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Crystallographic data of α-glucoheptonic acid γ-lactone.

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Crystals of β-glucoheptonic acid γ-lactone, prepared by recrystallization of the commercial compound from water, were found to be orthorhombic with

$$a_0 = 7{\cdot}61,\ b_0 = 17{\cdot}25,\ c_0 = 6{\cdot}60\ \text{Å}\ (\text{all } \pm 0{\cdot}02\ \text{Å})\ .$$

The density was measured by flotation as 1·587 g.cm.$^{-3}$, (calculated: 1·596 g.cm.$^{-3}$); therefore the cell contains four molecules of $C_7H_{12}O_7$. Weissenberg patterns showed odd orders of $h00$, $0k0$, and $00l$ to be the only systematic absences, thus establishing the space group as D_2^4–$P2_12_12_1$.

Principal powder diffraction lines are given in Table 1. A camera with a diameter of 114·6 mm. and Cr $K\alpha$ radiation (λ = 2·285 Å) were used. Relative intensities were determined visually and no corrections for absorption were applied.

Optical data were kindly furnished by H. A. Rose of these laboratories:

Refractive indices (5893 Å, 25° C.):

$$\alpha = 1{\cdot}498 \pm 0{\cdot}002,\quad \beta = 1{\cdot}562 \pm 0{\cdot}002,\quad \gamma = 1{\cdot}574 \pm 0{\cdot}002\ .$$

Optic axial angle: (−)45° (calculated from α, β, and γ).
Optic axial plane: 100.
Acute bisectrix: $\alpha = b$.

Form and habit: The crystals are equant and may be found lying on any face. They show the forms: prism {110}, brachydome {011}, and brachypinacoid {010}.

Table 1

Observed spacing (Å)	I/I_1	*HKL*	Calculated spacing (Å)
8·63	0·07	020	8·63
7·00	0·03	110	7·00
6·18	0·50	011	6·18
5·26	0·13	021	5·25
4·80	1·00	111	4·80
4·59	0·33	130	4·59
4·31	1·00	040	4·31
3·81	0·03	200	3·81
3·75	0·67	210, 140	3·74, 3·76
3·60	0·33	041	3·61
3·31	0·07	002	3·30
3·25	0·20	012	3·25
3·15	0·03	150	3·17
3·10	0·03	022, 051, 221	3·10, 3·09, 3·10
2·86	0·27	240	2·86
2·69	0·27	160	2·70
2·62	0·13	042, 061	2·62, 2·64
2·55	0·07	250	2·56
2·50	0·07	202	2·50
2·39	0·03	052	2·39
2·36	0·07	301, 170	2·37, 2·35
2·33	0·07	330	2·33
2·29	0·13	260, 232	2·30, 2·29
2·27	0·13	321	2·28
2·20	0·07	340	2·19
2·10	0·13	113	2·10
2·05	0·07	350, 123	2·05, 2·05
1·96	0·07	043, 322	1·96, 1·96
1·90	0·07	400	1·90
1·67	0·07	0, 10, 1	1·67

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A preliminary X-ray study of some alkali aminedisulphonates and methanedisulphonates.

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As a result of goniometric measurements, Münzing (1888) and Zirngiebl (1902) concluded that crystals of ammonium and potassium aminedisulphonates (or imidosulphates) are isomorphous. In addition Zirngiebl pointed out the crystallographic similarities between these salts and ammonium and potassium methanedisulphonates (or methionates); the morphological constants of these and other alkali metal methionates have been determined by Backer & Terpstra (1929).

The cell dimensions and space groups of five of these sulphonates have now been determined from single-crystal X-ray photographs with Cu $K\alpha$ radiation, and are recorded in Table 1. Ammonium aminedisulphonate was prepared by deammonation of ammonium sulphamate; and potassium aminedisulphonate by hydrolysis of the trisulphonate, $K_3(N(SO_3)_3)2H_2O$; rubidium aminedisulphonate, no previous reference to which could be found in the literature, was obtained by double decomposition of the more soluble ammonium salt. Suitable specimens of ammonium and potassium methionates were crystallized from material kindly supplied by Prof. H. J. Backer.

Systematic extinctions showed the space groups of $(KSO_3)_2NH$, $(KSO_3)_2CH_2$ and $(NH_4SO_3)_2CH_2$ to be Cc or $C2/c$, and statistical examination of the intensities of the aminedisulphonate indicated that the centrosymmetrical $C2/c$ should be chosen. The isomorphism of the three compounds is shown not only by the similarity of cell dimensions and space group but by correspondences between low-order intensities. The ammonium and ru-

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Table 1. *Crystallographic data*

	a (Å)	b (Å)	c (Å)	β (°)	Mols/ unit cell	Density (g.cm.$^{-3}$) Obs.	Density (g.cm.$^{-3}$) Calc.	Space group
Ammonium aminedisulphonate $NH(SO_3NH_4)_2$	12·72	3×7·74=23·2	7·49	92·8	12	2·00	1·90	—
Potassium aminedisulphonate $NH(SO_3K)_2$	12·43	7·46	7·18	91·2	4	2·49	2·47	$C2/c$
Rubidium aminedisulphonate $NH(SO_3Rb)_2$	12·80	5×7·68=38·4	7·45	91·9	20	3·00	3·12	—
Ammonium methanedisulphonate $CH_2(SO_3NH_4)_2$	12·70	7·85	7·65	92·6	4	1·83	1·63	$C2/c$
Potassium methanedisulphonate $CH_2(SO_3K)_2$	12·55	7·75	7·30	90·5	4	2·37	2·37	$C2/c$

The accuracy of the above cell dimensions is about 0·05 Å, except for potassium aminedisulphonate for which it is about 0·01Å.

bidium aminesulphonates also have structures very closely similar to that of the potassium salt, but extremely weak layer lines on b-axis photographs show that these two salts have superstructures of respectively three and five times the unit-cell volume of the potassium salt.

The results of a detailed three-dimensional analysis of potassium aminedisulphonate will be published separately (with Prof. G. A. Jeffrey).

I am grateful to Prof. E. G. Cox for suggesting this investigation and to the British Rayon Research Association for financial support.

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The unit cell and space group of the complex tridipyridyl-nickel sulfate.

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The complex tridipyridyl–nickel sulfate, which is isomorphous with the analogous copper salt, has the formula $Ni(\alpha\alpha'\,dip.)_3SO_4.7H_2O$. Jaeger & Van Dyck (1938) found that it forms prismatic monoclinic crystals with

$$a:b:c = 1{\cdot}547:1:0{\cdot}908,\ \beta = 97^\circ\ 52'\ .$$

We have made rotation diagrams round the b and c axes and equi-inclination Weissenberg photographs of the levels $h0l$, $h1l$, $hk0$ and hkl. As no faces of the zone [100] are developed, the determination of a had to be made from the central lattice line spacing of zero-layer Weissenberg photographs.

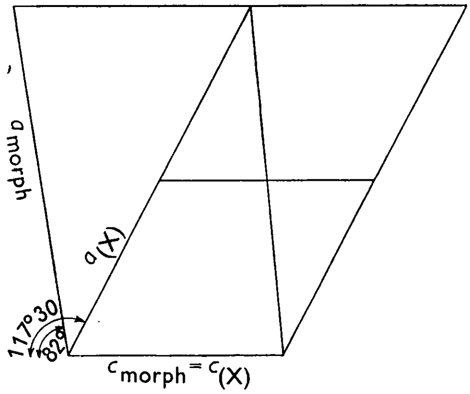

Fig. 1.

The cell dimensions are

$$a = 22{\cdot}90,\ b = 14{\cdot}19,\ c = 24{\cdot}80\ \text{Å};\ \beta = 117^\circ\ 3'\ .$$

As is seen in Fig. 1, the X-ray cell is not identical with the morphological cell. The b axis, perpendicular to the plane of the paper, is twice as large in the morphological cell as in the X-ray cell.

The density, determined by flotation, was 1·45 g.cm.$^{-3}$, requiring eight formula weights per unit cell. Systematic extinctions were found in the reflexions $h00$, $0k0$, $00l$ when k, k, l respectively are odd. No reflexions were observed for hkl when $h+k$ is odd, indicating a C end-centred lattice. These absences characterize the space groups Cc and $C2/c$; in view of the external symmetry we propose

Table 1. *Powder-pattern data*

d_{hkl}	I	d_{hkl}	I
11·11	10	3·65	9
9·09	1	3·33	1
7·27	3	3·03	1
6·55	10	2·81	2
5·59	3	2·52	4
5·14	6	2·31	1
4·61	2	2·10	3
4·34	8	1·84	2
4·02	8		