Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

trans-Dichlorobis(3,5-dimethylpyrazole-*N*)-bis(triphenylphosphine-*P*)ruthenium(II)

Suchada Chantrapromma,[a]* Hoong-Kun Fun,[b] Ibrahim Abdul Razak,[b] Qian-Feng Zhang[c] and Xinquan Xin[c]

[a]Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [c]Coordination Chemistry Institute and Department of Chemistry, Nanjing University, Nanjing 210093, People's Republic of China

Correspondence e-mail:
suchada@ratree.psu.ac.th

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.109
Data-to-parameter ratio = 20.5

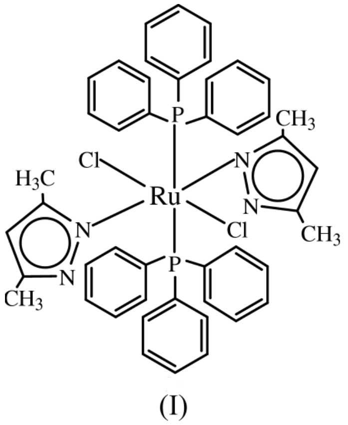
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 17 April 2001
Accepted 26 April 2001
Online 30 April 2001

In the title centrosymmetric Ru^{II} mononuclear complex, *trans*-$Ru(PPh_3)_2(3,5\text{-}Me_2pz)_2Cl_2$ (pz = pyrazole), $[RuCl_2(C_5H_7N_2)_2(C_{18}H_{15}P)_2]$, the Ru^{II} ion exhibits an octahedral coordination environment, consisting of the two *trans* P atoms of PPh_3, the two *trans* N atoms of 3,5-Me_2pz and the two *trans* Cl atoms. The 3,5-Me_2pz ligands coordinate terminally to the central Ru^{II} ion.

Comment

The chemistry of ruthenium complexes containing pyrazolate ligands (pz) has attracted much attention during the last decade (Monica & Ardizzoia, 1997). Much of this interest arises from their role in important catalytic reactions; in particular, attention has focused on the complexes containing electron-rich metal centers (Okoroafor *et al.*, 1988). Also, these studies have revealed that pyrazolates exhibit rich coordination modes (Monica & Ardizzoia, 1997). Although reports on the Ru^{II} complexes with phosphine ligands are common, however, the number of Ru^{II} complexes with phosphine and pz mixed ligands is limited (Sherlock *et al.*, 1989). Several hetero-binuclear complexes formed by pz bridges have recently appeared (Garcia *et al.*, 1990*a*,*b*). In this context, we present the crystal structure of the title complex, (I).



(I)

The asymmetric unit of (I) contains one-half of the title complex, with the other half generated by inversion through the Ru atom; the Ru atom lies at the origin. A displacement ellipsoid plot with the numbering scheme is shown in Fig. 1. The structure of (I) consists of mononuclear $[Ru(PPh_3)_2(3,5\text{-}Me_2pz)_2Cl_2]$ units. The Ru atom is in a slightly distorted octahedral environment. This coordination is formed by two P atoms from *trans*-triphenylphosphine ligands, two N atoms from *trans*-pz ligands and two Cl atoms. The Ru—P bond

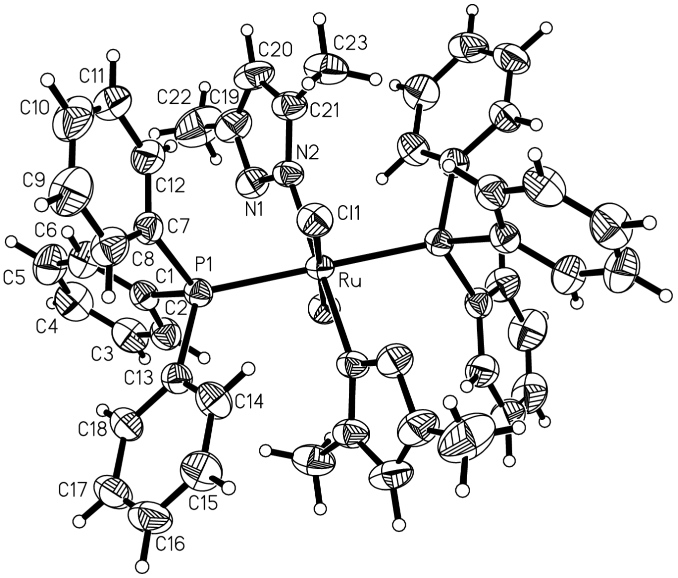


Figure 1
The structure of the title complex showing 50% probability displacement ellipsoids and the atom-numbering scheme.

length in (I) (Table 1) is obviously longer than that in $RuCl(PPh_3)_2\{HB(pz)3\}]$ [2.340 (3) Å; Alcock *et al.*, 1992] with two *cis*-PPh_3 ligands. The Ru—N bond distance in (I) with terminally coordinated pz ligand is significantly shorter than those in the related complexes with bridging pz ligands, such as $[(PPh_3)_2(OC)HRu(m\text{-}pz)_2Rh(cod)]$ (cod = cycloocta-1,5-diene) [2.283 (5) Å; Garcia *et al.*, 1990*a*] and $[(PPh_3)_2(OC)HRu(m\text{-}Cl)(m\text{-}pz)Ir(tfb)]$ (tfb = tetrafluorobenzobarrelene) [2.302 (4) Å; Garcia *et al.*, 1990*b*]. The Ru—Cl bond length is normal. There are two intramolecular C—H···Cl weak interactions, C2—H2*A*···Cl1^{i} [symmetry code: (i) −*x*, −*y*, −*z*] and C23—H23*C*···Cl1, with H···Cl distances of 2.65 and 2.74 Å, respectively.

Experimental

The title complex was obtained from the reaction of $Ru(PPh_3)_3Cl_2$ and 3,5-Me_2pz (molar ratio 1:2) in tetrahydrofuran (THF) (Stephenson & Wilkinson, 1966). To a solution of $Ru(PPh_3)_3Cl_2$ (0.104 mmol) in THF (15 ml) was added 3,5-Me_2pz (0.21 mmol). The mixture was stirred for 2 h and then the solvent was pumped off and the residue washed with hexane. Red crystals suitable for X-ray data collection were obtained by recrystallization from dichloromethane/hexane at room temperature.

Crystal data

$[RuCl_2(C_5H_7N_2)_2(C_{18}H_{15}P)_2]$
$M_r = 886.76$
Monoclinic, $P2_1/c$
$a = 11.6466\ (2)$ Å
$b = 17.1438\ (3)$ Å
$c = 10.5361\ (2)$ Å
$\beta = 95.603\ (1)°$
$V = 2093.66\ (6)$ Å^3
$Z = 2$
$D_x = 1.407$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8192 reflections
$\theta = 3.0–28.4°$
$\mu = 0.62$ mm^{-1}
$T = 293\ (2)$ K
Slab, red
0.28 × 0.26 × 0.20 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.846$, $T_{\max} = 0.887$
14 880 measured reflections
5169 independent reflections
3919 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.052$
$\theta_{\max} = 28.3°$
$h = -15 \rightarrow 15$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.109$
$S = 0.99$
5169 reflections
252 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.023$
$\Delta\rho_{\max} = 1.06$ e Å^{-3}
$\Delta\rho_{\min} = -0.91$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ru—N2	2.1278 (19)	Ru—Cl1	2.4491 (6)
Ru—P1	2.4116 (6)	P1—C13	1.839 (2)
N2—Ru—N2^{i}	180.0	P1—Ru—Cl1^{i}	96.84 (2)
N2—Ru—P1	87.59 (6)	N2—Ru—Cl1	93.76 (6)
N2—Ru—P1^{i}	92.41 (6)	P1—Ru—Cl1	83.16 (2)
P1—Ru—P1^{i}	180.0	Cl1^{i}—Ru—Cl1	180.0
N2—Ru—Cl1^{i}	86.24 (6)		

Symmetry code: (i) −*x*, −*y*, −*z*.

After checking their presence in the difference map, all H atoms were geometrically fixed and allowed to ride on their attached atoms with $U_{\rm iso} = 1.2U_{\rm eq}$ for the attached atoms and $U_{\rm iso} = 1.5U_{\rm eq}$ for methyl H atoms. The highest peak in the difference Fourier map lies close to the Ru atom (0.97 Å)

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

The authors would like to thank the Ministry of Education of China, the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 305/PFIZIK/610942.

References

Alcock, N. W., Burns, I. D., Claire, K. S. & Hill, A. F. (1992). *Inorg. Chem.* **31**, 2906–2908.
Garcia, M. P., Lopez, A. M., Esteruelas, M. A., Lahoz, F. J. & Oro, L. A. (1990*a*). *J. Organomet. Chem.* **388**, 365–377.
Garcia, M. P., Lopez, A. M., Esteruelas, M. A., Lahoz, F. J. & Oro, L. A. (1990*b*). *J. Chem. Soc. Dalton Trans.* pp. 3465–3471.
Monica, G. L. & Ardizzoia, G. A. (1997). *Prog. Inorg. Chem.* **46**, 151–238.
Okoroafor, M. O., Shen, L.-H., Honeychuck, R. V. & Brubaker, C. H. Jr (1988). *Organometallics*, **7**, 1297–1303.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL Software Reference Manual.* Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Sherlock, S. J., Cowie, M., Singleton, E. & Steyn, M. M. de V. (1989). *J. Organomet. Chem.* **361**, 353–365.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Stephenson, T. A. & Wilkinson, G. J. (1966). *Inorg. Nucl. Chem.* **28**, 945–951.

m208 Suchada Chantrapromma *et al.* • $[RuCl_2(C_5H_7N_2)_2(C_{18}H_{15}P)_2]$ *Acta Cryst.* (2001). E**57**, m207–m208