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Neutron Diffraction Study of Diammonium Trihydrogen Periodate, (NH₄)₂H₃IO₆, and Its Deuterium Analogue, (ND₄)₂D₃IO₆

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Abstract

Diammonium trihydrogen periodate, $(NH_4)_2H_3IO_6$, crystallizes in the trigonal space group $R\bar{3}$ with $Z = 3$. The unit-cell parameters are $a = 6.9233$ (5), $c = 11.1491$ (8) Å for the non-deuterated substance and $a = 6.9308$ (6), $c = 11.1613$ (16) Å for the deuterated one. Full-matrix least-squares refinements based on neutron ($\lambda = 1.04$ Å) intensities yielded $R(F) = 0.057$ and 0.061, $R_w(F^2) = 0.088$ and 0.094 for 634 and 663 reflections of the non-deuterated and deuterated substance respectively. The crystal structure consists of layers of nearly regular IO_6 octahedra and NH_4 tetrahedra, linked within the layer by nearly linear [177.1 (3) and 176.7 (2)°] N–H···O hydrogen bonds [with the H···O distance 1.853 (3) and the D···O distance 1.838 (3) Å]. The layers are perpendicular to

the trigonal axis and the neighbouring layers are bonded together by nearly linear [177.0 (5) and 175.8 (3)°] O–H···O hydrogen bonds, with the O–H distance 1.018 (5) and the O–D distance 1.015 (4) Å. The H atoms in these bonds are dynamically disordered; their positions 0.712 (8) and 0.718 (6) Å apart are related by the centre of symmetry in the middle of the O–H···O bond. The IO_6 octahedra are identical (within experimental errors) in both structures [I–O = 1.888 (2), O···O = 2.674 (3) and 2.654 (3) Å], while in the NH_4 group the N–H distances [1.034 (6) and 1.046 (3) Å] are significantly shorter (by ~5σ) than the N–D bonds [1.067 (4) and 1.055 (2) Å].

Introduction

Diammonium trihydrogen periodate has attracted a great deal of attention (for an exhaustive literature

review see Gränicher, Kind, Meier & Petter, 1968; Kind, 1971) because of its phase-transition order–disorder phenomena of hydrogen bonding and because of the shift of phase-transition temperature from 253 K for the non-deuterated substance to 266 K for the deuterated one. The structural formula and crystal structure were determined by Helmholz (1937) and confirmed by Gränicher et al. (1968).

This neutron diffraction study was undertaken to determine H positions, which was not possible in previous X-ray studies because of the presence of the I atom, and to see what effect (if any) the deuteration of the substance has on the hydrogen bonding. This work is a part of a more comprehensive study of phase transitions of ammonium and silver periodates (Rüegg, 1977).

Experimental

Samples for the measurement were cut from colourless, transparent plate-like crystals grown by W. Huber, Laboratory of Solid State Physics, ETH Zürich. The samples were stuck by a contact adhesive (Cyanolite) to thin vanadium rods mounted on a goniometer head and dipped into paraffin oil to cover them with a thin layer which prevented their decomposition by moisture in the air.

An accurate re-determination of the unit-cell parameters from 25 accurately centred high-angle reflections [four-circle Syntex $P2_1$ diffractometer, λ(Mo $K\alpha_1$) = 0·709300 (1) Å] was undertaken for both non-deuterated and deuterated samples cut from the same crystals as the samples for the neutron work. The deuteration caused a significant expansion ($\Delta a \sim 10\sigma$, $\Delta c \sim 7\sigma$) of both unit-cell dimensions by 0·11%.

Neutron diffraction measurements were performed on a four-circle neutron diffractometer at the Saphir reactor of the Swiss Federal Institute for Reactor Research at Würenlingen. The space-group extinctions and magnitudes of the unit-cell parameters (an absence of superstructure reflections in view of the sensitivity of the neutron diffraction to H positions) were verified by measuring reflections which should be systematically extinct by space-group extinction: $-h + k + l \neq 3n$ and reflections $h/2$, $k/2$, $l/2$ in $\sin\theta/\lambda \leq 0·25$ Å^{-1}. No violations were observed and no superstructure reflections were found.

Integrated intensities were derived from reflection profiles and their standard deviations $\sigma(I)$ from counting statistics. Intensities of unobserved reflections were given a value that was 50% of the 'threshold' of measurable intensity with a $\sigma(I)$ of the same value and included in the subsequent least-squares refinement. Linear absorption coefficients μ were determined by transmission of pin-hole-collimated neutrons through crystal platelets, and absorption corrections and mean path lengths (for extinction corrections) were com-

Table 1. *Crystal and experimental data for diammonium trihydrogen periodates (see also Abstract)*

	Non-deuterated sample	Deuterated sample
Molecular weight	262·002	273·068
Calculated density (Mg m^{-3})	2·842	2·952
Principal growth faces	(101) and (001)	
Data collection		
Wavelength	1·040 Å	
Monochromator	Pyrolytic graphite	
$\lambda/2$ contamination	~1%	
Scan length (°)	1·45 (0·44 + 2·26 tan θ)	
Scanning mode	ω:2θ, 20–30 points per reflection	
Monitor count	40 000–400 000	
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0·725	0·744
Sample mounting	(101) and (001) planes parallel with diffractometer φ axis	
Number of reference reflections and their behaviour	3, no significant variation	
Interval between measurement of reference reflections	~24 h	
Range of intensities (relative scale)	1–144	1–630
Crystal sample and absorption corrections		
Weight	178·6 mg	83·1 mg
Volume	~63 mm^3	~28 mm^3
Shape	Polyhedron, 4·9 × 4·8 × 2·7 mm	Polyhedron, 4·5 × 4·5 × 1·8 mm
Number of boundary planes	6	8
Linear absorption coefficient μ (including incoherent scattering)	0·290 mm^{-1}	0·07844 mm^{-1}
Range of transmission factor	0·301–0·558	0·736–0·892
Range of mean path length	1·731–3·774 mm	1·400–3·736 mm

puted. Crystal data and experimental details are summarized in Table 1.

Structure determination and refinement

The refinement was performed in the space group No. 148, $R\bar{3}$, using a non-primitive hexagonal unit cell (with lattice points 0,0,0; $\frac{2}{3},\frac{1}{3},\frac{1}{3}$ and $\frac{1}{3},\frac{2}{3},\frac{2}{3}$, i.e. with the 'obverse' orientation of rhombohedral to hexagonal axes, which is the orientation adopted as standard in *International Tables of X-ray Crystallography*, 1952). The values of the atomic coordinates determined by Helmholz (1937), $x_O = 0·232$ (3), $y_O = 0·216$ (3), $z_O = 0·105$ (5)* and $z_N = 0·340$ (10) which refer to the 'reverse' orientation, were transformed to the 'obverse' one according to the equation

$$x,y,z_{obv} = y, y - x, z_{rev}$$

and thus the values $x_O = 0·216$, $y_O = -0·016$, $z_O = 0·105$ and $z_N = 0·340$ were used as starting parameters. Structure factors, calculated without the contribution of H atoms ($R \simeq 0·20$), were used in a difference Fourier synthesis [computed with a local version of the XRAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972)], which revealed the positions of the H atoms and a consecutive difference synthesis showed that the position of the H(1) atom which forms the hydrogen bond between the IO$_6$ octahedra is split into two positions related by the centre of symmetry in the middle of the hydrogen bond.

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* The value $z_O = 0·135$ (5) of Kind (1971) is in error.

In an early stage of the refinement the necessity for including extinction corrections was recognized. This was done only for isotropic secondary extinction according to Zachariasen (1967) with a modified version of the *ORFLS* program (Busing, Martin & Levy, 1962) on non-averaged sets of structure factors, to allow for different mean path lengths of the beam in the sample for symmetry-related reflections. The occupancy factors and all scattering lengths (Bacon, 1972), except that for substituted deuterium atoms, were kept fixed during the full-matrix least-squares refinement. For the starting value of the scattering length of the substituted D atoms we used $b_D = 5.32$ fm which corresponds to a mixture of $87 \pm 1\%$ D atoms and 13% H atoms, determined by infrared spectroscopy. At a later stage of the refinement the scattering lengths $b[D(1)]$, $b[D(2)]$ and $b[D(3)]$ were relaxed and they converged to values which, when averaged, correspond to an almost complete deuteration of the sample ($95.5 \pm 2.5\%$). For a discussion of this discrepancy see *Conclusions*.

At the end of the refinement an attempt was made to refine the third cumulants (Johnson, 1969) as well. For both substances this attempt resulted in a non-positive semi-definite form of $\sum b_{ij} h_i h_j$ for atoms O and H(1) and therefore the results are not presented.

Details of the refinement are given in Table 2. Difference Fourier maps computed at the end of the refinement for all independent reflections with F_{obs} corrected for extinction and F_{calc} showed no spurious

Table 2. *Summary of the least-squares refinement*

The extinction coefficient y is defined (Zachariasen, 1967) by $F^2_{measured} = y\,F^2_{kinematical}$. $F_{corr} = F_{obs}/\sqrt{y}$ is the observed structure factor corrected for extinction.

Formula	$(NH_4)_2H_3IO_6$	$(ND_4)_2D_3IO_6$
Number of parameters refined (*n*)	36	39
Number of observations, including zero intensities (*m*)	634	663
Number of independent reflections (m_1)	320	345
Ratio m_1/n	8.89	8.45
Maximum shift/e.s.d. ratio in the final LS cycle	<0.06	<0.05
$R(F) = \sum |F_{corr} - |F_{calc}||/\sum F_{corr}$	0.0572	0.0612
$R_w(F^2) = |\sum w(F^2_{corr} - F^2_{calc})^2/\sum wF^2_{corr}|^{1/2}$	0.0881	0.0942
$S = |\sum w(F^2_{corr} - F^2_{calc})^2/(m-n)|^{1/2}$	8.7584	14.0112
Extinction parameter r^* (isotropic, secondary only)	$13\,404 \pm 21$	$4\,408 \pm 39$
Reflection most affected by extinction	009	113
Its value y	0.274	0.281
Weighting scheme	$w = 1/\sigma^2(F^2)$ where $\sigma(F^2) \sim \sigma(I)$	
Refinement based on	F^2	
Function minimized	$\sum w(F^2_{corr} - F^2_{calc})^2$	
Neutron scattering lengths used in refinement (fm)	$b_I = 5.30$	
	$b_O = 5.80$	
	$b_N = 9.40$	
	$b_H = -3.74$	$b_D = 6.67$
Neutron scattering lengths of partially	—	$b[D(1)] = 5.73\ (8)$
substituted H → D atoms (refined) (fm)	—	$b[D(2)] = 6.30\ (11)$
	—	$b[D(3)] = 6.40\ (7)$
Degree of deuteration {based on refined	—	D(1): $91.0 \pm 1.3\%$
values of $b[D(i)]$ and tabulated value	—	D(2): 96.5 ± 1.7
$b_D = 6.67$ fm}	—	D(3): 97.4 ± 1.1
Average degree of deuteration from above values	—	$95.5 \pm 2.5\%$
Average degree of deuteration determined by infrared spectroscopy	—	$87 \pm 1\%$

Table 3. *Fractional atomic coordinates* ($\times 10^5$) *and equivalent values of* B_{iso} (*computed from anisotropic* b_{ij}) *of* $(NH_4)_2H_3IO_6$ *and* $(ND_4)_2D_3IO_6$

Estimated standard deviations (in parentheses) refer to last significant figures.

$(NH_4)_2H_3IO_6$

	x	y	z	B_{iso} (Å²)
I	0	0	0	0.33
O	21850 (21)	−750 (22)	9662 (12)	0.63
N	0	0	35937 (15)	1.15
H(1)	30118 (88)	12492 (87)	14891 (47)	1.31
H(2)	0	0	27201 (51)	4.28
H(3)	15620 (47)	3842 (53)	38720 (30)	2.61

$(ND_4)_2D_3IO_6$

	x	y	z	B_{iso} (Å²)
I	0	0	0	0.53
O	21751 (25)	−887 (26)	9638 (13)	0.80
N	0	0	35904 (17)	1.36
D(1)	29935 (56)	12471 (57)	14781 (30)	1.01
D(2)	0	0	26835 (34)	4.34
D(3)	15876 (32)	4225 (34)	38808 (17)	2.41

peaks. Resultant atomic coordinates are given in Table 3.*

Description of the structure

The non-deuterated and deuterated title substances are isostructural. Table 4 (interatomic distances) shows that significant differences occur only in the bonds where H/D atoms participate. For the sake of brevity, in the following text only one value for a bond or angle will be given; when the differences are significant, the value for the non-deuterated substance will be given first and the corresponding value for the deuterated substance, separated by a ‖ sign, will be second. Estimated standard deviations (e.s.d.'s) refer to last significant figures.

The structure consists of layers perpendicular to the trigonal axis. Within the layer, the I atoms, in the middle of the IO_6 octahedra, form a regular triangular net and the ammonium NH_4 pyramids, directed alternately upwards and downwards, are located in the middle of these triangles. The layers are stacked in the sequence *ABCABCABC*... and along the trigonal *c* axis there is therefore a sequence

$$...-O_3,I,O_3-H(2),N,H(3)_3-H(3)_3,N,H(2)-IO_6-$$
$$NH_4-NH_4-IO_6-NH_4-NH_4-...$$
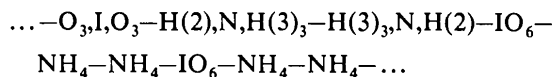
* A list of structure factors, the table of anisotropic thermal parameters and the table of main axes of ellipsoids of thermal vibration have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35090 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. Selected interatomic distances (Å) and angles (°)

The values were calculated with the program ORFFE (Busing, Martin & Levy, 1964) with the variance–covariance matrix and the e.s.d.'s of cell parameters. Bond lengths N—H were corrected for riding motion, all other bond lengths for independent vibration of the atoms. E.s.d.'s in parentheses refer to the last significant figures.

Symmetry code

None	x	y	z	(iii)	$-y$	$x-y$	z
(i)	$-y+\frac{1}{3}$	$x-y-\frac{1}{3}$	$z-\frac{1}{3}$	(iv)	y	$-x+y$	$-z$
(ii)	$-x+\frac{2}{3}$	$-y+\frac{1}{3}$	$-z+\frac{1}{3}$	(v)	$-x+y+\frac{2}{3}$	$-x+\frac{1}{3}$	$z+\frac{1}{3}$

	$(NH_4)_2H_3IO_6$		$(ND_4)_2D_3IO_6$	
	Uncorrected	Corrected	Uncorrected	Corrected
IO_6 octahedron				-
I—O	1·879 (1)	1·887 (1)	1·878 (1)	1·889 (1)
O···O^{iii}	2·666 (2)	2·673 (2)	2·666 (3)	2·674 (3)
O···O^{iv}	2·648 (2)	2·655 (2)	2·645 (3)	2·653 (3)
O—I—O^{iii}	90·40 (6)		90·45 (6)	
O—I—O^{iv}	89·60 (6)		89·55 (6)	
NH_4 tetrahedron				
N—H(2)	0·974 (6)	1·034 (6)	1·012 (4)	1·067 (4)
N—H(3)	1·024 (3)	1·046 (3)	1·039 (2)	1·055 (2)
H(2)···H(3)	1·613 (5)	1·673 (5)	1·661 (4)	1·718 (4)
H(2)—N—H(3)	107·63 (21)		108·18 (15)	
Hydrogen bonding				
O—O^{ii}	2·636 (3)	2·643 (3)	2·656 (3)	2·664 (3)
H(1)—H(1^{ii})	0·657 (9)	0·712 (8)	0·681 (6)	0·718 (6)
H(1)—O	0·992 (5)	1·018 (5)	0·992 (3)	1·015 (4)
H(1)···O^{ii}	1·645 (4)	1·662 (4)	1·666 (4)	1·680 (4)
O—H(1)—H(1^{ii})	172·56 (1·37)		169·78 (82)	
O—H(1)···O^{ii}	177·04 (54)		175·84 (33)	
O···N^{i}	2·852 (1)	2·860 (2)	2·851 (2)	2·861 (2)
O···H(3^{i})	1·829 (3)	1·853 (3)	1·813 (2)	1·838 (2)
N—H(3)···O^{v}	177·14 (32)		176·66 (20)	
O···H(2)	2·489 (5)	2·516 (5)	2·460 (3)	2·489 (3)

Fig. 1. Hydrogen-bonding scheme. Intralayer hydrogen bonds N—H(3)···O hold together IO_6 octahedra and NH_4 tetrahedra. The interlayer hydrogen bonding between the stacked layers (upper layer is represented by IO_6 octahedron with I—O bonds filled) is between O atoms of the IO_6 octahedra through strong, dynamically disordered O—H(1)···O bonds. Disordered positions of H(1) are dotted.

Fig. 2. Stereoview (drawn with ORTEP II, Johnson, 1971) of the unit-cell contents of three formula units. Crystallographically independent atoms are labelled and their coordinates are given in Table 3. Thermal ellipsoids enclose (in both figures) 33% probability.

The IO_6 and NH_4 groups are basic building units of the structure. The former are almost regular octahedra, identical for both structures within experimental errors [with distances I—O = 1·888 (2) and O···O = 2·674 (3) and 2·654 (3) Å], while the latter show a pronounced isotope effect, found also in other substances with hydrogen bonds (Hamilton & Ibers, 1968). The N—H bond between the N atom and the H(2) atom, not participating in the hydrogen bonding, is 0·03 Å longer in the deuterated substance [1·034 (6)||1·067 (4) Å], while the N—H(3) bond [the atom H(3) forms a hydrogen bond to the next IO_6 octahedron] is longer by only 0·01 Å [1·046 (3)|| 1·055 (2) Å].

The IO_6 octahedra and NH_4 tetrahedra are held together by hydrogen bonding (see Fig. 1). Intralayer bonds are nearly linear [177·1 (3)||176·7 (2)°], not disordered hydrogen bonds —N—H(3)···O with the H(3)···O distance 1·853 (3)||1·838 (3) Å. In these bonds the O atoms are linked just to one H(3) atom and vice versa. Almost isotropic vibration of the H(3) atom (see Table 3) indicates that no hindered rotation of the NH_4 group about the trigonal axis takes place.

Neighbouring layers are hydrogen bonded by the interlayer O—H(1)···O hydrogen bonds [with the
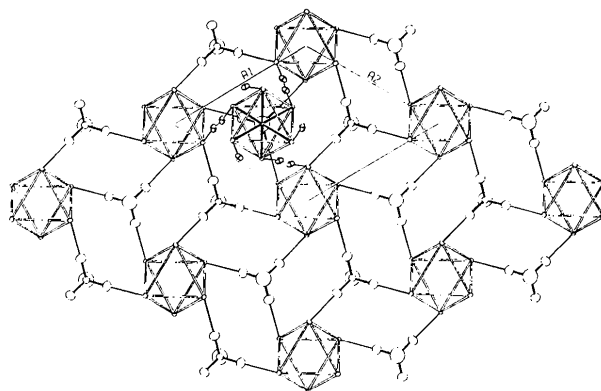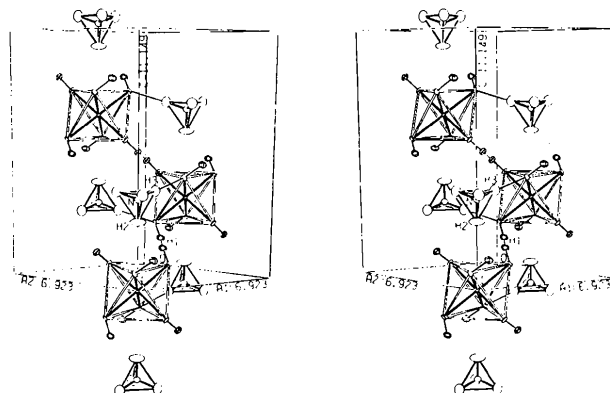
O—H(1) distance 1·017 (5) Å] which are nearly linear [177·0 (5)||175·8 (3)°] and dynamically disordered. The H(1) atom spends 50% of its time in one of the two positions related by the centre of symmetry and 0·715 (7) Å apart along the O—H(1)···O bond. The O···O distances 2·643 (3)||2·664 (3) Å between neighbouring IO_6 octahedra [bonded by the hydrogen bonds O—H(1)···O] are shorter than the O···O distances within IO_6 octahedra for the non-deuterated substance, while for the deuterated one they are of about the same magnitude.

The hydrogen environment of one O atom is illustrated in Fig. 2. The O atom is bonded to one H(3) atom 100% of the time and simultaneously 50% of the time to the H(1) atom, which oscillates between the O atoms of neighbouring IO_6 octahedra. Further, there

are van der Waals contacts [2·516 (5)‖2·489 (4) Å] between the H(2) atom of the ammonium NH_4 group above the IO_6 octahedron and three near O atoms.

Conclusions

Both structures were refined with anisotropic temperature parameters including H atoms. For both structures the extinction corrections amount to more than 70%, *i.e.* less than 30% of the kinematical intensity was really measured for the strongest reflections. Choosing a smaller sample for the deuterated substance did not help in this respect and we think that too high a quality of the crystals, rather than their size, was responsible for the magnitude of the extinction effects.

Although the degree of deuteration determined by infrared spectroscopy ($87 \pm 1\%$) differs by 8·5% from our value of $95 \cdot 5 \pm 2 \cdot 5\%$, computed from resultant least-squares values of b_D, the difference amounts to $\sim 3\sigma$ only and it is encouraging that, even though the scale factor, the refined scattering lengths b_D and the thermal parameters were highly correlated, no scattering length b_D of the substituted D atoms ever exceeded during the refinement the value $b_D = 6 \cdot 67$ fm for pure deuterium. Therefore we believe that the positional parameters of all H and D atoms, including the dynamical disorder in the interlayer $O-H_{disordered}\cdots O$ hydrogen bonds, are well established by this study.

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Hydrogen Bond Studies.
CXXXVIII.* Neutron Diffraction Studies of LiNO₃.3H₂O at 120 and 295 K

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Abstract

LiNO₃.3H₂O has been studied by neutron diffraction at 120 and 295 K. The final R_{w,F^2} values are 4·5% (120 K) and 4·9% (295 K). The crystal structure found in a previous room-temperature X-ray study is confirmed: one water molecule is involved in six hydrogen bonds; the other independent water molecule in the structure has a tetrahedral environment comprising two Li⁺ ions and two medium–strong hydrogen bonds. The four different O⋯O hydrogen-bond lengths increase by 0·02–0·03 Å going from 120 to 295 K. The mean-