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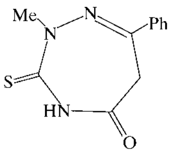
The novel title ruthenium(II) complex, $[RuCl(C_{10}H_{14})(C_{10}H_{10}N_3OS)]$, was synthesized from the reaction of 1,2,4-triazepine, a new class of bidentate ligands, with $[Ru(p\text{-cymene})Cl_2]_2$. The 1,2,4-triazepine ligand is coordinated to the metal centre through the N-4 and S atoms, forming a four-membered chelate ring. This is the first structural example of a transition metal complex containing a 1,2,4-triazepine ligand.

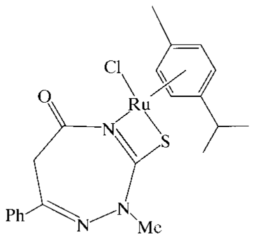
Comment

Heterocylic compounds have found widespread application in both pharmacology as active drugs (Pauwels *et al.*, 1990; Koop *et al.*, 1991) and chemistry as useful reaction intermediates in organic synthesis (Anderson & Raju, 1989; Steinmeyer & Neef, 1992). They are also good ligands which coordinate readily with transition metals to provide versatile and useful complexes (Ghosh *et al.*, 1998; Wills, 1998; Shi *et al.*, 1999). However, the preparation of transition metal complexes with 1,2,4-tiazepines as ligands is not well documented. This can probably be ascribed to the difficulty of synthesis of these heterocycles and to the triazepine nucleus instability (Ait Itto *et al.*, 1997).

As part of our research on transition metal complexes (El Firdoussi *et al.*, 1993, 1997; Abouhamza *et al.*, 1999), we report here the crystal structure of a new triazepine based ruthenium(II) complex, namely chloro(η^6-*p*-cymene)(2-methyl-5-oxo-7-phenyl-5,6-dihydro-2*H*-1,2,4-triazepine-3-thiolato-κ^2N^4,*S*)ruthenium(II), (II). This kind of complex which has a great conformational rigidity could lead to catalysts of high selectivity. The title compound was prepared by reaction in 2-propanol of the 1,2,4-triazepine (I) with $[Ru(p\text{-cymene})Cl_2]_2$ in the presence of triethylamine. Crystals were isolated and characterized by X-ray diffraction. Various complexes of the type [Ru(arene)(heteroatomic ligand)Cl] have been crystallographically characterized (Nishiyama *et al.*, 1989; Suzuki *et al.*, 1996).



(I)



(II)

Complex (II) is, to the best of our knowledge, the first structural example of a transition metal complex containing a 1,2,4-triazepine ligand. The latter acts as a bidentate ligand (through N1 and S1), forming a four-membered chelate ring. The *p*-cymene and Cl ligands complete the coordination sphere of the Ru^{II} atom.

The crystal structure has two similar molecules in its asymmetric unit pseudo-centrosymmetrically arranged, but no additional crystallographic symmetry was found (Le Page, 1987, 1988). Both molecules have the same arrangement and have very close geometries. Average distances involving ruthenium coordination: Ru—N 2.097 (8), Ru—Cl 2.404 (2), Ru—S 2.438 (3) and Ru–centroid 1.67 (1) Å. Average angles involving ruthenium coordination: N—Ru—Cl 85.9 (2), Cl—Ru—S 82.08 (9), N—Ru—S 66.8 (2), N—Ru–centroid 133.0 (3), S—Ru–centroid 135.3 (3) and Cl—Ru–centroid 128.3 (3)°.

Experimental

Complex (II) was prepared by reaction of $[Ru(p\text{-cymene})Cl_2]_2$, 1,2,4-triazepine (I) and triethyamine (molar ratio 1:4:8) in 2-propanol at 353 K. The resulting solid was filtered off and recrystallized from $CHCl_3$.

Crystal data

$[RuCl(C_{10}H_{14})(C_{10}H_{10}N_3OS)]$
$M_r = 503.01$
Monoclinic, *Cc*
$a = 20.647$ (2) Å
$b = 11.835$ (1) Å
$c = 17.275$ (2) Å
$\beta = 96.253$ (5)°
$V = 4196.2$ (7) Å^3
$Z = 8$
$D_x = 1.592$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 8671 reflections
$\theta = 4.31$–$68.16°$
$\mu = 8.281$ mm^{-1}
$T = 293$ (2) K
Prismatic, colourless
0.7 × 0.2 × 0.2 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: empirical (using ΔF; Parkin *et al.*, 1995)
$T_{min} = 0.050$, $T_{max} = 0.189$
8671 measured reflections
5007 independent reflections
4791 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 68.16°$
$h = -24 \rightarrow 23$
$k = 0 \rightarrow 14$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0718P)^2 + 9.2057P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.049$	$(\Delta/\sigma)_{\max} = 0.001$
$wR(F^2) = 0.119$	$\Delta\rho_{\max} = 0.95$ e Å^{-3}
$S = 1.033$	$\Delta\rho_{\min} = -0.80$ e Å^{-3}
5007 reflections	
584 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL* (Otwinowski & Minor, 1997); data reduction: *HKL*; program(s) used to solve structure: *DIRDIF*92 (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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