Report for 1944 of the American Society for X-Ray and Electron Diffraction

THE ASXRED held a joint meeting with the AAAS X-Ray Conference, at Gibson Island, Maryland, August 21–25, 1944. It was the only meeting of the year. The officers of the ASXRED were: L. H. Germer, president; W. H. Zachariasen, vice president; and J. D. H. Donnay, secretary-treasurer. Those of the conference were: L. H. Germer, chairman; P. P. Debye, vice-chairman.

The following papers were presented:

*Dorothy Wrinch, Smith College, Northampton, Massachusetts. **Fourier transforms and structure factor.**

*W. O. Milligan, Rice Institute, Houston, Texas. **X-rays and catalysis.**

Newell S. Gingrich, University of Missouri. Present address, 7903 Chicago Avenue, Silver Spring, Maryland. **Diffraction of x-rays by liquid elements.**

C. G. Shull, The Texas Company, Beacon, New York. **Some experimental studies of small angle scattering.**

*Richard S. Bear, Massachusetts Institute of Technology. **Small-angle diffraction studies on native protein fibers.**

E. J. Bicek, Illinois Institute of Technology. **Long spacing studies of high polymers.**

*Maurice L. Huggins, Kodak Research Laboratories, Rochester, New York. **Photography of crystal structures by optical Fourier synthesis.**

*J. C. M. Brentano, Northwestern University. **The comparative evaluation of x-ray powder patterns.**

James W. Ballard, Mine Safety Appliances Company, Pittsburgh, Pennsylvania. **Routine quantitative analysis by x-ray diffraction, photometric technique and method.**

M. J. Buerger, Massachusetts Institute of Technology. **The precession method of photographing the reciprocal lattice.**

*G. Tunell, Geophysical Laboratory, Washington, D. C. **The two-circle goniometer.**

F. E. Haworth, Bell Telephone Laboratories, Murray Hill, New Jersey. **Some new x-ray and electron diffraction techniques.**

*I. Fankuchen, Polytechnic Institute, Brooklyn, New York. **Microtechniques in x-ray diffraction.**

The council for 1945 is as follows: W. H. Zachariasen, president; David Harker, vice president; J. D. H. Donnay, secretary; C. C. Murdock, treasurer; L. H. Germer, past-president.

Abstracts of Papers

A1. Small-Angle Diffraction Studies on Protein Fibers. RICHARD S. BEAR.—A survey of the small-angle patterns of collagen (tendon), α-keratin (porcupine quill), β-keratin (feather) and clam muscle has disclosed the following: (1) As many as 20 to 40 or more layer lines are observable on the patterns of the fibers studied. The layer line positions lead to the following values for the fiber-axis periods: 95, 198, 640 and 725A, for feather, porcupine quill, collagen and clam muscle, respectively, and for normal dried materials. With collagen, appropriate treatments have succeeded in producing changes in the x-ray diagrams corresponding to period variations in the range 550–680A. (2) In most of the protein fiber diagrams, the larger part of the diffractions occur on the meridian of the pattern and on prominent row lines, one to each side of the meridian. The lateral spacings calculable from the row lines are 34, 83, and about 325A for feather, porcupine quill and clam muscle, respectively. Collagen patterns show no detectable large spacings other than those on the meridian, which appear to be solely the diffraction orders of the fiber-axis period. (3) Collagen is also distinctive in the manner of intensification of the meridional diffractions. The first order is predominant and corresponds to a slowly changing, longitudinal density variation extending over the entire 640A fiber period. This can be seen in electron microscope photographs of collagen fibrils as a banded structure of the same average period length. The other fibers yield patterns whose innermost intense meridional orders are higher than the first (the fourth, third, and fifth, respectively, for feather, porcupine quill and clam muscle). The clam muscle pattern is particularly interesting in this regard, emphasizing all meridional orders whose indices are multiples of five (to the fortieth). This feature is also recognizable in electron micrographs of clam muscle fibrils as a banded structure whose band period is 145A in longitudinal extension, representing a sub-pattern one-fifth the full 725A fiber period. (4) The diffuse wide-angle interferences are, in reality, clusters of very high orders of the large fundamental spacings. Though the wide-angle fine structure is only partially resolvable, it becomes more apparent the smaller are the large periods of a given material. (5) In other respects, the wide-angle and small-angle diffractions seem independent. The collagen large period can be varied 20 percent without detectable alteration in the positions of similarly oriented (meridional) wide-angle diffractions. Porcupine quill and clam muscle wide-angle patterns are, except for fine structure, very closely alike, but their small-angle diffractions are quantitatively very different. The electron microscope studies referred to are the work of F. O. Schmitt, C. E. Hall and M. A. Jakus.

* The papers marked by an asterisk are abstracted below.

A2. The Comparative Evaluation of X-ray Powder Patterns. J. C. M. BRENTANO.—Methods are discussed related to the evaluation of the individual line of a powder diffraction pattern (*a* and *c*), to "improving" the intensity distribution of a line (*b*) and to the way of utilizing the line groups of the chemical or morphological constituents of a mixed powder for evaluating the relative amounts present (*d*). In comparing x-ray diffraction lines of various constituents and at different angles, it is in a general way not justified to assume equal intensity distribution throughout the width of the lines. The typical case is that of lines with a comparatively narrow central peak region and a more-or-less extended low density foot or tail. (a) As a method for obtaining the integrated intensities of a line, it is proposed to measure the x-ray exposure by the intensity of the (111) reflection of the silver forming the photographic image. For not too high densities, this is proportionate to the exposure so that the integrated intensity can be obtained by a single measurement. This can be done by a counter. The characteristics of the method are similar to those of scatter densitometry,[1] but it can be applied in a higher density range and is not sensitive to surface scratches. (b) The uneven intensity distribution, which is unfavorable for evaluation by absorption densitometry requiring a point-to-point conversion, can be improved by recording the line pattern on a film rocked with uniform velocity through a range wider than the central dense part of the line, but smaller than its foot. The peak range of the line is thus broadened, and the ratio between densities in that range and the foot region is improved. The contour of the line is thus made more favorable for measurement with less increase of line width than by the use of wide slits or extra focal settings. (c) A rapid way of carrying out the actual point-to-point evaluation by absorption densitometry was found by superposing on the micro-densitometer tracing of the line pattern that of a stepped wedge, both recorded with the same densitometer setting. The tracing of the steps is used to correlate the densitometer record with a blackening curve by projecting the tracing on the curve. By combining (b) and (c) the point-to-point evaluation of each line can be done in one to two minutes. (d) To determine in the quantitative evaluation of the lines of a mixed powder which lines can be utilized and which are affected by superpositions, a separate pattern is taken for each constituent. The intensities of these lines should be proportionate to those of the corresponding line group of the mixed pattern; superpositions lead to departure from proportionality. A graphical method is given which shows up such lines and permits one to utilize all the remaining lines in evaluating the mass ratios of the constituents present in the powder. The method requires that the relative intensities should not be affected by absorption. This can be obtained with a powder rod of negligible absorption or a flat powder layer conforming with the focusing condition discussed by the speaker.[2]

[1] J. C. M. Brentano, Baxter and Cotton, Phil. Mag. **17**, 370 (1934).
[2] J. C. M. Brentano, Proc. Phys. Soc. **47**, 932 (1935) and **49**, 61 (1937).

A3. Some Techniques for the Growth and Preparation of Micro Specimens for X-ray Diffraction Studies. I. FANKUCHEN.—This paper presented techniques for the recrystallization on a micro scale of materials for x-ray study from both solution and the melt. Techniques were also presented for mounting such specimens both in air and sealed in capillary tubes. The use of a micro-camera for the study of local variation in structure was also discussed. The common feature of all apparatus and techniques was that preparation and mounting were all done under the microscope.

A4. Photography of Crystal Structures by Optical Fourier Synthesis. MAURICE L. HUGGINS.—Crystal structure analysts are making increasing use of electron density projections and Patterson vector projections obtained by Fourier series summations of the x-ray diffraction data. Since the direct summations are long and tedious, various methods have been developed for speeding up the summation process. One of these is a modification[1] of Sir Lawrence Bragg's[2] method of photographic superposition of patterns of light and dark bands, the orientation and spacing of the bands, and the times of exposure of the band patterns being determined by the x-ray data. A suitable set of band patterns for this purpose has been prepared on a roll of 35 mm film. With this, typical electron density and Patterson projections have been made in less than an hour. The electron density projections, so obtained, are true pictures of the unit of structure, magnified 100,000,000 or more times. The atoms are readily distinguished, as in the corresponding contour maps drawn from the results of the direct calculations. It is expected that copies of these rolls of band patterns will be made available soon, at a nominal cost, to others engaged in crystal structure work.

[1] M. L. Huggins, J. Am. Chem. Soc. **63**, 66 (1941).
[2] W. L. Bragg, Zeits. f. Krist. **A70**, 475 (1929); *The Crystalline State* (The Macmillan Company, New York and London, 1934), p. 229.

A5. A Review of the Application of X-Ray and Electron Diffraction Methods to Contact Catalysis. W. O. MILLIGAN.—The results of x-ray and electron diffraction examination of contact catalysts is reviewed from the point of view of (a) identification of compounds in catalysts; (b) compounds formed during treatment or use; (c) small angle scattering; and (d) relation between catalytic activity and (1) lattice constants, (2) intensities, (3) crystal size, (4) orientation. The mechanism of the formation of active catalyst is discussed from the point of view of (a) protective action, (b) solid solutions, (c) amorphous and crystalline states, (d) order and disorder, (e) distortion of crystal lattices. The state of absorbed substances and the potentialities of electron microscopic examination of catalysts are considered briefly. A bibliography of 135 references is included.

A6. The Two-Circle Goniometer in the X-ray Laboratory. G. TUNELL.—The principal uses of the two-circle goniometer in the x-ray laboratory are the orientation and

adjustment of faceted crystals for x-ray diffraction photographs and the preliminary determination of axial elements. Faceted crystals can be oriented and adjusted with the two-wide goniometer more rapidly and easily than any other way. Moreover, the adjustment is very flexible; it can be accomplished by a zone of faces, by a pole face alone, by a ring of faces, and in other ways. There are three types of two-circle goniometer: the Goldschmidt, the Czapski, and the Fedorov. None of these is available in the United States at the present time. If one is to be manufactured in this country, it is imperative that the adjustment head be made interchangeable with the Weissenberg, oscillation, and Laue cameras on the market. The method of measurement and calculation with the Goldschmidt goniometer, which is the most generally useful type in the x-ray laboratory, is described in the following works:

V. Goldschmidt, *Index der Krystallformen der Mineralien* (Verlagsbuchhandlung Julius Springer, Berlin, 1886).
V. Goldschmidt, *Krystallographische Winkeltabellen* (Verlagsbuchhandlung Julius Springer, Berlin, 1897).
E. T. Wherry and Charles Palache, "The Goldschmidt Two-circle Method," a collection of papers in *American Mineralogist*. Vol. 5 (1920).
V. Goldschmidt, *Kursus der Kristallometrie* (Gebrüder Borntraeger, Berlin, 1934).
A. Weissberger, "Laboratory Techniques of Organic Chemistry," Interscience (*in press*).

A7. Fourier Transforms and Structure Factor. DOROTHY WRINCH.—This paper summarizes the properties of Fourier transforms with special reference to their use in crystal analysis. Every distribution has its own characteristic Fourier transform and it is shown how, once it is recorded, it can be used for the determination of its contribution to the structure factor of any crystal in which it is part of the unit of pattern, since the Fourier transform of any lattice of points is also a lattice of points, namely the reciprocal lattice. Among those whose Fourier transforms are recorded are many simple point distributions, such as the vertices of a cube, a tetrahedron, an octahedron, finite point sets on a triangular or a hexagonal mesh, etc., which yield the Fourier transforms of like atoms placed at such point sets when multiplied by the atomic scattering factor. Fourier transforms are also recorded for a number of mega-distributions (comprising large numbers of points) including patterned cube and octahedral volumes and the corresponding shells and surfaces. The application of these transforms to the structure factors of small crystals is discussed. The Fourier transforms of a number of finite continuous distributions through cube and octahedral volumes and shells and on cube and octahedral surfaces are also included.