The Crystal Structure of Diammonium Bis(oxalato)monaquooxovanadate(IV) Monohydrate

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Diammonium bis(oxalato)monaquooxovanadate(IV) monohydrate is monoclinic with $a=11\cdot162$, $b=7\cdot919$, $c=14\cdot370$ Å, $\beta=117\cdot45°$, $Z=4$. Intensities were collected on a Hilger–Watts four-circle diffractometer with Mo $K\alpha$ radiation ($\lambda=0\cdot7107$ Å). The structure was solved by Patterson and Fourier methods and refined by full-matrix least-squares calculations. R for 1413 observed reflexions was 0·039. The V atom is coordinated to six O atoms in the form of a distorted octahedron. Two pairs of chelated oxalato O atoms form a *cis* arrangement, one of these atoms occupying an apical position. The other apical site is taken by the vanadyl O and the remaining equatorial position by a water O atom. The carboxylato groups are non-planar, the dihedral angles being 10·0 and 12·8°. The relative lengths of the V–O apical bonds demonstrate the well-known *trans* influence: the shorter is 1·594 (3) Å and the other 2·184 (3) Å. The lengths of the equatorial bonds are 1·985 (3), 2·006 (3), 2·022 (3) and 2·033 (3) Å. All O atoms are involved in hydrogen bonding in the form of either inter- or intramolecular contacts. The O–H$\cdots$O bonds range from 2·621 (4) to 3·056 (4) Å. The ammonium ions form N–H$\cdots$O bonds to terminal carboxylato O, coordinated carboxylato O and the lattice water molecule. These contacts range from 2·828 (4) to 3·379 (5) Å. There is also evidence of bifurcation among the N–H$\cdots$O contacts.

Introduction

In a previous publication (Form, Raper, Oughtred & Shearer, 1972) we reported the structure of $(NH_4)_2[VO(C_2O_4)_2H_2O].H_2O$ (AVO) from photographic data. The analysis revealed a six-coordinate complex of the VO^{2+} ion containing a *cis* arrangement of oxalato groups and a coordinated water molecule. This structure was in contradiction to the spectroscopic data which suggested a five-coordinate complex containing *trans* oxalato groups and no coordinated water molecule.

Such an arrangement merited a more precise study, and this has been undertaken with data obtained with a Hilger–Watts four-circle diffractometer.

Experimental

(i) Crystal data

AVO was prepared by the method described by Palmer (1954). Single crystals suitable for X-ray analysis were obtained by recrystallization from aqueous isopropyl alcohol. Comparison of the infrared spectra of the original and recrystallized material confirmed the identity of the product.

The crystals were allocated to space group $P2_1/c$ from Weissenberg and precession photographic data. Cell dimensions were determined on a Hilger–Watts four-circle diffractometer with Mo $K\alpha_1$ ($\lambda=0\cdot70926$Å) radiation (Table 1).

Table 1. *Crystal and experimental data*

Formula	$C_4H_{12}O_{11}N_2V$
F.W.	315
Unit cell	
a	11·162 (1) Å
b	7·919 (1)
c	14·370 (2)
β	117·450 (5)°
V	1200·0 Å³
Systematic absences	$0k0: k=2n+1$
	$h0l: l=2n+1$
Space group	$P2_1/c$
D_c	1·76 g cm⁻³
D_m	1·81
Z	4
μ(Mo $K\alpha$)	9·47 cm⁻¹
$F(000)$	644

(ii) Data collection and reduction

A crystal, $0\cdot40\times0\cdot30\times0\cdot08$ mm, and Zr-filtered Mo radiation ($\lambda=0\cdot7107$Å) were used to collect the data. A $\theta-2\theta$ scan technique was employed and the intensity sets $h,k\pm l$ and $-h,k,\pm l$ were collected out to a value of $\theta=26°$. Each scan consisted of 75 steps at intervals of 0·01°, the total counting time for each reflexion being 150 s, plus 25 s for each of the two background counts performed before and after the scan. The 300, $10\bar{4}$ and 035 reflexions were measured as internal standards every 50 reflexions. 2218 unique reflexions were measured and of these 1413 were greater than 3σ and were considered observed.

The data were corrected for Lorentz and polarization effects but not for absorption.

Structure determination and refinement

The coordinates of the non-hydrogen atoms were obtained by Patterson and Fourier methods and refined by least-squares calculations. The weighting scheme was

$$\sqrt{\omega} = \frac{2N}{F_o\{S + K^2(B_1 + B_2) + (PN)^2\}^{1/2}}$$

where S is the peak count, B_1 and B_2 are the measured background counts and N is the net count. K is the ratio of the time taken for the peak measurement to that for the two backgrounds. P was assigned a value of 0·06. The unobserved reflexions were given zero weight.

Anisotropic, block-diagonal least-squares refinement of the heavy atoms gave $R = 0.054$. At this point a dif-

Table 2. *Final atomic parameters of non-hydrogen atoms*

(a) Fractional coordinates ($\times 10^5$) (e.s.d.'s in parentheses)

	x	y	z
V	32512 (6)	3772 (8)	18203 (5)
O(1)	33540 (26)	−13294 (34)	12734 (20)
O(2)	43742 (28)	17816 (33)	13262 (20)
O(3)	48834 (25)	−611 (30)	32140 (19)
O(4)	61444 (27)	7504 (32)	48641 (19)
O(5)	42806 (28)	34141 (34)	44282 (19)
O(6)	34258 (25)	26157 (32)	27621 (18)
O(7)	15663 (26)	14925 (33)	7904 (18)
O(8)	−4923 (29)	21323 (40)	5650 (23)
O(9)	−1580 (31)	−4401 (46)	19959 (25)
O(10)	20026 (25)	−4517 (34)	23725 (19)
O(11)	−19010 (32)	5599 (37)	34851 (25)
N(1)	−26543 (31)	14369 (41)	11804 (24)
N(2)	11098 (41)	8375 (52)	42888 (30)
C(1)	51829 (39)	9389 (47)	39925 (30)
C(2)	42237 (36)	24668 (49)	37245 (29)
C(3)	5622 (43)	13474 (50)	9899 (30)
C(4)	7797 (41)	249 (53)	18475 (29)

ference map indicated the sites of the 12 H atoms. The coordinates of these atoms were included in the subsequent calculations but not refined. Full-matrix refinement with anisotropic temperature factors for the heavy atoms and an isotropic temperature factor of 5·0 Å² for H, gave $R = 0.039$.

At this stage no shift was greater than one third of the corresponding standard deviation and refinement was terminated. A weighting analysis showed that $\sum \omega^2/N$ was independent of both $|F_o|$ and $\sin^2 \theta$. A difference map based on the final atomic coordinates, showed no significant electron density.

Scattering factors for the heavy atoms were taken from *International Tables for X-ray Crystallography* (1962). The correction (0·3 e) for anomalous dispersion by the V atom (Templeton, 1962) was applied. The scattering factor for H was from Stewart, Davidson & Simpson (1966).

The final positional and thermal parameters are shown in Table 2.*

Description of the structure

A diagram of the anion VO $[(C_2O_4)_2H_2O]^{2-}$ is shown in Fig. 1.

The interatomic distances in the octahedron are given in Table 3 and the bond angles in Table 4. The bond parameters of the oxalato groups are listed in Table 5. Mean planes and atomic displacements are given in Table 6.

The contents of the cell in **b** projection are shown in Fig. 2.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31125 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2 (*cont.*)

(b) Anisotropic temperature factors ($\times 10^5$). The values of b_{ij} are defined by $\exp[-\frac{1}{4}(h^2a^{*2}b_{11} + 2hka^*b^*b_{12} + \ldots)]$ (e.s.d.'s in parentheses).

	b_{11}	b_{22}	b_{33}	b_{23}	b_{13}	b_{12}
V	615 (8)	598 (10)	251 (4)	−36 (12)	366 (8)	7 (17)
O(1)	780 (34)	867 (51)	441 (19)	−373 (52)	478 (43)	−35 (67)
O(2)	1249 (40)	972 (51)	407 (19)	−385 (54)	1040 (47)	−711 (76)
O(3)	674 (30)	722 (51)	295 (16)	−86 (46)	338 (37)	253 (60)
O(4)	650 (31)	870 (53)	294 (18)	70 (46)	188 (40)	−39 (61)
O(5)	1094 (37)	970 (53)	296 (18)	−282 (53)	611 (42)	360 (76)
O(6)	718 (32)	790 (47)	235 (17)	−1 (45)	236 (39)	336 (62)
O(7)	712 (33)	900 (50)	253 (16)	292 (47)	317 (39)	203 (65)
O(8)	704 (37)	1454 (65)	583 (23)	450 (62)	273 (47)	466 (79)
O(9)	794 (37)	2584 (82)	733 (25)	1135 (80)	959 (53)	511 (95)
O(10)	607 (31)	1061 (50)	329 (17)	461 (53)	388 (38)	260 (72)
O(11)	1050 (39)	1154 (60)	723 (25)	−456 (63)	665 (53)	−265 (81)
N(1)	651 (39)	957 (62)	400 (23)	31 (63)	407 (49)	−45 (82)
N(2)	1224 (56)	1899 (89)	543 (29)	−614 (79)	667 (68)	245 (109)
C(1)	513 (45)	637 (63)	359 (27)	122 (68)	503 (59)	−158 (84)
C(2)	570 (45)	665 (66)	330 (27)	43 (70)	437 (58)	−47 (87)
C(3)	716 (51)	759 (76)	282 (25)	−174 (70)	195 (59)	55 (102)
C(4)	625 (47)	1151 (90)	321 (25)	−63 (71)	395 (57)	64 (95)

Discussion

(i) The structure of the complex anion

Sathyanarayana & Patel (1965) have suggested, on the basis of infrared and thermal analysis data, that the anion consists of a five-coordinate V atom with O atoms in a square pyramidal arrangement; the basal plane has at its corners two oxalato groups in the *trans* position, the vanadyl O being located at the apex. Both

Table 3. *Interatomic distances (e.s.d.'s in parentheses)*
(Å)

V——O(1)	1·594 (3)	O(2)—O(7)	2·871 (5)
V——O(2)	2·033 (3)	O(3)—O(1)	2·704 (4)
V——O(3)	2·022 (3)	O(3)—O(10)	2·883 (4)
V——O(6)	2·184 (3)	O(3)—O(6)	2·567 (4)
V——O(7)	1·985 (3)	O(10)-O(1)	2·729 (4)
V——O(10)	2·006 (3)	O(10)-O(7)	2·599 (4)
O(2)-O(3)	2·896 (4)	O(10)-O(6)	2·814 (4)
O(2)-O(6)	2·798 (4)	O(7)—O(1)	2·860 (4)
O(2)-O(1)	2·700 (4)	O(7)—O(6)	2·776 (3)

Table 4. *Bond angles (e.s.d.'s in parentheses)* (°)

O(1)–V–O(2)	95·49 (14)	O(6)–V–O(10)	84·27 (11)
O(1)–V–O(3)	96·09 (13)	O(6)–V–O(2)	83·06 (11)
O(1)–V–O(6)	171·02 (13)	O(6)–V–O(3)	75·12 (10)
O(1)–V–O(7)	105·57 (13)	O(7)–V–O(10)	81·27 (11)
O(1)–V–O(10)	97·97 (13)	O(3)–V–O(10)	91·44 (11)
O(6)–V–O(7)	83·34 (11)	O(2)–V–O(3)	91·19 (11)
		O(2)–V–O(7)	91·20 (12)

Table 5. *Bond parameters of the oxalato groups (e.s.d.'s in parentheses)*

Bond lengths (Å)

C(1)–C(2)	1·542 (6)	C(3)–C(4)	1·549 (6)
C(1)–O(3)	1·283 (4)	C(3)–O(7)	1·284 (6)
C(1)–O(4)	1·226 (5)	C(3)–O(8)	1·217 (6)
C(2)–O(6)	1·259 (4)	C(4)–O(10)	1·275 (5)
C(2)–O(5)	1·237 (5)	C(4)–O(9)	1·217 (6)

Bond angles (°)

O(3)C(1)O(4)	124·7 (4)	O(7)C(3)O(8)	125·7 (4)
O(3)C(1)C(2)	113·2 (3)	O(7)C(3)C(4)	113·9 (4)
O(4)C(1)C(2)	122·0 (4)	O(8)C(3)C(4)	120·3 (4)
O(6)C(2)O(5)	125·8 (4)	O(10)C(4)O(9)	125·4 (4)
O(6)C(2)C(1)	114·0 (3)	O(10)C(4)C(3)	113·8 (4)
O(5)C(2)C(1)	120·2 (3)	O(9)C(4)C(3)	120·8 (4)
VO(3)C(1)	120·8 (3)	VO(7)C(3)	114·7 (3)
VO(6)C(2)	114·9 (2)	VO(10)C(4)	114·8 (3)
O(3)VO(6)	75·1 (1)	O(7)VO(10)	81·3 (1)

water molecules were thought to exist as lattice water, uncoordinated to the metal atom.

The present work indicates that the V atom is six-coordinate, one of the O atoms being provided by a water molecule. As shown in Fig. 1, the two oxalato groups have a *cis* arrangement. A similar arrangement has been found in triammonium bis(oxalato)dioxovanadate(V) dihydrate (Scheidt, Tsai & Hoard, 1971) and in tripotassium bis(oxalato)dioxovanadate(V) (Drew, Einstein & Gransden, 1974).

Table 6. *Planes and atomic displacement in the complex anion*

The general equation for the plane is $lX + mY + nZ = P$ where X, Y and Z are the fractional coordinates with respect to the orthogonal axes a, b and c^* and P is the origin to plane distance in Å.

Plane I	V, O(2), O(3), O(7), O(10):	$0·2424X - 0·8349Y - 0·4942Z = 1·1115$
Plane II	V, O(1), O(3), O(6), O(7):	$0·8084X + 0·4595Y - 0·3679Z = 1·2123$
Plane III	V, O(1), O(2), O(6), O(10):	$-0·4987X + 0·3420Y - 0·7965Z = 2·9788$
Plane IV	C(1), C(2), O(3), O(4), O(5), O(6):	$0·8119X + 0·5495Y - 0·1973Z = 1·9607$
Plane V	C(1), C(2), O(3), O(4):	$0·8051X + 0·5229Y - 0·2799Z = 1·4996$
Plane VI	C(1), C(2), O(5), O(6):	$0·8140X + 0·5699Y - 0·1123Z = 2·4084$
Plane VII	C(3), C(4), O(7), O(8), O(9), O(10):	$0·0331X - 0·7330Y - 0·6794Z = 1·6385$
Plane VIII	C(3), C(4), O(7), O(8):	$-0·0741X - 0·7107Y - 0·6996Z = 1·6367$
Plane IX	C(3), C(4), O(9), O(10):	$0·1427X - 0·7415Y - 0·6556Z = 1·6200$
Plane X	O(2), O(11), N(1), N(2):	$0·0523X - 0·9867Y - 0·1539Z = 1·4815$

Displacements from the mean plane (Å)

	I	II	III	IV	V	VI	VII	VIII	IX	X
V	0·302 (1)	0·030 (1)	0·024 (1)							
O(1)		0·051 (3)	−0·121 (3)							
O(2)	0·069 (3)		0·118 (3)							0·039 (3)
O(3)	−0·068 (2)	−0·057 (3)		−0·093 (3)	0·002 (3)					
O(4)				0·095 (3)	0·003 (3)					
O(5)				−0·091 (3)		0·000 (3)				
O(6)		0·056 (3)	−0·113 (2)	0·102 (3)		0·000 (3)				
O(7)	−0·076 (3)	−0·050 (3)					0·128 (3)	0·001 (2)		
O(8)							−0·119 (3)	0·001 (3)		
O(9)							0·115 (3)		−0·004 (3)	
O(10)	0·076 (3)		0·116 (3)				−0·133 (3)		−0·004 (3)	
O(11)										0·128 (3)
C(1)				−0·007 (4)	−0·007 (4)	0·000 (4)				
C(2)				0·00 (4)	0·002 (4)	0·001 (4)				
C(3)							−0·002 (4)	−0·003 (3)	−0·003 (4)	
C(4)							0·012 (4)	0·001 (4)	0·010 (4)	
N(1)										−0·069 (3)
N(2)										−0·098 (4)

The V atom is elevated from the equatorial plane (plane I) by 0·302(1) Å and forms a short bond with the apical O(1). The shortness of this bond [1·594(3) Å] is evidence of multiple-bonding and it is typical of V=O lengths found in similar structures. The length [2·184(3) Å] of the other axial bond V–O(6) is a further example of the *trans* influence of the vanadyl ion.

Bond lengths in six-coordinate oxovanadate(IV) complexes are compared in Table 7.

(ii) Bond parameters in the oxalato groups

The average bond lengths and angles in some complex oxalates are compared in Table 8.

The bond parameters vary according to whether the O atom concerned is coordinated (O_c) or uncoordi-

Fig. 1. Perspective diagram of the complex anion.

nated (O_u). This has previously been observed by Scheidt *et al.* (1971) and Drew *et al.* (1974).

A consequence of the *trans* effect (also noted by these authors) is that the strongest of the C–O_c bonds occur where the O atom concerned is in the *trans* position to the vanadyl O atom. Table 9 presents data on complex oxalates wherein these bonds are distinguished. These parameters show that the *trans* effect is transmitted throughout the oxalato groups although the consequent difference in the lengths of the terminal C–O_u bonds is small.

The C–C lengths are in agreement with values generally found in complex oxalates and correspond to the simple type of bond to which Sutton (1965) has assigned a value of 1·537 ± 0·005Å.

(iii) Non-planarity of the oxalato groups

Both oxalato ligands are non-planar (planes IV and VII). However, all four C–CO_2^- residues are virtually planar. Planes V and VI give a dihedral angle of 10·0° about C(1)–C(2) and planes VIII and IX give 12·8° about C(3)–C(4).

Non-planar hydrated complexes have previously been reported by Drew *et al.* (1974) and Viswamitra (1962), and planar configurations in this type of compound have been found by Glen, Silverton & Hoard (1963) and Kojić-Prodić, Liminga & Šćavničar (1973). Stomberg (1970) found that in anhydrous $K_2[MoO(O_2)_2(C_2O_4)]$ the oxalato groups have a dihedral angle of 5·1°. This would suggest that crystal forces other than hydrogen-bonding may be capable of twisting the planes of the carboxyl groups.
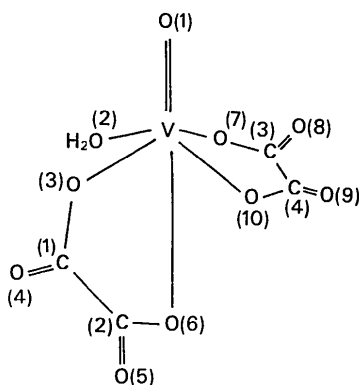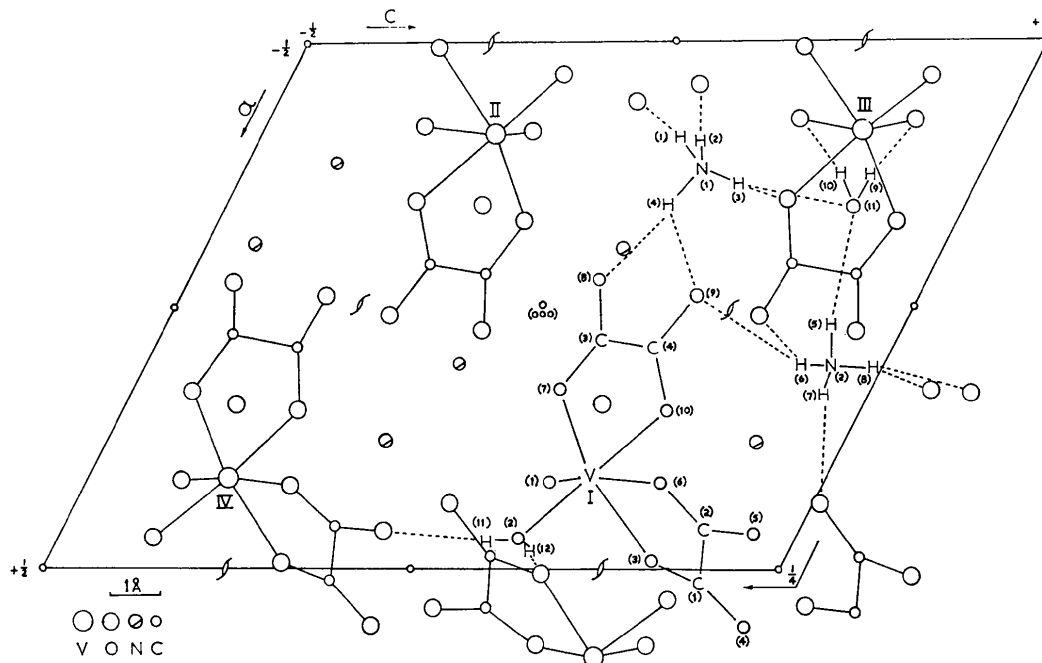

Fig. 2. Projection down **b** of diammonium bis(oxalato)monaquooxovanadate(IV) monohydrate showing hydrogen-bonding scheme.

(iv) Hydrogen-bonding

The water O and the N atoms lie approximately on a plane (X). The 12 H atoms form a complex network of intra- and intermolecular contacts. The uncoordinated water O(11) links the pair of ammonium ions within the same asymmetric unit, while the two H atoms of this water molecule form bonds with the apical O atoms in the coordination octahedron of a symmetry-related unit. The ammonium ions and the coordinated water molecule form hydrogen bonds involving all the remaining O atoms, coordinated and uncoordinated. N(1) is linked to the uncoordinated pair O(8) and O(9) by a bifurcated hydrogen bond.

The interatomic distances pertinent to hydrogen bonding in crystalline AVO are given in Table 10. The

Table 7. *Bond lengths (Å) in octahedral oxovanadate*(IV) *complexes*

Compound	Apical (vanadyl) V=O	Apical (*trans*) V–O	Equatorial V–O	Reference
AVO	1·594 (3)	2·184 (3)	2·033 (3) 2·022 (3) 1·985 (3) 2·006 (3) Av. = 2·011	This work
VOSO₄	1·59 (4)	2·28 (4)	2·00 (5) 2·01 (6) 2·05 (6) 2·05 (6) Av. = 2·03	Kierkegaard & Longo (1965)
(NH₄)₂VO(NCS)₄.5H₂O	1·62 (6)	2·22 (5)	V–N Av. = 2·04 (3)	Hazell (1963)
VOC₇H₃NO₄.H₂O	1·591 (11)	V–N 2·184 (2)	V–O 2·027 (12) 2·027 (12) 2·017 (10) 2·017 (10) Av. = 2·022	Bersted, Belford & Paul (1968)

Table 8. *Average bond lengths and angles in some complex oxalates*

Complex oxalate	Bond length (Å)			Angle (°)			Reference
	C–C	C–O_c	C–O_u	O_c–C–O_u	C–C–O_c	C–C–O_u	
(NH₄)₂[VO(C₂O₄)₂H₂O].H₂O	1·545	1·275	1·224	125·4	113·7	120·8	This work
(NH₄)₃[VO₂(C₂O₄)₂].2H₂O	1·538	1·274	1·234	127·1	113·9	118·6	Scheidt et al. (1971)
K₃[VO₂(C₂O₄)₂].3H₂O	1·550	1·274	1·226	125·9	114·1	120·0	Drew et al. (1974)
Na₄[Zr(C₂O₄)₄].3H₂O	1·545	1·275	1·222	126·3	113·4	120·2	Glen et al. (1963)
K₂[MoO(O₂)₂(C₂O₄)]	1·561	1·277	1·210	124·4	115·4	120·0	Stomberg (1970)
Na(NH₄)[MoO₃(C₂O₄)].2H₂O	1·525	1·360	1·260	125	115	121	Beagley & Small (1964)

Table 9. C–O *lengths* (Å)

Compound	C–O_c (*trans*)	C–O_c (*non-trans*)	C–O_u	Reference
(NH₄)₂[VO(C₂O₄)₂H₂O].H₂O	1·259 (4)		1·237 (5)	This work
		1·283 (4)	1·226 (4)	
		1·284 (6)	1·217 (6)	
		1·275 (5)	1·217 (6)	
(NH₄)₃[VO₂(C₂O₄)₂].2H₂O	1·263 (3)		1·239 (3)	Scheidt et al. (1971)
	1·262 (4)		1·241 (4)	
		1·291 (3)	1·221 (3)	
		1·280 (4)	1·234 (4)	
K₃[VO₂(C₂O₄)₂].3H₂O	1·262 (3)		1·224 (4)	Drew et al. (1974)
	1·257 (3)		1·240 (3)	
		1·291 (4)	1·221 (3)	
		1·287 (3)	1·220 (3)	
K₂[MoO(O₂)₂(C₂O₄)]	1·266 (20)		1·213 (23)	Stomberg (1966)
		1·287 (23)	1·207 (25)	

lengths of O–H···O bonds range from 2·621(4) to 3·056(4)Å and N–H···O from 2·828(4) to 3·379(5)Å.

Table 10. *Hydrogen-bonding parameters*

Symmetry code

(I)	x,	y,	z;
(II)	$-x$,	$-y$,	$-z$;
(III)	$-x$,	$\frac{1}{2}+y$,	$\frac{1}{2}-z$;
(IV)	x,	$\frac{1}{2}-y$,	$\frac{1}{2}+z$;
(V)	$x-1$,	$\frac{1}{2}-y$,	$-\frac{1}{2}+z$;
(VI)	$-x$,	$-\frac{1}{2}+y$,	$\frac{1}{2}-z$;
(VII)	$-x+1$,	$-y$,	$-z+1$;
(VIII)	$-x$,	$-y$,	$-z+1$;
(IX)	x,	$\frac{1}{2}-y$,	$z-\frac{1}{2}$;
(X)	$-x+1$,	$\frac{1}{2}+y$,	$\frac{1}{2}-z$;

	Distance between bonded atoms (Å)
$N(1^I)$—$H(1^I)$—$O(4^V)$	2·828 (4)
$N(1^I)$—$H(2^I)$—$O(5^{VI})$	2·886 (4)
$N(1^I)$—$H(4^I)$—$O(8^I)$	2·979 (5)
$N(1^I)$—$H(4^I)$—$O(9^I)$	2·887 (5)
$N(1^I)$—$H(3^I)$—$O(10^{III})$	3·087 (4)
$N(1^I)$—$H(3^I)$—$O(11^I)$	3·097 (4)
$N(2^I)$—$H(6^I)$—$O(9^I)$	3·096 (5)
$N(2^I)$—$H(6^I)$—$O(9^{III})$	3·379 (5)
$N(2^I)$—$H(5^I)$—$O(11^I)$	3·016 (5)
$N(2^I)$—$H(7^I)$—$O(4^{VII})$	3·003 (6)
$N(2^I)$—$H(8^I)$—$O(7^{IV})$	2·893 (5)
$N(2^I)$—$H(8^I)$—$O(11^{VIII})$	3·104 (5)
$O(2^I)$—$H(11^I)$—$O(5^{IX})$	2·685 (4)
$O(2^I)$—$H(12^I)$—$O(3^X)$	2·621 (4)
$O(11^I)$-$H(9^I)$—$O(1^{III})$	3·056 (4)
$O(11^I)$-$H(10^I)$—$O(6^{VI})$	2·955 (4)

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The Crystal Structure of Sodium Acetate Trihydrate

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The molecular structure of the title compound has been determined by direct methods from photographic data. $Na^+(CH_3COO)^-.3H_2O$, monoclinic, $a=12\cdot475$, $b=10\cdot407$, $c=10\cdot449$ Å, $\beta=112\cdot65°$, space group $C2/c$, 475 reflexions, $R=0\cdot11$. The Na^+ ion has distorted octahedral coordination with six oxygen atoms. Adjacent octahedra share an edge and form a continuous chain along the z axis. Along the chain the separations between the Na^+ ions are $3\cdot39$ and $3\cdot56$ Å and the Na^+–O distances are in the range $2\cdot35$–$2\cdot56$ Å. The C–O bond lengths are $1\cdot23$ and $1\cdot31$ Å and both oxygen atoms are hydrogen bonded to two water molecules.

Sodium acetate trihydrate is one of the better known salts of one of the simple organic acids; as such its crystal structure has been determined.

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Experimental

Crystals of sodium acetate trihydrate, grown by slow evaporation from aqueous solution, are small hexagons. Weissenberg and oscillation photographs established that the crystals were monoclinic in space