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Chloro(dimethylformamide-κO)(hydridotripyrazolylborato)(tripyrrol-1-ylphosphine-κP)-ruthenium(II)

Christian Slugovc,[a] Karl Kirchner[b] and Kurt Mereiter[c]*

[a]Institute for Chemistry and Technology of Organic Materials (ICTOS), Graz University of Technology, Stremayrgasse 16, A-8010 Graz, Austria, [b]Institute of Applied Synthetic Chemistry, Vienna University of Technology, Getreidemarkt 9/163, A-1060 Vienna, Austria, and [c]Institute of Chemical Technologies and Analytics, Vienna University of Technology, Getreidemarkt 9/164, A-1060 Vienna, Austria

Correspondence e-mail:
kurt.mereiter@tuwien.ac.at

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Key indicators

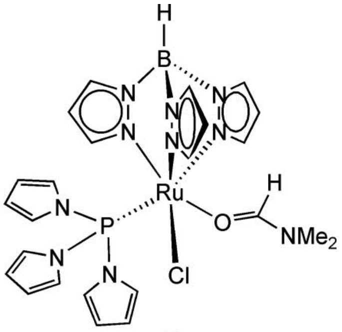
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.025
wR factor = 0.058
Data-to-parameter ratio = 23.3

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

The title compound, [Ru($C_9H_{10}BN_6$)Cl(C_3H_7NO)($C_{12}H_{12}N_3P$)], has the Ru atom in octahedral coordination. The Ru—P bond length of 2.2022 (5) Å is some 0.107 Å shorter than that in the corresponding triphenylphosphine complex; this difference is attributable to the electronic donor–acceptor properties of the phosphine ligand.

Comment

Ruthenium(II) hydridotripyrazolylborate complexes, Ru(Tp), are of interest for stoichiometric and catalytic transformations of organic molecules (Pavlik *et al.*, 2005*a*,*b*, and references therein). In these complexes, phosphine coligands, such as PPh_3 (triphenylphosphine), PCy_3 (tricyclohexylphosphine) or Ph_2PNHPh (phenylaminodiphenylphosphine), are important as electron σ-donors and π-acceptors, which stabilize the metal centre and influence its reactivity but may also take part in ligand migrations, migratory insertions or C—H activations.



(I)

In order to supplement a previous study of [Ru(Tp)Cl(PPh_3)(DMF)] (DMF is dimethylformamide) (Slugovc *et al.*, 1997), we were interested in the corresponding complex with tri(*N*-pyrrolyl)phosphine ($PPyrl_3$) because this coligand, rarely used as yet, is known to be a much weaker σ-donor and a stronger π-acceptor than PPh_3 (Moloy & Petersen, 1995) while being practically isosteric with the latter (Burrows, 2001). Therefore, the title compound, [Ru(Tp)Cl($PPyrl_3$)(DMF)], (I), was synthesized and investigated by X-ray diffraction. In (I), the Ru atom adopts a fairly regular octahedral coordination (Fig. 1 and Table 1) and shows a ligand disposition that is similar to that of [Ru(Tp)Cl(PPh_3)(DMF)], but has a different phosphine group orientation (Fig. 2). The most significant difference in the Ru coordinations of these two compounds is that the Ru—P bond length in (I), 2.2022 (5) Å, is shorter by 0.107 Å than that in [Ru(Tp)Cl(PPh_3)(DMF)], where it is 2.309 (1) Å. This difference agrees qualitatively with the finding on a few other transition metal

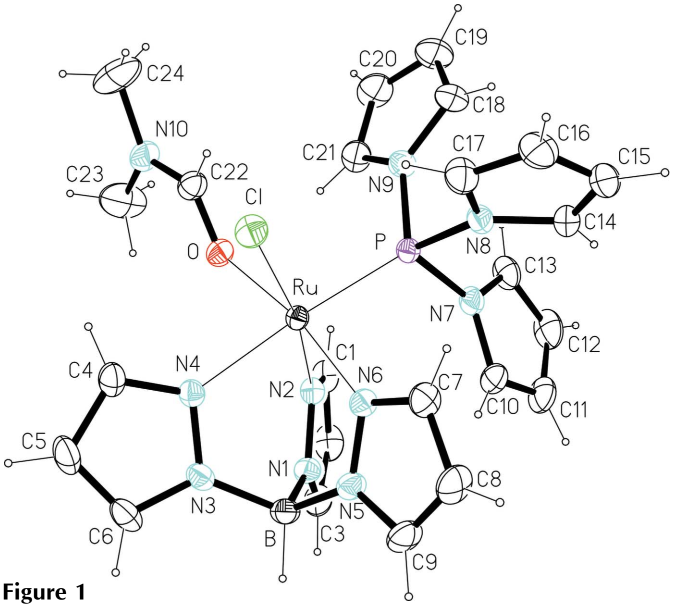


Figure 1
Perspective view of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for the H atoms).

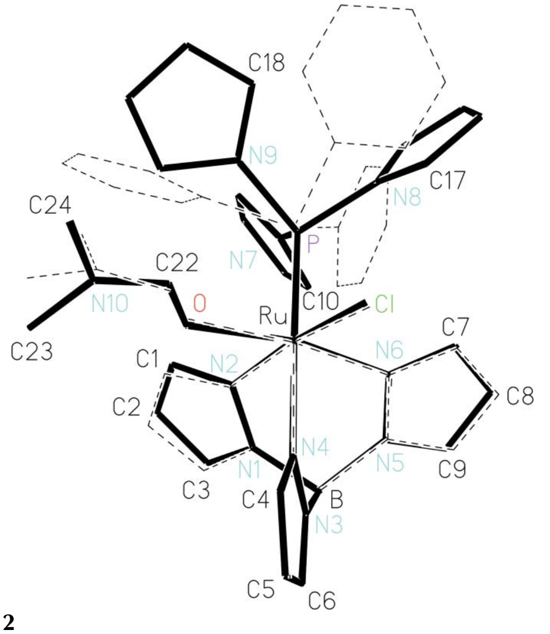


Figure 2
Superposition plot of the Ru complexes in (I) (full lines) and [Ru(Tp)Cl(PPh$_3$)(DMF)] (broken lines; Slugovc *et al.*, 1997). The weighted r.m.s. deviation of the fitted atoms Ru, Cl, P, O, B and N1–N6 is 0.068 Å.

complexes, for which pairs of PPyrl$_3$- and PPh$_3$-containing compounds were studied and the metal–P bonds to PPyrl$_3$ were systematically shorter by 0.05–0.10 Å (Moloy & Petersen, 1995; Huang *et al.*, 1998; Trzeciak *et al.*, 1997). The PPyrl$_3$ ligand in (I) exerts a notable *trans* influence, which causes the Ru—N4 bond to be 0.067 Å longer than the mean of the Ru—N2 and Ru—N6 bond lengths (Table 1). The corresponding *trans* influence in [Ru(Tp)Cl(PPh$_3$)(DMF)] results in a bond-length difference of 0.087 Å. Fig. 2 demonstrates that the phosphines in (I) and [Ru(Tp)Cl(PPh$_3$)(DMF)] both adopt propeller conformations (Burrows, 2001) but do this in a reversed sense and with differing orientations of the propeller arms. This feature is connected with the crystal packing as the two compounds are clearly not isostructural {[Ru(Tp)Cl(PPh$_3$)(DMF)]: monoclinic, $P2_1/c$, a = 9.929 Å, b = 15.293 Å, c = 20.129 Å, β = 94.33°, V = 3048 Å^3}. Coherence in both crystal structures is essentially provided by numerous intermolecular C—H$\cdots\pi$ interactions, whereas π–π stacking is absent.

Experimental

Compound (I) was synthesized from [RuTpCl(COD)] (COD is cycloocta-1,5-diene) (Gemel *et al.*, 1996). A suspension of [RuTpCl(COD)] (140 mg, 0.306 mmol) in dimethylformamide (4 ml) was treated with PPyrl$_3$ (70 mg, 0.306 mmol) and the mixture heated under reflux for 1 h, after which the solvent was removed under vacuum. The yellow solid was washed with *n*-hexane and dried *in vacuo* (yield 169 mg, 85%). Crystals for X-ray diffraction were obtained by layering a solution in CH$_2$Cl$_2$ with diethyl ether.

Crystal data

[Ru(C$_9$H$_{10}$BN$_6$)Cl(C$_3$H$_7$NO)(C$_{12}$H$_{12}$N$_3$P)]
M_r = 651.87
Monoclinic, $P2_1/n$
a = 10.046 (2) Å
b = 22.749 (4) Å
c = 12.683 (2) Å
β = 96.78 (1)°
V = 2878.3 (9) Å^3
Z = 4
D_x = 1.504 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.73 mm^{-1}
T = 298 (2) K
Prism, yellow
0.59 × 0.38 × 0.31 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.641, T_{max} = 0.797
23769 measured reflections
8277 independent reflections
7117 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 30.1°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.058
S = 1.06
8277 reflections
355 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.023P)^2 + 0.8425P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00085 (15)

Table 1
Selected geometric parameters (Å, °).

Ru—N2	2.0652 (13)	Ru—Cl	2.4204 (6)
Ru—N4	2.1315 (13)	P—N7	1.7261 (14)
Ru—N6	2.0631 (13)	P—N8	1.7161 (14)
Ru—O	2.1217 (12)	P—N9	1.7188 (14)
Ru—P	2.2022 (5)		
N8—P—N9	98.41 (7)	N8—P—Ru	120.75 (5)
N8—P—N7	97.82 (7)	N9—P—Ru	121.06 (5)
N9—P—N7	98.92 (7)	N7—P—Ru	115.29 (5)

All H atoms were placed in calculated positions (C—H = 0.93–0.96 Å, B—H = 1.10 Å) and thereafter treated as riding. A torsional parameter was refined for each methyl group. The constraints U_{iso}(H) = 1.2U_{eq}(C$_{aryl,formyl}$,B) and U_{iso}(H) = 1.5U_{eq}(C$_{methyl}$) were applied.

Acta Cryst. (2006). E**62**, m1356–m1358 Slugovc *et al.* • [Ru(C$_9$H$_{10}$BN$_6$)Cl(C$_3$H$_7$NO)(C$_{12}$H$_{12}$N$_3$P)] **m1357**

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*, *SADABS* and *XPREP* in *SHELXTL* (Bruker, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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