Refinement of the Structure of D-Tartaric Acid by X-ray and Neutron Diffraction*

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The crystal structure of D-tartaric acid determined by Stern & Beevers [Acta Cryst. 3, 341 (1950)] was refined by using three-dimensional intensity data (Cu K α) collected on a computer-controlled diffractometer operated by an IBM 1620 machine under a stored data collection program. The crystal-lographic data are: $a = 7.71_5 \pm 0.003$, $b = 6.00_4 \pm 0.003$, $c = 6.23_1 \pm 0.003$ Å, $\beta = 100.1 \pm 0.1^\circ$, with space group $P2_1$. The parameters were refined by using a full-matrix least-squares program; all the hydrogen atoms were located by a difference Fourier synthesis and refined by the least-squares method. The results of this refinement are compared with a similar result obtained from neutron diffraction data.

The molecule is made of two $-C^*H$. OH. COOH parts, each part consisting of a planar carboxyl group and a tetrahedral asymmetric carbon atom; the α -hydroxyl oxygen atom is also situated close to the carboxyl plane. There is a slight but interesting difference in the overall shape of these two parts; the angle between the planes of the carboxyl groups in these two parts is 54.6°.

The molecules are held in the structure by a three-dimensional network of O-H...O hydrogen bonds of usual strength. The hydrogen bond scheme in the crystal was unequivocally determined by the present analysis.

Introduction and experimental

The crystal structure of D-tartaric acid was determined some fifteen years ago by Stern & Beevers (1950) by elegant deconvolution of the Patterson function. Although their results revealed an interesting network of O-H...O hydrogen bonds and the general shape of the molecule, no attempt was made to refine the structure enough for the bond lengths and angles of this important oxyacid to be discussed with confidence. The present paper[†] deals with the refinement of the structure based on three-dimensional X-ray intensity data obtained by counter measurement on CCXD, a computer-controlled diffractometer (Cole, Okaya & Chambers, 1963) and on two projections of neutron diffraction data.

A single crystal of D-tartaric acid was ground into a sphere of approximately 0.5 mm in diameter and mounted on a General Electric Goniostat; the *b* axis of the crystal was set almost parallel to the φ axis of the Goniostat. Full three-dimensional intensity data within the range of sin $\theta \le 0.96$ for Cu K α (Ni-filtered) were then recorded by CCXD operated under a stored data-collection program in an IBM 1620 computer (Okaya, 1964). For each reflection, the crystal setting and the accuracy of the equipment were first tested by step-scanning around the ω and χ axes and then the

integrated intensity data were recorded by $(\theta - 2\theta)$ step scanning. While the ω setting was studied, the maximum and minimum counts were recorded and if the difference between them did not exceed the statistical fluctuation, the reflection was then treated as nonobserved. Out of 647 reflections recorded by the diffractometer there were fewer than five such reflections. The intervals between the steps were so chosen that the first three and the last three out of the total of twenty-four θ -2 θ steps for each reflection could be taken to represent the background at the 2θ value of the reflection; they are 0.09° for 2θ less than 70° , 0.11° for $70^\circ \le 2\theta < 140^\circ$ and 0.13° thereafter. The integrated intensity data were calculated from data on the 2θ step scan as a time-shared program on the IBM 1620 computer. The observed F values were corrected for the absorption effect ($\mu r = 0.8$). The crystal belongs to the monoclinic system with space group $P2_1$, and the cell dimensions measured on the diffractometer are a = $7.71_5 \pm 0.003$, $b = 6.00_4 \pm 0.003$, $c = 6.23_1 \pm 0.003$ Å, $\beta =$ $100.1 \pm 0.1^{\circ}$; these values agree well with the values given in the previous paper.

Starting from the atomic coordinates given by Stern & Beevers the refinement of the structure was made in the usual manner by using a full-matrix least-squares program on an IBM 7094 computer. After several cycles of refinement with only the contribution of the non-hydrogen atoms, the positions of the hydrogen atoms were obtained by an $F_o - F_{(c+o)}$ synthesis. The atomic coordinates thus obtained for the hydrogen atoms were then subjected to the least-squares treatment; only isotropic temperature factors were used for the hydrogen atoms. The atomic coordinates, their standard devi-

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[†] A short account of this work was given at the 1964 annual meeting of the American Crystallographic Association (Bozeman, Montana).

Table $1(a)$.	Atomic	coordinates	in fractions	of cell	edges d	and thei	r estimated	standard	deviations
	Standard	deviations ar	e in units of	10−4 Å ·	for C an	nd O, and	1 10-3 Å for	H atoms.	

Atoms	x	$\sigma(x)$	У	$\sigma(y)$	z	$\sigma(z)$
O(1)	0.3382	26	0.0343	25	0.5102	21
O(2)	0.4296	27	-0.0325	27	-0.0219	23
O(3)	0.6084	24	0.0020	26	0.3005	22
O(4)	0.3063	25	0.4173	25	0.2335	22
O(5)	-0.0765	28	0.0792	32	0.1790	28
0(6)	-0.0217	27	0.4072	33	0.3496	29
C(1)	0.2941	33	0.0159	34	0.2832	30
C(2)	0.4640	34	-0.0068	35	0.1911	31
C(3)	0.2003	32	0.2273	33	0.1811	29
C(4)	0.0219	35	0.2528	35	0.2496	31
H (1)	0.241	36	-0.116	36	0.236	29
H(2)	0.182	33	0.207	35	0.028	31
H(3)	0.253	93	0.037	85	0.555	72
H(4)	0.515	81	-0.046	69	-0.090	57
H(5)	0.342	54	0.438	64	0.367	56
H(6)	-0.182	85	0.049	67	0.203	54
Neutron	diffraction res	ults				
H(1)	0· 2 101	21	-0.1385	28	0.2256	34
H(2)	0-1699	15	0.1993	45	0.0032	18
H(3)	0.2359	14	-0.0177	35	0.5724	19
H(4)	0.5360	15	-0.0427	26	-0.0931	23
H(5)	0.3470	21	0.4329	46	0.3900	22
H(6)	-0.1928	12	0.0725	27	0.2315	24

Table 1(b). Anisotropic temperature factors

The β 's are used in the expression exp $\{-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)\}$

	β_{11}	β22	β ₃₃	β_{12}		β_{13}	β_{23}
D(1)	0.00695	0.02355	0.01149	-0.000	006	0.00568	0.00578
D(2)	0.00725	0.02713	0.01412	0.003	372	0.00814	- 0.00598
D(3)	0.00578	0.02374	0.01634	0.002	233	0.00606	0.00881
D(4)	0.00648	0.01708	0.01018	-0.004	400	0.00343	0.00116
D(5)	0.00640	0.02688	0.02362	-0.002	763	0.01023	-0.01724
D(6)	0.00920	0.02641	0.02492	-0.000	004	0.01310	-0.01773
C(1)	0.00510	0.01411	0.01266	0.002	222	0.00388	0.00336
C(2)	0.00665	0.01161	0.01502	-0.000	024	0.00557	0.00333
C(3)	0.00442	0.01532	0.00954	-0.000	009	0.00284	0.00067
C(4)	0.00496	0.01802	0.01203	0.00	164	0.00052	-0.00043
	Isotro	pic temperature	factors for h	ydrogen at	oms, in 1	0 ⁻¹⁶ cm ²	
	H(1)) H(2)	H(3)	H(4)	H(5)	H(6)	
	$-1\cdot3$	$-1\cdot 2$	6.4	4.4	1.8	5.3	

ations and thermal parameters after five cycles of further refinement are shown in Table 1. The final conventional error index $R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$ is 0.043 including unobserved reflections (as $F_0 = 0$). Comparison between the observed and calculated structure factors is shown in Table 2(a). The atomic scattering factors used in the computation were those values listed in International Tables for X-ray Crystallography (1962). The parameter shifts at the last stage were negligible in comparison with the standard deviations. The weighting scheme used in the refinement procedure was: $\sqrt{w} = 1.0$ for $F_{obs} < 10.0$, $\sqrt{w} = 10.0/F_{obs}$ for $F_{obs} \ge$ 10.0, and zero weight for unobserved reflections. As shown in Table 1, the isotropic temperature factors of two hydrogen atoms have become negative; although it is doubtful that any real significance can be attributed to such a result, one notices that these two hydrogen atoms are those bonded to the carbon atoms.

Table 1(c). Anisotropic temperature factorsfrom neutron diffraction*

	β ₁₁	β22	β33	β_{12}	β ₁₃
O(1)	0.0082	0.0157	0.0054	0.0021	0.0039
O(2)	0.0055	0.0164	0.0083	0.0037	-0.0009
O(3)	0.0060	0.0174	0.0178	0.0017	0.0048
O(4)	0.0047	0.0121	0.0142	-0.0019	0.0045
0(5)	0.0049	0.0214	0.0277	-0.0012	0.0028
O(6)	0.0028	0.0182	0.0252	0.0037	0.0056
C(1)	0.0034	0.0137	0.0057	0.0112	0.0005
C(2)	0.0043	0.0090	0.0094	-0.0013	0.0001
C(3)	0.0049	0.0113	0.0046	0.0013	0.0009
C(4)	0.0021	0.0157	0.0096	0.0000	0.0020
H(1)	0.0136	0.0142	0.0232	-0.0022	0.0100
H(2)	0.0089	0.0288	0.0125	-0.0033	0.0001
H(3)	0.0080	0.0298	0.0225	-0.0004	0.0023
H(4)	0.0079	0.0146	0.0308	-0.0016	0.0085
H(5)	0.0192	0.0377	0.0155	0.0014	0.0008
H(6)	0.0068	0.0230	0.0343	0.0014	-0.0019

* Since only hol and hk0 data were used, no β_{23} was calculated.

Table 2(a). Observed and calculated structure factors from X-ray diffraction ($\times 10$)

н 0	K000000	L 12345	FOBS 10 324 36 198 34	FCAL 17 342 35 196 28	н 8	к 1 1	L 3 4 0 1	FOBS 32 49 14 29	FCAL 33 50 16 27	H 7	к 3333		FOBS 57 9 28 14	FCAL 58 9 29 15	н" 5	x L 6 1 6 2 6 3 6 0	FOBS 20 18 32 9 21	FCAL 18 17 31 8	н 7	K L 1 -3 1 -4 1 -5 1 -6	FOBS 61 44 10 36	FCAL 61 46 7 35	н 8	K L 3 -3 3 -4 3 -5 3 -1 3 -2	F085 27 40 21 51	FCAL 26 41 22 55 7
ı	00000	0123	103 242 239 96	107 239 225 90	9 0	1	2 3 0 1	27 42 45 63	29 41 44 57	0 0	33 4444	1 2 0 1 2	19 25 170 59 46	19 25 173 61 45	0	6 2 7 1 7 2 7 3	48 21 28	45 20 29		1 -2 1 -3 1 -4 1 -5 1 -6	26 25 18 46 109	19 28 16 45 103	1	3 -3 3 -4 4 -1 4 -2 4 -3 6 -6	67 20 27 39 29	64 24 26 42 26
2	0000 00	4567 01	0 50 14 137	66 0 47 13 132 173		2222222	234567	139 107 48 58 11	138 102 50 58 14	1	1444 44	3 4 5 6 0	59 79 45 22 125 148	59 81 46 21 124 146	z	7 0 7 1 7 2 7 0 7 1 7 2	10 52 35 1.3 3	13 50 33 12 1 16	9	1 -1 1 -2 1 -3 1 -4 1 -1 1 -2	45 6 55 30 67	43 6 59 56 27 66	2	4 -5 4 -6 4 -1 4 -2 4 -3	37 47 126 100 64	38 50 126 103 64
	0000000	234567	7C 44 118 18 40 26	62 42 119 16 40 27	1	2 2 2 2 2 2 2 2 2 2 2 2	012345	390 227 70 116 126 53	345 256 53 119 126 65		44444	23456	50 49 39 51 40	41 49 39 51 39	3 1	7 0 7 1 0 -1 0 -2	8 41 112 93	10 38 119 93	1	1 -3 2 -1 2 -2 2 -3 2 -4	46 106 58 75 136	54 105 57 68 135	3	4 -4 4 -5 4 -6 4 -1 4 -2	54 50 38 64 53	57 51 38 54 54
3	00000	0 1 2 3 4	340 38 82 9 117	316 37 79 5 117	2	2 2 2 2 2 2	6 7 0 1 2	13 30 80 205 106	5 31 72 211 107	2	444444	012345	22 40 32 42 30 24	11 37 31 42 29 23	_	0 -3 0 -4 0 -5 0 -6 0 -7	181 178 60 20 45	188 185 60 6 43	z	2 -5 2 -6 2 -7 2 -1 2 -2	30 17 55 94 152	29 19 56 98 151	4	4 -3 4 -4 4 -5 4 -6 4 -1	38 31 81 38 52	42 31 83 39 54
4	00000	5 6 0 1 2	71 28 27 9 37	68 26 35 8 28		22222	34567	75 131 49 82 9	73 136 48 89 15	3	4 4 4 4 4 4 4	6 0 1 2 3	22 36 44 52 36	25 16 38 52 34	2	0 -1 0 -2 0 -3 0 -4 0 -5 0 -6	15 78 188 30 80 2	14 78 185 28 78 1		2 -3 2 -4 2 -5 2 -6 2 -7	123 45 97 52 17	121 46 100 49 16	E	4 -2 4 -3 4 -4 4 -5 4 -6	74 45 80 51 52	77 45 82 49 53
5	0000	3456 01	107 45 11 36	105 43 9 32	,	2222222	0123456	189 33 131 64 91 49	175 28 133 65 91 49	4	14 4444	5 0 1 2 3	50 51 33	49 51 31	3	0 -2 0 -3 0 -4 0 -5 0 -6	117 118 59 105	120 118 59 106 31	ſ	2 -2 2 -3 2 -4 2 -5 2 -6 2 -7	58 59 81 106 65 17	58 53 78 106 64	,	4 -2 4 -3 4 -4 4 -5 4 -6	60 41 31 12 36	59 39 31 13 40
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8	00000	1234	94 46 152 48 75	88 48 149 45 75	6	2 2 2 2 2 2 2 2	4 5 0 1 2	17 8 57 89 66	18 10 55 90 66	ז ס	.444	0 1 2 1	47 37 12 146	45 36 10 147	6	0 -4 0 -5 0 -6 0 -7 0 -1	60 26 2 8 217	62 25 2 6 227	6	2 -6 2 -7 2 -1 2 -2 2 -3	33 29 226 70 46	32 28 237 68 46	1	5 -2 5 -3 5 -4 5 -5	29 85 48 21	30 85 49 22
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Z	1	767 012	17 59 14 137 90 79	14 59 13 138 94 82	ı	3333	0 1 2 3 4	56 129 58 59 54	52 128 69 58 56	4	555555	1 2 3 4 0	68 11 61 24 44	65 6 59 23 43	1	0 -3 0 -4 1 -1 1 -2 1 -3	23 0 190 68 72	24 3 191 69 71		3 -3 3 -4 3 -5 3 -6 3 -7	103 77 47 34 36	103 91 49 35 37	6	5 -3 5 -4 5 -5 5 -1 5 -2	33 13 55 25 34	32 11 54 23 32
	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	34567	57 147 27 27 10	53 149 24 27 11	2	3333	5 6 0 1 2	87 41 201 44 94	88 41 187 46 94	5	5555	1 2 4 0 1	13 17 33 31 31	14 20 32 32 32	2	1 -4 1 -5 1 -6 1 -7 1 -1 1 -2	124 94 12 8 120	124 97 13 9	2	3 -1 3 -2 3 -3 3 -4 3 -5 3 -6 3 -6	68 139 50 25 58 47 29	67 140 63 24 60 49 27	7	5 -3 5 -4 5 -1 5 -2	24 6 24 17	24 7 25 17
3		0123456	129 47 127 37 93 54 34	126 50 131 35 93 52 37	3	33333333	3 4 5 6 0 1	150 97 74 6 143 126	137 97 77 7 137 130	6	5 5 5 5	2 3 0 1 2	24 21 63 38 51	61 35 52		1 -2 1 -3 1 -4 1 -5 1 -6 1 -7	197 76 44 59 30	209 80 41 58 29	3	3 -1 3 -2 3 -3 3 -4 3 -5	142 120 25 47 55	142 120 25 43 56	2	5 -2 6 -3 6 -4 6 -1 6 -2	40 17 0 45 7	40 16 1 44 7
4	1 1 1 1	01234	87 259 96 49 63	82 263 94 47 72	,	3333	23456	50 57 52 62 19	52 59 51 63 22	7 9	5 6 6 6 6 6	0 1 2 3 4	53 40 39 35 29	54 40 37 30 28	3	1 -1 1 -2 1 -3 1 -4 1 -5 1 -6 1 -7	244 190 50 88 54	236 184 90 47 88 62 46	4	3 -6 3 -7 3 -1 3 -2 3 -3 3 -4	47 14 71 85 56 53	48 16 74 89 57 64	3	6 -3 6 -4 6 -1 6 -2 6 -3 6 -4	37 33 42 58 15 54	37 34 43 59 13 54
5	1	5 6 0 1 2 3	77 26 72 69 62 42	77 30 71 68 60 42	4	3333	0 1 2 3 4 5	68 79 28 16 60 47	74 28 12 61 44	1	66666	1 0 1 2 3 4	86 19 50 18 16	88 17 50 19 16	4	1 -1 1 -2 1 -3 1 -4 1 -5	159 48 70 75 173	162 48 68 74 181	5	3 -5 3 -6 3 -1 3 -2 3 -3	77 21 114 83 70	76 19 119 85 71	4	6 -1 6 -2 6 -3 6 -4	20 14 5 10	21 16 10
6	1	245 012	10 46 59 114 47	4 47 53 118 49	5	33333	0 1 2 3 4 5	107 90 57 73 25 28	114 92 65 82 21 30	2	5 6 6 6 6	0 1 2 3 4	31 17 7 22 9	36 14 6 22 9	5	1 - 6 1 - 7 1 - 1 1 - 2 1 - 3	51 13 76 30 155	48 10 80 30 160	6	3 -4 3 -5 3 -6 3 -1 3 -2	89 46 21 22 131	90 45 22 21 134	5 6 ,	6 -1 6 -2 6 -3 6 -1	31 16 10 19	30 16 11 25
7	1	3 4 5 0 1 2	48 11 26 107 70 73	48 11 28 109 69 72	ė	3	0 1 2 3 4	128 26 71 10	133 26 72 10 14	٦ 4	6666.6	0 1 2 3 0	38 21 10 9 21	40 20 10 10	6	1 - 4 1 - 5 1 - 6 1 - 7 1 - 7 1 - 1 1 - 2	14 14 43 41 53 22	11 12 40 39 50 16	7	3 -4 3 -5 3 -6 3 -1 3 -2	48 11 40 37 33	46 12 40 41 32	2	7 -2 7 -3 7 -1 7 -2	5 18 32 15	3 19 36 12

Table 2(b). Observed and calculated structure factors from neutron diffraction (\times 100)

7 0 262 246 9 4 0 170 -5 9 0 19 -12 3 161 164		12 0 12 129 0 6 137 140 9 2 0 67 -5 5 0 70 -12 2 233 275		5 5 511 514 11 5 105 96 6 4 97 117 9 1 178 141 5 7 0 7 -19 1 0 89					5 0 100 7/ 11 0 0 1/ 6 0 0 59 6 1 151 118 -5 4 569 579 -11 4 297 257	b 0 0 0 1 1 2 5 4 20 5 6 5 5 7 1 2 1 0 5 6 7 6 7 0 7 0 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	P3 20 3 2 3 6 7 1 5 13 22 20 13 20 4 5 6 6 7 7 1 7 1 6 7 2 3 22 20 13 20 1 20 21 20 13 20 14 20 1 20 10 10 10 10 10 10 10 10 10 10 10 10 10	h.0.20000000000000000000000000000000000	Ro 2 4 6 6 5 7 0 2 4 0 0 2 7 4 5 1 2 5 7 5 7 5 4 4 5 7 5 7 5 4 5 6 7 5 1 1 2 5 7 5 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7	Γ 200 0 1173 223 0 1173 223 2113	1011111111111111111111111111111111111	k ¹ :136 D.06 D.06 D.06 D.112 P. 67 15 1721 9929 13 27 6 5 5 5 5 22 72 73 72 9 - 9 - 9 - 9 - 9 - 9 - 9 - 9 - 9 - 9
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226 & -77 & 1 & 245 & 226 & 217 & -42 & 1 & 0 & 110 & -77 & 5 & 223 & 221 & 210 & 117 & -77 & 7 & 6 & 223 & 221 & 210 & 117 & -77 & 7 & 6 & 223 & 221 & 210 & 117 & -77 & 7 & 6 & 223 & 221 & -77 & 5 & 255 & 516 & -77 & 6 & 235 & 226 & -77 & 6 & 235 & -77 & 6 & 235 & 226 & -77 & 19 & 9 & 77 & -7 & 6 & 235 & 226 & -77 & 19 & 9 & 177 & -76 & -7 & 7 & 29 & 99 & 26 & -77 & 19 & 9 & 177 & -76 & -76 & 10 & 0 & 127 & -76 & -76 & 100 & 177 & -76 & -76 & -76 & 110 & -77 & 6 & 236 & -76 & 110 & -77 & -76 & 235 & 226 & -76 & -76 & 110 & -77 & -76 & -76 & -76 & -77 & -7$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{c} 1 & 7 & 7 & 50 & 6 & 6 & 109 & 106 & 1 & 2 & 234 & 221 & 0 & 2 & 555 & 516 & -1 & 6 & 160 & 134 & -7 & 7 & 4 & 50 & 65 \\ 1 & 0 & 7 & 74 & 6 & 7 & 826 & 287 & 1 & 5 & 224 & 129 & 0 & 1 & -7 & 5 & 825 & 221 \\ 2 & 0 & 556 & 346 & 7 & 0 & 246 & 248 & 1 & 5 & 112 & 1120 & 0 & 6 & 556 & 266 & -2 & 1 & 0 & 110 & -7 & 6 & 825 & 221 \\ 2 & 0 & 105 & 7 & 1 & 469 & 449 & 1 & 6 & 1450 & 146 & -1 & 7 & 0 & 99 \\ 2 & 1 & 276 & 276 & 7 & 1 & 469 & 449 & 1 & 6 & 1450 & 146 & -1 & 1 & 1317 & 566 & -2 & 1 & 86 & 556 & 556 & -7 & 9 & 90 & 0 & 13 \\ 2 & 0 & 105 & 7 & 1 & 469 & 449 & 1 & 6 & 1450 & 146 & 1 & 1 & 1317 & 556 & -7 & 6 & 235 & 229 \\ 2 & 1 & 276 & 276 & 7 & 1 & 469 & 449 & 1 & 6 & 1450 & 146 & 2 & 7 & 166 & 190 & -2 & 6 & 50 & 0 & 27 \\ 2 & 5 & 1160 & 157 & -7 & 7 & 2 & 541 & 326 & 1 & 1 & 0 & 511 & 1 & 237 & 256 & -2 & 1 & 4 & 145 & 1150 & -7 & 9 & 0 & 0 & 13 \\ 2 & 5 & 100 & 157 & 7 & 1 & 2 & 541 & 326 & 1 & 1 & 0 & 51 & 1 & 2 & 526 & 251 & -7 & 6 & 255 & 229 \\ 2 & 5 & 1113 & 16 & 7 & 7 & 1239 & 42 & 0 & 0 & 511 & 2 & 5 & 256 & 56 & 0 & 0 & 277 & -4 & -4 & -5 & 0 & 0 & 67 \\ 2 & 0 & 112 & 113 & 6 & 7 & 0 & 200 & 201 & 2 & 2 & 1 & 0 & 0 & 52 & 5 & 5 & 5 & 5 & 6 & 0 & 0 & 277 & -4 & -4 & -5 & 0 & 0 & 56 \\ 2 & 0 & 112 & 113 & 6 & 0 & 200 & 201 & 2 & 2 & 0 & 0 & 55 & 5 & 5 & 1 & 477 & 477 & 477 & -77 & 272 & 272 & 272 \\ 2 & 0 & 112 & 113 & 6 & 0 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To confirm and refine the hydrogen positions, two (h0l and hk0) zones of neutron diffraction data were taken by the (θ -2 θ) scan method. Because of an accident, the h0l data had to be collected on two different crystals.

Refinement with the neutron data was carried out by means of least-squares analysis, starting with the X-ray parameter set. The carbon and oxygen positional parameters (as given in Table 1) were held constant. Refinement was carried out on all the hydrogen parameters, the carbon and oxygen temperature parameters and three scale factors. The refinement proceeded with isotropic temperature factors, followed by six rounds of least squares with anisotropic temperature factors. The weights are $\sqrt{w} = 1/(\sigma(F) + 0.025F)$ where $\sigma(F)$ is



Fig. 1. Bond distances from X-ray diffraction.



Fig. 2. Bond angles from X-ray diffraction.



Fig. 3. The conformation of the molecule. C(3) is above the plane of carboxyl I by 1.323 Å. The deviations (Å) of atoms from the group plane are also shown for each part. Note the difference between the O(1)-O(3) and O(4)-O(6) distances.

based on counting statistics. Non-observed reflections were given zero weight. The resultant parameters are given in Table 1. The final R was 0.059 (omitting unobserved reflections). The observed and calculated structure factors (multiplied by 100) are given in Table 2(b). Both X-ray and neutron least squares minimized $\Sigma w(F_o - F_c)^2$.

Discussion

Bond distances and angles have been calculated from the X-ray atomic coordinates in Table 1(a); they are shown in Figs. 1 and 2 respectively. Around C(1) and C(2), only the average of the three angles involving the hydrogen atom is shown for each atom. The results of the hydrogen determination by neutron diffraction are given in Table 4 for comparison. The molecule consists of two $-C^*H$. OH. COOH parts, each part containing a planar carboxyl group and a tetrahedral $-C^*H$. OH – configuration. In each of the two parts, the α -hydroxyl oxygen atom stays close to the carboxyl plane; it is interesting to note that a similar situation also exists in the mesotartaric ion (Kroon, Peerdeman & Bijvoet, 1965). It may be seen from the Figures that although these two parts are similar in over-all shape, there exist slight differences. The carboxyl group of part I, C(1), C(2), O(2), and O(3), is less planar than that of part II and the O(1)–O(3) distance is much shorter than the corresponding O(4)–O(6) distance in part II; this is mainly due to the large C(4)–C(3)–O(4) angle. It is interesting to notice that such seemingly

Table 3. Hydrogen-bond system in the D-tartaric acid crystal

The second values for	or H–O, H···O and an	gles around hydrogen ato	oms are those obtained by	the neutron study
Bonds	A	В	С	D
From to of Hydrogen involved Distances (Å) O-H HO	O(1) O(6) $(-x, -\frac{1}{2}+y, 1-z)$ H(3) 2.839 0.8, 0.98 2.1, 1.86	O(4) O(3) $(1-x, \frac{1}{2}+y, 1-z)$ H(5) 2.909 0.8, 0.97 2.1, 1.95	O(2) O(4) $(1-x, -\frac{1}{2}+y, -z)$ H(4) 2.633 0.9, 1.00 1.8, 1.64	O(5) O(3) (-1+x, y, z) H(6) 2·707 0·9, 1·00 1·9, 1·71
Angles around hydrogen atoms	157°, 171°	172°, 169°	176°, 172°	152°, 168°
Separations listed by Stern & Beevers (1950)	O(1)-O(10)	O(7)–O(5)	O(4)-O(7)	O(5)-O(9)



Fig. 4. Projection of the structure along the *a* axis. Hydrogen bonds are shown by chain lines. For the four hydrogen bonds, *A* to *D*, see Table 3. *Bond *D* is formed from O(5) to O(3) of the tartrate ion which is one unit cell below in the *a* direction.

equivalent groups start to take slightly different configurations. It is difficult to decide whether this asymmetry of the molecule is due only to differences in the hydrogen-bond formation or is inherent in the tartrate ion itself. The question might be answered by accurate crystal structure analyses of various crystals with tartaric as well as mesotartaric ions. The planes of the two parts make an angle of 54.6° (Fig. 3). In these two carboxyl groups, which retain their protons, there are two distinct C-C-O angles; a C-C-OH angle of around 110° and a wider C-C=O angle of about 125°. The differences between the C-O and C=O distances are about 0.1 Å for the two groups. This situation is found in many crystal structures of molecules with carboxyl groups; when a carboxyl group loses its proton, the two C-C-O angles become almost equivalent and are about 118°. The dependence of the shape of carboxyl groups upon the state of ionization has been exhibited by various acid salts of dicarboxylic acids, e.g. ammonium hydrogen D-tartrate (Bommel & Bijvoet, 1958), dipotassium ethylenetetracarboxylate (Kumra & Darlow, 1965), potassium acid phthalate (Okaya, 1965) and others.

The structure consists of a complicated network of $O-H \cdots O$ hydrogen bonds. Figs. 4 and 5 are the structures projected down the *a* and *b* axes, showing the hydrogen-bond scheme which is essentially the same as that given by Stern & Beevers (1950); the fifth contact given in their paper is ruled out as a hydrogen bond. Donohue (1952) discussed the hydrogen-bond system in the crystal and proposed two possible schemes; the difference between these two schemes is

based on the position of the proton on carboxyl I, *i.e.* the choice between O(2) and O(3) for the hydroxyl oxygen of this group. The difference in the two C-C-O angles given by Stern & Beevers indicates conclusively the position of the proton without locating its position; therefore, the hydrogen-bond system could have uniquely been assigned (Scheme A of Donohue) if the relation between the shape of carboxyl groups and proton positions had been fully understood at that

Table 4.

(a) Bond distances involving hydrogen atoms from coordinates determined by the neutron study

	O(1)-H(3) O(2)-H(4) O(4)-H(5) O(5)-H(6)	$\begin{array}{c} 0.98 \pm 0.02 \text{ \AA} \\ 1.00 \pm 0.02 \\ 0.97 \pm 0.02 \\ 1.00 \pm 0.02 \end{array}$
	C(1)-H(1) C(3)-H(2)	1.15 ± 0.03 1.14 ± 0.02
(b)	Bond angles around oxygen atoms	
	C(1)-O(1)-H(3) C(2)-O(2)-H(4) C(3)-O(4)-H(5) C(4)-O(5)-H(6)	$108 \pm 3^{\circ}$ 115 ± 3 112 ± 3 115 ± 4
	around C(1)	
	H(1)-C(1)-O(1) H(1)-C(1)-C(2) H(1)-C(1)-C(3)	114 ± 3 107 ± 3 110 ± 3
	around C(2)	
	H(2)-C(3)-O(4) H(2)-C(3)-C(1) H(2)-C(3)-C(4)	111 ± 4 107 \pm 4 105 \pm 4



Fig. 5. Projection of the structure along the *b* axis showing the hydrogen bond scheme. Figs. 4 and 5 have been drawn on an IBM 1627 X - Y plotter based on calculation done on an IBM 7094 machine (Okaya, 1966).

time. As is evident from the Figures and Table 3, the two carboxyl groups do not have identical surroundings; possible implications of the situation in relation to the asymmetry of the tartrate ion have been discussed in the previous paragraph.

The neutron diffraction data confirm the hydrogenbonding scheme deduced from the X-ray data. As has been noted in previously determined compounds, the values of the O-H and C-H bond distances (Table 4) from neutron data are about 0.15 Å longer than those determined from X-ray data (Fig. 3). This discrepancy is due to an inadequate description of the X-ray scattering from a bound hydrogen atom (for form factors of bonded hydrogen atoms, see Stewart, Davidson & Simpson, 1965). The bond distances derived from the neutron diffraction data are closer to accepted values. In addition, the two O-H-O hydrogen bond angles (Table 3) which were found to be less than 160° by X-rays are shown to be closer to 170°. We note that the expected inverse relationship between O-H and O-O distance in hydrogen bonds seems to hold in this compound, although the accuracy of the determination is not really high enough to be certain about it.

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X-ray Diffraction Study of Cold-worked α-CuIn and α-CuSn Alloys

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X-ray diffraction line profiles from filings of copper-indium and copper-tin alloys in the solid solution range were recorded by a Geiger counter X-ray diffractometer. Information regarding stacking fault densities α and β was obtained from peak-position and peak-asymmetry measurements and both parameters were found to increase with increasing solute concentration. The same general behaviour of the stacking-fault parameter α with respect to solute concentration was observed in the two systems and α increases in the order Cu–In, Cu–Sn. The broadening of the powder peaks was studied by Fourier analysis of line shapes, and the anisotropic values of the effective particle sizes $[D_e]_{nkl}$ and the root mean squared strains $[\langle e_L^2 \rangle]_{hkl}^{1/2}$ were obtained in all cases. The measured effective particle sizes are primarily a consequence of deformation and twin faulting. A fair agreement was observed for the compound fault probability $(1 \cdot 5\alpha + \beta)$ obtained by two different methods.

1. Introduction

Cold working or plastic deformation of metals and alloys has been found to produce appreciable changes in the intensity distribution of diffracted X-rays. The changes in position, shape and width of X-ray powder diffraction line profiles from cold-worked metals and alloys are evidences of microstructural changes in the materials. The earlier X-ray studies in this field were usually confined to measurements of line-widths and it was suggested that line broadening is produced either by lattice strains or by lattice strains and small particle size simultaneously (Greenough, 1952). It was Barrett (1950), who first suggested that plastic deformation of face-centred cubic metals may introduce stacking faults on the (111) planes. Subsequently, Paterson (1952), Warren & Warekois (1955), and Wagner (1957*a*, *b*) developed the effects of deformation and twin stacking faults on the diffraction profiles of f.c.c. structures. If the normal stacking sequence of (111) planes is *ABCABC*, then a deformation fault is a break in this sequence *ABC'BCABC* where the prime indicates the fault plane. A reversal in the sequence *ABCACBA* represents a twin fault. Deformation faults give rise to a symmetrical broadening and peak-shift, while twin faults produce an asymmetrical broadening and a negligibly small peak-shift. In addition to the broadening due to faulting, the peaks of cold-worked