16.X-07 A SYNCHROTRON RADIATION CAMERA AND DATA ACQUISITION SYSTEM FOR TIME RESOLVED X-RAY SCATTERING STUDIES. J. Bordas and M.H.J. Koch, European Molecular Biology Lab., DESY, Hamburg, FRG.

Until recently,time resolved measurements of X-ray scattering have not been feasible because laboratory sources were not bright enough and suitable detectors unavailable.New developments in these fields have changed the situation. The combination of the bright X-ray beam produced by the storage ring DORIS (DESY) with suitable optics,detector and data acquisition system has enabled us to obtain time resolved diffraction patterns in the submillisecond range.

To achieve this performance the camera has to deliver a highly collimated and focused beam. The detector and data acquisition system must handle rates in excess of 10^6 photons/second. Facilities for external triggering,synchronization,time slicing and cycling have to be provided.Data reduction "in situ" is required to be able to define at any time the protocol of the next experiment.

We present and discuss the concepts behind the system and expand on some of the solutions we have chosen.The performance of the instrument will be illustrated with examples from recent experiments on biological systems.

Improvements and developments being carried out at present will also be discussed.

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16.X-08 TWO-DIMENSIONAL POSITION-SENSITIVE DETECTORS FOR NEUTRON DIFFRACTION EXPERIMENTS USING BIOLOGICAL MATERIALS. By B.P. Schoenborn, Dept. of Biology, Brookhaven Natl. Lab., Upton, N.Y. 11973, USA.

The use of neutron scattering for biological structure analysis was made possible largely by the development of linear and two-dimensional position-sensitive counters. These detectors simultaneously collect diffraction data over a large area, have a high and uniform counting efficiency with good resolution and positional accuracy. Over the last ten years, such detectors have been developed at Brookhaven (Alberi, Fischer, Radeka, Rogers & Schoenborn, Nucl. Instrum. Methods 1975, 127: 507-523), the Institute Laue-Langevin (Allemand, Bourdel, Roudaut, Convert, Ibel, Jacobe, Cotton & Farnoux, Nucl. Instrum. Methods 1975, 126: 29-42) and Oak Ridge National Lab. (Kopp, Rev. Sci. Instrum. 1977, 48, 383-388). The instruments developed at Brookhaven use charge division with a gas mixture of He^3 and Ar at a pressure of 10 atm to provide a counting efficiency of 80% for neutrons at 1.5 A wavelength. The resolution is 2.8 mm with active areas ranging from 17 x 17 cm to 50 x 50 cm.

The use of such detectors largely overcomes the low flux that characterizes neutron experiments. In small angle scattering experiments, these two-dimensional detectors are used rather like film in X-ray work.

In neutron protein crystallography, the use of such counters controlled by a modern data acquisition system permits new approaches to data collection strategies. Instead of dealing with conventional scans like the θ-2θ scan that provides an integrated intensity as a function of a rotational parameter, the computer linked counter can be used to produce a three-dimensional reflection profile. As the crystal steps ($\Delta\omega$) through a reflection, the observed data is stored for each step in an external memory as a function of extent in 2θ and height (y) of a reflection. A typical array size for each reflection would be $20(2\theta) \times 20(\omega) \times 10(y)$. In this space the reflection will be a three-dimensional distribution with dimensions determined by the basic geometrical conditions like $\Delta\lambda$, crystal size, mosaic spread and beam collimation parameters. Knowledge of these basic parameters will allow a delineation of the reflection from the background and permit therefore an accurate intensity determination. This is particularly important in protein crystallography where high background occurs due to the large incoherent scattering of numerous hydrogen atoms.

16.2-01 CONSIDERATIONS OF EFFICIENCY AND PRECISION IN FOUR-CIRCLE DIFFRACTOMETRY. By William Clegg, Anorganisch-Chemisches Institut der Universität, Tammannstr. 4, D-3400 Göttingen, Fed. Republic of Germany.

The need to write a complete new control program for a Stoe-Siemens four-circle diffractometer has provided us with the opportunity of incorporating some novel features. Of prime importance were high data collection rates, flexibility and ease of use, without loss of precision in either unit cell geometry or intensity measurements. Key points of interest are:-

(1) A simple program command structure, extensive use of default parameters, and the possibility of queuing a sequence of commands for subsequent operation.
(2) Initial reflection search either with or without photographic information.
(3) Considerable enhancement of the Auto-indexing procedure (R.A.Sparks, Crystallographic Computing Techniques (Munksgaard, 1976), p.456) for cell determination.
(4) A choice of reflection centring procedures, with precautions to minimise systematic errors produced by instrument misalignment, backlash, etc.
(5) Matrix and cell refinement with and without symmetry constraints.
(6) Cell reduction, with output designed to assist in locating higher symmetry.
(7) Oscillation photographs about any lattice vector.
(8) Optical determination of crystal measurements and face indices (e.g. for absorption corrections).

(9) A high-speed peak intensity measurement procedure, which is valuable in space-group determination and in selecting suitable reflections for cell refinement before data collection; in some cases, it can also serve as a means of collecting a rough preliminary data set.
(10) Intensity data collection by a profile-fitting technique (W.Clegg, Acta Cryst.(1981) A37, 22) to give high precision at high speed.
(11) Variation of counting times to give approximately constant-precision intensity measurements.
(12) Special optional routines, integrated into the data collection, for azimuthal scans and Friedel pair measurements.
(13) Monitoring of crystal orientation, with automatic corrective action if it changes significantly.
(14) Flexible handling of a multi-purpose reflection list.

Local computing considerations dictated the use of BASIC as the program language on a Data General Eclipse S/250 mini-computer, diffractometer operation being time-shared with structure determination (Fortran programs), plotting, and other BASIC users.

The total diffractometer time required for a crystal, including setting up for data collection, is about 1.5 hours per independent non-H atom, giving a turnround of 2-3 medium-sized structures per week. Final R indices for crystals of medium to high quality are typically 0.03-0.06.

16.2-02 RUBY SPHERES FOR ALIGNING SINGLE-CRYSTAL DIFFRACTOMETERS. By L.D. Calvert, E.J. Gabe and Y. Le Page, Chemistry Division, N.R.C., Ottawa, Canada, K1A 0R6.

A standard crystal for the calibration of single-crystal diffractometers should ideally have the following properties. It should have long-term chemical stability and no phase transition over a wide range of temperature. It should be insoluble in most solvents and not be subject to radiation damage. It should have high diffracting power, even at high 2θ angles and have many symmetrically equivalent reflections. It is most important that the material should be readily available as single-crystal spheres of suitable diameter. Pure α-Al_2O_3 (corundum) satisfies all these criteria except its availability as spherical crystals. However, synthetic ruby spheres (α-Al_2O_3 with small amounts of Cr) are commercially available.

The organizing committee of the present Congress has obtained a supply of such spheres (0.15 mm diameter) to distribute to participants at the Congress and thereafter on receipt of written requests. The cell data for corundum are well known (N.B.S. (U.S.) Circ. 539, 9, 3 (1960)). Cell and structural data relevant to the particular ruby spheres will be given at the Congress and distributed with the spheres.

16.2-03 A NEW MICROPROCESSOR-BASED AXIS POSITIONER. By Kenneth Baldwin, William Huebsch, and Charles Prewitt, Dept. Earth & Space Sci., SUNY at Stony Brook N.Y. 11794, USA

A new, general-purpose, programmable axis positioner suitable for use in x-ray diffractometry is under development. The positioner makes use of a commercially-available Z80A-based single card computer with programmable ROM firmware for long range versatility and adaptability.

An external clock and associated circuitry will be added to provide programmable speeds ranging from 0.1°/min to 6.0°/sec and ramped slew. The positioner is capable of performing incremental and absolute positioning up to a maximum rotation of 360 degrees. When used with a five-phase stepping motor in half-step mode, the resolution for a conventional diffractometer axis is 0.001 degrees.

Communication to and from the positioner is in ASCII via an RS232 serial line at programmable speeds of 110 to 9600 baud. A simple command string structure has been implemented to allow off-line control from a terminal as well as on-line control from a host computer.

By clustering multiple positioners with a communications priority, only a single RS232 serial line is needed to control all axes simultaneously. In addition to conventional diffractometry, this axis positioner concept can be used in any application requiring control of stepping motors, such as for equipment on a synchrotron beam line.

16.2-04 EFFECT OF SCAN TYPE ON INTEGRATED INTENSITY. By. R.G. Hazell, F.K. Larsen, S. Kræmmer, Department of Chemistry, Aarhus University, DK-8000 Aarhus C, and B. Lebech, Physics Department, Risø National Laboratory, DK-4000 Roskilde, Denmark.

For a crystal with large mosaic spread an ω-scan gives a higher integrated intensity than a θ-2θ scan at moderate θ. This has in some cases led us to collect data using the ω-scan technique. In neutron diffraction where the collimation before and after the monochromator allows a fairly large wave length spread the counter aperture is only wide enough to accept all of the diffracted beam at low θ-values. Comparison of ω and θ-2θ data from the same crystal collected at Risø, DK, shows a constant ratio between the two data sets for $0<\theta\leq30°$ but for $\theta>30$ the ratio falls, and our data in the range $30<\theta<55°$ show a linear fall off with θ or with tanθ. The point where the cut off starts and its slope depend on the apparatus, the size of the sample and possibly its mosaicity. A least-squares refinement with two extra parameters to describe this permitted a satisfactory refinement using the ω-data. Sequeira (Acta Cryst. (1974) A30, 839) gives formulae which, if all collimations and mosaicities are known, allow the calculation of the intensity loss for any scan type. The collimators are, however, assumed to give Gaussian intensity distributions which is not fulfilled by our open tubes and only a qualitative agreement with the experiment is obtained. We attempt to use the resolution ellipsoid approach of Lebech and Nielsen (Abstracts, Petten, 1975, p. 467) to illustrate the problem and calculate numerically the loss for non-Gaussian collimators for different scan directions. Failure to take the loss of intensity into account leads to incorrect thermal parameters, unless the θ-2θ scan technique is used and the mosaicity is small or isotropic. The error is worst for neutron data but can also be important in the X-ray case.