

International Union of Crystallography

Commission on Crystallographic Apparatus

Phase II of the I. U. Cr. Single Crystal Intensity Measurement Project

An International Project which will be phase II of the Single Crystal Intensity Measurement Project, SCIMP (Abrahams, Hamilton & Mathieson, 1970), is proposed by the Commission on Crystallographic Apparatus of the International Union of Crystallography. The plan of the phase II project is for each participant (individual or group) to derive for a specific material, α -glycine, a set of experimental structure factors, F_o , as independent of the individual crystal as possible. To this end, corrections for absorption and extinction will be obligatory. The aim of the project is to determine the measure of accord achieved by the participants over a range of crystals, diffractometers and techniques and also to obtain some guide as to the efficacy of experimental procedures for the diagnosis and correction of extinction effects.

The reasons for initiating phase II of SCIMP derive from the results of Phase I, the report on which clearly indicated that, for the material used, D(+)-tartaric acid, the particular characteristics of the individual crystal constituted the major source of error in the derivation of accurate F_o values. The functional trend of the differences between F values for different crystals has identified extinction as the main error factor. There is therefore an obvious need to extend the earlier intensity measurement project to a second stage in which the effects of differences between individual crystals are diagnosed and minimized. To reduce measurements from different crystals to a more or less common base, it is necessary that corrections be applied, not only for simple absorption but also, and probably more importantly, for extinction. Correction procedures for simple absorption are relatively straightforward but there is no single preferred procedure for the estimation of extinction effects. Since theoretical F_c values are dependent on the particular model adopted, reference to such values as a guide or measure of experimental accuracy is questionable. It seems therefore more appropriate to test, as far as possible, *experimental procedures which are intended to diagnose and correct for extinction*.

There are a number of feasible procedures for the estimation of extinction which are essentially experimental. These involve either measurements on the one specimen or measurements on two (or more) specimens – for example:

- (a) with a single specimen,
 - (i) measurements with a polarization analyser, *e.g.* Chandrasekhar *et al.* (1969);
 - (ii) measurements made at two or more wavelengths, *e.g.* DeMarco & Weiss (1962);
 - (iii) measurements of integrated intensity with varying path lengths as achieved, for instance, by rotation about the scattering vector, *e.g.* Willis (1962).
- (b) with more than one specimen,
 - (i) comparison with measurements on powdered specimens of the same material, *e.g.* Stewart & Jensen (1969);
 - (ii) measurements on single crystals of different size, or extinction condition, and appropriate extrapolation, *e.g.* Cochran (1953).

Any other suitable procedure, not specified above, but of an experimental nature may be used.

The Commission welcomes the cooperation of crystallographers in this project. Its successful operation will obviously depend critically on their willingness to participate. It is recognized that the proposed project is not a simple straightforward one but, just as phase I focused attention on the potential gross effect of specimen variation, so phase II could be most valuable and instructive in highlighting the magnitude and significance of intensity-dependent factors in experimental measurements and should assist in assessing the scope of operational procedures for estimating extinction.

Any individual crystallographer or group wishing to have further details should contact the Chairman – Dr A. McL. Mathieson, Division of Chemical Physics, C.S.I.R.O., P.O. Box 160, Clayton, Victoria 3168, Australia.

References

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International Tables for X-ray Crystallography

The Editors of Volume I of *International Tables for X-ray Crystallography* regret that there is an omission in Table 3.4.1 (page 30) of the 1969 edition. In the cubic system the Laue Class of highest symmetry has been omitted. This should be inserted below the Laue Class **m3** as follows:

432
43m
m3m