SHORT-FORMAT PAPERS

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Acta Cryst. (1990). C**46**, 889–890

Structure of *cis*-Tetraamminebis(hydrogensulfito)ruthenium(II)

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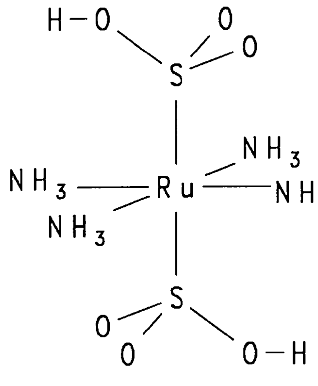
Abstract. $[Ru(HSO_3)_2(NH_3)_4]$, $M_r = 331{\cdot}3$, monoclinic, $P2_1/c$, $a = 6{\cdot}2019\,(15)$, $b = 7{\cdot}0213\,(11)$, $c = 11{\cdot}761\,(2)$ Å, $\beta = 115{\cdot}30\,(2)^\circ$, $V = 463{\cdot}0\,(3)$ Å^3, $Z = 2$, $D_x = 2{\cdot}38$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 21{\cdot}0$ cm^{-1}, $F(000) = 332$, $T = 293$ K, $R = 0{\cdot}019$ for 1179 reflections with $F_o^2 > 3\sigma(F_o^2)$. The complex lies on an inversion center with Ru–N distances of 2·141 (2) Å and a Ru–S distance of 2·2785 (5) Å. All of the H atoms are linked to O atoms of neighboring hydrogensulfite ligands in an extensive network of H bonds.

Experimental. The title complex (I) was obtained by the procedure described by Vogt, Katz & Wiberley (1965). Colorless crystals were obtained by slow cooling of the reaction mixture. Data crystal was 0·12 × 0·20 × 0·32 mm and was mounted with epoxy on a glass fiber. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans of 4–16° min^{-1} in θ. Unit cell determined from least-squares analysis of angle data for 25 reflections with $17 < 2\theta < 22^\circ$. Analytical absorption correction varied from 0·79 to 1·00. Data collected to $(\sin\theta)/\lambda = 0{\cdot}68$ Å^{-1}, $0 \leq h \leq 8$, $0 \leq k \leq 9$, $-16 \leq l \leq 16$. Three standard reflections ($\bar{3}00$; $0\bar{2}4$; $2\bar{1}1$) indicated crystal decomposition of less than 1·1% over 13·5 h of data collection. 1563 reflections measured, 1342 unique ($R_{\text{int}} = 0{\cdot}02$), 162 reflections with $I < 3\sigma(I)$ where $\sigma^2(I) = \sigma^2_{\text{cs}}(I) + (0{\cdot}05I)^2$; $\sigma_{\text{cs}}(I)$ is standard deviation of I based on counting statistics. Solved by Patterson map analysis and Fourier methods. Full-matrix least squares minimized $\sum w(F_o - F_c)^2$. H atoms refined isotropically and all non-H atoms refined anisotropically for a total of 90 parameters. $R = 0{\cdot}019$, $wR = 0{\cdot}030$, GOF = 1·2, where non-Poisson $w^{-1} = [\sigma^2(I) + (0{\cdot}05I)^2]/4F^2$; final value of the refined extinction coefficient, $g = 1{\cdot}03 \times 10^{-6}$; final $(\Delta/\sigma)_{\text{max}} < 0{\cdot}01$; $\Delta\rho_{\text{max}} = 0{\cdot}64\,(5)$ and $\Delta\rho_{\text{min}} = -0{\cdot}55\,(5)$ e Å^{-3} on final difference map. Atomic scattering factors and anomalous-dispersion correc-



(I)

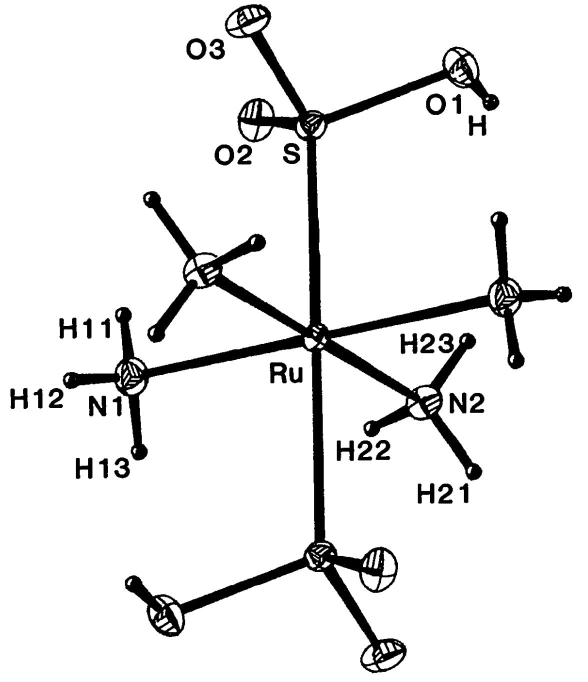


Fig. 1. *ORTEP* (Johnson, 1976) drawing and numbering scheme for (I). The ellipsoids are drawn at the 30% probability surface and H atoms have been given arbitrary radii for figure clarity.

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Table 1. *Fractional coordinates and isotropic or equivalent isotropic thermal parameters*

The thermal parameters are of the form $B = 8\pi^2U$ and $B_{eq} = (8\pi^2/3)\sum_i\sum_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B/B_{eq}(Å^2)
Ru	0·000	0·000	0·000	0·987 (4)
S	−0·13953 (7)	0·26052 (6)	0·06300 (4)	1·253 (7)
O(1)	−0·3144 (3)	0·3833 (2)	−0·0575 (1)	2·03 (2)
O(2)	0·0505 (3)	0·3998 (2)	0·1383 (1)	1·89 (3)
O(3)	−0·2957 (2)	0·2198 (2)	0·1256 (1)	2·07 (3)
N(1)	0·3121 (4)	0·0071 (2)	0·1751 (2)	1·77 (4)
N(2)	0·1701 (3)	0·1817 (3)	−0·0834 (1)	1·54 (3)
H	−0·238 (5)	0·435 (5)	−0·082 (3)	3·6 (7)*
H(11)	0·300 (6)	0·101 (6)	0·204 (3)	6 (1)*
H(12)	0·311 (5)	−0·073 (5)	0·219 (3)	4·2 (8)*
H(13)	0·430 (6)	−0·010 (3)	0·159 (3)	2·8 (8)*
H(21)	0·184 (5)	0·126 (5)	−0·146 (3)	4·1 (8)*
H(22)	0·303 (5)	0·199 (5)	−0·032 (3)	4·6 (8)*
H(23)	0·099 (5)	0·284 (4)	−0·105 (2)	3·7 (7)*

* Refined isotropically.

tions from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*. Table 1 gives the atomic coordinates and Table 2 gives selected bond distances and angles.* Fig. 1 shows the complex with the numbering scheme.

Table 2. *Selected bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Ru—S	2·2785 (5)	S—O(2)	1·496 (1)
Ru—N(1)	2·141 (2)	S—O(3)	1·474 (2)
Ru—N(2)	2·142 (2)	O(1)—H	0·75 (4)
S—O(1)	1·617 (1)	N—H	0·81 (ave)
		N—H	0·76–0·87 (range)
S—Ru—N(1)	88·93 (6)	O(1)—S—O(3)	101·74 (8)
S—Ru—N(2)	90·03 (5)	O(2)—S—O(3)	110·54 (8)
N(1)—Ru—N(2)	90·39 (7)	S—O(1)—H	107 (3)
Ru—S—O(1)	110·41 (6)	Ru—N—H	108 (mean)
Ru—S—O(2)	113·87 (6)	Ru—N—H	103–111 (range)
Ru—S—O(3)	115·39 (6)	H—N—H	110 (mean)
O(1)—S—O(2)	103·53 (8)	H—N—H	105–119 (range)

Related literature. Previous structural reports of complexes containing the bisulfite ion as a ligand were of [Ru(bpy)$_2$(py)(HSO$_3$)]PF$_6$ and Ru(bpy)$_2$(HSO$_3$)$_2$ (Allen, Jeter, Cordes & Durham, 1988) and Na$_4$[Ru(SO$_3$)$_2$(HSO$_3$)$_2$(NH$_3$)$_2$] (Johnson, Jeter & Cordes, 1987). The related structural aspects of SO$_2$ as a ligand have been reviewed by Ryan, Kubas, Moody & Eller (1981).

We thank the National Science Foundation and the State of Arkansas for financial support.

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* Lists of anisotropic temperature factors and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52536 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of *cis*-Tetrachlorobis(tetrahydrofuran)hafnium(IV)

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Abstract. [HfCl$_4$(C$_4$H$_8$O)$_2$], $M_r = 464{\cdot}52$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}914$ (2), $b = 12{\cdot}806$ (3), $c = 14{\cdot}357$ (3) Å, $V = 1455$ (1) Å^3, $Z = 4$, $D_x = 2{\cdot}120$ g cm^{-3}, Mo $K\alpha$ ($\lambda = 0{\cdot}71073$ Å), $\mu = 78{\cdot}3$ cm^{-1}, $F(000) = 220$, $T = 298$ K, $R = 0{\cdot}0359$ for 1494 reflections with $I \geq 3\sigma(I)$ and 137 variables. The HfCl$_4$(THF)$_2$ compound has a distorted *cis* octahedral stereochemistry. The bond angles about the Hf atom are consistent with this structure type. The important bond distances are: Hf—Cl 2·363 (4), 2·377 (4) Å (*trans* to THF's), 2·401 (3), 2·388 (4) Å (*cis* to THF's) and Hf—O 2·197 (8), 2·194 (8) Å.

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