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mer-Trichlorotris(dimethyl sulfide)ruthenium(III)

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Key indicators

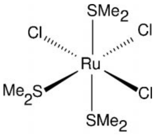
Single-crystal X-ray study
T = 120 K
Mean σ(S–C) = 0.003 Å
R factor = 0.029
wR factor = 0.064
Data-to-parameter ratio = 27.2

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

The title compound, *mer*-[$RuCl_3(C_2H_6S)_3$], contains discrete molecules with six-coordinate Ru atoms [Ru—Cl 2.3389 (10)–2.3562 (8) Å and Ru—S 2.3801 (8)–2.3879 (10) Å]. The structure is a polymorph of the form previously reported by Jaswal *et al.* (1990). [*Can. J. Chem.* **68**, 1808–1817].

Comment

The title compound, (I), was prepared as a precursor to studies of ruthenium catalysts. It has been prepared previously by two routes. The first was the direct reaction of $RuCl_3{\cdot}3H_2O$ with Me_2S in ethanol (Chatt *et al.*, 1971) and the second (unexpectedly) from $RuCl_3{\cdot}3H_2O$ and Me_2SO in boiling concentrated HCl (Jaswal *et al.*, 1990). The latter study also determined the crystal structure and confirmed the *mer* geometrical isomer proposed from earlier IR studies.



(I)

We used the preparation of Chatt *et al.* (1971) and measured the cell dimensions to confirm the identity of the product. The unit cell did not agree with that reported by Jaswal *et al.* (1990) and hence the structure was determined. The structure consists of discrete pseudo-octahedral molecules in the *mer* conformation (see Fig. 1 and Table 1) and is a polymorph of the form reported earlier (Jaswal *et al.*, 1990).

The structure could not be solved in the space group *Pnam* and a satisfactory solution was obtained in the non-centrosymmetric space group $Pna2_1$. The bond lengths agree well with the earlier study, the average Ru—Cl and Ru—S distances being essentially identical. The geometry of the

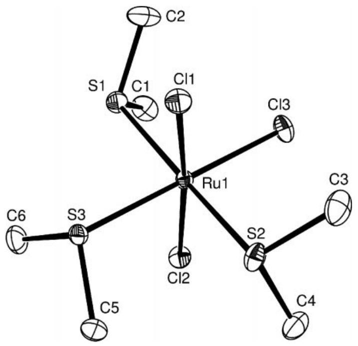


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

coordinated dimethylsulfide ligand is likewise the same. The only point of note is the hint of a *trans* influence among the Ru—Cl and Ru—S distances. The longest bond to Cl is Ru1—Cl3 (by 0.011 Å), and Ru1—S3 is the shortest S distance (by 0.007 Å) with Cl3 and S3 in a *trans* relationship (Fig. 1). This is consistent with S > Cl in *trans* influence to Ru^{III}.

Experimental

$\text{RuCl}_3\cdot3\text{H}_2\text{O}$ (0.20 g, 0.76 mmol) was dissolved in a solution of ethanol (12.5 ml) and concentrated hydrochloric acid (1.25 ml). To this was added an excess of Me_2S (0.7 ml, 9.5 mmol) and the mixture was heated to reflux under nitrogen for 1 h and allowed to cool to room temperature. Partial removal of solvent followed by cooling to 263 K afforded the first batch of crystals, which were washed with hexane; this was repeated to obtain further product. The crystal used in the structure determination was obtained from the preparation.

Crystal data

$[\text{RuCl}_3(\text{C}_2\text{H}_6\text{S})_3]$
$M_r = 393.80$
Orthorhombic, $Pna2_1$
$a = 16.391$ (2) Å
$b = 8.540$ (1) Å
$c = 10.482$ (1) Å
$V = 1467.3$ (3) Å^3
$Z = 4$
$D_x = 1.783$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5262 reflections
$\theta = 2.9$–27.5°
$\mu = 2.00$ mm^{-1}
$T = 120$ (2) K
Block, red
0.18 × 0.18 × 0.10 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{\min} = 0.629$, $T_{\max} = 0.818$
11276 measured reflections
3263 independent reflections
2912 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.058$
$\theta_{\max} = 27.5°$
$h = -21 \rightarrow 21$
$k = -11 \rightarrow 10$
$l = -12 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.064$
$S = 1.04$
3263 reflections
120 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0171P)^2 + 0.0078P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.79$ e Å^{-3}
$\Delta\rho_{\min} = -0.65$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0045 (3)
Absolute structure: Flack (1983), 1504 Friedel pairs
Flack parameter = −0.04 (3)

Table 1
Selected geometric parameters (Å, °).

Ru1—Cl1	2.3389 (10)	Ru1—S2	2.3876 (11)
Ru1—Cl2	2.3451 (9)	Ru1—S3	2.3801 (8)
Ru1—Cl3	2.3562 (8)	S1—C1	1.796 (3)
Ru1—S1	2.3879 (10)	S1—C2	1.791 (4)
Cl1—Ru1—Cl2	173.19 (3)	S3—Ru1—S1	90.10 (3)
Cl1—Ru1—Cl3	94.72 (3)	S2—Ru1—S1	174.22 (4)
Cl2—Ru1—Cl3	92.03 (3)	C2—S1—C1	99.11 (17)
Cl3—Ru1—S3	179.37 (3)	C4—S2—C3	99.71 (17)
S3—Ru1—S2	88.48 (3)	C5—S3—C6	97.90 (17)

H atoms were placed in calculated positions (C—H = 0.98 Å) with a common refined isotropic displacement parameter. The largest peak in the difference electron-density map was 0.9 Å from Ru1.

Data collection: *COLLECT* (Hooft, 1998) and *DENZO* (Otwinowski & Minor, 1997); cell refinement: *COLLECT* and *DENZO*; data reduction: *COLLECT* and *DENZO*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976).

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