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Neutron Diffraction Studies of Polyiodides.

I. Potassium Triiodide Monohydrate

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Abstract

The crystal structure of $\text{KI}_3 \cdot \text{H}_2\text{O}$ has been determined directly from neutron diffraction data giving $R_F^2 = 0.027$. This compound crystallizes in the monoclinic space group Pc with $a = 4.629$ (1), $b = 10.047$ (3), $c = 9.662$ (3) Å, $\beta = 116.51$ (1)°, $Z = 2$. The linear I_3^- ions are arranged in planes in a mutually perpendicular manner and are found to be very nearly symmetric [$\text{I}-\text{I} = 2.925$ (3), 2.935 (3) Å]. The K^+ ion and the water molecule are between the iodine planes, each water molecule being coordinated to two K^+ ions.

Introduction

The I_3^- ion is of interest because its structure is malleable to its crystal environment and, because of this, it has received considerable theoretical treatment (Brown & Nunn, 1966; Wiebenga & Kracht, 1969; Gabes & Nijman-Meester, 1973). Several structures containing the I_3^- ion have been determined, usually containing a large cation or a cation solvated with organic molecules (see Table 4, in which are listed structural details of I_3^- salts but excluding the linearly extended polyhalides found in channel inclusion complexes and I_3^- units which form part of higher polyiodides). In order to investigate further the effect of the crystal environment on the structures of poly-

iodides, this study of the polyiodides of hydrated cations was commenced. This report represents the first structural determination of a polyiodide of a simple hydrated cation, which in the past has proved to be difficult by conventional X-ray diffraction because of the poor stability of such compounds to X-radiation. However, because it was possible to grow large crystals the stability problem was overcome by the use of neutron diffraction.

An account of the preparation of $\text{KI}_3 \cdot \text{H}_2\text{O}$ was first published by Johnson (1877) and the macroscopic crystallography was described by Wells, Weeler & Penfield (1892). An initial X-ray examination was carried out by Clark & Duane (1923) and the structure has provided the basis for a doctoral thesis (Finney, 1973).

Preparation and mounting of the crystal

$\text{KI}_3 \cdot \text{H}_2\text{O}$ was prepared by dissolving a mixture of analytical grade KI and I_2 in the proportion 31:60 in a minimum of water to produce a saturated solution on the solubility arc of $\text{KI}_3 \cdot \text{H}_2\text{O}$ as indicated by the ternary phase diagram of Briggs, Clack, Ballard & Sassaman (1940). Slow evaporation over sulphuric acid at room temperature produced a crystal $3.8 \times 4.6 \times 5.5$ mm. The composition of the crystals was checked by analysis ($\text{I}_2 = 58.00\%$; $\text{KI}_3 \cdot \text{H}_2\text{O}$ requires $\text{I}_2 = 57.99\%$).

The mounting of the crystal presented two problems. The compound is corrosive, especially to organic material, so that the crystal cannot be glued to a pin, and it is also very hygroscopic. A holder consisting of a cradle appropriate to the size of the crystal was formed on the end of a piece of silica rod which was glued upright on to a flat glass base. The crystal, while still wet with mother liquor, was placed in the holder and then dried in a desiccator to make it stick to the silica. A cap made from silica tubing of 1 mm wall thickness was placed over the crystal and glued to the base to exclude moisture.

Experimental

The structure was determined directly from neutron diffraction data collected on a four-circle diffractometer provided by the Australian Institute of Science and Engineering (AINSE), at the Australian Atomic Energy Commission (AAEC) research establishment. The wavelength of the neutrons, $\lambda = 1.2401 \text{ \AA}$, was determined from diffraction measurements of a LiF crystal ($a = 4.0262 \text{ \AA}$). The monoclinic cell dimensions obtained by Finney (1973) were refined from the orientation angles of some forty selected reflections. The density was not redetermined, since that found by Finney was in good agreement with our calculated density of 3.60 Mg m^{-3} for $Z = 2$ (Table 1). Reflections were systematically absent for $h0l$, $l = 2n$, indicating the two space groups $P2/c$ or Pc . On the basis of intensity statistics, the non-centrosymmetric space group Pc was chosen. The linear absorption coefficient ($\mu = 0.0554 \text{ mm}^{-1}$) was calculated from the sum of the values for the absorption and scattering cross sections of K, I and O ($3.58, 8.04, 4.24 \times 10^2 \text{ fm}^2$ respectively at $\lambda = 1.24 \text{ \AA}$) from Bacon (1975), and the value for the slow-neutron scattering cross section for H in water from Melkonian (1949) ($40 \times 10^2 \text{ fm}^2$ at $\lambda = 1.24 \text{ \AA}$). Two sets of equivalent reflections were collected for $2\theta < 70^\circ$, corrected for absorption and by the Lorentz factor, analysed, and combined to give 378 independent reflections.

Table 1. *Crystal data*

KI ₃ ·H ₂ O	
Crystal system: monoclinic	
Space group: Pc	
$a = 4.629 (1) \text{ \AA}$	$Z = 2$
$b = 10.047 (3)$	$\mu_c = 0.0554 \text{ mm}^{-1}$ *
$c = 9.662 (3)$	$D_c = 3.60 \text{ Mg m}^{-3}$
$\beta = 116.51 (1)^\circ$	$D_o = 3.60 (1) \text{ (Finney, 1973)}$

* Calculated for neutrons, $\lambda = 1.2401 \text{ \AA}$.

Structure determination and refinement

MULTAN 76 (Main, Woolfson, Lessinger, Germain & Declercq, 1976) was used to solve the structure. The values used for the coherent neutron scattering lengths, obtained from Shull (1972), were 3.70, 5.28, 5.80 and -3.74 fm for K, I, O and H respectively. *MULTAN* produced numerous combinations of phases having similar figures of merit, which was a result of translations of the cell in x and z . In this space group these translations do not affect the relative coordinates of the atoms and in order to locate the cell, the x and z coordinates of I(3) were fixed. The initial atomic coordinates of the non-hydrogen atoms so obtained were refined isotropically with *LINUS* (Coppens & Hamilton, 1970), and the positions of the H atoms were determined from a difference map. The introduction of anisotropic temperature factors for all the atoms gave a final $R_{F^2} = \frac{\sum ||F_o^2| - |F_c^2||}{\sum |F_o^2|} = 0.027$.* The positional parameters are listed in Table 2.

The bond lengths and angles calculated by *ORFFE* (Busing, Martin & Levy, 1964) are listed in Table 3. The H—O distances averaged over thermal motion, the H being assumed to ride on the O atom, are O—H(1) = $1.007 (8)$ and O—H(2) = $1.016 (8) \text{ \AA}$.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35473 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Positional parameters with e.s.d.'s in parentheses*

	x	y	z
K	0.0273 (10)	0.7635 (4)	0.1218 (5)
I(1)	0.3326 (5)	0.4540 (2)	0.4054 (3)
I(2)	0.3660 (10)	0.2438 (3)	0.2021 (3)
I(3)	0.3887 (0)	0.0316 (2)	0.0
O	0.6876 (10)	0.7334 (3)	0.2915 (4)
H(1)	0.7557 (15)	0.7924 (6)	0.3711 (7)
H(2)	0.7153 (14)	0.6502 (5)	0.3373 (6)

Table 3. *Interatomic distances (Å) and angles (°) with e.s.d.'s in parentheses*

Symmetry code: (') $x, 1 - y, \frac{1}{2} + z$; (") $x, -y, \frac{1}{2} + z$.			
I(1)—I(2)	2.935 (3)	O—H(1)	0.908 (7)
I(2)—I(3)	2.925 (3)	O—H(2)	0.928 (7)
I(1)⋯I(2')	4.130 (3)	O—I(2)	3.661 (4)
I(2)⋯I(3'')	3.960 (3)	H(1)—I(3)	3.074 (6)
K—O	2.766 (3)	H(2)—I(1)	2.915 (5)
K—O'	2.749 (3)		
I(1)—I(2)—I(3)	178.8 (2)	H(1)—O—H(2)	105.2 (5)
I(2)—I(1)—I(2')	175.2 (1)	O—K—O'	114.2 (1)
O—H(2)—I(1)	138.5 (3)		

Discussion

$KI_3 \cdot H_2O$ contains planes of linear I_3^- units which are arranged within the plane, normal to each other in a T-shaped pattern, the central I atom of one unit being almost collinear with the line of the neighbouring unit (Figs. 1, 2). This motif is very similar to that found in the triiodides of Rb^+ , Cs^+ , NH_4^+ and Et_4N^+ , whereas in most of the other triiodide structures the anions are arranged parallel (for references, see Table 4). Because this structure is not centrosymmetric, there are two distances (3.960, 4.130 Å) between the end of one I_3^- unit and the central I atom of the next. This separation between the units is slightly less than two van der Waals radii for I (4.30 Å, Pauling, 1960), but considerably greater than the intermolecular distance of solid I_2 (3.496 Å, van Bolhuis, Koster & Migchelsen, 1967), and indicates that there is only a weak interaction between the I_3^- units. That there is at least some interaction between the units is indicated by the

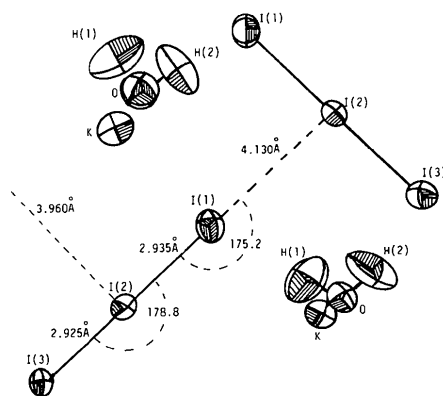


Fig. 1. A perspective view of potassium triiodide monohydrate (ORTEP, Johnson, 1965) in the bc plane in which the triiodide anions form this T-shaped motif.

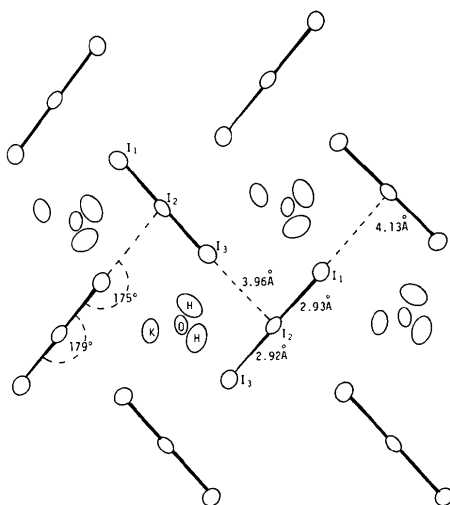


Fig. 2. A view of potassium triiodide monohydrate in the bc plane.

Table 4. Dimensions of triiodide ions

(a) Symmetric triiodide ions (in order of bond length)

	I(1)–I(2)	I(1)···I(3)	Reference
[Mo(diams) ₂ (CO)Cl]I ₃	2.903 Å	5.806 Å	(1)
[Mo(OC)(CNCH ₃) ₄]I ₃	2.914	5.828	(2)
[Ph ₄ As]I ₃	2.92	5.840	(3)
Eu(ep) ₂ I ₃	2.925	5.850	(4)
[Et ₄ N]I ₃	2.928	5.856	(5)
[(C ₂ H ₅) ₂ Fe]I ₃	2.93	5.86	(6)
KI, KI ₁ , valinomycin	2.93	5.86	(7)
RbI(I ₃)(BiI ₃)·2H ₂ O	2.935	5.870	(8)
KI(xanthotoxin) ₂	2.939	5.878	(9)
[Et ₄ N]I ₃	2.943	5.886	(5)
KI, KI ₁ , 6(CH ₃ CONHCH ₃)	2.945	5.890	(10)

(b) Asymmetric triiodide ions (in order of their degree of asymmetry)

	I(1)–I(2)	I(2)–I(3)	Δ	I(1)···I(3)	Reference
KI ₃ ·H ₂ O	2.925 Å	2.935 Å	0.010 Å	5.860 Å	(11)
[MoCl ₄ (diams) ₂]I ₃	2.890	2.912	0.022	5.812	(12)
[(C ₂ H ₅) ₂ N ₂] ₂ I ₃	2.899	2.931	0.032	5.830	(13)
[Et ₄ N]I ₃	2.912	2.961	0.049	5.873	(5)
[W(CO) ₄ (diams) ₂]I ₃	2.895	2.957	0.062	5.852	(14)
[Et ₄ N]I ₃	2.895	2.981	0.089	5.873	(5)
[(C ₂ H ₅) ₂ Fe]I ₃	2.85	2.97	0.12	5.82	(15)
[(C ₂ H ₅) ₂ Fe]I ₃	2.86	3.00	0.14	5.86	(15)
CsI ₃	2.840	3.042	0.202	5.882	(3)
RbI ₃	2.792	3.016	0.224	5.808	(16)
NH ₄ I ₃	2.802	3.114	0.312	5.916	(17)

References: (1) Drew & Wilkins (1973). (2) Lam, Lewis & Lippard (1976). (3) Runsink, Swen-Walstra & Migchelsen (1972). (4) Sohn, Schlueter, Hendrickson & Gray (1974). (5) Migchelsen & Vos (1967). (6) Bernstein & Herstein (1968). (7) Neupert-Laves & Dobler (1975). (8) Lazarini (1977). (9) Kapon & Herstein (1974). (10) Toman, Honzl & Jeený (1965). (11) This work. (12) Drew & Wilkins (1974). (13) Pritzkow (1975). (14) Drew, Egginton & Wilkins (1974). (15) Bats, de Boer & Bright (1971). (16) Finney (1973). (17) Cheesman & Finney (1970).

shiny black lustre of these crystals compared to the pale yellow of tetraphenylarsonium triiodide when the units are separated by 5.20 Å (Runsink, Swen-Walstra & Migchelsen, 1972). The separation of the I_3^- units in $KI_3 \cdot H_2O$ is similar to that in the four similar structures mentioned earlier.

A remarkable feature of this triiodide is that it is very nearly symmetrical, the difference in the two I–I lengths being only 0.01 Å, rather than what might be expected from the trend of the four similar structures, which would indicate that the smaller the cation, the more asymmetrical the I_3^- anion. It has, however, been well established that the symmetry of the I_3^- ion depends not so much on the size of the cation but upon whether the environment of the ion within the crystal lattice is symmetrical or not (Migchelsen & Vos, 1967; Wiebenga & Kracht, 1969). The distances of the cations from the I atoms in the I_3^- ion for $KI_3 \cdot H_2O$ and CsI_3 are listed in Table 5. When these are compared with the sum of the ionic radii for $K^+ - I^-$, 3.49 Å, and $Cs^+ - I^-$, 3.85 Å, it is at once apparent that the K–I distances always exceed the sum of the ionic radii, while those for Cs–I do not, particularly for the I atom on the end of the I_3^- ion containing the longer bond. On the other hand, the asymmetry of the I_3^- anions found in ammonium triiodide and a number of tetraalkylammonium triiodides [Me_4N^+ , Et_4N^+ , $(n-Bu)_4N^+$] by Nakamura & Kubo (1975), where the point-charge effect of the cation is negligible, can be accounted for

Table 5. Distances of atoms from the triiodide ion (Å) with e.s.d.'s in parentheses

	KI ₃ ·H ₂ O	CsI ₃
I(1)—M ⁺	3.674 (3), 3.713 (4), 3.880 (4)	3.70, 3.84, 3.88
I(2)—M ⁺	3.839 (4)	4.10
I(3)—M ⁺	3.778 (3), 3.863 (4)	3.94, 4.02, 4.25
I(1)—O	3.662 (4), 3.833 (4)	
I(2)—O	3.812 (3)	
I(3)—O	3.745 (4), 3.785 (3), 3.918 (3)	
I(1)—H	2.915 (5), 3.287 (6), 3.880 (6)	
I(2)—H	3.206 (6), 3.627 (5)	
I(3)—H	3.074 (5), 3.163 (5)	

by an unequal covalent interaction of I in neighbouring anions on to the terminal I atoms of the I₃⁻ ion.

This view is similar to that taken by Mooney Slater (1959) and fits in with the approach of Hach & Rundle (1951), that the I₃⁻ ion is a resonating system of an I⁻ ion and an I₂ molecule in which equivalent bonds would be expected. Any asymmetry within the ion can then be attributed to an unequal distribution of charge about the end I atoms which would cause a difference in the ratio of the contributions of the two resonating structures. They considered that the I₃⁻ ion might have different dimensions in different environments but it would appear that the overall length of the I₃⁻ ion deforms according to the influence of its surroundings rather than the opposite view that the asymmetry is fundamental and the symmetric ions are constrained to equivalence by suitable surroundings. Furthermore, the classification of triiodides by Nakamura & Kubo (1975) into three groups, *viz* (i) triiodides having symmetric I₃⁻ ions, (ii) those having nearly symmetric I₃⁻ ions, and (iii) those having markedly asymmetric I₃⁻ units, seems superfluous. From the structural data of triiodides listed in Table 4, it would appear that an I₃⁻ ion may be symmetrical or have any degree of asymmetry according to the requirements of its environment.

Whether the I₃⁻ ion in KI₃·H₂O is, in fact, asymmetric is a moot point, since the difference in bond lengths could be due to experimental error, the difference, 0.01 Å, being only 3σ. The precise symmetry of all the symmetric I₃⁻ ions listed in Table 4 is a consequence of crystal symmetry whereas, in this case, the central I atom is not on a special position. The apparent symmetry is, however, unlikely to be due to the disorder of asymmetric units equally distributed throughout the crystal lattice, in view of the good refinement of this non-centrosymmetric structure and shape of the thermal ellipsoids (Figs. 1, 3) which have a major axis at right angles to the line of the I₃⁻ unit. The view expressed by Bats, de Boer & Bright (1971) that, when no crystallographic symmetry is imposed on the anion, invariably two significantly different I—I distances are found in the same structure certainly does not appear to hold here. Nuclear quadrupole resonance

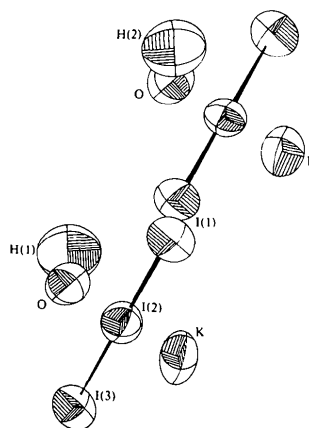


Fig. 3. A perspective view of potassium triiodide monohydrate (ORTEP, Johnson, 1965) parallel to the plane of the iodine layer. Only one hydrogen ellipse for each water molecule is included for clarity since the two hydrogens are superimposed in this view.

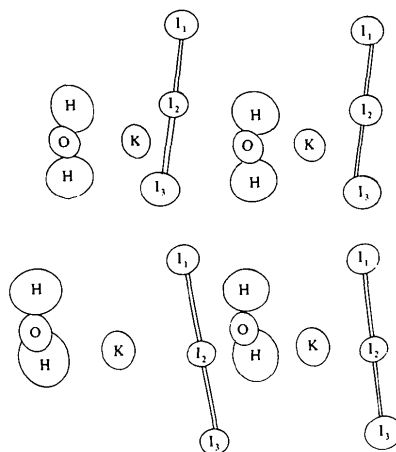


Fig. 4. A view of potassium triiodide monohydrate parallel to the plane containing the I₃⁻ ions.

is one spectroscopic technique which has been used successfully to confirm the crystallographically determined symmetry of several I₃⁻ ions and has predicted its symmetry in a number of other compounds (Nakamura & Kubo, 1975). It is quite possible that this technique could confirm the symmetry of the anion in KI₃·H₂O, although the end I atom resonances in I₃⁻ cannot always be detected (Hacobian, 1979).

The K⁺ ion and the water molecule are positioned between the planes containing the I atoms, Fig. 4, in contrast to the structures of the triiodides of Cs⁺, Rb⁺ and NH₄⁺, in which the cation is within the plane of the I₃⁻ anion. The K⁺ ion is equidistant to two water molecules, the cation—H₂O—cation angle being 114.2°. Although this compound is not known in the anhydrous state, the hydrogen bonding in this structure appears to be only weak. The minimum I—H distances are 2.915 and 3.075 Å, only a little less than the sum of the van der Waals radii of 3.15 Å (Bondi, 1964).

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Liaisons Hydrogène dans les Cristaux de Sulfate de Vanadyle Trihydraté (VOSO₄·3H₂O).

Comparaison Structurale de Quatre Sulfates de Vanadyle Hydratés

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Abstract

The crystal structure of VOSO₄·3H₂O has been redetermined from three-dimensional X-ray data collected by a Nonius CAD-4 diffractometer with Mo K α

radiation. The space group is $P2_1/n$, with $a = 7.387$ (2), $b = 7.401$ (2), $c = 12.046$ (2) Å, $\beta = 106.57^\circ$, $Z = 4$, $d_m = 2.25$, $d_x = 2.28$ Mg m⁻³, $V = 631.23$ Å³, 2117 observed reflections. The H atoms were located from difference syntheses. Structural