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$[2,6\text{-Bis}(3,4\text{-dihydro-}2H\text{-pyrrol-5-yl})\text{pyridine-}\kappa^3N]$trichlororhodium(III) monohydrate

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In the solid state, the title mononuclear octahedral rhodium(III) complex, $[RhCl_3(C_{13}H_{15}N_3)]\cdot H_2O$, shows a hydrogen bond between the equatorial Cl atom and the solvent water molecule.

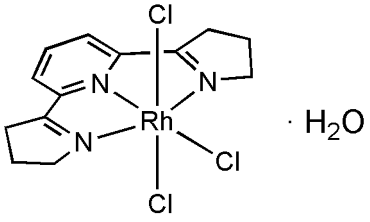
Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.008 Å
R factor = 0.036
wR factor = 0.079
Data-to-parameter ratio = 16.5

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

Comment

The tridentate ligand 2,6-bis(3,4-dihydro-2*H*-pyrrol-5-yl)-pyridine, *L* (Bernauer & Gretillat, 1989), has been used extensively for the synthesis of mononuclear complexes containing one *L* (Cabort, Therrien *et al.*, 2003) or two *L* units around a transition metal (Cabort, Therrien, Stoeckli-Evans *et al.*, 2002; Cabort, Michel *et al.*, 2003). To the best of our knowledge, *L* has never been used as a monodentate, bidentate, or bridging ligand. To investigate these other possible coordination modes, we decided to react *L* with $[Cp^*{}_2Rh_2Cl_2(\mu\text{-}Cl)_2]$ (White *et al.*, 1992). However, under the reaction conditions of refluxing MeOH in the presence of $NaBF_4$, the 1,2,3,4,5-pentamethylcyclopentadienyl (Cp*) moiety is removed, and the title mononuclear complex, $[RhCl_3(L)]$, (I), is formed. Complex (I) has been reported previously by Cabort *et al.* (Cabort, Therrien, Bernauer & Süss-Fink, 2002). Unlike Cabort *et al.*, where compound (I) crystallized with two benzene molecules per asymmetric unit, we have obtained crystals of complex (I) with one molecule of water per asymmetric unit, giving rise to a different packing arrangement and to the formation of an intermolecular hydrogen bond between the equatorial Cl atom and the water molecule.



(I)

The three N atoms of the tridentate ligand, along with the three Cl atoms, form a distorted octahedral geometry around the metal in (I) (Fig. 1, Table 1). The bond distances and angles are similar to those found in other $[RhCl_3(\eta^3\text{-}L)]$ complexes, where *L* is bis(oxazolinyl)pyridine (Nishiyama *et al.*, 1991), bis(pyrazolyl)pyridine (Christenson *et al.*, 1995), 2,6-bis-(ethylidyneimino)pyridine (Haarman *et al.*, 1997), 2,3,6-tris(2-pyridyl)-1,3,5-triazine (Paul *et al.*, 1998), 2,2′:6′,2″-terpyridine (Ziegler *et al.*, 1999; Kwong *et al.*, 2001) or 2,6-bis(3,4-dihydro-

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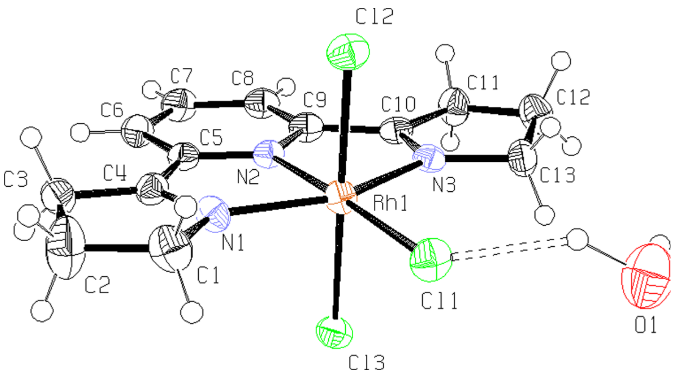

Figure 1
ORTEP-3 plot (Farrugia, 1997) of (I). Displacement ellipsoids are drawn at the 50% probability level.

2*H*-pyrrol-5-yl)pyridine (Cabort, Therrien, Bernauer & Süss-Fink, 2002).

The formation of five-membered chelate rings significantly distorts the geometry around the Rh atom in (I). The N1—Rh1—N2 [78.12 (15)°], N2—Rh1—N3 [78.03 (14)°] and N1—Rh1—N3 [156.08 (15)°] angles are significantly smaller than the values of 90 and 180° expected for an ideal octahedral geometry. The equatorial plane formed by *L* and atoms Rh1 and Cl1 is planar, with an average deviation of 0.069 Å; only atoms C3 and C12 of the pyrrole fragment and atom Cl1 are significantly out of this plane, by −0.135 (4), −0.178 (5) and 0.145 (2) Å, respectively. The distance between Rh and the central N2 atom [1.975 (3) Å] is shorter than the other Rh—N bonds [Rh1—N1 2.064 (4) and Rh1—N3 2.071 (4) Å].

In the crystal of (I), a weak intermolecular hydrogen bond is formed between atom Cl1 and the water molecule, with Cl1—O1 3.153 (5) and Cl1···H1*W*—O1 142°.

Experimental

To a methanol solution (10 ml) of $[Cp^*_2Rh_2Cl_2(\mu\text{-}Cl)_2]$ (30 mg, 0.05 mmol) were added 2,6-bis(3,4-dihydro-2*H*-pyrrol-5-yl)pyridine (20 mg, 0.1 mmol) and $NaBF_4$ (11 mg, 0.1 mmol). The orange solution was stirred and refluxed for 12 h. After cooling to room temperature, a brown precipitate was observed. The precipitate was dissolved in a methanol–water solution. Crystals of (I) were observed after two weeks. ^{1}H NMR (200 MHz, $CDCl_3$, δ, p.p.m.): 8.44 (*t*, 1H), 8.32 (*d*, 2H), 4.14 (*m*, 4H), 3.48 (*m*, 4H), 2.48 (*m*, 4H). MS (ESI, *m*/*z*): 423. $[RhCl_3(L)]$ analysis, calculated for $C_{13}H_{15}Cl_3N_3Rh\cdot H_2O$: C 35.44, H 3.89, N 9.54%; found: C 35.30, H 3.98, N 9.48%.

Crystal data

$[RhCl_3(C_{13}H_{15}N_3)]\cdot H_2O$
$M_r = 440.56$
Monoclinic, $C2/c$
$a = 18.682$ (4) Å
$b = 14.821$ (3) Å
$c = 14.819$ (3) Å
$\beta = 126.57$ (3)°
$V = 3295.4$ (17) Å^3
$Z = 8$
$D_x = 1.776$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6717 reflections
$\theta = 1.9$–25.9°
$\mu = 1.52$ mm^{-1}
$T = 153$ (2) K
Block, red
0.23 × 0.18 × 0.13 mm

Data collection

Stoe IPDS diffractometer
φ oscillation scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{\min} = 0.798$, $T_{\max} = 0.820$
8555 measured reflections
3136 independent reflections
2127 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.052$
$\theta_{\max} = 25.9°$
$h = -21 \rightarrow 22$
$k = -16 \rightarrow 18$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.079$
$S = 0.89$
3136 reflections
190 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0406P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.47$ e Å^{-3}
$\Delta\rho_{\min} = -0.44$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—Rh1	2.064 (4)	Cl1—Rh1	2.3677 (12)
N2—Rh1	1.975 (3)	Cl2—Rh1	2.3431 (16)
N3—Rh1	2.071 (4)	Cl3—Rh1	2.3389 (15)
N2—Rh1—N1	78.12 (15)	N2—Rh1—Cl1	177.27 (10)
N2—Rh1—N3	78.03 (14)	N1—Rh1—Cl1	99.64 (11)
N1—Rh1—N3	156.08 (15)	N3—Rh1—Cl1	104.25 (11)
Cl3—Rh1—Cl2	178.70 (4)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*W*···Cl1	1.21	2.11	3.153 (5)	142

The H atoms were included in calculated positions and treated as riding atoms (C—H = 0.97 and 0.97 Å; $U_{\rm iso} = 1.5U_{\rm eq}$ of the parent atom), except for the H atoms of the water molecule which were located from a Fourier difference map and fixed at their positions.

Data collection: *EXPOSE* in *IPDS* (Stoe & Cie, 2000); cell refinement: *CELL* in *IPDS*; data reduction: *INTEGRATE* in *IPDS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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