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Tetrachloropentakis(dimethyl sulfoxide)diruthenium(II)

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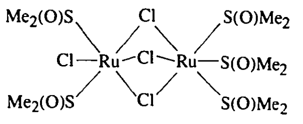
Abstract

Tri-μ-chloro-chloropentakis(dimethyl sulfoxide)diruthenium(II), $[Ru_2Cl\{(CH_3)_2SO\}_5(\mu\text{-}Cl)_3]$ has a face-sharing bioctahedral structure with the common face constructed by three bridging Cl atoms. The metal atoms are separated by 3.245 (2) Å.

Comment

The chemistry of halogen–dimethyl sulfoxide–ruthenium(II) complexes is of considerable current interest because of their antitumour activities (Allesio *et al.*, 1988, 1991). A number of such complexes of Ru^{II} and Ru^{III}, including the title complex, have been reported, but all the structurally characterized species are limited to mononuclear complexes only (Allesio *et al.*, 1991; Davies, Einstein, Farell, James & McMillan, 1978).

Herein we report the first crystal structure of a binuclear complex of this category, namely $[Ru_2Cl(Me_2SO)_5(\mu\text{-}Cl)_3]$, (I), whose ^1H NMR and IR spectra are known (Heath, Lindsay & Stephenson, 1982; Hudali, Kingston & Tayim, 1979).



(I)

All the Me_2SO ligands are bonded terminally to the metal through sulfur. The two metal atoms are bridged by three Cl atoms, leading to a face-sharing bioctahedral structure. The bridging atoms have a staggered conformation with respect to the terminal donor atoms, which in turn are mutually eclipsed when viewed down the Ru(1)–Ru(2) axis. The average Ru—Cl(bridging) bond length [2.461 (3) Å] is longer than the Ru—Cl(terminal) bond length [2.394 (4) Å]. The average Ru(1)—S bond length [2.233 (3) Å] is shorter than the average Ru(2)—S bond length [2.275 (3) Å], indicating greater π back bonding in the former which possibly arises from greater σ donation from the terminal Cl atom.

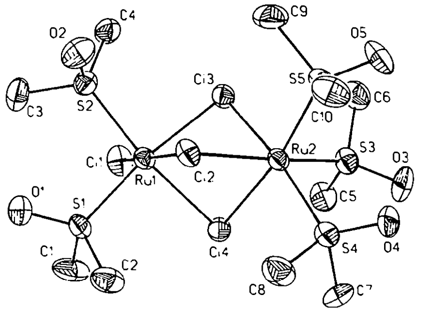


Fig. 1. Molecular structure showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.

Experimental

Synthesis was performed by literature method (Heath, Lindsay & Stephenson, 1982). Single crystals were grown by diffusion of a dichloromethane solution of the complex into *n*-hexane.

Crystal data

$[Ru_2Cl_4(C_2H_6OS)_5]$
$M_r = 734.6$
Monoclinic
$P2_1/n$
$a = 11.095\ (7)$ Å
$b = 15.692\ (8)$ Å
$c = 15.173\ (7)$ Å
$\beta = 109.83\ (4)°$
$V = 2485\ (2)$ Å^3
$Z = 4$
$D_x = 1.963$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 35 reflections
$\theta = 8\text{–}16°$
$\mu = 2.084$ mm^{-1}
$T = 295$ K
Parallelepiped
0.33 × 0.24 × 0.18 mm
Red

Data collection

Siemens *R3m/V* diffractometer
ω scans
Absorption correction: empirical
$T_{min} = 0.89$, $T_{max} = 1.00$
5045 measured reflections
4278 independent reflections
3016 observed reflections $[F > 6\sigma(F)]$

$R_{int} = 0.0430$
$\theta_{max} = 25°$
$h = -13 \rightarrow 12$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 18$
2 standard reflections monitored every 98 reflections intensity decay: 3%

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Refinement

Refinement on F
$R = 0.0548$
$wR = 0.0631$
$S = 2.08$
3016 reflections
235 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F) + 0.0001F^2]$
$(\Delta/\sigma)_{\max} = 0.005$
$\Delta\rho_{\max} = 0.72$ e Å^{-3}
$\Delta\rho_{\min} = -0.79$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1990)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$U_{\rm eq}$
Ru(1)	1.1315 (1)	0.1159 (1)	0.3002 (1)	0.028 (1)
Ru(2)	0.8836 (1)	−0.0082 (1)	0.2250 (1)	0.027 (1)
Cl(1)	1.2465 (3)	0.1669 (2)	0.2029 (2)	0.047 (1)
Cl(2)	1.0001 (2)	0.0506 (2)	0.3805 (2)	0.032 (1)
Cl(3)	1.1010 (2)	−0.0256 (2)	0.2215 (2)	0.034 (1)
Cl(4)	0.9191 (2)	0.1376 (2)	0.1774 (2)	0.037 (1)
S(1)	1.1268 (3)	0.2441 (2)	0.3630 (2)	0.035 (1)
S(2)	1.3171 (2)	0.0757 (2)	0.4064 (2)	0.035 (1)
S(3)	0.7985 (3)	−0.0597 (2)	0.0768 (2)	0.036 (1)
S(4)	0.6848 (2)	0.0261 (2)	0.2261 (2)	0.036 (1)
S(5)	0.8849 (3)	−0.1382 (2)	0.2920 (2)	0.034 (1)
O(1)	1.2232 (8)	0.2675 (5)	0.4535 (6)	0.058 (3)
O(2)	1.3137 (8)	0.0351 (6)	0.4927 (5)	0.050 (3)
O(3)	0.6631 (7)	−0.0845 (6)	0.0427 (6)	0.054 (3)
O(4)	0.5945 (7)	−0.0428 (5)	0.2260 (6)	0.048 (3)
O(5)	0.8319 (9)	−0.2122 (5)	0.2319 (5)	0.051 (3)
C(1)	1.1260 (14)	0.3264 (8)	0.2825 (11)	0.060 (6)
C(2)	0.9776 (12)	0.2634 (8)	0.3756 (9)	0.048 (5)
C(3)	1.4380 (11)	0.1551 (9)	0.4377 (9)	0.052 (5)
C(4)	1.3958 (11)	0.0011 (8)	0.3554 (8)	0.047 (4)
C(5)	0.8183 (13)	0.0145 (8)	−0.0061 (8)	0.049 (4)
C(6)	0.8880 (13)	−0.1479 (8)	0.0556 (9)	0.051 (5)
C(7)	0.6027 (11)	0.0946 (8)	0.1328 (10)	0.055 (5)
C(8)	0.6925 (11)	0.0935 (7)	0.3220 (9)	0.048 (5)
C(9)	1.0454 (12)	−0.1670 (8)	0.3611 (10)	0.056 (5)
C(10)	0.8147 (13)	−0.1362 (7)	0.3811 (8)	0.049 (5)

Table 2. *Selected geometric parameters* (Å, °)

Ru(1)—Cl(1)	2.394 (4)	Ru(2)—Cl(2)	2.454 (3)
Ru(1)—Cl(2)	2.421 (3)	Ru(2)—Cl(3)	2.446 (3)
Ru(1)—Cl(3)	2.490 (3)	Ru(2)—Cl(4)	2.470 (3)
Ru(1)—Cl(4)	2.483 (3)	Ru(2)—S(3)	2.271 (3)
Ru(1)—S(1)	2.234 (3)	Ru(2)—S(4)	2.276 (3)
Ru(1)—S(2)	2.232 (3)	Ru(2)—S(5)	2.277 (3)
Cl(1)—Ru(1)—Cl(2)	171.9 (1)	Cl(2)—Ru(2)—Cl(3)	82.0 (1)
Cl(1)—Ru(1)—Cl(3)	90.9 (1)	Cl(2)—Ru(2)—Cl(4)	81.3 (1)
Cl(1)—Ru(1)—Cl(4)	93.5 (1)	Cl(2)—Ru(2)—S(3)	172.8 (1)
Cl(1)—Ru(1)—S(1)	93.2 (1)	Cl(2)—Ru(2)—S(4)	95.7 (1)
Cl(1)—Ru(1)—S(2)	89.2 (1)	Cl(2)—Ru(2)—S(5)	89.2 (1)
Cl(2)—Ru(1)—Cl(3)	81.8 (1)	Cl(3)—Ru(2)—Cl(4)	80.9 (1)
Cl(2)—Ru(1)—Cl(4)	81.7 (1)	Cl(3)—Ru(2)—S(3)	91.2 (1)
Cl(2)—Ru(1)—S(1)	93.5 (1)	Cl(3)—Ru(2)—S(4)	172.7 (1)
Cl(2)—Ru(1)—S(2)	94.7 (1)	Cl(3)—Ru(2)—S(5)	93.0 (1)
Cl(3)—Ru(1)—Cl(4)	79.8 (1)	Cl(4)—Ru(2)—S(3)	95.4 (1)
Cl(3)—Ru(1)—S(1)	171.4 (1)	Cl(4)—Ru(2)—S(4)	92.0 (1)
Cl(3)—Ru(1)—S(2)	92.1 (1)	Cl(4)—Ru(2)—S(5)	169.3 (1)
Cl(4)—Ru(1)—S(1)	92.5 (1)	S(3)—Ru(2)—S(4)	90.7 (1)
Cl(4)—Ru(1)—S(2)	171.5 (1)	S(3)—Ru(2)—S(5)	93.5 (1)
S(1)—Ru(1)—S(2)	95.5 (1)	S(4)—Ru(2)—S(5)	93.9 (1)

H atoms were fixed at ideal positions with fixed isotropic displacement parameters ($U_{\rm iso}$ = 0.08 Å^2). Structure solution and refinement used *SHELXTL-Plus* (Sheldrick, 1990) on a MicroVAX II computer.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1006). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(2,3,7,8,12,13,17,18-Octaethylporphinato)-perchloratomanganese(III)

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Abstract

The crystal structure of [Mn(OEP)(OClO$_3$)] (where OEP = $C_{36}H_{44}N_4$) has been determined. The axial Mn—O bond length is 2.183 (2) Å and the equatorial Mn—N$_p$ bond lengths have an average value of 2.000 (5) Å. The molecules form weak dimers in the solid state with inter-ring distances of 3.49 Å and a lateral shift of 3.27 Å. The compound is found to be isomorphous with the iron derivative [Fe(OEP)(OClO$_3$)]. A brief comparison of the structural parameters for the two molecules is given.

Comment

The structure determination of the title compound, (I), was undertaken to identify the reaction intermediate