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The Crystal Structure of Tetraqua-*cis*-dichloroiron(III) Tetrachloroferrate(III) Monohydrate (Ferric Chloride $2\frac{1}{2}$ -Hydrate)*

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Abstract

The crystal structure of ferric chloride $2\frac{1}{2}$ -hydrate, $\text{FeCl}_3 \cdot 2\frac{1}{2}\text{H}_2\text{O}$, $[\text{FeCl}_2(\text{H}_2\text{O})_4]^+ \cdot [\text{FeCl}_4]^- \cdot \text{H}_2\text{O}$, has been determined from single-crystal X-ray diffractometer data using an average of three equivalent data sets, and refined to $R = 0.035$ for the 1617 unique reflections collected using Mo $K\alpha$ radiation. The structure is orthorhombic, space group $Pbc2_1$, with $a = 6.272$ (3), $b = 12.945$ (3) and $c = 16.654$ (4) Å, $Z = 4$, and consists of a somewhat distorted tetrahedral FeCl_4^- anion, a distorted octahedral tetraqua-*cis*-dichloroiron(III) cation and a single solvate water molecule. The tetrahedral Fe–Cl bonds range from 2.169 (4) to 2.220 (4) Å. The octahedral Fe–Cl bonds are 2.236 (4) and 2.251 (4) Å, and the Fe–water distances range from 2.004 (8) to 2.116 (7) Å. An extensive network of hydrogen bonds links the ions and the solvate water molecule.

Introduction

The ferric chloride–water phase diagram (Roozeboom, 1892) reveals four hydrates: $\text{FeCl}_3 \cdot 2\text{H}_2\text{O}$, $\text{FeCl}_3 \cdot 2\frac{1}{2}\text{H}_2\text{O}$, $\text{FeCl}_3 \cdot 3\frac{1}{2}\text{H}_2\text{O}$ and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. The last of these has been examined crystallographically by Lind (1967), and shown to consist of tetraqua-*trans*-dichloroiron(III) ions, with a Cl^- ion and two solvate water molecules. The crystal structure of ferric chloride $2\frac{1}{2}$ -hydrate was determined to provide a known structural background for the interpretation and use of parameters determined from infrared, Mössbauer and ultraviolet–visible absorption spectra of both the solid compound and the acidic ferric chloride solutions used to leach sulfide ores. Concentration and structural information about the complexes in these solutions is a necessary first step in the study of reaction mechanisms of these leaching reactions. This paper reports the results of the first of three crystal structure determinations of the three lower ferric chloride hydrates.

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Experimental

Ferric chloride $2\frac{1}{2}$ -hydrate melts at 329 K, and is extremely deliquescent. Even crystals mounted in sealed glass capillaries acquired a liquid layer around them within a period of a few days, and this contributed to crystal movement within the capillaries making them unsuitable for X-ray data collection. Eventually, dried fused silica tubes were used, and when sealed in these the crystals remained dry. The crystals themselves appeared to decompose under X-ray bombardment, however, as evidenced by the decrease in intensities of monitored standard reflections after about a week. Comparable experimental difficulties, in terms of both deliquescence and crystal decomposition, were reported in the structure determination of hexaaquairon(III) nitrate trihydrate (Hair & Beattie, 1977). Fuller details of the experimental procedures used in preparing, analyzing and loading of the crystals will be given in a separate paper (Francis & Wiles, 1979).

Preliminary examination of the compound with a precession camera revealed that the structure was orthorhombic, space group *Pbcm* or *Pbc2₁*. A single-crystal fragment, approximately 0.3 mm across, and roughly equidimensional, was set in a silica tube and mounted in a general orientation on a Picker four-circle diffractometer. The cell dimensions given below were determined from a least-squares refinement (Busing, 1970) of the 2θ , χ and ω angles of 34 reflections. The parameters were refined as a triclinic cell, and it was found that the maximum deviation from orthogonality was about $1'$ of arc, and well within one standard deviation.

Intensity data were collected to a limit of $2\theta = 55^\circ$, using graphite-monochromated Mo $K\alpha$ radiation, and a θ - 2θ scan mode. A scan speed of 2° min^{-1} was used, with a 2θ peak width of 2° , increasing with 2θ to account for α_1 - α_2 dispersion. Backgrounds were measured for 10 s on each side of the peak. The *hkl*, $\bar{h}\bar{k}l$, $h\bar{k}l$ and $\bar{h}kl$ octants of data were collected (equivalent in the noncentrosymmetric space group *Pbc2₁*). Three standard reflections were measured every fifty reflections to maintain a check on crystal alignment, instrument stability and decomposition of the crystal. The standard reflections remained constant during the collection of the first three segments of data, but started decreasing significantly during the collection of the fourth segment, $\bar{h}\bar{k}l$, and these data were consequently discarded. Spherical absorption corrections were applied to the intensity data ($\mu = 3.36 \text{ mm}^{-1}$) but no corrections for the absorption by the silica tube were attempted. The three useable segments of data were averaged, and standard deviations were calculated for each reflection from counting statistics and the relative agreement of the three measurements. Of the 1617 unique reflections examined, 326 were considered unobserved on the criterion that $I < 1.65\sigma(I)$. The

agreement factor between the three segments of data [$\sum (I - \bar{I}) / \sum I$] was 0.034 for all reflections and 0.029 for the observed data. Structure factors were derived by the application of Lorentz and polarization corrections, with $\sigma(F)$ set to $\frac{1}{2}\sigma(I)(I \text{ Lp})^{-1/2}$. The subsequent least-squares refinements were weighted by $w = 1/\sigma^2(F)$. Unobserved reflections were included at the minimum observable threshold, and used in the refinement only if $F(\text{calc.}) > F(\text{threshold})$ for a given *hkl*.

Crystal data

Molecular formula: $[\text{FeCl}_2(\text{H}_2\text{O})_4]^+ \cdot [\text{FeCl}_4]^- \cdot \text{H}_2\text{O}$; formula weight: 414.49; crystal system: orthorhombic; systematic absences: $0kl, k = 2n + 1; h0l, l = 2n + 1$; possible space groups: *Pbcm* (No. 57), or *Pbc2₁* (No. 29, *Pca2₁* with b, a, \bar{c}), *Pbc2₁* confirmed by the structure analysis; cell dimensions: $a = 6.272$ (3), $b = 12.954$ (3), $c = 16.654$ (4) Å at 298 K; $\lambda(\text{Mo } K\alpha_1) = 0.70930$ Å; linear absorption coefficient: $\mu(\text{Mo } K\alpha) = 3.36 \text{ mm}^{-1}$; density: $D_{\text{calc}} = 2.05 \text{ Mg m}^{-3}$; $Z = 4$ (density was not measured); intensity data: 1617 reflections measured three times ($2\theta < 55^\circ$); 1293 observed with $I > 1.65\sigma(I)$.

Structure solution and refinement

The Fe atom positions were determined from an $E^2 - 1$ map assuming the centrosymmetric space group *Pbcm*. This showed that there were two different Fe atoms in the asymmetric unit, and their positions were approximately $[\text{Fe}(1)] 0.05, \frac{1}{4}, \frac{1}{2}$, and $[\text{Fe}(2)] 0.54, 0.01, \frac{1}{4}$. Phasing the data from these positions revealed the tetrahedrally bonded $[\text{FeCl}_4]^-$ molecule and a water molecule on the mirror plane at $z = \frac{1}{4}$, but the inclusion of the one O and four Cl atoms did not clearly reveal the coordination around the second Fe atom. Even the successive choice of possible coordination sites did not lower the reliability factor *R* to below 0.24.

Examination of the statistics of the normalized structure factors (*E*) suggested that the true space group might be noncentrosymmetric, *Pbc2₁*, but with pseudosymmetry approximating the centrosymmetric *Pbcm*. The constraints of the mirror plane at $z = \frac{1}{4}$ and the twofold axis through Fe(1) were released, and successive least squares and Fourier maps revealed the complete structure. The anisotropic refinement converged to $R = 0.039$.

At this stage, examination of the model of the structure indicated the probable hydrogen-bonding network, and a comparison of the calculated positions of the H atoms with a difference synthesis revealed resolved peaks at all of the ten H positions. Attempts to refine these positions were unsuccessful, probably due to the inaccuracies of the data, resulting from the use of

spherical absorption corrections and the absence of absorption corrections for the silica tube. The positions of the H atoms quoted in Table 3 are thus the presumed positions with no refinement.

The effect of scattering curves for the Fe and Cl atoms was examined. With H atoms included but not refined, the structure refined to $R = 0.0352$ using the curves of Cromer & Mann (1968) for the neutral atomic species Fe, Cl and O, and the anomalous-dispersion corrections of Cromer & Liberman (1970) for these same atoms. A difference synthesis showed negative troughs at the Fe positions, and positive peaks at the Cl positions. The refinement was continued using the ionic-species curves Fe^{3+} and Cl^- from the same source, instead of the neutral species. The temperature factors changed, as expected, but the resultant difference synthesis was considerably more featureless around the Fe and Cl atoms. Although the final residual was only fractionally better ($R = 0.0346$), the evidence of the difference syntheses was taken to favor the use of ionic curves. For H atoms, the curve of Stewart, Davidson & Simpson (1965) was used.

All the structural computations were performed using the XRAY system of programs (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). The refinements were carried out using the full-matrix least-squares program *CRYLSQ* (written for the XRAY system by F. Kundell) with anisotropic thermal parameters for all atoms heavier than H. The mean parameter shift in the last cycle was less than 0.1σ , with a maximum shift of about 0.3σ in one of the thermal parameters.

Attempts to re-refine the structure in the centrosymmetric space group *Pbcm*, starting from the appropriate mean positions of the atoms in space group *Pbc2₁*, were totally unsuccessful. The R value did not fall below 0.20, with the thermal parameters of all atoms around Fe(1) ever increasing and unreasonably

large. The values of the refined positional parameters in space group *Pbc2₁* are given in Table 1.*

Description of the structure

Fig. 1 shows the structure viewed in the *bc* plane, and clearly indicates the two different ions stacked in alternate layers. The layers at $z \sim 0, \frac{1}{2}$ are tetraaqua-*cis*-dichloroiron(III) cations in a somewhat distorted octahedral coordination. A very approximate twofold axis of rotation, through the Fe atoms, relates Cl(5) to Cl(6), O(2) to O(4), and O(3) to O(5). The H atoms are

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34479 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

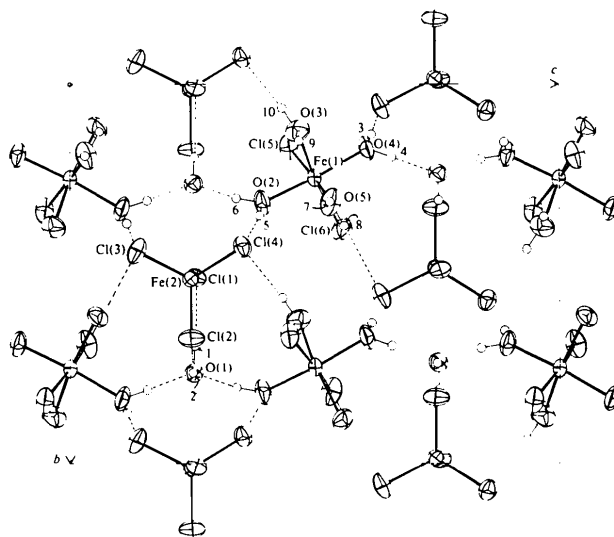


Fig. 1. The projection of the unit cell along *a*. The thermal ellipsoids are drawn to include 50% probability, and the atoms of the asymmetric unit are labelled. H atoms are indicated as circles, and numbered in accordance with Table 3. Part of the hydrogen-bonding network is indicated by broken lines.

Table 1. Atomic fractional coordinates with standard deviations

	<i>x</i>	<i>y</i>	<i>z</i>
Fe(2)	0.4624 (2)	0.5139 (1)	0.2470 (1)
O(1)	0.0765 (8)	0.7637 (4)	0.2524 (6)
Cl(1)	0.1143 (3)	0.5096 (2)	0.2573 (2)
Cl(2)	0.5775 (3)	0.6736 (2)	0.2477 (3)
Cl(3)	0.5658 (5)	0.4406 (3)	0.1365 (2)
Cl(4)	0.5901 (5)	0.4296 (2)	0.3527 (2)
Fe(1)	0.0542 (2)	0.2484 (2)	0.5*
Cl(5)	-0.2279 (5)	0.1666 (2)	0.4481 (2)
Cl(6)	-0.1106 (5)	0.3852 (2)	0.5558 (2)
O(2)	0.1229 (14)	0.3133 (7)	0.3938 (4)
O(3)	0.2483 (13)	0.1241 (6)	0.4662 (5)
O(4)	0.0622 (14)	0.1708 (7)	0.6062 (5)
O(5)	0.3522 (11)	0.3118 (7)	0.5334 (5)

* The z coordinate of Fe(1) was fixed at $\frac{1}{2}$ to define the origin of the polar space group.

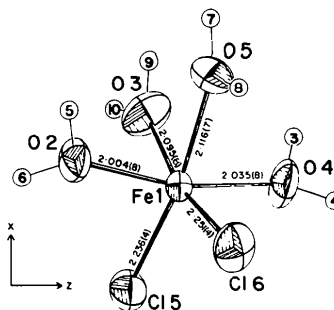


Fig. 2. The tetraaqua-*cis*-dichloroiron(III) cation, viewed along the *y* direction.

also similarly related. This relationship would be exact if the structure were centrosymmetric. The molecule, showing the bond lengths, is illustrated in Fig. 2.

Between the cation layers at $z \sim 0, \frac{1}{2}$, there are layers of tetrahedrally coordinated $[\text{FeCl}_4]^-$ anions and solvate water molecules at $z \sim \frac{1}{4}, \frac{3}{4}$. The tetrahedra are somewhat distorted, the individual bond lengths varying from 2.169 (4) to 2.220 (4) Å, and the angles varying from 106.6 (1) to 110.7 (2)°. The molecule is illustrated in Fig. 3, which also gives the bond lengths. Table 2 gives the details of the angular geometry of both anion and cation.

In the determination of the hydrogen-bond network, the O—H distance was taken as 0.95 Å, and the H—O—H angles were taken as 104.5°. The hydrogen-bond network was deduced as follows. The O atom of the lone solvate molecule, O(1), is in close proximity to Cl(1) (3.30 Å) and Cl(2) (3.35 Å), and to O(2) (2.74 Å) and O(4) (2.72 Å), in an approximately tetrahedral environment (see Fig. 4). This solvate O must, therefore, act as a donor to the two Cl atoms, and as an acceptor from the other two bonded water molecules. The four hydrogen bonds around O(1) are thus close to tetrahedral. Now, referring to Fig. 1, if it is accepted that a bonded water molecule [say O(4), H(3), H(4)], which acts as a donor to O(1) [O(4)—H(4)···O(1)], will have a second donor H related to the first [H(4)] by

a twofold axis along the Fe(1)—O(4) bond, it is found that this second H atom [H(3)] is positioned to act as an intermolecular bridge, binding Cl(3) of the adjacent anion to the cation. Similarly, the water molecule O(2), H(5), H(6) [*trans* to O(4), H(3), H(4)] is related by the approximate mirror planes at $z = \frac{1}{4}, \frac{3}{4}$ to the symmetry-related water molecule O(4), H(3), H(4), and the H atoms bind an adjacent anion *via* O(2)—H(5)···Cl(4) and another symmetry-related solvate O atom [O(1)] *via* O(2)—H(6)···O(1).

The remaining two bonded water molecules O(3), H(9), H(10) and O(5), H(7), H(8) are *cis* to each other and are close to Cl(5) and Cl(6) of the cation related by the translation $x = +1$. This gives rise to the intermolecular bonds Fe(1)—O(5)—H(7)···Cl(6)—Fe(1) and Fe(1)—O(3)—H(9)···Cl(5)—Fe(1), which bind cation to cation in the x direction. Finally, if the positions of H(7) and H(9) are fixed, and the Fe(1)—O—H fragments are rotated by 180° about the Fe(1)—O bonds, the last two H atoms are found in positions to bind adjacent anions Fe(1)—O(5)—H(8)···Cl(3) and Fe(1)—O(3)—H(10)···Cl(4). As previously explained, resolved peaks were found in the difference synthesis in all the proposed H positions, but refinement of these parameters was not found possible.

Table 3 gives the calculated H positions and the details of the hydrogen-bonding network.

Table 2. Bond angles (°) with standard deviations

(1) Tetrahedral FeCl_4^- anion; angles around Fe(2)

	Cl(2)	Cl(3)	Cl(4)
Cl(1)	110.6 (1)	110.7 (1)	106.6 (1)
Cl(2)	—	108.6 (2)	110.0 (2)
Cl(3)	—	—	110.5 (1)

(2) Octahedral $[\text{FeCl}_2(\text{H}_2\text{O})_4]^+$ cation; angles around Fe(1)

	Cl(6)	O(2)	O(3)	O(4)	O(5)
Cl(5)	99.7 (1)	91.6 (3)	89.6 (2)	97.0 (3)	169.9 (2)
Cl(6)	—	97.7 (3)	169.1 (2)	92.3 (3)	89.5 (2)
O(2)	—	—	87.7 (3)	165.5 (4)	83.1 (3)
O(3)	—	—	—	80.8 (3)	81.7 (3)
O(4)	—	—	—	—	86.6 (3)

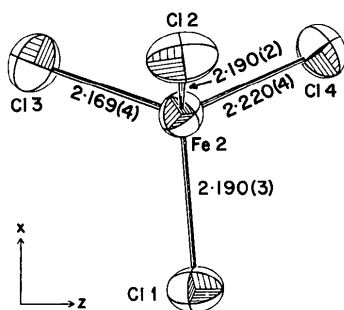


Fig. 3. The tetrachloroferrate(III) anion, viewed along the y direction.

Discussion

Hair & Beattie (1977) in their discussion of hexaaqua-iron(III) nitrate trihydrate have presented a comprehensive review of metal-ligand distances in Fe^{II} and Fe^{III} solvated complexes. They note that both the Fe^{III} structures containing the $[\text{FeCl}_2(\text{H}_2\text{O})_4]^+$ ion so far examined (Lind, 1967; Ferrari, Cavalca & Tani, 1957) are *trans*-dichloro, though both the Fe^{III} structures

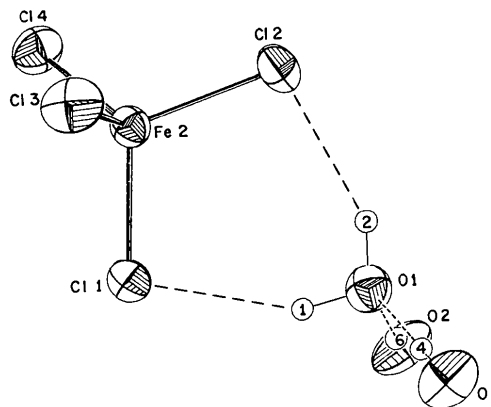


Fig. 4. Hydrogen bonding around the solvate water molecule O(1), H(1), H(2). O(1) is surrounded by two donor and two acceptor H atoms, in approximately tetrahedral coordination.

Table 3. Proposed hydrogen-bonding network

	<i>x</i>	<i>y</i>	<i>z</i>	Donor oxygen atom	Hydrogen-bonded atom	H...acceptor distance (Å)	Donor oxygen—acceptor distance (Å)	O—H...acceptor angle (°)
H(1)	0.0371	0.6929	0.2544	O(1)	Cl(1)	2.42	3.300 (6)	153
H(2)	0.2277	0.7630	0.2504	O(1)	Cl(2)	2.48	3.353 (6)	153
H(3)	0.1885	0.1328	0.6161	O(4)	Cl(3) ¹	2.57	3.509 (9)	170
H(4)	0.0165	0.1928	0.6580	O(4)	O(1) ⁶	1.77	2.722 (12)	178
H(5)	0.2568	0.3466	0.3879	O(2)	Cl(4)	2.42	3.365 (9)	172
H(6)	0.0760	0.3016	0.3402	O(2)	O(1) ²	1.81	2.743 (11)	164
H(7)	0.5034	0.3106	0.5306	O(5)	Cl(6) ³	2.64	3.521 (8)	154
H(8)	0.3197	0.3745	0.5604	O(5)	Cl(3) ⁴	2.80	3.673 (9)	153
H(9)	0.3893	0.1505	0.4690	O(3)	Cl(5) ³	2.43	3.344 (9)	160
H(10)	0.2600	0.0639	0.4338	O(3)	Cl(4) ⁵	2.39	3.308 (9)	161

The superscripts for hydrogen-bonded atoms refer to equivalent positions which may be generated from the atomic coordinates in Table 1 by the following symmetry operations:

(1)	<i>x</i> ,	$\frac{1}{2} - y$,	$\frac{1}{2} + z$	(4)	$1 - x$,	$1 - y$,	$\frac{1}{2} + z$
(2)	$-x$,	$-\frac{1}{2} + y$,	<i>z</i>	(5)	$1 - x$,	$-\frac{1}{2} + y$,	<i>z</i>
(3)	$1 + x$,	<i>y</i> ,	<i>z</i>	(6)	$-x$,	$1 - y$,	$\frac{1}{2} + z$

found containing the $[\text{Fe}(\text{H}_2\text{O})_4(\text{SO}_4)_2]$ molecule (Thomas, Robinson & Fang, 1974; Fanfani, Nunzi & Zanazzi, 1970) are *cis*. The present structure departs from this pattern, in that the ion is clearly a somewhat distorted *cis*-dichloro octahedron. Hair & Beattie further state that their $\text{Fe}^{\text{III}}-\text{O}$ distance of 1.986 (7) Å in hexaquaairon(III) nitrate trihydrate, and the identical distance [1.98 (2) Å] in paracoquimbite (Robinson & Fang, 1971), represents the shortest $\text{Fe}^{\text{III}}-\text{O}$ distance for coordinated water molecules, and that these shortest distances occur in $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$ octahedra. With coordinated chloride ions, this distance increases to 2.07–2.08 Å.

In the present structure, due to the *cis*-dichloro ligands, the $\text{Fe}^{\text{III}}-\text{O}$ distances should *not* be averaged, but rather considered individually in terms of the well known *trans* effect of ligands.* There are two longer Fe—O distances, and two shorter ones. The longer ones are both *trans* to Cl, and the shorter ones are *trans* to each other, as expected, since Cl has a greater *trans* effect than water. The shorter Fe—Cl bond [2.236 (4) Å] is *trans* to the longest Fe—O distance [2.116 (7) Å], and the longer Fe—Cl bond [2.251 (4) Å] is *trans* to the next-longest Fe—O distance [2.095 (8) Å]. It is probably valid to consider the average [2.02 (2) Å] of the shortest Fe—O distances [2.004 (8), 2.035 (8) Å] as being representative of the 'unaffected' Fe—O distance. Comparison of this average bond distance with the $\text{Fe}^{\text{III}}-\text{water}$ distance in tetraqua-*trans*-dichloroiron(III) chloride dihydrate (ferric chloride hexahydrate) (Lind, 1967) shows the latter to be longer [2.07 (2) Å]. This difference may well be due to the

different hydrogen bonding around the two ions. Similarly, both $\text{Fe}^{\text{III}}-\text{Cl}$ distances in the present structure are shorter than in ferric chloride hexahydrate [2.30 (2) Å], again presumably because in the latter case the *trans* bond-lengthening effect of each Fe—Cl bond acts on the other, whereas in the present structure it is manifested in the lengthening of the two longer Fe—O bonds.

There are distortions of the octahedral angles, and these can be qualitatively accounted for in the present structure. $\text{Cl}(5)-\text{Fe}-\text{Cl}(6) = 99.7 (1)^\circ$, $\text{Cl}(5)-\text{Fe}-\text{O}(2) = 91.6 (3)^\circ$, $\text{Cl}(5)-\text{Fe}-\text{O}(4) = 97.0 (3)^\circ$, $\text{Cl}(6)-\text{Fe}-\text{O}(2) = 97.7 (3)^\circ$ and $\text{Cl}(6)-\text{Fe}-\text{O}(4) = 92.3 (3)^\circ$ are all evidence of greater ligand—ligand repulsions when one or both ligands are Cl. However, an explanation for the deviations from *mm* symmetry, found in this distorted ion, must be sought in the hydrogen-bonding network that holds the whole structure together.

If the hydrogen-bond potential-energy function, in terms of distance, is neither symmetrical about the minimum, nor linear with respect to distance (and there is no reason why it should be), then a twist of the cation from the centrosymmetric orientation will result in a lengthening of one O—H...Cl bond, and a shortening of its partner previously related by the twofold axis. This may result in a *net gain* of energy, depending on the shape of the potential-energy function for the O—H...Cl and the O—H...O bonds, and the distances involved. When this effect is summed over the eight hydrogen bonds in which the cation is involved, the result is an ion distorted away from the twofold axis, and with no molecular symmetry of its own. A detailed analysis of these factors, however, is beyond the scope of the present structure determination.

Similarly, the distortions of the $[\text{FeCl}_4]^-$ anion from tetrahedral symmetry are difficult to explain by factors

* For a detailed discussion of the *trans* effect, and the many theories used to explain it, see Basolo & Pearson (1958). The electrostatic theory of the *trans* effect predicts and explains the weakening (and hence presumably lengthening) of bonds *trans* to strongly electronegative ligands.

other than the hydrogen-bonding forces discussed above. The variation in Fe—Cl bond distances found in this structure, 2.169 (4) to 2.220 (4) Å, are very significant, and much greater than in the $[\text{FeCl}_4]^-$ anion in $[\text{PCl}_4][\text{FeCl}_4]$ (Kistenmacher & Stucky, 1968) where the agreement between the bonds is remarkable (2.185 ± 0.003 Å). However, the deviations in the angles from tetrahedral in the latter structure (108.2 to 113.5°) are somewhat greater than in the present structure (106.6 to 110.7°). It is worth pointing out that the two Fe—Cl bonds which are equal in this structure, Fe—Cl(1) [2.190 (3) Å] and Fe—Cl(2) [2.190 (2) Å], are nearly identically affected by the hydrogen-bonding network, as both Cl atoms are bound to the same solvate water molecule at very similar distances (Fig. 4). This would seem to contradict the argument previously used to explain the distortions from the centrosymmetric structure. A possible explanation is that, in fact, these O—H...Cl distances are close to the minimum in the potential-energy function, and hence are stabilized.

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Structure Cristalline de $\text{Sb}_2\text{Mo}_{10}\text{O}_{31}$

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Abstract

$\text{Sb}_2\text{Mo}_{10}\text{O}_{31}$ crystallizes in the space group *Pma2* with $a = 20.03$ (2), $b = 8.09$ (1) and $c = 7.17$ (1) Å ($d_m = 4.87 \pm 0.05$, $d_c = 4.83$ Mg m⁻³, $Z = 2$). The crystal structure has been determined by the Patterson method on the xOz and xyO projections. Least-squares

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refinement with anisotropic temperature factors of the three-dimensional atomic parameters gives a final *R* of 0.055. The Mo atoms have octahedral coordination and the environment of the Sb atoms can be considered as being similar to the classical AX_3E ($E = \text{lone pair}$) polyhedra. Hexagonal tunnels formed by the MoO_6 octahedra are occupied by the Sb atoms.

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