

Orthorhombic *cis*-dichlorotetrakis(dimethyl sulfoxide)ruthenium(II) at 120 KRadhey S. Srivastava<sup>a</sup> and  
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## Key indicators

Single-crystal X-ray study

T = 120 K

Mean  $\sigma(S-C) = 0.004 \text{ \AA}$ 

R factor = 0.046

wR factor = 0.088

Data-to-parameter ratio = 37.4

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

Refinement of the structure of the *Pccn* polymorph of the title compound,  $[\text{RuCl}_2(\text{C}_2\text{H}_6\text{OS})_4]$ , using low-temperature data, has led to an increase in precision by a factor of 2–3 over the previous room-temperature results [Attia & Calligaris (1987). *Acta Cryst.* **C43**, 1426–1427] and those for the *P2<sub>1</sub>/n* polymorph [Mercer & Trotter (1975). *J. Chem. Soc. Dalton Trans.* pp. 2480–2483; Alessio *et al.* (1988) *Inorg. Chem.* **27**, 4099–4106]. The chloro ligands are *cis*, with Ru–Cl distances of 2.4165 (8) and 2.4352 (8) Å, and the dimethyl sulfoxide (DMSO) ligands *trans* to them are S-bonded, with Ru–S distances of 2.2717 (8) and 2.2734 (9) Å. One of the two mutually *trans* DMSO ligands is O-bonded, and the Ru–S distance *trans* to it is shortened, at 2.2480 (9) Å.

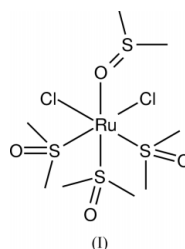
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## Comment

During the course of our studies of ruthenium complexes containing coordinated dimethyl sulfoxide and tetramethylene sulfoxide, *mer*- $\text{RuCl}_3(\text{DMSO})_3$  was treated with 1,2-bis(diphenylphosphino)ethane (dppe) in the presence of excess DMSO in an attempt to introduce the dppe ligand onto the metal center. The resulting orange crystals were determined by crystal structure analysis to be *cis*- $\text{RuCl}_2(\text{DMSO})_4$ , (I), for which both monoclinic (Mercer & Trotter, 1975; Alessio *et al.*, 1988) and orthorhombic (Attia & Calligaris, 1987) polymorphs have been previously reported at room temperature.



The sample reported here is identical to the orthorhombic polymorph, and refinement with 120 K data has led to an increase in precision by a factor of 2.5–3.0, with excellent agreement. These results are also approximately twice as precise as the room-temperature results for the monoclinic (*P2<sub>1</sub>/n*) polymorph. Of the four DMSO ligands, three are S-coordinated, including both *trans* to Cl. As reported by Mercer & Trotter (1975), the Ru–S distance *trans* to the O-bonded DMSO is significantly shortened, being more than 0.02 Å shorter than the other two. The S–O distance in the O-bonded DMSO ligand is indicative of less double-bond character than the other three, being about 0.05 Å longer.

Alessio *et al.* (1991) have reported the structures of *trans*-RuCl<sub>4</sub>(DMSO)<sub>2</sub>, which has S-bonded DMSO ligands, and also *mer*-RuCl<sub>3</sub>(DMSO)<sub>3</sub>, which has one O-bonded DMSO. However, in that case, the O-bonded DMSO is *trans* to Cl rather than to S-bonded DMSO. That paper also reports a scheme for the reduction of *mer*-RuCl<sub>3</sub>(DMSO)<sub>3</sub> to the title compound.

## Experimental

To 0.264 g (0.597 mmol) of *mer*-RuCl<sub>3</sub>(DMSO)<sub>3</sub> dissolved in chloroform (10 ml) and 2 ml of DMSO were added 0.10 g (0.25 mmol) of solid dppe. The mixture was stirred for 24 h, yielding a brown solution. The solution was concentrated to one third of its volume on a rotary evaporator and was slowly evaporated at room temperature to yield orange crystals. The product was filtered, washed with diethyl ether and then vacuum dried (70% yield).

### Crystal data

[RuCl <sub>2</sub> (C <sub>2</sub> H <sub>6</sub> OS) <sub>4</sub> ]	Mo K $\alpha$ radiation
$M_r = 484.48$	Cell parameters from 6991 reflections
Orthorhombic, <i>Pccn</i>	$\theta = 2.5\text{--}33.1^\circ$
$a = 28.167(7) \text{ \AA}$	$\mu = 1.66 \text{ mm}^{-1}$
$b = 10.817(2) \text{ \AA}$	$T = 120 \text{ K}$
$c = 11.648(2) \text{ \AA}$	Fragment, orange
$V = 3548.9(13) \text{ \AA}^3$	$0.20 \times 0.17 \times 0.12 \text{ mm}$
$Z = 8$	
$D_x = 1.813 \text{ Mg m}^{-3}$	

### Data collection

KappaCCD diffractometer (with Oxford Cryostream cooler)	6724 independent reflections
$\omega$ scans with $\kappa$ offsets	3830 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (HKL SCALEPACK; Otwinowski & Minor, 1997)	$R_{\text{int}} = 0.085$
$T_{\text{min}} = 0.748$ , $T_{\text{max}} = 0.819$	$\theta_{\text{max}} = 33.2^\circ$
25051 measured reflections	$h = -43 \rightarrow 43$
	$k = -16 \rightarrow 16$
	$l = -17 \rightarrow 17$

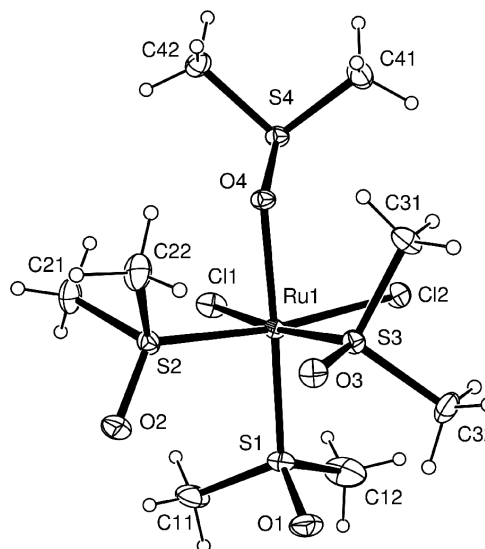
### Refinement

Refinement on $F^2$	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.046$	$w = 1/[\sigma^2(F_o^2) + (0.0256P)^2]$
$wR(F^2) = 0.088$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.95$	$(\Delta/\sigma)_{\text{max}} = 0.001$
6724 reflections	$\Delta\rho_{\text{max}} = 0.86 \text{ e \AA}^{-3}$
180 parameters	$\Delta\rho_{\text{min}} = -0.95 \text{ e \AA}^{-3}$

**Table 1**

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

Ru1—O4	2.140 (2)	Ru1—Cl2	2.4352 (8)
Ru1—S1	2.2480 (9)	S1—O1	1.475 (2)
Ru1—S2	2.2734 (9)	S2—O2	1.480 (3)
Ru1—S3	2.2717 (8)	S3—O3	1.488 (2)
Ru1—Cl1	2.4165 (8)	S4—O4	1.536 (2)
O4—Ru1—S1	177.96 (6)	S3—Ru1—Cl2	87.29 (3)
S3—Ru1—S2	93.90 (3)	Cl1—Ru1—Cl2	86.28 (3)
S2—Ru1—Cl1	92.01 (3)	S4—O4—Ru1	122.64 (12)
S3—Ru1—S1—O1	16.53 (12)	S1—Ru1—S3—O3	−81.48 (12)
S1—Ru1—S2—O2	17.16 (12)	C41—S4—O4—Ru1	−119.66 (17)



**Figure 1**

The title compound, showing the atom-numbering scheme, with displacement ellipsoids drawn at the 50% probability level.

H atoms were placed in idealized positions, with C—H bond distances of 0.98  $\text{\AA}$ , utilizing difference maps in the expected toruses, and thereafter treated as riding, with a torsional parameter refined for each methyl group. Displacement parameters for H were assigned as  $U_{\text{iso}} = 1.5U_{\text{eq}}$  of the attached atom.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; method used to solve structure: coordinates of Attia & Calligaris (1987); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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