Discussion. Atomic coordinates and geometrical parameters of the anion are given in Tables 1 and 2.* The structure of the anion is shown in Fig. 1. The compound is isomorphous, and presumably isostructural, with $[PPh_4]_2[MoOS_3(CuCl)_3]$ (Müller, Schimanski & Schimanski, 1983; coordinates have not been published for this structure). The same Mo-centred anion occurs in the double salt $[PPh_4]_3[MoOS_3(CuCl)_3][CuCl_2]$ (Clegg, Garner, Nicholson & Raithby, 1983) and differs from the W-centred anion only in having slightly shorter bonds to Mo than to W. The MOS_3Cu_3 core (M = Mo, W) is also observed in $[MOS_3(CuPPh_3)_3Cl]$, in which the eighth vertex of the MS_3Cu_3Cl cube, opposite M, is occupied by Cl (Müller, Bögge & Schimanski, 1983); this eighth vertex is vacant in the $[MOS_3(CuCl)_3]^{2-}$ anions. A cube cluster occurs as the ReS_3Cu_3Cl core of $[ReS_4(CuCl)_3Cl]^{2-}$ (Scattergood, Garner & Clegg, 1987), again with Cl in the eighth vertex position. By contrast, all four S atoms are involved in bonding to Mo and Cu in $[MoS_4(CuCl)_3]^{2-}$, giving a T-shaped $MoCu_3$ arrangement instead of a pyramidal one (Clegg, Garner & Nicholson, 1983). In $[MS_4(CuCl)_4]^{2-}$ (M = Mo or W), a square of Cu atoms surrounds the central M atom, with all S atoms involved in bonding to Mo and Cu; Cl bridges link the anions together into polymeric chains (M = Mo; Nicholson, Flood, Garner & Clegg, 1983) or dimers (M = W; Clegg, Scattergood & Garner, 1987).

Thus we find that a pseudo-cubane core is adopted in complexes of this type for a central MOS_3 unit, where S bonds to the remaining metal atoms and O does not, and for a central MS_4 unit when an additional atom can occupy the eighth vertex position to complete the distorted cube. Without the additional atom, a central MS_4 unit forms complexes in which all the S atoms bond to the other metal atoms.

We thank the SERC for financial support.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a complete list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44332 (32 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-Dichlorobis(ethylenediamine)rhodium(III) Nitrate and Hydrogen Oxalate Salts

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Abstract. (1) $[RhCl_2(C_2H_8N_2)_2]NO_3$, M_r = 356·0, monoclinic, $P2_1/n$, a = 6·441 (2), b = 9·275 (3), c = 10·005 (3) Å, β = 102·45 (2)°, V = 583·6 (5) Å^3, Z = 2, D_x = 2·026 (3) g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ = 19·0 cm^{-1}, $F(000)$ = 356, T = 295 (1) K, final R = 0·016 for 1401 observed reflections (θ–2θ scan) with $F_o^2 > 2\sigma(F_o^2)$. (2) $[RhCl_2(C_2H_8N_2)_2][C_2HO_4].2H_2O$, M_r = 419·1, monoclinic, $P2_1/m$, a = 6·434 (2), b = 15·391 (4), c = 7·432 (2) Å, β = 93·72 (2)°, V = 734·4 (6) Å^3, Z = 2, D_x = 1·895 (3) g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ = 15·4 cm^{-1}, $F(000)$ = 424, T = 295 (1) K, final R = 0·023 for 1226 observed reflections (θ–2θ scan) with $F_o^2 > 2\sigma(F_o^2)$. Both compounds contain centrosymmetric dichloro octahedral rhodium(III) complexes with two chelated ethylenediamine ligands in *gauche* conformation in a square plane. The Rh—Cl distances are (1) 2·3325 (3) and (2) 2·3291 (4) Å, and the Rh—N distances are (1)

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2·063 (2) and (2) 2·067 (2) Å. In compound (2) the hydrogen oxalate ion and the two water molecules of crystallization are located on the mirror planes. The hydrogen oxalate ion has a C—C distance 1·553 (4) Å and C—O distances of 1·255 (3), 1·240 (3), 1·205 (3) and 1·310 (4) Å, the latter being the protonated oxygen.

Introduction. In the course of our studies of amine and oxime chelates of rhodium(III) we have prepared salts of *trans*-dichlorobis(ethylenediamine)rhodium(III) as simple model compounds for comparison with the more complicated nitrogen-coordinated complexes. The crystal structure of the nitrate salt has been reported (Baidina, Podberezskaya, Belyaev & Bakakin, 1979) but is here, at higher precision, compared with the hydrogen oxalate salt to determine the extent, if any, of anion effects on the detailed geometry of the rhodium(III) complex.

Experimental. Yellow crystals of $[Rh(en)_2Cl_2]NO_3$ were obtained by slow evaporation of the reaction mixture (Johnson & Basolo, 1962) without recrystallization. Light yellow crystals of $[Rh(en)_2Cl_2][C_2HO_4].2H_2O$ were obtained by slow evaporation of a slightly wet methanol solution. Both crystals had approximate dimensions $0\cdot1 \times 0\cdot2 \times 0\cdot3$ mm. Data collection and reduction were handled for both compounds as follows. The crystal was placed in random orientation on an Enraf–Nonius CAD-4 diffractometer. After optical centering, 25 reflections ($2\theta = 18$–$36°$) were located by an automatic search of reciprocal space and recentered twice. The setting angles of those reflections were used to determine the cell dimensions. Three standard reflections every 7200 s of X-ray exposure (no significant variation), three orientation standards after every 200 reflections with recentering of all 25 if any significant angular error, five ψ scans to obtain empirical absorption curve, θ–2θ scan technique. For compound (1), 2869 reflections (hkl, $\bar{h}kl$, $\bar{h}\bar{k}\bar{l}$ and $h\bar{k}\bar{l}$) from $2\theta = 4$ to 56°, empirical transmission correction range from 0·90 to 1·00, systematic absences $0k0$ with $k = 2n+1$ and $h0l$ with $h+l = 2n+1$, 1504 independent reflections ($R_{int} = 0\cdot017$), 1401 reflections with $F_o^2 > 2\sigma(F_o^2)$ used in structure solution and refinement. For compound (2), 2033 reflections (hkl, $hk\bar{l}$ from $2\theta = 4$ to 50° and also $\bar{h}\bar{k}\bar{l}$ and $\bar{h}\bar{k}l$ from $2\theta = 4$ to 40°), empirical transmission correction range 0·88 to 1·00, systematic absences $0k0$ with $k = 2n+1$, 1307 independent reflections ($R_{int} = 0\cdot022$), 1226 reflections with $F_o^2 > 2\sigma(F_o^2)$ used in structure solution and refinement. Both structures solved by Patterson and difference Fourier methods, full-matrix least squares minimized $\sum w(|F_o| - |F_c|)^2$, H atoms from difference Fourier map and calculation were refined with isotropic thermal parameters [fixed B's for (2)], all other atoms anisotropically, scattering factors including f' and f'' from *International Tables for X-ray Crystallography* (1974); computation on PDP 11/34 and 11/24 computers using Enraf–Nonius (1984) *SDP* programs. Compound (1), 118 variables, $R = 0\cdot016$, $wR = 0\cdot031$, $S = 1\cdot40$, $w = 4F_o^2/[\sigma^2_{counting} + (0\cdot040F_o^2)^2]$, max. shift/e.s.d. = 0·03, isotropic extinction (Zachariasen, 1967) [$g = 2\cdot26\ (5) \times 10^{-6}$], max. and min. $\Delta\rho$ on final difference Fourier map 1·0 (near Rh) and −0·5 e Å^{-3}. Compound (2), 135 variables, $R = 0\cdot023$, $wR = 0\cdot035$, $S = 1\cdot79$, $w = 4F_o^2/[\sigma^2_{counting} + (0\cdot030F_o^2)^2]$, max. shift/e.s.d. = 0·12, isotropic extinction (Zachariasen, 1967) [$g = 1\cdot15\ (5) \times 10^{-7}$], max. and min. $\Delta\rho$ on final difference Fourier map 1·3 and −0·6 e Å^{-3}.

Table 1. *Positional parameters and e.s.d.'s for compound* (1)

	x	y	z	B_{eq}(Å^2)
Rh	0·000	0·000	0·000	1·363 (3)
Cl	0·29088 (5)	0·15545 (3)	0·05238 (3)	2·336 (6)
O(1)	0·4698 (4)	0·0606 (3)	0·6133 (2)	4·27 (5)
O(2)	0·4385 (5)	0·0550 (4)	0·3945 (3)	6·13 (7)
O(3)	0·3504 (4)	0·0902 (3)	0·4738 (3)	5·01 (6)
N(1)	−0·1704 (2)	0·1335 (1)	0·1016 (1)	1·94 (2)
N(2)	−0·0895 (2)	0·0962 (1)	−0·1896 (1)	2·09 (2)
N(3)	0·500	0·000	0·500	3·15 (7)
C(1)	−0·1004 (2)	0·1096 (2)	0·2512 (1)	2·47 (2)
C(2)	0·0599 (2)	0·0494 (2)	−0·2753 (1)	2·64 (3)

Anisotropically refined atoms are given in the form of the equivalent isotropic displacement parameter defined as $\frac{4}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab(\cos\gamma)B_{12} + ac(\cos\beta)B_{13} + bc(\cos\alpha)B_{23}]$.

Table 2. *Positional parameters and e.s.d.'s for compound* (2)

	x	y	z	B_{eq}(Å^2)
Rh	0·000	0·000	0·000	1·562 (5)
Cl	0·26672 (9)	0·04097 (4)	−0·17974 (8)	2·36 (1)
O(1)	0·2242 (4)	0·250	−0·4483 (3)	2·80 (5)
O(2)	0·2846 (4)	0·250	0·0280 (3)	2·59 (5)
O(3)	0·5136 (4)	0·250	−0·2734 (4)	3·39 (6)
O(4)	−0·0019 (4)	0·250	−0·1576 (3)	2·52 (5)
O(*W*1)	0·2904 (5)	−0·250	−0·1195 (4)	6·7 (1)
O(*W*2)	−0·1762 (5)	0·250	−0·5324 (4)	7·9 (1)
N(1)	0·0838 (3)	0·0951 (1)	0·1874 (3)	2·09 (4)
N(2)	0·2062 (3)	−0·0743 (1)	0·1592 (3)	2·03 (4)
C(1)	0·2653 (4)	0·0649 (2)	0·3054 (4)	2·57 (5)
C(2)	0·2426 (4)	−0·0318 (2)	0·3374 (3)	2·62 (5)
C(3)	0·1896 (5)	0·250	−0·1248 (4)	2·01 (6)
C(4)	0·3260 (6)	0·250	−0·2898 (5)	2·44 (7)

Anisotropically refined atoms are given in the form of the equivalent isotropic displacement parameter defined as $\frac{4}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab(\cos\gamma)B_{12} + ac(\cos\beta)B_{13} + bc(\cos\alpha)B_{23}]$.

Discussion. Final parameters for nonhydrogen atoms are in Table 1 [compound (1)] and Table 2 [compound (2)]*. The molecular structure as observed by Baidina

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* Lists of structure factors, H-atom parameters, anisotropic thermal parameters, and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44319 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

et al. (1979) involves *trans*-dichloro ligands and two chelated ethylenediamine ligands with *gauche* conformation in the octahedral Rh^{III} cations. The nitrate ion is normal (trigonal planar) but is on a center of symmetry so that each O has two equally occupied positions. The hydrogen oxalate ion lies completely in the mirror plane. The rhodium complexes are shown in Figs. 1(*a*) [compound (1)] and 1(*b*) [compound (2)]. Table 3 gives the important bond distances and angles with comparison to previous studies. From Fig. 1 it is clear that the complex cation structure is nearly identical for the two salts. The Rh and N atoms are coplanar in both cases with C(1) ±0·30 (1) and ±0·25 Å (2) and C(2) ±0·40 (1) and ±0·43 Å (2) from that plane; center of symmetry in each complex produces the *gauche* conformations. Least-squares planes containing the *trans*-chloro ligands, the Rh atom, and pairs of *trans* N atoms are nearly perpendicular to each other [96·93 (2) in (1) and 97·08 (3)° in (2)] and to the Rh, 4N plane [89·51 (2) and 89·76 (2)° in (1) and 90·32 (3) and 91·38 (3)° in (2)]. There are no unusually short intermolecular contacts except for hydrogen bonding. Table 4 summarizes the hydrogen bonding, which is extensive in both complexes. Fig. 2 shows the hydrogen bonds in compound (2) where all oxygen H atoms and one H from each amine is involved. The oxalate H is involved in a bifurcated interaction with a water molecule

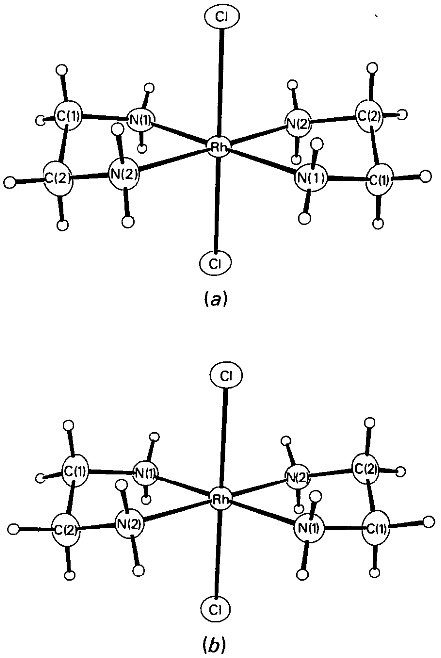

Fig. 1. (*a*) Perspective view of $[Rh(en)_2Cl_2]^+$ from the nitrate salt. (*b*) Perspective view of $[Rh(en)_2Cl_2]^+$ from the hydrogen oxalate salt.

Table 3. *Selected bond distances* (Å) *and angles* (°)

	(1)	(1)†	(2)	*cis*-(1)*†
Rh–Cl	2·3325 (3)	2·335 (2)	2·3291 (4)	2·362 (2)
Rh–N(1)	2·062 (2)	2·067 (6)	2·068 (2)	2·052 (5),2·053 (5)
Rh–N(2)	2·064 (1)	2·065 (6)	2·065 (2)	2·049 (4),2·057 (5)
N(1)–C(1)	1·484 (4)	1·51 (1)	1·490 (3)	1·480 (7),1·473 (7)
N(2)–C(2)	1·485 (3)	1·50 (1)	1·482 (3)	1·464 (14),1·482 (7)
C(1)–C(2)	1·508 (6)	1·52 (1)	1·515 (3)	1·516 (7),1·518 (12)
N(3)–O(1)	1·316 (2)	1·30 (1)		1·245 (5)
N(3)–O(2)	1·162 (2)	1·16 (1)		1·241 (6)
N(3)–O(3)	1·261 (2)	1·25 (1)		1·226 (7)
C(3)–C(4)			1·533 (4)	
C(4)–O(1)			1·310 (4)	
C(4)–O(3)			1·205 (3)	
C(3)–O(2)			1·255 (3)	
C(3)–O(4)			1·241 (3)	
Cl–Rh–Cl	180	180	180	90·65 (7)
Cl–Rh–N(1)	90·31 (5)	90·2 (2)	91·43 (5)	90·4 (2),93·6 (2),92·2 (2)
Cl–Rh–N(2)	90·52 (5)	90·6 (2)	90·50 (5)	89·6 (2),89·5 (2),87·9 (2)
N(1)–Rh–N(1)	180	180	180	93·7 (2),93·5 (2)
N(2)–Rh–N(2)	180	180	180	91·9 (2)
N(1)–Rh–N(2)	83·06 (6)	83·6 (2)	82·90 (7)	83·1 (2),83·5 (2)

* Values are for related distances and angles.
† These values are from Baidina *et al.* (1979).

Table 4. *Details of hydrogen bonds* (*distances in* Å, *angles in* °)

X–H···*Y*	*X*···*Y*	*X*–H	H···*Y*	*X*–H···*Y*
(2)				
O(1)–H(O1)···O(*W*2)	2·612 (3)	0·68	1·95	163
O(1)–H(O1)···O(4)	2·681 (3)	0·68	2·36	111
O(*W*1)–H(10*W*1)···O(4)	2·861 (3)	0·78	2·08	177
O(*W*1)–H(20*W*1)···O(2)	2·775 (3)	0·76	2·03	165
O(*W*2)–H(10*W*2)···O(3)	2·862 (3)	0·48	2·49	139
O(*W*2)–H(20*W*2)···O(*W*1)	2·642 (3)	0·72	1·93	179
N(1)–H(2N1)···O(2)	2·992 (3)	0·87	2·16	162
N(2)–H(2N2)···O(4)	3·007 (3)	0·91	2·11	172
(1)				
N(1)–H(1N1)···O(3)	2·880 (3)	0·72	2·18	163
N(1)–H(1N1)···O(1)	2·972 (3)	0·72	2·29	159
N(1)–H(1N1)···Cl	3·122 (2)	0·72	2·99	93*
N(1)–H(2N1)···Cl	3·404 (2)	0·79	2·67	155
N(1)–H(2N1)···Cl	3·105 (2)	0·79	2·82	104*
N(2)–H(1N2)···O(1)	3·105 (3)	1·00	2·18	154
N(2)–H(1N2)···O(2)	3·039 (3)	1·00	2·20	141
N(2)–H(1N2)···Cl	3·128 (2)	1·00	2·92	101*
N(2)–H(2N2)···O(2)	3·338 (3)	0·81	2·55	163
N(2)–H(2N2)···O(3)	3·400 (3)	0·81	2·75	139
N(2)–H(2N2)···Cl	3·100 (2)	0·81	2·92	95*

* Intramolecular (weak interaction).

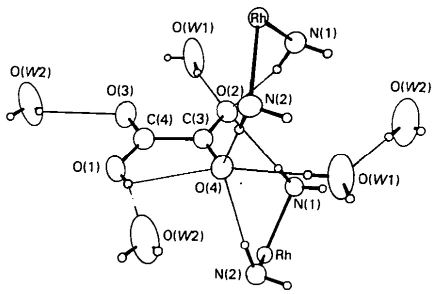

Fig. 2. Hydrogen-bonding network in $[Rh(en)_2Cl_2][HC_2O_4].2H_2O$.

[(O*W*2)] and intramolecularly with an oxalate oxygen [O(4)]. This latter oxygen is an acceptor for three additional hydrogen bonds, while O(2) accepts three hydrogen bonds from water [O(*W*1)] and amine hydrogens and O(3) accepts one from water [O(*W*2)]. The protonated oxalate oxygen [O(1)] does not accept any hydrogen bonds. The intramolecular hydrogen bond O(1) to O(4) is of moderate strength [$\text{O}\cdots\text{O} = 2{\cdot}681\ (3)$ Å]. In compound (1) the hydrogen bonding involves all amine H atoms with the nitrate oxygen atoms as primary acceptors and apparent weak intramolecular interactions with the coordinated chloro ligands.

Despite these rather large differences in crystalline environments for the rhodium complexes in the two salts there is little difference in molecular structure (Fig. 1, Table 3), suggesting that this structure may be very similar to that in solution.

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Structures of *N*-Benzylpiperazinium Tetrabromocuprate(II) Hydrate and Bis[methyl(2-phenylethyl)ammonium] Tetrabromocuprate(II)

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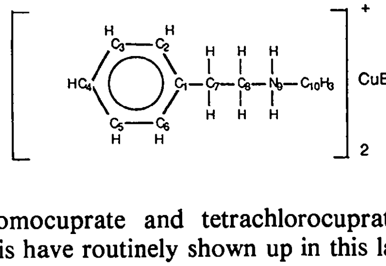
Abstract. $[C_{11}H_{18}N_2][CuBr_4].H_2O$, $M_r = 579{\cdot}5$, monoclinic, $P2_1/c$, $a = 7{\cdot}413\ (2)$, $b = 16{\cdot}459\ (10)$, $c = 14{\cdot}961\ (8)$ Å, $\beta = 104{\cdot}78\ (4)°$, $V = 1765$ Å^3, $Z = 4$, $D_x = 2{\cdot}18$ g cm^{-3}, $F(000) = 1108$, $\mu = 102{\cdot}1$ cm^{-1}, Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å), $T = 293$ K. 2720 unique reflections of which 1863 had $F \geq 3\sigma(F)$ were refined to a final $R = 0{\cdot}0588$ for the 3σ data set ($wR = 0{\cdot}0511$). The structure consists of discrete $CuBr_4^{2-}$ anions, *N*-benzylpiperazinium cations and water molecules, held together in the lattice with hydrogen bonding. The $CuBr_4^{2-}$ ion has nearly tetrahedral geometry and is therefore different from other $CuCl_4^{2-}$ and $CuBr_4^{2-}$ ions seen in similar compounds. $[NH_2(CH_3)(C_8H_9)]_2[CuBr_4]$, $M_r = 655{\cdot}6$, monoclinic, $C2/c$, $a = 25{\cdot}98\ (2)$, $b = 9{\cdot}045\ (5)$, $c = 10{\cdot}986\ (5)$ Å, $\beta = 111{\cdot}08\ (4)°$, $V = 2408{\cdot}6$ Å^3, $Z = 4$, $D_x = 1{\cdot}81$ g cm^{-3}, $F(000) = 1276$, $\mu = 74{\cdot}9$ cm^{-1}, Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å), $T = 293$ K. 1130 unique reflections, 748 reflections with $F > 3\sigma(F)$ were refined to a final $R = 0{\cdot}0618$ for the 3σ set ($wR = 0{\cdot}0595$). The structure consists of discrete $CuBr_4^{2-}$ anions and methyl(2-phenylethyl)ammonium cations. The $CuBr_4^{2-}$ anion has geometry intermediate between square planar and tetrahedral. The crystal packing shows distinct layers of anions and cations, held together in the lattice with hydrogen bonding.

Introduction. A series of copper chloride and copper bromide salts containing quasi-planar $Cu_nX_{2n+2}^{2-}$ ions has been sought in order to study their magnetic and structural correlations (Geiser, Willett, Lindbeck & Emerson, 1986). The structure of methyl(2-phenylethyl)ammonium heptachloromonoethanolatetricuprate(II) has been studied (Grigereit, 1986; Grigereit, Ramakrishna, Place, Willett, Pellacani, Manfredini, Menabue, Bonamartini-Corradi & Battaglia, 1987) and shown to contain the unusual $Cu_3Cl_7(EtOH)^-$ anion. In an attempt to make the analogous bromide salt, the crystalline product turned out to be bis[methyl-(2-phenylethyl)ammonium] tetrabromocuprate(II), hereafter $(NMPH)_2CuBr_4$ (I).

(I)



Tetrabromocuprate and tetrachlorocuprate anions such as this have routinely shown up in this laboratory in the attempted preparation of such oligomeric species, resulting in a separate study of the varying geometries

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