

unit cell there is a normal octahedral Ni(3) mononuclear complex containing a methanol molecule and a water molecule forming a *cis* octahedral structure.

From the crystal data and the analytical values mentioned above the crystals obtained in this work contain one binuclear $[(\text{acac})(\text{tmen})\text{Ni}(\text{CO}_3)\text{Ni}(\text{tmen})(\text{acac})]$ and one octahedral mononuclear unit $[\text{Ni}(\text{acac})(\text{tmen})(\text{H}_2\text{O})(\text{CH}_3\text{OH})]\text{BPh}_4$ in the same unit cell. There is little reported literature of carbonate existing as a bidentate bridging ligand and of two different structural units occupying the same unit cell, although the crystal of this material was obtained quite accidentally. It is interesting to note that the mononuclear cation in the present crystal has an octahedral arrangement with BPh_4 as the counterion. Recently, we have solved the structure of an Ni^{II} complex containing tmen and acac which forms an octahedral cation with ClO_4 as counterion,

$[\text{Ni}(\text{acac})(\text{tmen})(\text{H}_2\text{O})(\text{CH}_3\text{OH})]\text{ClO}_4$, and a square-planar one with BPh_4 , $[\text{Ni}(\text{acac})(\text{tmen})]\text{BPh}_4$.

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Structure of Acridinium *trans*-Tetrachlorobis(dimethyl sulfoxide)ruthenate(III)

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Abstract. Acridinium (OC-6-11)-Tetrachlorobis(dimethyl sulfoxide)ruthenate(III), $[\text{C}_{13}\text{H}_{10}\text{N}][\text{RuCl}_4(\text{C}_2\text{H}_6\text{OS})_2]$, $M_r = 579.38$, monoclinic, $P2_1/c$, $a = 10.536(1)$, $b = 13.372(1)$, $c = 16.619(1)$ Å, $\beta = 102.25(1)^\circ$, $V = 2288.1(3)$ Å³, $Z = 4$, $D_x = 1.682$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71073$ Å, $\mu = 13.3$ cm⁻¹, $F(000) = 1164$, $T = 294$ K, $R = 0.031$, $wR = 0.038$ for 4321 unique observed reflections. The structure of the anion is characterized by a *trans* arrangement of the two sulfur-bonded dimethyl sulfoxide ligands. Average bond distances are Ru—Cl 2.349 (8) and Ru—S 2.35 (1) Å.

Introduction. In the course of a general study on halide–dimethyl sulfoxide (dmsO) ruthenium complexes (Alessio *et al.*, 1991), *mer*- $\text{RuCl}_3(\text{dmsO})_3$ was treated with acridine (acr), in an attempt to introduce this ligand into the coordination sphere of Ru^{III} . 2.27 mmol of acridine, dissolved in 5 ml of CHCl_3 , were added to a solution of 0.27 mmol of

mer- $\text{RuCl}_3(\text{dmsO})_3$ in 10 ml of CHCl_3 . After 1 h of stirring at room temperature, a light-green solution was obtained, which was filtered and concentrated to half of its original volume. After addition of a small amount of diethyl ether a yellow precipitate was obtained within 24 h at 277 K. This precipitate was filtered off, washed with cold ethanol and vacuum dried. The complex was recrystallized at room temperature from dichloromethane/diethyl ether to give reddish brown crystals (yield 60%).

X-ray analysis of the product showed that acridine did not coordinate to the metal atom and the structure was determined to be acridinium tetrachlorobis(dimethyl sulfoxide)ruthenate(III) $[(\text{acr})\text{H}][\text{Ru}(\text{dmsO})_2\text{Cl}_4]$. The overall reaction between *mer*- $\text{RuCl}_3(\text{dmsO})_3$ and acridine in chloroform is the addition of one mole of hydrogen chloride and elimination of one dmsO molecule.

The results are of interest in view of the small amount of structural data available on Ru^{III} –dmsO complexes. They provide accurate Ru^{III} –Cl bond distances comparable to those of $[(\text{dmsO})_2\text{H}][\text{Ru}(\text{dmsO})_2\text{Cl}_4]$, the first fully characterized Ru^{III} –

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chloride–dmso complex (Alessio *et al.*, 1991; Jaswal, Rettig & James, 1990). In fact, in the latter complex, the Cl atoms of one of the two crystallographically independent molecules could not be located with great accuracy because of either thermal motion (Alessio *et al.*, 1991) or statistical occupancy disorder effects (Jaswal *et al.*, 1990).

Experimental. Crystal dimensions approximately $0.3 \times 0.4 \times 0.5$ mm. Weissenberg and precession photographs indicated crystals to be monoclinic. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters refined by least-squares fit of 25 reflections in the range $14 < \theta < 18^\circ$. Intensity data measured by the $\omega/2\theta$ technique, ω -scan angle = $(0.70 + 0.35\tan\theta)^\circ$; ω -scan rate, $1-10^\circ \text{ min}^{-1}$. Three standard reflections, monitored every 83 min, showed no decay. 7129 reflections collected ($2 \leq \theta \leq 30^\circ$; $-14 \leq h \leq 14$, $0 \leq k \leq 18$, $0 \leq l \leq 23$), 4321 significant [$F_o \geq 3.0\sigma(F_o)$]; $\sigma(F_o)$ based on counting statistics. Empirical absorption correction applied to data using ψ scans of four reflections at $\theta > 15^\circ$ (correction factors in the range 0.93–1.00, average transmission factor 0.97). Lorentz and polarization corrections. Structure solved using the heavy-atom method. H atoms located at idealized positions on the basis of ideal bond lengths (C—H, N—H 0.95 Å) and angles and not included in the least-squares refinement; $B = 1.3B_{\text{eq}}$ of the corresponding bonded C or N atom. Refinement using full-matrix least-squares method with anisotropic thermal factors for non-H atoms led to $R = 0.031$, $wR = 0.038$, $S = 1.10$, $(\Delta/\sigma)_{\text{max}} = 0.48$. Function minimized: $\sum_i w_i (|F_o| - |F_c|)^2$, $w = 4F_o^2 / [\sigma^2(I) + (0.04F_o)^2]$. Maximum residual $\Delta\rho$ 0.49 (8) $e \text{ \AA}^{-3}$. Atomic scattering factors, anomalous-dispersion terms and programs as in *MolEN* (Enraf–Nonius, 1990). All computations carried out on a MicroVAX 2000 computer.

Discussion. Final fractional coordinates and equivalent isotropic thermal parameters of non-H atoms are given in Table 1* and bond lengths and angles in Table 2. The structure of the title complex is shown in Fig. 1. It consists of [(acr)H]⁺ cations hydrogen bonded to the O atom [N⋯O(1) 2.768 (3) Å; O(1)⋯H—N 160°] of one of the two dmso molecules of the [Ru(dmso)₂Cl₄][−] anion.

Bond lengths and angles of the acridinium ion are very close to those previously reported for 9-(chloromethyl)acridine hydrochloride (Zacharias & Glusker,

Table 1. Final fractional coordinates and equivalent isotropic thermal parameters (\AA^2) with their e.s.d.'s in parentheses

$$B_{\text{eq}} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}
Ru	0.15082 (2)	0.15436 (2)	0.17267 (1)	2.070 (4)
Cl(1)	0.1011 (1)	0.23113 (7)	0.04211 (5)	3.78 (2)
Cl(2)	-0.05514 (8)	0.07799 (6)	0.14131 (6)	3.28 (2)
Cl(3)	0.19305 (9)	0.07381 (6)	0.30076 (5)	3.31 (2)
Cl(4)	0.35409 (8)	0.23296 (7)	0.20007 (6)	3.74 (2)
S(1)	0.25273 (8)	0.01805 (6)	0.12248 (5)	2.58 (1)
S(2)	0.06514 (7)	0.28923 (6)	0.23289 (5)	2.45 (1)
O(1)	0.3672 (2)	-0.0241 (2)	0.1787 (2)	4.11 (6)
O(2)	-0.0679 (2)	0.2828 (2)	0.2463 (2)	4.38 (6)
N	0.4965 (3)	-0.0375 (2)	0.3418 (2)	2.86 (5)
C(1)	0.3034 (4)	0.0474 (3)	0.0302 (2)	4.63 (9)
C(2)	0.1471 (4)	-0.0837 (3)	0.0907 (3)	4.18 (8)
C(3)	0.0769 (4)	0.4011 (3)	0.1792 (3)	4.31 (9)
C(4)	0.1652 (4)	0.3193 (3)	0.3289 (2)	4.75 (9)
C(5)	0.4679 (3)	-0.1063 (2)	0.3952 (2)	2.68 (6)
C(6)	0.3513 (3)	-0.1616 (3)	0.3762 (2)	3.62 (7)
C(7)	0.3269 (4)	-0.2305 (3)	0.4312 (3)	4.50 (9)
C(8)	0.4135 (4)	-0.2452 (3)	0.5067 (3)	4.8 (1)
C(9)	0.5248 (4)	-0.1927 (3)	0.5268 (2)	4.03 (8)
C(10)	0.5571 (3)	-0.1217 (3)	0.4715 (2)	2.89 (6)
C(11)	0.6701 (3)	-0.0653 (3)	0.4874 (2)	3.08 (7)
C(12)	0.6972 (3)	0.0050 (3)	0.4323 (2)	2.82 (6)
C(13)	0.8123 (4)	0.0640 (3)	0.4458 (2)	3.89 (8)
C(14)	0.8343 (4)	0.1284 (3)	0.3879 (3)	4.43 (9)
C(15)	0.7443 (4)	0.1382 (3)	0.3130 (3)	4.68 (9)
C(16)	0.6323 (4)	0.0855 (3)	0.2969 (2)	3.91 (8)
C(17)	0.6066 (3)	0.0173 (3)	0.3565 (2)	2.86 (6)

1974), as well as to those of 9-acridineethanol (Toma, Ray, Stowell & Byrn, 1990). The cation is planar within 0.059 (7) Å and roughly parallel [dihedral angle 6.1 (3)°] to the RuCl₄ coordination plane [planar within 0.013 (2) Å]. The whole molecule approaches C_s symmetry [after interchange of O(2) and C(4)]. The approximate mirror plane passes through N, Cl(1) and the two Ru—S bonds, bisecting the C(11)—Ru—Cl(2) and Cl(3)—Ru—Cl(4) bond angles.

The hydrogen-bonding interaction between one dmso ligand and [(acr)H]⁺ causes significant distortions in the Ru anion. In fact, the S(1)—Ru—Cl(1) [92.29 (3)°] and S(1)—Ru—Cl(2) [93.09 (3)°] bond angles are wider than the S(1)—Ru—Cl(3) [87.31 (3)°] and S(1)—Ru—Cl(4) [87.10 (3)°] angles, with a resulting S(1)—Ru—S(2) bond angle of 174.82 (3)° and slightly different Ru—S bond lengths. Analogously, the Ru—Cl(1) and Ru—Cl(2) bond distances [average 2.356 (2) Å] are longer than Ru—Cl(3) and Ru—Cl(4) [average 2.3423 (4) Å]. The Ru—Cl bond distances [average 2.349 (8) Å] are close to the average value of 2.347 (8) Å, found in the well defined molecule of [(dmso)₂H]-[Ru(dmso)₂Cl₄] (Jaswal *et al.*, 1990; Alessio *et al.*, 1991) as well as in [(tmso)H][Ru(tmso)₂Cl₄] (tmso = tetramethylene sulfoxide) (Alessio *et al.*, 1990). Furthermore, they compare well to the average values of 2.347 (1), 2.344 (8) and 2.346 (6) Å found

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55572 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances (Å) and angles (°)

[RuCl ₄ (dms ₂) ₂] ⁻		[(acr)H] ⁺	
Ru—Cl(1)	2.3567 (9)	N—C(5)	1.357 (4)
Ru—Cl(2)	2.3545 (8)	N—C(17)	1.350 (4)
Ru—Cl(3)	2.3426 (8)	C(5)—C(6)	1.411 (5)
Ru—Cl(4)	2.3421 (9)	C(5)—C(10)	1.424 (4)
Ru—S(1)	2.3560 (9)	C(6)—C(7)	1.361 (7)
Ru—S(2)	2.3350 (8)	C(7)—C(8)	1.401 (6)
S(1)—O(1)	1.472 (2)	C(8)—C(9)	1.346 (6)
S(1)—C(1)	1.771 (4)	C(9)—C(10)	1.413 (5)
S(1)—C(2)	1.766 (4)	C(10)—C(11)	1.386 (5)
S(2)—O(2)	1.467 (3)	C(11)—C(12)	1.384 (5)
S(2)—C(3)	1.760 (4)	C(12)—C(13)	1.424 (5)
S(2)—C(4)	1.762 (4)	C(12)—C(17)	1.419 (4)
		C(13)—C(14)	1.348 (6)
		C(14)—C(15)	1.402 (6)
		C(15)—C(16)	1.352 (6)
		C(16)—C(17)	1.414 (5)
Cl(1)—Ru—Cl(2)	88.32 (3)	C(5)—N—C(17)	123.5 (3)
Cl(1)—Ru—Cl(3)	177.71 (3)	N—C(5)—C(6)	120.8 (3)
Cl(1)—Ru—Cl(4)	89.94 (3)	N—C(5)—C(10)	119.0 (3)
Cl(1)—Ru—S(1)	92.29 (3)	C(5)—C(6)—C(7)	119.0 (3)
Cl(1)—Ru—S(2)	91.58 (3)	C(6)—C(7)—C(8)	121.1 (4)
Cl(2)—Ru—Cl(3)	89.44 (3)	C(7)—C(8)—C(9)	121.1 (4)
Cl(2)—Ru—Cl(4)	178.26 (3)	C(8)—C(9)—C(10)	120.5 (4)
Cl(2)—Ru—S(1)	93.09 (3)	C(5)—C(10)—C(9)	118.1 (3)
Cl(2)—Ru—S(2)	90.45 (3)	C(5)—C(10)—C(11)	118.0 (3)
Cl(3)—Ru—Cl(4)	92.30 (3)	C(9)—C(10)—C(11)	123.9 (3)
Cl(3)—Ru—S(1)	87.31 (3)	C(10)—C(11)—C(12)	122.1 (3)
Cl(3)—Ru—S(2)	88.96 (3)	C(11)—C(12)—C(13)	124.1 (3)
Cl(4)—Ru—S(1)	87.10 (3)	C(11)—C(12)—C(17)	118.2 (3)
Cl(4)—Ru—S(2)	89.47 (3)	C(13)—C(12)—C(17)	117.7 (3)
S(1)—Ru—S(2)	174.82 (3)	C(12)—C(13)—C(14)	120.7 (3)
Ru—S(1)—O(1)	116.4 (1)	C(13)—C(14)—C(15)	120.6 (4)
Ru—S(1)—C(1)	112.3 (2)	C(14)—C(15)—C(16)	121.6 (4)
Ru—S(1)—C(2)	113.3 (2)	C(15)—C(16)—C(17)	119.2 (3)
O(1)—S(1)—C(1)	106.1 (2)	N—C(17)—C(16)	119.2 (3)
O(1)—S(1)—C(2)	105.9 (2)	N—C(17)—C(12)	120.5 (3)
C(1)—S(1)—C(2)	101.4 (2)	C(12)—C(17)—C(16)	120.3 (3)
Ru—S(2)—O(2)	119.2 (1)		
Ru—S(2)—C(3)	111.3 (2)		
Ru—S(2)—C(4)	110.7 (2)		
O(2)—S(2)—C(3)	107.4 (2)		
O(2)—S(2)—C(4)	106.4 (2)		
C(3)—S(2)—C(4)	99.9 (2)		

for the *trans* Cl—Ru^{III}—Cl group in *mer*-[RuCl₃(dms₂)₃] (Alessio *et al.*, 1991), *mer*-[RuCl₃(dms)₃] (dms = dimethyl sulfide) (Jaswal *et al.*, 1990) and [(im)H][*trans*-Ru(im)₂Cl₄] (im = imidazole) (Keppler, Rupp, Juhl, Endres, Niebl & Balzer, 1987), respectively. Longer values have been found in [(4-Meim)H][*trans*-Ru(4-Meim)₂Cl₄], average 2.364 (4) Å, (Keppler, Rupp *et al.*, 1987) and in [(im)H]₂[*trans*-Ru(im)Cl₅], average 2.37 (1) Å (Keppler, Wehe, Endres & Rupp, 1987). The bond lengthening in these compounds could be due to a greater electron density on the metal atom, which should weaken the Ru—Cl bonds.

Neglecting the bond distances of the last two compounds, the average Ru^{III}—Cl, *trans* to Cl, bond length is 2.347 (6) Å, lengthening to 2.35 (1) Å if all values are considered. This confirms the previous observations (Jaswal *et al.*, 1990; Alessio *et al.*, 1990, 1991), that the Ru^{III}—Cl distance is markedly shorter than the Ru^{II}—Cl distance, such as found in *trans*-[RuCl₂(dms₂)₄] [2.402 (2) Å (Alessio *et al.*,

1988), 2.432 (1) Å (Jaswal *et al.*, 1990)], *trans,cis,cis*-[RuCl₂(dms₂)₂(NH₃)₂].H₂O [2.408 (7) Å (Henn, Alessio, Mestroni, Calligaris & Attia, 1991)], *trans,trans,trans*-[RuCl₂(dms₂)₂(im)₂] [2.414 (1) Å (Keppler, 1991)] and *trans*-[RuCl₂(1,5-dtco-o)] (1,5-dtco-o = 1,5-dithiacyclooctane 1-oxide) [2.413 (4) Å (Arbuckle, Bharadwaj & Musker, 1991)].

The Ru^{II}—Cl, *trans* to Cl, bond distances average 2.413 (11) Å, giving an average shortening of 0.06 (2) Å, from Ru^{II} to Ru^{III} species. A difference of 0.09 Å is found for the Ru—Cl, *trans* to S, bond distances, comparing Ru^{III}-dms and Ru^{II}-sulfoxide complexes: *e.g.* 2.3417 (8) Å in *mer*-[RuCl₃(dms)₃] (Jaswal *et al.*, 1990), 2.427 (4) Å in *cis*-[RuCl₂(tms₂)₄] (Alessio *et al.*, 1990) and 2.428 (11) Å in different Ru^{II}-dms₂ complexes (Alessio *et al.*, 1988). The greater difference can be explained in terms of a lower *trans* influence of dms with respect to the dms₂ and tms₂ ligands.

A shortening of 0.040 (6) Å has been reported for other Ru (*t_{2g}*)⁶—(*t_{2g}*)⁵ couples and has been attributed to the electrostatic effect of the greater positive charge on Ru³⁺ atoms (Stynes & Ibers, 1971). The Ru^{III}—S, *trans* to S, bond distances of 2.3350 (8) and 2.3560 (9) Å [average 2.35 (1) Å] compare well to the average values of 2.348 (1) and 2.33 (1) Å found in the [(dms₂)₂H]⁺ and [(tms₂)H]⁺ analogues, respectively. It is interesting to observe that they do not show any appreciable reduction with respect to those found in *trans* S—Ru^{II}—S groups: *e.g.* 2.360 (1) in *trans*-[RuBr₂(dms₂)₄] (Oliver & Riley, 1984), 2.352 (2) Å (Alessio *et al.*, 1988) and 2.3534 (9) Å (Jaswal *et al.*, 1990) in *trans*-[RuCl₂(dms₂)₄] and 2.355 (6) Å in *cis*-[RuCl₂(tms₂)₄] (Alessio *et al.*, 1990).

The different trends in the Ru—Cl and Ru—S bond distances, passing from Ru^{II} to Ru^{III}, have been interpreted (Alessio *et al.*, 1990, 1991) in terms of a strong reduction of π back bonding in the Ru^{III}—S(dms₂) bonds, assumed to be present in the Ru^{II} complexes. Thus, the reduction of the bond

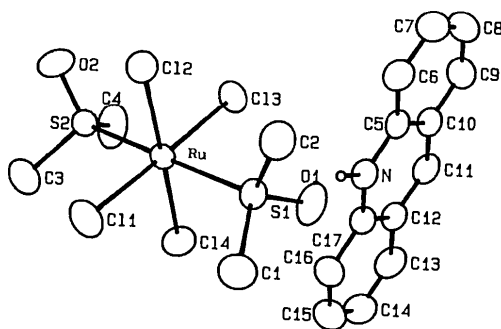


Fig. 1. ORTEP (Johnson, 1976) drawing and atom-numbering scheme for [(acr)H][*trans*-Ru(dms₂)₂Cl₄]. Only the H⁺ atom of the acridinium ion, bonded to O(1), is shown.

length, passing from the +2 to the +3 oxidation state, would be counterbalanced by the decrease in the Ru—S bond order.

On the other hand, if the Ru^{III}—S bonding has a prevailing σ character, this would induce a positive charge on the S atom and thereby would increase the $p\pi$ — $d\pi$ overlap between oxygen and sulfur orbitals, with a consequent increase in the S=O bond order (Davies, 1981) and decrease of the S—O bond lengths. In fact, the S(2)—O(2) bond length of 1.467 (3) Å, comparable to those of 1.467 (5) Å in the [(dmsO)₂H]⁺ analogue and 1.465 (4) Å in the tmso derivative, appears to be shorter than the S—O bond lengths in Ru^{II} S-bonded dmsO complexes, which average 1.48 (1) Å (Alessio *et al.*, 1988), as has already been indicated. The S(1)—O(1) bond distance of 1.472 (2) Å is slightly longer, owing to hydrogen-bonding effects, as also found in the tmso derivative [1.488 (2) Å] (Alessio *et al.*, 1990).

The strengthening of the S—O bond in Ru^{III} complexes is clearly shown by the higher ν (S=O) stretching frequencies, *e.g.* 1129 cm⁻¹ in [(dmsO)₂H]-[Ru(dmsO)₂Cl₄] versus 1089 cm⁻¹ in *trans*-[RuCl₂(dmsO)₄] (Alessio *et al.*, 1988).

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Structure of Dichloro(2-pyrimidinol- κ N,O)mercury(II)

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Abstract. [HgCl₂(C₄H₄N₂O)], $M_r = 367.6$, monoclinic, $P2_1/c$, $a = 10.506$ (3), $b = 4.187$ (1), $c = 17.337$ (12) Å, $\beta = 92.97$ (7)°, $V = 761.6$ (6) Å³, $Z = 4$, $D_x = 3.206$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 20.848$ mm⁻¹, $F(000) = 656$, $T = 295$ K, final $R = 0.059$ for 1704 unique observed reflections. The Hg atom is coordinated by the two Cl and one N atom and the carbonyl O atom of 2-pyrimidinol, forming a four-membered chelate ring. A hydrogen bond N—H...O and the weak interaction of Cl atoms with neighbouring Hg atoms results in a ladder-like structure along the b axis.

Introduction. Metal complexes of pyrimidine derivatives have, for a long time, been widely studied, especially pyrimidines that play an important role in biochemistry (Beauchamp & Simard, 1984). Surprisingly enough, we have found very few references to 2-pyrimidinol (Goodgame & Jeeves, 1979), despite the fact that it is one of the simplest of the family, the only crystal structure report being of a copper(II) complex (Cartwright, Reynolds & Skapski, 1977). As part of our work on the coordination chemistry of pyrimidines (Romero, Salas, Simard, Quirós & Beauchamp, 1990), we report here the synthesis and crystal structure of the compound obtained by the reaction of mercury oxide with 2-pyrimidinol hydrochloride.

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