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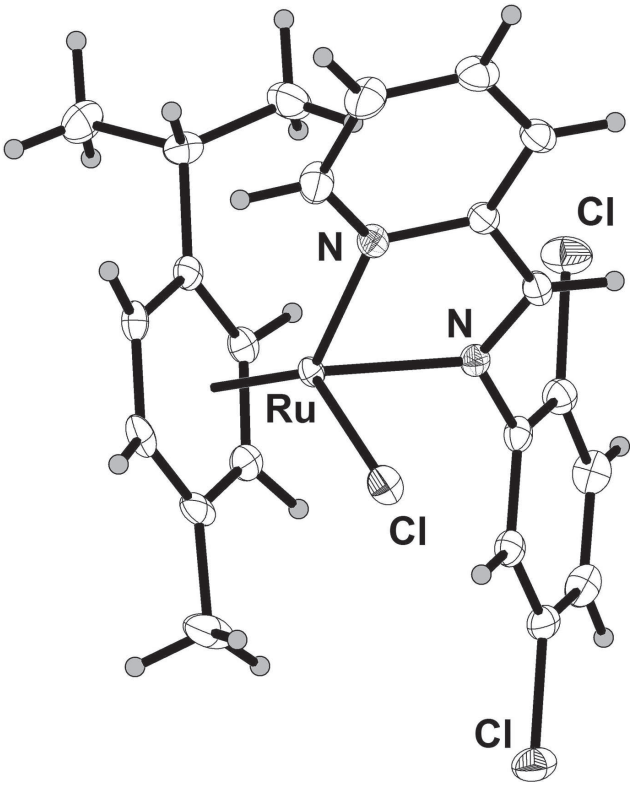
Crystal structure of (η^6-1-isopropyl-4-methyl benzene)-(*N*-(2,5-dichlorophenyl)-1-(pyridin-2-yl)methanimine-κ^2*N*,*N*′)ruthenium(II) perchlorate, $C_{22}H_{22}Cl_4N_2O_4Ru$



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Abstract

$C_{22}H_{22}Cl_4N_2O_4Ru$, monoclinic, $P2_1/c$ (no. 14), $a =$ 15.0181(3) Å, $b = 10.3983(2)$ Å, $c = 16.6496(3)$ Å, $\beta =$ 112.459(1)°, $V = 2402.84(8)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0180$, $wR_{ref}(F^2) = 0.0474$, $T = 173(2)$ K.

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The title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.26 × 0.23 × 0.22 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.13 mm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX-II, φ and ω-scans
θ_{max}, completeness:	28.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	44260, 5964, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5622
$N(param)_{refined}$:	301
Programs:	Bruker programs [1], SHELX [2], WinGX & ORTEP [3]

Source of material

To a suspension of [(η^6-*p*-cymene)Ru(μ—Cl)Cl]$_2$ (0.2 mmol) in methanol (20 mL) was added the ligand *N*-(2,5-dichlorophenyl)-1-(pyridin-2-yl)methanimine (0.42 mmol). The mixture was stirred at room temperature for 3 h followed by the reduction in volume of the solvent *in vacuo* to about 10 mL before adding $NaClO_4$ in slight excess (0.42 mmol). The mixture was then cooled in an ice bath while stirring for 2 h leading to a precipitate, which was collected by filtration. The filtrate was washed with diethyl ether and dried *in vacuo*. Crystals of the title compound suitable for single-crystal X-ray diffraction studies were grown by the liquid diffusion method where the solutions of the compounds in acetone were layered with hexane and left undisturbed for 2 days. Orange crystals, yield 82%, m.p. 210 °C (decomp.).

Experimental details

All hydrogen atoms were placed in idealized positions and refined in riding models with U_{iso} assigned the values of 1.2 times those of their parent atoms and the distances of C—H were constrained to 0.93 Å for all the aromatic H atoms, 0.96 Å for methyl hydrogens and 0.98 Å for methine hydrogen.

Discussion

Half-sandwich arene ruthenium complexes represent one of the most sought after organometallic compounds due

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
Ru1	0.73293(2)	0.97102(2)	0.06453(2)	0.01087(4)
N1	0.76030(8)	1.11564(11)	−0.00928(7)	0.0142(2)
N2	0.85109(7)	1.06443(11)	0.15235(7)	0.0128(2)
O1	0.01825(8)	0.40214(11)	0.21584(7)	0.0279(2)
O2	0.12567(8)	0.57521(12)	0.24527(7)	0.0312(3)
O3	−0.00697(8)	0.56582(12)	0.11131(7)	0.0289(2)
O4	−0.03102(10)	0.60840(14)	0.24073(9)	0.0414(3)
Cl1	0.64386(2)	1.13436(3)	0.10190(2)	0.01749(7)
Cl2	1.06256(2)	0.97839(4)	0.22049(2)	0.02525(8)
Cl3	0.79524(3)	0.96298(3)	0.42910(2)	0.02279(7)
Cl4	0.02582(2)	0.53860(3)	0.20303(2)	0.01834(7)
C1	0.70876(10)	1.13973(14)	−0.09356(9)	0.0206(3)
H1	0.655494	1.085967	−0.124436	0.025*
C2	0.73111(11)	1.24100(15)	−0.13687(9)	0.0243(3)
H2	0.694188	1.254638	−0.196875	0.029*
C3	0.80704(11)	1.32202(14)	−0.09273(9)	0.0222(3)
H3	0.823439	1.390882	−0.121991	0.027*
C4	0.85903(10)	1.30076(13)	−0.00456(9)	0.0177(3)
H4	0.910450	1.356212	0.027959	0.021*
C5	0.83410(9)	1.19684(12)	0.03476(8)	0.0140(2)
C6	0.88282(9)	1.16419(13)	0.12590(8)	0.0145(2)
H6	0.935165	1.213626	0.163901	0.017*
C7	0.89304(9)	1.02237(12)	0.24069(8)	0.0143(2)
C8	0.98773(10)	0.97846(13)	0.27821(9)	0.0170(3)
C9	1.02231(10)	0.92808(14)	0.36198(9)	0.0205(3)
H9	1.086814	0.897934	0.387400	0.025*
C10	0.96326(10)	0.92166(13)	0.40845(9)	0.0200(3)
H10	0.986514	0.886066	0.465310	0.024*
C11	0.86954(10)	0.96787(12)	0.37099(9)	0.0172(3)
C12	0.83338(10)	1.01688(12)	0.28747(9)	0.0156(2)
H12	0.768709	1.046438	0.262250	0.019*
C13	0.65143(10)	0.81852(13)	0.10166(9)	0.0181(3)
C14	0.74979(10)	0.78628(12)	0.13582(9)	0.0167(2)
H14	0.785159	0.786251	0.200331	0.020*
C15	0.80424(9)	0.78390(12)	0.08177(8)	0.0146(2)
H15	0.876134	0.780060	0.109682	0.018*
C16	0.76031(9)	0.80956(12)	−0.00819(8)	0.0143(2)
C17	0.65915(9)	0.83759(12)	−0.04329(9)	0.0161(2)
H17	0.629621	0.872971	−0.103601	0.019*
C18	0.60638(9)	0.84407(13)	0.01014(9)	0.0182(3)
H18	0.540569	0.883204	−0.013369	0.022*
C19	0.81548(10)	0.80802(14)	−0.06756(9)	0.0183(3)
H19	0.780516	0.863671	−0.118972	0.022*
C20	0.91817(11)	0.85847(16)	−0.02393(10)	0.0259(3)
H20A	0.916842	0.944551	−0.000447	0.039*
H20B	0.948291	0.862814	−0.066717	0.039*
H20C	0.955357	0.800479	0.023401	0.039*
C21	0.81590(12)	0.66978(16)	−0.09979(11)	0.0282(3)
H21A	0.848488	0.613288	−0.050074	0.042*
H21B	0.850048	0.667337	−0.139523	0.042*
H21C	0.749420	0.640480	−0.130368	0.042*
C22	0.59511(12)	0.83266(15)	0.15891(11)	0.0278(3)
H22A	0.639000	0.826735	0.219989	0.042*
H22B	0.546878	0.764039	0.145720	0.042*
H22C	0.562735	0.916411	0.148318	0.042*

to their potential applications in catalytic and biological applications [4]. Their unique properties, which include mild reaction conditions required for synthesis, high yields and, for some, a wide range of stability and solubility under aqueous conditions, have made them be of great interest in organometallic chemistry [5–8]. The coordinated arene moiety is relatively inert towards substitution and acts as spectator ligand [6]. These stabilize and protect the metal centre and thereby prevent rapid oxidation of the Ru(II) to Ru(III) [6].

The asymmetric unit of the title compound contains a Ru(II) title complex and one perchlorate anion. The title compound is similar to our previously published compounds in a series of half-sandwich ruthenium(II) complexes [7–11].

The cationic complex features the known "pseudo-octahedral three-legged piano stool" structure. In this arrangement, the ruthenium centre is coordinated to the N,N′-bidentate ligand through the N atom of the pyridine and the N atom of the imine group and a chlorido ligand at the base of the stool and the *p*-cymene ring at the apex of the stool. The Ru—N bond lengths are 2.0606(12) and 2.0804(13) Å. This value is comparable to those reported for other arene ruthenium complexes with N,N′ donor ligands [9–16]. The N—Ru—N bond angles was 76.52(6)°. The N—Ru—Cl bond angles were 84.04(4)° and 84.05(5)°. These values are close to those reported for related compounds [9–16].

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