temperature factors. The following summarizes the crystal data:

$$a_0 = 4{\cdot}06, \quad b_0 = 5{\cdot}15, \quad c_0 = 8{\cdot}87\ \text{A}.$$

Laue symmetry, *mmm*.

Extinctions, $h0l$ absent for $l \neq 2n$.

A strongly positive test for pyroelectricity indicated the space group $Pmc2_1$. The calculated density was 1·677 g.cm.$^{-3}$, observed 1·658 g.cm.$^{-3}$, giving two molecules per unit cell. These results are not compatible with the monoclinic cell previously assigned on the basis of an optical study (Cook, 1914), nor with the handbook value of 2·454 g.cm.$^{-3}$ for the density.

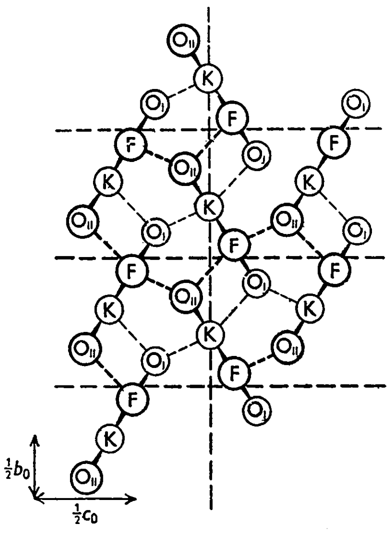

Fig. 1. Projection of KF.2H$_2$O on (100) plane.

An easily interpretable Patterson projection on (001) gave approximate x and y co-ordinates, the x co-ordinates then being fixed at exactly 0 and $\frac{1}{2}$ by symmetry and packing considerations. These co-ordinates were:

	x	y
K	$\frac{1}{2}$	0·4
F	0	0·1
H_2O_I	$\frac{1}{2}$	0·2
H_2O_{II}	0	0·3

That the water molecules are in twofold rather than in fourfold positions is in keeping with the presence of the mirror planes only 2 A. apart along the a axis, which can accommodate these units only in those planes.

The x and y co-ordinates permitted the calculation of the phase angles, 0 or π, for the $hk0$ planes, enabling further refinement by means of Fourier projections on (001). These latter calculations were carried out at millicycle intervals using both unmodified values of $F_{\text{obs.}}$ and, later, values corrected for series termination, as suggested by Booth and Cochran (1948). It was not necessary to change any of the phase angles throughout the refinement procedure.

When packing considerations were applied to the displacements of the units in the x, y directions it became apparent that in the plane $(0, y, z)$ the fluoride ion and the water molecule, H_2O_{II}, differed in their z co-ordinates by one-fifth of a cell edge. Likewise, in $(\frac{1}{2}, y, z)$ the potassium ion and H_2O_I differed by three-tenths. A Patterson projection parallel to the [110] axis showed the fluoride and potassium ions to be $0{\cdot}117c$ apart.

A one-dimensional projection using the $(00l)$ data was then made, giving the z parameters listed in Table 1. Although one cannot expect high accuracy for parameters determined from a projection using only the five available orders of $(00l)$, the parameters so determined gave a value of 0·18 for the reliability index for the 34 reflections in the $\{hhl\}$ zone. Consequently, since the projection parallel to [110] does not have a center of symmetry, no further refinement was made. The reliability index,

$$R = \Sigma \,||\, F_o \,| - |\, F_c \,||\, \div \Sigma \,|\, F_o \,|,$$

had the value of 0·15 for 33 $hk0$ reflections and an overall value of 0·19 for 124 planes available from the four Weissenberg photographs. It may be pointed out that, since the scattering factor of the potassium ion is two to three times that of any of the others, the positions of the fluoride ion and water molecules are probably not as accurately fixed as that of the potassium ion.

Table 1. *Final parameters*

	x	y	z
K	$\frac{1}{2}$	0·391	0
F	0	0·091	0·117
H_2O_I	$\frac{1}{2}$	0·189	0·705
H_2O_{II}	0	0·307	0·405

The crystal is made up of distorted octahedra about both the potassium and fluoride ions. About the water molecules are distorted tetrahedra of two cations and two anions, as is usually found in hydrates.

The two water molecules appear to form two different length hydrogen bonds with the fluoride ion. The H_2O_I–F distance is 2·61 A., whereas the H_2O_{II}–F distance is 2·79 A., as compared with the accepted radius sum of 2·76 A. for the oxide and fluoride ions.

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Acta Cryst. (1951). **4**, 182

Preliminary study of the structure of citrinin.

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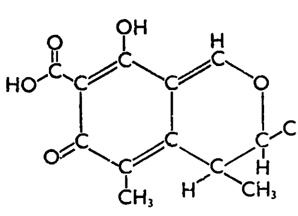
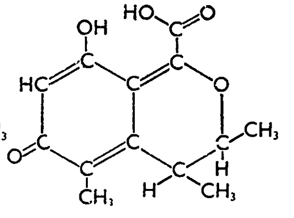
Citrinin is produced as a crystalline metabolic product of the mold *Penicillium citrinum*, and has been of interest experimentally from the standpoint of its antibiotic properties. As a consequence of chemical studies of its degradation products, Gore, Panse & Venkataraman (1946) and Cartwright, Robertson & Whalley (1949 *a*, *b*, *c*) have each proposed structures for the molecule which differ only in the positions suggested for the carboxyl group:

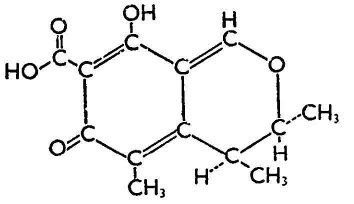


Cartwright *et al.*



Gore *et al.*

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

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

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 $D_2^4\text{-}P2_12_12_1$. 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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