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(6) Two Instrumental Systems for Automatic Diffraction Data Collection from Single Crystals

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With the extensive development of high-speed computing methods in crystallography, the strengthening of statistical phase-determining methods for centric structures or projections and the direct anomalous-dispersion method for non-centric structures, and the excellence of proportional and scintillation counter techniques for accurate intensity measurement,[3] the real bottleneck in x-ray structure analysis has suddenly become the tedious, time-consuming operations required for collection of three-dimensional scattering data from moderately to highly complex single crystals. The anomalous dispersion methods[4] in particular render photographic recording obsolete,[5] because high accuracy of intensity measurement (often to within ± 1 per cent) becomes of large importance, and can be achieved only by direct photon-counting techniques.

Several schemes for automatic single-crystal diffractometry have been developed. Two of these, both already under operational testing, are reported here.

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SCADAC, for single-crystal automatic diffractometer and analogue computer, is designed for rapid, accurate x-ray measurements, and includes an internal (analogue) computer which solves Laue's equation by an analogue to Ewald's construction in reciprocal space. The computer requires reciprocal lattice vectors as input; these vectors can of course be determined on the diffractometer. The computer then provides information to servo amplifiers which motor-drive the crystal and scintillation counter detector to positions satisfying a Laue condition. The machine then samples the peak intensity, inserts a proper filter, and scans the entire peak (into the background) an integral number of times sufficient to provide required accuracy. The counts are then printed out at pre-determined intervals of scan angles, and are also totalized, substracted from background, and the integrated intensities printed out, on a paper tape, along with the Miller indices of the reflection, the filter factor and the number of scans. Relay circuits permit the avoidance of measurements at reciprocal lattice points where translational symmetries produce absences; but the symmetry must of course be established in advance.

The diffractometer scans points on one row of a particular reciprocal lattice level, and then moves to the next row at that level. It can also be programmed to scan along any given curve or over any selected area at one level. It is constructed in equatorial mounting, to simplify the analogue computations for non-zero-level lattice planes. Change to a new reciprocal lattice level is done manually. The operating speed is about one reflection per minute. One machine does the work of approximately a dozen standard single-crystal goniometers using photographic recording; and of course the accuracy of intensity measurement is much higher than is achievable photographically.

SCADAC, located at Penn State, includes regulated power supplies: an adjustable constant-potential HV source, and an x-ray filament supply which is regulated to produce constant tube current. The entire system is mounted on four standard relay racks. The power, x-ray and mechanical systems occupy the two central racks; and the two outer racks, carrying the computer and counter circuitry, are at angles to form an open U.

A second type of automatic machine has been designed and constructed for single-crystal neutron observations at Brookhaven National Laboratory.[6] The diffractometer is a miniaturized neutron goniometer previously described.[7] This has been fitted with separate motor drives for both the crystal and counter-arm shafts; and geared to these shafts as well are very accurate digitalized angle counter devices, which electrically signal the angular settings of the shafts. The digitalizers provide five-decade signals for each angle.

Instructions for angle settings are provided to the machine in the form of five pre-set decade switch settings, corresponding to the crystal or counter positions for a given Laue peak, for each digitalizer. The crystal and counter-arm motors turn these shafts until the five-decade angle digitalizers correspond in settings to the pre-set counters. The crystal and counter then scan the reflection, and all measurements and settings are automatically recorded on paper tape, as in SCADAC.

The pre-set counter control system for the machine at present consists of ten panels, each carrying the ten decade switches necessary for a single reciprocal lattice node. Ten reflections can thus be programmed at one time. This is adequate for neutron diffraction, where measurement of one reflection requires from one to several hours. These panels will eventually be replaced by punched card or paper tape control. Then one control unit can program a battery of goniometers.

The Brookhaven system is preferable for neutron studies, because of the necessarily slow rate at which neutron measurements can be accomplished. For x-ray diffraction a machine of the SCADAC type seems preferable, and one machine will provide enough data for a sizable laboratory.

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[3] W. Parrish and T. R. Kohler, Rev. Sci. Instr. 27, 795 (1956).

[4] R. Pepinsky and Y. Okaya, Proc. Nat. Acad. Sci. U. S. 42, 286 (1956).

[5] Arguments over the relative superiority of direct photon counting *versus* photographic recording plus photometering are also obsolete.

[6] B. C. Frazer, F. Langdon and R. Pepinsky, Paper No. 43, Fourteenth Annual Pittsburgh Diffraction Conference, Nov., 1956.

[7] R. Pepinsky and B. C. Frazer, Rev. Sci. Instr. 26, 402 (1955).