

Synthesis and X-Ray Crystal Structure Analysis of Δ, Λ - μ -Hydroxo- μ -trifluoroacetato-bis[bis(ethylenediamine)chromium(III)] Bromide Triperchlorate Monohydrate

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Reaction of the dinuclear complex cation Δ, Λ - $[(en)_2Cr(OH)_2Cr(en)_2]^{4+}$ in aqueous CF_3COOH led to the formation of the singly bridged species Δ, Λ - $[(CF_3COO)(en)_2Cr(OH)Cr(en)_2(CF_3COO)]^{3+}$ which was isolated as a perchlorate salt (91 %). This species hydrolyzed in aqueous solution to give the heterobridged cation Δ, Λ - $[(en)_2Cr(OH)(CF_3COO)Cr(en)_2]^{4+}$ which was isolated as a bromide salt (73 %). The latter cation was characterized by single-crystal structure analysis of the monobromide triperchlorate monohydrate salt. These crystals are orthorhombic, space group *Pnma* with $Z = 4$, unit cell dimensions: $a = 17.687(3)$; $b = 11.703(3)$; and $c = 15.304(4)\text{\AA}$. The structure was solved and refined to $R = 0.059$ from 1104 diffractometer-collected intensities. The complex ion contains a crystallographic mirror plane and the conformations of the chelate rings are $\lambda^2\Delta, \Lambda^2\delta$. The bridging six-membered ring is nearly planar. The Cr–Cr separation is $3.616(2)\text{\AA}$. The CF_3 group and the ClO_4^- ions are disordered.

During the last ten years, there has been considerable interest in hydroxo-bridged chromium(III) complexes. Many new dinuclear- as well as higher oligomeric species have been synthesized and characterized by X-ray crystal structure analysis.^{1–16} The magnetochemical properties of this class of compounds have been studied with special emphasis on the relationship between geometric structural parameters and the singlet-triplet energy separation of the ground levels, and significant progress has been made in this respect.^{17–20}

The hydrolytic behaviour of these complexes has also been studied extensively.^{4–15} The kinetics and thermodynamics of bridge cleavage and formation reactions of di- μ -hydroxo chromium(III) ammine and amine complexes have been studied in detail,^{4–8} and the observed trends have proven to be valid also for the related rhodium(III) and iridium(III) systems.^{21–23}

Bridge cleavage of the dihydroxo-bridged complexes in the presence of nucleophiles, X^{n-} , other

than water has also been examined, although to a lesser extent, and such reactions have recently been studied for the *meso* (Δ, Λ) isomers of the complexes $[(en)_2M(OH)_2M(en)_2]^{4+}$ ($M = Cr$ or Rh) and have led to the synthesis and characterization of novel doubly-bridged complexes of the type $[(en)_2M(OH)(X)M(en)_2]^{(5-n)+}$ with $X^{n-} = HCOO^-$, CH_3COO^- , SO_4^{2-} or amino acidato anions for $M = Cr(III)$,^{1,10,11} and $X = O_2^{2-}$ or O_2^- for $M = Rh(III)$.^{24,25} The present paper describes the reaction of Δ, Λ - $[(en)_2Cr(OH)_2Cr(en)_2]^{4+}$ with trifluoroacetate in acidic solution and reports the X-ray crystal structure of one of the isolated products, Δ, Λ - $[(en)_2Cr(OH)(CF_3COO)Cr(en)_2] Br \cdot (ClO_4)_3 \cdot H_2O$.

Experimental

Materials. Δ, Λ - $[(en)_2Cr(OH)_2Cr(en)_2](ClO_4)_4$ was prepared as described in the literature.⁹ All other reagents were of analytical grade.

Instruments. A Zeiss DMR 21 was used for all spectrophotometric measurements in the visible region. For the spectrophotometric data presented, the molar absorption coefficient ϵ is given in $l\ mol^{-1}\ cm^{-1}$ and the wavelength, λ , in nm. The molarity of solutions of the binuclear complexes is in all cases defined in terms of the number of moles of complex, rather than the number of moles of chromium(III), per litre of solution. An Enraf Nonius CAD4 diffractometer equipped with a graphite monochromator was used for the crystal structure determination.

Analysis. Chromium analyses by atomic absorption spectrophotometry were performed by Karen Jørgensen, Chemistry Department I, H. C. Ørsted Institute: C, H, N, F, Cl and Br analyses were performed by the Microanalytical Laboratory at the H. C. Ørsted Institute, Copenhagen.

Preparations. 1. Δ, Λ - μ -Hydroxo-bis[trifluoroacetatobis(ethylenediamine)chromium(III)] perchlorate monohydrate, Δ, Λ -[(CF₃COO)(en)₂Cr(OH)Cr(en)₂(CF₃COO)](ClO₄)₄·H₂O.

Trifluoroacetic acid (2M, 12 mL) was added to a solution of Δ, Λ -[(en)₂Cr(OH)₂Cr(en)₂](ClO₄)₄ (2.00 g, 2.58 mmol) in water (25 mL) at room temperature (~23 °C). The colour of the solution changed within about 10 min from reddish purple to red. After 1 h, a saturated solution of sodium perchlorate (10 mL) was added, whereupon, red crystals of the perchlorate salt precipitated. The crystals were filtered off, washed with 96 % ethanol, then with diethyl ether and dried in air. Yield 2.13 g (91 %). The product was pure as shown by reprecipitation: 2.00 g were dissolved in ice-cold 0.1 M hydrochloric acid (120 mL). The filtered solution was stirred and cooled in ice, and a saturated (room temperature) solution of sodium perchlorate (40 mL) was added. Microscopic red crystals of the perchlorate immediately precipitated and were isolated as above. Yield 1.44 g (72 %). The visible absorption spectra of both this and the former product were identical. Anal. [Cr₂(C₂H₈N₂)₄(OH)(C₂F₃O₂)₂](ClO₄)₃·H₂O: Cr, C, N, H, Cl. Spectral data in 0.1 M hydrochloric acid at 0 °C and extrapolated to the time of dissolution: (ϵ, λ)_{max} = (204,504), (115,377); (ϵ, λ)_{min} = (42.4,430). The same values (at t_0) were obtained in 0.1 M HCl or in 4 M HCl at 25 °C. In 2 M CF₃COOH at 25 °C, the spec-

trum was constant for at least 20 min, with spectral data identical to those given above.

2. Δ, Λ - μ -Hydroxo- μ -trifluoroacetato-bis[bis(ethylenediamine)chromium(III)] bromide tetrahydrate, Δ, Λ -[(en)₂Cr(OH)(CF₃COO)Cr(en)₂]Br₄·4H₂O.

A solution of Δ, Λ -[(CF₃COO)(en)₂Cr(OH)Cr(en)₂(CF₃COO)](ClO₄)₃·H₂O (5.0 g, 5.53 mmol) in 0.1 M HCl (200 mL) was kept at ~23 °C for 20 min and a saturated aqueous solution of sodium bromide (200 mL) was then added. After 5 min, the orange-red crystals were filtered off, washed with 96 % ethanol then diethyl ether and dried in air. This gave 3.52 g (73 %) of a pure product. Anal. [Cr₂(C₂H₈N₂)₄(OH)(C₂F₃O₂)₂]Br₄·4H₂O: Cr, C, N, H, Br. Spectral data in 0.1 M hydrochloric acid at 0 °C and extrapolated back to the time of dissolution: (ϵ, λ)_{max} = (204,503), (74,370); (ϵ, λ)_{sh} = (66,408); (ϵ, λ)_{min} = (59.8,431). Spectral data (at t_0) in 4 M HCl and in 0.1 M pyridine/0.1 M pyridinium chloride were identical to those obtained for 0.1 M HCl.

The visible absorption spectrum was the same for products reprecipitated by any of the following three procedures:

(a) The bromide salt (3.0 g) was dissolved in 0.1 M HCl (190 mL) at 0 °C. The filtered solution was stirred and cooled in ice, and a saturated (room temperature) solution of sodium bromide (190 mL) added. The resulting precipitate was isolated as above. Yield 2.70 g (90 %).

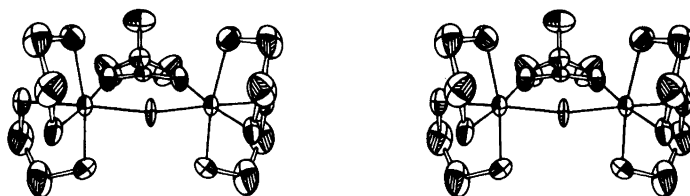
(b) The bromide salt (3.0 g) was reprecipitated as above, but at 25 °C. Yield 2.95 g (98 %).

(c) The bromide salt (3.0 g) was dissolved in 0.1 M HCl (190 mL) at 25 °C. After 2 min, the solution was cooled in ice and the complex precipitated as above. Yield. 2.35 g (78 %).

3. Δ, Λ - μ -Hydroxo- μ -trifluoroacetato-bis[bis(ethylenediamine)chromium(III)] bromide triperchlorate monohydrate, Δ, Λ -[(en)₂Cr(OH)(CF₃COO)Cr(en)₂]Br(ClO₄)₃·H₂O.

The tetrabromide (1.25 g, 1.44 mmol) was added to a mixture of 0.2 M HClO₄ (10 mL) and a saturated aqueous NaClO₄ solution (20 mL) at room temperature. The salt dissolved rapidly and the solution was cooled in ice with stirring. Crystallization began after a few minutes (it may be necessary to induce crystallization by scratching with a spatula) and the crystals were isolated by filtration after 10 min. They were washed with 96 % ethanol then diethyl ether and dried in air. This gave 0.95 g (76 %). Anal. [Cr₂(C₂H₈N₂)₄

Fig. 1. Stereoscopic ORTEP drawing of the complex cation (the labelling is shown in Fig. 2). The thermal ellipsoid encloses 50% probability. The cation has a crystallographic mirror plane and the conformation is Δ, Λ . Hydrogen atoms have been omitted.



$(\text{OH})(\text{C}_2\text{F}_3\text{O}_2)]\text{Br}(\text{ClO}_4)_3 \cdot \text{H}_2\text{O}$: Cr, C, N, H, Cl, Br (ionic).

The visible absorption spectrum was identical to that of the tetrabromide. The crystals which were used for the X-ray structure determination were made by this procedure but with the following modifications: only half the volume given above for the saturated aqueous NaClO_4 solution was used; the temperature was $\sim 15^\circ\text{C}$; seeding crystals from a previous experiment were used.

Crystal structure determination. The crystal used for X-ray investigation was prepared as described above under *Preparations*. X-ray diffraction photographs showed that the crystals were orthorhombic, and the systematic absences ($0kl$: $k+l$ odd, $hk0$: h odd) characterized the space group as $Pn2_1a$ (No. 33) or $Pnma$ (No. 62). Diffraction data were collected from a red prismatic crystal ($0.3 \times 0.25 \times 0.25$ mm) using $\text{MoK}\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$). Peaks were scanned in an ω - θ mode with the use of variable scan times determined on the basis of a rapid prescan, the maximum scan time employed being 180 s. A unique data set having $1.85^\circ \leq \theta \leq 22.5^\circ$ was collected.

The intensities and centering of 3 standard reflections were monitored after every 100 reflections, and the crystal was automatically recentered whenever crystal movement and/or intensity decline was detected. After 2080 reflections, the intensity declined rapidly and data collection was ceased. (About 50 reflections in the range $20^\circ < \theta < 22.5^\circ$ plus the 120 reflection are missing in the data set). The data were corrected for background counts, for a decline in intensity which amounted to 10%, and for Lorentz polarisation effects. No absorption correction was applied. A total of 1104 reflections with intensities greater than 2.5 times their e.s.d.s, as obtained from counting statistics, were considered ob-

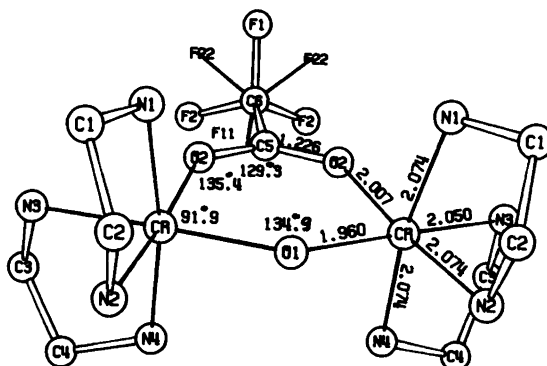
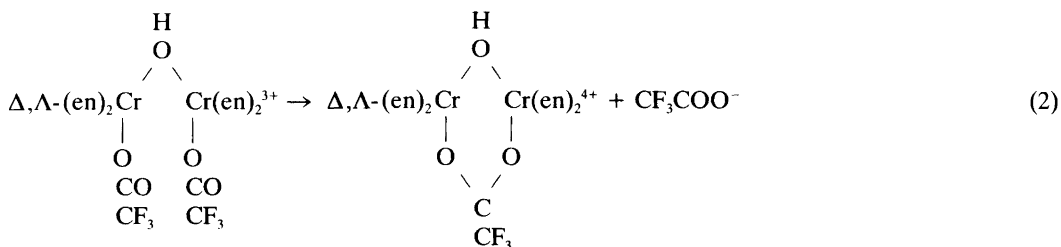
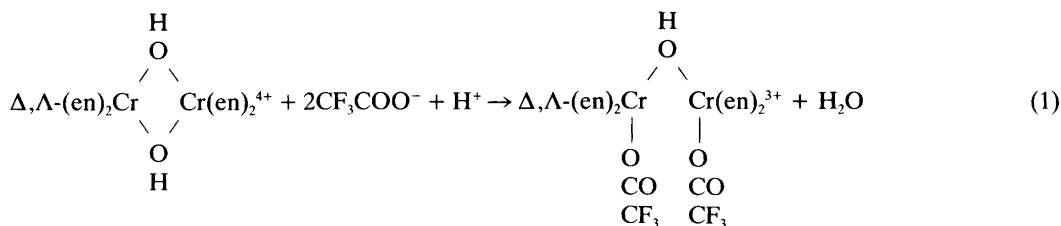


Fig. 2. Bond distances in the coordination octahedra and the bridging system. The angles of the ring are shown.

served and were employed in the subsequent analysis. For the crystallographic calculations, use was made of the following computer programs: the Vanderbilt system²⁶ for diffractometer operation; a data reduction program of local origin; MULTAN²⁷ and the X-Ray System²⁸ for the structure solution and refinement; and ORTEP II²⁹ for the illustrations. The scattering factor used for neutral atoms were those of Cromer and Mann;³⁰ for the hydrogen atoms, those of Stewart *et al.*³¹ The anomalous dispersion corrections of the scattering factors for Br, Cr and Cl were those calculated by Cromer and Liberman.³²

Crystal data. Δ, Λ - μ -hydroxo- μ -trifluoroacetato-bis[bis(ethylenediamine)chromium(III)] bromide triperchlorate monohydrate, $(\text{C}_{10}\text{H}_{35}\text{Cr}_2\text{F}_3\text{N}_8\text{O}_3)^{4+}$, Br^- , $3(\text{ClO}_4^-)$, H_2O : $M = 870.7$, space group $Pnma$ (No. 62); $a = 17.687(3)$, $b = 11.703(3)$, $c = 15.304(4)$ \AA ; $V = 3167.8$ \AA^3 , $Z = 4$, D_m (floatation) = 1.81 g cm^{-3} , $D_x = 1.83$ g cm^{-3} , $\mu(\text{MoK}\alpha) = 24.1$ cm^{-1} .



Determination and refinement of the structure. The structure was solved by direct methods. A standard run of MULTAN³⁰ assuming the space group *Pnma* gave starting positions for the Cr, Br and Cl atoms. The remaining non-hydrogen atoms were located by conventional Fourier methods. The parameters, including anisotropic temperature factors, were refined by the method of full matrix least-squares, minimizing $w(F_o - F_c)^2$. The hydrogen atoms were inserted at calculated positions (C-H = 0.95 and N-H = 0.90 Å). The hydrogen atoms of the water molecule and the bridging hydroxo group could not be located in a difference electron density map and their contribution to F_c was neglected. The hydrogen positions were not refined and the isotropic temperature factors were fixed to $U = 0.06 \text{ \AA}^2$. The convergence of the refinement was very slow, probably due to difficulties in finding a good model for the trifluoroacetato group and the perchlorate ions. The relatively small number of reflections did not permit the introduction of extra parameters in the achiral space group *Pn2₁a*, or the refinement of occupancy factors.

Finally, the CF₃ group was situated in two positions turned 60° relative to each other with occupancy factors of 0.5. The anisotropic thermal parameters for the fluorine atoms and for the oxygen atoms of the perchlorate ions are not very satisfactory. The final difference density map showed several peaks, especially around these atoms.

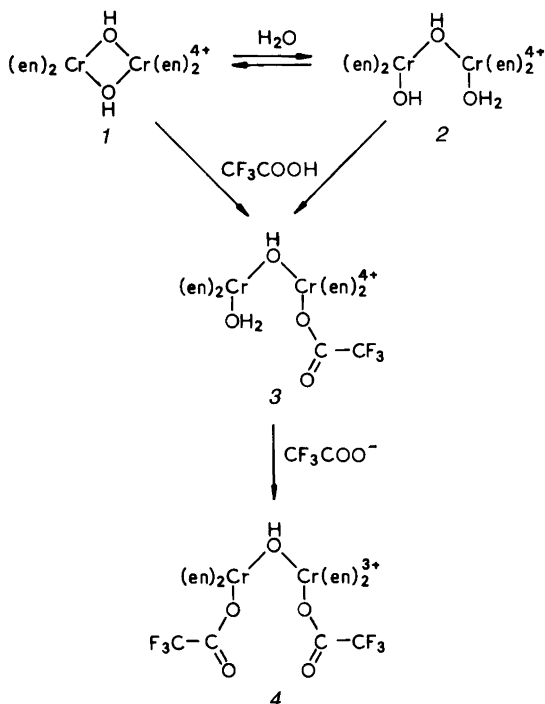
The highest peak was 0.9 e \AA^{-3} , but no chemically meaningful conclusions could be drawn. The last cycles of refinement employed a weighting scheme which gave unit weight to most reflections, but downweighted those with $F_o > 75$ and/or $\sin \theta < 0.33$. $W = x.y$ where $x = 1$ or $75/F_o$, $y = 1$ or $\sin \theta/0.33$. In the last cycle of the least-squares refinement, the maximum shift/error was 0.36 and the unit weighted and weighted residuals, R and R_w , where 0.059 and 0.071, respectively*.

Listings of observed and calculated structure amplitudes, calculated hydrogen atom positions, and anisotropic thermal parameters may be obtained from the authors.

Results and discussion

Reaction of $\Delta, \Lambda\text{-}[(\text{en})_2\text{Cr}(\text{OH})_2\text{Cr}(\text{en})_2]^{4+}$ in 2 M CF₃COOH led to the formation of a singly-bridged species, as shown in eqn. (1). The singly-bridged species was isolated as a red perchlorate

*At this point, it became clear that much effort spent on refinement could have been saved if the data set had been recollected at low temperature and an absorption correction had been applied. However, since the question posed by the chemistry involved had been answered, this was not considered worthwhile.



Scheme 1.

monohydrate salt in 91% yield. Preliminary spectrophotometric kinetic studies showed that the reaction in 1 M CF_3COOH was quantitative and that there was an appreciable build-up of at least one intermediate. This is not surprising since the reaction must occur in (at least) two steps via the singly-bridged intermediate Δ, Λ - $[(\text{H}_2\text{O})(\text{en})_2\text{Cr}(\text{OH})\text{Cr}(\text{en})_2(\text{CF}_3\text{COO})]^{4+}$, which, as shown in Scheme 1, can be formed by two pathways. The kinetics of the equilibration reaction between the mono and dihydroxo-bridged species, 1 and 2 in Scheme 1, have been studied previously¹⁶ and the reaction is much faster ($t_1 = 43$ s at 25°C) than the formation of 4 which has an overall half-life of 5–10 min in 1 M CF_3COOH at 25°C. These observations are consistent with both pathways shown in Scheme 1.

The assignment of the Δ, Λ configuration with both trifluoroacetato ligands coordinated in a position *cis* to the hydroxo bridge is based upon the known structure of the dihydroxo-bridged complex and the well-established tendency of such chromium(III) amine complexes to undergo ther-

mal substitution without isomerization. Furthermore, the facile formation of a doubly-bridged Δ, Λ complex, as described in the following, supports this assignment.

Whereas the bis(trifluoroacetato) complex is fairly stable in 1 M CF_3COOH , it is unstable in pure water and in acidic solution with no added CF_3COOH . In these latter media, it forms a μ -hydroxo- μ -carboxylato complex, as shown in eqn. (2). The reaction is relatively fast ($t_1 \sim 5$ min in 0.1 M HCl at 25°C), and the carboxylato-bridged complex has been isolated as a tetrabromide tetrahydrate in 73% yield. A bromide triperchlorate salt has also been isolated.

The visible absorption spectra of aqueous neutral or acidic solutions of salts of the carboxylato-bridged complex undergo a rapid change, even at 0°C, and the spectra, therefore, had to be extrapolated back to the time of dissolution. Kinetic measurements³³ indicate that these spectral changes are due to rapid equilibration between the doubly-bridged complex and a monohydroxo-bridged species, Δ, Λ - $[(\text{H}_2\text{O})(\text{en})_2\text{Cr}(\text{OH})\text{Cr}(\text{en})_2(\text{CF}_3\text{COO})]^{4+}$. Contamination with this cation was therefore a possibility which had to be considered, but the fact that several different reprecipitation procedures for the tetrabromide gave products with identical spectra indicated that no such contamination was present. The observation that both the tetrabromide and the bromide triperchlorate salts have identical spectra which did not change for either salt upon repeated reprecipitation also indicates that the products are pure. Finally, visual examination of the samples showed only one kind of crystal to be present for each salt.

The fact that the visible absorption spectra (at t_0) of aqueous solutions of Δ, Λ - $[(\text{en})_2\text{Cr}(\text{OH})(\text{CF}_3\text{COO})\text{Cr}(\text{en})_2] \text{Br}_4 \cdot 4\text{H}_2\text{O}$ at pH = 0 and 5.5 are identical points strongly to a doubly-bridged structure. A mono-bridged structure would require the presence of terminally coordinated water ligand(s) and would therefore lead to spectral changes due to the deprotonation of the aqua ligands. The μ -carboxylato- μ -hydroxo structure was finally established unambiguously by a crystal structure analysis of the bromide triperchlorate salt.

The structure contains Δ, Λ - $[(\text{en})_2\text{Cr}(\text{OH})(\text{CF}_3\text{COO})\text{Cr}(\text{en})_2]^{4+}$ cations, Br^- and ClO_4^- anions and water molecules held together by electrostatic forces and hydrogen bonds. The

Fig. 3. Stereoscopic illustration of the content of the unit cell. Thermal ellipsoids enclosing 50% probability are shown for bromide ion and the water molecule. The orientations are $a \uparrow$ and $c \leftarrow$.

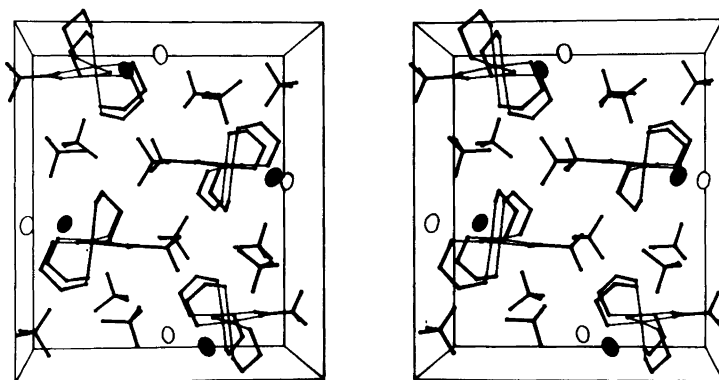


Table 1. Final atomic positions (and e.s.d.s) of non-hydrogen atoms $\times 10^4$ and $U_{eq} \times 10^3$.

The other half of the complex ion (atoms designated by a prime in Fig. 2) is generated by $x' = x$, $y' = 1.5 - y$, and $z' = z$. $U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$.

	x	y	z	U_{eq}
Cr	1158(0)	5955(1)	2569(1)	28.0(0.1)
O(1)	1190(6)	7500(0)	3062(6)	32(5)
O(2)	1294(5)	6553(6)	1349(5)	38(4)
N(1)	2305(5)	5677(9)	2746(6)	50(5)
N(2)	1116(6)	5281(9)	3805(6)	57(6)
N(3)	1050(5)	4372(8)	1973(6)	44(5)
N(4)	-6(5)	5950(9)	2422(8)	60(6)
F(1)	2017(21)	7500(0)	-340(23)	84(18)
F(11)	698(21)	7500(0)	-276(21)	113(33)
F(2)	1026(21)	8378(27)	-361(16)	113(17)
F(22)	1719(31)	8332(31)	-271(19)	124(27)
C(1)	2425(9)	4947(12)	3522(10)	59(8)
C(2)	1872(9)	5266(15)	4200(9)	72(8)
C(3)	269(12)	4271(15)	1601(12)	78(10)
C(4)	-276(10)	4796(16)	2207(17)	85(13)
C(5)	1294(9)	7500(0)	1007(10)	28(9)
C(6)	1356(13)	7500(0)	4(12)	40(9)
O(3)	661(9)	7500(0)	4785(9)	78(7)
Br	706(1)	2500(0)	3665(1)	72(1)
Cl(1)	3816(4)	7500(0)	4516(4)	70(3)
O(11)	3151(10)	7500(0)	4017(13)	94(10)
O(12)	4488(12)	7500(0)	4046(13)	142(12)
O(13)	3823(12)	6705(32)	5065(20)	429(30)
Cl(2)	3051(4)	2500(0)	1797(5)	87(3)
O(21)	3536(12)	2500(0)	2523(16)	110(11)
O(22)	2325(13)	2500(0)	2067(17)	182(17)
O(23)	3144(19)	3353(28)	1323(20)	297(26)
Cl(3)	3529(4)	7500(0)	1284(5)	62(3)
O(31)	3492(9)	7500(0)	2204(10)	78(8)
O(32)	4236(12)	7500(0)	1032(13)	296(34)
O033)	3156(13)	6639(21)	946(13)	210(15)

packing is shown in Fig. 3. The complex ion is shown in Fig. 1.

The atomic labelling and the bond distances in the inner coordination sphere and in the bridging ring are shown in Fig. 2. Bond distances and angles are given in Table 2. The complex has crystallographically imposed $Cs(m)$ symmetry. Atoms O(1), C(5) and C(6) lie in a mirror plane at $y = 1/4$. The quality of the data does not permit a detailed discussion of bond lengths and angles. A ring-formed electron density was found for the fluorine atoms, indicating either disorder or strong thermal vibration of the trifluoroacetato group. This phenomenon has been observed

Table 2. Selected interatomic distances/Å and angles $^\circ$.

Cr ...Cr'	3.616(2)	N(4)-C(4)	1.47(2)
Cr -O(1)	1.960(5)	C(1)-C(2)	1.47(2)
Cr -O(2)	2.007(8)	C(3)-C(4)	1.47(3)
Cr -N(1)	2.074(11)	O(2)-C(5)	1.226(11)
Cr -N(2)	2.050(11)	C(5)-C(6)	1.54(2)
Cr -N(3)	2.074(10)	C(6)-F(1)	1.28(5)
Cr -N(4)	2.074(9)	C(6)-F(11)	1.24(5)
N(1)-C(1)	1.48(2)	C(6)-F(2)	1.31(4)
N(2)-C(2)	1.47(2)	C(6)-F(22)	1.24(5)
N(3)-C(3)	1.50(2)		
O(1)-Cr-O(2)	91.9(4)	N(1)-Cr -N(3)	90.5(4)
O(1)-Cr-N(1)	93.8(5)	N(2)-Cr -N(3)	93.4(4)
O(1)-Cr-N(2)	90.0(4)	N(2)-Cr -N(4)	93.7(4)
O(1)-Cr-N(4)	94.2(5)	N(3)-Cr -N(4)	81.8(4)
O(2)-Cr-N(1)	93.4(4)	Cr -O(1)-Cr'	134.9(6)
O(2)-Cr-N(3)	85.1(4)	Cr -O(2)-C(5)	135.5(9)
O(2)-Cr-N(4)	91.1(4)	O(2)-C(5)-O(2)'	129.3(1.5)
N(1)-Cr-N(2)	81.6(5)	O(2)-C(5)-C(6)	115.3(8)

for other trifluoroacetate complexes.^{34,35} The geometry of the trifluoroacetate group is in agreement with that found in tetrakis- μ -trifluoroacetato-bis[triaqua(trifluoroacetato) praseodymium(III)].³⁴ The four independent Cr–N distances are essentially identical $\langle \text{Cr–N} \rangle = 2.068(12) \text{ \AA}$. The Cr–O distances are 1.960(5) Å to the OH group and 2.007(8) Å to the CF₃COO group. None of these parameters differ appreciably from those found for similar compounds.^{1–3} The C–C bonds of the ethylenediamine rings are nearly parallel to the pseudo-three-fold axis of the coordination octahedra, such that the conformation is all *lel* $\lambda\Delta, \Lambda\delta$. The six-membered bridging ring is almost planar, the torsion angles given in Table 3 revealing a slight chair form distortion. The six atoms of the ring lie within 0.07 Å from a least-squares plane through these atoms.

Table 3. Torsion angles/ $^\circ$ of the bridging ring.

Cr –O(1) –Cr' –O(2)'	–11.0(9)
O(1)–Cr' –O(2)'–C(5)	9.1(1.4)
Cr' –O(2)'–C(5) –O(2)	–10.1(2.6)

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