

Chromium(III) Complexes of Macrocyclic Ligands. I. Crystal Structure of *trans*-Bis(*O*-carbamato)(1,4,8,11-tetraazacyclotetradecane)chromium(III) Perchlorate Sesquihydrate

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trans-[Cr(cyclam)(OCONH₂)₂]₂ClO₄·1½H₂O crystallizes in the *C2/c* space group with $a = 15.174(6)$ Å, $b = 17.257(10)$ Å, $c = 16.964(8)$ Å, $\beta = 109.05(3)^\circ$. The structure contains two independent centrosymmetrical complex ions which are almost identical except for the orientation of the carbamate ligands. The macrocyclic tetraamine ligand has the 5-membered ring in the *gauche* and the 6-membered ring in the *chair* conformation. The perchlorate ion is disordered. The final *R*-values were $R \sim 0.047$ and $R_w \sim 0.053$ (4817 refl.).

The cyclic tetraamine 1,4,8,11-tetraazacyclotetradecane (cyclam) forms complexes with most transition metal ions but until recently only compounds with the ligand in the *cis* configuration were readily obtained for chromium(III).^{1,2} During preparative work on the *trans* series of complexes an orange compound, present as an impurity in crude *trans*-[Cr(cyclam)(NH₃)₂]₂Cl₃ prepared from *trans*-[Cr(cyclam)Cl₂]₂Cl and NH₃(l), was isolated. Various types of evidence suggested that coordinated carbamate could be present, a situation so far not encountered in chromium(III) chemistry. To settle this question, and also to determine the cyclam ligand conformation, which could possibly explain both the increased robustness of *trans*-cyclam' complexes compared to other *trans*-tetraamine chromium(III) complexes, and also the unusually high spectrochemical Δ -value for the cyclam ligand, this structural investigation was undertaken.

EXPERIMENTAL

Suitable crystals of *trans*-[Cr(cyclam)-OCONH₂)₂]₂ClO₄·1½H₂O for the crystallographic work were obtained by slowly cooling a saturated aqueous solution from 50 to 0°C. The yield is low, probably due to decomposition reactions. The identity between this recrystallized product and the analyzed starting material was established through Hägg-Guinier powder diagrams. The monoclinic orange crystals are strongly pleochroitic and of different habitus with well-developed faces, where a prism after [010] and plates with the well-developed form {010} often occur.

The crystals could be assigned to the space group *Cc* or *C2/c* through Weissenberg and precession photographs. Intensity data for a 0.28 × 0.18 × 0.18 mm³ large crystal were collected on a Picker FACS-1 diffractometer with graphite monochromated MoK α radiation and 4817 independent reflections including 1008 classified as 'less thans' [$I < 3\sigma(I)$] were measured. The data were corrected for crystal decay, but not for absorption.

Crystal data. *trans*-[Cr(C₁₀H₂₄N₄)(OCONH₂)₂]₂·ClO₄·1½H₂O. Monoclinic *C2/c* (*Cc*). $a = 15.174(6)$ Å, $b = 17.257(10)$ Å and $c = 16.964(8)$ Å; $\beta = 109.05(3)^\circ$. $Z = 8$. $\mu(\text{MoK}\alpha) = 7.63 \text{ cm}^{-1}$. $D_m = 1.564$, $D_x = 1.579 \text{ g cm}^{-3}$. $V = 4199 \text{ \AA}^3$. $M = 498.9 \text{ g mol}^{-1}$. $F(000) = 2096$.

Structure determination and refinement. The structure was solved by conventional methods. Possible positions of Cr and Cl atoms were indicated in the Patterson function, and a subsequent Fourier synthesis carried out in the space group *Cc* revealed the positions of all C, N and O atoms. Least squares refinement with anisotropic temperature factors for all these atoms gave an agreement factor of $R = 0.073$. The result of this calculation suggested

that the cations were centrosymmetrical and the space group possibly $C2/c$. A difference electron density synthesis clearly showed the hydrogen atoms at reasonable positions. Weighted least squares refinement in the space group $C2/c$ including hydrogen atoms with isotropic temperature factors and with weights derived from counting statistics but modified to downweigh the most intense reflections, *i.e.*:

$$1/w \sim \sigma^2(F_{\text{obs}}) + 0.005F_{\text{obs}} + 0.00003F_{\text{obs}}^2$$

gave agreement factors $R=0.054$ and $R_w=0.061$. Large temperature factors of the perchlorate oxygen atoms indicated a static or dynamic disorder of this

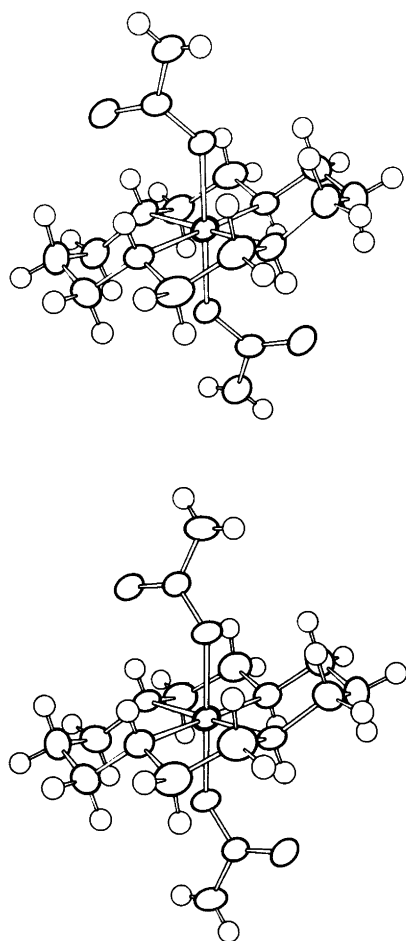


Fig. 1. The two different complex cations. Upper drawing: Cr1-complex, lower drawing: Cr2-complex. Cr-, O-, N- and C-atoms are drawn as 50% probability ellipsoids, H-atoms as spheres with a radius of 0.15 Å.

Table 1. Fractional coordinates for $trans-[Cr(\text{cyclam})(\text{OCONH}_2)_2]\text{ClO}_4 \cdot 1\frac{1}{2}\text{H}_2\text{O}$.

Atom	X	Y	Z
Cr1	.25000	.25000	.50000
N101	.12568 (15)	.19091 (13)	.45412 (13)
N102	.25910 (15)	.27423 (13)	.38402 (13)
N103	.07261 (19)	.43664 (15)	.47577 (19)
O101	.18324 (11)	.34652 (10)	.50346 (10)
O102	.09373 (13)	.36748 (11)	.37123 (11)
Cl01	.11414 (21)	.14213 (17)	.37969 (19)
Cl02	.12759 (21)	.18742 (20)	.30800 (18)
Cl03	.22730 (21)	.21190 (18)	.32075 (17)
Cl04	.35636 (20)	.29904 (18)	.39889 (19)
Cl05	.38445 (20)	.35193 (17)	.47381 (20)
Cl06	.11668 (16)	.38155 (13)	.44749 (16)
H101N	.0860 (18)	.2245 (15)	.4416 (15)
H102N	.2240 (21)	.3125 (18)	.3663 (19)
H103M	.0257 (25)	.4565 (21)	.4436 (22)
H103N	.0803 (21)	.4377 (17)	.5238 (20)
H101A	.1594 (20)	.0985 (17)	.3959 (17)
H101B	.0515 (22)	.1202 (17)	.3630 (18)
H102A	.1068 (24)	.1571 (21)	.2602 (23)
H102B	.0902 (22)	.2359 (18)	.2956 (19)
H103A	.2709 (19)	.1687 (16)	.3407 (17)
H103B	.2327 (20)	.2315 (17)	.2664 (19)
H104A	.3930 (21)	.2508 (17)	.4085 (18)
H104B	.3609 (21)	.3251 (18)	.3529 (20)
H105A	.4503 (23)	.3712 (18)	.4871 (18)
H105B	.3458 (20)	.3960 (17)	.4651 (17)
Cr2	.75000	.25000	.50000
N201	.73479 (16)	.13890 (13)	.45301 (15)
N202	.88179 (14)	.23125 (13)	.58232 (14)
N203	.88402 (20)	.33755 (17)	.34217 (16)
O201	.79575 (11)	.28499 (11)	.41061 (10)
O202	.94716 (13)	.25555 (12)	.44765 (13)
C201	.77411 (22)	.07604 (17)	.51431 (21)
C202	.87609 (22)	.08820 (19)	.56125 (24)
C203	.89981 (21)	.15456 (19)	.62311 (20)
C204	.90274 (22)	.29709 (19)	.64091 (17)
C205	.86591 (21)	.37124 (19)	.59398 (20)
C206	.87771 (17)	.29038 (14)	.40293 (15)
H201N	.7602 (22)	.1403 (19)	.4202 (20)
H202N	.9141 (19)	.2340 (16)	.5534 (17)
H203M	.9389 (24)	.3444 (18)	.3415 (20)
H203N	.8404 (22)	.3598 (17)	.3182 (18)
H201A	.7378 (20)	.0726 (18)	.5522 (19)
H201B	.7645 (24)	.0270 (22)	.4840 (22)
H202A	.9002 (23)	.0406 (20)	.5889 (20)
H202B	.9082 (22)	.0922 (19)	.5231 (20)
H203A	.8655 (21)	.1517 (17)	.6615 (19)
H203B	.9682 (23)	.1517 (18)	.6593 (19)
H204A	.8767 (22)	.2857 (18)	.6830 (20)
H204B	.9680 (22)	.3021 (18)	.6704 (19)
H205A	.8761 (19)	.4152 (18)	.6318 (17)
H205B	.8949 (19)	.3822 (16)	.5523 (18)
C41	.6903 (4)	.4975 (6)	.2115 (7)
O11	.7551 (11)	.4532 (12)	.1875 (11)
O12	.7209 (6)	.5111 (6)	.2981 (6)
O13	.6789 (7)	.5697 (7)	.1683 (6)
O14	.6009 (6)	.4621 (5)	.1918 (6)
C42	.3121 (6)	.5021 (7)	.2952 (8)
O21	.2522 (10)	.4404 (13)	.3063 (10)
O22	.3075 (14)	.4958 (9)	.2125 (9)
O23	.2720 (6)	.5755 (6)	.2991 (6)
O24	.3965 (7)	.4945 (7)	.3550 (8)
O1	.41779 (25)	.05982 (24)	.34304 (20)
H101W	.3680 (42)	.0498 (35)	.3309 (36)
H102W	.4402 (35)	.0319 (29)	.3119 (31)
O2	.0	.47101 (18)	.25000
H201W	.0344 (21)	.4437 (17)	.2844 (18)

anion, in agreement with parameters for two rather different perchlorate ions in the initial Cc space group refinements. Further calculations were therefore carried out with two perchlorate ions, each of

a population of $\frac{1}{2}$ in $C2/c$. This gave the final agreement factors $R \sim 0.047$ and $R_w \sim 0.053$ without significant changes of the remaining parameters. The scattering factors for Cr(0) and the corrections for anomalous dispersion for Cr and Cl were taken from Ref. 3. The scattering factors for the other atoms, *i.e.* Cl(0), O(0), N(0), C(0) and H(0), were those of the XRAY system,⁴ which was used for the calculations. The determined positional parameters are given in Table 1. Lists of observed and calculated structure factors as well as temperature factors are available from the authors.

RESULTS AND DISCUSSION

Two chromium atoms are placed at two non-equivalent centres of symmetry. This gives two independent complex cations in which, however, bond lengths and angles in both the cyclam and carbamate ligand sets are almost identical. This is shown in Figs. 1 and 2, and Table 2.

The macrocyclic ligand has a conformation in which the five-membered ring has the *gauche*, and the six-membered ring the *chair* conformation, respectively. It has a crystallographic symmetry centre and is close to having a mirror plane orthogonal to the CrN_4 -plane and containing the symmetry centre and the middle carbon atom of the six-membered ring. This conformation is predicted by semi-empirical calculations to have the lowest energy⁵ and is also found in a number of other compounds including $(cyclamH_2)(ClO_4)_2$,⁶ *trans*-

$[Ni(cyclam)Cl_2]$,⁷ $[Co(cyclam)](ClO_4)_2$ ⁸ and $[Cu(cyclam)](ClO_4)_2$.⁹

The bond lengths in the present complex and in the uncoordinated diprotonated ligand are almost identical, *cf.* Table 3. The average value for the Cr–N bond length of 2.059 Å is very close to both the value of 2.07 Å calculated semiempirically to be the “ideal” metal–nitrogen bond length in *trans*-‘cyclam’ complexes⁵ and also to the experimentally found chromium–nitrogen bond lengths in a number of complexes containing ammonia, 1,2-ethanediamine (en) and 1,3-propanediamine (tn), *cf.* Table 4. The data of Table 4 do not support a correlation between bond lengths and spectrochemical Δ -values, and the high Δ -value of *trans*-‘cyclam’ complexes of chromium(III) cannot therefore reasonably be ascribed to a statically strained metal–nitrogen bond, but is more likely the result of a dynamically very rigid ligand.

The N–Cr–N angles deviate from the average values found in a number of chelate diamine complexes, *cf.* Table 4, but accord with a greater flexibility of 6-membered chelate rings *vs.* 5-membered.

In solution the axial ligands in *trans*-‘cyclam’ complexes show an increased substitution inertness compared to that of most other tetraamine chromium(III) complexes.¹⁰ Both the pentaamine- and pentaquachromium(III) series of complexes are known to react by an associative interchange mechanism,¹¹ but plausible transition state structures¹² for *trans*-‘cyclam’ complexes reacting by

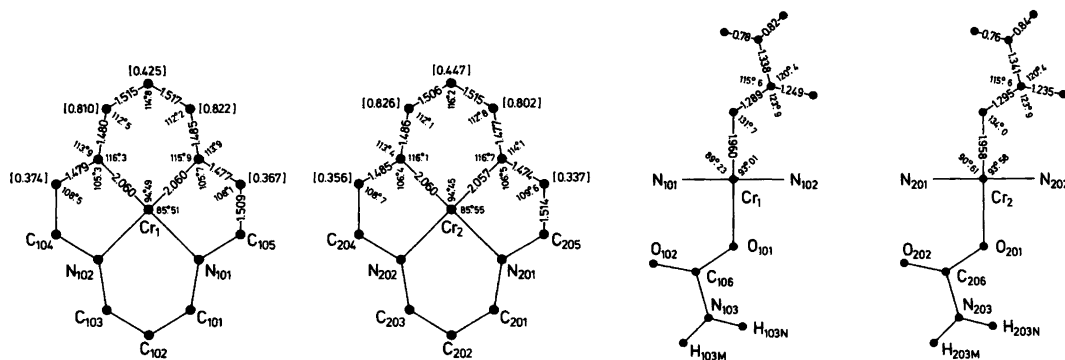


Fig. 2. Numbering scheme and bond lengths and angles for left: The two macrocyclic tetraamine rings, with carbon atom distances from the CrN_4 -plane shown in square brackets, and right: The two carbamate ligands for which the O–Cr–N angles are to those two nitrogen atoms of the macrocyclic tetraamine ligand which are closest to the uncoordinated carbamate oxygen atom, and are donor atoms of a 6-membered chelate ring, *cf.* Fig. 1. Standard deviations are almost constant for each bond and angle type and are: $\sigma(Cr-X) \sim 0.002$ Å ($X=N,O$); $\sigma(C-X) \sim 0.004$ Å ($X=C,N,O$); $\sigma(X-Cr-Y) \sim 0.09^\circ$ ($X,Y=N,O$); and $\sigma(X-Y-Z) \sim 0.2^\circ$ ($X,Y,Z=C,N,O$).

Table 2. Summary of C–H, N–H, O–H and Cl–O bond lengths, [Å], and angles [°] in *trans*-[Cr(cyclam)(OCONH₂)₂]ClO₄·1½H₂O.

	No. of indep. determinations.	Average (Std.dev.)	Range
<i>d</i> (C–H)	20	0.970(7)	0.92–1.02
∠(H–C–H)	10	106.6(9)	101.6–110.9
∠(H–C–N)	12	108.1(5)	105.4–111.1
∠(H–C–C)	28	110.2(4)	105.2–113.6
<i>d</i> (N–H)	8	0.803(12)	0.76–0.84
∠(Cr–N–H)	4	104.2(11)	103.2–105.6
∠(C–N–H)	8	108.0(7)	106.5–110.2
<i>d</i> (O–H)	3	0.800(24)	0.74–0.86
∠(H–O–H)	2	107.1(23)	105.7–107.4
<i>d</i> (Cl–O)	8	1.409(6)	1.355–1.452
∠(O–Cl–O)	12	109.4(3)	103.7–118.3

Table 3. Comparison of bond lengths [Å] in *trans*-[Cr(cyclam)(OCONH₂)₂]⁺ with literature data for [cyclamH₂]²⁺.

	No. of indep. determinations.	Average	Range	Ref.
C–N	{ 8	1.480(2)	1.474–1.486	This work
	{ 4	1.482(3)	1.464–1.504	6
C–C	{ 6	1.513(2)	1.506–1.517	This work
	{ 3	1.509(4)	1.503–1.520	6
N–N	{ 4	2.059(2)	2.057–2.060	This work
	{ 2	2.045	1.98 and 2.11	6

such a mechanism are seen to be sterically hindered, particularly by the axial hydrogen atoms of two of the carbon atoms in the 6-membered rings, *cf.* Fig. 1. The increased robustness of *one* of the axial ligands in *trans*-difluorido-(1,4,8,11-tetraazaundecane)-chromium(III), which is similarly shielded by the hydrogen atoms of the equatorial amine ligand¹³ supports this explanation.

The two independent carbamate ligands are planar within the estimated uncertainties and close to being congruent, but their orientations towards the cyclam ligand are different and seem to be dominated by differences in the hydrogen bonds, primarily to the uncoordinated oxygen atom as described in Table 5. The distances from the coordinated oxygens to chromium are the same, *cf.* Fig. 2 B, and the average value, 1.959 Å, is close to the values found for a number of other chromium oxygen bond lengths, *i.e.* Cr–OH₂ ~ 2.00₇ Å, Cr–OH

Table 4. Comparison with selected average literature values for Cr–N bond lengths, N–Cr–N angles and spectrochemical Δ-values of chromium(III) complexes containing ammonia and 5- and 6-membered chelate diamine ligand rings.

Amine	<i>d</i> (Cr–N) [Å]	∠(N–Cr–N) [°]	Δ [μm ⁻¹]
cyclam	2.059	85.53 94.47	~ 2.44
a ^a	2.06 ₈	–	2.111 ^d
en ^b	2.07 ₇	82.4	2.166 ^e
tn ^c	2.08 ₉	– 89.5	2.164 ^e

^a a = ammonia. Data for [Cr₆][CuCl₅]¹⁷ [Cr₆][CuBr₅]¹⁸ and [Cr₆][Ni(CN)₅].2H₂O.¹⁹
^b en = 1,2-ethanediamine. Data for [Cren₃]₃.H₂O (118 K),²⁰ [Cren₃](SCN)₃.¾H₂O,²¹ (+)_D-[Cren₃]-Cl₃.2H₂O (123 K),²² ¾H₂O,²¹ (-)_D-[Cren₃](SCN)₃,²³ [Cren₂tn]Br₃.H₂O²⁴ and [Cren₂tn]₃.H₂O.²⁴
^c tn = 1,3-propanediamine. Data from Ref. 24 and for [Cr₃tn₃][Ni(CN)₅].2H₂O.¹⁹ ^dRef. 25. ^eRef. 26.

Table 5. Selected intermolecular hydrogen bonds with $d(\text{H}\cdots\text{B})$ less than 2.75 Å in *trans*-[Cr(cyclam)-(OCONH₂)₂]₂ClO₄·1½H₂O, cf. Fig. 3.

A—H⋯B	$d(\text{A—H})$ [Å]	$d(\text{H}\cdots\text{B})$ [Å]	$d(\text{A}\cdots\text{B})$ [Å]
N101—H101N⋯O202	0.81(3)	2.21(3)	2.898(3)
N102—H102N⋯O21	0.84(3)	2.53(4)	3.143(21)
⋯O11	0.84(3)	2.65(4)	3.298(21)
N103—H103M⋯O1	0.82(3)	2.64(3)	3.414(5)
—H103N⋯O1	0.78(4)	2.25(4)	3.030(5)
N201—H201N⋯O23	0.77(4)	2.40(4)	3.048(11)
⋯O13	0.77(4)	2.35(4)	3.023(13)
N202—H202N⋯O202 ^a	0.80(3)	2.05(3)	2.803(4)
N203—H203M⋯O102	0.84(4)	2.27(4)	3.100(4)
—H203N⋯O11	0.76(3)	2.71(3)	3.367(18)
⋯O21	0.76(3)	2.54(3)	3.219(17)
O1—H101W⋯O12	0.74(6)	2.22(6)	2.954(9)
⋯O22	0.74(6)	2.69(7)	3.418(20)
⋯O23	0.74(6)	2.55(5)	3.106(9)
—H102W⋯O2	0.86(6)	1.91(6)	2.771(5)
O2—H201W⋯O102	0.80(3)	1.96(3)	2.745(3)

^a This hydrogen bond is intramolecular.

(bridge)~1.95₃ Å and Cr—O (oxalat)~1.97₄ Å.*

In Table 6 selected bond lengths in the coordinated carbamate are compared with the analogous distances in the carbamate ion of ammonium-carbamate¹⁴ and in ethylcarbamate.¹⁵ In all three cases a planar carbamate group is found and the present carbamate ligand is seen to show intermediate bond lengths between the free anion and the organic derivative.

* Data for: *trans*-[Cr(OH₂)₄Cl₂]₂Cl₂H₂O,^{27,28} [Cr(chel)-(OH₂)OH]₂·4H₂O,²⁹ [Cr(Cl-dipic)(OH₂)OH]₂·2H₂O,²⁹ Na₄[Crox₂OH]₂·6H₂O,³⁰ K[Cr en ox₂]₂·KI₂H₂O³¹ and K₃[Cr ox₃]₂·3H₂O.³²

The variation in C—H, N—H and O—H bond lengths is as expected, and since it has been shown in accurate studies as *e.g.* that of urea¹⁶ that bond lengths involving hydrogen are highly dependent on the model used to interpret the data, the results in Table 2 will not be discussed further.

The present perchlorate salt contains only one independent perchlorate anion in the space group *C2/c*. The data were, however, as stated above, better represented using two anions, each with a population of ½, for which the average results are given in Table 2. The temperature factors of the oxygen atoms were rather large, but primarily in directions perpendicular to the Cl—O bond, in

Table 6. Comparison with average literature values for bond lengths [Å] in carbamate type derivatives.

	$d(\text{C—O}')$	$d(\text{C—O}'')$	$d(\text{C—N})$	Ref.
	1.28 ₄	1.28 ₄	1.36 ₁	14
	1.29 ₂	1.24 ₂	1.34 ₀	This work
	1.33 ₈	1.22 ₁	1.34 ₅	15

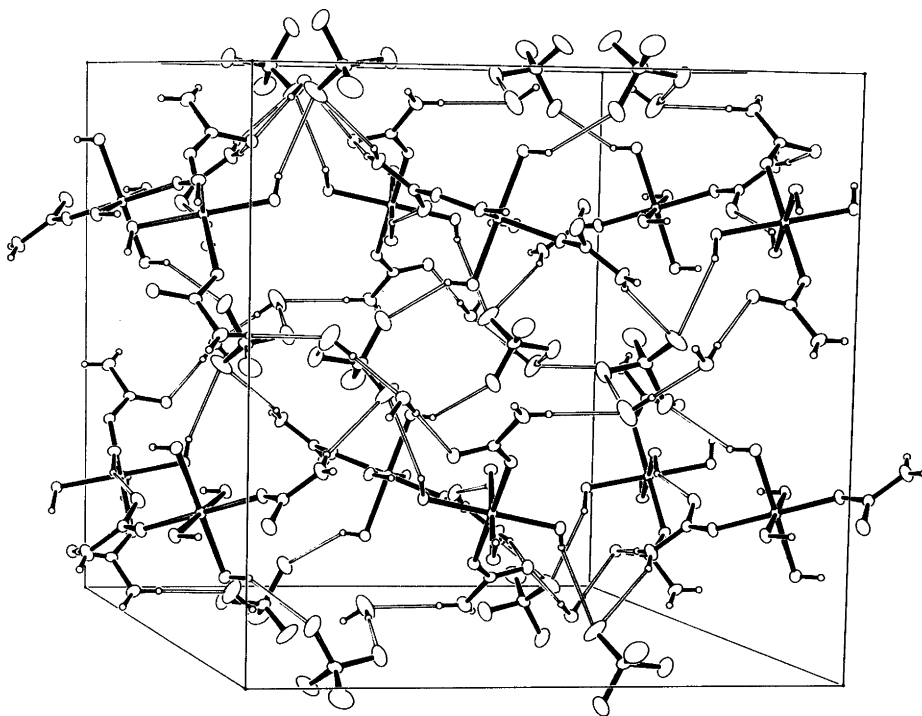


Fig. 3. Packing of the unit cell, *b*-axis vertical, showing the hydrogen bonds between the complex ions, the perchlorate ions and the water molecules, *cf.* Table 5. CH₂-groups of the 'cyclam' ligands are omitted and only the Cl1-perchlorate ion has been included. H-atoms are drawn as spheres with a radius of 0.10 Å, and all other atoms as 35% probability ellipsoids.

agreement with a statically or dynamically disordered anion.

The rather complicated net of hydrogen bonds between complex ions, perchlorate ions and water molecules is shown in Fig. 3. The corresponding distances are given in Table 5.

All hydrogen atoms bound to nitrogen or oxygen are seen to be involved in hydrogen bonding to various types of oxygen atoms. Rather short bonds are formed to both water and carbamate oxygens, whereas the bond to the disordered perchlorate oxygens are somewhat longer.

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