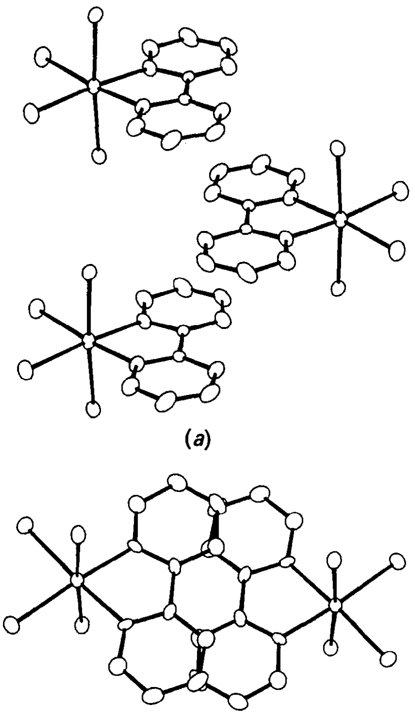
(a)

(b)

Fig. 2. The anion stacking as viewed (*a*) normal to the **c** direction and (*b*) parallel to the **c** direction.

tions (C—H = 0·95 Å) with fixed isotropic *B* values of 1·2 times the *B* value of the attached C atoms. All non-H atoms were refined anisotropically for a total of 83 parameters. $R = 0·051$, $wR = 0·062$, GOF = 1·3, for reflections with $I > 3\sigma(I)$ where non-Poisson $w^{-1} = [\sigma^2(I) + (0·06I)^2]/4F^2$. Final $(\Delta/\sigma)_{\max} < 0·01$, $\Delta\rho_{\max} = 1·4\,(1)$ (two peaks within 1 Å of the Ru atom) and $\Delta\rho_{\min} = -0·7\,(1)$ 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 atomic coordinates and Table 2 selected bond distances and angles. Fig. 1 shows the anion with the numbering scheme, and Fig. 2 shows the overlap of the anions parallel to the *c* axis of the crystal.

Related literature. The utility of this anion has been extensively studied by Dwyer, Goodwin & Gyarfas (1963). Structural aspects of ruthenium complexes with bipyridine ligands have been discussed by Durham, Wilson, Hodgson & Meyer (1980).

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* Tables of H-atom coordinates, anisotropic temperature factors and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52239 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of $(H_4cyclam)^{4+}.ReCl_6^{2-}.2Cl^-.4(CH_3)_2SO$

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Abstract. 1,4,8,11-Tetraazoniacyclotetradecanehexachlororhenate(IV) dichloride–dimethyl sulfoxide (1/4), $[C_{10}H_{28}N_4][Cl_6Re]Cl_2.4C_2H_6OS$, $M_r = 986·7$, triclinic, $P\bar{1}$, $a = 9·6478\,(9)$, $b = 9·9716\,(12)$, $c = 10·9887\,(9)$ Å, $\alpha = 66·470\,(7)$, $\beta = 89·033\,(4)$, $\gamma = 84·813\,(4)$°, $V = 965·1$ Å^3, $Z = 1$, $D_x = 1·697$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 3·987$ mm^{-1}, $F(000) = 495$, $T = 295$ K, $R = 0·0228$ for 2480 unique observed reflections. The quadruply protonated cyclam cation and the $ReCl_6^{2-}$ anion lie

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Table 1. *Atomic coordinates and isotropic thermal parameters with e.s.d.'s*

	x	y	z	U_{iso}(Å^2)
Re	0·0000	0·0000	0·0000	0·02758 (13)
Cl(1)	0·23591 (10)	0·03998 (12)	−0·04801 (10)	0·0503 (5)
Cl(2)	0·01845 (9)	−0·18755 (10)	−0·08070 (9)	0·0410 (5)
Cl(3)	0·06516 (11)	−0·17362 (11)	0·21517 (9)	0·0444 (5)
Cl(4)	0·56272 (11)	0·68903 (10)	−0·45376 (9)	0·0449 (5)
C(3)	−0·7090 (3)	1·1075 (4)	0·2615 (3)	0·0429 (8)
N(4)	−0·5690 (3)	1·1631 (3)	0·25266 (24)	0·0336 (7)
C(5)	−0·4488 (4)	1·0475 (3)	0·2975 (3)	0·0399 (8)
C(6)	−0·3158 (3)	1·1145 (3)	0·3005 (3)	0·0383 (8)
C(7)	−0·1949 (3)	1·0043 (4)	0·3691 (3)	0·0415 (8)
N(8)	−0·2201 (3)	0·9099 (3)	0·5141 (3)	0·0361 (7)
C(9)	−0·2496 (3)	0·9905 (3)	0·6012 (3)	0·0406 (8)
S(1)	0·51187 (10)	0·41810 (11)	−0·13305 (9)	0·0434 (5)
O(1)	0·4641 (4)	0·3373 (4)	0·0038 (3)	0·0810 (8)
C(11)	0·69356 (21)	0·4303 (3)	−0·1240 (3)	0·0675 (8)
C(12)	0·5172 (3)	0·29278 (24)	−0·21146 (23)	0·0699 (8)
S(2)	0·04155 (11)	0·57582 (12)	0·64198 (12)	0·0611 (6)
O(2)	0·0201 (3)	0·7389 (3)	0·6030 (3)	0·0617 (8)
C(21)	0·1089 (3)	0·5010 (3)	0·80753 (21)	0·0752 (8)
C(22)	0·19509 (24)	0·5477 (3)	0·56074 (25)	0·0893 (8)

Table 2. *Bond lengths* (Å), *angles* (°) *and torsional angles* (°) *with e.s.d.'s*

Re—Cl(1)	2·3607 (11)	C(7)—N(8)	1·522 (4)
Re—Cl(2)	2·3610 (10)	N(8)—C(9)	1·485 (4)
Re—Cl(3)	2·3597 (10)	S(1)—O(1)	1·483 (4)
C(3)—N(4)	1·493 (4)	S(1)—C(11)	1·775 (3)
C(3)—C(9′)	1·504 (5)	S(1)—C(12)	1·776 (3)
N(4)—C(5)	1·497 (4)	S(2)—O(2)	1·502 (3)
C(5)—C(6)	1·507 (5)	S(2)—C(21)	1·777 (3)
C(6)—C(7)	1·508 (5)	S(2)—C(22)	1·775 (3)
Cl(1)—Re—Cl(2)	90·35 (4)	C(7)—N(8)—C(9)	115·8 (3)
Cl(1)—Re—Cl(3)	90·15 (4)	C(7)—N(8)—H(81)	118·6 (5)
Cl(2)—Re—Cl(3)	90·37 (3)	C(7)—N(8)—H(82)	103·9 (4)
N(4)—C(3)—C(9′)	115·3 (3)	C(9)—N(8)—H(81)	101·4 (4)
C(3)—N(4)—C(5)	115·4 (3)	C(9)—N(8)—H(82)	92·2 (4)
C(3)—N(4)—H(41)	107·5 (4)	H(81)—N(8)—H(82)	122·3 (6)
C(3)—N(4)—H(42)	112·9 (4)	C(3)—C(9′)—N(8′)	112·8 (3)
C(5)—N(4)—H(41)	108·4 (4)	O(1)—S(1)—C(11)	107·80 (17)
C(5)—N(4)—H(42)	101·8 (4)	O(1)—S(1)—C(12)	106·07 (17)
H(41)—N(4)—H(42)	110·7 (6)	C(11)—S(1)—C(12)	98·43 (13)
N(4)—C(5)—C(6)	110·7 (3)	O(2)—S(2)—C(21)	105·64 (16)
C(5)—C(6)—C(7)	114·3 (3)	O(2)—S(2)—C(22)	105·17 (16)
C(6)—C(7)—N(8)	113·7 (3)	C(21)—S(2)—C(22)	98·60 (13)
H(31)—C(3)—N(4)—H(41)	49·9 (6)	H(41)—N(4)—C(5)—C(6)	−52·2 (5)
H(31)—C(3)—N(4)—H(42)	−72·4 (6)	H(42)—N(4)—C(5)—C(6)	64·5 (5)
H(32)—C(3)—N(4)—H(41)	−175·5 (6)	N(4)—C(5)—C(6)—C(7)	168·8 (3)
H(32)—C(3)—N(4)—H(42)	62·2 (6)	C(5)—C(6)—C(7)—N(8)	−60·5 (4)
C(9′)—C(3)—N(4)—C(5)	60·7 (4)	C(6)—C(7)—N(8)—C(9)	−59·5 (4)
C(9′)—C(3)—N(4)—H(41)	−60·4 (5)	C(6)—C(7)—N(8)—H(81)	61·4 (6)
C(9′)—C(3)—N(4)—H(42)	177·3 (5)	C(6)—C(7)—N(8)—H(82)	−158·9 (5)
N(4)—C(3)—C(9′)—N(8′)	70·4 (4)	H(81′)—N(8′)—C(9′)—C(3)	−43·0 (5)
C(3)—N(4)—C(5)—C(6)	−172·8 (3)	H(82′)—N(8′)—C(9′)—C(3)	80·6 (5)

Primed atoms are related to their unprimed equivalents by inversion through $(-\frac{1}{2}, 1, \frac{1}{2})$.

respectively across and on crystallographic inversion centres. The conformation of the cyclam cation is determined by N—H···O and N—H···Cl interactions, part of an extensive network of hydrogen bonding.

Experimental. The compound prepared by reaction of K_2ReCl_6 and 1,4,8,11-tetraazacyclotetradecane (cyclam) in water, crystals obtained from dmso/CH_2Cl_2. Pale-green columnar crystal, 0·73 × 0·29 × 0·33 mm, Stadi-4 four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, cell parameters from 2θ values of 34 reflections measured at $\pm\omega$ (26 $< 2\theta <$ 45°). For data collection, ω–2θ scans with ω-scan width $(0{\cdot}99 + 0{\cdot}347\tan\theta)°$, $2\theta_{max} = 45°$, h $-10\rightarrow 10$, k $-9\rightarrow 10$, l $0\rightarrow 11$, slight crystal decay (*ca* 8%) corrected for during processing, initial absorption correction using ψ scans, 2528 unique reflections, giving 2480 with $F > 6\sigma(F)$ for structure solution [from a Patterson synthesis (Re) followed by iterative cycles of least-squares refinement and

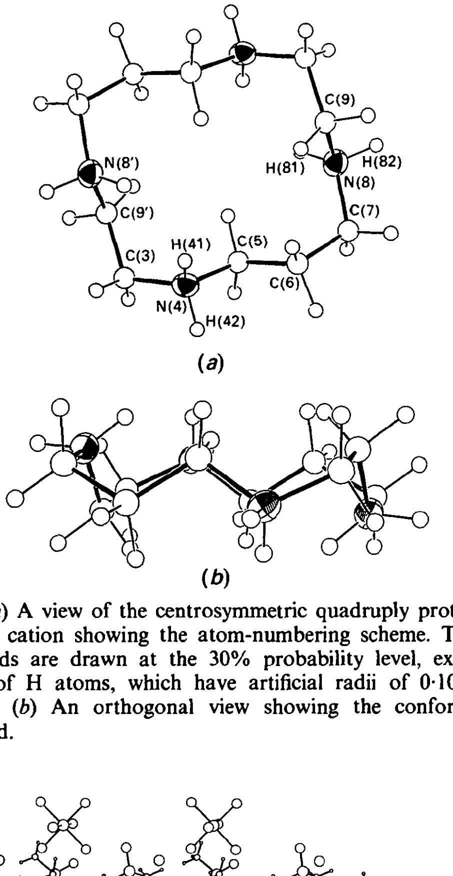

Fig. 1. (*a*) A view of the centrosymmetric quadruply protonated cyclam cation showing the atom-numbering scheme. Thermal ellipsoids are drawn at the 30% probability level, excepting those of H atoms, which have artificial radii of 0·10 Å for clarity. (*b*) An orthogonal view showing the conformation adopted.

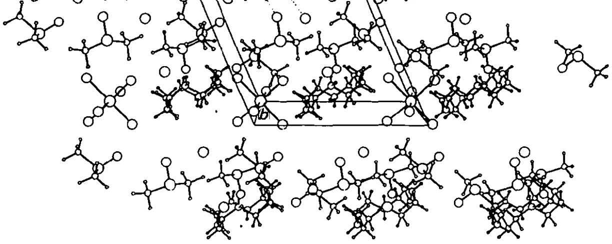

Fig. 2. A packing diagram of the structure showing the unit-cell contents and part of the extensive hydrogen-bonding network.

difference Fourier synthesis] and refinement [using full-matrix least squares on F (Sheldrick, 1976)]. At isotropic convergence, final absorption corrections (min. 0·895, max. 1·171) applied empirically using *DIFABS* (Walker & Stuart, 1983). C—H and N—H bonds were constrained to be 1·08 and 1·00 Å, respectively; HCH and HCS angles in the dmso molecules were constrained to be tetrahedral. At final convergence, $R = 0{\cdot}0228$, $wR = 0{\cdot}0338$, $S = 1{\cdot}164$ for 257 refined parameters, $(\Delta/\sigma)_{\max}$ in final cycle 0·66, max. and min. residues in final ΔF synthesis 0·83, $-0{\cdot}73$ e Å^{-3} respectively. The weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}002487F^2$ gave satisfactory agreement analyses. Scattering factors were inlaid (Sheldrick, 1976) except for Re (Cromer & Mann, 1968). Atomic coordinates are listed in Table 1, while selected bond lengths, angles and torsion angles appear in Table 2.* The atom-numbering scheme for the quadruply protonated cyclam is shown in Fig. 1, which was generated using *ORTEP* (Mallinson & Muir, 1985): a packing diagram of the structure, produced using *PLUTO* (Motherwell, 1976), appears as Fig. 2. Molecular-geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

Related literature. Diprotonated cyclam has been reported as the diperchlorate salt (Nave & Truter, 1974). The complex *trans*-$[Re(O)_2(cyclam)]Cl.2(BPh_3.H_2O)$ (Blake, Greig & Schröder, 1988), containing neutral cyclam coordinated to dioxo-Re^V, also has an extensive hydrogen-bonding network.

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Diaquabis(oxo-5 prolinato)zinc(II)

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Abstract. $[Zn(C_5H_6NO_3)_2(H_2O)_2]$, $M_r = 357{\cdot}6$, monoclinic, $C2$, $a = 14{\cdot}834\ (8)$, $b = 5{\cdot}888\ (3)$, $c = 7{\cdot}920\ (6)$ Å, $\beta = 98{\cdot}34\ (5)^\circ$, $V = 684\ (1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}735$ Mg m^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}7107$ Å, $\mu = 1{\cdot}88$ mm^{-1}, $F(000) = 368$, $T = 293\ (1)$ K, $R = 0{\cdot}016$ for 1070 independent observed reflections. The mol-