

layer is rotated through  $-120^\circ$  relative to the other layers.

These arrangements are shown diagrammatically in Fig. 6, where each circle represents a layer and the single, double and triple arrow-heads denote the  $a_1$ ,  $a_2$  and  $a_3$  axes. The arrows between the circles indicate the sequence of rotations and displacements. These results suggest that displacements along the  $a_2$  axis are more common than those along the  $a_1$  axis. The evidence is perhaps too slender to justify drawing a general conclusion that the layer displacements tend to avoid the direction of the symmetry plane, but it certainly is a feature of the results so far obtained. It may also be significant that the two crystals of type B which were examined showed some streaking along Debye-Scherrer lines indicating misalignment of mosaic fragments; such streaking was not obtained with crystals of types A, C and D.

A strong point in favour of the idea of rotations comes from the disordered structures (cf. Fig. 3 (b) and (f)), which, when described in terms of fixed axes, are disordered with respect to  $b$  but ordered with respect to  $a$ , a state of affairs which scarcely seems natural. In terms of rotations, however, it can be said that the layers are randomly rotated by multiples of  $120^\circ$ , with accompanying  $-\frac{1}{3}a$  displacements. By analogy with the ordered structures, the displacements may, in any one crystal, be along a particular  $a$  axis of the layer. Whether or not this is the case cannot yet be stated,

but further work is in progress on the disordered structures.

Finally, we wish to thank all who have supplied us with specimens of chlorites, especially Dr F. A. Bannister of the British Museum and Monsieur G. Vigneron of Louvain. We thank also the Department of Scientific and Industrial Research for a grant to one of us (G. W. B.) for the provision of a research assistant, and to another (B. M. O.) for a maintenance grant, and the Government Grant Committee of the Royal Society for funds to purchase special equipment.

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## The Crystal Structure of Potassium Sodium *dl*-Tartrate Tetrahydrate, $\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$

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The crystal structure of potassium sodium *dl*-tartrate tetrahydrate has been studied by X-rays, using the Weissenberg and rotation method ( $\text{MoK}\alpha$ ,  $\lambda = 0.710 \text{ \AA}$ ). The lattice has the dimensions:

$$a_0 = 9.80 \text{ \AA}, \quad b_0 = 9.66 \text{ \AA}, \quad c_0 = 8.21 \text{ \AA}, \\ \alpha = 110^\circ 52', \quad \beta = 101^\circ 28', \quad \gamma = 119^\circ 44'.$$

There are two molecules of  $\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$  in the unit cell. The space group is  $C_2^1 - P1$ . The tartrate molecules are like those in Rochelle salt, with the difference that a *d*- and an *l*-molecule form a pair, probably linked by a hydrogen bond between oxygen atoms belonging to the carboxyl groups. The potassium atom has a six co-ordination, being surrounded by one oxygen atom and one hydroxyl group from the tartrate groups and by four water molecules. The sodium atom also has a six co-ordination consisting of two oxygen atoms and one hydroxyl group from the tartrate groups and three water molecules. Water molecules themselves are in the middles of tetrahedra or of triangles formed of other atoms or atom groups. The structural relation between Rochelle salt and *dl*-tartrate is such that the two structures may be thought of as derived by different principles from the ideal building units whose features are common to them.

$\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$ , potassium sodium *dl*-tartrate tetrahydrate, is a racemoid, its optically active counterpart

being the well-known Rochelle salt. Though known since Mitscherlich (1842) (*Gmelins Handbuch* . . . , 1938,

vol. 22, p. 1163), who first synthesized the compound, we know it crystallographically very little in contrast with Rochelle salt, the crystal structure of which has been successfully analysed by Beevers & Hughes (1940, 1941).

The present study of this compound has been undertaken in anticipation that it might, in relation to Rochelle salt, afford a further example of polysynthetic structures as conceived and expounded recently by Ito (1950).

### Material

The specimen examined, a colourless transparent crystal of dimensions  $1.5 \times 1 \times 0.1$  cm., was synthesized by Y. Tsuzuki of the National Industrial Laboratory of Tokyo and offered to us for study.

The results of an investigation by Y. Takano of the Mineralogical Institute, who carried out measurements with a Goldschmidt two-circle goniometer, follow. The crystals, belonging to the triclinic holohedral class, are usually platy in habit with  $(10\bar{1})$  extended, other faces developed being  $(010)$ ,  $(001)$ ,  $(100)$ ,  $(1\bar{1}0)$ ,  $(01\bar{1})$  and  $(20\bar{1})$  (Fig. 1). The crystallographic constants, based on the measurements

$$(100) \wedge (010) = 51^\circ 44', \quad (010) \wedge (001) = 57^\circ 40',$$

$$(001) \wedge (100) = 62^\circ 24', \quad (100) \wedge (10\bar{1}) = 63^\circ 29',$$

$$(010) \wedge (01\bar{1}) = 63^\circ 14', \text{ are}$$

$$a : b : c = 1.012 : 1 : 0.8515,$$

$$\alpha = 110^\circ 52', \quad \beta = 101^\circ 28', \quad \gamma = 119^\circ 44'.$$

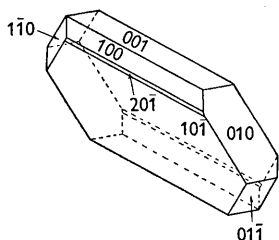


Fig. 1. A crystal of potassium sodium *dl*-tartrate tetrahydrate (measured and drawn by Y. Takano).

### Experimental

Mo  $K\alpha$  radiation ( $\lambda = 0.710$  Å.) was used throughout the experiments. Rotation photographs about  $[100]$ ,  $[010]$  and  $[001]$  gave the dimensions

$$a_0 = 9.80, \quad b_0 = 9.66, \quad c_0 = 8.21 \text{ Å.},$$

with the axial ratios,

$$a : b : c = 1.014 : 1 : 0.8499,$$

in agreement with the values obtained morphologically. For axial angles we adopted the morphological values given above, which have been checked by measurements on the Weissenberg-Buerger photographs, namely,

$$\alpha = 111^\circ, \quad \beta = 101.5^\circ, \quad \gamma = 120^\circ.$$

It may be remarked here that these are the unique constants of the lattice, conforming to the reduction (Ito, 1950, p. 189). There are in the unit cell two molecules of  $\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$  giving the calculated specific gravity 1.677, against  $1.71 \pm 0.01$  as measured by T. Sameshima, formerly of the Mineralogical Institute.

The space group is  $C_2^1 - P\bar{1}$ .

The intensities were estimated visually, using the multiple-film technique of de Lange, Robertson & Woodward (1939), in the Weissenberg-Buerger and rotation photographs, taking into account the shape of specimens in relation to absorption. The crystals easily lose their transparency, giving rise to a number of extra spots in the photographs. To avoid the possible complications in intensity measurement, efforts were therefore made to take photographs quickly before the crystals absorbed moisture (or dehydrated) and partly decayed.

Intensities observed were referred to the absolute scale by taking the intensity of the strong  $(30\bar{2})$  reflexion to be just half the value it would have if all the atoms in the cell were in phase ( $F = 170$ ). No correction for extinction and other factors was made.

### Analysis

At the outset of analysis it was assumed that the same form of molecule of  $\text{C}_4\text{H}_4\text{O}_6$  as found in Rochelle salt by Beevers & Hughes is to be found also in the structure of *dl*-tartrate. Though this molecule may be twisted about the single C-C bonds and may have different configurations, it can, to a first approximation, be regarded as a giant group of atoms occupying a certain definite volume impenetrable by other atoms. To simplify the analysis we tried to accommodate this group in the unit cell, along with the metallic atoms (K and Na) and  $\text{H}_2\text{O}$  molecules, so as to explain roughly the intensity of the  $(h0l)$  spectra. For this purpose we chose sets of strong (e.g.  $30\bar{2}$ ,  $10\bar{2}$ ,  $20\bar{2}$ ,  $30\bar{1}$ ,  $10\bar{4}$ ) and very weak (e.g.  $10\bar{3}$ ,  $40\bar{3}$ ,  $10\bar{3}$ ,  $30\bar{4}$ ,  $20\bar{5}$ ) reflexions and, using diagrams similar to one used by Warren & Bragg (1928) in their study of diopside, tried to find areas in the projection on a plane normal to  $[010]$  into which the respective atoms and atom groups could be put. These led us to fix the positions of four metallic atoms in the  $xz$  plane at

$$x = 0, z = 0; \quad x = 0, z = \frac{1}{2};$$

$$x \doteq 0.10, z \doteq 0.75; \quad x \doteq 0.90, z \doteq 0.25$$

$$\text{or at} \quad x \doteq 0.70, z \doteq 0.50; \quad x \doteq 0.30, z \doteq 0.50;$$

$$x \doteq 0.60, z \doteq 0.90; \quad x \doteq 0.40, z \doteq 0.10.$$

In the former alternative, the tartrate molecule must have its centre at such points as  $x \doteq 0.50, z \doteq 0.50$  or  $x \doteq 0.50, z \doteq 0.25$ , whereas in the latter it must be close to the corners of the cell, i.e.  $x \doteq 0, z \doteq 0$ . At this stage of analysis, in order to decide between these positions, we considered  $(hkl)$  spectra. Since most of these can be accounted for only by the latter combination, we

regarded it as an approximate structure of *dl*-tartrate and started to determine the positions of atoms by trial and error. Bond angles and distances usual in organic compounds and also found in Rochelle salt were used to locate the individual atoms. The parameters of atoms, however, were determined in such a way that the experimental data were best explained, without regard to the possible slight deviations of bond angles and distances from the usual values. Before we arrived at the final structure, Fourier projections upon a plane normal to [010] were used to refine some of the parameter values by successive approximations. Fourier projections upon a plane normal to [101] were used to obtain  $y$  parameters of metallic atoms and the parameters of  $H_2O$  molecules. In Table 1 are given the parameters of the atoms and in Table 2 the  $F$  values observed and those calculated with these parameters. Calculation showed that it was not possible to obtain agreement between the observed and calculated  $F$  values of reflexions of the lower orders to such a degree as we wished without interfering with the majority of other reflexions. This is probably due to the fact that the presence of hydrogen atoms in the structure was not taken into account.

Table 1. *Parameters of atoms in potassium sodium dl-tartrate tetrahydrate*

	$x/a$	$y/b$	$z/c$
K	0.680	0.173	0.471
Na	0.590	0.723	0.900
O (1)	0.270	0.940	0.033
O (2)	0.105	0.910	0.793
O (3)	0.745	0.797	0.226
O (4)	0.820	0.606	0.208
OH (5)	0.795	0.640	0.775
OH (6)	0.050	0.022	0.200
$H_2O$ (7)	0.580	0.480	0.892
$H_2O$ (8)	0.643	0.948	0.596
$H_2O$ (9)	0.362	0.575	0.604
$H_2O$ (10)	0.406	0.770	0.001
C (1)	0.125	0.848	0.904
C (2)	0.960	0.740	0.938
C (3)	0.995	0.842	0.151
C (4)	0.833	0.737	0.190

### Description of the structure

Fig. 2 shows the structure projected on a plane normal to [010] and Fig. 3 the corresponding Fourier diagram. In Fig. 4 is shown a perspective drawing of a tartrate

molecule, including the bond distances and angles. Four carbon atoms lie exactly in a plane, and the molecule is almost of the same configuration as that of Rochelle salt. The two middle carbon atoms carry respectively OH and H, while the carbons at both ends form carboxyl groups. Each molecule is related by a centre of symmetry to its counterpart. The closest approach of the two molecules is between OH of one and O of the other at the extremities of the chains. This OH-O linking is evidenced by the unusual short O-O distance (2.52 Å.) which suggests a probable hydrogen bond between them.

The potassium atom occupies a general position, being sixfold co-ordinated by one oxygen of a tartrate molecule, one hydroxyl group of another, and four water molecules. The sodium atom, also occupying a general position, touches one oxygen and one hydroxyl group of a tartrate molecule, one oxygen of another and three water molecules, these forming a distorted octahedron.

The water molecule  $H_2O(8)$  is 2.62 Å. from two potassium atoms and 2.95 Å. from a hydroxyl group of a tartrate molecule, forming a triangular bond distribution in a plane. The molecule  $H_2O(9)$  is 2.90 Å. from a potassium atom, 2.27 Å. from a sodium atom, 2.92 Å. from an oxygen atom and 2.58 Å. from a hydroxyl group, forming a tetrahedral bond distribution. The molecule  $H_2O(10)$  has its neighbours also tetrahedrally arranged, the distance being 3.48 Å. to a potassium atom, 2.30 Å. to a sodium atom, 2.56 Å. to an oxygen atom, and 2.90 Å. to  $H_2O(7)$ . Finally the molecule  $H_2O(7)$  can also be given four bonds, though their distribution is not as regular, being 2.32 Å. to a sodium atom, 2.90 Å. to  $H_2O(10)$  (whose bond may be considered as directed from  $H_2O(10)$  to  $H_2O(7)$ ), 2.54 Å. to an oxygen atom and 2.52 Å. to a hydroxyl group. An unusually long distance (2.75 Å.) between OH(5) and the sodium atom may be due to repulsion between sodium and the hydrogen atom of  $H_2O(7)$ , which should bind the hydroxyl.

Beevers & Hughes have traced in Rochelle salt a uni-directional series of bonds (carboxyl-water) which may possibly be responsible for its anomalous dielectric properties. No such type of bond could be found in the structure of the racemoid. In Table 3 interatomic distances are given.

Table 2. *Intensities of reflexions of potassium sodium dl-tartrate tetrahydrate*

$hkl$	$F_{obs.}$	$F_{calc.}$	$hkl$	$F_{obs.}$	$F_{calc.}$	$hkl$	$F_{obs.}$	$F_{calc.}$
100	17	-10.8	30 $\bar{2}$	85	87.6	40 $\bar{3}$	7	-13.2
10 $\bar{1}$	27	-22.0	102	65	-69.4	103	9	-30.2
001	23	43.6	20 $\bar{3}$	12	1.6	204	50	-52.6
20 $\bar{1}$	10	10.0	10 $\bar{3}$	6	-3.2	400	33	-33.8
101	22	34.4	300	32	28.2	304	—	-0.8
10 $\bar{2}$	42	-43.0	30 $\bar{3}$	26	24.8	104	55	48.4
200	26	-30.0	003	29	-46.6	40 $\bar{1}$	21	28.6
20 $\bar{2}$	38	-39.6	40 $\bar{2}$	10	-11.6	50 $\bar{2}$	8	10.0
002	13	14.2	202	34	34.2	004	16	15.0
30 $\bar{1}$	13	-15.8	40 $\bar{1}$	6	-1.4	302	12	17.8
201	23	48.2	301	55	-54.8	50 $\bar{3}$	22	28.4

Table 2 (cont.)

<i>hkl</i>	<i>F</i> <sub>obs.</sub>	<i>F</i> <sub>calc.</sub>	<i>hkl</i>	<i>F</i> <sub>obs.</sub>	<i>F</i> <sub>calc.</sub>	<i>hkl</i>	<i>F</i> <sub>obs.</sub>	<i>F</i> <sub>calc.</sub>
50 $\bar{1}$	33	51.6	807	9	10.2	34 $\bar{3}$	17	-2.0
203	12	16.4	405	14	13.6	15 $\bar{1}$	17	18.6
401	6	6.0	90 $\bar{2}$	1	-8.6	43 $\bar{4}$	6	8.0
504	26	-20.6	702	7	10.4	515	1	0.6
305	16	-12.4	107	8	-0.4	515	8	8.4
205	4	-4.2	90 $\bar{6}$	—	2.2	252	4	-5.8
104	9	-10.8	708	10	10.8	252	15	20.6
500	7	8.6	108	3	2.4	525	5	-5.2
405	12	-14.0	306	12	13.4	525	16	-16.6
105	6	9.0	90 $\bar{1}$	26	-27.0	444	4	3.2
603	—	1.8	801	1	5.0	444	21	-26.8
602	15	16.8	907	22	29.0	353	27	-36.2
402	12	-15.0	808	6	-7.4	535	1	-4.4
303	2	-8.0	509	11	11.4	535	9	-12.6
505	11	19.2	409	17	-18.6	353	19	-26.2
005	16	19.2	207	9	-11.2	060	—	0.0
604	4	7.2	10.0.4	11	7.4	161	5	8.4
60 $\bar{1}$	32	-29.8	008	—	2.0	16 $\bar{1}$	21	25.4
501	4	6.0	10.0.5	17	-22.8	616	1	3.2
204	12	-11.8	609	16	-21.6	616	3	-4.4
306	21	-16.6	10.0.3	12	-14.2	262	6	-9.6
406	37	39.6	604	11	14.6	262	—	-2.2
206	13	-12.0	309	9	-12.6	454	12	14.6
605	12	19.8	900	—	0.8	545	36	-42.4
600	10	-7.8	505	2	-0.8	626	2	3.0
105	13	28.0	703	11	12.0	626	15	-16.6
506	13	-17.6	10.0.6	23	21.0	454	5	-5.6
703	11	-13.8	709	1	-4.6	545	2	6.0
106	9	12.6	10.0.2	11	13.8	363	24	-29.2
403	21	24.4	406	18	-20.4	636	16	17.4
702	8	14.2	209	15	19.6	363	17	-21.6
704	16	8.0	802	7	-12.6	636	6	-4.0
502	17	-26.2	908	4	9.8	070	—	2.6
304	5	4.8	108	14	-21.8	171	5	6.2
701	21	23.2	10.0.7	16	-15.4	555	13	17.0
606	8	10.6	809	2	-5.2	171	2	2.6
601	29	-54.0	307	4	5.4	464	4	-3.0
705	—	1.0	10.0.1	6	13.2	272	3	-7.6
006	9	-9.0	109	6	-9.0	717	1	-6.6
205	13	-17.6	901	4	-9.2	555	26	34.8
407	—	0.4	010	35	35.0	646	9	-11.2
307	—	-2.6	111	55	61.8	717	21	-18.4
700	3	2.8	111	9	-8.2	272	24	-26.6
507	12	15.2	020	13	-20.6	464	16	19.6
207	22	30.2	121	2	-3.2	727	19	24.4
706	23	28.2	212	—	4.2	646	32	36.2
804	5	-2.0	121	25	26.2	727	11	-5.2
106	15	-16.4	212	11	-3.2	373	7	-11.0
503	—	-2.4	222	14	12.6	373	4	7.6
404	13	-16.6	222	23	-41.0	737	—	2.0
802	3	-3.2	030	13	-18.2	565	6	7.8
607	5	3.2	131	22	-12.2	656	16	-24.4
602	—	-0.4	131	37	42.2	737	40	45.4
805	22	23.0	313	33	-47.6	565	8	12.4
107	22	-29.6	313	16	-9.4	474	7	-15.0
305	6	-7.6	232	32	21.2	656	3	5.0
803	32	41.0	323	—	-1.4	181	14	20.8
801	23	27.8	232	13	23.4	181	21	-25.8
701	16	18.8	323	—	-3.8	747	3	8.8
707	13	-15.8	040	6	9.4	474	17	10.2
806	13	-18.2	141	29	-46.8	282	—	-1.8
007	22	-21.4	141	26	20.0	818	29	-28.6
206	3	5.2	333	18	23.0	282	21	28.0
408	24	21.2	414	8	5.2	818	6	13.2
508	10	-8.0	414	13	14.2	747	9	16.4
308	12	-3.2	333	6	7.8	828	—	-3.4
904	13	-17.4	242	22	-24.2	383	7	13.4
800	13	-13.2	424	16	-17.6	383	14	23.4
903	6	-8.2	242	25	25.2	828	11	10.6
504	2	-6.6	424	2	7.6	838	—	2.2
608	27	-36.2	343	16	18.8	090	27	-28.8
905	1	-3.0	050	14	17.4	191	3	3.4
603	15	-16.0	434	33	-40.0			
208	—	2.8	151	9	-15.0			

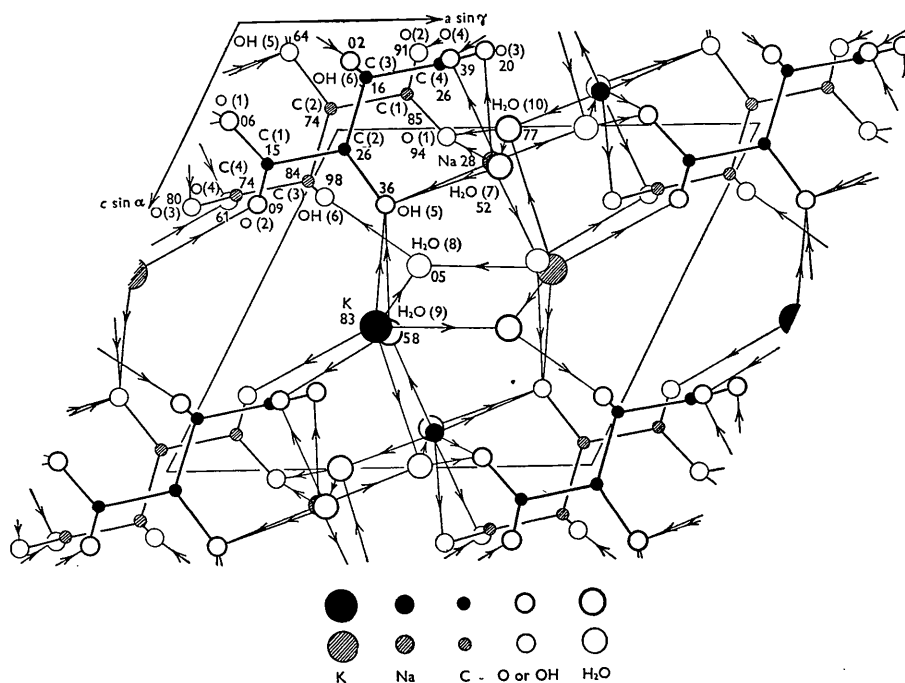


Fig. 2. The structure of potassium sodium *dl*-tartrate tetrahydrate,  $\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$ , as projected on a plane normal to the [010] axis. Bond directions are shown by arrows. Numbers give the heights of atoms from (010) as a percentage of the  $b$  translation.

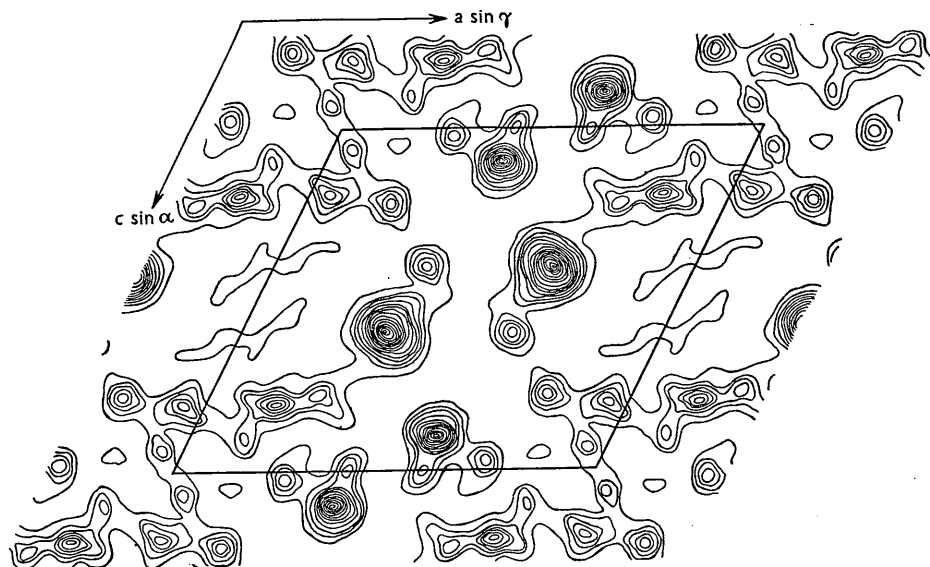


Fig. 3. A Fourier projection of electron density of potassium sodium *dl*-tartrate tetrahydrate. The projection corresponds to Fig. 2. Contours at an interval of  $2 \text{ e.A.}^{-2}$ .



This relationship between the structures of Rochelle salt and *dl*-tartrate may now be visualized, using diagrams similar to those adopted by Ito (1950) and others. Representing by  $T$  and  $T'$  a configuration having the composition  $(\text{KNa})\frac{1}{2}\text{C}_4\text{H}_4\text{O}_6 \cdot \text{H}_2\text{O}$  and its mirror image, by  $H_1$  and  $H'_1$  a configuration having the composition  $(\text{KNa})\frac{1}{2} \cdot 3\text{H}_2\text{O}$  and its mirror image, by  $H_2$  and  $H'_2$  a configuration having the composition  $(\text{KNa})\frac{1}{4} \cdot 2\text{H}_2\text{O}$  and its mirror image, and by  $H_3$  and  $H'_3$  a configuration having the composition  $(\text{KNa})\frac{1}{4} \cdot \text{H}_2\text{O}$  and its mirror image, we have

	$T$	$H_1$	$T$		$T'$	$H'_1$	$T'$
Rochelle salt	$H_1$	$T$	$H_1$ or		$H'_1$	$T'$	$H'_1$
	$T$	$H_1$	$T$		$T'$	$H'_1$	$T'$

Potassium sodium	$H_2$	$T$	$H_3$
<i>dl</i> -tartrate	$H'_3$	$T'$	$H'_2$
tetrahydrate	$H_2$	$T$	$H_3$

In contrast with inorganic compounds, in which, as shown by Ito, the polysynthetic structures often take place in strict or nearly strict sense of terms, they appear to be realized here but approximately, the terms being applicable only to the broad structural principles and not to the structures in every detail. The concept, nevertheless, is very useful in discussing organic structures as illustrated by the present example, enabling us to elucidate relationships between kindred compounds which would otherwise be easily overlooked, and to describe the complicated structures in a straightforward and simple way.

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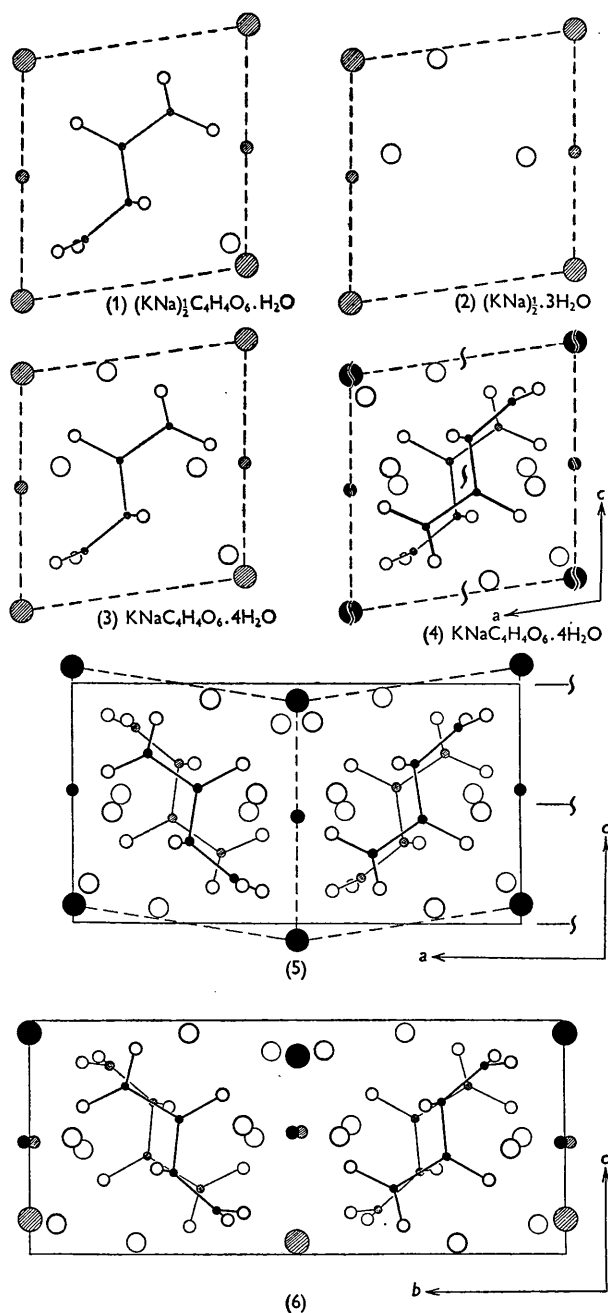


Fig. 5(a).

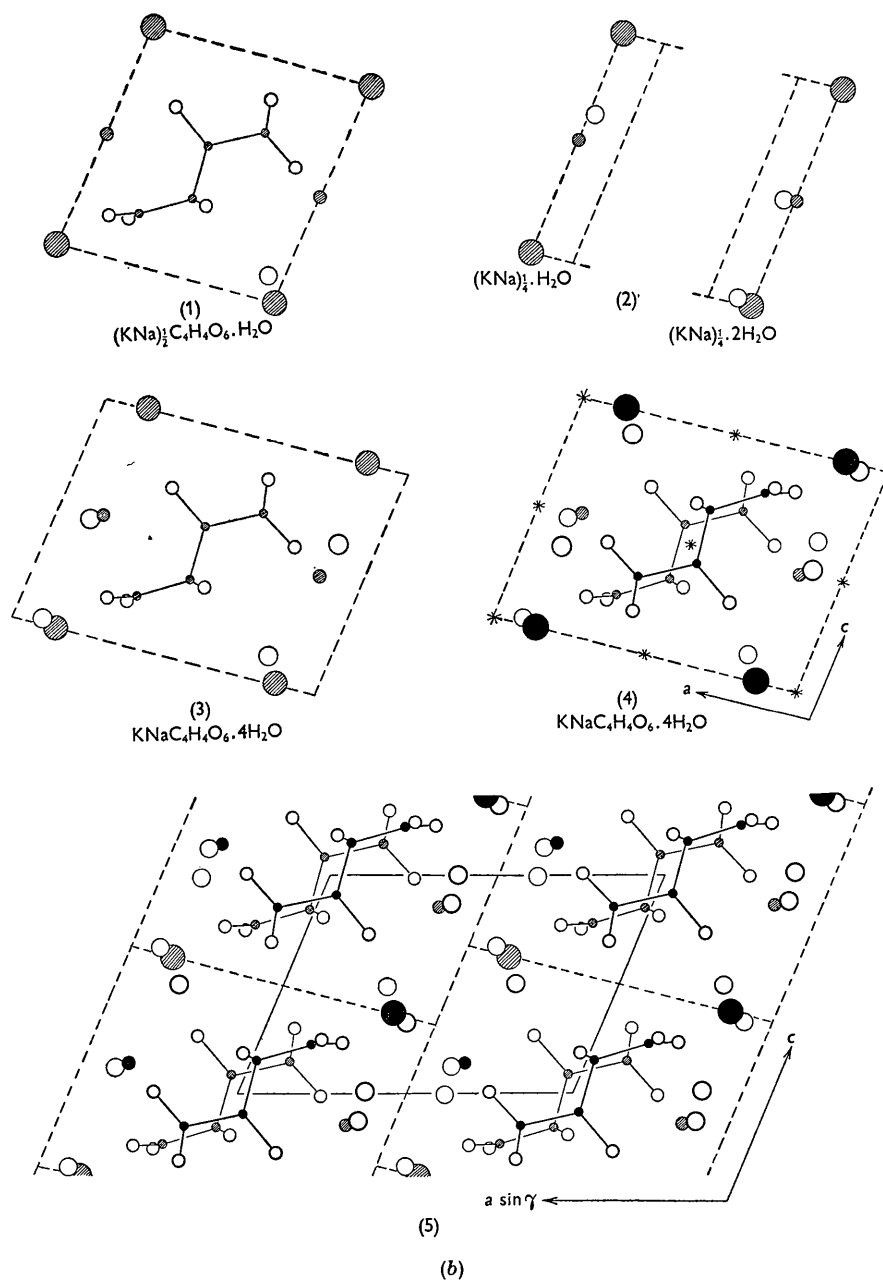


Fig. 5. A way in which both the structure of Rochelle salt and that of potassium sodium *dl*-tartrate tetrahydrate are derived from the common building units (see text). (a) Rochelle salt. (b) Potassium sodium *dl*-tartrate tetrahydrate. Notation as in the previous figures.