# SHORT-FORMAT PAPERS

*Contributions intended for publication under this heading should follow the format given in the* Checklist for Authors [*Acta Cryst.* (1985). C**41**, 1–4].

*Acta Cryst.* (1987). C**43**, 2001–2002

## Structure of Sodium Diamminebis(hydrogensulfito)disulfitoruthenate(II) Hexahydrate

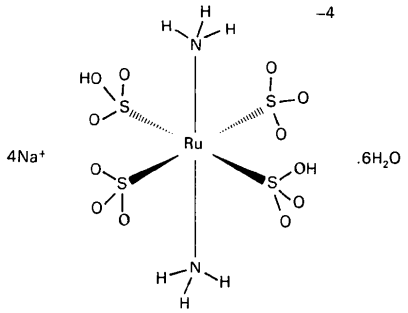
BY D. A. JOHNSON, D. Y. JETER AND A. W. CORDES

*Department of Chemistry and Biochemistry, University of Arkansas, Fayetteville, AR* 72701, *USA*

(*Received* 13 *March* 1987; *accepted* 22 *May* 1987)

**Abstract.** $Na_4[Ru(SO_3)_2(HSO_3)_2(NH_3)_2].6H_2O$, $M_r = 657{\cdot}4$, monoclinic, $P2_1/c$, $a = 9{\cdot}559$ (2), $b = 6{\cdot}347$ (2), $c = 16{\cdot}345$ (2) Å, $\beta = 100{\cdot}16$ (1)°, $V = 976{\cdot}1$ (7) Å$^3$, $Z = 2$, $D_x = 2{\cdot}24$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·7107 Å, $\mu = 13{\cdot}7$ cm$^{-1}$, $F(000) = 660$, $T = 293$ K, $R = 0{\cdot}044$ for 1753 unique observed reflections. The centrosymmetric complex anion is pseudo-octahedral with intramolecular hydrogen bonding between pairs of sulfite and bisulfite ligands. One sodium ion is surrounded by six oxygen atoms from the ligands and the other by two ligand oxygen atoms and four water molecules.

**Experimental.** The title compound (I) was prepared by the treatment of the mother liquor from the preparation of *trans*-$[Ru(SO_3)_2(NH_3)_4]$ (Vogt, Katz & Wiberley, 1965) with excess $Na_2S_2O_5$. Crystals obtained from aqueous solution. Colorless platelet data crystal 0·06 × 0·10 × 0·16 mm mounted on glass fiber. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using $\omega$–$2\theta$ scans of 4 to 16° min$^{-1}$ in $\theta$. Unit cell determined from least-squares analysis of angle data for 25 reflections with $14 < 2\theta < 23°$. Analytical absorption correction based on crystal shape varied from 0·70 to 1·00. Data collected to $(\sin\theta)/\lambda$ of 0·70 Å$^{-1}$, $0 \le h \le 13$, $0 \le k \le 8$, $-23 \le l \le 23$. Three standard reflections (008, $1\bar{3}\bar{5}$, $4\bar{1}3$) varied ±0·9% over 28·3 h of data collection; 2985 reflections measured, 2826 unique ($R_{int} = 0{\cdot}03$), 1073 reflections with $I < 3\sigma(I)$ considered unobserved. Solved by Patterson and Fourier methods. Full-matrix least squares minimized $\sum w(\Delta F)^2$. H atoms constrained to positions found on difference Fourier maps, with isotropic $B$ values = 1·2 × $B$ of bonded N or O. All other atoms refined anisotropically for 133 variables. $R = 0{\cdot}044$, $wR = 0{\cdot}054$, $S = 1{\cdot}11$, where non-Poisson $w^{-1} = [\sigma^2(I) + 0{\cdot}0036I^2]/4F^2$. Final $(\Delta/\sigma)_{max} < 0{\cdot}18$, $\Delta\rho_{max} = 0{\cdot}9$ (1) and $\Delta\rho_{min} = -0{\cdot}5$ (1) e Å$^{-3}$ on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*. Table 1 gives the atom coordinates and Table 2 selected bond distances and angles.* Fig. 1 shows the complex anion with the numbering scheme.




(I)

**Related literature.** The title compound has been prepared previously by the reaction of sulfite or metabisulfite ions with several different Ru ammine complexes (Gleu, Breuel & Rehm, 1938; Lever & Powell, 1969). Other structures reported with Ru–S bonds include an $SO_2$ complex (Vogt, Katz & Wiberly, 1965) and a dimethyl sulfoxide complex (McMillan, Mercer, James & Trotter, 1975). Infrared studies on related complexes have also been reported (Hall & Griffith, 1981).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44083 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/87/102001-02$01.50

Table 1. *Fractional atomic coordinates and thermal parameters*

Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as $\frac{4}{3}(a^2B_{11} + b^2B_{22} + c^2B_{33} + acB_{13}\cos\beta)$.

| | $x$ | $y$ | $z$ | $B_{eq}/B$(Å$^2$) |
|---|---|---|---|---|
| Ru | 0·000 | 0·000 | 0·000 | 0·718 (8) |
| S(1) | 0·1262 (1) | 0·1074 (2) | 0·12889 (8) | 0·93 (2) |
| S(2) | 0·1716 (1) | 0·1225 (2) | −0·07373 (8) | 1·02 (2) |
| Na(1) | 0·0349 (3) | 0·6132 (4) | 0·2293 (2) | 1·58 (4) |
| Na(2) | 0·6135 (3) | 0·3543 (5) | 0·2841 (2) | 1·91 (5) |
| O(1) | 0·0679 (5) | 0·2994 (7) | 0·1618 (3) | 1·68 (8) |
| O(2) | 0·1308 (5) | −0·0648 (7) | 0·1918 (3) | 1·52 (8) |
| O(3) | 0·2839 (4) | 0·1557 (7) | 0·1280 (3) | 1·49 (8) |
| O(4) | 0·3230 (4) | 0·1357 (9) | −0·0171 (3) | 1·90 (9) |
| O(5) | 0·1990 (4) | −0·0183 (8) | −0·1415 (2) | 1·77 (8) |
| O(6) | 0·1414 (4) | 0·3378 (7) | −0·1075 (3) | 1·67 (8) |
| O(7) | 0·3777 (4) | 0·4813 (8) | 0·2442 (3) | 1·91 (8) |
| O(8) | 0·6358 (5) | 0·3924 (9) | 0·1382 (3) | 2·5 (1) |
| O(9) | 0·5686 (5) | 0·2118 (9) | 0·4140 (3) | 2·6 (1) |
| N(1) | −0·1117 (5) | 0·2913 (8) | −0·0149 (3) | 1·35 (9) |
| H(1) | −0·1074 | 0·3906 | 0·0371 | 1·7 |
| H(2) | −0·1973 | 0·2773 | −0·0371 | 1·7 |
| H(3) | −0·0859 | 0·3887 | −0·0547 | 1·7 |
| H(4) | 0·3242 | 0·1680 | 0·0547 | 2·3 |
| H(5) | 0·3496 | 0·3887 | 0·2051 | 2·3 |
| H(6) | 0·3047 | 0·4434 | 0·2793 | 2·3 |
| H(7) | 0·6973 | 0·5000 | 0·1309 | 3·0 |
| H(8) | 0·6289 | 0·3906 | 0·2031 | 3·0 |
| H(9) | 0·6094 | 0·3340 | 0·3711 | 3·1 |
| H(10) | 0·5957 | 0·2578 | 0·4687 | 3·1 |

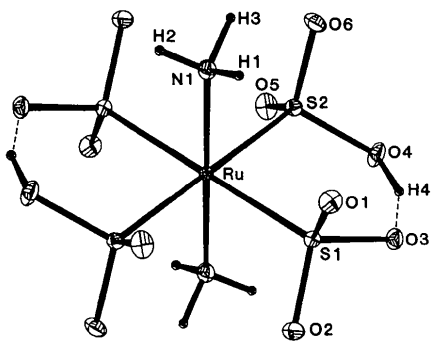



Fig. 1. *ORTEP*II diagram (Johnson, 1976) and atom-numbering scheme. Non-H ellipsoids at 30% probability level, H atoms given arbitrary radii.

Table 2. *Selected interatomic distances* (Å) *and angles* (°)

| | | | |
|---|---|---|---|
| Ru–S(1) | 2·337 (1) | S(2)–O(4) | 1·577 (4) |
| Ru–S(2) | 2·333 (1) | S(2)–O(5) | 1·482 (4) |
| Ru–N(1) | 2·128 (5) | S(2)–O(6) | 1·482 (4) |
| S(1)–O(1) | 1·481 (4) | O(4)–H(4) | 1·189 |
| S(1)–O(2) | 1·497 (4) | O(3)···H(4) | 1·324 |
| S(1)–O(3) | 1·540 (4) | | |

| | | | |
|---|---|---|---|
| S(1)–Ru–S(2) | 94·0 (1) | O(2)–S(1)–O(3) | 104·0 (2) |
| S(1)–Ru–N(1) | 91·1 (1) | O(4)–S(2)–O(5) | 101·9 (2) |
| S(2)–Ru–N(1) | 92·0 (1) | O(4)–S(2)–O(6) | 105·5 (3) |
| O(1)–S(1)–O(2) | 108·6 (3) | O(5)–S(2)–O(6) | 109·1 (3) |
| O(1)–S(1)–O(3) | 105·8 (3) | | |

| Cation–anion | | Cation–water | |
|---|---|---|---|
| Na(1)···O(1) | 2·325 (5) | Na(2)···O(7) | 2·374 (5) |
| Na(1)···O(1$^{i}$) | 2·479 (5) | Na(2)···O(7$^{vi}$) | 2·416 (6) |
| Na(1)···O(2$^{ii}$) | 2·364 (5) | Na(2)···O(8) | 2·443 (5) |
| Na(1)···O(2$^{i}$) | 2·483 (5) | Na(2)···O(9) | 2·415 (6) |
| Na(1)···O(5$^{iii}$) | 2·473 (5) | | |
| Na(1)···O(6$^{iv}$) | 2·392 (5) | | |
| Na(2)···O(2$^{v}$) | 2·461 (5) | | |
| Na(2)···O(3$^{v}$) | 2·488 (5) | | |

Symmetry code: (i) $-x$, $0{\cdot}5+y$, $0{\cdot}5-z$; (ii) $x$, $1+y$, $z$; (iii) $x$, $0{\cdot}5-y$, $0{\cdot}5+z$; (iv) $-x$, $1-y$, $-z$; (v) $1-x$, $0{\cdot}5+y$, $0{\cdot}5-z$; (vi) $1-x$, $-0{\cdot}5+y$, $0{\cdot}5-z$.

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

**References**

Enraf–Nonius (1982). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.

GLEU, K., BREUEL, W. & REHM, K. (1938). *Z. Anorg. Chem.* **235**, 201–210.

HALL, J. P. & GRIFFITH, W. P. (1981). *Inorg. Chim. Acta*, **48**, 65–71.

*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

LEVER, F. M. & POWELL, A. R. (1969). *J. Chem. Soc. A*, pp. 1477–1482.

MCMILLAN, R. S., MERCER, A., JAMES, B. R. & TROTTER, J. (1975). *J. Chem. Soc. Dalton Trans.* pp. 1006–1010.

VOGT, L. H., KATZ, J. L. & WIBERLY, S. E. (1965). *Inorg. Chem.* **24**, 1157–1163.

*Acta Cryst.* (1987). C**43**, 2002–2004

# Structure of $PbPSe_3$

BY HOSEOP YUN AND JAMES A. IBERS

*Department of Chemistry, Northwestern University, Evanston, Illinois* 60201, *USA*

(*Received* 16 *April* 1987; *accepted* 12 *May* 1987)

**Abstract.** Lead phosphorus triselenide, $M_r = 475{\cdot}04$, monoclinic, $P2_1/n$, $a = 6{\cdot}897$ (3), $b = 7{\cdot}642$ (3), $c = 9{\cdot}696$ (4) Å, $\beta = 91{\cdot}51$ (1)°, $V = 510{\cdot}9$ Å$^3$, $Z = 4$, $D_x = 6{\cdot}174$ g cm$^{-3}$, $\lambda$(Mo $K\alpha_1$) = 0·7093 Å, $\mu = 545{\cdot}7$ cm$^{-1}$, $F(000) = 780{\cdot}61$ (including anomalous dispersion), $T = 123$ K, $R(F^2) = 0{\cdot}070$ for 1301

0108-2701/87/102002-03$01.50