

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

(a) The coordination polyhedron

Sb—I	3.523 (1)	S(1)—Sb—S(4)	69.2 (1)
Sb—I'	3.365 (1)	S(2)—Sb—S(3)	69.7 (1)
Sb—S(1)	2.521 (3)	Sb—S(1)—C(1)	88.7 (4)
Sb—S(2)	2.665 (4)	Sb—S(4)—C(1)	83.2 (4)
Sb—S(3)	2.502 (3)	Sb—S(2)—C(2)	83.2 (4)
Sb—S(4)	2.697 (4)	Sb—S(3)—C(2)	88.5 (4)
S(1)···S(4)	2.967 (5)		
S(2)···S(3)	2.954 (5)		

Symmetry code: (i) 1+x, y, z.

(b) The dithiocarbamate ligands

S(1)—C(1)	1.72 (1)	S(1)—C(1)—S(4)	118.8 (7)
S(4)—C(1)	1.72 (1)	S(2)—C(2)—S(3)	118.6 (7)
S(2)—C(2)	1.72 (1)	S(1)—C(1)—N(1)	119.3 (9)
S(3)—C(2)	1.71 (1)	S(4)—C(1)—N(1)	121.9 (9)
C(1)—N(1)	1.32 (2)	S(2)—C(2)—N(2)	121.7 (9)
C(2)—N(2)	1.33 (2)	S(3)—C(2)—N(2)	119.7 (9)
N(1)—C(3)	1.50 (2)	C(1)—N(1)—C(3)	125.6 (10)
N(1)—C(9)	1.51 (2)	C(1)—N(1)—C(9)	123.1 (10)
N(2)—C(4)	1.50 (2)	C(2)—N(2)—C(4)	124.9 (10)
N(2)—C(10)	1.49 (2)	C(2)—N(2)—C(10)	123.4 (10)
C(3)—C(5)	1.53 (2)	N(1)—C(3)—C(5)	103 (1)
C(5)—C(7)	1.45 (3)	N(1)—C(9)—C(7)	102 (1)
C(7)—C(9)	1.50 (2)	N(2)—C(4)—C(6)	104 (1)
C(4)—C(6)	1.49 (3)	N(2)—C(10)—C(8)	103 (1)
C(6)—C(8)	1.43 (3)	C(3)—C(5)—C(7)	106 (1)
C(8)—C(10)	1.53 (2)	C(5)—C(7)—C(9)	110 (2)
		C(4)—C(6)—C(8)	110 (2)
		C(6)—C(8)—C(10)	109 (2)

chain. From ^{121}Sb Mössbauer spectroscopic studies of analogues of trigonal-bipyramidal Sb(dtc) complexes (Stevens & Trooster, 1979) it is known that in these complexes the stereochemical lone pair is in an axial or equatorial position. The I atoms, which bridge the $\text{Sb}[\text{S}_2\text{CN}(\text{CH}_2)_4]_2$ units, lie out of the SbS_4 side, as observed in the crystal structure of $\text{Sb}(\text{Et}_2\text{dtc})_2\text{I}\cdot\text{CHCl}_3$ (McKie *et al.*, 1981). The distances Sb—I 3.365 (1), 3.523 (1) Å are shorter than the sum of the van der Waals radii: 2.2 Å for Sb and 2.15 Å for I (Pauling,

1967). The dithiocarbamate-ligand geometry is very similar to that in other dtc complexes whose crystal structures have been solved. The C≡S bond lengths are in the range 1.71 (1) to 1.72 (1) Å and bond lengths of 1.32 (2) and 1.33 (2) Å confirmed double character of the C≡N bond. These parameters are generally a good approximation to the parameters in other dtc complexes. The high degree of π -conjugation in the S_2CNC_2 ligand fragments is reflected in the good planarity of these fragments. The Sb atom lies 0.019 (1) and 0.089 (1) Å out of the S_2CNC_2 ligand planes. The shortest intermolecular distances are between the S(2), C(4) and C(6) atoms and indicate the presence of weak S···C bonds probably through the H atoms of the corresponding C atoms.

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Sodium *trans*-Bis(1,2-ethanediamine)disulfitorhodate(III) Trihydrate,
 $\text{Na}[\text{Rh}(\text{C}_2\text{H}_8\text{N}_2)_2(\text{SO}_3)_2]\cdot 3\text{H}_2\text{O}$

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Abstract. $M_r = 460.26$, monoclinic, $P2_1/a$, $a = 106.74 (3)^\circ$, $V = 1526 (1) \text{ \AA}^3$, $Z = 4$, $D_m = 1.98$, $D_x = 13.730 (3)$, $b = 13.660 (3)$, $c = 8.495 (5) \text{ \AA}$, $\beta = 2.00 \text{ Mg m}^{-3}$, $\lambda(\text{Ag K}\alpha) = 0.55830 \text{ \AA}$, $\mu = 0.746 \text{ mm}^{-1}$, $F(000) = 936$, $T = 298 \text{ K}$, $R = 0.067$, $wR = 0.067$ for 3366 independent non-zero reflections.

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The two independent Rh atoms on inversion centers are octahedrally coordinated by two S-bonded sulfite ligands in *trans* positions and four N atoms of the chelating 1,2-ethanediamine molecules. Relevant average bond lengths are Rh–S 2.323 (1) (elongated by *trans* influence), Rh–N 2.073 (5) and S–O 1.487 (5) Å. The complex anions, arranged in pseudotetragonal layers within the (001) planes, are held together by Na⁺ cations and hydrogen bonds of medium strength involving the water molecules.

Introduction. In the course of systematic investigations of the *trans* influence in sulfite complexes of Pt metals (Breitinger, Petrikowski & Bauer, 1982, and references therein), spectrometric and X-ray studies of appropriate Rh complexes have been performed (Petrikowski, 1984). With respect to a subsequent normal coordinate treatment the structure of the title compound has been solved.

Experimental. Single crystals of colorless Na[Rh(en)₂(SO₃)₂].3H₂O were obtained from an aqueous solution of rhodium(III) chloride, Na₂SO₃ and 1,2-ethanediamine (en) (details and analysis deposited). *D_m* by flotation in trichloromethane–dibromomethane mixtures. Crystal 0.2 × 0.15 × 0.15 mm; unit-cell parameters by least-squares refinement of 25 reflections (2θ ≈ 25°); systematic absences indicate space group *P2₁/a*; Philips PW 1100 four-circle diffractometer, graphite-monochromated Ag Kα radiation; intensity data (maximum *h,k,l*: 17,17,10) recorded in ω-scan mode for 3° ≤ θ ≤ 23°, sinθ/λ ≤ 0.70 Å⁻¹; 3366 independent reflections measured (*R*_{int} = 0.031), 3214 with *I* > 2σ(*I*); three standard reflections (622, 622, 622) measured every 90 min with no significant variation; data not corrected for absorption; scattering factors and correction for anomalous dispersion from *International Tables for X-ray Crystallography* (1974).

Rh, S and Na positions from three-dimensional Patterson synthesis, remaining non-hydrogen atoms from subsequent Fourier maps; refinement (on *F*) by blocked-matrix least-squares method, H atoms not localized; refinement of anisotropic (Rh and S) and isotropic (C, N, O and Na) temperature factors gave *R* = 0.067, *wR* = 0.067, weighting scheme *w* = 0.0034/[σ²(*F_o*) + 10⁻⁵*F_o*²]; final difference map showed no maxima greater than those for the expected H positions; (Δ/σ)_{max} 2.72 for *z* of C(3), others 1.19 for *y* of C(4) and 1.12 for *x* of C(3), otherwise ≤ 1.0; calculations carried out with the *SHELX76* system (Sheldrick, 1976); no correction for secondary extinction.

Discussion. The structure discussed here is isotypic with that of the corresponding Co^{III} complex already reported (Fallon, Raston, White & Yandell, 1980). Fig. 1 shows a view onto the (001) plane and the atom

numbering scheme. The final atomic parameters and *U* values are given in Table 1.* Table 2 shows interatomic distances and angles in the complex anions.

* Lists of structure factors and anisotropic thermal parameters, details of the preparative procedure and the analytical data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39899 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

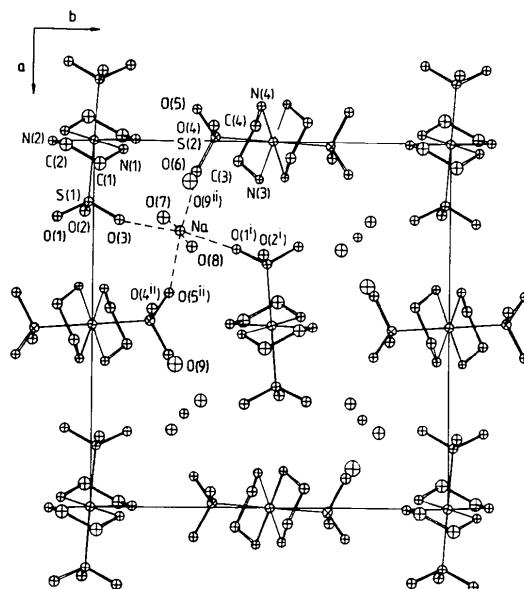


Fig. 1. Projection of the structure of Na[Rh(en)₂(SO₃)₂].3H₂O onto the (001) plane, showing non-hydrogen atoms with labeling (ORTEP; Johnson, 1965). The ellipsoids correspond to 50% probability.

Table 1. Fractional coordinates (×10⁴) and isotropic thermal parameters (Å² × 10⁴), with e.s.d.'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i>
Rh(1)	0	0	0	149 (1)*
Rh(2)	0	5000	0	155 (1)*
S(1)	1617 (1)	-137 (1)	-333 (2)	184 (3)*
S(2)	41 (1)	3383 (1)	858 (2)	190 (3)*
Na	2454 (2)	2418 (2)	103 (3)	250 (5)
O(1)	2167 (3)	-1002 (3)	538 (4)	261 (8)
O(2)	1546 (3)	-235 (3)	-2122 (5)	327 (8)
O(3)	2216 (3)	741 (3)	330 (5)	291 (8)
O(4)	41 (3)	3335 (3)	2630 (5)	315 (8)
O(5)	-884 (3)	2871 (3)	-141 (4)	256 (8)
O(6)	953 (3)	2870 (3)	686 (5)	299 (8)
O(7)	1608 (3)	1953 (3)	-2646 (5)	467 (10)
O(8)	3414 (3)	2755 (3)	3043 (5)	393 (9)
O(9)	6912 (5)	2319 (4)	4616 (7)	685 (12)
N(1)	622 (3)	825 (3)	2089 (5)	253 (9)
N(2)	331 (3)	-1161 (3)	1649 (5)	242 (9)
N(3)	710 (3)	4625 (3)	-1765 (5)	211 (8)
N(4)	-1297 (3)	4662 (3)	-1876 (5)	229 (8)
C(1)	1245 (6)	171 (6)	3417 (9)	497 (13)
C(2)	735 (6)	-786 (5)	3380 (8)	483 (13)
C(3)	-13 (4)	4031 (4)	-3098 (7)	301 (10)
C(4)	-1061 (4)	4500 (4)	-3464 (7)	327 (11)

* $U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta)$.

Table 2. Bond lengths (Å) and angles (°) in the complex anions

Anion 1		Anion 2	
Rh(1)—S(1)	2.325 (1)	Rh(2)—S(2)	2.321 (1)
S(1)—O(1)	1.480 (4)	S(2)—O(4)	1.508 (4)
S(1)—O(2)	1.500 (5)	S(2)—O(5)	1.484 (4)
S(1)—O(3)	1.472 (4)	S(2)—O(6)	1.478 (4)
Rh(1)—N(1)	2.068 (4)	Rh(2)—N(3)	2.075 (5)
Rh(1)—N(2)	2.078 (4)	Rh(2)—N(4)	2.071 (4)
N(1)—C(1)	1.498 (8)	N(3)—C(3)	1.510 (7)
N(2)—C(2)	1.503 (8)	N(4)—C(4)	1.492 (8)
C(1)—C(2)	1.480 (11)	C(3)—C(4)	1.524 (8)
O(1) S(1)—O(2)	108.1 (2)	O(4)—S(2)—O(5)	107.7 (2)
O(1)—S(1)—O(3)	108.1 (2)	O(4)—S(2)—O(6)	108.8 (3)
O(2)—S(1)—O(3)	109.0 (2)	O(5)—S(2)—O(6)	109.2 (2)
O(1)—S(1)—Rh(1)	111.8 (2)	O(4)—S(2)—Rh(2)	110.4 (2)
O(2)—S(1)—Rh(1)	110.2 (2)	O(5)—S(2)—Rh(2)	109.2 (2)
O(3)—S(1)—Rh(1)	109.6 (2)	O(6)—S(2)—Rh(2)	111.5 (2)
S(1)—Rh(1)—N(1)	88.6 (1)	S(2)—Rh(2)—N(3)	91.0 (1)
S(1)—Rh(1)—N(2)	89.4 (1)	S(2)—Rh(2)—N(4)	88.1 (1)
N(1)—Rh(1)—N(2)	83.2 (2)	N(3)—Rh(2)—N(4)	82.4 (2)
Rh(1)—N(1)—C(1)	109.1 (3)	Rh(2)—N(3)—C(3)	109.3 (3)
Rh(1)—N(2)—C(2)	110.3 (3)	Rh(2)—N(4)—C(4)	111.5 (3)
N(1)—C(1)—C(2)	110.3 (4)	N(3)—C(3)—C(4)	107.8 (3)
N(2)—C(2)—C(1)	111.1 (4)	N(4)—C(4)—C(3)	108.4 (3)

The unit cell comprises two independent anions, both with Rh atoms on inversion centers. Coordination around the Rh atoms is pseudo-octahedral. The S-bonded sulfite groups are in *trans* positions. Owing to the *trans* influence of the sulfite ligand the average Rh—S bond length of 2.323 (1) Å is significantly longer than in *fac*-Na₃[Rh(NH₃)₃(SO₃)₃].6H₂O [2.291 (3) Å; Petrikowski (1984)]. The geometries of the sulfite ligands are similar to those found in the analogous Co^{III} compound (Fallon *et al.*, 1980), with average S—O bonds of 1.487 (5) and 1.489 (6) Å, respectively. The elongations of the bonds S(1)—O(2) and S(2)—O(4) (Table 2) arise from involvements of the respective O atoms in hydrogen bonding (see below and Table 4).

The Rh—N bond lengths of 2.068 (4) to 2.078 (4) Å are similar to those reported for the [Rh(en)₃]³⁺ cation [2.056 (7) and 2.067 (7) Å; Whuler, Brouty, Spinat & Herpin (1976)]. In both anions the two chelate rings adopt envelope conformations with opposite chiralities (δ and λ) yielding internally compensated *meso* forms of the complexes. The dihedral angles viewed along the C—C bonds are 46.3° on average. Distances of the ring atoms from the equatorial reference planes normal to the S—Rh—S axes are as follows: in anion 1 N(1) 0.051 (4), N(2) 0.022 (4), C(1) 0.522 (8), C(2) -0.054 (8); in anion 2 N(3) -0.037 (4), N(4) 0.068 (4), C(3) 0.453 (5), C(4) -0.157 (5) Å. For the influence of the intracomplex N—H...O hydrogen bonds and the relative orientations of the sulfite groups on the conformation of the chelate rings a similar reasoning as given by Fallon *et al.* (1980) applies. The cohesion around the (001) plane is mainly effected by electrostatic interactions between complex anions and pseudo-octahedrally coordinated Na⁺ cations showing a narrow range of Na—O distances [except Na—O(8),

Table 3]. However, several intercomplex hydrogen bonds N—H...O [2.870 (6) to 3.012 (6) Å] and the system O(2ⁱ)...H—O(8)—H...O(4ⁱⁱ) (Table 4) (*cf.* data given by Fallon *et al.*, 1980) also contribute to bonding within the layers. The connection between these layers is achieved by a branched hydrogen-bond system (Fig. 2 and Table 4), which is obviously broken on grinding the substance (the 001 reflection disappears). Although the H atoms have not been localized it may be concluded from the geometry around O(7ⁱⁱⁱ) and from IR absorptions above 3500 cm⁻¹ that one O(7ⁱⁱⁱ)—H bond is probably not involved in hydrogen bonding.

Table 3. Environment of the Na⁺ ions (distances in Å, angles in °)

	O(3)	O(5 ⁱⁱ)	O(6)	O(7)	O(8)	
O(1)	2.322 (4)	168.5 (2)	81.3 (1)	94.3 (1)	95.6 (2)	88.5 (1)
O(3)	2.330 (4)		90.3 (1)	95.1 (2)	77.2 (2)	98.3 (1)
O(5 ⁱⁱ)	2.383 (5)			170.9 (2)	94.6 (2)	82.8 (1)
O(6)	2.337 (5)				93.8 (2)	89.1 (1)
O(7)	2.376 (5)					174.8 (2)
O(8)	2.508 (4)					

Symmetry code: (i) 0.5 - x, 0.5 + y, z; (ii) 0.5 + x, 0.5 - y, z; (iii) x, y, z + 1.

Table 4. Environments of the H₂O molecules with oxygen atoms O(7ⁱⁱⁱ), O(8) and O(9ⁱⁱ) (distances in Å, angles in °)

Environment of O(7 ⁱⁱⁱ)				
		O(2 ⁱⁱ)	O(3 ⁱⁱⁱ)	O(9 ⁱⁱ)
Na ⁱⁱⁱ	2.376 (5)	98.4 (1)	50.7 (2)	127.0 (2)
O(2 ⁱⁱ)	3.028 (6)		47.9 (2)	120.9 (2)
O(3 ⁱⁱⁱ)	2.935 (6)			151.9 (5)
O(9 ⁱⁱ)	2.671 (8)			
Environment of O(8)				
		O(2 ⁱ)	O(4 ⁱⁱ)	O(9 ⁱⁱ)
Na	2.508 (4)	87.1 (1)	89.3 (1)	102.9 (1)
O(2 ⁱ)	2.860 (6)		113.5 (2)	104.1 (2)
O(4 ⁱⁱ)	2.789 (6)			141.0 (4)
O(9 ⁱⁱ)	2.763 (9)			
Environment of O(9 ⁱⁱ)				
		O(7 ⁱⁱⁱ)	O(8)	
O(4)	2.783 (7)	107.4 (2)	112.5 (2)	
O(7 ⁱⁱⁱ)	2.671 (8)		139.1 (4)	
O(8)	2.763 (9)			

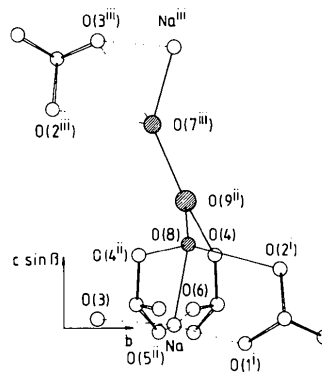


Fig. 2. Environments of the H₂O molecules with oxygen atoms O(7ⁱⁱⁱ), O(8) and O(9ⁱⁱ) (hatched), viewed down the *a* axis. Heavy lines indicate the bond system connecting the layers around (001).

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Crystal Structures of Ethylenediaminetetraacetato Metal Complexes. VII. Structures containing the $[\text{Fe}(\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_8)(\text{H}_2\text{O})]^-$ Anion: Ethylenediammonium (EDA) and Tetraethylammonium (TEA) Salts

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Abstract. (EDA,Fe): $[\text{C}_2\text{H}_{10}\text{N}_2][\text{Fe}(\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_8)(\text{H}_2\text{O})]_2 \cdot 6\text{H}_2\text{O}$, $M_r = 894.36$, monoclinic, $C2/c$, $a = 30.460$ (3), $b = 7.504$ (1), $c = 15.450$ (2) Å, $\beta = 95.50$ (2)°, $V = 3515$ (1) Å³, $D_x = 1.69$ Mg m⁻³, $Z = 4$, $F(000) = 1872$, $\lambda(\text{Mo } K\alpha) = 0.7093$ Å, $\mu(\text{Mo } K\alpha) = 0.957$ mm⁻¹. (TEA,Fe): $[\text{N}(\text{C}_2\text{H}_5)_4][\text{Fe}(\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_8)(\text{H}_2\text{O})]_2 \cdot 2\text{H}_2\text{O}$, $M_r = 528.36$, orthorhombic, $Pcab$ (non-standard setting of $Pbca$), $a = 22.484$ (4), $b = 15.221$ (3), $c = 14.228$ (3) Å, $V = 4869$ (2) Å³, $D_x = 1.44$ Mg m⁻³, $Z = 8$, $F(000) = 2248$, $\lambda(\text{Mo } K\alpha) = 0.7093$ Å, $\mu(\text{Mo } K\alpha) = 0.700$ mm⁻¹. The structures were refined by full-matrix least squares to R values of 0.041 and 0.065 for 2436 and 2837 observed reflections, respectively. A half-normal probability plot comparison between the $[\text{EDTA}(\text{H}_2\text{O})\text{Fe}]^-$ anions of different structures in the literature shows that the Fe coordination polyhedron in (EDA,Fe) is similar to that in the crystal structure of (Ag,Fe), while the polyhedron in (TEA,Fe) is similar to that in the crystal structure of (K,Fe) [Solans, Font Altaba & Garcia-Oricain (1984). *Acta Cryst.* **C40**, 635–638].

Introduction. A systematic crystal structure study of ethylenediaminetetraacetato complexes is being carried out in the authors' departments (Solans, Font-Altaba & Garcia-Oricain, 1984a,b).

In preceding papers (parts V and VI) the crystal structures of $X[\text{EDTA}(\text{H}_2\text{O})\text{Fe}]_m \cdot n\text{H}_2\text{O}$ with $X = \text{Ag}^+$, Na^+ , K^+ and Tl^+ (Solans *et al.*, 1984a) and Ba^{2+} (Solans *et al.*, 1984b) were determined and compared with the structures where $X = \text{Li}^+$ and Rb^+ (Lind, Hamor, Hamor & Hoard, 1964). Two kinds of $[\text{EDTA}(\text{H}_2\text{O})\text{Fe}]$ bipyramidal-pentagonal coordination polyhedra were found. In this paper the influence of organic cations is studied; two ions have been selected, one with spherical symmetry $[\text{N}(\text{C}_2\text{H}_5)_4]^+$, tetraethylammonium] and the other without this symmetry ($\text{C}_2\text{H}_{10}\text{N}_2^+$, ethylenediammonium).

Experimental. (EDA,Fe): yellow-brown prism $0.1 \times 0.1 \times 0.2$ mm. (TEA,Fe): red prism $0.2 \times 0.2 \times 0.3$ mm. Philips PW 1100 diffractometer, graphite monochromator, Mo $K\alpha$. Cell parameters in both structures from 25 reflections ($4 \leq \theta \leq 12^\circ$). ω -scan technique, scan width 0.8° , scan speed $0.03^\circ \text{ s}^{-1}$. Three standard reflections every 2 h, no variations in reference intensities. (EDA,Fe): 2454 reflections measured; $\theta \leq 30^\circ$; h,k,l : $\pm 36,8,18$; 2436 with $I \geq 2.5\sigma(I)$. (TEA,Fe): 2891 reflections measured; $\theta \leq 30^\circ$; h,k,l : 24,18,16; 2837 with $I \geq 2.5\sigma(I)$. Lp corrections, absorption ignored. (EDA,Fe) determined by direct methods with *MULTAN80* (Main, Fiske, Hull,