1969). In retrospect it is clear that this band should instead be assigned to the N–H group which is, as we have seen, weakly bonded to a fluorine atom. In Table 7 it is shown that the $N\cdots F$ distance and N–H stretching frequency fit very well into the approximately linear relationship demonstrated by Nakamoto, Margoshes & Rundle (1955). This $N–H\cdots F$ bond must, incidentally, be one of the weakest ever reported, and yet its authenticity seems assured. It does not seem possible to identify the band corresponding to $N^+–H$ stretching. It should presumably be rather broad, and may be lost in the region of C–H stretching.

Table 7. *Bond distance and stretching frequency for* $N–H\cdots F$ *bonds*

(Adapted from Nakamoto, Margoshes & Rundle, 1955). Non-bonded N-H frequency, 3400 cm^{-1}.

	Bond distance	Stretching frequency	$\Delta\nu$
$N_2H_4.2HF$	2·62 (2) Å	2548 cm^{-1}	852 cm^{-1}
NH_4HF_2	2·80 (3)	2910	490
NH_3BF_3	3·01 (3)	3338	62
This work	3·040 (5)	3367	33

Computer programs used in this work are those of Ahmed, Hall, Pippy & Huber (1966), Gantzel & Trueblood (*MGTLS, thermal motion analysis*) and C. K. Johnson (*ORTEP, thermal ellipsoid plot*). The specimen material was supplied by Dr G. A. Neville, with whom the author acknowledges much helpful discussion.

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The Crystal Structure of Strontium Di-iodate(V) Monohydrate

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The crystals of strontium di-iodate(V) monohydrate, $Sr(IO_3)_2.H_2O$, are monoclinic, with unit-cell constants $a=13{\cdot}156$ (16), $b=7{\cdot}741$ (6), $c=8{\cdot}914$ (15) Å, $\beta=132{\cdot}9$ (3)°, space group $C2/c$ and $Z=4$. The structure consists of pyramidal anions IO_3^- with bond distances I–O = 1·786, 1·806 and 1·825 Å, of Sr^{2+} cations and of water molecules. The environment of each iodine atom is completed by four oxygen atoms of three different pyramidal anions and the resulting coordination polyhedron can be described as a distorted pentagonal bipyramid. Sr^{2+} cations are surrounded by nine oxygen atoms (one from the water molecule): the coordination polyhedron is a trigonal prism with non-basal faces centred. The bond distances Sr–O are in the range 2·531–3·136 Å.

Introduction

In connexion with researches on salts of oxyacids of iodine(V) and iodine(VII), the crystal structure of strontium diiodate(V) monohydrate, $Sr(IO_3)_2.H_2O$, has been determined by X-ray diffraction methods.

Experimental

Preparation

The crystals were prepared in the form of stable, shiny, colourless prisms by concentrating solutions obtained by dissolving strontium carbonate in aqueous iodic acid.

Crystal data

Compound: Strontium di-iodate(V) monohydrate, $Sr(IO_3)_2.H_2O$;
Formula weight: 455·49;
Crystal class: monoclinic prismatic;
Unit-cell parameters (determined from rotation and Weissenberg photographs and refined on an automated single crystal diffractometer, Mo $K\alpha$ radiation, $\lambda=0\cdot71069$ Å):
$a=13\cdot156$ (16), $b=7\cdot741$ (6), $c=8\cdot914$ (15) Å;
$\beta=132\cdot9$ (3)°; $V=665\cdot6$ Å^3; $Z=4$;
$D_x=4\cdot54$, $D_m=4\cdot52$ g.cm^{-3};
μ(Mo $K\alpha$)$=177\cdot5$ cm^{-1}; $F(000)=808$;
Space group: $C2/c\,[C_{2h}(6)$ – No. 15] or $Cc[C_s(4)$ – No. 9] from systematic absences; $C2/c$ was chosen and confirmed by structure determination.

Intensity data

The intensity data were collected on a single-crystal automated Siemens diffractometer on line with a Siemens 304/P computer using Zr-filtered Mo $K\alpha$ radiation and the ω–2θ scan technique. A thin prismatic crystal of approximate dimensions $0\cdot040\times0\cdot055\times0\cdot250$ mm was aligned with its [001] axis along the φ axis of the diffractometer and all the reflexions with $2\theta\leq72°$ were collected. Intensities of 1588 independent reflexions were measured and as many as 1281 of them, having $I>2\sigma(I)$, were used in the crystal analysis $\{\sigma^2(I)=[\text{total counts}+(0\cdot005\ I)^2]\}$.

Calculations

After the usual Lorentz and polarization corrections, the intensities were put on absolute scale by Wilson's (1942) method. No correction for absorption effects was made ($\mu\bar{R}_{[001]}\simeq0\cdot4$).

The positions of the heavy atoms were located from a three-dimensional Patterson function. A Fourier synthesis was then computed and the positions of the remaining non-hydrogen atoms were determined. The refinement was carried out by means of cycles of full-matrix least-squares, minimizing the function $\sum w|\Delta F|^2$; at the beginning isotropic and in the last cycles anisotropic thermal parameters were introduced. Unit weights were used at first, then the scheme: $1/w=(A+BF_o)^2$ was assumed; $A(=2\cdot300)$ and $B(=0\cdot0215)$ were determined by plotting ΔF against $|F_o|$. In this way the R index improved down to the final value of 3·9%.

The final atomic coordinates are given in Table 1 and the thermal parameters in Table 2. A list of the observed and calculated structure factors is available from the authors on request.

Table 1. *Fractional atomic coordinates* (× 10^4) *with e.s.d.'s*

	x	*y*	*z*
Sr	5000	3414 (1)	7500
I	3097 (1)	0308 (1)	2952 (1)
O(1)	1418 (4)	−0665 (7)	1207 (7)
O(2)	3138 (5)	0868 (6)	4960 (7)
O(3)	4160 (4)	−1642 (6)	4052 (6)
H_2O	5000	6683 (10)	7500

The atomic scattering factors used were those of Cromer & Mann (1968). All the calculations were performed on the CDC 6600 computer of the Centro di

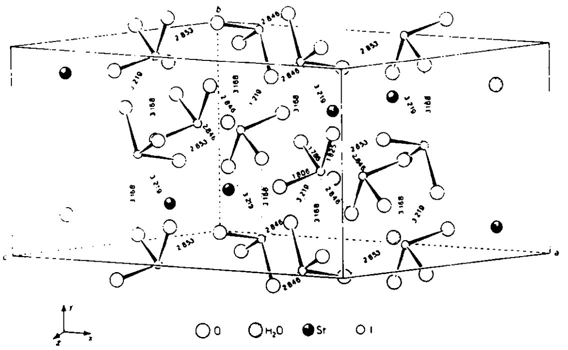

Fig. 1. Clinographic projection of the structure.

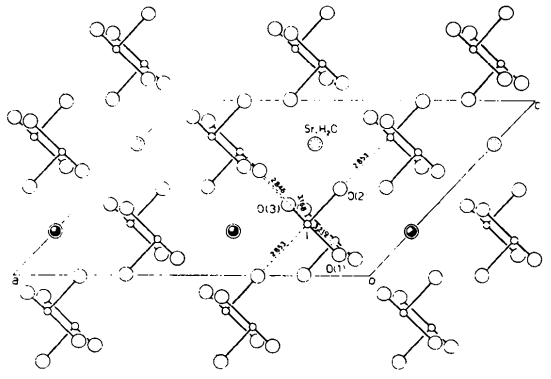

Fig. 2. Projection of the structure on the plane (010).

Table 2. *Anisotropic thermal parameters with e.s.d.'s* (Å^2)

The B_{ij} values refer to the formula:

$$\exp\left[-(b_{11}h^2+b_{22}k^2+b_{33}l^2+b_{12}hk+b_{13}hl+b_{23}kl)\right] \text{ in which } b_{11}=\tfrac{1}{4}a^{*2}B_{11},\ b_{12}=\tfrac{1}{2}a^*b^*B_{12},\ etc.$$

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Sr	1·818 (24)	1·227 (21)	0·797 (18)	0	0·628 (18)	0
I	0·673 (8)	0·833 (9)	0·488 (8)	0·002 (7)	0·265 (7)	−0·027 (6)
O(1)	0·797 (116)	2·040 (164)	1·076 (125)	−0·329 (109)	0·314 (106)	−0·139 (115)
O(2)	2·100 (156)	1·448 (138)	1·195 (123)	−0·192 (124)	1·240 (127)	−0·391 (113)
O(3)	1·441 (130)	1·340 (130)	0·701 (106)	0·583 (112)	0·431 (102)	0·308 (103)
H_2O	10·948 (886)	0·756 (211)	6·780 (624)	0	8·074 (711)	0

Calcolo Elettronico Interuniversitario dell'Italia Nord-Orientale (Bologna), using the programs written by Immirzi (1967).

Description and discussion of the structure

The clinographic projection of the structure is shown in Fig. 1. The structure consists of pyramidal anions IO_3^-, of Sr^{2+} cations and of water molecules. The anions are joined to one another by I···O interactions; the whole framework can be regarded as formed by layers whose average plane is $(10\bar{1})$ (Fig. 2). The anions of the layer are held together alternately by short interactions I···O = 2·846, 2·846 Å and longer interactions I···O = 3·168, 3·168, 3·219, 3·219 Å. The distance between the average planes through the layers is $\frac{1}{2}d_{10\bar{1}}$. The layers are connected *via* interactions I···O = 2·853 Å and also *via* Sr^{2+} cations and water molecules.

The iodate anion is pyramidal with iodine–oxygen distances I–O(1) = 1·786, I–O(2) = 1·806, I–O(3) = 1·825 Å and angles O–I–O = 98·6, 99·2, 99·9°. The values (Table 3) are comparable with those found in other iodates (Braibanti, Manotti Lanfredi, Pellinghelli & Tiripicchio, 1971). The O(1) and O(3) atoms bridge iodine atoms belonging to the same layer; the O(2) atoms bridge iodine atoms of different layers.

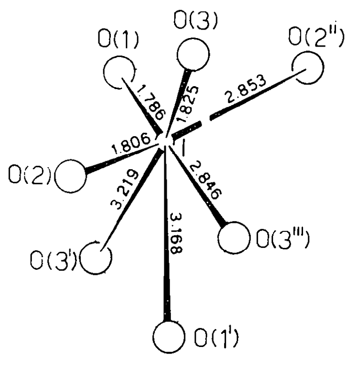

Fig. 3. The coordination polyhedron around the iodine atom.

The environment of each iodine atom in the IO_3^- anion is completed by I···O interactions with other anions; the coordination polyhedron can be represented as a rather deformed pentagonal bipyramid (Fig. 3).

The inter-anion iodine–oxygen distances resemble those found in calcium diiodate(V) hexahydrate and do not have particularly shorter values (~2·5 Å) than those found in other iodates (Braibanti, Manotti Lanfredi, Pellinghelli & Tiripicchio, 1971).

The coordination polyhedron around the strontium ion (Fig. 4) can be described as a trigonal prism with non-basal centred faces. The six corners of the prism are occupied by oxygen atoms belonging to pyramidal anions of different layers. One face is centred by the water molecule, the other two faces are centred by two oxygen atoms belonging to pyramidal anions of different layers. The bonds Sr–O (Table 4) are in the range 2·531–3·136 Å the shortest of which is the bond with the water molecule. This type of coordination has been found in $SrCl_2.6H_2O$ (Jensen, 1940) and in $Sr(VO_3)_2.4H_2O$ (Sedlacek & Dornberger-Schiff, 1965). However, in the compound $Sr(MnO_4)_2.3H_2O$ (Ferrari, Braibanti, Bigliardi & Manotti Lanfredi, 1966) the coordination number is ten, instead of nine, the coordination polyhedron being substantially the same as for ninefold coordination, the additional atom lying along a line passing through the center of one basal face.

In $Sr(OH)_2.H_2O$ (Bärnighausen & Weidlein, 1967), Sr has coordination number eight and the co-

Table 3. *Environment of the iodine atom*

Pyramidal anion				
	I–O(1)	1·786 (8) Å	0(1)–O(2)	2·723 (13) Å
	I–O(2)	1·806 (9)	O(1)–O(3)	2·750 (13)
	I–O(3)	1·825 (6)	O(2)–O(3)	2·778 (11)
	O(1)–I–O(2)	98·6 (3)°	O(2)–O(1)–O(3)	61·0 (2)°
	O(1)–I–O(3)	99·2 (2)	O(1)–O(2)–O(3)	60·0 (2)
	O(2)–I–O(3)	99·9 (2)	O(1)–O(3)–O(2)	59·0 (2)
Interanion contacts				
	I———O(1^{i})	3·168 (6) Å	I———O(3^{i})	3·219 (8) Å
	I———O(2^{ii})	2·853 (11)	I———O(3^{iii})	2·846 (11)
	O(1)—I–O(3^{i})	72·5 (2)°	O(2^{ii})–I–O(3)	75·1 (2)°
	O(3^{i})—I–O(1^{i})	51·0 (1)	O(2^{ii})–I–O(1)	81·4 (2)
	O(1^{i})—I–O(3^{iii})	59·9 (1)	O(2^{ii})–I–O(3^{iii})	100·0 (2)
	O(3^{iii})–I–O(3)	77·1 (2)	O(2^{ii})–I–O(1^{i})	114·0 (1)
	O(2)—I–O(3^{iii})	79·6 (2)	O(2^{ii})–I–O(3^{i})	108·2 (1)
	O(2)—I–O(1^{i})	70·4 (2)	O(2^{ii})–I–O(2)	174·9 (2)
	O(2)—I–O(3^{i})	76·6 (2)		

Asymmetric units							
i	$\frac{1}{2}-x$	$\frac{1}{2}+y$	$\frac{1}{2}-z$	vi	x	$\bar{y}$	$\frac{1}{2}+z$
ii	x	$\bar{y}$	$-\frac{1}{2}+z$	vii	$\frac{1}{2}+x$	$\frac{1}{2}+y$	$1+z$
iii	$1-x$	$\bar{y}$	$1-z$	viii	$\frac{1}{2}+x$	$\frac{1}{2}-y$	$\frac{1}{2}+z$
iv	$\frac{1}{2}-x$	$\frac{1}{2}-y$	$1-z$	ix	x	$1+y$	z
v	$1-x$	y	$\frac{3}{2}-z$	x	$1-x$	$1+y$	$\frac{3}{2}-z$

Table 4. *Coordination around the strontium atom*

For asymmetric units, see Table 3.

Sr——H_2O	2·531 (8) Å	Sr——$O(2^{iv})$	3·136 (15) Å
Sr——$O(3^{iii})$	2·664 (11)	Sr——$O(2^{viii})$	3·136 (15)
Sr——$O(3^{vi})$	2·664 (11)	Sr——O(2)	2·731 (8)
Sr——$O(1^{i})$	2·553 (13)	Sr——$O(2^{v})$	2·731 (8)
Sr——$O(1^{vii})$	2·553 (13)		

O(2)—$O(2^{iv})$	3·061 (10) Å	O(2)—$O(2^{v})$	3·782 (17) Å
$O(2^{iv})$-$O(1^{i})$	3·131 (19)	$O(2^{iv})$-$O(1^{vii})$	4·617 (20)
$O(1^{i})$—O(2)	3·075 (8)		

H_2O—Sr——$O(3^{iii})$	121·0 (1)°	H_2O—Sr——$O(2^{iv})$	79·8 (1)°
$O(3^{iii})$-Sr——$O(3^{vi})$	118·0 (2)	H_2O—Sr——$O(1^{vii})$	73·8 (1)
$O(2^{iv})$-Sr——$O(1^{i})$	65·9 (2)	$O(3^{iii})$-Sr——$O(1^{i})$	70·6 (2)
$O(2^{iv})$-Sr——O(2)	62·4 (2)	$O(3^{iii})$-Sr——$O(2^{viii})$	68·9 (2)
$O(1^{i})$—Sr——O(2)	71·1 (2)	$O(3^{iii})$-Sr——O(2)	69·7 (2)
$O(2^{iv})$-Sr——$O(1^{vii})$	108·1 (2)	$O(3^{iii})$-Sr——$O(2^{v})$	66·7 (2)
O(2)—Sr——$O(2^{v})$	87·6 (1)		

$O(2^{iv})$-O(2)——$O(1^{i})$	61·4 (2)°	$O(1^{vii})$-$O(2^{iv})$-O(2)	76·6 (2)°
$O(2^{iv})$-$O(1^{i})$——O(2)	59·1 (2)	$O(2^{iv})$-O(2)——$O(2^{v})$	104·8 (2)
$O(1^{i})$—$O(2^{iv})$—O(2)	59·5 (2)	O(2)——$O(2^{v})$—$O(1^{vii})$	90·9 (2)
$O(2^{iv})$-$O(1^{i})$——$O(2^{viii})$	104·0 (2)	$O(2^{v})$—$O(1^{vii})$-$O(2^{iv})$	87·7 (2)
$O(1^{i})$—$O(2^{viii})$-$O(1^{vii})$	75·8 (2)		

ordination polyhedron can be described as a trigonal prism with only two non-basal centred faces.

The water molecule is surrounded by a strontium cation and six oxygen atoms of pyramidal anions of different layers. The distances between the water molecule and the oxygen atoms are in the range 2·779–3·051 Å (Table 5) of which $H_2O\cdots O(3^{ix})=2{\cdot}779$ Å and its symmetry related distance form the most acceptable pair of hydrogen bonds. The water molecule presents a very high thermal motion in the (xz) plane but any explanations of this fact seem at the moment to be only speculative.

Table 5. *Coordination around the* H_2O *molecule*

For asymmetric units, see Table 3.

H_2O-Sr	2·531 (8) Å
H_2O-$O(1^{i})$	3·051 (12)
H_2O-$O(1^{vii})$	3·051 (12)
H_2O-$O(1^{viii})$	2·891 (15)
H_2O-$O(1^{iv})$	2·891 (15)
H_2O-$O(3^{ix})$	2·779 (11)
H_2O-$O(3^{x})$	2·779 (11)

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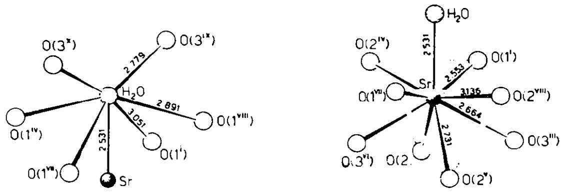

Fig. 4. The environments of the water molecule and of the strontium ion.

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