

particularly striking in the $\equiv\text{PCINMe}_2$ groups, where the exocyclic N and Cl atoms are always very nearly eclipsed despite the large difference between the P–Cl and P–NMe₂ bond lengths.

Valency angles

The pattern of the endocyclic angles is consistent with the expected mirror symmetry of the ring across the N(1)···P(4) diagonal. Thus, the angles are $118.8 \pm 0.2^\circ$ at P(2) and P(6) and $119.4 (2)^\circ$ at P(4); $120.1 \pm 0.3^\circ$ at N(3) and N(5) and $121.6 (3)^\circ$ at N(1). The three exocyclic Cl–P–NMe₂ angles which are 104.9 , 104.2 and $105.7 (2)^\circ$ are significantly different from each other, presumably for the reasons stated previously in the discussion of bond lengths. The two Me groups at N(7) and those at N(8) are symmetrical about their respective P–N bonds, but those at N(9) are not symmetrical about P(6)–N(9). The angles P(6)–N(9)–C(5) and P(6)–N(9)–C(6) are $120.8 (4)$ and $117.7 (4)^\circ$, respectively. Despite this considerable asymmetry, the mean angles at the three exocyclic N atoms are nearly equal (117.2 , 116.9 and 117.6°).

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The Crystal Structure of Sodium Chlorite Trihydrate, $\text{NaClO}_2 \cdot 3\text{H}_2\text{O}$

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$\text{NaClO}_2 \cdot 3\text{H}_2\text{O}$ is triclinic, with $a=6.960 (2)$, $b=8.842 (3)$, $c=5.504 (2) \text{ \AA}$, $\alpha=92.36 (7)$, $\beta=119.09 (7)$, $\gamma=104.73 (7)^\circ$, space group $P\bar{1}$, $Z=2$. The structure was solved by Patterson methods and refined to an R_w of 4.0% by full-matrix least-squares methods based on 2227 independent counter reflexions. Only one oxygen of ClO_2^- is coordinated to Na^+ ; the other shares a hydrogen bond. Chains of edge-sharing irregular Na octahedra run along c ; connexion between the chains is achieved by a system of hydrogen bonds in which all the hydrogens are involved. Cl–O distances: 1.557 (3) and 1.570 (3) \AA ; O–Cl–O angle: $108.37 (8)^\circ$; Na–O distances range between 2.367 (3) and 2.496 (3) \AA .

Introduction

The title compound is of some importance in the textile industry and of growing interest in water

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deposition. In crystal chemistry it stands out as a gap in the systematics of the simplest compounds. Moreover, as the crystal structures of only a few chlorites have been determined, *i.e.* of ammonium chlorite (Gillespie, Sparks & Trueblood, 1959), silver chlorite (Cooper & Marsh, 1961) and lanthanum chlorite trihydrate (Coda, Giuseppetti & Tadini, 1965), a new contribution to the crystal chemistry of these compounds seemed useful.

scheme was based on the experimental $\sigma(F_{\text{obs}})$ derived from the counting statistics, and R_w dropped to 0.073. Then a likely system of hydrogen bonds was deduced from a survey of the structure, and in a difference synthesis the corresponding six hydrogen peaks were found as the most prominent maxima. The hydrogen coordinates derived from this map fit poorly the known geometry of the water molecule; moreover the hydrogen coordinates obtained by least-squares refinement were unsatisfactory for the same reason. Therefore theoretical hydrogen coordinates were obtained in the following way (O' , O'' and H_2O are the oxygens involved in the hydrogen-bonding system and H' , H'' are the hydrogens pointing respectively towards O' and O''): rather than starting from the literature 'average water', the conformation was derived from the linear correlations between the angles $O'-H_2O-O''$ and $H'-H_2O-H''$, and between the bond distances $O'-H_2O$ (or $O''-H_2O$) and $H'-H_2O$ (or $H''-H_2O$) (Ferraris & Franchini-Angela, 1972); then the water molecule with this geometry was placed in the O' , O'' , H_2O plane, so that $(H_2O-O')^2 + (H_2O-O'')^2$ was a minimum. The hydrogen coordinates were adjusted after each cycle of least-squares refinement; a fixed thermal parameter $B_H = 4 \text{ \AA}^2$ was assigned to all of them.

The coordinates of the non-hydrogen atoms were then refined anisotropically by four full-matrix least-squares cycles. At convergence R_w was 4.0%.

The refinement was performed with a locally modified *ORFLS* program (Busing, Martin & Levy, 1962). The scattering factors are those listed by Hanson, Herman, Lea & Skillman (1964).

The final positional and thermal parameters are given in Tables 2 and 3; the analysis of the thermal ellipsoids is in Table 4. The molecular dimensions are reported in Tables 5, 6 and 7; in the last the distances and angles in the hydrogen bonds are quoted.*

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30788 (18 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1 NZ, England.

Table 4. Analysis of the thermal ellipsoids

Root-mean-square displacements (\bar{u} , in \AA) and angles (α, β, γ , in degrees) that each principal direction of the thermal ellipsoid makes respectively with the a, b, c crystallographic axes. The $H_2O(2)$ ellipsoid is a degenerated revolution ellipsoid.

		\bar{u}	α	β	γ
Na	1	0.165	120.2	109.8	19.6
	2	0.179	30.7	113.8	88.9
	3	0.192	95.2	31.8	70.4
Cl	1	0.175	109.0	24.5	69.1
	2	0.197	143.5	104.8	38.8
	3	0.224	60.1	109.1	59.0
O(1)	1	0.173	106.2	41.2	122.9
	2	0.192	49.4	63.1	94.2
	3	0.278	134.9	61.4	33.3
O(2)	1	0.178	49.4	56.9	126.9
	2	0.206	78.9	88.7	43.0
	3	0.236	137.2	33.1	71.1
H ₂ O(1)	1	0.158	71.2	34.9	117.0
	2	0.192	27.6	117.0	92.8
	3	0.208	109.5	69.7	27.1
H ₂ O(2)	1	0.164			
	2	0.165			
	3	0.222	133.1	34.1	63.1
H ₂ O(3)	1	0.160	11.3	93.7	118.0
	2	0.184	97.7	60.3	43.3
	3	0.192	98.3	30.0	119.9

Table 5. Interatomic distances (\AA) and angles ($^\circ$)

Estimated standard deviations for distances are 0.003 \AA , for angles 0.08 $^\circ$.

i	1-x, 1-y, 1-z	vi	x, y, 1+z
ii	x, y, z-1	vii	x, 1+y, z-1
iii	x, y-1, 1+z	viii	-x, 1-y, 2-z
iv	-x, 1-y, -z	ix	1-x, -y, 2-z
v	-x, 1-y, 1-z	x	1-x, 1-y, 2-z

Cl-O(1)	1.557	O(1 ⁱ)—Na-H ₂ O(3 ⁱⁱ)	171.89
Cl-O(2)	1.570	O(1 ⁱ)—Na-H ₂ O(3 ⁱⁱ)	100.19
Na-O(1 ⁱ)	2.367	H ₂ O(1 ⁱⁱ)—Na-H ₂ O(2)	101.82
Na-H ₂ O(1 ⁱⁱ)	2.402	H ₂ O(1 ⁱⁱ)—Na-H ₂ O(2 ⁱⁱ)	162.06
Na-H ₂ O(2)	2.416	H ₂ O(1 ⁱⁱ)—Na-H ₂ O(3 ⁱⁱ)	92.03
Na-H ₂ O(2 ⁱ)	2.496	H ₂ O(1 ⁱⁱ)—Na-H ₂ O(3 ⁱⁱ)	79.15
Na-H ₂ O(3 ⁱⁱ)	2.442	H ₂ O(2)—Na-H ₂ O(2 ⁱ)	93.69
Na-H ₂ O(3 ⁱ)	2.373	H ₂ O(2)—Na-H ₂ O(3 ⁱⁱ)	82.86
		H ₂ O(2)—Na-H ₂ O(3 ⁱ)	168.77
O(1)—Cl-O(2)	108.37	H ₂ O(2 ⁱ)—Na-H ₂ O(3 ⁱⁱ)	80.91
O(1 ⁱ)—Na-H ₂ O(1 ⁱⁱ)	94.34	H ₂ O(2 ⁱ)—Na-H ₂ O(3 ⁱⁱ)	83.88
O(1 ⁱ)—Na-H ₂ O(2)	90.91	H ₂ O(3 ⁱⁱ)—Na-H ₂ O(3 ⁱ)	85.94
O(1 ⁱ)—Na-H ₂ O(2 ⁱ)	94.37		

Table 6. Angles around the oxygens ($^\circ$)

Estimated standard deviations for angles are less than 0.08 $^\circ$. For symmetry code see Table 5. The vertex of each angle is shown at the left; the angles involving hydrogen bonds are the first for every oxygen.

O(1)	H ₂ O(3 ⁱⁱ)...H ₂ O(3 ⁱⁱ)	81.23	H ₂ O(3 ⁱⁱ)...Cl	109.06
	H ₂ O(3 ⁱⁱ)...Cl	109.26	H ₂ O(3 ⁱⁱ)...Na ⁱ	86.63
	H ₂ O(3 ⁱⁱ)...Na ⁱ	114.92	Cl...Na ⁱ	134.89
O(2)	H ₂ O(1 ⁱ)...H ₂ O(1 ^{vi})	76.31	Cl...H ₂ O(1 ⁱ)	107.46
	H ₂ O(1 ⁱ)...H ₂ O(2 ^{iv})	126.12	Cl...H ₂ O(1 ^{vi})	115.27
	H ₂ O(1 ^{vi})...H ₂ O(2 ^{iv})	120.80	Cl...H ₂ O(2 ^{iv})	108.00
H ₂ O(1)	O(2 ⁱ)...O(2 ⁱⁱ)	103.69	Na ^{vi} ...O(2 ^{iv})	100.45
	O(2 ⁱ)...H ₂ O(2)	100.49	Na ^{vi} ...O(2 ⁱⁱ)	117.88
	O(2 ⁱⁱ)...H ₂ O(2)	132.48	Na ^{vi} ...H ₂ O(2)	96.72
H ₂ O(2)	H ₂ O(1)...O(2 ^{iv})	106.53	Na ⁱ ...H ₂ O(1)	98.65
	Na...H ₂ O(1)	118.81	Na ⁱ ...O(2 ^{iv})	135.20
	Na...O(2 ^{iv})	111.57	Na...Na ⁱ	86.31
H ₂ O(3)	O(1 ^v)...O(1 ^{vi})	98.77	Na ^{vi} ...O(1 ^v)	114.34
	Na ⁱ ...O(1 ^v)	119.24	Na ^{vi} ...O(1 ^{vi})	101.38
	Na ⁱ ...O(1 ^{vi})	128.02	Na ⁱ ...Na ^{vi}	94.06

The projection of the crystal structure is shown in Figs. 1 and 2; all the atoms cited in the text, and in Tables 5 and 7, are drawn with the symmetry code of Table 5.

Description of the structure

The ClO_2^- anion, considered as an isolated unit, exhibits geometrical features very similar to those already found; the Cl—O distances 1.557 (3), 1.570 (3) Å are comparable to the 1.57 (3) Å (-35°C) in ammonium chlorite (Gillespie, Sparks & Trueblood,

1959), 1.55 (5) Å in silver chlorite (Cooper & Marsh, 1961) and 1.54 (4) Å in lanthanum chlorite trihydrate (Coda, Giuseppetti & Tadini, 1965). The O—Cl—O angle is $108.37 (8)^\circ$ compared respectively with 110.5 ± 1.4 , 111 ± 3 and $109.7 \pm 2.5^\circ$ in the chlorites just quoted. However, unlike the latter chlorites, the two ClO_2^- oxygen atoms are non-equivalent in their structural role [the same happens with the three water molecules: compare also the tensimetric analysis carried out by Riganti (1959)]: only one of them, O(1), belongs to the coordination sphere of the Na^+ cation and shows the

Table 7. *Interatomic angles ($^\circ$) and distances (Å) involved in the hydrogen bonds*

Estimated standard deviations are in parentheses. The figures lacking parentheses refer to distances and angles involving hydrogens. The lower-case roman numerals have the same meaning as those of Table 5.

Vertex:	$\text{H}_2\text{O}(1)$	$\text{H}_2\text{O}(2)$	$\text{H}_2\text{O}(3)$		
$\angle \text{O}(2^{\text{i}}) \cdots \text{O}(2^{\text{iii}})$	103.69 (5)	$\angle \text{H}_2\text{O}(1) \cdots \text{O}(2^{\text{iv}})$	106.53 (6)	$\angle \text{O}(1^{\text{v}}) \cdots \text{O}(1^{\text{vi}})$	98.77 (5)
$\text{H}_2\text{O}(1) \cdots \text{O}(2^{\text{i}})$	2.823 (2)	$\text{H}_2\text{O}(2) \cdots \text{H}_2\text{O}(1)$	2.844 (2)	$\text{H}_2\text{O}(3) \cdots \text{O}(1^{\text{v}})$	2.739 (2)
$\text{H}_2\text{O}(1) \cdots \text{O}(2^{\text{iii}})$	2.843 (2)	$\text{H}_2\text{O}(2) \cdots \text{O}(2^{\text{iv}})$	2.877 (2)	$\text{H}_2\text{O}(3) \cdots \text{O}(1^{\text{vi}})$	3.017 (2)
$\angle \text{H}(11) \cdots \text{H}(12)$	107.10	$\angle \text{H}(21) \cdots \text{H}(22)$	107.39	$\angle \text{H}(31) \cdots \text{H}(32)$	106.57
$\text{H}_2\text{O}(1) \cdots \text{H}(11)$	0.96	$\text{H}_2\text{O}(2) \cdots \text{H}(21)$	0.96	$\text{H}_2\text{O}(3) \cdots \text{H}(31)$	0.94
$\text{H}_2\text{O}(1) \cdots \text{H}(12)$	0.96	$\text{H}_2\text{O}(2) \cdots \text{H}(22)$	0.96	$\text{H}_2\text{O}(3) \cdots \text{H}(32)$	0.97
$\text{O}(2^{\text{i}}) \cdots \text{H}(11)$	1.86	$\text{H}_2\text{O}(1) \cdots \text{H}(21)$	1.88	$\text{O}(1^{\text{v}}) \cdots \text{H}(31)$	2.08
$\text{O}(2^{\text{iii}}) \cdots \text{H}(12)$	1.88	$\text{O}(2^{\text{iv}}) \cdots \text{H}(22)$	1.92	$\text{O}(1^{\text{vi}}) \cdots \text{H}(32)$	1.77
Other angles of interest					
$\angle \text{O}(2^{\text{i}}) \cdots \text{H}_2\text{O}(2)$	100.49 (6)				
$\angle \text{O}(2^{\text{iii}}) \cdots \text{H}_2\text{O}(2)$	132.48 (6)				

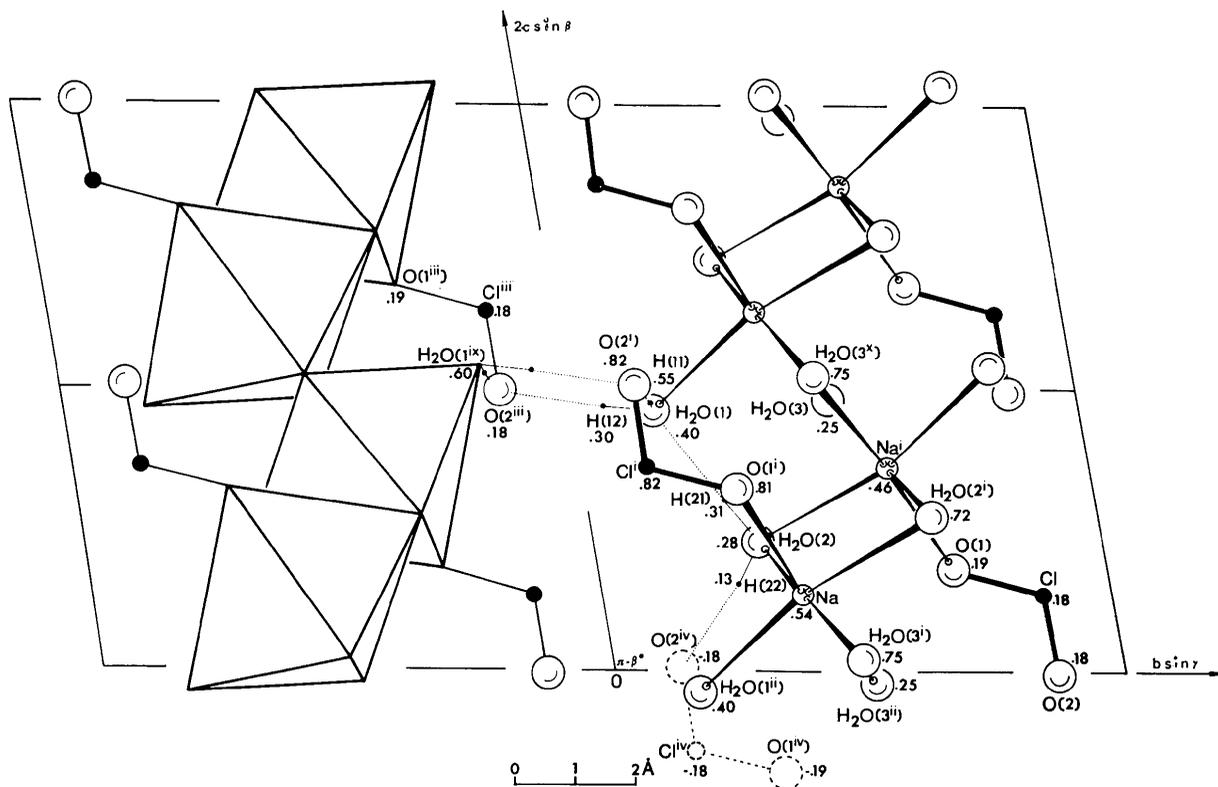


Fig. 1. Four cells of the crystal structure projected along the a axis; the figures quote the x/a coordinates of the relevant atoms. Only the hydrogen bonds around $\text{H}_2\text{O}(1)$ and $\text{H}_2\text{O}(2)$ are shown. Note that the $\text{O}(2^{\text{iv}})\text{—Cl}^{\text{iv}}\text{—O}(1^{\text{v}})$ anion is one cell below, along the projection axis, with respect to the other similar anions.

shortest Cl–O distance: 1.557 (3) against 1.570 (3) Å for O(2). Both oxygens are involved in hydrogen bonds with water molecules (Table 7): O(1) forms the shortest of all, 2.739 (2) Å, and another fairly long, 3.017 (2) Å, with two different H₂O(3) oxygens, while O(2) forms three hydrogen bonds of medium length, about 2.85 Å, with two different H₂O(1)'s and one H₂O(2).

The Na coordination octahedron is moderately distorted. The deviations from regularity may be appreciated from Table 5: the angles O–Na–O concerning pairs of contiguous oxygens range between 79.15 (8) and 101.82 (8)°, while those involving pairs of opposite oxygens range between 162.06 (8)° and 171.89 (8)°; the oxygens H₂O(1ⁱⁱ), H₂O(2), H₂O(2ⁱ) and H₂O(3ⁱ) lie almost in a plane, as do O(1ⁱ), H₂O(2), H₂O(3ⁱⁱ) and H₂O(3ⁱ): the largest distance from their best plane is 0.05 Å; however the four oxygens O(1ⁱ), H₂O(1ⁱⁱ), H₂O(2ⁱ) and H₂O(3ⁱⁱ) do not fit a plane so well: the shortest distance from their least-squares plane is 0.15 Å. The H₂O(2) as well as the H₂O(3) oxygens participate twice in the Na coordination: in both cases a pair of oxygens related by a symmetry centre is involved; O(1) as well as H₂O(1) participate only once in the Na coordination. The Na–O distances range between 2.367 (3) and 2.496 (3) Å.

The Na octahedra form chains along *c* (Figs. 1 and 2) by sharing the edges H₂O(2)–H₂O(2ⁱ) and H₂O(3)–H₂O(3ⁱ), *i.e.* the edges with vertices related by symmetry centres. The different chains are connected *via* hydrogen bonds which involve all the hydrogens of the structure.

A four-membered ring entirely based on the hydrogens of H₂O(1), both shared with O(2), is shown in Fig. 1, lettered as H₂O(1)–O(2ⁱ)–H₂O(1^{ix})–O(2ⁱⁱⁱ); rings of this kind connect pairs of Na octahedra along *b*, H₂O(1) and H₂O(1^{ix}) both being directly bound to Na. This link is reinforced *via* two ClO₂⁻ anions, ending respectively in O(1ⁱ) and O(1ⁱⁱⁱ), which again coordinate directly Na⁺ ions of different chains.

A similar four-membered ring, H₂O(3)–O(1^v)–H₂O(3^{viii})–O(1^{vi}), entirely based on the hydrogens of H₂O(3), both shared with O(1), is shown in Fig. 2; a collection of such rings connects pairs of Na octahedra along *a*, from H₂O(3) to O(1^v), both directly bound to Na. Unlike the former H₂O(1) ring, all four vertices of the H₂O(3) ring are directly bound to Na⁺ ions; thus H(31) establishes a bridge, H₂O(3)–O(1^{vi}), between contiguous Na⁺ ions of the same chain.

A similar bridge through H(21) connects H₂O(2) and H₂O(1), belonging to two shared Na octahedra, *i.e.* of

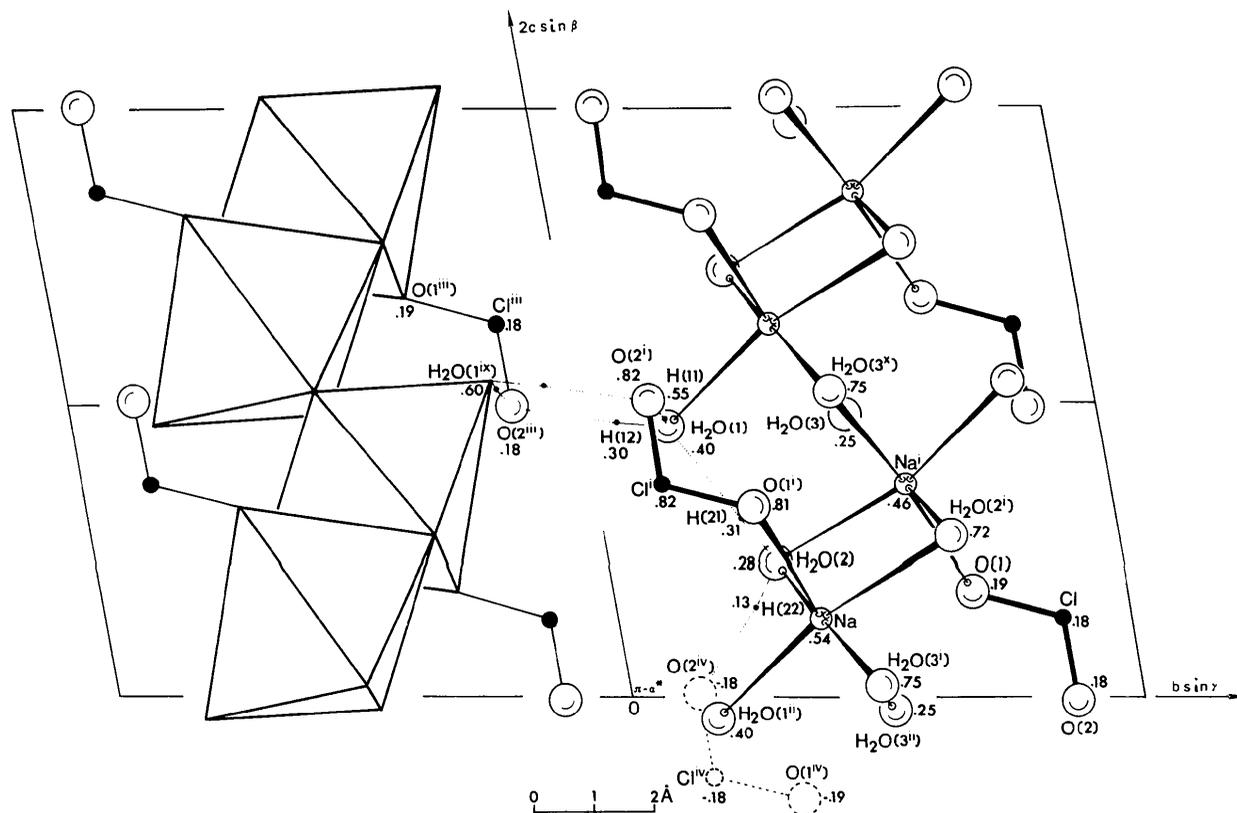


Fig. 2. Four cells of the crystal structure projected along the *b* axis; the figures quote the *y/b* coordinates of the relevant atoms. Only the hydrogen bonds around H₂O(2) and H₂O(3) are shown.

the same chain (Figs. 1 and 2); however the second $\text{H}_2\text{O}(2)$ hydrogen, $\text{H}(22)$, links pairs of different chains along **a**, from $\text{O}(1)$ to $\text{O}(2^{\text{iv}})$ through a ClO_2^- anion: $\text{O}(1^{\text{iv}})-\text{Cl}^{\text{iv}}-\text{O}(2^{\text{iv}})$ in Fig. 2. Thus the number of connexions along **a**, higher than along **b**, explains the fact that the only good cleavage follows $\{010\}$.

Each of the six Na-coordinating oxygens is involved in a hydrogen bond with one oxygen of the neighbouring Na octahedra of the same chain, so reinforcing the link obtained by edge-sharing. The vertices of the shared edge $\text{H}_2\text{O}(3)-\text{H}_2\text{O}(3^*)$ form two H-bonds, in opposite directions, with the $\text{O}(1)$'s of the neighbouring Na^+ ions; a similar pair of H-bonds starts from the shared edge $\text{H}_2\text{O}(2)-\text{H}_2\text{O}(2')$ towards two $\text{H}_2\text{O}(1)$'s.

If ionic strengths of +0.25 and +0.75 are assigned, as contributions of each hydrogen, respectively to the farthest and the nearest oxygen involved in a hydrogen bond, the balance of electrostatic valences looks satisfactory for all oxygens except $\text{O}(2)$, which has an excess positive charge of +0.25; three different hydrogen atoms surround this oxygen.

Table 6 shows that each of the five oxygens in this structure exhibits a roughly tetrahedral environment, if all kinds of chemical interactions are taken into account, *i.e.* ionic, covalent and hydrogen-bonding. $\text{O}(2)$ and $\text{H}_2\text{O}(1)$ behave similarly in that both are involved in three different hydrogen bonds of similar length (the obvious difference between them is that $\text{O}(2)$ is never the nearest oxygen to any hydrogen).

The amount and the anisotropy of the thermal mo-

tion are largest for the ClO_2^- anion (Tables 3 and 4); the longest axes of the $\text{O}(1)$ and $\text{O}(2)$ ellipsoids lie roughly in the (100) plane, normal to the respective Cl-O bonds; the longest axis of the Cl ellipsoid forms an angle of 60° with **a** and lies roughly in the plane normal to (100), bisecting the $\text{O}(1)-\text{Cl}-\text{O}(2)$ angle.

There are no abnormally short non-bonding distances.

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The Structure of $\text{K}_3\text{RhCl}_6 \cdot \text{H}_2\text{O}$; a Comparison of Two Independent X-ray Structure Determinations

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Two independent structure analyses of $\text{K}_3\text{RhCl}_6 \cdot \text{H}_2\text{O}$ are compared. The first is derived from data collected on a four-circle diffractometer [Cresswell, Fergusson, Penfold & Scaife. *J. Chem. Soc. Dalton*, (1972), pp. 254-262] which led to a final *R* of 0.072. The authors collected data on a linear diffractometer for two rotation axes and the final scaled and merged data led to a structure giving a final *R* of 0.046. The methods and extent of data collection and their treatment are compared. There was no significant difference in the cell dimensions and the derived atomic positions are shown to be identical within the standard deviations estimated by both groups. The r.m.s. amplitudes of vibration of the atoms differ significantly between the two studies and this is attributed to absorption effects. Final atomic and thermal parameters for the authors' investigation are tabulated.

Introduction

Although there has been considerable interest in the comparison of crystallographic data for a particular crystal collected by different groups on different in-

struments, this is often undertaken as part of a programme where care is taken to standardize experimental conditions, especially in the selection of the crystal. An example is the recent study published by the International Union of Crystallography Commission on