Short Communications

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Preliminary X-ray crystallographic data for methyl 3-*O*-(1-carboxyethyl) shikimate δ-lactone.

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Optically active methyl 3-*O*-(1-carboxyethyl) shikimate δ-lactone (I) was synthesized by Sprecher & Sprinson (1962) as part of their studies of aromatic amino acid biosynthesis. The exact configuration of the substituents on the carbon α to the carbonyl group in the lactone ring is unknown.

Compound (I) was crystallized by slow cooling from benzene. The procedure yields highly birefringent triclinic laths, elongated along **c**, and frequently twinned on the (100) face. Traces of α and β lie in the (100) plane.

The X-ray crystallographic data were obtained from oscillation and precession photographs (Cu $K\alpha$). The density was determined by the modified density gradient tube procedure (Low & Richards, 1952; Richards & Thompson, 1952).

$a = 11{\cdot}32 \pm 0{\cdot}03$ Å	$\alpha = 101{\cdot}8 \pm 0{\cdot}6°$
$b = 8{\cdot}48 \pm 0{\cdot}03$	$\beta = 99{\cdot}3 \pm 0{\cdot}6$
$c = 6{\cdot}52 \pm 0{\cdot}03$	$\gamma = 102{\cdot}1 \pm 0{\cdot}6$

Space group $P1$

$D_m = 1{\cdot}413 \pm 0{\cdot}005$ g.cm^{-3}.

Molecular weight as determined, assuming two molecules per unit cell, 248 ± 6.

Formula weight for $C_{11}O_6H_{14}$, 242.

No further work on this structure is planned.

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A simple multi-film intensity technique for the precession camera.

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A recent short communication by Williams (1965) described a modified Zoltai screen (Zoltai, 1963) for the Buerger precession camera, which enables any screen setting to be achieved, hence allowing the photography of zero and upper levels. Among other things the screen eliminates the progressive doubling of spots on successive films of a multi-film pack (caused by the wrong crystal-to-film distance) by the non-recording of one reflexion of each pair. Thus intensity data can be recorded much more rapidly, especially for upper levels where the exposure times are longer than for the zero level.

A much simpler method to overcome the disadvantage of partial doubling of spots on a pack of films without any adaptation of the precession camera is to misset deliberately the cassette position so as to produce doubling of spots on all films of the pack. This method works for any level which can be photographed in the ordinary way. The convenience of the method is limited by the spacings of the spots recorded on the films; if the spacings are too small the photographs become difficult to interpret owing to partial overlap of spots with different indices. The method can thus not be used for crystals with large repeat distances. The doubling becomes zero at the limit of the recorded area of the reciprocal-lattice plane, since points there enter and leave the sphere of reflexion at the same time.

For zero-level photographs the film cassette must be displaced towards the crystal, whereas for upper levels the film cassette can be displaced away from the crystal (giving photographs similar to that figured by Buerger, 1964, p. 95). For zero-level photographs a displacement of the film cassette towards the crystal of 0·5–1 mm is usually adequate, if the crystal is not too large. For upper-level photographs the cassette must be sufficiently displaced away from the crystal so that the distance of the film nearest the crystal