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**Structure of  $\mu$ -Carbonato- $1\kappa^2O^1, O^2:2\kappa^2O^1, O^3$ -bis[(acetylacetonato)-  
(*N,N,N',N'*-tetramethylethylenediamine)nickel(II)] (Acetylacetonato)aqua-  
(methanol)(*N,N,N',N'*-tetramethylethylenediamine)nickel(II) Tetraphenylborate**

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**Abstract.**  $[\text{Ni}_2(\text{C}_5\text{H}_7\text{O}_2)_2(\text{CO}_3)(\text{C}_6\text{H}_{16}\text{N}_2)_2][\text{Ni}(\text{C}_5\text{H}_7\text{O}_2)(\text{C}_6\text{H}_{16}\text{N}_2)(\text{CH}_4\text{O})(\text{H}_2\text{O})](\text{C}_{24}\text{H}_{20}\text{B})$ ,  $M_r = 1251.32$ , triclinic,  $P\bar{1}$ ,  $a = 16.965$  (9),  $b = 18.847$  (8),  $c = 12.108$  (7) Å,  $\alpha = 97.41$  (6),  $\beta = 95.29$  (6),  $\gamma = 117.83$  (3)°,  $V = 3344$  (3) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.243$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 8.94$  cm<sup>-1</sup>,  $F(000) = 1336$ ,  $T = 295$  K,  $R = 0.058$ ,  $wR = 0.066$  for 11 335 independent reflections. This crystal contains a  $\mu$ -carbonato-bridged nickel(II) binuclear complex moiety, a mononuclear octahedral nickel(II) complex cation coordinated with a water and a methanol molecule and a tetraphenylborate moiety as counterion.

**Introduction.** Continuing our work on mixed-ligand complexes containing *N*-alkylated ethylenediamine [*N,N,N',N'*-tetramethylethylenediamine (tmen)] and  $\beta$ -diketonate ligands [acetylacetonate (acac)], we obtained bluish green lozenge-shaped crystals of the title complex from a methanol solution. Its crystal structure has been determined, by a single-crystal X-ray analysis, to be made up of [(acac)(tmen)Ni(CO<sub>3</sub>)Ni(tmen)(acac)] and [Ni(acac)(tmen)(H<sub>2</sub>O)-

(CH<sub>3</sub>OH)]BPh<sub>4</sub>. This structure is consistent with the results of elemental analysis: H: 7.45 (7.65), C: 56.90 (56.63), N: 6.86 (6.72)%; the values in parentheses are calculated for C<sub>59</sub>H<sub>95</sub>BN<sub>6</sub>Ni<sub>3</sub>O<sub>11</sub>. It is noteworthy that (a) it is a novel example of a binuclear complex which contains one carbonate acting as a bridging bidentate ligand and (b) in the same unit cell, there are two kinds of complex, binuclear and mononuclear.

**Experimental.** Synthesis: [Ni(acac)(tmen)]BPh<sub>4</sub> was dissolved in a mixture of acetonitrile and methanol, and to this solution oxamide and potassium hydroxide dissolved in water were added to form a bluish green solution. After drying, bluish green crystals were obtained, which were recrystallized from methanol. Approximate crystal dimensions 0.3 × 0.3 × 0.3 mm. Rigaku AFC-5R off-centered diffractometer with graphite-monochromatized Mo *K*α radiation. Cell parameters refined by least-squares method on the basis of 17 independent 2θ values, 20 < 2θ < 25°. Three-dimensional intensity data collected up to 2θ = 50°; range of *hkl*, -20 to 20, -22 to 22 and 0 to 14. 2θ-ω scan technique with scan widths of Δω = (1.0 + 0.35tanθ)°, scan speed 4° min<sup>-1</sup>(2θ). Three

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standard reflections were monitored every 50 reflections and no significant change in intensity was observed. 12 392 intensities measured, 11 335 with  $|F_o| > 3\sigma(|F_o|)$  considered observed and used for the structure determination. Corrections made for Lorentz and polarization effects but not for absorption. Reflection data for 110,  $\bar{1}20$ ,  $\bar{1}11$  and  $1\bar{1}1$  were omitted at the final stage of refinement, it being considered that these were influenced by extinction effects. The structure was solved by direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined by full-matrix least squares in three blocks with *SHELX76* (Sheldrick, 1976). The quantity minimized was  $\sum w(|F_o| - |F_c|)^2$ , where  $w = [\sigma(|F_o|)^2 + 0.004|F_o|^2]^{-1}$ . The refinements were made with anisotropic thermal parameters for all the non-H atoms. H atoms found in the difference Fourier maps and calculated geometrically (C—H, 1.0 Å) were refined isotropically. In the course of refinement, an additional peak of about  $1 \text{ e } \text{Å}^{-3}$  was found near the C(18) atom, forming a five-membered ring having appropriate geometry with the C(19), N(11), N(12) and Ni atoms. This suggests that the C(18) atom is slightly disordered. However, the minor occupancy was estimated to be so small that the disorder was ignored in order to avoid the complexity of the structure. Max.  $\Delta/\sigma$  and max.  $\Delta\rho$  (except for the peak mentioned above) in the final refinement were 0.076 and  $0.49 \text{ e } \text{Å}^{-3}$ . Final *R* and *wR* were 0.058 and 0.066, respectively. *S* was 0.993; number of refined parameters 1075. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations were carried out on an IBM 4381-R24 computer at the Information Processing Center of Ochanomizu University.

**Discussion.** Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.\* The crystal structure of [Ni<sub>2</sub>(acac)<sub>2</sub>(tmen)<sub>2</sub>(CO<sub>3</sub>)] [Ni(acac)(tmen)(H<sub>2</sub>O)(CH<sub>3</sub>OH)]BPh<sub>4</sub> is shown in Fig. 1. This structure is composed of a uncharged binuclear complex (I), a mononuclear cationic complex (II) and tetraphenylborate as counter ion. *ORTEP* (Johnson, 1965) drawings of (I) and (II) with numbering of atoms are shown in Fig. 2, and selected bond distances and angles are listed in Table 2 for the binuclear moiety and Table 3 for the mononuclear complex cation.

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and all bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55566 (39 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1009]

Table 1. *Final atomic coordinates* ( $\times 10^5$  for Ni, and  $\times 10^4$  for B, C, N and O) and *equivalent isotropic thermal parameters*

$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	<i>B</i> <sub>eq</sub> (Å <sup>2</sup> )
Ni(1)	61383 (2)	84231 (2)	56929 (3)	3.7
Ni(2)	64839 (2)	74936 (2)	85928 (3)	3.5
O(1)	6350 (1)	8015 (1)	7185 (2)	3.6
O(2)	5392 (1)	7763 (1)	8383 (2)	3.8
O(3)	5253 (1)	8313 (1)	6878 (2)	4.5
C(1)	5650 (2)	8035 (2)	7491 (3)	3.6
O(11)	5338 (2)	7229 (1)	4993 (2)	5.3
O(12)	5603 (2)	8733 (2)	4418 (2)	5.4
O(21)	7299 (1)	8630 (1)	9530 (2)	4.5
O(22)	7560 (1)	7369 (1)	8321 (2)	4.7
N(11)	7017 (2)	9703 (2)	6402 (2)	4.5
N(12)	7267 (2)	8498 (2)	4984 (2)	4.3
N(21)	5652 (2)	6243 (2)	7692 (2)	4.3
N(22)	6253 (2)	6963 (2)	10054 (2)	4.5
C(11)	4700 (3)	6937 (2)	4158 (3)	5.7
C(12)	4104 (4)	6044 (3)	3928 (5)	10.1
C(13)	4521 (3)	7409 (3)	3479 (4)	6.4
C(14)	4966 (2)	8253 (3)	3611 (3)	5.3
C(15)	4678 (4)	8648 (4)	2804 (5)	8.8
C(16)	7386 (5)	9887 (3)	7591 (4)	11.3
C(17)	6587 (4)	10193 (3)	6278 (7)	11.7
C(18)	7696 (3)	9932 (3)	5670 (6)	9.5
C(19)	8004 (3)	9349 (3)	5403 (4)	6.3
C(20)	7076 (3)	8340 (4)	3742 (4)	8.1
C(21)	7526 (3)	7918 (3)	5367 (4)	7.1
C(31)	8133 (2)	9057 (2)	9476 (3)	4.6
C(32)	8592 (3)	9935 (3)	10092 (4)	7.0
C(33)	8647 (2)	8767 (2)	8934 (3)	5.3
C(34)	8358 (2)	7957 (2)	8444 (3)	4.8
C(35)	9033 (3)	7735 (3)	7988 (4)	7.2
C(36)	4682 (3)	5998 (2)	7367 (4)	5.8
C(37)	5982 (3)	6078 (2)	6653 (4)	6.0
C(38)	5780 (3)	5778 (2)	8517 (4)	5.7
C(39)	5638 (3)	6075 (2)	9661 (4)	6.0
C(40)	7115 (3)	7105 (3)	10693 (4)	6.3
C(41)	5828 (3)	7322 (3)	10778 (3)	6.4
Ni(3)	71547 (2)	21230 (2)	19839 (3)	3.8
O(1 <sup>w</sup> )	6089 (1)	2233 (1)	1031 (2)	4.4
O(Me)	6277 (2)	1689 (2)	3140 (2)	5.5
C(Me)	6275 (3)	1248 (3)	3982 (4)	6.5
O(31)	7990 (1)	1804 (2)	2783 (2)	5.0
O(32)	6504 (2)	1017 (1)	950 (2)	5.3
N(31)	7806 (2)	3319 (2)	2971 (3)	5.2
N(32)	8064 (2)	2665 (2)	840 (3)	5.7
C(51)	7907 (2)	1098 (2)	2634 (3)	5.2
C(52)	8635 (3)	1035 (3)	3390 (4)	7.4
C(53)	7238 (3)	418 (2)	1881 (4)	6.0
C(54)	6592 (3)	403 (2)	1088 (4)	5.6
C(55)	5927 (3)	-393 (3)	284 (4)	7.7
C(56)	8183 (4)	3369 (3)	4134 (4)	9.2
C(57)	7188 (3)	3667 (3)	3045 (4)	7.3
C(58)	8553 (4)	3806 (3)	2407 (6)	9.9
C(59)	8497 (6)	3533 (4)	1333 (7)	15.7
C(60)	7577 (4)	2498 (5)	-320 (5)	12.0
C(61)	8676 (4)	2342 (4)	711 (6)	11.8
B	751 (2)	3366 (2)	7431 (3)	4.2
C(71)	580 (2)	2412 (2)	7267 (3)	4.4
C(72)	615 (3)	2027 (2)	8140 (4)	5.8
C(73)	458 (3)	1208 (3)	7980 (4)	6.7
C(74)	269 (3)	773 (2)	6904 (5)	6.5
C(75)	222 (3)	1139 (2)	6003 (4)	5.8
C(76)	380 (2)	1932 (2)	6189 (3)	5.3
C(77)	-222 (2)	3362 (2)	7453 (3)	4.5
C(78)	-1052 (2)	2710 (2)	6901 (3)	5.4
C(79)	-1843 (3)	2771 (3)	6828 (4)	6.8
C(80)	-1828 (3)	3478 (4)	7262 (5)	7.5
C(81)	-1024 (3)	4125 (3)	7872 (6)	8.3
C(82)	-242 (3)	4064 (2)	7944 (5)	6.9
C(83)	1161 (2)	3741 (2)	6330 (3)	4.7
C(84)	745 (3)	4006 (2)	5578 (3)	5.9
C(85)	1106 (4)	4292 (3)	4619 (4)	7.8
C(86)	1891 (4)	4305 (3)	4407 (4)	8.2
C(87)	2336 (3)	4064 (3)	5141 (4)	7.3
C(88)	1971 (3)	3769 (2)	6078 (4)	6.0
C(89)	1430 (2)	3939 (2)	8624 (3)	4.4
C(90)	2293 (2)	4606 (2)	8681 (4)	5.4
C(91)	2821 (3)	5102 (3)	9720 (5)	7.7
C(92)	2518 (4)	4941 (3)	10707 (4)	7.6
C(93)	1678 (4)	4291 (3)	10687 (4)	7.4
C(94)	1156 (3)	3811 (3)	9663 (4)	6.4

Table 2. Selected bond distances (Å) and angles (°) for  $[\text{Ni}_2(\text{acac})_2(\text{tmen})_2(\text{CO}_3)]$ 

Ni(1)—O(1)	2.118 (2)	Ni(2)—O(21)	2.026 (2)
Ni(1)—O(3)	2.131 (3)	Ni(2)—O(22)	1.997 (3)
Ni(1)—O(11)	2.018 (3)	Ni(2)—N(21)	2.164 (3)
Ni(1)—O(12)	2.004 (3)	Ni(2)—N(22)	2.122 (3)
Ni(1)—N(11)	2.156 (3)	O(1)—C(1)	1.292 (4)
Ni(1)—N(12)	2.123 (3)	O(2)—C(1)	1.281 (4)
Ni(2)—O(1)	2.119 (2)	O(3)—C(1)	1.272 (4)
Ni(2)—O(2)	2.139 (2)		
O(1)—Ni(1)—O(3)	62.2 (1)	O(1)—Ni(2)—N(22)	163.2 (1)
O(1)—Ni(1)—O(11)	86.0 (1)	O(2)—Ni(2)—O(21)	90.2 (1)
O(1)—Ni(1)—O(12)	163.8 (1)	O(2)—Ni(2)—O(22)	161.5 (1)
O(1)—Ni(1)—N(11)	94.9 (1)	O(2)—Ni(2)—N(21)	92.9 (1)
O(1)—Ni(1)—N(12)	99.1 (1)	O(2)—Ni(2)—N(22)	100.9 (1)
O(3)—Ni(1)—O(11)	89.5 (1)	O(21)—Ni(2)—O(22)	90.7 (1)
O(3)—Ni(1)—O(12)	102.1 (1)	O(21)—Ni(2)—N(21)	175.5 (1)
O(3)—Ni(1)—N(11)	92.2 (1)	O(21)—Ni(2)—N(22)	92.2 (1)
O(3)—Ni(1)—N(12)	160.9 (1)	O(22)—Ni(2)—N(21)	87.4 (1)
O(11)—Ni(1)—O(12)	90.1 (1)	O(22)—Ni(2)—N(22)	97.5 (1)
O(11)—Ni(1)—N(11)	178.3 (1)	N(21)—Ni(2)—N(22)	84.0 (1)
O(11)—Ni(1)—N(12)	93.7 (1)	Ni(1)—O(1)—Ni(2)	174.7 (1)
O(12)—Ni(1)—N(11)	89.4 (1)	Ni(1)—O(1)—C(1)	89.2 (2)
O(12)—Ni(1)—N(12)	96.8 (1)	Ni(2)—O(1)—C(1)	90.2 (2)
N(11)—Ni(1)—N(12)	84.7 (1)	Ni(2)—O(2)—C(1)	89.5 (2)
O(1)—Ni(2)—O(2)	62.4 (1)	Ni(1)—O(3)—C(1)	89.8 (2)
O(1)—Ni(2)—O(21)	89.8 (1)	O(1)—C(1)—O(2)	117.9 (3)
O(1)—Ni(2)—O(22)	99.2 (1)	O(1)—C(1)—O(3)	117.9 (3)
O(1)—Ni(2)—N(21)	94.5 (1)	O(2)—C(1)—O(3)	124.2 (3)

Table 3. Selected bond distances (Å) and angles (°) for  $[\text{Ni}(\text{acac})(\text{tmen})(\text{H}_2\text{O})(\text{CH}_3\text{OH})]^+$ 

Ni(3)—O(W)	2.160 (3)
Ni(3)—O(Me)	2.094 (3)
Ni(3)—O(31)	1.999 (3)
Ni(3)—O(32)	2.014 (3)
Ni(3)—N(31)	2.114 (3)
Ni(3)—N(32)	2.151 (4)
O(W)—Ni(3)—O(Me)	86.5 (1)
O(W)—Ni(3)—O(31)	169.7 (1)
O(W)—Ni(3)—O(32)	80.0 (1)
O(W)—Ni(3)—N(31)	96.2 (1)
O(W)—Ni(3)—N(32)	91.8 (1)
O(Me)—Ni(3)—O(31)	89.7 (1)
O(Me)—Ni(3)—O(32)	90.8 (1)
O(Me)—Ni(3)—N(31)	91.1 (1)
O(Me)—Ni(3)—N(32)	175.3 (1)
O(31)—Ni(3)—O(32)	90.4 (1)
O(31)—Ni(3)—N(31)	93.5 (1)
O(31)—Ni(3)—N(32)	92.7 (1)
O(32)—Ni(3)—N(31)	175.7 (1)
O(32)—Ni(3)—N(32)	93.2 (1)
N(31)—Ni(3)—N(32)	84.7 (1)

Both of the nickel(II) ions of (I) are coordinated with two tmen N atoms, two acac O atoms and two carbonate O atoms to give octahedral geometry. The bond lengths are 2.123 (3)–2.164 (3) Å (Ni—N for Ni—tmen) and 1.997 (3)–2.026 (2) Å (Ni—O for Ni—acac) and are similar to those of  $[\text{Ni}(\text{acac})(\text{tmen})(\text{H}_2\text{O})(\text{CH}_3\text{OH})]\text{ClO}_4$ : Ni—N 2.157 (6), 2.131 (6) Å and Ni—O 1.989 (5), 2.032 (4) Å (Yamada, Hori & Fukuda, 1992). Bond lengths of C—O in the bridging carbonate are 1.272 (4)–1.292 (4) Å, indicating that the three C—O bonds are equivalent. The Ni(1)—O(1)—Ni(2) bond angle formed between the two Ni atoms and the bridging carbonate O atom is 174.7 (1)° in this case. A similar bond angle of 174.6 (5)° between two metal ions and the bridging carbonate group has been reported (Mikuriya, Murase, Asato & Kida, 1989; Murase,

Vuckovic, Kodera, Harada, Matsumoto & Kida, 1991). On the other hand, because of the small bite angle of this carbonate anion acting as a four-membered bidentate chelate ring to the Ni<sup>II</sup> ions, the angles O(1)—Ni(1)—O(3) and O(1)—Ni(2)—O(2) are 62.2 (1) and 62.4 (1)°, respectively, which are smaller than the corresponding bond angles [O(21)—Ni(1)—O(22), O(23)—Ni(2)—O(24)] formed with the acetylacetonate ligand. Consequently, the geometries of the Ni<sup>II</sup> moieties in this binuclear unit are distorted octahedral. On the other hand, in the same

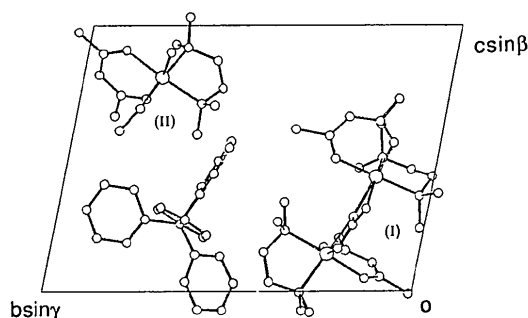
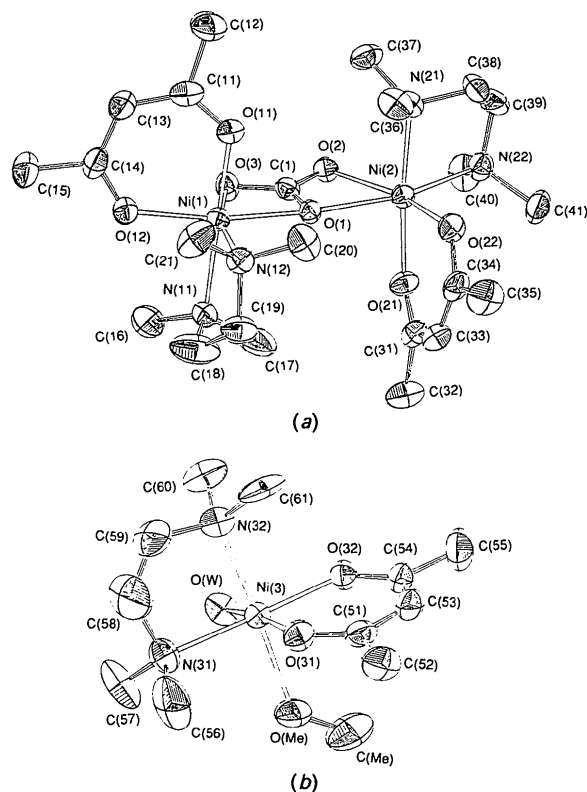
Fig. 1. Crystal structure viewed along the *a* axis.

Fig. 2. ORTEP drawing (a) of the complex (I) and (b) of (II). The thermal ellipsoids are drawn at the 50% probability level.

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 [(acac)(tmen)Ni(CO<sub>3</sub>)Ni(tmen)(acac)] and one octahedral mononuclear unit [Ni(acac)(tmen)(H<sub>2</sub>O)(CH<sub>3</sub>OH)]BPh<sub>4</sub> 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<sub>4</sub> as the counterion. Recently, we have solved the structure of an Ni<sup>II</sup> complex containing tmen and acac which forms an octahedral cation with ClO<sub>4</sub> as counterion,

[Ni(acac)(tmen)(H<sub>2</sub>O)(CH<sub>3</sub>OH)]ClO<sub>4</sub>, and a square-planar one with BPh<sub>4</sub>, [Ni(acac)(tmen)]BPh<sub>4</sub>.

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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), [C<sub>13</sub>H<sub>10</sub>N][RuCl<sub>4</sub>(C<sub>2</sub>H<sub>6</sub>OS)<sub>2</sub>], *M<sub>r</sub>* = 579.38, monoclinic, *P*2<sub>1</sub>/*c*, *a* = 10.536 (1), *b* = 13.372 (1), *c* = 16.619 (1) Å, β = 102.25 (1)°, *V* = 2288.1 (3) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.682 g cm<sup>-3</sup>, λ(Mo Kα) = 0.71073 Å, μ = 13.3 cm<sup>-1</sup>, *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*-RuCl<sub>3</sub>(dmsO)<sub>3</sub> was treated with acridine (acr), in an attempt to introduce this ligand into the coordination sphere of Ru<sup>III</sup>. 2.27 mmol of acridine, dissolved in 5 ml of CHCl<sub>3</sub>, were added to a solution of 0.27 mmol of

*mer*-RuCl<sub>3</sub>(dmsO)<sub>3</sub> in 10 ml of CHCl<sub>3</sub>. 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) [(acr)H][Ru(dmsO)<sub>2</sub>Cl<sub>4</sub>]. The overall reaction between *mer*-RuCl<sub>3</sub>(dmsO)<sub>3</sub> 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<sup>III</sup>-dmsO complexes. They provide accurate Ru<sup>III</sup>-Cl bond distances comparable to those of [(dmsO)<sub>2</sub>H][Ru(dmsO)<sub>2</sub>Cl<sub>4</sub>], the first fully characterized Ru<sup>III</sup>-

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