Then Ω, the solid angle shielded is given by

$$\Omega = 2\int_{\gamma-\beta}^{\alpha}\int_{0}^{F(\theta)} \sin\theta \, d\theta \, d\varphi$$
$$= 2\int_{\gamma-\beta}^{\alpha} \sin\theta \, F(\theta) \, d\theta \, .$$

Finally we get the 'shielded fraction' by dividing by the total solid angle subtended by oxygen O(1).

$$\text{Shielded fraction} = \frac{\Omega}{2\pi\,(1-\cos\alpha)} \, .$$

Case 3. *Large fraction shielded*

If $\gamma<\beta<\alpha+\gamma$, overlap occurs and the width of the area shielded lies between the radius and the diameter of the atom being shielded. In this case the solid angle shielded $= A+B$,

$$\text{where } A = \int_{0}^{\beta-\gamma}\int_{0}^{2\pi} \sin\theta \, d\theta \, d\varphi$$
$$= 2\pi[1-\cos(\beta-\gamma)]$$
$$\text{and} \quad B = 2\int_{\beta-\gamma}^{\alpha} \sin\theta \, \mathrm{F}(\theta) \, d\theta \, .$$

Then for the 'shielded fraction' we have

$$\text{'Shielded fraction'} = \frac{A+B}{2\pi(1-\cos\alpha)} \, .$$

Case 4. *Complete shielding*

If $\beta\geq\alpha+\gamma$ the oxygen atom is completely shielded and

'Shielded fraction' = 1.

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Neutron Diffraction Refinement of the Structure of Potassium Oxalate Monohydrate

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The structure of potassium oxalate monohydrate has been refined to a high degree of precision using three-dimensional neutron diffraction data. The conventional R value is 0·026 and the estimated standard deviations of atomic coordinates are between 0·001 and 0·003 Å. The oxalate ion is not quite planar with a separation of 0·037 (6) Å between the two parallel carboxyl planes. The C–C bond length is 1·581 (2) Å and the two C–O bond lengths are 1·262 (2) and 1·256 (2) Å. The O–H distance in the hydrogen bonds from the water molecule to the oxalate ion is 0·963 (3) Å; the O · · · O distance is 2·754 (2) Å, the O–H---O angle is 169·66 (22)° and the H–O–H angle, 107·62 (33)°. The root-mean-square amplitude of thermal motion of the water oxygen atom is considerably larger than that of the hydrogen atoms in some directions. The structure is compared with a recent X-ray structure of the same crystal and the agreement is excellent.

Introduction

In an earlier paper (Chidambaram, Sequeira & Sikka, 1964) (CSS) we reported a two-dimensional neutron diffraction study of the structure of potassium oxalate monohydrate. In that study the water molecule was shown to have a rather unusual type of coordination with neither of its two lone-pairs of electrons being specifically directed. The water molecule in this crystal has recently also been the subject of extensive studies by proton magnetic resonance (McGrath & Paine, 1964; Pederson, 1966, 1968). McGrath & Paine observed that, unlike the case for $Ba(ClO_3)_2.H_2O$, $Li_2SO_4.H_2O$ or gypsum, the proton-doublet splitting in this crystal showed surprisingly little or no temperature dependence. In view of the unusual coordination and behaviour of the water molecule it seemed worth while to investigate this structure further using three-dimensional neutron diffraction data. Towards the completion of this investigation, however, an accurate X-ray diffraction study using diffractometer data (Hodgson & Ibers, 1969) (HI) came to our attention. The results of the X-ray study have been compared with the present results, the two being of comparable precision.

Experimental

Neutron intensity data were collected using the 4-circle neutron diffractometer 3-D FAD (Momin, Sequeira & Chidambaram, 1968) at the CIRUS reactor in Trombay. A cylindrical crystal specimen, 4·2 mm in diameter and 4·7 mm long (weight 126 mg) with its axis parallel to the crystallographic b axis, was prepared from a large single crystal grown by slow evaporation from a saturated aqueous solution. The crystal was dipped several times in liquid nitrogen so as to reduce extinction effects and mounted in the 'symmetric' position with its b axis parallel to the φ axis. The orientation of the specimen and the cell parameters were first refined on the basis of the optimized 2θ, χ and φ values of some 20 arbitrary reflexions, using our program *REFINE* (Srikanta, 1968*a*). The refined values of the cell constants (space group $C2/c$) agree well with the more reliable values obtained by HI which are as follows: $a=9{\cdot}222$ (3), $b=6{\cdot}197$ (2), $c=10{\cdot}690$ (5) Å and $\beta=110{\cdot}70$ (3)°. The intensities of 414 independent reflexions within the limit $\sin\theta/\lambda=0{\cdot}575$ ($\lambda=1{\cdot}406$ Å) were then recorded using the θ–2θ coupled step-scanning technique. The scan-lengths ranged from 4 to 6° in steps of 0·1°. As the automation was not completed at the time, the recording was done manually at the rate of two reflexions per hour. Two standard reflexions were measured after every 20 reflexions to keep a check on the stability of the crystal and that of the counting equipment. The peak intensities of the $0k0$ reflexions showed little variation as a function of rotation about the scattering vector, indicating that the effects of multiple reflexions were small. It was not practicable to check the other reflexions for the effects of multiple reflexion.

The data were reduced using our program *DATARED* (Srikanta, 1968*b*). The signal counts were corrected for background in the standard way to obtain the net counts C_N, whose standard deviations $[\sigma(C_N)]$ were estimated using the following relation (Busing & Levy, 1957)

$$\sigma^2(C_N)=[C_S+(N_S/N_B)^2C_B+(0{\cdot}1C_N)^2]\,,$$

where C_S is the total signal count in N_S steps and C_B is the background count in N_B steps. C_N and $\sigma(C_N)$ were then converted to F^2 and $\sigma(F^2)$ respectively, by applying the Lorentz and absorption corrections. An absorption coefficient of 0·75 cm^{-1} (measured) was used. The values of F^2 were averaged* for the few reflexions measured more than once.

Refinement

The atomic positions and isotropic temperature factors reported in the two-dimensional study of CSS were taken as the starting parameters for the refinement. An initial value of the scale factor was also estimated by comparison with the two-dimensional data. The least-squares refinement was carried out on F^2 with weights $\omega=1/\sigma^2(F^2)$, using the program *ORFLS* (Busing, Martin & Levy, 1962) incorporating modifications by Hamilton, Ibers & Johnson. Three cycles of isotropic refinement followed by three cycles of anisotropic refinement resulted in values of 0·061 and 0·117 respectively, for $R_1(=\Sigma||F_o|-|F_c||/\Sigma|F_o|)$ and $R_2(=\Sigma|F_o^2-F_c^2|/\Sigma F_o^2)$. At this stage it was obvious that the F_o^2 values of the intense reflexions were affected by extinction and hence an empirical extinction correction of the form

$$F_o^2(\text{corrected})=F_o^2\exp(gQ)\,,$$

where $Q\equiv F_o^2(\text{corrected})/\sin 2\theta$, was applied. A value of 0·00202 for the constant g was obtained by plotting $\log(F_o^2/F_c^2)$ as a function of $F_c^2/\sin 2\theta$. This reduced R_1 and R_2 to 0·044 and 0·068 respectively in two more cycles of refinement. An error-analysis carried out at this stage gave the following relation for the average discrepancies between observed and calculated F^2's:

$$\langle|\Delta(F^2)|\rangle_{\text{av}}=0{\cdot}15+0{\cdot}05Q\,.$$

Two more cycles of anisotropic refinement using a weighting scheme $\omega^{-1/2}=\langle|\Delta(F^2)|\rangle_{\text{av}}$ and a revised value of $g=0{\cdot}00218$ reduced R_1 and R_2 to 0·038 and 0·054. An examination of the data at this stage showed that for a few (11) weak reflexions the discrepancies ($|\Delta(F^2)|$) were greater than 5σ. These reflexions were rejected leaving 403 independent reflexions. The error-analysis was repeated ($\langle|\Delta(F^2)|\rangle=0{\cdot}138+0{\cdot}0374Q$) and further anisotropic refinement was carried out omitting observations for which $F_o^2<2\sigma$. Finally the scattering amplitudes of K, O and C atoms were also allowed to vary, the scattering amplitude of H being fixed at $-0{\cdot}378\times10^{-12}$ cm. Their values (in units of 10^{-12} cm) converged to 0·373 (8), 0·593 (10) and 0·675 (12) for K, O and C respectively. The final R values were as follows:

Number of observations	R_1	R_2	R_ω^*
403	0·029	0·045	0·061
375 ($F_o>2\sigma$)	0·026	0·045	0·060

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* The mutual agreement in the intensities of the repeated reflexions was generally better than 1 per cent.

Table 1. *Positional and thermal parameters for* $K_2C_2O_4.H_2O$

The X-ray parameters are from Hodgson & Ibers, and our numbering system corresponds to theirs. The values of all the parameters have been multiplied by 10^5, and the standard deviations are given in parentheses. The differences Δ are expressed in terms of the pooled error $\sigma_p \equiv (\sigma^2_{\text{x-ray}}+\sigma^2_{\text{neutron}})^{1/2}$.

		x	y	z	β_{11}*	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
K	Neutron	13202 (19)	83024 (27)	13207 (16)	681 (28)	1363 (64)	408 (20)	−12 (25)	194 (15)	103 (21)
	X-ray	13179 (3)	83038 (5)	13194 (3)	701 (4)	1331 (8)	427 (3)	−10 (4)	181 (3)	106 (3)
	Δ/σ_p	1·2	0·5	0·8	0·7	0·5	1·0	0·1	0·9	0·1
O(1)	Neutron	12622 (12)	26965 (19)	9153 (11)	770 (17)	1568 (36)	589 (13)	−192 (16)	475 (11)	−207 (14)
	X-ray	12580 (10)	26910 (20)	9130 (10)	810 (10)	1710 (30)	620 (10)	−190 (20)	470 (10)	−170 (10)
	Δ/σ_p	2·6	2·0	1·4	2·0	3·0	1·8	0·1	0·3	2·2
O(2)	Neutron	31972 (13)	48648 (17)	9262 (11)	866 (18)	1297 (38)	534 (13)	−268 (17)	357 (11)	−235 (14)
	X-ray	31960 (10)	48690 (20)	9240 (10)	900 (10)	1310 (20)	580 (10)	−260 (10)	320 (10)	−230 (10)
	Δ/σ_p	0·8	1·6	1·4	0·8	0·3	2·9	0·4	2·5	0·3
O(3)	Neutron	0	49578 (35)	0·25	1755 (37)	1319 (57)	916 (25)	0	918 (27)	0
	X-ray	0	49740 (30)	0·25	1960 (40)	1270 (40)	940 (20)	0	980 (30)	0
	Δ/σ_p		4·0		3·7	0·7	0·8	0	1·5	0
C	Neutron	23433 (10)	32553 (13)	5279 (8)	527 (15)	918 (33)	306 (11)	−55 (13)	213 (8)	−45 (11)
	X-ray	23470 (10)	32570 (20)	5320 (10)	590 (10)	1080 (20)	336 (9)	30 (10)	192 (9)	10 (10)
	Δ/σ_p	2·6	0·8	3·2	3·5	4·3	2·1	5·1	1·7	3·4
H	Neutron	3497 (27)	40404 (37)	19367 (23)	1333 (39)	2000 (89)	747 (31)	67 (36)	495 (22)	−105 (32)
	X-ray	2200 (400)	42000 (400)	19900 (300)	2400 (600)	1300 (800)	1200 (400)	−1000 (600)	90 (40)	40 (50)
	Δ/σ_p	3·2	4·0	1·8	1·8	0·9	1·1	1·8	9·5	2·5

* The form of the anisotropic temperature factor is $\exp\,[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)]$.

Discussion

The final positional and thermal parameters with standard deviations in parentheses are given in Table 1, where they are also compared with the corresponding values of the parameters found by HI. For the non-hydrogen atoms, the standard deviations of the parameters in the two sets are comparable and the overall agreement between them is excellent. The bond distances and bond angles are compared in Table 2; the O–H distance found by the X-ray method is too low, in agreement with the general trend in X-ray determinations. The final values of F_o^2 and F_c^2 are listed in Table 3.

The structure is as described by CSS and by HI. Numbering of the atoms in this paper corresponds to that of HI. A complete description of the coordination of the water molecule and its hydrogen bonding has been given by CSS and will not be repeated here.

Oxalate ion

The oxalate ion with its two parallel O(1)–C–O(2) planes separated by 0·037 (6) Å is not quite planar, although the sum of the bond angles around the carbon atom is 359·98 (16)°. The values of 1·5698 (17), 1·2559 (14) and 1·2487 (15) Å for the C–C, C–O(1) and C–O(2) bond lengths are in good agreement with the values of 1·5740 (24), 1·2595 (16) and 1·2473 (17) Å obtained by HI. The bond between the carbon atom and the hydrogen-bonded oxygen atom [C–O(1)] is somewhat longer than the other carbon–oxygen [C–O(2)] bond; a still larger difference between C–O(1) and C–O(2) was found by CSS in their earlier, less accurate two-dimensional neutron study and ascribed tentatively to the partial covalent character of the hydrogen bond for which O(1) is the acceptor. HI have also found a difference between the two C–O bonds and in the same direction, though of about twice the magnitude of that found in this investigation.

Motion of the water molecule

The mean-square amplitudes (u_{ij}) of thermal motion of the water oxygen and hydrogen atoms referred to the principal axes of the water molecule (X_1 along the H–H bond, X_2 along the twofold axes and X_3 along the plane normal) are given in Table 4.

The excellent agreement between the neutron and X-ray values of the thermal parameters of the non-hydrogen atoms suggests that these values must be realistic. It is surprising that the mean-square amplitude of the hydrogen atom normal to the molecular plane is considerably less than that of the oxygen atom. In view of this a 'riding' model (Busing & Levy, 1964) is invalid and it is not possible to get a meaningful correction of the O–H bond distance for thermal motion.

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* $R_w = [\omega(F_o^2 - F_c^2)^2/\omega F_o^4]^{1/2}$.

Table 2. *Bond distances (in Å) and angles (in degrees)*

The standard deviations are given in parentheses.

Bond	This work	Hodgson & Ibers	Δ/σ_p
C—C	1·5698 (17)	1·5740 (24)	1·4
C—O(1)	1·2559 (14)	1·2595 (16)	1·7
C—O(2)	1·2487 (15)	1·2473 (17)	0·6
O(1)-C—O(2)	125·89 (9)	126·27 (12)	2·5
C—C—O(1)	116·24 (10)	115·76 (14)	2·8
C—C—O(2)	117·86 (9)	117·96 (13)	0·6
O(3)—O(1)	2·7536 (17)	2·7602 (17)	2·7
O(3)-H	0·9628 (33)	0·80 (3)	5·4
H—H*	1·5541 (49)		
H—O(3)-H†	107·62 (33)	106·9 (32)	0·2
O(1)-O(3)—O(1)	118·82 (9)		
H—O(3)—O(1)	6·74 (14)		
O(3)-H—O(1)	169·66 (22)	165·9 (29)	1·3

* The angle from the *a* axis to the H–H vector measured away from the *c* axis is 46·45° (20).

† The dihedral angle between the H-O(3)-H and O(1)-O(3)-O(1) planes is 4·50° (17).

Table 3. *Observed and calculated structure factors for* $K_2C_2O_4.H_2O$

The nuclear scattering lengths used are (in units of 10^{-12} cm): potassium 0·373, carbon 0·675, oxygen 0·593 and hydrogen −0·378. The four columns in each set contain the Miller indices *h*, *l*, $100F_o^2$ and $100F_c^2$.

h	*l*	$100F_o^2$	$100F_c^2$
k = 0			
−10	2	2388	2316
	4	6851	6377
	6	248	259
−8	2	295	282
	4	604	592
	6	4717	4981
	8	10	02
	10	425	442
−6	2	10107	10277
	4	334	318
	6	150	149
	8	15889	15256
	10	2560	2397
−4	2	389	341
	4	15020	14626
	6	2407	2242
	8	4318	4149
	10	9523	9820
	12	35	00
−2	2	385	345
	4	2930	2883
	6	209	196
	8	6571	6548
	10	156	146
0	2	5842	5797
	4	3210	3300
	6	12803	13310
	8	76	62
	10	545	522
2	0	7843	7792
	2	7035	7674
	4	25	00
	6	1626	1577
	8	2249	2345
	10	47	61
4	0	749	761
	2	68	55
	4	2169	2175
	6	725	696
	8	27	15
6	0	102	78
	2	1340	1311
	4	945	956
	6	2273	2281
8	0	3418	3462
	2	1263	1219
	4	185	186
k = 1			
−9	1	49	53
	2	177	166
	3	177	171
	4	139	120
	5	3515	3765
	6	473	466
	7	1994	1979
	8	142	151
	9	79	69
−7	1	1241	1188
	2	37	32
	4	1399	1396
	5	158	154
	6	3611	3670
	7	6408	6688
	8	167	155
	9	3811	4005
	10	19	23
	11	226	210
−5	1	555	538
	2	931	928
	3	622	609
	4	2834	2756
	5	17	11
	6	119	107
	7	133	131
	8	4499	4078
	9	6264	6785
	10	1500	1437
	11	3470	3585
−3	1	202	177
	2	1444	1377
	3	214	184
	4	1028	968
	5	88	73
	7	107	110
	8	1644	1577
	9	04	03
	10	33	33
	11	2572	2684
−1	1	5231	4946
	2	1277	1185
	3	51	45
	4	10715	9889
	5	3285	3375
	6	61	44
	7	1678	1587
	8	35	32
	9	121	121
	10	2739	2749
	11	136	119
1	0	564	533
	1	2380	2583
	2	2236	2385
	3	3637	4093
	4	565	563
	5	12	00
	6	819	816
	7	4322	4818
	8	1457	1498
	9	2320	2252
	10	24	18
3	0	1952	2004
	1	1934	1936
	2	1400	1393
	3	727	722
	4	93	96
	5	2117	2213
	6	513	481
	7	15	05
	8	895	876
	9	3332	3579
5	0	128	109
	1	213	187
	2	22	11
	3	332	305
	4	160	153
	5	63	56
	6	74	54
	7	565	531
7	0	53	49
	1	2057	2062
	2	641	622
	3	501	440
	4	23	08
	5	05	00
9	0	1416	1417
	1	1336	1300
k = 2			
−8	1	2271	2291
	2	2691	2807
	3	1304	1275
	4	4941	4913
	5	552	513
	6	4508	4859
	7	502	467
	8	1844	1856
	9	183	161
−6	1	02	01
	2	939	939
	3	3369	3448
	4	123	110
	5	1214	1137
	6	7612	7909
	8	718	723
	9	851	830
	10	85	65
−4	1	52	36
	2	390	355
	3	15	14
	4	625	558
	5	5175	5327
	6	2512	2456
	7	317	316
	8	523	484
	9	64	53
	10	150	128
	11	1191	1248
−2	1	3606	3720
	2	9469	8688
	3	680	639
	4	128	98
	5	77	56
	6	238	237
	8	3029	3090
	9	150	157
	10	1552	1559
	11	615	590
0	0	1034	1006
	1	414	335
	2	1370	1337
	3	6290	5849
	4	651	593
	5	905	865
	6	241	182
	7	83	62
	8	960	918
	9	670	698
	10	217	216
2	0	304	260
	1	164	157
	2	6154	7036
	3	983	992
	4	6416	6793
	5	2619	2642
	6	865	867
	7	430	424
	8	2176	2087
	9	13	10
4	0	986	956
	1	1426	1431
	2	3260	3341
	3	229	225
	4	3517	3589
	5	960	951
	6	153	118
	7	1362	1262
	8	2397	2396
6	0	3061	3013
	2	35	24
	3	430	407
	4	12	00
	5	598	554
	6	44	28
8	0	4545	4470
	1	13	10
	2	796	810
	3	385	342
k = 3			
−9	2	1730	1758
	3	579	586
	4	3015	3074
	5	427	409
−7	1	1027	1012
	2	2644	2675
	3	1062	1070
	4	3783	3916
	5	1604	1623
	6	274	210
	7	909	936
	8	368	358
	9	171	167
−5	1	12	03
	2	7745	8311
	3	55	48
	4	84	71
	5	1513	1515
	6	13	13
	7	455	435
	8	35	28
	9	330	332
−3	1	593	606
	2	1220	1246
	3	602	579
	4	1208	1201
	5	28	11
	6	1306	1205
	7	3059	3005
	8	415	393
	9	31	21
	10	699	696
−1	1	242	210
	2	831	780
	3	517	493
	4	180	170
	5	149	145
	6	2573	2630
	7	209	207
	8	30	19
	9	857	862
	10	124	109
1	0	147	133
	1	3284	3575
	2	2775	2927
	3	338	326
	4	2246	2300
	5	69	73
	6	5348	5988
	7	513	456
	8	3324	3385
	9	1062	1066
3	0	4075	3836
	1	176	158
	2	214	199
	3	2770	2946
	4	175	163
	5	299	298
	6	5310	5796
	7	276	272
	8	2686	2662
5	0	1151	1166
	1	11	05
	2	225	211
	3	415	400
	4	3784	3923
	5	245	220
	6	348	336
7	0	2104	2207
	1	97	85
	2	958	934
	3	64	66
k = 4			
−8	1	1418	1380
	2	71	61
	3	5136	5528
	4	150	106
	5	231	237
−6	1	171	190
	2	656	567
	3	2611	2669
	4	2509	2643
	5	4343	4529
	6	1094	1112
	7	135	122
	8	1258	1211
−4	1	399	373
	2	82	72
	3	304	318
	4	2297	2300
	5	466	463
	6	956	968
	7	1841	1852
	9	107	98
−2	1	2268	2361
	2	1044	939
	3	142	140
	4	1253	1241
	5	172	168
	6	214	189
	7	311	308
	8	279	242
	9	779	777
0	0	6126	6228
	1	1809	1869
	3	2336	2443
	4	376	335
	5	671	693
	6	3556	3560
	7	892	932
	8	24	00
	9	35	20
2	0	651	537
	1	2000	2057
	2	545	429
	3	2494	2696
	4	1357	1311
	5	3997	4562
	6	30	00
	7	745	696
4	0	196	190
	1	534	498
	2	5059	5823
	3	04	03
	4	46	05
	5	3036	3069
	6	453	420
6	0	2049	2099
	1	133	124
	2	905	920
	3	247	223
k = 5			
−7	3	270	270
−5	1	1077	1067
	2	205	186
	3	1961	2007
	4	636	616
	5	1629	1682
	6	2299	2249
	7	102	69
−3	1	2826	2937
	2	1600	1577
	3	216	197
	4	1348	1278
	5	1764	1707
	6	744	712
	7	743	749
−1	1	89	81
	2	519	495
	3	2237	2218
	4	6774	6827
	5	731	714
	6	320	309
	7	1118	1069
1	0	832	845
	1	332	334
	2	4835	5205
	3	446	396
	4	818	813
	5	1407	1348
	6	1073	1048
	7	08	03
3	0	2052	2013
	1	1229	1296
	2	84	66
	3	27	00
	4	416	377
	5	13	02
5	0	12	00
	1	4908	5119
	2	769	736
	3	780	849
k = 6			
−4	1	184	165
	2	1076	1064
	3	131	105
−2	1	114	99
	2	4442	4499
	3	488	467
	4	184	118
	5	38	16
0	0	8044	7917
	1	953	1016
	3	1233	1314
	5	274	278
2	0	919	788
	1	73	75
	2	2112	2026
	3	458	405
	4	1523	1455
4	0	2192	2312
	1	25	20

Table 4. *Mean-square amplitudes of* O(3) *and* H, *in* Å²

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(3)	0·01988	0·02566	0·06434	0	0·00038	0
H	0·02853	0·03891	0·04428	0·00429	0·00290	0·00003

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