Experimental

Powder diffraction photographs were obtained with a 9 cm. Bradley–Jay type low-temperature camera (Hawes, 1959) using Cu $K\alpha$ radiation in conjunction with a slit collimator.

The halogen specimens were prepared by quickly freezing liquid chlorine enclosed in thin-walled glass capillaries. Because of the rapid rate of recrystallization of solid chlorine at temperatures close to its melting point it was necessary to use four different specimens to obtain the photograph taken at 158·2 °K. Each specimen was photographed for approximately thirty minutes and was then discarded and replaced by one freshly prepared. The rate of recrystallization was sufficiently slow at 77·4 °K. to permit the use of a single capillary throughout the exposure without excessive spottiness in the diffraction patterns. The chlorine used in this work was prepared by the oxidation of hydrochloric acid by potassium permanganate and was purified through fractional distillation.

The temperature of 77·4 °K. was maintained by a cooling bath of slowly boiling liquid nitrogen which surrounded the entire camera, while the temperature of 158·2 °K. was maintained by the manual addition, as required, of small amounts of liquid nitrogen to a sand bath surrounding the camera. This latter temperature was measured by a standardized copper-constantan thermocouple held near the specimen, and it is probable that the stated temperature of 158·2 °K. is reliable to within a degree.

Determination of the physical constants

Existing data by Keesom & Taconis (1936) on the structure of solid chlorine (tetragonal, $P4/ncm$) enabled the films to be indexed. Each film yielded fifteen good diffraction lines suitable for the determination of lattice constants. The lattice constants of chlorine, calculated by the method of least squares, are as follows:

1) at 77·4 °K.: $a = 8{\cdot}550$, $c = 6{\cdot}221$ Å;
2) at 158·2 °K.: $a = 8{\cdot}596$, $c = 6{\cdot}239$ Å.

The overall degree of accuracy of the results is estimated to be approximately one part in two thousand. On this basis the expressions relating lattice parameters in Å to absolute temperature are:

$$a = (8{\cdot}506+5{\cdot}2\times10^{-4}T)\pm0{\cdot}005\ ,$$
$$c = (6{\cdot}204+2{\cdot}2\times10^{-4}T)\pm0{\cdot}003\ .$$

The mean linear coefficients of thermal expansion are

$$\alpha_a = 66{\cdot}4\times10^{-6}\pm3{\cdot}0\times10^{-6}\ °C.^{-1}\ ,$$
$$\alpha_c = 35{\cdot}7\times10^{-6}\pm3{\cdot}9\times10^{-6}\ °C.^{-1}\ .$$

The mean volume coefficient of expansion is

$$\beta = 167{\cdot}5\times10^{-6}\pm5{\cdot}0\times10^{-6}\ °C.^{-1}\ .$$

The density is

$$\varrho = (2{\cdot}098-3{\cdot}5\times10^{-4}T)\pm0{\cdot}002\ \text{g.cm.}^{-3}\ .$$

The values reported by Keesom & Taconis (1936) are

$$a = 8{\cdot}56,\ c = 6{\cdot}12\ \text{Å},\ \varrho = 2{\cdot}09\ \text{g.cm.}^{-3}$$

at a temperature estimated to be −185 °C.

We wish to express our grateful thanks to the Electrolytic Zinc Co. of Australasia Ltd. for making available to us unlimited quantities of liquid nitrogen in this work. One of us (L. L. H.) also thanks Australian Titan Products, Proprietary Limited, for a grant which made this work possible.

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Crystallographic data for valinomycin and evolidine iodoacetate.

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During the selection of compounds suitable for structural analysis, a preliminary investigation of valinomycin and evolidine iodoacetate was made. The chemical formulae of these compounds is best given as follows:

Valinomycin: *cyclo*[*ABAB*]
where
A = D(−)-α-hydroxypropionyl-D(−)valyl ,
and
B = D(−)-α-hydroxy*iso*valeryl-D(−)valyl .

Evolidine:
cyclo [Ser. Phe. Leu. Pro. Val. Asp(β-NH_2).Leu]
(Law, Millar, Springall & Birch, 1958).

As no further work on either is contemplated, the observations are recorded here (Table 1).

The crystals of valinomycin were all twinned and single crystals had to be isolated by careful cutting. The molecular symmetry suggested by the chemical formula (Brockmann & Geeren, 1957) is not reflected in the space group and number of molecules in the unit cell.

Evolidine iodoacetate was found to decompose in the X-ray beam for an integral exposure too small to permit sufficiently extensive diffraction data to be recorded for one layer (even when held at −150 °C.). Although they turn brown due to release of halogen, the crystals do not decompose to a gum, as in the case of bromamphenicol (Dunitz, 1952), or to a mass of dis-oriented crystals; they change to a single crystal of a new phase as the old phase disappears. The new phase is orthorhombic, the relation of the new to old axes being given in Table 1. Evolidine itself belongs to the triclinic system (Eastwood *et al.*, 1955).

Table 1. *Crystallographic data*

Compound	Valinomycin	Evolidine Iodoacetate	Evolidine (Transf. prod.)
Formula	$C_{36}H_{60}O_{12}N_4$	$C_{40}H_{59}O_{10}N_8I$	?
Unit cell dimensions			
a	10·4$_4$(Å)	9·35Å	9·23Å
b	14·47	22·5	23·0
c	22·2$_2$	24·9	25·3
$\alpha°$	105·0°	—	—
$\beta°$	86·9°	—	—
$\gamma°$	90·4°	—	—
Space group	$P1$	$P2_12_12_1$	$P2_12_12_1$
Density (g.cm.$^{-3}$)			
Meas.	1·15	—	—
Calc.	1·13	1·15	—
Number of molecules	3	4	—

I am extremely grateful to Dr L. C. Vining of the Prairie Regional Laboratory, National Research Council, Saskatoon, Saskatchewan, Canada for the preparation and gift of beautiful crystals of valinomycin, to Prof. A. J. Birch, of the University of Manchester, U. K. for his kindness in presenting a quantity of the rare natural product, evolidine, and to Mr A. F. Beecham of this Division for preparing the iodoacetate derivative of evolidine.

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A new aid for the rapid determination of absorption corrections by Albrecht's method.

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Albrecht's (1939) method for graphically determining the correction for absorption of the X-ray beam by a crystal is useful when the crystal cannot be ground into a sphere or cylinder and when automatic computing machinery is not available. Recently Rogers & Moffett (1956) and Henshaw (1958) have published aids to facilitate the procedure. Tho following modification of the methods already described is an added timesaver.

Albrecht approximated the absorption correction for a crystal of constant cross-section by the expression

$$A = N^{-1} \sum_{j=1}^{N} \exp\{-\mu(l_i+l_r)_j\},$$

where μ is the linear absorption coefficient, l_i and l_r are respectively the lengths of the jth incident and diffracted rays, and N is the total number of regularly-spaced points in the cross-section, at which

$$\exp\{-\mu(l_i+l_r)_j\}$$

was evaluated.

In previous methods l_i and l_r were determined in separate operations. It is possible, however, to measure (l_i+l_r) directly and evaluate each term of the summation in a single step. In Fig. 1, a ray enters the crystal at N, is reflected at P, and emerges at M. If a point N' on the extension of PM can be located such that NP is equal in length to $N'P$, the length $N'M$ must represent (l_i+l_r). Because the triangle NPN' is isosceles, the line NN' makes equal angles with NP and $N'P$, and this angle is the complement of the Bragg angle. The line through NN', therefore, must be parallel to the reciprocal lattice vector for the reflection under consideration and is the same for all diffracted rays originating on the same incident ray. By measuring $N'M$ with an exponential scale, one obtains a term of the summation for each chosen point P.

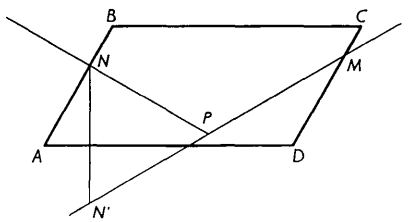

Fig. 1. The path of X-rays reflected at a single point in a crystal. *ABCD* is the outline of a crystal, *NP* is the incident ray, and *PM* is the diffracted ray.

The measurement is made with the aid of one drawing and four overlays. The lowermost sheet (as suggested by Rogers & Moffett, 1956) has a plot of the reciprocal lattice and a correctly oriented outline of the crystal on the scale $\alpha = m\mu x$, where x is the actual dimension, α the corresponding distance in the outline, and m a constant dependent on the exponential scale on which the path lengths within the crystal are measured. Sheets two and three are Bernal circles for the incident and diffracted ray, on the same scale as the reciprocal lattice. Both of these sheets have equally spaced parallel lines for establishing the network of points at which the absorption correction will be evaluated, as described by Rogers & Moffett (1956). Sheets four and five carry the exponential scales for reading the individual absorption terms.

The two scales are mounted on rectangular sheets of clear celluloid. Each sheet must have one straight edge and an inscribed line parallel to it, both lines being the same distance (e.g. 2·5 cm.) from the edge. The scale for l_i, termed the entrance scale, is made by marking a zero point, Z, near one end of a narrow strip of paper. The exit scale, on which (l_i+l_r) is actually measured, is