*Acta Cryst.* (1963). **16**, 930

## Crystallographic data for an *N*-benzyl-*N*-methylcamphidinium iodide.

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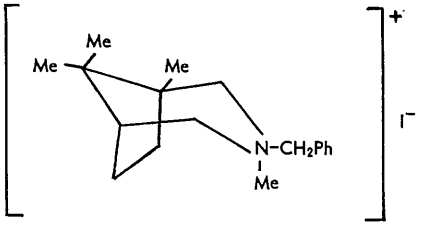
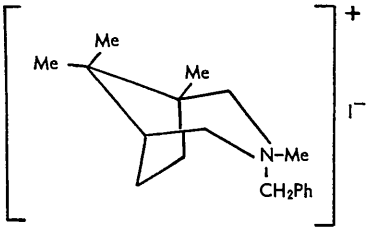
(*Received* 21 *March* 1963)

In the course of some studies on diastereoisomeric pairs of quaternary ammonium salts a (±)-*N*-benzyl-*N*-methylcamphidinium iodide was obtained stereospecifically from the reaction of *N*-benzylcamphidine with methyl iodide in acetone under reflux (McKenna & White, 1963). The well-formed crystals are believed on chemical grounds to be the (±) form of I (the piperidine chair is however almost certainly distorted) rather than the isomeric substance II.




I




II

An X-ray examination yielded the following data. $C_{18}H_{28}NI$ ($M = 385{\cdot}3$) is orthorhombic with

$$a = 20{\cdot}54,\ b = 7{\cdot}91,\ c = 10{\cdot}63\ \text{Å},\ \text{all}\ \pm 0{\cdot}05\ \text{Å}.$$

$\varrho_o = 1{\cdot}432$ g.cm$^{-3}$; $\varrho_c = 1{\cdot}482$ g.cm$^{-3}$ for $Z = 4$. The space group is $Pca2_1$ ($C_{2v}^5$). Systematic absences also permit $Pcam$ ($D_{2h}^{11}$) but, as the molecule possesses no plane of symmetry and the (±) form was studied, this space group with its eightfold general positions can be eliminated.

A partial powder pattern is shown below: the intensities were estimated visually.

No further work on this substance is planned.

| $I$ | $d_o$ | $d_c$ | $hkl$ |
|---|---|---|---|
| 10 | 5·317 Å | 5·315 Å | 002 |
| 8 | 4·733 | 4·720 | 202 |
| 8 | 4·383 | 4·411 | 012 |
| 9 | 3·717 | 3·709 | 312 |
| 4 | 3·542 | 3·487 | 221 |
| 9 | 3·034 | 3·032 | 222 |
| 3 | 2·937 | 2·924 | 313 |
| 5 | 2·689 | 2·699 | 422 |
| 2 | 2·588 | 2·588 | 620 |
| 6 | 2·513 | 2·519 | 614 |
| | | 2·515 | 621 |
| | | 2·511 | 522 |
| 5 | 2·375 | 2·380 | 811 |
| 1 | 2·313 | 2·312 | 802 |
| 2 | 2·169 | 2·173 | 713 |
| | | 2·172 | 531 |
| 3 | 2·100 | 2·100 | 324 |
| | | 2·099 | 604 |
| 3 | 2·031 | 2·029 | 614 |
| 2 | 1·9676 | 1·9683 | 140 |
| | | 1·9666 | 315 |
| 1 | 1·8537 | 1·8544 | 624 |
| | | 1·8535 | 042 |
| | | 1·8528 | 922 |
| 1 | 1·7370 | 1·7384 | 832 |
| 3 | 1·6910 | 1·6914 | 914 |
| | | 1·6906 | 641 |
| | | 1·6900 | 12,0,1 |

### Reference

McKenna, J. & White, J. (1963). *J. Chem. Soc.* In the press.

*Acta Cryst.* (1963). **16**, 930

## Fine angle adjustments. A new use of the Weissenberg goniometer.

By I. Fankuchen, *Polytechnic Institute of Brooklyn, Brooklyn* 1, *N. Y., U. S. A.*

(*Received* 2 *July* 1963)

In many fields of scientific work there is need for methods for adjusting a plane to make a very precise angle with a direction. In X-ray work two obvious examples are in obtaining rocking curves of crystals and in the study of the total reflection of X-rays at glancing incidence. In each of these cases, it is necessary to change the angle between the surface and an X-ray beam by a second or so of arc at a time. In the past, rather fancy equipment has been used for this kind of work: long lever arms generally pushed by micrometer screws. It is the purpose of this paper to show that the equi-inclination Weissenberg goniometer can be used for this kind of work with no modifications whatsoever; this despite the fact that no angular reading on the Weissenberg goniometer or

on the arcs can be made with an accuracy better than 5′ of arc. To illustrate the method a rocking curve (Fig. 1) and a total reflection curve (Fig. 2) will be used.

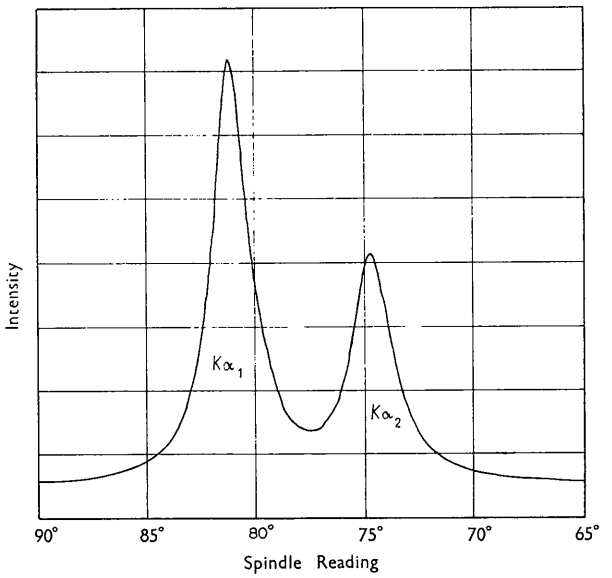



Fig. 1. Rocking curve of calcite in anti-parallel position. Actual angular separation of $K\alpha_2 - K\alpha_1$ is 4·7′ of arc.

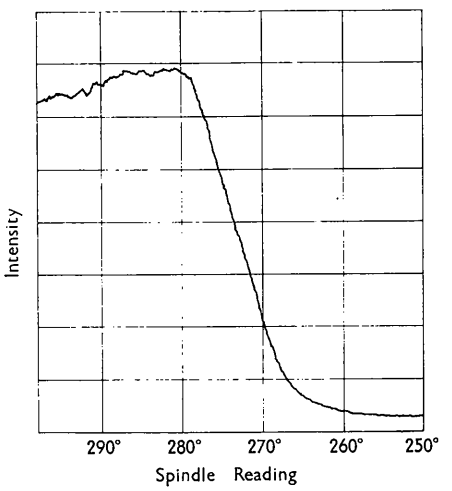



Fig. 2. Total reflection curve of Cu $K\alpha$ from plate glass surface. 10° rotation = 5′ change in glancing angle.

Suppose one wants a rocking curve from a set of planes. Imagine the crystal mounted on a goniometer head on a Weissenberg goniometer so that the set of planes is exactly at right angles to the axis of rotation. Suppose the equi-inclination angle is set at the critical angle, $\theta$, for say $K\alpha_1$. If the crystal is rotated on the spindle of the Weissenberg goniometer, no change will be observed in the reflected ray if it is monitored by some direct reading device. Now turn the spindle so that one arc is in the plane defined by the incident X-ray beam and the spindle axis. Set this arc off by an amount $\Delta\theta$. With conventional goniometer heads, $\Delta\theta$ can be made conveniently as small as 5′ of arc. The angle that the X-ray beam now makes with the set of planes is $\theta + \Delta\theta$; 180° later it would make an angle of $\theta - \Delta\theta$. In other words, a change in the angle that the X-ray beam makes with the set of planes of $2\Delta\theta$ would be accomplished by rotating the crystal 180° on the spindle axis. As the crystal is rotated on the spindle axis, the planes will pass during the rotation through the critical angles for the $K\alpha_1$ and $K\alpha_2$ lines. Fig. 1 illustrates such a rocking curve for a calcite cleavage surface, using Cu $K\alpha$ radiation previously monochromatized by reflection from a calcite cleavage surface. The 'magnification' obtained by this method depends upon $\Delta\theta$. The smaller $\Delta\theta$ the bigger the 'magnification'. In some experiments the 'magnification' has been made large enough for a rotation of 1° on the spindle axis to correspond to 1″ of arc in the change of the angle between the incident beam and the set of planes. The rate of change in angle accomplished by this method is not linear but is close to it in the central part of the 180° range.

This method can obviously also be used to do experiments in total reflection, and such experiments have already resulted in some new findings which will be reported elsewhere. A typical total reflection curve obtained by this method from a plate glass surface is illustrated in Fig. 2. Here the magnification was again not too high; 10° of rotation actually corresponded to a change in glancing angle of 5′ of arc. At 270°, the glancing angle was about 14′.

An interesting by-product of this work is a fast and accurate method of setting a set of crystal planes accurately perpendicular to an axis of rotation. This, in principle, can be done nicely on an equi-inclination Weissenberg goniometer, if one sets the equi-inclination angle to be accurately the critical angle for the $K\alpha_1$ line. If one can do this, then all one has to do is adjust the arcs to maximize the reflected intensity and the set of planes will be automatically perpendicular to the axis of rotation. However, on no commercial instrument can one set the equi-inclination angle accurately enough within a few seconds of arc to permit this procedure to be carried on directly.

Suppose the equi-inclination angle is set a little off from the correct critical angle. If the set of crystal planes is slightly mis-set from perpendicular to the spindle axis, rotation will cause two maxima per 360° rotation. The further off the equi-inclination angle is from being correct, the closer will be the two points in the rotation at which the maxima occur. Ideally, if the critical angle is exactly right for $K\alpha_1$, the $K\alpha_1$ peaks should be separated by 180°. Suppose they are not. Then all one has to do is turn the spindle to the angle setting at which the maximum should have been and tap the equi-inclination angle until the reflection is a maximum. A few such quick adjustments will separate the maxima by 180°. The equi-inclination adjustment can now be locked because the angle will be the critical angle. All that now needs to be done is to adjust the goniometer arcs to constant maximum of reflected intensity. Obviously the same procedure can be used to align crystals *e.g.* on Eulerian cradles.