

## Refinement

Refinement on  $F^2$  $R = 0.038$  $wR = 0.048$  $S = 1.47$ 

3017 reflections

245 parameters

H atoms riding: see below

 $w = 1/[\sigma^2(F) + 0.001F^2]$  $(\Delta/\sigma)_{\max} = 0.003$  $\Delta\rho_{\max} = 0.31 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$ 

Extinction correc-

tion:  $F^* = F[1 + 0.002\chi F^2/\sin(2\theta)]^{-1/4}$ 

Extinction coefficient:

 $\chi = 0.0007(4)$ 

Scattering factors from

*SHELXTL-Plus* (Sheldrick, 1991)

Absolute configuration: none

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Cu1—O1	1.948 (2)	N1—C7	1.501 (4)
Cu1—O2	1.943 (2)	N1—C8	1.474 (5)
Cu1—N1	2.046 (3)	N2—C10	1.479 (6)
Cu1—N2	2.000 (3)	N2—C11	1.492 (4)
O1—Cu1 <sup>i</sup>	2.291 (2)	O3—O2 <sup>ii</sup>	2.857 (5)
O2—C17	1.343 (4)	O3—N1 <sup>iii</sup>	3.016 (4)
O1—Cu1—O2	88.5 (1)	O2—Cu1—N2	93.9 (1)
O1—Cu1—N1	92.9 (1)	N1—Cu1—N2	87.3 (1)
O2—Cu1—N1	154.7 (1)	Cu1—O1—Cu1 <sup>i</sup>	96.4 (1)
O1—Cu1—N2	173.9 (1)		

Symmetry codes: (i)  $-x, 1-y, -z$ ; (ii)  $x, y, 1+z$ ; (iii)  $-x, 1-y, 1-z$ .

The data collection nominally covered over a full sphere of reciprocal space, by a combination of three sets of exposures; each set had a different  $\varphi$  angle for the crystal and each exposure covered  $0.3^\circ$  in  $\omega$ . The crystal-to-detector distance was 5.89 cm. Coverage of the unique set is over 86% complete to at least  $25.6^\circ$  in  $\theta$ . Crystal decay was monitored by remeasuring the first frame at the end of the data collection, and found to be negligible. H atoms were placed geometrically and refined with a riding model and with  $U_{\text{iso}}$  constrained to be  $0.08 \text{ \AA}^2$ .

Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1436). Services for accessing these data are described at the back of the journal.

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*trans*-[RuCl<sub>2</sub>(dmf)<sub>4</sub>]CF<sub>3</sub>SO<sub>3</sub>.CH<sub>2</sub>Cl<sub>2</sub>

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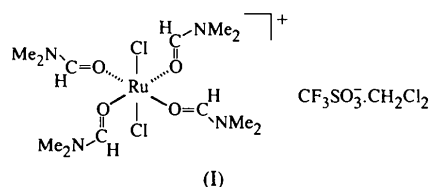
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## Abstract

The asymmetric unit of the structure of the title complex, *trans*-dichlorotetrakis(*N,N*-dimethylformamide-*O*)-ruthenium(III) trifluoromethylsulfonate dichloromethane solvate, [RuCl<sub>2</sub>(C<sub>3</sub>H<sub>7</sub>NO)<sub>4</sub>]CF<sub>3</sub>SO<sub>3</sub>.CH<sub>2</sub>Cl<sub>2</sub>, contains an ordered [RuCl<sub>2</sub>(dmf)<sub>4</sub>]<sup>+</sup> cation, a CF<sub>3</sub>SO<sub>3</sub><sup>−</sup> anion and a CH<sub>2</sub>Cl<sub>2</sub> solvent molecule. Within the cation, the chlorine ligands adopt a mutually *trans* arrangement, with four O-bonded dmf molecules ligated equatorially to the Ru<sup>III</sup> centre [Ru—Cl 2.3464(16) and 2.3376(15), and Ru—O 2.043(4), 2.045(4), 2.049(4) and 2.055(4) Å], giving a distorted octahedral stereochemistry overall.

## Comment

The X-ray structure (Fig. 1) of the title compound, (I), shows a distorted octahedral arrangement around the central Ru<sup>III</sup> ion, with two mutually *trans* chlorine ligands and four essentially planar O-bonded dmf ligands; Ru—Cl 2.3376(15) and 2.3464(16), and Ru—



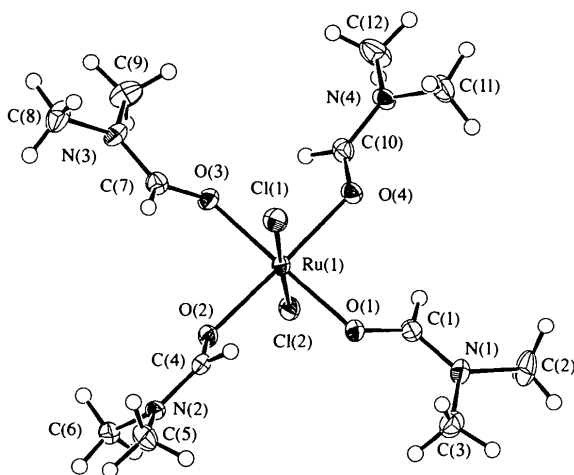


Fig. 1. View of the structure of *trans*-[RuCl<sub>2</sub>(dmf)<sub>4</sub>]<sup>+</sup> with the numbering scheme adopted. Anisotropic displacement parameters are shown at the 40% probability level.

O 2.043 (4)–2.055 (4) Å. These bond distances compare well with those reported for [Ru(dmfa)<sub>6</sub>]<sup>3+</sup> [*d*(Ru—O) 2.01 (1)–2.03 (1) Å] and [Ru(dmfa)<sub>6</sub>]<sup>2+</sup> [*d*(Ru—O) 2.079 (2)–2.098 (2) Å] (Judd *et al.*, 1995).

The Ru—O—C—N fragments all adopt a *trans* configuration. The angles subtended at the Ru<sup>III</sup> atom lie in the ranges 87.99 (17)–91.72 (12) and 178.20 (6)–178.88 (15)°, and thus do not deviate significantly from the 90 and 180° expected for a regular octahedron. Also, the C—O and C—N bond distances involved in the amide functions are very similar to those reported for the few other structurally characterized examples of dmf complexes (Deppisch, Gladrow & Kummer, 1984; Holt, Alcock, Sumner & Asplund, 1979).

The IR spectrum of [RuCl<sub>2</sub>(dmf)<sub>4</sub>]CF<sub>3</sub>SO<sub>3</sub> shows a low frequency shift in  $\nu(\text{CO})$  from 1673 cm<sup>-1</sup> in free dimethylformamide to 1637 cm<sup>-1</sup> in the title compound. This compares with 1635 cm<sup>-1</sup> for [Ru(dmfa)<sub>6</sub>]<sup>2+</sup> and 1642 cm<sup>-1</sup> for [Ru(dmfa)<sub>6</sub>]<sup>3+</sup> (Judd *et al.*, 1995).

## Experimental

The title compound was prepared by reaction of [Ru(dmfa)<sub>6</sub>](CF<sub>3</sub>SO<sub>3</sub>)<sub>3</sub> (Judd *et al.*, 1995) with two molar equivalents of LiCl in refluxing EtOH for ~2.5 h. Concentration of the solution *in vacuo* and careful addition of diethyl ether gave the product as a yellow solid which was filtered and dried. The selected crystal was coated with mineral oil, mounted on a glass fibre and immediately placed in a stream of cold N<sub>2</sub> gas.

### Crystal data

[RuCl<sub>2</sub>(C<sub>3</sub>H<sub>7</sub>NO)<sub>4</sub>]CF<sub>3</sub>O<sub>3</sub>S·  
CH<sub>2</sub>Cl<sub>2</sub>  
*M<sub>r</sub>* = 698.35  
Triclinic  
*P*1̄  
Mo *K*α radiation  
λ = 0.71073 Å  
Cell parameters from 23  
reflections  
θ = 15.35–18.15°

*a* = 12.050 (7) Å  
*b* = 14.901 (10) Å  
*c* = 8.363 (7) Å  
α = 103.60 (6)°  
β = 99.13 (6)°  
γ = 74.71 (5)°  
*V* = 1400 (2) Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 1.656 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

μ = 1.074 mm<sup>-1</sup>  
*T* = 150.0 K  
Block  
0.30 × 0.22 × 0.18 mm  
Yellow

### Data collection

Rigaku AFC-7S diffractometer  
ω/2θ scans  
Absorption correction:  
ψ scans (North, Phillips  
& Mathews, 1968)  
*T<sub>min</sub>* = 0.757, *T<sub>max</sub>* = 0.824  
5186 measured reflections  
4932 independent reflections

3827 reflections with  
*I* > 3σ(*I*)  
*R<sub>int</sub>* = 0.028  
θ<sub>max</sub> = 25.06°  
*h* = 0 → 14  
*k* = -17 → 17  
*l* = -9 → 9  
3 standard reflections  
every 150 reflections  
intensity decay: none

### Refinement

Refinement on *F*  
*R* = 0.045  
*wR* = 0.053  
*S* = 2.787  
3827 reflections  
307 parameters  
H atoms not refined  
*w* = 1/[σ<sup>2</sup>(*F*)]

(Δ/σ)<sub>max</sub> = 0.0130  
Δρ<sub>max</sub> = 0.62 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = -0.89 e Å<sup>-3</sup>  
Extinction correction: none  
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. Selected geometric parameters (Å, °)

Ru(1)—Cl(1)	2.3464 (16)	N(1)—C(2)	1.456 (7)
Ru(1)—Cl(2)	2.3376 (15)	N(1)—C(3)	1.468 (8)
Ru(1)—O(1)	2.055 (4)	N(2)—C(4)	1.310 (7)
Ru(1)—O(2)	2.049 (4)	N(2)—C(5)	1.450 (7)
Ru(1)—O(3)	2.043 (4)	N(2)—C(6)	1.457 (7)
Ru(1)—O(4)	2.045 (4)	N(3)—C(7)	1.305 (7)
O(1)—C(1)	1.266 (7)	N(3)—C(8)	1.462 (8)
O(2)—C(4)	1.261 (7)	N(3)—C(9)	1.451 (8)
O(3)—C(7)	1.273 (7)	N(4)—C(10)	1.309 (7)
O(4)—C(10)	1.266 (7)	N(4)—C(11)	1.446 (8)
N(1)—C(1)	1.306 (7)	N(4)—C(12)	1.455 (8)
Cl(1)—Ru(1)—Cl(2)	178.20 (6)	Cl(2)—Ru(1)—O(3)	89.59 (12)
Cl(1)—Ru(1)—O(1)	90.03 (17)	Cl(2)—Ru(1)—O(4)	90.24 (12)
Cl(1)—Ru(1)—O(2)	91.72 (12)	O(1)—Ru(1)—O(2)	91.17 (15)
Cl(1)—Ru(1)—O(3)	90.73 (12)	O(1)—Ru(1)—O(4)	89.91 (15)
Cl(1)—Ru(1)—O(4)	87.99 (17)	O(2)—Ru(1)—O(3)	89.95 (15)
Cl(2)—Ru(1)—O(1)	89.61 (12)	O(3)—Ru(1)—O(4)	88.97 (15)
Cl(2)—Ru(1)—O(2)	90.05 (12)		

Data collection used a Rigaku AFC-7S four-circle diffractometer equipped with an Oxford Cryosystems low-temperature attachment. The temperature for data collection was 150 K. As there were no identifiable faces, the data were corrected for absorption using ψ scans. The structure was solved by direct methods using *SIR92* (Altomare *et al.*, 1994) and developed using iterative cycles of full-matrix least squares and difference Fourier syntheses (*TEXSAN*; Molecular Structure Corporation, 1994). This showed an ordered [RuCl<sub>2</sub>(dmf)<sub>4</sub>]<sup>+</sup> cation and CF<sub>3</sub>SO<sub>3</sub><sup>-</sup> anion in the asymmetric unit. During refinement, a fully occupied CH<sub>2</sub>Cl<sub>2</sub> solvent molecule was also

identified within the asymmetric unit. All non-H atoms were refined anisotropically, and H atoms were included in fixed calculated positions with  $d(\text{C—H})$  0.96 Å and  $U_{\text{iso}}(\text{H}) = U_{\text{iso}}(\text{C})$  of the parent C atom.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *SIR92*. Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1100). Services for accessing these data are described at the back of the journal.

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## (8-Aminoquinoline-*N,N'*)dichlorodimethyltin(IV)

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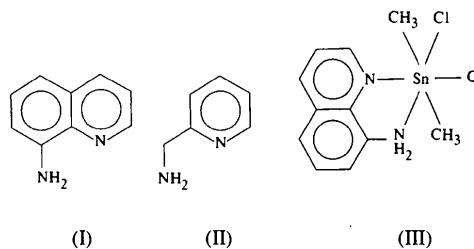
## Abstract

The title compound, [SnCl<sub>2</sub>(CH<sub>3</sub>)<sub>2</sub>(C<sub>9</sub>H<sub>8</sub>N<sub>2</sub>)], has been prepared from dimethyltin dichloride and 8-aminoquinoline. The coordination of the Sn atom is octahedral,

with the Cl atoms *trans* to the N atoms of 8-aminoquinoline, with bond distances Sn—Cl 2.547 (3) and 2.619 (3), Sn—N<sub>sp<sup>3</sup></sub> 2.322 (7), Sn—N<sub>sp<sup>2</sup></sub> 2.357 (8), and Sn—C 2.115 (10) and 2.122 (10) Å. Both amino protons are intermolecularly hydrogen bonded to Cl atoms of neighbouring molecules.

## Comment

Extensive studies have been carried out on the 1:1 adducts of diorganotin dichloride with symmetric (*e.g.* 1,10-phenanthroline) and asymmetric *N,N'*-chelating agents (*e.g.* 2-aminomethylpyridine) owing to a potential link between antitumour activity, the Cl—Sn—Cl angle and the Sn—N bond distance (Crowe, Smith & Atassi, 1984; Saxena & Huber, 1989). However, there have been no structural reports on diorganotin dihalide adducts of 2-aminomethylpyridine even though some of these adducts were reported to possess antitumour activity (Crowe *et al.*, 1984). We have studied the reactions of 8-aminoquinoline, (I), which is an asymmetric *N,N'*-bidentate ligand closely resembling 2-aminomethylpyridine, (II).



An X-ray structural determination of diphenyldichloro(8-aminoquinoline)tin(IV) revealed that the Cl atoms occupied the two axial positions of the six-coordinated Sn atom (Bengtson *et al.*, 1996). In a continuation of our attempts to prepare compounds displaying antitumour activity, we now report the synthesis and crystal structure of the dimethylchloro(8-aminoquinoline)tin(IV) adduct, (III). IR measurements show two medium intensity bands (567 and 494 cm<sup>-1</sup>) arising from the Sn—CH<sub>3</sub> stretching vibrations, suggesting that the two methyl groups attached to the Sn atom are *trans* to each other (Petrosyan, Yashina & Reutov, 1976). Furthermore, calculations using equations (1) and (3) of Lockhart & Manders (1986), based on the <sup>2</sup>J<sub>Sn—CH<sub>3</sub></sub> coupling constant value (113.4 Hz) obtained from the proton spectrum of the title adduct give a Me—Sn—Me angle of 166.8°, while the <sup>1</sup>J<sub>Sn—C</sub> coupling constant of 987.0 Hz gives an angle of 163.3°. An X-ray structure analysis confirmed the proposed *trans* configuration but found a C—Sn—C angle closer to 180°. The Sn—Cl distances are longer than those found for diphenyldichloro(8-aminoquinoline)tin(IV), 2.502 (1) and 2.518 (1) Å. This is to be expected (Hall & Tiekink, 1996) because dimethyltin dichloride is a weaker Lewis