If the crystal is very badly out of setting the procedure must be repeated a few times because the adjustments found are only approximate, though they generally converge very rapidly towards the correct positions of the arcs.

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Note on Stadler's double-slit Weissenberg technique.

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Stadler (1950) has described a new method of recording the *n*th and zero layers of the reciprocal lattice simultaneously by means of a Weissenberg goniometer with two screen slits.

We have found that the Unicam double-crystal Weissenberg goniometer requires only a minor transformation in order to make it suitable for that method. The layer-line screen of this instrument (Fig. 1 (*a*)) has the form of four half-cylinders B^1, B^2 and T^1, T^2. These are fixed on the two brass rings R^1, R^2 which can be moved along the rotation axis on the two brass cylinders C^1, C^2 to allow the positioning of the layer-line slit. The brass rings have on one end a rim *r* against which, normally, both half-cylinders are pushed. If the top half of the rim of R^1 is removed by milling and the key-hole-type slot of the top half of the layer-line screen T^1 is prolonged in the opposite direction, it becomes possible to position both screen halves T^1 and B^1 independently from one another (Fig. 1 (*b*)). In this way a setting may be obtained where the *n*th layer of the reciprocal lattice is recorded on the bottom half and the zero layer on the top half of the film, or vice versa. Or, if two crystals are used, the zero layers of the standard crystal and the unknown crystal may be recorded simultaneously on the top half of the film, whereas the *n*th layer of the unknown crystal is recorded alone on the bottom half of the film.

Besides the mechanical simplicity of its realization, the method has the advantage that the background fogging is not augmented because both slit halves act as one full slit only.

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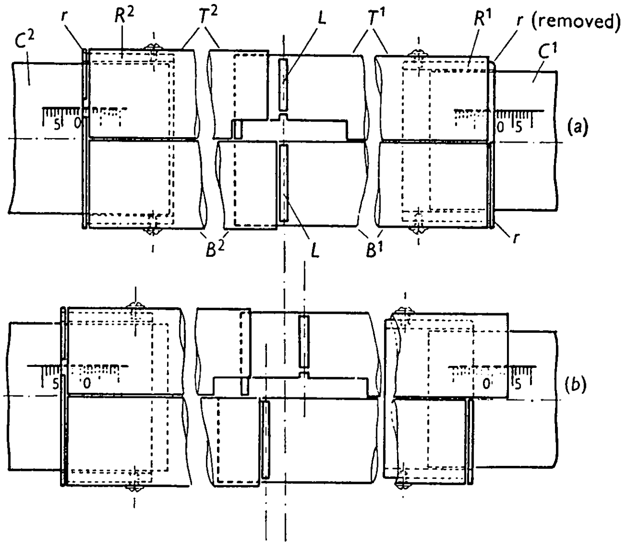

Fig. 1. (*a*) Normal arrangement of screens. (*b*) Arrangement of screens for simultaneously recording two layer lines.

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The space group and optical constants of glucuronolactone.

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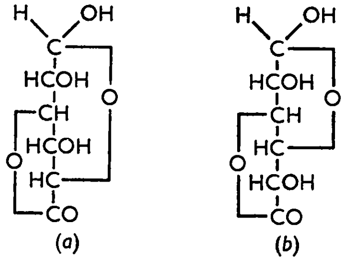
Interest in glucuronolactone has developed recently because of its experimental application in the treatment of arthritis (Smith & Staveley, 1950). It may be obtained by the slow evaporation of an aqueous solution of glucuronic acid. The latter substance is rather widely distributed in conjugated forms in many animal and plant products.

Pryde & Williams (1931) showed that glucuronic acid has a typical pyranoid ring, and later reported a double-ring structure for the lactone (Pryde & Williams, 1933). It was first believed that the lactone contained a six-membered pyranose and a five-membered lactone ring (*a*), but later work by Reeves (1940) and Smith (1944) on trimethyl-glucurono-lactone indicated that the structure consisted of two five-membered rings (*b*). A study of anhydro sugars by Haworth, Owen & Smith (1941) has shown that structures with two five-membered rings in



(*a*) (*b*)

Fig. 1.

the molecule are more stable than those with a five- and six-membered ring. Smith (1944) states that glucuronolactone is probably one of the few examples of crystalline furanose sugar derivatives.

Grünling (1883) measured crystals of glucuronolactone which he reported as monoclinic with apparent point-group symmetry $2/m$. He found

$$a:b:c=1\cdot289:1:1\cdot233;\quad \beta=91^\circ\ 35'.$$

Cleavage was parallel to (101) and (100).

Oscillation and Weissenberg photographs were taken using Grünling's axes. No reflections were observed for (hkl) with $(h+l)$ odd, which indicated a B end-centered lattice. Photographs taken about a new set of axes chosen to give a primitive cell showed absent reflections only for $(0k0)$ with k odd. This characterized the space group as either C_{2h}^2-$P2_1/m$ or C_2^2-$P2_1$. A positive piezoelectric effect was observed in the crystals and was kindly verified for us by Dr R. G. Stokes of the Naval Research Laboratory, Washington, D.C. This definitely established $P2_1$ as the correct space group.

The unit-cell dimensions were found to be

$$a=6\cdot81,\quad b=7\cdot50,\quad c=6\cdot64\ \text{A.};\quad \beta=93^\circ\ 16'.$$

The density, measured by the flotation method, is 1·75 g.cm.$^{-3}$ at 25° C., which requires two (calc. 2·03) molecules in the cell. The refractive indices are

$$\alpha=1\cdot548,\quad \beta=1\cdot578,\quad \gamma=1\cdot588\ (\|b).$$

Cleavage occurs parallel to $(\bar{1}01)$ and (001).

The above results were obtained in a preliminary study of the crystal in the course of an investigation of the structure of glucuronolactone. This investigation was made possible through a fellowship grant by the Corn Products Refining Company, Argo, Illinois, U.S.A.

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Structure transition and antiferromagnetism in magnetite.*

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Introduction

It has long been known that magnetic and specific-heat anomalies are shown by magnetite, Fe_3O_4, in the region of 125° K. A rise in magnetic susceptibility at about this temperature was reported by Weiss & Forrer (1929), and in the same year Millar detected an apparently associated increase in the specific heat. Li (1932) found that single crystals of magnetite developed a strong magnetic anisotropy when cooled below 114·5° K.

More than one attempt has been made to detect any change in crystal structure associated with these low-temperature phenomena, but without success. Ellefson & Taylor (1934) by powder methods, and Shôji (1935) by means of Laue photographs, studied the crystal structure, and concluded that there was no alteration of symmetry, although Ellefson & Taylor's measurements indicated that anomalous changes in the cubic structure cell constant occurred. Snoek (1947, p. 24) discussed the difficulty of explaining the existence of a symmetry of the magnetic anisotropy lower than that of the crystal anisotropy; he suggested the formation of an ordered arrangement of $3d$ electrons at 114·5° K.

The purpose of this note is to report the results of a new investigation of the crystal structure of magnetite at temperatures down to that of liquid air (approximately 95° K.). By means of X-ray powder photographs it has been possible to show that a structure transition occurs at low temperature, and that this comprises a rhombohedral deformation of the ideal cubic lattice.

Results of X-ray examination

The technique employed was similar to that adopted in our studies of monoxides of the transition elements (Tombs & Rooksby, 1950). A 19 cm. diameter X-ray powder camera was modified so as to allow liquid air or a suitably cooled liquid to stream over the rod-type powder specimen during a photographic exposure. The use of either Co $K\alpha$ or Fe $K\alpha$ radiations yielded good definition of a wide range of reflexions of significance in the disclosure of changes in crystal structure. Various samples of both natural and synthetic magnetite were examined, and notable differences in the definition of the X-ray powder lines were observed; oxides prepared synthetically at low temperatures suffered particularly from line-broadening of a magnitude sufficient to spoil resolution of the $K\alpha$ doublet. A well-crystallized, strain-free material was obtained by firing at 1350° C. in nitrogen a pressed plug of magnetite produced by reduction of a Turkey red pigment α-Fe_2O_3. Although sintering occurred in the high-temperature firing the material could be broken down to a powder of suitable particle size without the introduction of any appreciable lattice distortion. One or two per cent of α-Fe_2O_3 was detectable in the material, but the structure-cell constant of 8·394 A. for the Fe_3O_4 itself corresponds well with published data.

The accompanying figure shows in direct comparison parts of the Co $K\alpha$ powder photographs of magnetite at approximately 295 and 95° K. The line-splitting which is manifest at 95° K. for all recorded reflexions, except 800, is consistent with a deformation of the cubic structure cell along a [111] direction, so that the symmetry becomes rhombohedral. Consideration of displacements and relative

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