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A *p*-cymene-ruthenium(II)–DMSO complex, $[(\eta^6\text{-}C_{10}H_{14})RuCl_2(DMSO)]$

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Key indicators

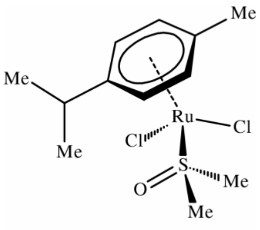
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.033
wR factor = 0.090
Data-to-parameter ratio = 17.1

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

The crystal structure of the neutral Ru^{II} complex $[RuCl_2(\eta^6\text{–}C_{10}H_{14})(C_2H_6OS)]$ is reported. The overall coordination geometry about the ruthenium centre is best described as a typical ‘piano stool’. In the *p*-cymene ligand, the six C atoms of the arene ring are in a planar configuration, as expected.

Comment

The homochiral sulfoxides have received considerable attention recently owing to their prominent role as intermediates in enantioselective synthesis and their potential use as precursors (Schenk *et al.*, 1997; Otto *et al.*, 2001). Because of our continuing interest in the area of arene ruthenium chemistry (Singh *et al.*, 2000), we reacted $[\{(\eta^6\text{–}C_{10}H_{14})RuCl_2\}_2]$ with 4-cyanophenyl imidazole, which contained dimethylsulfoxide as solvate. Surprisingly, we isolated the title complex, $[(\eta^6\text{-}C_{10}H_{14})RuCl_2(DMSO)]$, (I), instead of the expected arene–ruthenium–organonitrile complex.



(I)

The pertinent bond lengths and bond angles may be compared with the closely related 1,4,9,10-tetrahydroanthracene ruthenium complex $[(\eta^6\text{-}C_{14}H_{14})RuCl_2(DMSO)]$ (Beasley *et al.*, 1993). The title compound adopts a typical piano-stool geometry (a description commonly used for half-sandwich compounds) with a pseudo-tetrahedral arrangement of the *p*-cymene, the two chloride ligands, and the S atom of the coordinated DMSO ligand around the ruthenium metal centre. This is a similar arrangement to that of $[(\eta^6\text{-}C_{10}H_{14})RuCl_2(MePPh_2)]$, $[(\eta^6\text{-}C_{10}H_{14})RuCl_2(pz)_2]PF_6$ and the η^6-arene DMSO complex $[(\eta^6\text{-}C_{14}H_{14})RuCl_2(DMSO)]$ (Bennett *et al.*, 1972; Tocher *et al.*, 1983; Beasley *et al.*, 1993). The six-membered ring of the *p*-cymene is planar, with a maximum deviation of 0.013 (3) Å (C4) and an overall r.m.s. deviation of 0.008 Å. The C—C bond lengths within the *p*-cymene ring are similar, apart from a shortening of the C2—C3 bond. The Ru—C distances are almost equal, with an average of 2.199 (11) Å. The distance between the centroid of the *p*-cymene ring and ruthenium is 1.683 (3) Å, which is very close to that reported in other Ru^{II} arene complexes (Watkins

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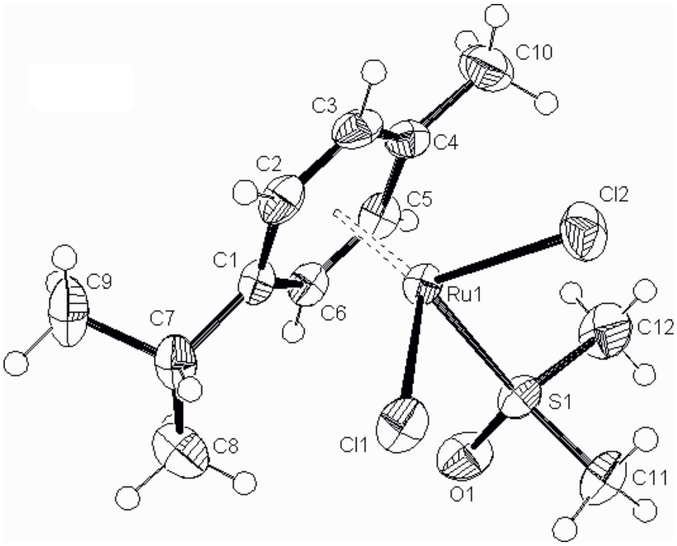

Figure 1
The structure of $[(\eta^6\text{-}C_{10}H_{14})RuCl_2(DMSO)]$ showing 50% probability displacement ellipsoids.

& Fronczek, 1982; McCormick *et al.*, 1993; Gupta *et al.*, 1998). The Ru—Cl1 and Ru—Cl2 bond lengths are similar to those in other Ru^{II} complexes (Bruce *et al.*, 1981). The DMSO ligand is bonded to ruthenium *via* the S atom, resulting in a roughly tetrahedral arrangement around sulfur. The Ru—S distance is comparable to those observed in the η^6-arene dimethyl sulfoxide complex $[(\eta^6\text{–}C_{14}H_{14})RuCl_2(DMSO)]$ (Beasley *et al.*, 1993) and the Ru^{II} complex $[RuCl_2(DMSO)_4]$ (Mercer & Trotter, 1975).

Experimental

The complex $[(\eta^6\text{-}C_{10}H_{14})RuCl_2(DMSO)]$ was prepared in a 68% yield by reaction of $[\{(\eta^6\text{-}C_{10}H_{14})RuCl_2\}_2]$ (0.612 g, 1 mmol) in dichloromethane (25 ml) with 4-cyanophenyl imidazole (0.338 g, 2 mmol) which was previously isolated from dimethyl sulfoxide and therefore contained DMSO as solvate. Orange–red crystals suitable for single-crystal analysis were obtained by slow diffusion of petroleum ether (b.p. 333–353 K) into a solution of the complex in dichloromethane.

Crystal data

$[RuCl_2(C_{10}H_{14})(C_2H_6OS)]$
$M_r = 384.31$
Triclinic, $P\overline{1}$
$a = 7.8522$ (16) Å
$b = 9.4124$ (9) Å
$c = 10.9151$ (17) Å
$\alpha = 106.850$ (11)°
$\beta = 94.395$ (15)°
$\gamma = 99.697$ (13)°
$V = 754.3$ (2) $Å^3$
$Z = 2$
$D_x = 1.692$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 5.2$–12.1°
$\mu = 1.51$ mm^{-1}
$T = 293$ (2) K
Needle, brown
0.4 × 0.1 × 0.1 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.836$, $T_{max} = 0.860$
2632 measured reflections
2632 independent reflections
2373 reflections with $I > 2\sigma(I)$
$\theta_{max} = 25.0°$
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 11$
$l = -12 \rightarrow 12$
3 standard reflections every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.090$
$S = 1.07$
2632 reflections
154 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.067P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.26$ e $Å^{-3}$
$\Delta\rho_{min} = -0.83$ e $Å^{-3}$

Table 1
Selected geometric parameters (Å, °).

Ru1—Cl1	2.4111 (10)	Ru1—C4	2.205 (4)
Ru1—Cl2	2.4045 (10)	Ru1—C5	2.192 (3)
Ru1—S1	2.3400 (10)	Ru1—C6	2.181 (3)
Ru1—C1	2.201 (3)	S1—O1	1.477 (3)
Ru1—C2	2.207 (4)	S1—C11	1.774 (4)
Ru1—C3	2.209 (3)	S1—C12	1.784 (4)
Cl1—Ru1—Cl2	88.05 (4)	C12—S1—Ru1	109.49 (16)
S1—Ru1—Cl1	86.28 (4)	O1—S1—C11	108.2 (2)
S1—Ru1—Cl2	86.74 (4)	O1—S1—C12	107.5 (2)
O1—S1—Ru1	115.71 (13)	C11—S1—C12	99.5 (2)
C11—S1—Ru1	115.07 (15)		

The highest residual peak lies 0.919 Å from Ru1. All H atoms were placed in calculated positions and refined with a riding model.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *maXus* (Mackay *et al.*, 1998); 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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