

## Laboratory Notes

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### X-ray diffraction using combined film and microprocessor techniques

An X-ray diffractometer with a climbing detector has been provided with the option of exchanging the detector with a cylindrical film cassette, and by synchronizing the specimen crystal rotation and the film motion by means of the microprocessor controller, Weissenberg-type and other exposures are easily obtained.

Whereas diffractometer techniques are indispensable for accurate intensity recording, film techniques have the advantage of providing overviews over extended regions of reciprocal space. The two techniques are therefore commonly combined. However, the sample has normally to be transferred between the Weissenberg or precession camera, say, and the diffractometer. Furthermore, low-temperature film cameras are rare, in particular for temperatures below that of liquid nitrogen.

We shall now describe an easy way of combining the two techniques in one instrument. The diffractometer used is a two-axis one, provided with a detector climbing motion in the vertical plane (see Fig. 1 for a sketch). The detector holder is so constructed that it can be exchanged with a film cassette.

Both flat and cylindrical cassettes are available, the latter being particularly useful. With the film cylinder axis vertical (parallel to the sample rotation axis) oscillation exposures can of course be taken in the normal way. Our present

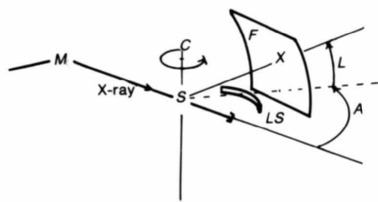


Fig. 1. Schematic view of the diffractometer. *M*: monochromator crystal; *S*: sample crystal; *LS*: Layer screen; *F*: film cassette. When detector is used it is placed at +. Angular motions: *A*: detector-arm motion in the horizontal plane around the vertical diffractometer axis, relative to the direct beam; *C*: sample crystal rotation around the vertical diffractometer axis; *L*: detector lift motion in the vertical plane around a horizontal axis making an angle  $A - 90^\circ$  with the direct beam.

cylinder cassette covers a range of about  $75^\circ$  of  $2\theta$ . By positioning the detector arm *A* (with the cassette) in the horizontal plane any desired range of  $2\theta$  may be covered.

More important, however, Weissenberg exposures may also be obtained. A layer-line screen is installed in front of the film, and the film is moved in the vertical direction synchronously with the sample rotation motion. The vertical motion is performed by means of the climbing motor. By choosing the cassette cylinder axis horizontal in this case one obtains a linear relation (for the equatorial plane) between the rotation angle of the crystal and the film coordinate, quite analogous to the normal Weissenberg case. For non-equatorial layers small deviations from linearity will be observed.

The method is illustrated by Fig. 2, which shows a picture taken with the horizontal-axis film cassette (see figure caption for details). One will see that we are dealing with a one-sided Weissenberg picture, which may be indexed in the normal way. Close examination will show some minor distortions from the normal Weissenberg geometry; for instance, the symmetry lines  $h00$  and  $0k0$  do not form perfectly straight lines. For the purpose of survey pictures this is of no importance.

The new method has the great advantage that the relation between the film and the sample motion may be



Fig. 2. Weissenberg-type ( $hk0$ ) exposure of furandicarboxylic acid obtained on the diffractometer with the cylindrical film cassette, horizontal cylinder axis, cylinder radius 95 mm, detector arm position at  $A = 35^\circ$ , crystal rotation  $200^\circ$ . Monochromated Cu radiation. Layer screen distance 90 mm, slit height 3 mm. Rotation step length of film  $\Delta L = 0.02^\circ$  and of crystal  $\Delta C = 0.08^\circ$ . Total exposure time 50 min. Notice that the vertical direction on the film corresponds to the film lift (*L*) direction, and the horizontal direction corresponds to  $2\theta$ .

chosen at will owing to the microprocessor control of the diffractometer. For instance, one may choose to focus attention on particular regions of reciprocal space, which may then be magnified and even reshaped. Scans other than the Weissenberg type may also be envisaged. The details of the method are being worked out and will be presented in due course (Samuelsen, Moret & Høier, to be published).

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## Crystallographers

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*This section is intended to be a series of short paragraphs dealing with the activities of crystallographers, such as their changes of position, promotions, assumption of significant new duties, honours, etc. Items for inclusion, subject to the approval of the Editorial Board, should be sent to the Executive Secretary of the International Union of Crystallography (J. N. King, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England).*

**William Louis (Bill) Kehl** died on November 4, 1986 after a long illness. He was educated at the University of Iowa where he obtained an M.S. degree in 1941. He was a research scientist at the Gulf Research and Development Laboratories in Harmorville, near Pittsburgh, Pennsylvania, USA. Until his retirement, both Bill and his wife Bettie were very active members of the United States crystallographic community. Bill served as Secretary of the American Crystallographic Association from 1964 to 1969 and as Secretary of the USA National Committee for Crystallography from 1971 through 1973. Bettie was in charge of the Polycrystal Book Service which sold crystallographic books, including all the publications of the International Union of Crystallography, to the North American crystallographic community.

G. A. Jeffrey writes 'I first met Bill in 1950 when I was a Visiting Professor at the University of Pittsburgh, and much appreciated the warm and helpful welcome that he gave me and my family when we returned permanently in 1952. Bill had the then novel idea of incorporating crystal structure analysis into industrial research. At that time, it was not so easy as it is today. It involved some adventurous winter trips over the Alleghenies to Penn State University to use Pepinsky's X-RAC. Bill developed a very successful research program at the Gulf Laboratories with X-ray diffraction

as the principal experimental tool. He was active in helping to organize the Pittsburgh Diffraction Conferences over many years. He was a great farmer and kept his friends well supplied with vegetables in the summer. Bettie's herb garden was equally appreciated, as were their summer picnics. He left Pittsburgh when he retired to live in Louisville, Ohio, and both he and Bettie were missed, particularly by the Pittsburgh crystallography community and their many friends.'

Professor **C. A. Taylor**, formerly Professor of Physics at University College, Cardiff, is the first recipient of the Michael Faraday Award for the furtherance of the public understanding of science. The award has been created by the Royal Society. Professor Taylor receives the award for his outstanding presentations of physics and applications of physics to the real world, aimed at all ages from six-year old primary school children to adults.

Professor **David H. Templeton** and Dr **Lieselotte K. Templeton**, Chemistry Department, University of California, Berkeley, California, USA, are the joint recipients of the third A. L. Patterson Award for their pioneering contributions to the understanding of anomalous scattering of X-rays. During the last ten years they have been engaged in the accurate measurement of anomalous-scattering terms at wavelengths near absorption edges and have co-authored many important papers.

Professor **M. Vijayan** of the Indian Institute of Science, Bangalore, has been awarded the Bhatnagar Prize of the Council of Scientific and Industrial Research, India, for his contributions to crystallographic studies on proteins and complexes of amino acids and other small molecules.

Professor **M. Vijayan** of the Indian Institute of Science, Bangalore, and Dr **K. K. Kannan** of the Bhabha Atomic Research Centre, Bombay, have been elected to the Indian National Science Academy in recognition of their contributions in the field of biological crystallography.

Professor **M. A. Viswamitra** of the Indian Institute of Science, Bangalore, has been awarded the triennial J. C. Bose Medal of the Indian National Science Academy for his work on crystallization and structural studies on oligonucleotides.

## New Commercial Products

*Announcements of new commercial products are published by the Journal of Applied Crystallography free of charge. The descriptions, up to 300 words or the equivalent if a figure is included, should give the price and the manufacturer's full address. Full or partial inclusion is subject to the Editor's approval and to the space available. All correspondence should be sent to the Editor, Professor M. Schlenker, Editor Journal of Applied Crystallography, Laboratoire Louis Néel du CNRS, BP166, F-38042 Grenoble CEDEX, France.*

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*J. Appl. Cryst.* (1987). **20**, 145

## Variable-Temperature Hall Mobility Measurements

A **variable temperature cryostat** for the **HL5200 Hall Measurement System** is now available.

The use of just two temperatures (room and liquid nitrogen) allows Hall measurements to be performed rapidly and routinely. However, for more demanding applications, a variable-temperature cryostat allows Hall measurements to be made over the temperature range 80–400K. The cryostat is software controlled to an accuracy of  $\pm 0.5$ K and a stability of  $\pm 0.05$ K.



The Polaron cryostat for the HL5200.

*Polaron Equipment Ltd, 53–63 Greenhill Crescent, Watford Business Park, Watford, Herts WD1 8QS, England*

*J. Appl. Cryst.* (1987). **20**, 145

## 100 years of apochromatic high-performance optics from Carl Zeiss

In 1886 Carl Zeiss launched the first apochromatic microscope objectives. In these objectives 'free from colour traces', the residual chromatic aberration ('secondary spectrum') present in conventional objectives was corrected by a complex sequence of lens elements. This made it possible to obtain true-to-colour images in the microscope even at very high magnifications.

The physicist Ernst Abbe – the then partner of Zeiss and the subsequent founder of Carl Zeiss Stiftung – only succeeded in designing these complex lens systems after years of painstaking calculations. Completely new types of glass also had to be developed.

The lenses of other optical instruments were also apochromatically corrected by Zeiss at a very early stage. In 1899 Max Pauly incorporated an apochromatic objective in a telescope. And when the Zeiss Tessar lens designed by Rudolph began its triumphal march in photography in 1902, an Aplanat-Tessar 1:10 for true-to-colour reproduction already existed.

In 1938, Plan-objectives with flat fields for photomicrography were designed for microscopes, a development which was to culminate a few years later in the legendary Planapochromats from Zeiss. After 100 years of experience in apochromatic high-performance optics with some outstanding highlights along the way, no end is yet in sight to development in this field.

*Carl Zeiss Oberkochen, Postfach 1369/1380, D-7082 Oberkochen, Federal Republic of Germany*

## Book Reviews

*Works intended for notice in this column should be sent direct to the Book-Review Editor (J. H. Robertson, School of Chemistry, University of Leeds, Leeds LS2 9JT, England). As far as practicable books will be reviewed in a country different from that of publication.*

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**Kristallstruktur und chemische Bindung.** By **A. Weiss** and **H. Witte**. Pp. xii + 396. Weinheim: Verlag Chemie (VCH Verlagsgesellschaft), 1983. Price DM98.00.

The main goal of this volume is a combined representation of the chemical and physical properties of the solid state and of its methods of investigation. The authors, Alarich Weiss and Helmut Witte have succeeded authoritatively in doing this.

The first part gives an introduction to basic crystallography, considering metrics and symmetry of the crystal lattice as well as of the single unit cell, and a survey of traditional models of chemical bonding in the solid state. The main part of the book is a well arranged summary of established techniques of solid-state investigations. The X-ray scattering chapter offers an extensive exposition of photographic methods but lacks a more detailed description of a full single-crystal structure determination as practised today. No mention of direct methods is made. Also missing is the determination of the absolute config-