Table 1. *A survey of observed and calculated intensities and their corrections*

E_p^0 is the primary extinction correction for a purely nuclear or magnetic reflection, E_p the corresponding value for a mixed reflection. $E(1)_s$ and $E(2)_s$ denote the secondary-extinction correction before and after quenching, respectively. $I(1)_{\text{obs.}}$ and $I(2)_{\text{obs.}}$ are the corresponding observed intensities. No changes were found in the E_p's after quenching.

$000l$	$F^2_{\text{nucl.}}$	$F^2_{\text{magn.}}$	$I_{\text{calc.}}$	E_p^0	E_p	$E(1)_s$	$E_pE(1)_sI_{\text{calc.}}$	$I(1)_{\text{obs.}}$	$E(2)_s$	$E_pE(2)_sI_{\text{calc.}}$	$I(2)_{\text{obs.}}$
3	72·4	165·6	338	0·23	0·19	0·69	44	58	0·78	50	85
6	28·9	2·9	21	0·85	0·82	0·90	15	22	0·94	16	25
9	55·7	981·6	487	0·34	0·32	0·40	62	85	0·55	86	98
12	19·9	2·4	8	0·97	0·96	0·96	7	13	0·97	7	12
15	105·5	171·1	77	0·76	0·62	0·76	36	31	0·83	40	31
18	209·7	286·3	113	0·72	0·57	0·68	44	40	0·78	50	44
21	1·1	15·1	3	0·99	0·99	0·98	3	2	0·99	3	—
24	21·0	0·6	4	0·99	0·99	0·98	4	1	0·99	4	—
27	340·8	40·4	56	1·00	0·99	0·72	40	36	0·81	45	39
30	22·1	0·4	3	1·00	0·99	0·98	3	10	0·99	3	7
33	24·2	16·9	5	1·00	0·99	0·97	5	7	0·98	5	6
36	2146·5	45·2	230	0·78	0·77	0·36	64	67	0·51	90	93
39	243·4	0·3	24	0·96	0·96	0·87	20	20	0·91	21	21
42	197·7	0·3	17	0·98	0·98	0·91	15	26	0·94	16	30
45	1499·2	0·1	118	0·87	0·87	0·55	56	51	0·67	69	64
48	10·2	0	1	1·00	1·00	0·96	1	1	1·00	1	0
51	343·0	0	23	1·00	1·00	0·87	20	20	0·92	21	20
54	282·9	0	18	1·00	1·00	0·90	16	14	0·93	17	13

culated values and a curve drawn through those of either predominantly nuclear or magnetic origin. The others are found to be somewhat below this curve, and the ratio of the calculated intensities estimated from the curve to the actual calculated intensities is equal to E_p/E_p^0. By using the aforementioned graphs, the values of E_p^0 and the E_p for these reflections are found. From these, and using (1) and (2), a mean mosaic block thickness of $t = 0{\cdot}00125$ cm was found both before and after heat treatment.

The secondary extinction coefficients E_s were now calculated, using the expression given by Hamilton (1957 and 1958*a*):

$$E_s = \exp\,(-c_s\,I_{\text{calc.}}E_p) \qquad (6)$$

where $c_s = 8\lambda^3A^{1/2}/\eta V_c^2(2\pi)^{3/2}$; A is the cross-sectional area of the crystal and η is the standard angular deviation of the mosaic blocks. Using the equation $I_{\text{obs.}} = E_sE_pI_{\text{calc.}}$ for some of the weaker reflections, c_s and hence η could be estimated. This yielded $\eta = 16''$ for the first run and $\eta = 24''$ for the second. These values may be compared with those found by Hamilton (1958*a*) for a laboratory-grown crystal of magnetite: $t = 0{\cdot}0014$ cm. and $\eta = 15{\cdot}4''$.

The final results are shown in Table 1. The intensities before extinction correction are defined as:

$$I_{\text{calc.}} = \frac{10^{23}}{\sin 2\theta}\,(F^2_{\text{nucl.}}+F^2_{\text{magn.}})\,\exp\,(-1{\cdot}31\,(\sin\theta/\lambda)^2)$$

the temperature factor being taken from Braun's X-ray work. The table shows how severe both primary and secondary extinction are for a crystal of this type, and it is seen that the applied heat treatment does little to improve the situation. Taking the large corrections into account, the agreement between observed and calculated intensities is seen to be quite reasonable, indicating that the spin alignment is correct. Some of the remaining discrepancy might of course also be due to errors in the light-atom positions which would influence the neutron data more strongly than the X-ray data. No improvement resulted by assuming an ordered arrangement of the Zn-atoms (Fig. 1). In view of the considerable difference in scattering power between Fe and Zn this provides further support for Braun's conclusion that the Zn and the Fe atom are randomly distributed over two positions.

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The thermal expansion of solid chlorine.

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The lattice parameters of solid chlorine have been determined at 77·4 °K. and at 158·2 °K. by the use of powder diffraction photographs. Values are given for the coefficients of linear and volume expansion as well as expressions relating lattice parameters and density to temperature.

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:

$$a = (8{\cdot}506 + 5{\cdot}2 \times 10^{-4}T) \pm 0{\cdot}005\ ,$$
$$c = (6{\cdot}204 + 2{\cdot}2 \times 10^{-4}T) \pm 0{\cdot}003\ .$$

The mean linear coefficients of thermal expansion are

$$\alpha_a = 66{\cdot}4 \times 10^{-6} \pm 3{\cdot}0 \times 10^{-6}\ {}^\circ\text{C.}^{-1}\ ,$$
$$\alpha_c = 35{\cdot}7 \times 10^{-6} \pm 3{\cdot}9 \times 10^{-6}\ {}^\circ\text{C.}^{-1}\ .$$

The mean volume coefficient of expansion is

$$\beta = 167{\cdot}5 \times 10^{-6} \pm 5{\cdot}0 \times 10^{-6}\ {}^\circ\text{C.}^{-1}\ .$$

The density is

$$\varrho = (2{\cdot}098 - 3{\cdot}5 \times 10^{-4}T) \pm 0{\cdot}002\ \text{g.cm.}^{-3}\ .$$

The values reported by Keesom & Taconis (1936) are

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

at a temperature estimated to be −185 °C.

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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).