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[*mer,trans*-(NN′N)RuCl$_2$(NC$_5$H$_5$)]·C$_7$H$_8$ {NN′N = 2,6-bis-[(dimethylamino)methyl]pyridine}

Stephan Back,[a] Gerd Rheinwald,[a]† Ignacio del Río,[b] Gerard van Koten[c] and Heinrich Lang[a]*

[a]Technische-Universität Chemnitz, Fakultät für Naturwissenschaften, Institut für Chemie, Lehrstuhl Anorganische Chemie, Straße der Nationen 62, D-09111 Chemnitz, Germany, [b]Instituto de Química Organometálica Enrique Moles, Departamento de Química Orgánica e Inorgánica, Facultad de Química, Universidad Oviedo, E-33071 Oviedo, Spain, and [c]Debye-Institute for Metal-Mediated Synthesis, Utrecht University, Padualaan 8, CH 3584 Utrecht, The Netherlands

† Author to whom correspondance should be directed pertaining to the crystallographic section of this manuscript.

Correspondence e-mail:
heinrich.lang@chemie.tu-chemnitz.de

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The title compound, *mer,trans*-{2,6-bis[(dimethylamino)-methyl]pyridine}dichloropyridineruthenium(II) toluene solvate, [RuCl$_2$(C$_{11}$H$_{19}$N$_3$)(C$_5$H$_5$N)]·C$_7$H$_8$, has metal–ligand dimensions of Ru—N$_{py}$(NN′N) 1.962 (2) Å, Ru—N(NN′N) 2.348 (3) Å, Ru—N(NN′N) 2.251 (3) Å, Ru—N$_{py}$ 2.096 (2) Å and Ru—Cl 2.3502 (7) or 2.3581 (7) Å (py = pyridine, C$_5$H$_5$N). Angles are N$_{py}$—Ru—N(NN′N) 178.37 (9)°, (NN′N)N—Ru—N(NN′N) 160.57 (9)° and Cl—Ru—Cl 177.19 (3)°.

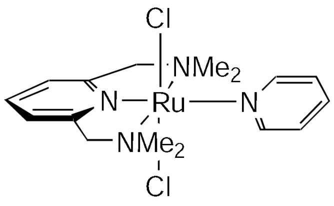
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.052
wR factor = 0.117
Data-to-parameter ratio = 23.4

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

Comment

Recently, interest has been focused on the chemistry of bis-orthochelating aromatic ligands (Rietveld *et al.*, 1997). This was due to their applicability to catalytic processes (Abbenhuis, del Río *et al.*, 1998) or materials science (Steenwinkel *et al.*, 1998). In this context, the dinitrogen-bridged bis-ruthenium complex {Ru}N≡N{Ru}, (I) [{Ru} = *mer,trans*-(C$_5$H$_3$N(CH$_2$NMe$_2$)$_2$-2,6)RuCl$_2$] (Abbenhuis, Boersma & van Koten, 1998), has been studied in the synthesis of bioactive arylpiperazines. In order to gain a deeper insight into the catalytic behaviour of (I), this homodinuclear complex was reacted with various *N*-donor molecules (del Río *et al.*, 2000). Treatment of (I) with two equivalents of py (py = pyridine, C$_5$H$_5$N) leads to the formation of the title compound {Ru}py·C$_7$H$_8$, (II), in high yield by loss of N$_2$. We report here on the structure of (II), which incorporates two different types of pyridine ligands, *i.e.* C$_5$H$_5$N and C$_5$H$_3$N(CH$_2$NMe$_2$)$_2$-2,6.



(II)

The X-ray diffraction study confirms the general structure of (II), suggested on the basis of the NMR (^{1}H, ^{13}C{^{1}H}) and IR data of (II) (Fig. 1).

In (II), the Ru^{II} centre occupies a distorted octahedral environment, with the three N-donor atoms of the NN′N-ligand in a meridional position. The Cl atoms are forced into the apical positions and are *trans*-orientated to each other. In general, the Ru—N$_{py}$, Ru—N$_{NMe}$ and Ru—Cl distances Ru1—Cl1 [2.3502 (7) Å], Ru1—Cl2 [2.3581 (7) Å], Ru1—N1 [1.962 (2) Å], Ru1—N2 [2.348 (3) Å] and Ru1—N3 [2.251 (3) Å], as well as the angles around the Ru^{II} centre Cl1—Ru1—Cl2 [177.19 (3)] and N2—Ru1—N3 [160.57 (9)°]

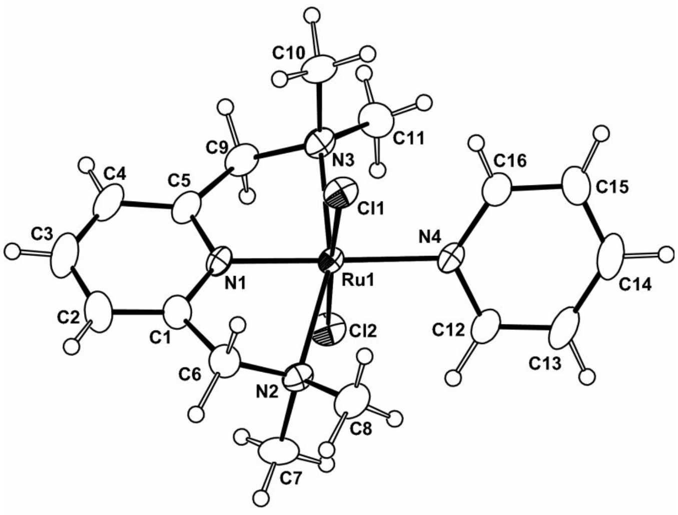


Figure 1
ZORTEP plot (50% probability) of complex (II) with molecular geometry and atom-numbering scheme (the toluene solvate molecule is not shown for clarity).

of the {Ru} building block, are consistent with those values reported for this type of complex fragment (del Rio *et al.*, 2000). The length of the Ru1—N4 bond of 2.096 (2) Å is elongated when compared to the Ru1—N1 bond, but lies in the range of distances reported for other Ru^{II}–pyridine complexes, *e.g.* $[Ru(py)_6]^{2+}$ with 2.10–2.14 Å (Templeton, 1979). Similar $Ru—N_{py}$ distances are found, *e.g.* in $[RuCl(py)_4(pz)]^+$ with 2.105 (4) Å (pz is pyrazine; Coe *et al.*, 1995). This relatively short Ru1—N1 distance can best be explained by the imbedding of the Ru^{II} centre into the framework of the bis-orthochelating NN′N ligand. The angle N1—Ru1—N4 [178.37 (9)°] illustrates the linearity of this array. The planes of the two pyridine ligands are tilted by 54.33 (10)° with respect to each other.

Experimental

A solution of {Ru}N≡N{Ru}, (I) (128 mg, 0.2 mmol), and py (32 mg, 0.4 mmol) in thf (30 ml) was stirred for 1 h at 298 K. During the course of the reaction, the colour of the solution turned from brown to red and the turbidity disappeared. The reaction mixture was then concentrated to 5 ml and *n*-pentane (50 ml) was added. The supernatant liquid was carefully decanted and the residue was dried *in vacuo* to yield (II) [150 mg, 95% yield based on (I)] as a red–brown solid. By diffusion of *n*-pentane into a dichloromethane/*n*-pentane solution of (II), with some added toluene, at 298 K, single crystals of (II) could be obtained. M.p.: 302 K (decomposition). ^{1}H NMR ($CDCl_3$, p.p.m.): [δ] 2.38 (*s*, 12H, NMe_2), 4.03 (*s*, 4H, CH_2), 7.1–7.2 (*m*, 2H, C_6H_3), 7.3–7.4 (*m*, 3H, C_5H_5N), 7.7–7.8 (*m*, 1H, C_5H_3N), 9.6–9.7 (*m*, 2H, C_5H_5N). $^{13}C\{^1H\}$ NMR ($CDCl_3$, p.p.m.): [δ] 53.6 (NMe_2), 72.1 (CH_2), 118.6 (CH/C_5H_3N), 123.5 (CH/C_5H_5N), 130.4 (*i*-C/C_5H_3N), 133.6 (CH/C_5H_5N), 156.4 (CH/C_5H_5N), 163.2 (CH/C_5H_3N). Analysis calculated for $C_{16}H_{24}Cl_2N_4Ru$ (440.40): C 43.24, H 5.44, N 12.61%; found: C 43.79, H 5.78, N 12.23%.

Crystal data

$[RuCl_2(C_{11}H_{19}N_3)(C_5H_5N)]\cdot C_7H_8$
M_r = 536.50
Orthorhombic, *Pbca*
a = 12.7109 (1) Å
b = 14.1115 (2) Å
c = 27.0317 (3) Å
V = 4848.67 (10) Å^3
Z = 8
D_x = 1.470 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 31 497 reflections
θ = 1.5–38.6°
μ = 0.88 mm^{-1}
T = 173 (2) K
Block, dark red
1.20 × 1.20 × 0.90 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 2000)
T_{min} = 0.374, T_{max} = 0.451
43 809 measured reflections

9359 independent reflections
7687 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 38.6°
$h = -10 \rightarrow 20$
$k = -13 \rightarrow 20$
$l = -10 \rightarrow 43$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.117
S = 1.19
9359 reflections
400 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0208P)^2 + 13.1818P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.015
$\Delta\rho_{max}$ = 2.07 e Å^{-3}
$\Delta\rho_{min}$ = −1.86 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.00058 (9)

Table 1
Selected geometric parameters (Å, °).

Ru1—Cl1	2.3501 (7)	Ru1—N2	2.348 (3)
Ru1—Cl2	2.3580 (7)	Ru1—N3	2.251 (3)
Ru1—N1	1.962 (2)	Ru1—N4	2.096 (2)
Cl1—Ru1—Cl2	177.19 (3)	N1—Ru1—N3	88.47 (10)
Cl1—Ru1—N4	90.31 (7)	N2—Ru1—N3	160.57 (9)
Cl2—Ru1—N4	86.93 (7)	N1—Ru1—N4	178.38 (9)
N1—Ru1—N2	72.10 (9)		

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai & Huttner, 1994); software used to prepare material for publication: *SHELX*97.

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