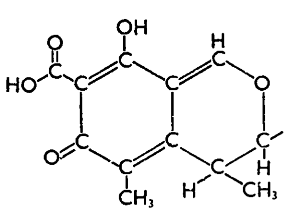
each proposed structures for the molecule which differ only in the positions suggested for the carboxyl group:



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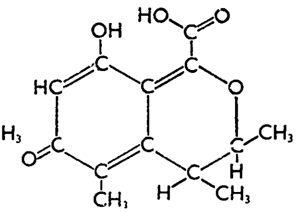

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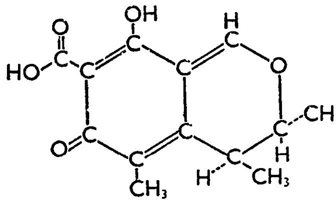
Cram (1950) has stated that the positions of the methyl groups on the two optically active carbon atoms would conform to the structure



or its mirror image.

The data presented in this communication were obtained in a study of the crystal preliminary to an attempt to establish the correct molecular structure by means of X-ray diffraction methods.

The crystals used were isolated by Ruoff & Hirshey (1942) in the course of a chemical study of citrinin. It crystallizes in the form of yellow orthorhombic needles elongated parallel to the x axis. The birefringence is large and negative with

$$\alpha = 1{\cdot}454\ \|a, \quad \beta = 1{\cdot}695\ \|b, \quad \gamma > 1{\cdot}700\ \|c.$$

Good cleavage occurs parallel to (100) only.

Oscillation and Weissenberg photographs gave the following cell dimensions:

$$a = 7{\cdot}38, \quad b = 12{\cdot}32, \quad c = 13{\cdot}69\ \text{A}.$$

All reflections observed show extinctions for odd orders of ($h00$), ($0k0$) and ($00l$), with no other systematic absences, hence the space group is D_2^4-$P2_12_12_1$. The density, measured by flotation, is 1·335 g.cm.$^{-3}$. The cell must therefor contain four (calculated 4·02) molecules based on the formula weight (250·24) of the proposed molecule.

It may be inferred from the above that the molecules approach a planar configuration with their planes arranged approximately parallel to (100). A complete structure analysis is now in progress.

The authors wish to acknowledge the kindness of Dr I. Fankuchen, Polytechnic Institute of Brooklyn, and Dr J. L. Hoard, Cornell University, in making available to them the facilities of their laboratories during portions of this investigation.

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The η phase of sodium 1-dodecanesulfonate.

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During the course of the investigation of the crystalline phases of paraffin-chain compounds in this laboratory, single crystals of seven different phases of the sodium 1-alkanesulfonates have been obtained. X-ray crystallographic data for three of these have been reported (Jensen & Lingafelter, 1944, 1946; Lingafelter & Jensen, 1950). The present report deals with that phase which we have designated the η phase. Crystals of sodium 1-dodecanesulfonate have been obtained in this phase by crystallization from water at temperatures between 20 and 31·5° C. At 31·5° C. the η-phase crystals occur mixed with crystals of the ϵ phase. The triclinic crystals of the η phase usually appear as poorly-formed needles elongated parallel to the a axis. The few well-formed crystals are thin tabular parallel to (001) and are outlined by ($01l$) and ($11l$).

X-ray diffraction data were obtained from rotation, Weissenberg, and precession photographs using copper radiation ($K\alpha$ 1·5418 A.). The constants of the reduced triclinic cell are

$$a_0 = 5{\cdot}60, \quad b_0 = 8{\cdot}60, \quad c_0 = 51{\cdot}51\ \text{A.},$$
$$\alpha = 94\tfrac{1}{4}^\circ, \quad \beta = 93^\circ, \quad \gamma = 91\tfrac{1}{2}^\circ.$$

An attempt was made to determine the degree of hydration of the η phase by a method to be described elsewhere, involving the measurement of the volume of water evolved by a weighed sample upon slow heating under 15 mm. pressure, but the crystals proved to be unstable at 15 mm. pressure of water vapor and room temperature, so that it was not possible to distinguish accurately between surface water and water of hydration. The results indicated, however, the presence of more than 1 molecule of water per molecule of sulfonate. The experimental density, 1·227 g.cm.$^{-3}$, determined by flotation, indicates 6 molecules of $C_{12}H_{25}SO_3Na.xH_2O$ per unit cell, and gives best agreement for a molecular weight corresponding to about 1·5 molecules of water per molecule of sulfonate.

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Six molecules per unit cell is rather unusual for paraffin-chain salts, having been found previously only for the ζ phase of the sodium alkane sulfonates, although Buerger (1945) found 12 chains per unit cell in the triclinic β form of sodium acid stearate. By analogy with other paraffin-chain salts, it may be concluded that the molecules lie in sheets parallel to (001) with the chains tilted more or less from the normal to this plane. There are four possible arrangements which might be considered, having respectively 6, 3, 2 or 1 molecule in the a–b cross-section. The first and third of these seem very unlikely, since the ionic forces between the sulfonate groups and the sodium ions almost certainly lead to a 'head-to-head' arrangement, requiring an even number of molecules in the c direction, as is the case for all paraffin-chain salts which have been investigated. The second possibility, 3 molecules in the a–b cross-section and 2 molecules in the c direction, appears to be rather unlikely from a consideration of the dimensions of the unit cell. In the first place, this leads to an effective molecular length of 25·6 A. which is considerably longer than the length observed for the alpha phase, 18·1 A. It does not seem possible that this difference could be accounted for by poorer 'end-packing' and the increased hydration. In the second place, this arrangement leads to a cross-sectional area per molecule of 16·04 A.^{2} In a recent paper reviewing X-ray crystallographic data on a large number of paraffin-chain compounds, Kohlhaas (1949) has shown that, for most paraffin-chain compounds, the cross-sectional area is usually 18–19 A.^{2} He lists no case in which the cross-section is less than 17·8 A.^{2} Vand, Aitken & Campbell (1949) have recently reported a cross-sectional area of 16·83 A.^{2} for silver hexanoate, but, since their results are based entirely on powder data, it is quite possible that they may be in error.

Thus it appears that, if the molecules in the η phase arrange themselves in sheets as is the case for other paraffin-chain compounds, there are 6 molecules in the c length and only one in the a–b cross-section. If we estimate the effective length of the sodium dodecanesulfonate molecule to be 18·1 A. as observed in the α phase, we can calculate the angle between the chain direction and the normal to the sheets to be about 61° $\left(\cos\tau=\dfrac{51\cdot28}{6\times18\cdot1}\right)$, and the effective chain cross-section to be 22 A.^{2} Because of the fact that the chains are tilted slightly in the α phase and also since the η phase is more highly hydrated than the α phase, the actual tilt of the chains is probably greater than 61° and the cross-section less than 22 A.^{2}

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The monohydrate of ethylenediamine *d*-tartrate: optical properties and X-ray diffraction data.

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Ethylenediamine d-tartrate monohydrate may be crystallized from an aqueous solution containing equimolecular proportions of ethylenediamine and d-tartaric acid. The hydrate is the stable form in contact with solution when the latter is saturated below approx. 41° C. The crystals belong to the orthorhombic sphenoidal class and exhibit the following forms: prisms {110}, domes {101}, {021} and sphenoids {111}.

The principal X-ray diffraction data and optical properties are given below and in Table 1.

$C_6H_{14}N_2O_6.H_2O$. Molecular weight = 228·20.

Specific gravity (18° C.) = $1\cdot52_4$.

Orthogonal structure cell dimensions (± 0·03 A.):

$a=11\cdot56$, $b=15\cdot04$, $c=5\cdot80$ A.

Molecules per unit cell = 4.

Probable space group $P2_12_12_1-V^4$.

Refractive indices (± 0·002), sodium light, 18° C.:

$\alpha=1\cdot542$ parallel to a,

$\beta=1\cdot552$ parallel to c,

$\gamma=1\cdot552$ parallel to b.

Birefringence negative, $\gamma-\alpha=0\cdot01$.

Optic axial angle ($2V$) approx. 26° at 18° C.

Table 1. *Principal 'powder' lines*

(Intensities visually estimated: d not corrected for absorption)

d (A.)	Intensity	d (A.)	Intensity	d (A.)	Intensity
9·23	0·5	3·78	2	2·58	2
7·51	0·5	3·63	10	2·46	8
6·31	3	3·46	1	2·44	3
5·80	2	3·24	4	2·36	1
5·44	1	3·17	5	2·31	3
5·22	4	3·04	1	2·25	3
4·91	0·5	2·93	4	2·15	3
4·55	10	2·87	5	2·12	3
4·31	4	2·78	4	2·06	3
4·13	3	2·72	5	1·99	3
3·99	9	2·59	1	—	—

With decrease in temperature, crossed-axial-plane dispersion was observed, crystals being in the uniaxial condition at − 34 ± 5° C. This is in contrast to the behaviour of the anhydrous tartrate (Steward, 1950), in which this effect occurred with increase in temperature. Both the anhydrous material and the hydrate are piezoelectric.

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