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Hydrogen bonding in thiacrown complexes: chlorobis(nicotinamide-κN)(1,4,7-trithiacyclononane-$\kappa^3 S$)ruthenium(II) hexafluorophosphate monohydrate

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The title complex, [RuClL_2([9]-ane-S_3)]PF_6·H_2O [where L is nicotinamide, $C_7H_6N_2O$) and [9]-ane-S_3 is 1,4,7-trithiacyclononane ($C_6H_{12}S_3$)], was isolated as a hexafluorophosphate salt from water–ethanol. The structure confirms that the amide moieties are available for possible mutual hydrogen-bond interactions. However, from aqueous solvents, these sites are involved in networks of interactions with water molecules.

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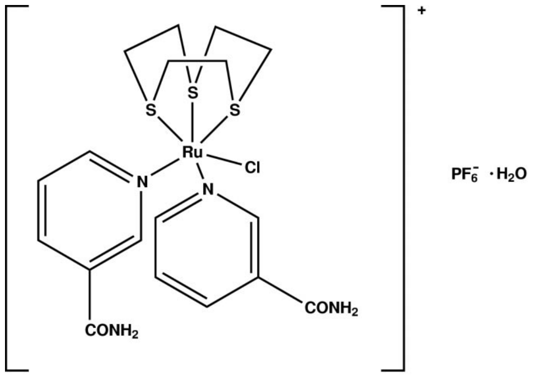
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.006 Å
R factor = 0.043
wR factor = 0.116
Data-to-parameter ratio = 17.8

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

Comment

The targeted design of long-range solid-state structures, or crystal engineering, is a fast-emerging area of chemistry. In this context, structures mediated by ligand coordination to specific metal centres has been particularly well pursued. However, comparatively less work has involved coordination complexes with coordinated ligands containing hydrogen-bonding sites. As part of a programme investigating the possible assembly of solution- and solid-phase host structures, we are synthesizing complexes based on Ru^{II} centres facially capped by thiacrown ligands. Coordination of two or three nicotinic and isonicotinic acid derivatives to these centres provides two- and three-dimensional synthons for crystal engineering. Solubility properties of the resultant complexes can be modulated by a change of counter-ion. The complexes are initially synthesized as chloride salts, but anion metathesis affords a route to a variety of other counter-ions. Using this methodology, we are exploring the effect of the counter-ion on the long-range structure of the resulting crystallographic architecture. We report here the structure of [RuClL_2([9]-ane-S_3)]PF_6 (where L is nicotinamide and [9]-ane-S_3 is 1,4,7-trithiacyclononane) grown from water–ethanol as a monohydrate, (I), in which the water molecules interact with the projecting amide groups.



(I)

Experimental

[$RuCl_2$(DMSO)([9]-ane-S_3)] (0.215 g, 0.5 mmol) and nicotinamide (0.122 g, 1.0 mmol) were heated at reflux for 4 h under a nitrogen atmosphere in 30 ml of a water–ethanol mixture (1:1). The reaction mixture was allowed to cool and any insoluble material was removed by filtration. Addition of ammonium hexafluorophosphate (0.163 g, 1.0 mmol) led to the crystallization of the final product. This was collected by filtration, washed with (3 × 10 ml) portions of water, ethanol and diethyl ether, and allowed to dry *in vacuo*. The product was obtained as a yellow powder (yield 0.178 g, 50%). Crystals suitable for X-ray crystallography were obtained via slow evaporation from the water:ethanol mother liquor.

Crystal data

[RuCl($C_6H_6N_2O$)$_2$($C_6H_{12}S_3$)](PF_6)·H_2O
M_r = 724.10
Monoclinic, $P2_1/c$
a = 12.9575 (15) Å
b = 9.8190 (11) Å
c = 20.974 (3) Å
β = 101.483 (2)°
V = 2615.1 (5) Å^3
Z = 4

D_x = 1.839 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4801 reflections
θ = 5.2–54.6°
μ = 1.08 mm^{-1}
T = 150 (2) K
Block, yellow
0.32 × 0.28 × 0.21 mm

Data collection

Bruker SMART 1000 diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.725, T_{max} = 0.806
28 430 measured reflections

5948 independent reflections
4693 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 27.6°
$h = -16 \rightarrow 16$
$k = -12 \rightarrow 12$
$l = -27 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.116
S = 1.05
5948 reflections
334 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 5.2806P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}

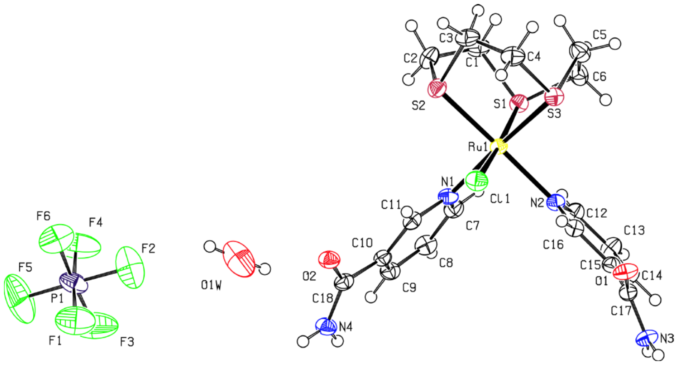


Figure 1
View of (I), showing the numbering scheme, with displacement ellipsoids drawn at the 50% probability level.

The H atoms were introduced at calculated positions and treated as riding atoms, with bond lengths of NH_2 0.88 Å, 0.95(C—H aromatic), and 0.99 Å (CH_2). The exception being the water O—H lengths which were found by low-theta difference Fourier, then restrained to 0.85 (1) Å. The thermal displacement parameters were all made equal to 1.2 times U_{eq} (parent N, O or C atom).

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

References

Bruker (1997). *SADABS, SMART, SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

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