

Commission on Crystallographic Apparatus

An international project for the calibration of absolute intensities in small-angle X-ray scattering

The importance of absolute intensity measurements in small-angle X-ray experiments has been recognized for many years, and a wide variety of methods have been reported for achieving such calibrations (Luzzati, 1960; Gerold, 1961; Kratky & Wawra, 1963; Damaschun & Müller, 1965; Kratky, Pilz & Schmitz, 1966). Apart from a comparison by Weinberg (1963) of the foil-attenuation method with the gas-scattering method and a comparison by Shaffer (1964) and Shaffer & Beeman (1970) of the data for zero-angle scattering for several gases, there has been no attempt to compare the many techniques. The problem of precision in measurements of absolute intensity, and the need for a comparison of the different techniques for a common standard sample, were discussed at the recent Second International Conference on Small-Angle Scattering of X-rays held in Graz, Austria, in August, 1970. The results of these discussions may be summarized as follows:

I. An international project should be established with the aims of (1) testing the precision of reproducibility and the comparative accuracy of the various calibration techniques in current use, and (2) clarifying the areas of difficulty in absolute intensity calibration.

II. There shall be no attempt to nominate a single absolute intensity calibration technique. Each participating laboratory will use its own preferred technique to carry out measurements on a set of standard specimens to be provided by the project organizer.

III. The secondary standards would be (1) chemically, thermally, and physically stable, (2) unaffected by long exposures to X-rays, (3) easily transported, and (4) easily handled. On the basis of these criteria, liquid samples were eliminated from consideration. Three solid samples were agreed upon as suitable standards: (1) glassy carbon, (2) polyethylene, and (3) cellulose acetate. Each specimen would be mounted in a specimen holder suitable for use in almost all small-angle scattering geometries.

IV. The project organizer would have the responsibility for (1) designing the specimen holders, (2) preparing the instructions to participants, (3) maintaining and distributing the standards, and (4) collecting and comparing the data.

Each participating laboratory will receive for calibration one of each of the three standard samples from the project organizer. The same three samples will be distributed sequentially to all participants in order to assist in separating technique errors from specimen errors. Detailed instructions regarding the kind and quantity of data required to make the comparison of results from different laboratories meaningful will be provided. Basically, data will be required that fully characterize (1) the geometry of the small-angle collimation system, (2) the X-ray generator and the focal spot, (3) the X-ray wavelength and monochromatization, and (4) the X-ray detection system. These data will be recorded on forms provided. Detailed descriptions of the calibration techniques and all raw data will be recorded. Equations and sample calculations for the data reduction must be shown, including the method of collimation corrections if any is used. The final result – the absolute differential X-ray scattering cross section for each sample – will be used to compare the results from the different laboratories. The data from participants will be analyzed with the assistance of L. B. Shaffer and a report prepared for publication. Complete anonymity of all participants will be maintained.

The standard samples and their mounts and the detailed instructions for participation are now being prepared and checked. All interested researchers are encouraged to communicate with the project organizer (address below) for further details.

Robert W. Hendricks
Metals and Ceramics Division
Oak Ridge National Laboratory
P.O. Box X
Oak Ridge
Tennessee 37830, U.S.A.

References

- DAMASCHUN, G. & MÜLLER, J. (1965). *Z. Naturforsch.* **20**, 1274.
GEROLD, V. (1961). *Phys. Stat. Solidi*, **1**, 37.
KRATKY, O., PILZ, I. & SCHMITZ, P. J. (1966). *J. Colloid Interface Sci.* **21**, 24.
KRATKY, O. & WAWRA, H. (1963). *Mh. Chem.* **94**, 981.
LUZZATI, V. (1960). *Acta Cryst.* **13**, 939.
SHAFFER, L. B. (1964). *Absolute X-ray Scattering Cross-Sections of Liquids and Solutions*. Ph.D. thesis, Univ. of Wisconsin.
SHAFFER, L. B. & BEEMAN, W. W. (1970). *J. Appl. Cryst.* **3**, 379.
WEINBERG, D. L. (1963). *Rev. Sci. Instrum.* **34**, 691.

Notes and News

Announcements and other items of crystallographic interest will be published under this heading at the discretion of the Editorial Board. The notes (in duplicate) should be sent to the Executive Secretary of the International Union of Crystallography (J. N. King, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England).

Conference on Framework Silicates and Metals Cambridge (England), 10 December 1971

The Crystallography Group of The Institute of Physics and The Physical Society and The Mineralogical Society are jointly holding a one-day meeting at the Cavendish Laboratory, Cambridge, in honour of Dr W. H. Taylor, who will retire from the position of Reader in Crystallography in September 1971. The meeting will have two sessions on topics which have been of particular interest to Dr Taylor;

in the morning the session will be devoted to *Framework Silicates* and in the afternoon the topic will be *Metals*. A Conference Dinner will be held in St John's College on the evening of 10 December.

Further information and registration forms will be available through the two societies. Accommodation for the nights of 9 and 10 December (if required) will be provided in a College. The Local Secretary (Dr P. Gay, Department of Mineralogy and Petrology, Downing Place, Cambridge, England) will be pleased to give advice to prospective participants.