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The Crystal Structure of Potassium Tetraoxalate, $K(HC_2O_4)(H_2C_2O_4).2H_2O$

By David J. Haas

Department of Biophysics, State University of New York at Buffalo, Roswell Park Graduate Division
Buffalo, N.Y., U. S. A.

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Potassium tetraoxalate $K(HC_2O_4)(H_2C_2O_4).2H_2O$ crystallizes in the triclinic system with the space group $P\bar{1}$ and lattice constants:

$$a = 7{\cdot}04_7\ \text{Å} \qquad \alpha = 101{\cdot}4_1^\circ$$
$$b = 10{\cdot}59_5 \qquad \beta = 100{\cdot}2_3$$
$$c = 6{\cdot}35_5 \qquad \gamma = 94{\cdot}0_8$$

The positions of the potassium ions were determined from a three-dimensional Patterson synthesis; the crystal structure was then solved by the heavy atom method. The crystal structure was fully refined with anisotropic temperature factors to an R value of 0·05.

The carbon–carbon distance for the oxalic acid molecules and the acid oxalate group is 1·55 Å. The interatomic distances and bond angles of the oxalic acid molecules and the corresponding part of the potassium acid oxalate group are consistent. Of the six crystallographically different hydrogen bonds, three are unusually short, being of the order of 2·5–2·6 Å.

Crystals deposited by slow evaporation out of an aqueous solution prepared from $K(HC_2O_4)$ and $Al(OH)_3$, according to a standard method (*Inorganic Syntheses*, 1939) for the preparation of potassium trioxalatoaluminum, were examined by X-ray diffraction. Initially a number of crystals were selected and examined, and all of these proved to be identical. We were, however, unable to identify them as being potassium trioxalatoaluminum (Herpin, 1958) and, therefore, by this preliminary X-ray investigation assumed that they were a different crystalline modification of the compound. The molecular weight of the unit-cell contents of these triclinic crystals was only slightly greater than that of the supposed formula: $K_3Al(C_2O_4)_3.3H_2O$, but when the positions of all the non-hydrogen atoms were established by X-ray diffraction methods, the unit cell was found to contain two formula weights of $K(HC_2O_4)(H_2C_2O_4).2H_2O$, as will appear below.

A portion of the crystalline sample from the original chemical preparation was later resolved into two components of different density by flotation. The lighter component (density 1·85 g.cm^{-3}) was found to be the compound described above, while the other (density $2{\cdot}02_1$ g.cm^{-3}) has cell dimensions very similar to, and systematic absences identical with, crystals of potassium trioxalatoaluminum trihydrate (Herpin, 1958).

This paper presents the results of the investigation of the atomic arrangement in the potassium tetraoxalate crystal whose composition was originally unknown. A review of the various potassium oxalate crystals has been given by Purdie (1925) and recent X-ray crystallographic studies have been performed on various oxalate (Beagley & Small, 1963) and oxalic acid crystals (Ahmed & Cruickshank, 1953).

Experimental

Preliminary X-ray investigation produced the following information:

Space group: $P1$ or $P\bar{1}$.
Lattice constants:

$$a=7{\cdot}04_7\ \text{Å},\ b=10{\cdot}59_5\ \text{Å},\ c=6{\cdot}35_5\ \text{Å};$$
$$\alpha=101{\cdot}4_1^\circ,\ \beta=100{\cdot}2_3^\circ,\ \gamma=94{\cdot}0_8^\circ.$$

Density:

$$D_m=1{\cdot}85\ \text{g.cm}^{-3},\ D_x=1{\cdot}86_2\ \text{g.cm}^{-3};\ Z=2\ .$$

The crystal was assigned to the space group $P1$ without testing for a center of symmetry (on the assumption that one could not be present inasmuch as the single trioxalate complex ion thought to be in the unit cell does not have a center of inversion).

Three-dimensional X-ray intensities were collected on the GE XRD 5 equipped with Single Crystal Orienter. A total of 1862 intensities were measured. Five hundred seventy three of these were weak (less than twice the background) and were eliminated from the calculations. The intensities were converted to $|F|^2$ in the usual way.

The crystal from which the intensities were collected has the dimensions: $0{\cdot}1 \times 0{\cdot}1 \times 0{\cdot}25$ mm. No serious X-ray absorption effects appeared to be present according to the criterion of Furnas (1957), and no absorption corrections were applied to the intensities.

Determination of the structure

In the asymmetric unit of the three-dimensional Patterson function, only one peak had a value that remained well above all others after weighting functions of various powers of sin θ were applied to the structure factors. (This tends to amplify the potassium–potassium vectors as opposed to the other vectors.) Since the origin of the unit cell is arbitrary for $P1$, one potassium atom was placed at the origin and another at the apparent potassium position derived from the Patterson function. At this time we were unable to explain why only this one Patterson peak remained large after applying the weighting function, inasmuch as the proposed cell contents included three potassium ions.

The phases of these two potassium ions were then used for the first electron density map. This calculation produced numerous peaks, among which was an arrangement that resembled an oxalate group. In addition, most of these apparent atomic positions correspond to Patterson peaks for vectors to light atoms from potassium ions. For purposes of computation, these small peaks were assumed to be atoms with scattering powers equal to that of nitrogen; by including these presumed atomic positions in a structure factor calculation, an R value of 0·25 was obtained. (The two potassium atoms alone gave an R value of 0·48.) Successive electron density calculations permitted us to locate 30 such atoms, and the R value decreased to 0·18. An $(F_o - F_c)$ electron density calculation of the complete cell established that all non-hydrogen atoms had been located. At this time, a model was built which made it obvious that the unit cell contained two potassium ions, four oxalate groups, and four water molecules. Accordingly, the formula of one unit cell could only be

$$2[K(HC_2O_4)(H_2C_2O_4).2H_2O] .$$

A study of the positions of the atoms in the unit cell indicated that centers of inversion might be present. An obvious place for one of these was midway between the two potassium atoms. By using this position as a center of inversion, it was found that all atoms in the structure had inversion images. It was concluded, therefore, that this position, plus those at $\frac{1}{2}00$, $0\frac{1}{2}0$, $00\frac{1}{2}$, $\frac{1}{2}\frac{1}{2}0$, $\frac{1}{2}0\frac{1}{2}$, $0\frac{1}{2}\frac{1}{2}$, and $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ from this point, are centers of inversion, and the correct space group is $P\bar{1}$.

The further refinement of the structure was carried out in the space group $P\bar{1}$. The positions of the 15 independent non-hydrogen atoms were refined on the IBM 1620 computer by the method of differential synthesis to an R value of 0·14. Final refinement by Block Diagonal Least Squares on the IBM 7090 computer gave the R values listed below (the unobserved reflections were not included in the calculations of these R values):

	R
Isotropic temperature factors; 2 cycles	0·11
Anisotropic temperature factors; 2 cycles	0·06
Anisotropic temperature factors plus hydrogen atoms; 1 cycle	0·05

Standard deviations of the atomic coordinates (except hydrogens), interatomic distances, and bond angles were calculated from the least-squares results (*International Tables for X-ray Crystallography*, 1959).

Table 1. *Coordinates of the atoms* (Å)

	x σ	y σ	z σ
K	0·1484 ± 0·001	0·2729 ± 0·001	0·1660 ± 0·001
O(1)	0·3337 ± 0·004	0·0679 ± 0·003	0·3283 ± 0·003
O(2)	0·9278 ± 0·004	0·1537 ± 0·003	0·5050 ± 0·003
O(3)	0·5187 ± 0·004	0·1570 ± 0·003	0·6538 ± 0·003
O(4)	0·1101 ± 0·004	0·0765 ± 0·003	0·7660 ± 0·003
O(5)	0·5600 ± 0·004	0·3085 ± 0·003	0·1821 ± 0·004
O(6)	0·8442 ± 0·003	0·4306 ± 0·003	0·2769 ± 0·004
O(7)	0·3470 ± 0·004	0·3555 ± 0·003	0·6432 ± 0·004
O(8)	0·6260 ± 0·003	0·4824 ± 0·003	0·7756 ± 0·004
$H_2O(1)$	0·9486 ± 0·003	0·3565 ± 0·003	0·7855 ± 0·003
$H_2O(2)$	0·7614 ± 0·004	0·1305 ± 0·003	0·0567 ± 0·004
C(1)	0·4533 ± 0·004	0·0632 ± 0·004	0·4884 ± 0·005
C(2)	0·0150 ± 0·005	0·0649 ± 0·004	0·5840 ± 0·005
C(3)	0·6688 ± 0·005	0·4189 ± 0·004	0·2465 ± 0·005
C(4)	0·4493 ± 0·005	0·4632 ± 0·004	0·7217 ± 0·005
H(1)	0·627 ± 0·2	0·242 ± 0·2	0·150 ± 0·2
H(2)	0·837 ± 0·2	0·388 ± 0·2	0·757 ± 0·2
H(3)	0·800 ± 0·2	0·075 ± 0·2	0·133 ± 0·2
H(4)	0·933 ± 0·2	0·241 ± 0·2	0·617 ± 0·2
H(5)	0·473 ± 0·2	0·225 ± 0·2	0·617 ± 0·2
H(6)	0·700 ± 0·2	0·092 ± 0·2	0·923 ± 0·2
H(7)	0·000 ± 0·2	0·417 ± 0·2	0·773 ± 0·2

Table 2. *Anisotropic thermal parameters*

($\times 10^4$)

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
K	103	38	163	21	−31	24
σ	2	1	3	2	4	3
O(1)	135	36	145	40	−71	24
σ	7	3	8	7	13	8
O(2)	188	31	136	59	−36	7
σ	8	3	8	7	14	8
O(3)	143	29	150	51	−63	−23
σ	7	3	9	7	13	8
O(4)	155	42	125	35	−48	5
σ	7	3	8	7	13	8
O(5)	111	27	288	−2	64	−12
σ	7	3	11	7	15	9
O(6)	87	39	237	25	12	29
σ	6	3	10	7	13	9
O(7)	129	25	223	22	−24	−15
σ	7	3	10	7	14	9
O(8)	96	44	266	32	1	16
σ	7	3	11	7	14	9
$H_2O(1)$	138	31	168	20	64	16
σ	7	3	8	7	13	8
$H_2O(2)$	177	45	129	63	−39	3′
σ	7	3	9	8	14	8
C(1)	91	31	117	23	32	17
σ	8	4	11	9	16	10
C(2)	105	38	120	38	41	24
σ	9	4	11	9	16	10
C(3)	101	27	118	22	5	15
σ	8	3	10	9	16	10
C(4)	103	29	106	27	2	10
σ	2	3	10	9	16	10

Table 3. *Observed and calculated structure amplitudes*

The entries are in the order h, k, l, F_o, F_c

0	0	1	17.2	-17.6
0	0	2	• 43.0	-62.5
0	0	3	20.3	-19.8
0	0	4	12.5	11.5
0	0	5	4.2	4.1
0	0	6	9.5	9.3
0	0	7	5.5	-5.6
1	0	1	15.3	13.0
1	0	-1	12.5	10.2
1	0	2	6.6	-6.4
1	0	-2	21.4	19.7
1	0	3	29.4	-28.2
1	0	-3	29.1	-28.5
1	0	4	23.2	22.4
1	0	-4	10.8	-10.1
1	0	5	24.1	24.0
1	0	-5	9.0	-8.4
1	0	-6	4.9	-5.2
1	0	7	4.6	-4.9
1	0	-7	10.8	10.6
2	0	0	22.7	23.3
2	0	1	38.7	-43.5
2	0	-1	22.7	-21.2
2	0	2	24.3	-23.9
2	0	-2	24.4	23.2
2	0	3	19.3	-18.6
2	0	-3	5.2	-4.7
2	0	4	22.6	22.3
2	0	-4	11.4	-11.7
2	0	5	13.4	13.7
2	0	-5	12.0	-12.0
2	0	7	4.8	-6.3
3	0	0	13.4	-14.1
3	0	1	19.0	-18.9
3	0	-1	16.7	-15.3
3	0	-2	18.4	17.0
3	0	3	8.6	8.3
3	0	-3	27.0	24.8
3	0	4	4.0	3.6
3	0	6	11.4	-11.6
3	0	-6	6.0	-5.8
4	0	0	25.3	-24.6
4	0	1	10.7	-9.6
4	0	2	27.1	26.0
4	0	-2	29.7	29.0
4	0	3	19.7	19.6
4	0	-3	4.6	5.0
4	0	4	9.2	-9.3
4	0	-4	3.1	2.9
4	0	5	8.6	-8.5
4	0	6	3.1	-3.3
4	0	-6	5.2	-5.5
5	0	0	5.5	-6.2
5	0	1	17.6	15.2
5	0	-1	4.0	-3.5
5	0	2	20.9	21.0
5	0	-2	17.3	-16.3
5	0	-3	6.9	-7.3
5	0	4	8.0	-8.0
5	0	-4	5.2	4.6
5	0	5	3.7	-4.0
5	0	-5	11.6	10.9
6	0	0	15.3	14.8
6	0	1	7.4	-7.4
6	0	2	11.9	-11.8
6	0	-2	10.5	-10.7
6	0	3	14.1	14.8
6	0	-3	10.8	-10.1
6	0	4	8.3	-9.4
6	0	-4	8.1	-7.7
6	0	-5	10.1	9.7
6	0	-6	3.7	3.7
7	0	0	5.7	5.7
7	0	1	4.5	5.1
7	0	-1	6.0	5.5
7	0	2	6.3	-6.4
7	0	3	10.8	-12.4
7	0	-3	6.3	-6.3
8	0	1	3.4	-3.4
8	0	-1	9.3	9.4
8	0	-2	8.1	8.5
8	0	-4	7.0	-8.6
0	1	0	3.4	-2.8
0	1	1	36.2	-38.2
0	-1	1	8.0	-8.6
0	1	2	3.3	2.2
0	-1	2	• 72.3	103.6
0	1	3	3.0	3.3
0	-1	3	7.5	7.0
0	1	4	3.4	3.4
0	-1	4	16.6	-16.2
0	-1	5	13.5	-13.2
0	1	6	8.0	7.9
0	1	7	11.7	-12.5
1	1	0	8.4	-9.4
1	-1	0	25.0	24.2
1	1	1	10.5	9.4
1	-1	1	11.7	-10.7
-1	1	1	3.3	3.4
1	1	-1	16.1	15.0
-1	1	2	40.1	-40.0
1	1	-2	29.5	-28.3
1	1	3	3.7	-3.0
-1	1	3	26.5	-25.4
1	1	-3	6.1	7.0
1	1	4	4.3	4.3
1	-1	4	10.2	-10.3
-1	1	4	5.2	-5.3
1	1	-4	38.4	38.7
1	1	5	15.8	15.5
-1	1	5	17.5	16.3
1	1	-5	19.1	-18.2
1	1	6	9.9	-10.2
1	-1	6	6.0	5.2
1	1	-6	25.6	-24.9
1	1	7	3.9	-4.6
1	-1	7	9.2	9.1
2	-1	0	19.4	19.3
2	1	1	8.1	-7.1
2	1	-1	• 81.3	-111.2
2	1	2	17.8	17.7
2	-1	2	16.1	-15.3
-2	1	2	6.1	-6.5
2	1	-2	37.1	37.5
2	1	3	18.8	18.3
2	-1	3	32.3	-29.7
-2	1	3	27.1	-26.4
2	1	-3	20.2	19.7
2	1	4	10.7	10.4
2	-1	4	9.3	-9.7
-2	1	4	4.6	-5.2
2	1	-4	5.4	5.2
2	1	5	11.3	-11.3
2	-1	5	8.1	7.8
-2	1	5	14.6	13.8
2	1	-5	10.7	-10.2
2	-1	6	5.2	4.9
-2	1	6	5.5	5.7
2	1	-6	8.0	-8.2
-2	1	7	5.2	5.1
2	1	-7	6.6	-7.4
5	-1	5	3.7	-3.7
-5	1	5	6.0	-5.5
5	1	-5	7.7	6.3
-5	1	6	14.9	-15.2
5	1	-6	5.4	5.6
-5	1	7	4.2	5.1
6	-1	0	13.2	-13.9
6	-1	1	7.5	7.7
-6	1	1	23.2	-22.6
6	1	2	9.3	-9.4
6	-1	2	10.7	10.4
6	1	-2	8.0	7.7
6	-1	3	5.4	4.9
-6	1	3	21.8	21.0
6	1	-3	9.5	-9.3
6	1	4	3.1	3.0
6	-1	4	13.4	-13.5
6	1	-4	7.8	-7.4
7	1	1	21.7	-22.7
-7	1	1	4.9	-3.9
7	1	-1	20.3	20.5
7	1	2	6.9	-6.8
7	-1	2	13.8	13.7
7	1	3	3.6	4.3
-7	1	3	4.8	-5.3
7	1	-3	8.6	-8.6
-7	1	4	7.8	8.1
-7	1	5	9.8	11.2
8	1	0	3.4	-3.4
8	-1	0	6.1	7.3
8	-1	1	8.7	9.4
8	1	-1	5.7	-5.9
-8	1	2	3.7	-3.7
8	1	-2	4.2	3.9
-8	1	3	5.1	-5.5
8	1	-3	6.0	6.7
8	1	-4	3.4	3.8
0	2	0	6.6	6.5
0	2	1	12.5	-11.7
0	-2	1	• 33.1	-43.3
0	2	2	9.2	-8.1
0	-2	2	14.1	-14.4
0	2	3	13.1	13.2
0	-2	3	34.5	37.3
0	2	4	8.4	8.2
0	-2	4	37.2	36.9
0	2	5	9.2	-9.3
0	-2	5	4.6	-7.9
0	2	6	16.7	-17.0
0	-2	6	12.6	-12.3
0	2	7	3.9	-3.9
1	2	0	22.6	-22.4
1	-2	0	18.8	-17.6
1	2	1	25.3	24.6
1	-2	1	9.7	4.0
-1	2	1	25.8	-26.5
1	2	2	36.8	36.7
-1	2	2	51.6	-54.8
1	2	-2	34.1	34.1
1	2	3	12.6	12.0
1	-2	3	6.4	6.2
-1	2	3	22.3	21.1
1	2	-3	19.9	18.4
1	2	4	23.5	-23.1
1	-2	4	5.5	-5.2
-1	2	4	15.2	15.0
1	2	-4	10.5	-9.9
1	2	5	3.4	2.8
-1	2	5	10.5	9.9
1	2	6	7.0	-8.2
1	-2	6	8.0	-8.2
-1	2	6	13.5	-13.2
1	2	-6	4.9	5.1
2	2	1	56.9	54.9
2	-2	1	41.6	43.0
-2	2	1	45.2	-46.5
2	2	-1	57.0	-60.2
2	2	2	26.8	25.9
2	-2	2	19.7	18.8
-2	2	2	39.3	-39.8
2	2	-2	17.6	15.9
2	2	3	10.4	-10.3
2	-2	3	17.8	-16.7
-2	2	3	37.7	37.4
2	2	-3	36.3	-33.7
2	2	4	5.7	-5.2
2	-2	4	23.2	-23.8
-2	2	4	23.0	22.8
2	2	-4	8.9	7.7
2	2	5	14.3	-13.9
-2	2	5	8.4	7.6
2	2	-5	8.9	8.6
2	2	6	10.7	10.9
2	-2	6	9.2	-10.1
-2	2	6	6.7	-6.3
2	-2	7	6.3	7.0
2	2	-7	5.1	-5.2
3	2	0	10.7	9.9
3	-2	0	45.7	47.4
3	2	1	29.8	-28.1
3	-2	1	60.3	66.4
-3	2	1	12.6	-11.0
3	2	-1	19.0	17.0
3	2	2	3.3	3.5
3	-2	2	29.8	-29.9
-3	2	2	14.6	-12.5
3	2	-2	9.3	-8.3
3	2	3	4.4	-4.9
3	-2	3	22.3	-22.6
-3	2	3	8.1	-7.9
3	2	-3	15.3	-15.2
3	2	4	16.9	-16.2
-3	2	4	10.7	-9.6
3	2	-4	15.3	-15.7
3	2	5	11.9	12.1
-3	2	5	25.5	25.5
3	2	-5	7.7	-7.2
3	2	6	7.7	7.8
3	-2	6	4.0	3.9
3	2	-6	16.9	16.3
-3	2	7	3.6	-4.0
3	2	-7	8.1	9.0
4	-2	0	6.1	6.3
4	2	1	17.8	-17.5
4	-2	1	7.8	7.7
-4	2	1	21.7	17.9
4	2	-1	9.2	8.5
4	2	2	3.7	3.0
4	-2	2	13.4	12.6
-4	2	2	26.1	-25.5
4	2	-2	38.7	39.2
4	2	3	8.4	-8.5
4	-2	3	13.5	-12.5
4	2	-3	28.3	-26.8
4	2	4	7.0	-7.2
4	-2	4	16.1	-16.3
-4	2	4	4.2	-4.0
4	2	-4	6.3	-5.8
4	-2	5	21.7	22.4
-4	2	5	6.7	-6.5
4	-2	6	5.4	5.7
0	-3	2	38.7	-44.0
0	3	3	17.3	-16.4
0	-3	3	11.6	-11.5
0	3	4	35.3	-35.4
0	-3	4	18.1	16.9
0	-3	5	22.4	22.6
0	3	6	7.4	7.3
1	3	0	36.6	37.6
1	-3	0	11.7	-10.6
1	-3	1	33.3	-34.8
1	3	-1	19.4	18.3
1	3	2	33.9	-34.0
1	-3	2	41.0	-41.2
1	3	-2	29.1	-28.9
1	3	3	11.1	-10.7
1	-3	3	20.0	19.3
-1	3	3	25.0	24.2
1	3	-3	9.5	-8.9
1	3	4	15.7	-14.8
1	-3	4	7.5	7.6
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2	8	4	9.2	8.8
2	-8	4	11.3	-11.4
-2	8	4	4.6	-5.1
2	8	-4	9.8	10.4
2	-8	5	11.6	11.9
-2	8	5	3.3	-4.1
2	8	-5	5.2	-5.0
2	-8	6	5.5	6.7
2	8	-6	7.2	-7.9
3	8	1	16.3	-16.4
3	-8	1	9.0	-8.4
-3	8	1	23.0	22.9
3	8	2	4.9	5.1
3	-8	2	28.9	-28.5
-3	8	2	12.9	12.7
3	8	-2	4.0	-3.9
3	8	3	4.8	5.1
-3	8	3	7.8	-7.0
3	-8	4	12.0	11.8
-3	8	4	3.9	-4.5
3	8	-4	11.4	11.3
3	-8	5	4.3	4.6
-3	8	5	3.1	3.3
3	8	-6	5.1	5.9
4	8	0	4.3	2.9
4	-8	0	7.0	-7.0
4	8	1	18.5	19.2
4	-8	1	4.3	-4.3
-4	8	1	19.0	15.2
4	8	-1	16.6	-16.7
4	8	2	4.6	3.2
4	-8	2	8.7	-9.1
4	-8	3	17.5	16.9
-4	8	3	9.3	10.1
4	8	-3	4.3	4.3
4	-8	4	15.2	14.8
4	8	-4	4.8	5.5
4	-8	5	4.5	-4.9
4	8	-5	6.0	7.4
5	8	0	9.9	8.9
5	-8	0	7.2	-7.3
5	8	1	4.8	4.9
-5	8	1	3.7	-4.1
5	8	-1	3.9	3.8
5	8	-2	6.9	-8.9
5	-8	3	12.8	12.3
-5	8	3	7.7	8.3
3	9	-3	8.7	-8.9
3	-9	4	11.1	-11.6
-3	9	4	7.4	-8.7
3	9	-4	3.7	4.3
3	9	-5	6.7	7.3
3	9	-6	3.7	4.5
4	9	0	13.7	13.3
-4	9	1	15.2	14.1
4	9	-1	17.6	16.9
4	9	2	8.4	-9.7
4	-9	2	7.7	-7.6
-4	9	2	7.8	8.9
4	9	-2	10.2	-9.6
-4	9	3	6.0	-6.3
4	9	-3	11.7	-12.0
4	-9	4	6.7	6.0
-4	9	4	7.4	-9.4
4	-9	5	5.5	6.4
4	9	-5	5.2	5.2
5	9	0	8.1	8.1
5	-9	0	5.4	-5.4
5	9	1	8.1	-9.1
5	-9	1	12.5	-11.8
-5	9	1	10.7	11.6
5	9	-1	6.3	5.9
5	-9	2	13.8	-12.6
-5	9	2	5.1	5.6
5	-9	3	9.2	9.2
-5	9	3	3.4	-3.9
5	-9	4	8.1	9.0
5	9	-5	4.9	-6.4
6	-9	0	13.8	-14.9
6	-9	1	19.1	-17.2
-6	9	1	3.4	3.3
6	-9	2	12.0	10.8
-6	9	2	4.5	4.5
0	10	0	17.6	-17.5
0	10	-1	20.2	19.1
0	10	2	4.5	4.4
0	10	-2	9.2	-9.1
0	10	-3	11.3	-11.0
0	-10	4	6.3	5.5
0	-10	5	20.6	19.3
1	10	0	16.9	15.9
1	-10	0	26.2	-25.5
1	10	1	6.7	6.8
1	-10	1	14.4	-13.7
1	10	-1	6.0	-5.4
1	-10	2	4.9	4.6
1	10	-2	16.9	-17.0
1	10	3	3.6	3.5
1	-10	3	14.3	13.7
-1	10	3	12.2	12.2
1	10	-3	11.4	10.9
2	10	0	3.4	2.9
2	-10	0	16.9	-16.3
2	10	1	9.3	9.0
2	-10	1	8.0	-7.9
-2	10	1	9.9	9.9
2	10	-1	17.0	16.8
2	10	2	5.2	-5.8
2	-10	2	6.7	5.9
-2	10	2	6.9	8.0
2	10	-2	9.0	-8.3
2	-10	3	10.1	10.2
-2	10	3	9.3	10.6
2	10	-3	8.0	-8.3
2	-10	4	8.7	-8.3
2	10	-4	8.3	-8.5
2	-10	5	11.1	-10.6
3	10	0	4.6	4.6
3	-10	0	4.6	-4.7
3	-10	1	12.5	11.9
-3	10	1	4.3	-4.9
3	10	2	8.0	9.1
3	-10	2	18.7	17.6
-3	10	2	7.4	-8.7
3	-10	3	7.5	7.1
3	-10	4	14.1	-13.9
3	10	-4	4.8	-5.3
3	-10	5	5.7	-6.3
3	10	-5	4.6	-4.9
4	10	0	7.8	-7.6
4	-10	0	4.5	4.4
4	10	1	4.0	-4.4
4	-10	1	7.8	8.0
4	-10	2	5.7	5.3
-4	10	2	4.3	-5.1
4	10	-2	5.1	5.5
4	-10	3	6.7	-5.9
-4	10	3	8.4	-10.9
4	10	-3	15.0	17.3
4	10	-4	6.6	-6.9
4	10	-5	7.8	-9.7
5	-10	0	12.6	13.4
5	-10	1	3.9	3.8
-5	10	1	4.8	-5.0
5	10	-1	5.5	-5.8
5	-10	2	12.0	-12.4
-5	10	2	6.7	-8.0
5	10	-2	6.0	6.5
5	10	-3	4.3	5.1
6	-10	0	7.4	7.6
6	-10	1	3.0	-3.0
0	11	0	8.0	7.7
0	11	2	7.8	-8.1
0	11	-2	9.6	-9.7
0	-11	4	10.2	-9.8
1	11	0	13.2	12.0
1	-11	0	4.5	5.5
-1	11	1	8.3	9.2
1	11	-1	16.6	16.1
1	11	2	15.8	-16.0
1	-11	2	18.1	-17.8
-1	11	2	3.6	3.0
1	11	-2	3.0	3.2
1	-11	3	14.4	-15.2
1	11	-3	10.1	-10.4
1	-11	4	3.4	3.7
1	11	-4	10.7	-11.2
1	-11	5	11.0	12.0
1	11	-5	10.1	10.4
2	11	0	8.4	-8.3
2	-11	0	10.1	9.3
2	11	1	8.9	-8.3
2	11	-1	19.7	20.2
2	-11	2	11.3	-11.1
-2	11	2	4.8	5.6
2	11	-3	5.2	5.4
2	-11	4	9.3	9.5
2	11	-5	5.2	-5.8
3	11	0	5.4	-5.1
3	-11	0	6.7	-7.4
3	-11	1	11.0	-10.9
3	11	-1	3.6	-3.7
3	-11	2	5.1	4.6
-3	11	2	11.7	12.3
3	11	-2	5.7	5.9
3	-11	3	14.4	14.8
3	11	-3	5.5	5.8
3	-11	4	4.0	4.4
3	11	-4	6.0	-6.3

Table 3 (*cont.*)

3	-1	0	6.1	-5.4
3	1	1	14.0	12.8
3	-1	1	9.5	-8.4
3	1	2	22.9	22.0
3	-1	2	12.2	-12.5
-3	1	2	22.7	22.3
3	1	-2	30.4	-27.0
3	1	3	6.0	-6.0
-3	1	3	25.3	25.3
3	1	-3	8.0	7.9
3	1	4	23.2	-23.2
-3	1	4	16.0	-14.8
3	1	-4	25.9	24.7
3	1	5	10.4	-9.9
3	-1	5	4.0	4.2
-3	1	5	11.7	-12.0
3	1	6	6.1	6.6
-3	1	6	4.3	-3.7
-3	1	7	15.0	17.0
4	1	0	25.8	24.1
4	-1	0	15.8	14.9
4	1	1	20.6	20.1
4	-1	1	12.5	-11.6
-4	1	1	6.7	7.0
4	1	-1	24.6	-23.2
4	1	2	9.8	8.9
4	-1	2	6.6	-6.0
4	1	-2	6.4	-5.3
4	1	3	20.2	-20.5
4	-1	3	13.8	14.0
-4	1	3	22.7	22.5
4	1	-3	5.7	-4.8
4	1	4	8.0	-7.7
-4	1	4	12.8	-11.8
4	1	-4	18.8	17.6
4	-1	5	14.4	15.2
-4	1	5	11.9	-11.5
4	1	-5	5.4	5.9
4	-1	6	3.1	-3.3
-4	1	6	4.0	-4.1
-4	1	7	3.6	3.6
5	1	0	19.6	20.0
5	-1	0	38.3	-40.1
5	1	1	8.9	8.8
-5	1	1	20.8	-14.9
5	1	-1	9.5	9.2
5	1	2	5.8	-5.3
5	-1	2	8.3	7.9
-5	1	2	23.6	22.2
5	1	-2	6.0	5.6
5	1	3	8.7	-8.1
5	-1	3	14.1	13.1
-5	1	3	12.2	11.4
5	1	-3	13.8	-13.0
5	1	4	6.4	-6.6
5	-1	4	11.6	11.9
-5	1	4	3.4	-3.6
5	1	-4	16.7	-16.0
5	1	5	6.4	7.3
4	2	-6	8.1	7.7
-4	2	7	5.8	6.9
4	2	-7	5.2	6.0
5	2	0	4.6	4.5
5	2	1	15.7	-15.5
5	-2	1	4.9	-5.1
5	2	-1	9.8	9.6
5	2	2	14.9	-14.7
5	-2	2	13.4	-12.8
-5	2	2	16.0	15.3
5	2	-2	19.9	19.6
5	2	3	11.6	11.2
5	-2	3	14.0	-14.2
-5	2	3	8.1	-8.1
5	2	-3	6.7	7.1
5	2	4	12.3	12.7
-5	2	4	20.2	-19.2
5	2	-4	3.4	-3.4
5	-2	5	14.6	15.2
5	2	-5	10.2	-9.8
5	2	-7	4.0	4.7
6	2	1	13.7	-13.2
6	-2	1	20.6	-21.2
-6	2	1	14.1	13.4
6	2	2	3.7	4.0
6	-2	2	9.9	-10.3
6	2	-2	7.4	6.7
6	2	3	9.9	9.9
6	-2	3	12.8	13.4
6	2	4	6.9	7.9
6	-2	4	3.0	3.3
6	2	-4	6.1	5.8
-6	2	5	4.2	4.2
6	2	-5	10.1	-10.3
-6	2	6	4.9	-5.2
7	2	0	10.2	-10.9
7	2	1	3.6	3.8
7	-2	1	11.4	-11.0
-7	2	1	4.3	-4.5
7	2	-1	20.5	-20.4
7	2	2	7.0	7.0
7	-2	2	7.2	7.2
-7	2	2	9.0	8.5
7	-2	3	5.8	5.8
-7	2	3	8.9	9.3
7	2	-3	12.9	13.1
-7	2	4	4.5	5.1
7	2	-4	3.9	3.9
-7	2	5	5.5	-6.9
7	2	-5	4.9	-4.3
8	2	1	8.3	9.9
-8	2	1	6.0	-6.4
8	2	-1	6.4	-6.1
-8	2	2	6.3	-6.3
8	2	-3	4.9	-5.3
8	2	-4	5.4	6.5
0	3	1	36.9	38.0
0	-3	1	22.6	-22.2
0	3	2	6.7	6.6
8	-3	0	13.2	-14.2
8	-3	1	12.0	-12.6
-8	3	1	5.1	5.3
-8	3	2	7.8	8.9
-8	3	3	3.4	4.1
8	3	-3	3.6	-3.5
0	4	0	21.1	-19.2
0	4	1	7.0	6.7
0	4	-1	29.2	28.9
0	4	2	21.4	-20.7
0	-4	2	5.5	-5.8
0	4	3	24.9	-24.2
0	-4	3	15.2	-15.8
0	4	4	4.2	-3.7
0	-4	4	9.6	-10.0
0	4	5	20.0	19.2
0	-4	5	12.9	-12.1
0	4	6	8.0	7.0
0	-4	7	23.3	21.9
1	4	0	48.6	-49.9
1	-4	0	40.7	42.2
1	4	1	13.7	12.4
1	-4	1	• 62.9	-71.1
-1	4	1	50.5	54.7
1	4	-1	5.4	5.0
1	4	2	5.8	-5.7
1	-4	2	20.0	-18.2
-1	4	2	13.8	-13.3
1	4	-2	48.1	44.6
1	4	3	3.0	-3.1
1	-4	3	16.3	15.3
-1	4	3	3.1	2.1
1	4	-3	20.2	19.6
1	4	4	13.8	-14.2
1	-4	4	12.9	-12.3
-1	4	4	15.2	-15.3
1	4	-4	27.6	-28.0
1	4	5	9.3	9.1
-1	4	5	3.4	2.8
1	4	-5	5.7	-4.6
1	-4	6	5.5	5.9
-1	4	6	12.3	12.9
1	4	-6	7.4	7.2
1	4	-7	7.0	7.8
2	4	0	20.6	-19.7
2	-4	0	12.3	12.1
2	4	1	14.7	-13.9
2	-4	1	24.4	23.6
-2	4	1	16.4	15.5
2	4	-1	10.8	10.7
2	-4	2	18.8	-19.0
2	4	3	24.3	23.8
2	-4	3	14.4	-14.4
2	4	-3	27.9	28.5
2	-4	4	15.8	15.5
-2	4	4	4.9	-5.0
2	-4	5	8.9	9.0
-2	4	5	8.6	-7.9
2	-5	3	8.6	-8.1
-2	5	3	24.6	-23.7
2	5	-3	23.3	22.8
2	5	4	10.2	-10.2
2	-5	4	7.0	-6.9
2	5	-4	5.1	5.0
2	5	5	17.9	-17.4
-2	5	5	14.6	15.0
2	-5	6	7.4	7.5
-2	5	6	3.3	3.5
2	5	-6	7.4	7.4
3	5	0	9.3	-8.4
3	-5	0	32.9	30.8
3	5	1	17.9	17.5
3	-5	1	9.8	-9.0
-3	5	1	13.7	5.6
3	5	-1	16.9	16.0
3	5	2	9.3	9.1
3	-5	2	16.3	-15.4
-3	5	2	4.0	-3.2
3	5	-2	10.4	-8.3
3	5	3	5.5	5.9
3	-5	3	28.5	-28.5
-3	5	3	27.1	-26.8
3	5	-3	9.9	-9.3
3	5	4	11.0	-11.3
3	-5	4	19.0	18.9
-3	5	4	4.6	-4.3
3	-5	5	7.7	7.3
3	5	-5	9.0	8.7
3	-5	6	10.5	10.2
-3	5	6	4.3	4.7
3	5	-7	3.0	-2.9
4	5	0	12.8	12.0
4	-5	0	6.1	5.9
4	5	1	17.3	17.2
4	-5	1	9.3	-9.6
-4	5	1	6.9	7.1
4	5	-1	4.5	-4.2
4	5	2	9.0	-8.7
4	-5	2	17.5	-17.4
-4	5	2	4.3	-4.1
4	5	-2	11.7	-11.1
4	5	3	4.5	-4.6
4	-5	3	16.0	-15.6
4	5	-3	13.1	-13.6
4	-5	4	9.0	9.4
4	5	-4	8.7	-9.0
4	-5	5	7.7	7.8
4	5	-5	6.4	6.5
4	-5	6	4.5	5.0
4	5	-6	7.2	7.8
5	5	0	15.5	-15.0
5	-5	0	14.1	13.6
5	-5	1	37.7	-38.7
-5	5	1	18.1	17.8
5	5	-1	22.7	22.8
5	5	2	10.2	-10.7
7	6	0	7.8	-8.3
7	-6	0	15.3	-15.1
7	-6	1	8.7	-8.9
-7	6	2	7.7	7.5
7	6	-2	6.7	-6.7
7	-6	3	5.8	6.7
-7	6	3	4.0	5.1
7	6	-4	6.9	7.5
8	-6	0	8.3	-9.3
0	7	0	6.9	6.4
0	7	1	26.5	26.9
0	7	2	4.2	3.7
0	7	3	9.2	-9.4
0	7	5	3.6	-3.2
0	-7	6	6.6	6.7
-1	7	1	8.4	-9.0
1	7	-1	3.7	-3.0
1	7	2	5.4	6.0
-1	7	2	25.9	25.7
1	7	3	13.5	-13.4
1	-7	3	7.0	-7.0
-1	7	3	12.6	12.5
1	7	-3	16.6	-16.8
1	-7	4	23.3	23.2
-1	7	4	13.4	-13.9
1	7	-4	4.9	-5.1
-1	7	5	22.6	-22.1
1	7	-5	11.4	-11.3
1	-7	6	5.4	5.5
1	7	-6	13.5	14.7
1	-7	7	5.2	-5.8
2	-7	0	17.5	-17.8
2	7	1	20.5	-20.7
2	-7	1	24.3	-23.5
-2	7	1	6.9	7.3
2	7	-1	9.2	8.2
2	7	2	21.1	-21.2
-2	7	2	5.2	5.0
2	7	3	5.7	5.9
2	-7	3	20.5	20.7
-2	7	3	5.8	5.3
2	7	4	8.3	8.3
-2	7	4	12.3	-12.4
2	7	-4	20.9	-21.2
2	7	-5	9.0	-9.3
2	-7	6	4.5	4.5
2	7	-6	9.8	10.3
2	7	-7	4.9	6.1
3	7	0	17.5	-17.1
3	-7	0	43.9	-44.9
-3	7	1	6.3	-6.2
3	7	-1	8.3	8.0
3	7	2	7.8	7.4
3	-7	2	18.5	18.1
-3	7	2	14.1	13.2
3	7	-2	12.9	13.2
3	7	3	11.0	-11.2
-3	7	3	6.7	6.7
5	8	-3	3.9	-4.3
5	-8	4	6.1	6.0
5	8	-5	4.5	-5.1
6	-8	1	10.1	9.3
-6	8	1	7.5	-7.7
6	8	-1	5.1	4.4
-6	8	2	12.3	-13.1
6	8	-2	4.3	4.2
6	-8	3	3.7	-3.2
6	8	-3	6.6	-6.9
6	8	-4	9.8	-10.4
7	-8	0	8.3	-8.3
7	-8	1	9.0	10.0
0	9	0	21.7	-21.1
0	9	1	22.1	21.8
0	9	-1	13.7	-13.2
0	9	2	6.6	-6.9
0	9	-2	10.4	10.0
0	-9	3	25.6	25.4
0	9	4	5.1	4.8
0	-9	4	3.1	2.8
0	-9	6	10.8	-10.4
1	9	0	5.1	-5.0
1	-9	0	5.1	-4.8
1	-9	1	27.3	26.3
-1	9	1	33.5	-33.9
1	9	-1	4.6	-4.0
1	9	2	11.7	11.0
1	-9	2	3.7	-3.6
-1	9	2	8.1	-7.6
1	9	3	8.6	8.8
1	-9	3	7.4	7.6
-1	9	3	9.9	10.0
1	9	-3	8.6	8.9
1	-9	4	4.0	-3.9
1	-9	5	9.6	-10.2
2	-9	0	12.6	-12.9
2	9	1	11.7	11.5
2	-9	1	8.0	7.9
-2	9	1	13.7	12.6
2	9	2	6.6	6.6
-2	9	2	10.2	-10.1
2	9	-2	26.7	-26.0
2	9	-3	4.0	-4.1
2	-9	4	13.7	-13.7
2	9	-4	9.2	9.0
2	-9	5	6.1	-6.0
2	9	-5	8.0	8.7
3	9	0	8.1	7.0
3	-9	0	4.6	5.3
3	9	1	9.5	10.2
3	-9	1	10.4	8.9
-3	9	1	14.1	13.8
3	9	-1	11.6	-11.7
3	9	-2	6.0	-5.4
3	9	3	11.0	-13.2
3	-9	3	4.8	4.0
-3	9	3	5.1	-5.6
4	-11	0	10.2	-10.4
4	-11	1	5.7	4.9
-4	11	1	10.5	-11.5
4	-11	2	9.2	8.3
-4	11	2	3.3	4.4
4	11	-2	7.7	-8.2
4	11	-3	6.0	7.5
-5	11	1	3.4	-4.4
0	12	0	4.3	3.9
0	12	1	10.7	-11.2
0	-12	4	3.3	-3.4
1	12	0	6.0	-5.6
1	-12	0	6.9	8.4
1	12	1	7.0	-7.5
1	-12	1	12.3	11.2
1	-12	3	9.5	-10.4
1	12	-3	13.8	13.8
1	-12	4	8.4	-9.1
1	12	-4	5.5	6.2
2	-12	0	3.6	3.8
2	12	-1	8.0	-8.2
2	12	-2	6.3	-6.4
2	-12	3	5.4	-5.5
2	12	-3	12.6	12.8
2	12	-4	4.9	5.8
3	-12	0	4.5	5.2
3	-12	1	10.8	-10.6
3	12	-1	8.0	-9.5
4	-12	0	8.3	-8.7
4	-12	1	11.0	-11.6
0	13	-1	10.7	-11.2
0	13	-3	4.8	5.4
1	13	-2	5.2	-5.7

The standard deviations for all angles were increased by the factor (bond angle in degrees/90 (Dunitz & Rollett, 1956).

After two cycles of least-squares refinement with anisotropic temperature factors, we calculated an $(F_o - F_c)$ electron density map; from this we were able to locate all seven hydrogen atoms. (No corrections were applied to the atomic positions determined from this map; the ratio of the average peak height to the background was 1·5.) These were included in the final cycle of the least-squares refinement. The results of these calculations are given in Tables 1, 2 and 3.

Description of the structure

Even though the space group of potassium tetraoxalate is $P\bar{1}$, it is convenient to draw the molecular arrangement referred to a unit cell with an origin not at a center of inversion; the origin of this cell has been chosen at one of the potassium ions. The two acid oxalate groups are nearly parallel to the (001) plane, while the oxalic acid molecules lie in a plane essentially perpendicular to this, parallel to (100). This structure is depicted in Fig. 1(*a*).

Although the asymmetric unit contains the atoms of only one molecule of oxalic acid, each half of this molecule is unrelated to the other by any symmetry element. Two independent oxalic acid molecules result from these halves by the operation of two different centers of inversion. This gives us two carbon–carbon distances for two symmetrically unrelated oxalic acid molecules. In the refinement, therefore, one carbon atom from each of the two oxalic acid molecules in the unit cell must be chosen, while both carbon atoms from the potassium acid oxalate group are required. These three distances are $1{\cdot}55_3$, $1{\cdot}55_4$, and $1{\cdot}55_1$ Å, the last being that of the potassium acid oxalate group (Fig. 2). It can be seen, therefore, that these results are consistent among themselves, and support the view that no shortening of the carbon–carbon bond occurs in either the oxalic acid molecule or the acid oxalate ion. This is in agreement with previous results (Ahmed & Cruickshank, 1953).

The remainder of the two symmetrically unrelated oxalic acid molecules and the un-ionized carboxylic acid group on the acid oxalate in does not appear to be significantly different. The primary difference is in the O(6)–C(3)–O(5) angle, but this does not seem to be chemically significant. On the other hand, the short C(4)–O(7) bond length indicated that O(7) is the ionized oxygen. This agreed with our belief that the acid oxalate ion could not have a center of inversion between its carbon atoms.

Best planes were calculated for the two symmetrically unrelated oxalic acid molecules and the acid oxalate group. The oxalic acid molecules are planar, whereas the acid oxalate group is not. The deviations of atoms from these best planes for each molecule are given in Table 4.

The potassium ion has nine nearest oxygen neighbors at an average distance of 2·95 ± 0·35 Å (or 2·92 ± 0·15 Å if atom O(2) is not included); these are listed in Table 5. The coordinating oxygen atoms around the potassium ion are arranged in the form

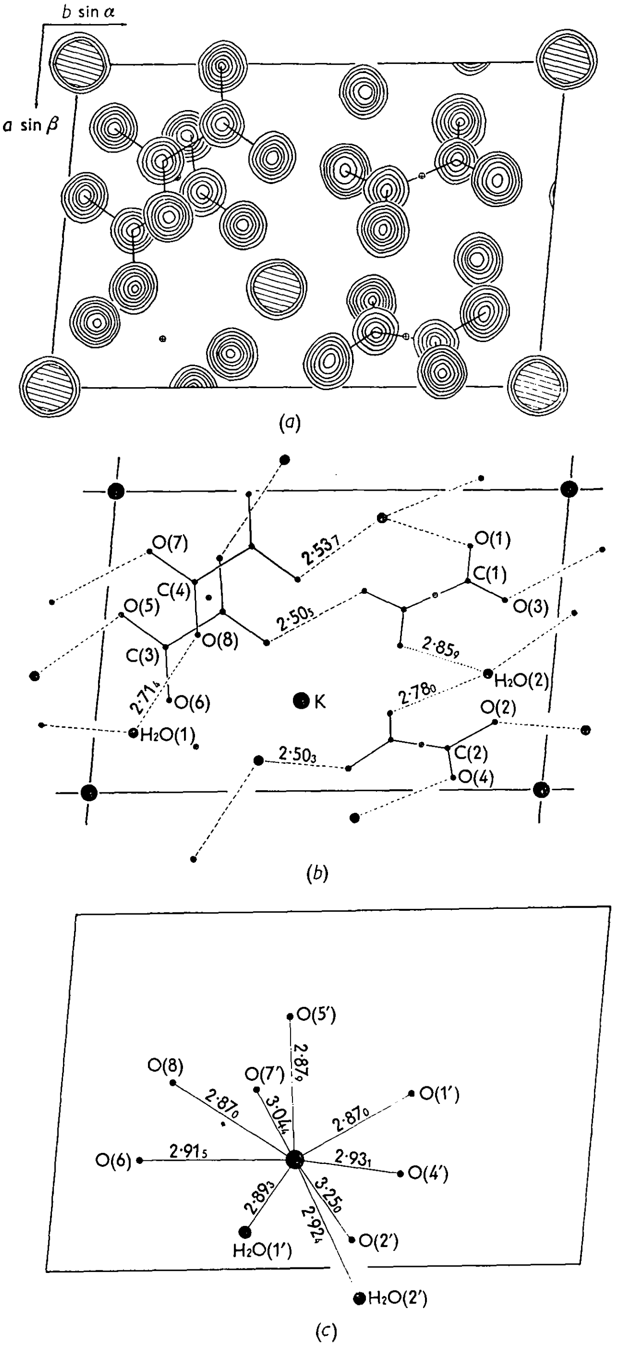

Fig. 1. (*a*). Composite electron density map with the origin of the unit cell at a potassium ion; contours at approximately 1 e.Å^{-3} levels. The centers of inversion are represented by crossed circles. (*b*) Hydrogen bonds of the structure; dotted bond crosses *C* face of the cell. The centers of inversion are represented by crossed circles. (*c*) Coordination of the potassium ion.

of an irregular nonaverticon; the arrangement is presented in Fig. 1(*c*).

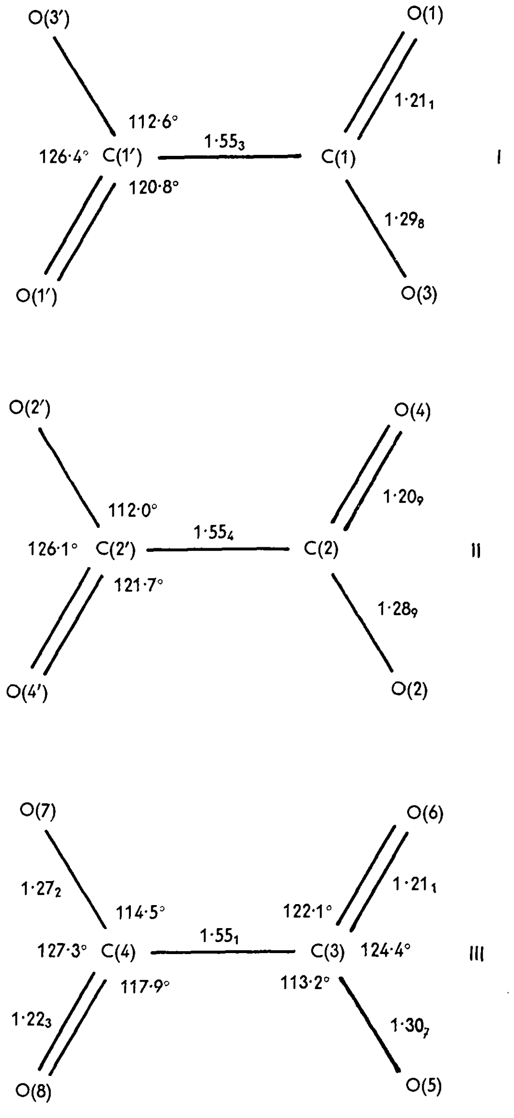

Fig. 2. Bond lengths (Å) and angles of the two symmetrically unrelated oxalic acid molecules (I, II) and the acid oxalate group (III).

The four water molecules in the unit cell are involved in the three-dimensional hydrogen bonding network (Fig. 1(*b*)). The two crystallographically different water molecules in the asymmetric unit coordinate the potassium ion and have different surroundings: $H_2O(2)$ has 6 neighboring oxygen atoms and forms three hydrogen bonds; $H_2O(1)$ has 4 neighboring oxygen atoms, two of which are hydrogen bonded to the water. Only one of these latter hydrogen bonds, however, uses a hydrogen atom belonging to the water molecule. The unbound hydrogen was located and is directed toward O(8); the distance from the water to this oxygen atom is 3·09$_8$ Å. The positions of all these hydrogen atoms were predicted from chemical consideration of the hydrogen bonding before the final refinement was begun.

In all, there are six hydrogen bonds, three of which are unusually short; all three of these cross planes

Table 4. *Deviations from the best planes*

Molecule I

Equation of plane $0{\cdot}81x+0{\cdot}38y-0{\cdot}45z=4{\cdot}17$

Standard deviation of distances from plane 0·001 Å

Atom	Distance to plane
C(1)	−0·002 Å
O(1)	0·000
O(3)	0·000
C(1′)	0·002
O(1′)	0·000
O(3′)	0·000

Molecule II

Equation of plane $0{\cdot}88x+0{\cdot}35y-0{\cdot}33z=7{\cdot}49$

Standard deviation of distances from plane 0·000 Å

Atom	Distance to plane
C(2)	0·000 Å
O(2)	0·000
O(4)	0·000
C(2′)	0·000
O(2′)	0·000
O(4′)	0·000

Molecule III

Equation of plane $-0{\cdot}18x-0{\cdot}29y+0{\cdot}94z=-0{\cdot}51$

Standard deviation of distances from plane 0·047 Å

Atom	Distance to plane
C(3)	0·002 Å
O(5)	0·056
O(6)	−0·058
C(4)	0·003
O(7)	0·054
O(8)	−0·059

Angle between planes C(3)–O(5)–O(6) and C(4)–O(7)–O(8) is 6·1°.

parallel to (010) at $y=0$, $\frac{1}{2}$ with respect to the potassium ion.

Owing to the high temperature for decomposition (the crystals lose water and change to powder at approximately 200 °C) and the low temperature factors derived from X-ray data, one can say that all the atoms are packed quite tightly to form the crystal structure. The anisotropic refinement indicates that vibrations of the oxalic acid molecules and acid oxalate groups are mainly perpendicular to the plane of the molecule.

No preferential cleavage direction would be expected, because hydrogen bonds and electrostatic attractions between the potassium and acid oxalate ions extend in all directions.

Table 5. *Significant interatomic distances and bond angles*

K–O(1)	2·870 ± 0·004 Å
K–O(2)	3·250 ± 0·004
K–O(4)	2·931 ± 0·004
K–O(5)	2·879 ± 0·004
K–O(6)	2·915 ± 0·004
K–O(7)	3·044 ± 0·004
K–O(8)	2·870 ± 0·004
K–$H_2O(1)$	2·893 ± 0·004
K–$H_2O(2)$	2·924 ± 0·004

Molecule I

O(1)–C(1)	1·211 ± 0·005 Å	O(1)–C(1)–O(3)	126·4 ± 0·6°
O(3)–C(1)	1·298 ± 0·005	O(1)–C(1)–C(1′)	120·8 ± 0·5
C(1)–C(1″)	1·553 ± 0·009	O(3)–C(1)–C(1′)	112·6 ± 0·5

Molecule II

O(4)–C(2)	1·209 ± 0·006 Å	O(4)–C(2)–O(2)	126·1 ± 0·6°
O(2)–C(2)	1·289 ± 0·006	O(4)–C(2)–C(2′)	121·7 ± 0·6
C(2)–C(2′)	1·554 ± 0·009	O(2)–C(2)–C(2′)	112·0 ± 0·5

Molecule III

O(6)–C(3)	1·211 ± 0·006 Å	O(6)–C(3)–O(5)	124·4 ± 0·6°
O(5)–C(3)	1·307 ± 0·006	O(6)–C(3)–C(4)	122·1 ± 0·5
C(3)–C(4)	1·551 ± 0·009	O(5)–C(3)–C(4)	113·2 ± 0·5
O(7)–C(4)	1·272 ± 0·006	O(7)–C(4)–O(8)	127·3 ± 0·6
O(8)–C(4)	1·223 ± 0·006	O(7)–C(4)–C(3)	114·5 ± 0·4
		O(8)–C(4)–C(3)	117·9 ± 0·4

Hydrogen bonds

O_a	H	O_b	O_a–H (Å)	O_a–O_b (Å)	H–O_a–O_b (°)
O(5)	H(1)	$H_2O(2)$	0·9 ± 0·2	2·537 ± 0·005	7·0 ± 13·0
$H_2O(1)$	H(2)	O(8)	0·9 ± 0·2	2·714 ± 0·005	11·3 ± 13·0
$H_2O(2)$	H(3)	O(4)	0·9 ± 0·2	2·780 ± 0·005	10·4 ± 13·0
O(2)	H(4)	$H_2O(1)$	1·0 ± 0·2	2·503 ± 0·005	3·6 ± 13·0
O(3)	H(5)	O(7)	0·9 ± 0·2	2·505 ± 0·005	16·7 ± 13·0
$H_2O(2)$	H(6)	O(1)	0·9 ± 0·2	2·859 ± 0·005	23·0 ± 13·0
$H_2O(1)$	H(7)	O(8)	0·8 ± 0·2	3·098 ± 0·005	

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