16.3-04 AREA DETECTOR DIFFRACTOMETER

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We report on a single crystal area detector diffractometer.It is based on monochromatic rotation technique, sequential registration of the Laue diagrams in steps of 0.02 degrees and analysis by means of digital image processing. X-ray quanta reach the entrance window of a single stage 2:1 reducing image intensifier that is fiberoptically coupled to a high sensitivity vidicon-type camera with low dark current.
The image analysis, carried out by a DEC LSI-11 minicomputer,includes the areas between Bragg reflexions and consists of:

a.Data reduction to intensities above background level,
b.peak detection and intensity integration,
c.reflexion position refinement and
d.indexing procedures.

The image is temporarily stored in a MOS mass storage device with a capacity of 196K x 12Bit Data transfer rate is increased by direct access to the video memory.
Tests show that exposure time and picture analysis of a Laue pattern take about 20 seconds.

16.3-05 HIGH-SPEED DATA COLLECTION SYSTEM FROM PROTEIN CRYSTAL USING AREA DETECTOR. By K. Mase, H. Hashizume and Y. Iitaka, Rigaku Corporation, and Faculty of Engineering, Faculty of Pharmacology, Tokyo University, Tokyo, Japan.

Since crystals with large unit cells like protein produce many reflections simultaneously or quasi-simultaneously, a data collection system using an area detector which can cover a broad effective area and distinguish reflections being collected simultaneously, is suitable for speed-up of measurement. However, to best utilize the feature of this detector, the diffractometer and detecting system must be online-controlled, making software that runs this system very important, yet complicated. We have realized a method of high-speed measurement of diffraction data by developing a multi-wire proportional chamber(effective area:64 x 64 mm^2) mountable on the simplest powder diffractometer. The crystal orientation is determined by the position of a reflection point on the detector and the diffraction angle(ϕ) that meets the diffraction condition. The ϕ angle and the position on the detector are estimated about reflection points to be measured and the related information is arranged in order of the ϕ angle. The diffractometer and detecting system are controlled by a dedicated minicomputer so that only the significant information is extracted from the intensity data and transferred. High-speed measurement to collect diffraction data and data processing are performed in parallel, thereby the information on the integrated intensities of many reflections and on backgrounds can be obtained successively. Measurement of a protein crystal(116 x 63 x 87 $Å^3$) was conducted to evaluate the measurement speed and data accuracy. The detector used allowed measurement at a 500 points/hr speed despite its rather small effective area. This measuring system is a practical one that displays satisfactory accuracy as well.

16.4-01 CENTRAL DATA COLLECTION FACILITY FOR PROTEIN CRYSTALLOGRAPHY, SMALL ANGLE DIFFRACTION AND SCATTERING at the Daresbury Laboratory Synchrotron Radiation Source (SRS). By J.R. Helliwell[1,2], T.J. Greenhough[1], P. Carr[1], P.R. Moore[2], A.J. Thompson[2], G. Hughes[2], M.M. Przybylski[2], P.A. Ridley[2], J.E.Bateman[3] J.F. Connolly[3] and R. Stephenson[3], Keele University[1] and Science Research Council, Daresbury Laboratory[2] and Rutherford Laboratory[3], England.

A centralised data collection facility for synchrotron X-ray diffraction experiments on crystals, fibres and solutions of biological macromolecules is nearing completion at the Daresbury Laboratory Synchrotron Radiation Source (SRS). The SRS is based on a 2 GeV electron storage ring with an initial maximum circulatory electron current of 370 mA with a critical wavelength of 3.8Å on the conventional X-ray line and 1.0Å on the wiggler beam line. The X-ray diffraction work-station consists of a vertically focussing platinum coated quartz mirror and a horizontally focussing 10.5° oblique cut Ge(111) triangular perfect monochromator crystal. Intensities at the focus of the optical system are expected to be two orders of magnitude stronger than conventional X-ray sources (rotating anode generators). Carefully controlled tungsten-aluminium slits both before and after the monochromator are available to minimise extraneous X-ray scatter. For protein crystallography an ARNDT-WONACOTT photographic rotation camera is available (see photo) modified for direct, CAMAC based, computer control. Sample cooling facilities over a range - 175°C to + 10°C are available. Electronic area detection facilities based on a TV image intensifier system are under development for the SRS by Enraf-Nonius Ltd., Delft. For small angle diffraction and scattering experiments a different camera is available allowing up to 5 m between specimen and detector providing exceptionally good spectral resolution (order to order and direct beam). A flat multiwire proportional chamber system is being commissioned with a 20 cm aperture and 1 mm wire pitch capable of handling rates up to 300 kHz with a dedicated histogramming memory to provide a real time acquisition facility for dynamic experiments.

Experimental results on a variety of systems will be presented.