

The Structure of Potassium Tetroxalate:* a Redetermination and a Cautionary Tale

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Abstract

The structure of potassium tetroxalate was carefully determined by X-ray work [Haas (1964). *Acta Cryst.* **17**, 1511–1516], the analysis being in $P\bar{1}$. This choice of space group has been questioned: implicitly by Groth [*Chemische Kristallographie* (1910), Vol. III, p. 140. Leipzig: Engelmann], who gave the crystal class as ‘asymmetrisch?’, and explicitly by Emsley, Jones & Kuroda [*J. Inorg. Nucl. Chem.* (1981), **43**, 2243–2246] who obtained a lower R value when refinement was in the noncentrosymmetric $P1$, and who claimed to have established a hydrogen-bonding scheme different from that of Haas. For these reasons, work based on an extensive set of X-ray data is reported here. With 2422 independent reflexions, the structure was refined in $P\bar{1}$ to $R = 3.0\%$. The present results confirm and amplify Haas’s work. However, the present X-ray measurements were severely affected by extinction errors; and the H atoms were located satisfactorily only after allowance had been made for these errors. An attempt to refine a desymmetrized structure in $P1$ failed. [Crystal data: $\text{KH}_3(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$, $M_r = 254.1$, triclinic, $P\bar{1}$, $a = 7.027(1)$, $b = 10.595(1)$, $c = 6.356(1) \text{ \AA}$, $\alpha = 101.40(5)$, $\beta = 100.12(5)$, $\gamma = 93.80(5)^\circ$, $V = 454.2 \text{ \AA}^3$, $Z = 2$, D_o (Haas) = 1.86 , $D_c = 1.859 \text{ Mg m}^{-3}$, $\mu(\text{Mo K}\alpha) = 0.618 \text{ mm}^{-1}$.]

Introduction

The tetroxalates – formerly quadroxalates – are superacid salts of formula $\text{MH}_3(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$, M being NH_4 , K, Rb, Cs or Tl¹. Stable and crystallizing well, one of them, the potassium salt (KTO), has been known for over 200 years under the names ‘salts of sorrel’, or ‘salts of lemon’, though these names were also formerly applied to the ordinary acid salt, KHC_2O_4 .

The classical crystallography of four of them is summarized by Groth (1910) (see also Porter, 1928).

They are isomorphous in the triclinic system. For NH_4 , Rb and Tl salts the crystal class is given as ‘pinakoidal’, which implies the space group $P\bar{1}$, but for KTO we find ‘asymmetrisch (?)’, implying the noncentrosymmetric $P1$. The doubt is not explained, however, and the crystal drawing shows holohedry.

The crystal structure of KTO was determined by Haas (1964). His X-ray work was of remarkable accuracy for that date: with 1289 reflexions measured by diffractometer, block-diagonal least-squares refinement, in $P\bar{1}$, converged at $R = 5\%$, and positions were found even for the H atoms. Shortly afterwards neutron diffraction work was performed on the ammonium salt (ATO) by Currie, Speakman & Curry (1967). Though of lower general accuracy, the analysis confirmed Haas’s heavy-atom structure, whilst the positions found for the seven relevant protons, now determined with better precision, agreed with those of Haas for the H atoms. The structures of various tetroxalates have been compared by Bulc, Golić & Šiftar (1979).

A recent X-ray redetermination of KTO has been briefly reported by Emsley, Jones & Kuroda (1981, EJK hereafter). They claim a much lower R value when the refinement was in the noncentrosymmetric $P1$ and propose a drastically amended hydrogen-bonding scheme. This claim is hard to assess because the report lacks not only important details – such as coordinates and standard deviations – but also any mention of the difficulties of refining, in $P1$, a structure whose atoms are in, or close to, the symmetrical positions of $P\bar{1}$ (see Schomaker & Marsh, 1979).

Analysis, results and discussion

These doubts led us to collect an extensive set of X-ray data for KTO. (Details will be given later.) We worked with a merged set of 2422 data, with $I > 3\sigma(I)$. Internal consistency was represented by $R_{\text{int}} = 1.4\%$, though this criterion would not reveal systematic errors that affected both members of a Friedel pair equally. Refinement was in $P\bar{1}$ (started from Haas’s coordinates for K, C and O, the H atoms being omitted) and, after introduction of anisotropic vibrational parameters, converged at $R = 5.4\%$. The geometry of the

* Potassium trihydrogen dioxalate dihydrate.

heavier-atom structure already showed high formal precision. With the H atoms, however, we had some initial difficulty: the 'difference-density' map carried a profusion of peaks. Amongst them could be found peaks for all seven H atoms, but they by no means dominated the map. After placing (isotropically vibrating) H atoms in suitable positions, refinement went only to 4.6%, and there were still embarrassing peaks in the difference density.

Examination of the structure factors revealed a number of strong, low-order reflexions with $|F_o| \ll |F_c|$. The two worst were $01\bar{2}$ (54.5, 104.7) and $2\bar{1}1$ (69.0, 113.5), and there were 35 others with highly significant negative discrepancies, whilst none were notably positive. [Haas's structure factor table shows similar, though rather smaller, discrepancies amongst the same reflexions: e.g. $01\bar{2}$ (72.3, 103.6) and $21\bar{1}$ (81.3, 111.2).] With a low R value, such blemishes cannot be erased by shifts of the H atoms. The only way in which we could account for them was by severe extinction errors.

In routine structure analyses, extinction is regarded as a minor source of trouble, usually to be evaded by merely omitting a few terms. In more ambitious work, proper correction is more important, and more difficult (see Coppens & Hamilton, 1970). The only routine we had to hand was the empirical formula which reduces $|F_c|$ for strong, low-order reflexions by refining a single 'isotropic' parameter, p . When this formula proved inadequate for dealing with the severe extinction in our KTO data, we adopted a two-stage tactic: omitting the 10 worst-affected terms, and dealing with the others by including p in our subsequent refinement. Though a few terms were overcorrected, R fell to 3.0%; the resulting structure was chemically acceptable and of high precision; and the difference map was clean, apart from two peaks at points of inversion ($00\frac{1}{2}$, $\frac{1}{2}0\frac{1}{2}$) at the centres of two oxalic acid units shown in Fig. 1.

Our problem with the H atoms may now be interpreted. The considerable number of terms suffering from extinction would put 'false' terms into the difference synthesis, and so produce bogus peaks comparable to the genuine ones due to H atoms. (EJK do not mention extinction. Should their intensity data have been similarly affected by uncorrected extinction, this would account for their initial difficulties.) There remains a gap between our final R (3.0%) and R_{int} (1.4%). We attribute it mainly to our approximate method of correcting for extinction.

However, we attempted refinement in $P1$. Our $P\bar{1}$ structure gave us no clue as to how the symmetry might be broken. We might, indeed, have started from the EJK coordinates; but these were not readily available. We therefore set out from our own coordinates for the 15 K, C and O atoms, and added 15 nearly centrosymmetrically related 'counter-atoms' with random positional shifts up to 0.05 Å. The first K

atom was fixed to secure our origin. Isotropic refinement, followed by anisotropic, led to $R = 5.1\%$. [The first K atom being fixed, there was no obstacle preventing the other K from moving into counter-coincidence. It did so. Any such propensity on the part of the other counter-atoms, however, is forbidden by the mathematics of least-squares refinement. See the discussion by Schomaker & Marsh (1979), where earlier references are given.] We then inserted 14 H atoms. (This necessitated fixing both K atoms to avoid putting the matrix out of bounds.) R fell to 4.1%, which became 3.1% when the terms badly affected by extinction were omitted from the calculation of R . At no stage was the $P1$ refinement satisfactory. Correlation coefficients between atom and counter-atom were high (132 ranging from $|0.71|$ to $|0.92|$), and convergence was slow and unstable; standard deviations were about five times higher than in $P\bar{1}$; and – which should be regarded as important – the banishment of symmetry resulted in distortions that were sometimes chemically implausible. Refinement by the block-diagonal approximation led to the same structure, with standard deviations more agreeable by a factor of about three. We feel it to be unlikely that useful information can be extracted from our data by further recourse to $P1$ (Marsh & Schomaker, 1979). In a structure of this sort, a rigorous disproof of $P1$ is impossible.

Haas's $P\bar{1}$ structure is essentially correct. Technical developments have enabled us to support it with results of higher precision. Fig. 1 shows the asymmetric unit, with our atom-numbering system, which differs a little from that used by Haas, but corresponds to that used

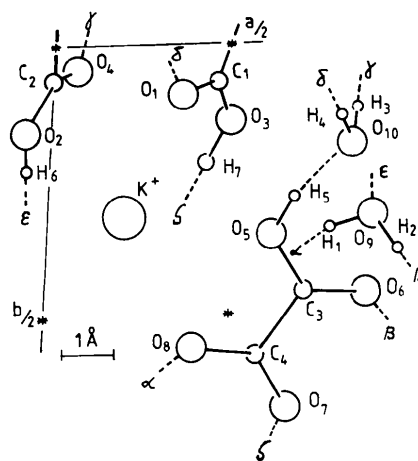


Fig. 1. The asymmetric unit of potassium tetroxalate, projected normal to (001). Asterisks mark centres of symmetry at $c/2$. The seven hydrogen bonds are indicated by broken lines. Where necessary, the connectivities in the hydrogen bonds are signified by pairs of Greek letters.

Table 1. *Potassium tetroxalate: fractional coordinates ($\times 10^5$, but $\times 10^4$ for H), and isotropic vibrational parameters (\AA^2 , $\times 10^4$), with standard deviations in parentheses*

	x	y	z	$U_{eq} \dagger$
K ⁺	14807 (3)	27271 (2)	16639 (4)	297 (2)
O(1)	33290 (11)	6742 (7)	32837 (13)	290 (4)
O(2)	-7193 (12)	15416 (8)	50492 (13)	320 (4)
O(3)	51922 (11)	15769 (7)	65332 (13)	288 (4)
O(4)	11070 (12)	7679 (7)	76632 (13)	299 (3)
O(5)	55896 (11)	30859 (7)	18309 (16)	353 (3)
O(6)	84492 (11)	43009 (7)	27718 (15)	310 (3)
O(7)	65340 (11)	64412 (7)	35584 (15)	316 (4)
O(8)	37308 (11)	51717 (8)	22384 (16)	356 (4)
O(9)	94938 (13)	35616 (7)	78609 (12)	291 (4)
O(10)	76099 (13)	13048 (8)	5638 (13)	318 (4)
C(1)	45457 (12)	6359 (9)	48842 (15)	219 (4)
C(2)	1519 (13)	6493 (9)	58407 (15)	234 (4)
C(3)	66872 (13)	41847 (9)	24658 (14)	227 (4)
C(4)	55135 (13)	53753 (8)	27856 (15)	230 (4)
H(1)	8469 (29)	3761 (20)	7801 (30)	439 (45)
H(2)	10077 (29)	4122 (20)	7471 (31)	520 (51)
H(3)	7800 (29)	762 (21)	1233 (34)	547 (51)
H(4)	7128 (34)	863 (24)	-796 (43)	725 (63)
H(5)	6262 (28)	2429 (21)	1417 (32)	549 (52)
H(6)	-577 (41)	2328 (31)	6154 (54)	1020 (93)
H(7)	4587 (29)	2311 (23)	6407 (37)	620 (56)

$$\dagger U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

Table 2. *Interatomic distances (\AA) and angles ($^\circ$) within the oxalate units, with standard deviations in parentheses*

C(1)–C(1 ⁱⁱ)	1.551 (2)	O(1)–C(1)–O(3)	126.3 (1)
C(1)–O(1)	1.218 (1)	C(1 ⁱⁱ)–C(1)–O(1)	120.7 (1)
C(1)–O(2)	1.288 (1)	C(1 ⁱⁱ)–C(1)–O(3)	113.0 (1)
C(2)–C(2 ⁱ)	1.543 (2)	O(2)–C(2)–O(4)	126.5 (1)
C(2)–O(2)	1.295 (1)	C(2 ⁱ)–C(2)–O(2)	111.9 (1)
C(2)–O(4)	1.211 (1)	C(2 ⁱ)–C(2)–O(4)	121.6 (1)
C(3)–C(4)	1.552 (1)	O(5)–C(3)–O(6)	125.0 (1)
C(3)–O(5)	1.303 (1)	C(4)–C(3)–O(5)	113.2 (1)
C(3)–O(6)	1.213 (1)	C(4)–C(3)–O(6)	121.8 (1)
C(4)–O(7)	1.257 (1)	C(7)–C(4)–O(8)	128.2 (1)
C(4)–O(8)	1.232 (1)	C(3)–C(4)–O(7)	114.6 (1)
		C(3)–C(4)–O(8)	117.2 (1)
K...O		C(1)–O(3)–H(7)	112 (2)
2.867 (1)		C(2)–O(4)–H(6)	111 (2)
2.868, 2.875		C(3)–C(5)–H(5)	112 (1)
2.889, 2.898			
2.907, 2.921			

The water molecules	
H(1)–O(9)–H(2)	104 (2)
H(3)–O(10)–H(4)	104 (2)

Symmetry code

- | | |
|-----------------------|----------------------|
| (i) $-x, -y, 1-z$ | (iv) $2-x, 1-y, 1-z$ |
| (ii) $1-x, -y, 1-z$ | (v) $1-x, -y, -z$ |
| (iii) $1-x, 1-y, 1-z$ | (vi) $-1+x, y, z$ |

for ATO. Coordinates are in Table 1.* The code needed for symmetry-related units and the principal molecular dimensions are in Table 2.

The CCO₂ atoms of each of the four carboxyl groups are coplanar. The dihedral angle, about C(3)–C(4) at the middle of the HC₂O₄ anion, is about 6°. Einspahr, Marsh & Donohue (1972) report a precise study of potassium binoxalate [potassium hydrogen oxalate, KHC₂O₄ (KHOx)]. Our results for the geometry of the half-anion agree closely with theirs. This unit in KTO has appreciable librational motion about an axis parallel to C(3)–C(4).

All the H and O atoms are involved in hydrogen bonding, details of which are in Table 3. The four bonds from the two water molecules are relatively weak. The three from oxalate units are all strong, though in no case do they qualify as symmetrical, or nearly symmetrical (Speakman, 1972) [for later work see Bacon, Walker & Speakman (1977) and McGregor, Speakman & Lehmann (1977)]. The bond O(2)–H(6)···O(9) is remarkably short for one to a water molecule. The P $\bar{1}$ hydrogen-bonding system of the tetroxalates is complicated. A stereodiagram was published by Currie, Speakman & Curry (1967); and the connectivities are indicated in Fig. 1.

We are indebted to a referee for drawing attention to a discrepancy at the foot of Haas's Table 5. A contact of 3.10 \AA is given between the water molecule, O(9), and O(8), though which symmetry-related O(8) is not specified. There must be something wrong here. Recalculation of inter-oxygen distances up to 4 \AA , using Haas's coordinates and cell dimensions, gave us, for O(9)···O(6), 2.73 \AA , which completes the agreement between Haas's hydrogen bonds and ours, and 3.29 and 3.44 \AA , which cannot represent hydrogen bonds. Haas's 3.10 \AA is evidently the non-bonded contact O(9)(2 – x, 1 – y, 1 – z)···O(7), which amounts to 3.081 (1) \AA from our parameters.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36924 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Geometrical details of the hydrogen bonds, with standard deviations in parentheses*

The symmetry code is given in Table 2.

	O...O (\AA)	O–H (\AA)	H...O (\AA)	O–H...O ($^\circ$)
O(9)–H(1)···O(8 ⁱⁱⁱ)	2.707 (1)	0.76 (2)	1.97 (2)	162 (2)
O(9)–H(2)···O(6 ^{iv})	2.741 (1)	0.80 (2)	1.95 (2)	167 (2)
O(10)–H(3)···O(4 ⁱⁱ)	2.791 (1)	0.79 (2)	2.03 (2)	162 (2)
O(10)–H(4)···O(1 ^v)	2.835 (1)	0.89 (2)	2.00 (2)	156 (2)
O(5)–H(5)···O(10)	2.526 (1)	0.89 (2)	1.64 (2)	175 (2)
O(2)–H(6)···O(9 ^{vi})	2.480 (1)	0.97 (3)	1.52 (3)	176 (2)
O(3)–H(7)···O(7 ⁱⁱⁱ)	2.499 (11)	0.92 (2)	1.58 (2)	173 (2)

Experimental

The crystal data are given in the *Abstract*, where the cell is that chosen by Haas (1964). It differs from the cell used by Groth and in the neutron work on ATO, the transformation, both ways, being by the matrix 001/010/100.

Intensity measurements

The crystal dimensions (mm) were $0.73 \times 0.25 \times 0.17$, the first being parallel to *c*. Measurements were made by a CAD-4 diffractometer, using Mo radiation with the $\omega/2\theta$ scan. They covered the complete sphere to $\theta = 30^\circ$ in two stages: first to 26° , and then the shell to 30° , some 300 reflexions being in duplicate in a total of 5677. The whole set was merged to yield 2629 independent terms, of which 207 had $|F_o| < 2.0\sigma(F_o)$ and were not used in the analysis. Standard deviations were calculated by $\sigma^2(F) = \sigma^2$ (counting statistics) + $0.04I$. Absorption corrections were applied though the transmission factors ranged only from 0.907 to 0.845. Intensity statistics, applied to all data, favoured the space group *P1*. The test is unreliable in structures with heavy atoms.

Refinement

The progress and outcome have been described above. All calculations were performed with the *SHELX* programs (Sheldrick, 1976). Scattering factors for C, H, O and K^+ were from *International Tables for X-ray Crystallography* (1974).

Two strong low-order reflexions had been used as standards. They held constant until two-thirds of the data had been collected; they then began to rise progressively, at different rates; at the end, their intensities were up by 13 and 31% respectively. Nevertheless, we felt justified in ignoring this calamity; for the particular reflexions we had chosen happen to be the two (01 $\bar{2}$ and $2\bar{1}1$) worst affected by extinction. Fortunately, the low-order terms liable to suffer from extinction were all measured before this vagary developed. All the indirect evidence, which was extensive in our data, showed the tube output to have been steady throughout.

Extinction corrections were made by reducing $|F_c|$ by the factor $[1 - 10^{-4}p(|F_c|^2)/\sin \theta]$. In our final cycles, after the 10 worst affected terms had been omitted, *p* refined to 0.11; such a value, had it been applied to the two worst, would have reduced $|F_c|$ to negative values. We have already drawn attention to the approximate nature of our method of correction. At final convergence, for 2412 terms, *R* was 3.02%, and *R_w* 3.58%. A weighting analysis was satisfactory, except for a slight over-weighting of weak, low-order terms.

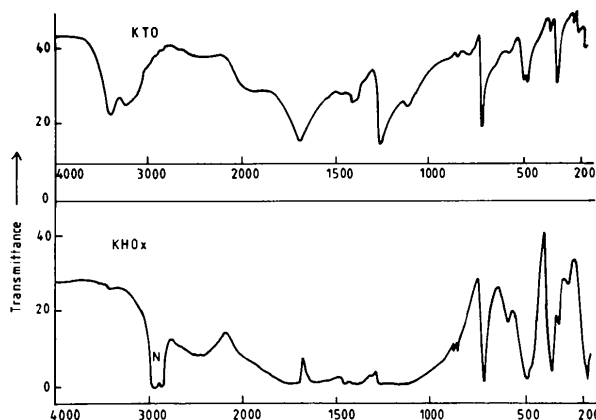


Fig. 2. Infrared spectra: top: potassium tetroxalate (KTO) in KBr disc; bottom: potassium binoxalate (KHOx) in Nujol mull (frequencies in cm^{-1}).

IR spectra

EJK draw support for their proposed hydrogen-bonding scheme from an IR spectrum, especially from the absence of strong absorption, associated with 'water of crystallization', near 3400 cm^{-1} ; they write: 'Clearly the strongly hydrogen bonded water molecules in the crystal have been shifted to lower wave numbers and compose part of the continuum'. Their spectrum is virtually identical with that in the lower part of our Fig. 2, apart from our Nujol peak. However, we obtained our spectrum from the binoxalate, KHC_2O_4 . [A clearer spectrum of KHOx has been published by de Villepin & Novak (1971).] The true spectrum of KTO is reproduced in the upper part of Fig. 2; there seems to be no previous published record of it.

Helpful comments have come from Drs D. Hadži, R. E. Marsh and J. H. Robertson, and from Sir Gordon Cox. The IR spectroscopy was performed by Mrs Lawrie and Mrs Malloch.

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The Structure of Tris(tetraphenyl imidodiphosphato)ytterbium(III)

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Abstract

The crystal structure of $C_{72}H_{60}N_3O_{18}P_6Yb$ has been determined by single-crystal X-ray diffraction techniques. The crystals are monoclinic, space group $P2_1/c$, with cell constants $a = 18.627$ (6), $b = 15.761$ (8), $c = 26.553$ (10) Å, $\beta = 113.76$ (2)°, $V = 7135$ (5) Å³, $Z = 4$, $D_c = 1.50$ Mg m⁻³. The structure was solved by Fourier and direct methods and refined by full-matrix least squares to an R factor of 0.051 for 7443 reflections including those below background. The structure consists of discrete $Yb\{N[PO(OC_6H_5)_2]_2\}_3$ molecules. The Yb atom is six coordinated. The low coordination number is due to the bulky ligands. The six O atoms of the three bidentate chelate rings form the coordination environment of the Yb^{3+} ion. The coordination polyhedron has virtual D_3 symmetry, more in the direction of a distorted octahedron than in the direction of a trigonal prism. The intrachelate O—O separation is 2.883 Å and the interchelate separation is 3.07–3.39 Å. The Yb atoms lie almost on a superposition lattice which has a quarter of the volume of the unit cell.

Introduction

Numerous structures of the complexes of rare-earth ions have been determined by X-ray diffraction in recent years, and their stereochemistry has been studied in detail (Moseley, 1975; Thompson, 1979). So far, a

large number of the investigations of such complexes in solution have been directed towards solvent-extraction separation. Neutral chelate complexes of tetraphenyl imidodiphosphate (idtph) with rare-earth ions of the type $Ln\{N[PO(OC_6H_5)_2]_2\}_3$ have been synthesized (Herrmann, Hoàng bá Nặng & Dreyer, 1979). We have carried out an X-ray structure analysis of the Yb complex, $Yb(idtph)_3$, in order to determine the structure, especially the exact coordination environment of the lanthanoid ions of such complexes in the solid state. We have started with the heavy rare-earth element Yb which has a small ionic radius. The relation of the stereochemistry of the structure to other information, e.g. extraction, IR, ³¹P NMR, ¹³C NMR and ¹H NMR measurements, will be discussed elsewhere. Moseley (1975) expected an octahedral environment with distortions towards a trigonal prism for the six-coordinate tris-bidentate rare-earth complexes. This was due to the decreasing 'bite' of the bidentate ligand. A six-coordinate Er complex, $Er(dpm)_3$ (dpm = 2,2,6,6-tetramethyl-3,5-heptanedionato), with trigonal-prismatic coordination for Er has been reported by de Villers & Boeyens (1971).

Experimental

The compound (m.p. 503–508 K) was prepared according to the procedure of Herrmann, Hoàng bá Nặng & Dreyer (1979). Suitable crystals were obtained by recrystallization from alcohol. Preliminary X-ray