	87.3 (4)	Ru—P(2)—C(201)	118.3 (3)
Cl(2)—Ru—M(2)	178.8 (3)	Ru—P(2)—C(207)	121.3 (3)

P(1)—Ru—P(2)	163.41 (11)	Ru—P(2)—C(213)	106.7 (4)
P(1)—Ru—M(1)	93.0 (4)	C(201)—P(2)—C(207)	98.6 (4)
P(1)—Ru—M(2)	98.6 (3)	C(201)—P(2)—C(213)	106.9 (5)
P(2)—Ru—M(1)	98.6 (4)	C(207)—P(2)—C(213)	103.5 (5)
P(2)—Ru—M(2)	93.0 (3)	P(2)—C(201)—C(202)	117.2 (6)
M(1)—Ru—M(2)	91.5 (4)	P(2)—C(201)—C(206)	122.4 (6)
Ru—P(1)—C(101)	119.2 (3)	P(2)—C(207)—C(208)	123.4 (6)
Ru—P(1)—C(107)	120.0 (3)	P(2)—C(207)—C(212)	116.5 (6)
Ru—P(1)—C(113)	105.5 (5)	P(2)—C(213)—C(214)	109.4 (9)
C(101)—P(1)—C(107)	99.7 (4)	C(213)—C(214)—C(215)	111.8 (9)
C(101)—P(1)—C(113)	105.2 (5)	C(214)—C(215)—C(216)	124.8 (10)
C(107)—P(1)—C(113)	105.7 (6)		

* M(1) and M(2) are the midpoints of the double bonds C(115)—C(116) and C(215)=C(216), respectively.

Programs used to solve structure: *SHELX76* (Sheldrick, 1976) followed by *DIRDIF* (Beurskens *et al.*, 1983). Program used to refine structure: *SHELX76* (Sheldrick, 1976). The crystal was pre-aligned photographically and mounted with *b* parallel to the spindle axis of the diffractometer. The ω -scan width was $[2.0+1.0(\sin\mu/\tan\theta)]^\circ$ at a scan rate of $1.2^\circ \text{ min}^{-1}$ and with static background counts for 10 s on each side of the peak position. Refinement was by full-matrix least squares. Phenyl rings were treated as rigid groups.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, and bond distances and angles, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55109 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1005]

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