

[RuCl{2,6-bis(3,4-dihydro-2H-pyrrol-5-yl)-pyridine}(PPh₃)₂]BF₄ dichloromethane solvate

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Key indicators

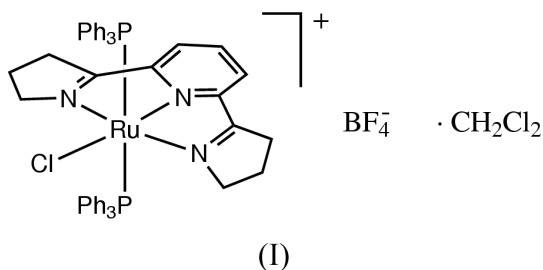
Single-crystal X-ray study
T = 153 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
Disorder in solvent or counterion
R factor = 0.031
wR factor = 0.076
Data-to-parameter ratio = 13.8

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

The tetrafluoroborate salt of the mononuclear octahedral ruthenium(II) complex, [RuCl(L)(PPh₃)₂]BF₄, where L = 2,6-bis(3,4-dihydro-2H-pyrrol-5-yl)pyridine, has been prepared as the dichloromethane solvate. The triphenylphosphine ligands adopt a *trans* configuration, with a P–Ru–P angle of 173.72 (2)°.

Comment

In the context of our study of hydrogenation reactions catalysed by ruthenium complexes, we have synthesized a ruthenium complex containing the tridentate ligand 2,6-bis(3,4-dihydro-2H-pyrrol-5-yl)pyridine, one chlorine, and two triphenylphosphines, [RuCl(L)(PPh₃)₂]⁺, where L = 2,6-bis(3,4-dihydro-2H-pyrrol-5-yl)pyridine. The cationic complex crystallizes as the tetrafluoroborate salt by slow diffusion of ether into a dichloromethane solution, yielding the dichloromethane solvate, (I). The molecular structure of the cationic moiety, depicted in Fig. 1, shows only small distortions from ideal octahedral geometry; selected bond lengths and angles are presented in Table 1.



The PPh₃ groups bend in the direction of the chloride ion to minimize steric interactions with the 2,6-bis(3,4-dihydro-2H-pyrrol-5-yl)pyridine ligand. The P–Ru–P angle is 173.72 (2)°, comparable to that in other *trans*-diphosphine complexes found in the literature, the values ranging between 173.64 (6) and 178.2 (2)° [Venegas-Yazigi *et al.* (2000); Perez *et al.* (1999) and references therein]. The Ru–N bond length *trans* to the chloride ion is shorter than the Ru–N_{pyrrole} bonds, Ru–N2 1.970 (2) Å compared to 2.102 (2) and 2.131 (2) Å for Ru–N1 and Ru–N3, respectively. The same observation has been made in the *trans*-[RuL1(Cl)(PMe₃)₂]⁺ cation, (Bessel *et al.*, 1991), where L1 = 2,6-bis(3-phenylpyrazol-1-yl)pyridine. As observed by Perez *et al.* (1999) in the complexes [RuCl(trpy)(PPh₃)₂] and [RuCl(trpy)(Ph₂PC₆H₄CH₂O(CO)-(CH₂)₃(CO)OCH₂C₆H₄PPh₂)], where trpy = 2,2':6',2''-terpyridine, the phenyl groups of the phosphine ligands influence the geometry in the equatorial plane of the metal. The chloride ligand is pushed by the phenyl groups, creating a

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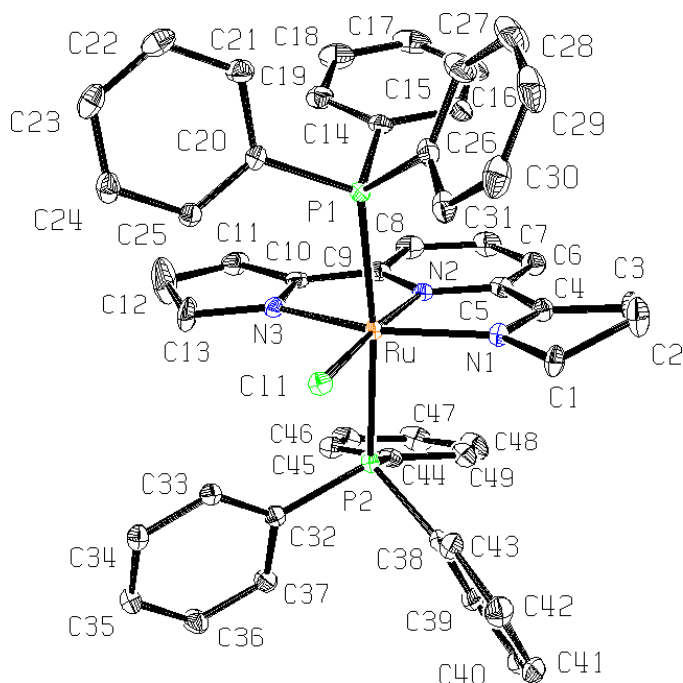


Figure 1
The molecular structure of $[\text{RuCl}(\text{L})(\text{PPh}_3)_2]^+$; for clarity tetrafluoroborate, dichloromethane and H atoms have been omitted. Displacement ellipsoids are drawn at the 50% probability level.

difference between the two $\text{Cl}-\text{Ru}-\text{N}_{\text{pyrrole}}$ bond angles, $\text{Cl}-\text{Ru}-\text{N}_3$ being greater by 12.32° than $\text{Cl}-\text{Ru}-\text{N}_1$.

Experimental

To a dichloromethane solution (15 ml) of $[\text{RuCl}_2(\text{PPh}_3)_3]$ (Holm, 1970) (100 mg, 0.104 mmol) was added, dropwise, a 5 ml solution of 2,6-bis(3,4-dihydro-2H-pyrrol-5-yl)pyridine (Bernauer & Gretillat, 1989) (24 mg, 0.11 mmol). The solution was stirred for 20 minutes and then NaBF_4 (15 mg, 0.14 mmol) was added. After 10 minutes the volume was reduced to 5 ml. A red solid was precipitated by addition of ether (3 ml). The solid was filtered and washed three times with ether to give $[\text{RuCl}(\text{L})(\text{PPh}_3)_2](\text{BF}_4)$ in 63% yield (63 mg). Suitable crystals for X-ray diffraction were grown by slow diffusion of ether into a dichloromethane solution at 277 K.

^1H NMR (CDCl_3): 7.39–7.45 (*m*, 12H), 7.29–7.34 (*m*, 7H), 7.18–7.23 (*m*, 14H), 3.75 (*m*, 4H), 2.75 (*m*, 4H), 1.60 (*m*, 4H). ^{31}P NMR (CDCl_3): 21.6 p.p.m. MS (ESI, *m/z*): 874 Analysis. Calculated for $\text{C}_{50}\text{H}_{47}\text{BCl}_3\text{F}_4\text{N}_3\text{P}_2\text{Ru}$: C, 57.41; N, 4.02 H, 4.53. Found: C, 57.22; N, 4.37; H, 4.72.

Crystal data

$[\text{Ru}(\text{C}_{13}\text{H}_{15}\text{N}_3)(\text{C}_{18}\text{H}_{15}\text{P})_2\text{Cl}]\text{BF}_4 \cdot \text{CH}_2\text{Cl}_2$
 $M_r = 1046.08$
 Triclinic, $P1$
 $a = 10.3368$ (8) Å
 $b = 12.7939$ (11) Å
 $c = 19.1336$ (16) Å
 $\alpha = 80.231$ (10) $^\circ$
 $\beta = 89.193$ (9) $^\circ$
 $\gamma = 67.943$ (9) $^\circ$
 $V = 2307.8$ (3) Å 3
 $Z = 2$
 $D_x = 1.505$ Mg m $^{-3}$
 $\text{Mo } K\alpha$ radiation
 Cell parameters from 8000 reflections
 $\theta = 2.1\text{--}25.9^\circ$
 $\mu = 0.64$ mm $^{-1}$
 $T = 153$ (2) K
 Rod, red
 $0.50 \times 0.30 \times 0.15$ mm

Data collection

Stoe IPDS diffractometer
 φ oscillation scans
 Absorption correction: refined from ΔF (DIFABS; Walker & Stuart, 1983)
 $T_{\text{min}} = 0.699$, $T_{\text{max}} = 0.880$
 16304 measured reflections
 8322 independent reflections
 6363 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$
 $\theta_{\text{max}} = 25.9^\circ$
 $h = -12 \rightarrow 11$
 $k = -15 \rightarrow 15$
 $l = -23 \rightarrow 23$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.076$
 $S = 0.96$
 8322 reflections
 604 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0452P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.56$ e Å $^{-3}$
 $\Delta\rho_{\text{min}} = -0.58$ e Å $^{-3}$

Table 1

Selected geometric parameters (Å, $^\circ$).

N1—Ru	2.102 (2)	P1—Ru	2.4051 (7)
N2—Ru	1.970 (2)	P2—Ru	2.3902 (7)
N3—Ru	2.131 (2)	Cl1—Ru	2.4717 (6)
N2—Ru—N1	77.47 (8)	N2—Ru—Cl1	173.88 (7)
N2—Ru—N3	77.35 (9)	N1—Ru—Cl1	96.43 (6)
N1—Ru—N3	154.81 (8)	N3—Ru—Cl1	108.75 (6)
P2—Ru—P1	173.72 (2)		

Three fluorine atoms of the tetrafluoroborate anion were treated as disordered with partial occupancy factors of 0.70:0.30. H atoms were included in calculated positions and treated as riding atoms, using *SHELXL97* default parameters.

Data collection: *EXPOSE* (Stoe & Cie, 2000); cell refinement: *CELL* (Stoe & Cie, 2000); data reduction: *INTEGRATE* (Stoe & Cie, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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