

Short Communications

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The significance of a stable crystal mounting in precision integrated X-ray intensity measurements.

By W. A. DENNE, *Division of Chemical Physics, CSIRO Chemical Research Laboratories, P. O. Box 160, Clayton, Victoria, Australia 3168.*

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Minute but relatively rapid changes of crystal orientation can lead to significant errors in integrated intensity measurements.

In spite of many recent proposals for modifications and new designs of crystal mountings for single-crystal diffraction work, *e.g.* Cole & Okraskinski (1962), Coppola (1965), Davies & Mathieson (1965), Jost (1966), Kalman (1967),

Carter & Sadler (1969), it appears that the stringent mechanical stability requirements for accurate intensity measurement are not widely appreciated. Indeed, in the analysis of the ACA intensity project (Abrahams, Alexander,

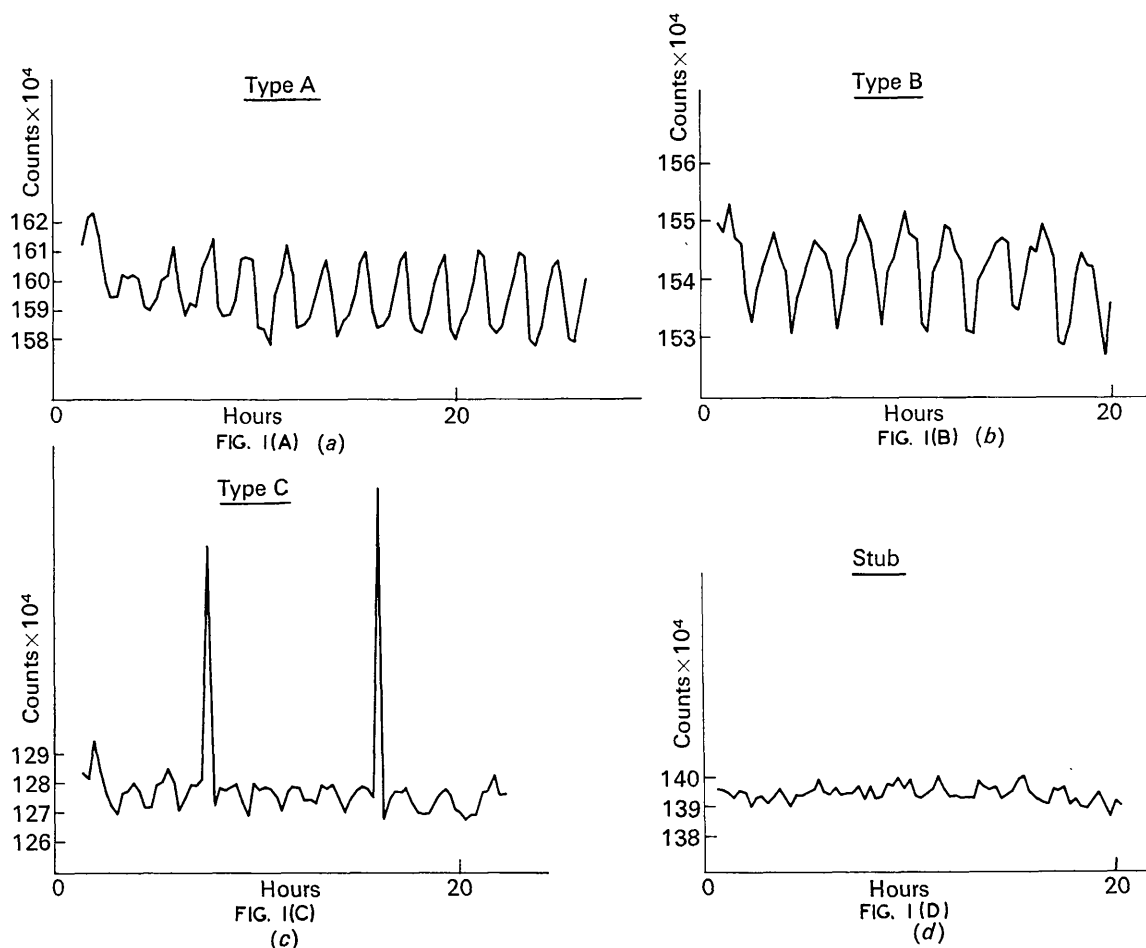


Fig. 1. Plots of integrated intensity against time for repeated measurements of the same reflexion using different mountings and an ambient temperature varying over 2°F . Figs. 1(a) to 1(c) were produced by commercial goniometer heads and Fig. 1(d) by a solid stub of brass. The counting statistical σ in all cases is about 0.1%.

Furnas, Hamilton, Ladell, Okaya, Young & Zalkin, 1967), it was stated with some confidence that in the course of measurements from a given crystal in several laboratories the orientation changed by less than 5 minutes of arc. However, changes of less than 1 minute of arc can cause significant errors in intensity measurements if they occur during the period of the integrating scan. For example, if a change of diffractometer setting causes a goniometer head to take up the slack in the slides with a resulting change in crystal orientation of $1'$, and say this change takes place uniformly throughout a scan of 1° , then the resulting scan velocity will necessarily be 1.7% high or low depending on the direction of the error. Should the change occur abruptly at the peak counting rate, with say a Gaussian profile of $\frac{1}{2}^\circ$ half-width, the resulting intensity error will be almost 4%.

Examination of a selection of arcs of different manufacture using an autocollimator revealed that some had up to 0.1° of slack. The following experimental test was therefore set up to assess this source of error in three different types of goniometer head and a brass stub mounting.

An α -glycine crystal was bonded with Araldite to a glass fibre, which was attached to the four mountings in turn, again using Araldite. The same integrated intensity (not spot value) was then measured repeatedly by ω - 2θ scan for mounting while, as a perturbing influence, the ambient temperature was allowed to cycle through 2°F every two hours. The resulting plots given in Fig. 1 indicate at least 2 to 3% errors for all mountings save the solid stub of brass, [Fig. 1(d)].

From this it is evident that deviations from uniform scan speed due to crystal movement can be a real source of error in precision intensity measurement and no doubt accounts for a significant fraction of the random errors in both the

I.U.Cr. (Abrahams, Hamilton & Mathieson, 1970) and ACA (Abrahams *et al.*, 1967; MacKenzie & Maslen, 1968) intensity projects.

If this source of error is to contribute less than $\frac{1}{4}\%$ in intensity measurements, it is clear from the example quoted that the play in the mounting must be less than about 0.0005° and angular drift rates less than $0.001^\circ\text{.min}^{-1}$. This not only makes great demands on the adhesive used, though Fig. 1(d) indicates that Araldite AY 105/HY 930 acquitted itself well in this series of measurements, but the machining tolerances required to bring most conventional designs of goniometer head up to this standard are almost unattainable. A new design of goniometer head has therefore been developed, details of which will be published shortly.

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The correction of measured integrated Bragg intensities for anisotropic thermal diffuse scattering. A correction.

By K. D. ROUSE and M. J. COOPER, *Materials Physics Division, Atomic Energy Research Establishment, Harwell, Berkshire, England*

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A correction to a previously published result for neutron scattering is given.

The paper of the above title (Rouse & Cooper, 1969) contains an error associated with the integrated thermal diffuse scattering (TDS) intensity for neutrons. Because of the nature of the scattering surfaces for neutrons it is not valid, in this case, to integrate the cross section over the scanned volume $dudvdw$. However, it can be shown (see Waller & Froman, 1952) that, under the conditions defined by the basic assumptions, the TDS intensity has the same form for 'faster-than-sound' neutrons as for X-rays; *i.e.* for faster-than-sound neutrons $1 - e^2(\mathbf{q})$ should not appear in equations (10) and (16) which then become identical with equations (5) and (14) respectively. Under these conditions,

therefore, the Jacobian has no effect on the neutron intensities. A more detailed consideration of the thermal diffuse scattering of neutrons will be given in a later paper.

The authors are grateful to Dr B. T. M. Willis for bringing this error to their attention.

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