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Automatic Single-Crystal Diffractometry for X-rays and Neutrons

During the Sixth Congress of the International Union of Crystallography in Rome, September 1963, the Commission on Crystallographic Apparatus held two Open Sessions on 'Automatic Single-Crystal Diffractometers for X-rays and Neutrons'. Three invited lectures were devoted to the X-ray and three to the neutron technique. Not all texts are available but, for the four which follow, the authors have been kind enough to permit presentation as a group. In this form they provide a valuable assessment of the contemporary state of development in single-crystal diffractometry. They have been prepared for publication by the Chairman of the Commission on Crystallographic Apparatus (Dr A. McL. Mathieson), and the Editors of *Acta Crystallographica* are grateful for his help. Abstracts of the two papers for which the full text is not available will be found on p. A 152 of Volume 16 of *Acta Crystallographica*.

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Analogue and Digital Single-Crystal Diffractometers

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Automatic diffractometers which have been described to date fall into one of three classes: those in which an automatic systematic search is made for the Bragg reflexions, those in which the angular positions of the crystal and detector axes are computed by means of an analogue computer which is generally an integral part of the diffractometer, and those in which these positions are computed by a digital computer. The first two types of instrument are commonly referred to as analogue diffractometers while diffractometers of the third type are called digital. In this last type the computer may or may not be directly linked to the diffractometer control circuits; in either case digital shaft positioning methods are used, similar to those employed in automatic machine tool control. Within all three classes of diffractometer a variety of different geometrical arrangements is possible.

The actual measurement of a reflexion in all these instruments requires the determination of the integrated intensity and of the background level near the Bragg peak. Stationary or moving crystal methods may be used.

The relative merits of automatic diffractometers must be assessed according to a variety of different considerations.

Introduction

The types of problem investigated by means of X-ray diffraction studies on single crystals cover a wide range. They include such widely divergent subjects as, on the one hand, very accurate electron density determinations on small molecules whose structures are already known to a high degree of approximation, and on the other hand the determination of the structures of very large completely unknown biological molecules; they cover high and low temperature studies, the investigation of thermal diffuse scattering, molecular weight determinations and investigations of crystal imperfections. For the thirty years or so during which the vast majority of crystallographic studies were carried out almost exclusively by photographic methods, a large number of special X-ray cameras were evolved and many different techniques were devised.

Developments in the reliability and sensitivity of X-ray detectors and associated circuitry, in techniques of automation and in the processing of experimental data by means of digital computers have stimulated the desire to replace X-ray cameras and densitometers by automatic diffractometers using quantum counters as detectors. There has been much mutually beneficial interplay between such developments in X-ray diffraction and in neutron diffraction where photographic techniques are more difficult and where these were developed relatively late in the growth of the subject.

As there is no universal photographic technique which will suit all crystallographic problems so there can be no one type of automatic diffractometer which is 'best' for all purposes. It should, perhaps, be said here that while much valuable work has been done, and continues to be done with non-automatic X-ray diffractometers, manually operated instruments repre-

sent only an interim stage; in the long run, a large degree of automation is essential if more than a very small volume of work is to be carried out. Until recently, there has been justifiable doubt whether such automation was basically capable of replacing the crystallographer's judgement and decision at every step of the process of data collection: a considerable number of automatic instruments have been described, many of which were summarized recently (Arndt & Phillips, 1963); however, relatively few results obtained with such instruments have so far been published in the literature and there have been disturbing rumours of lack of reproducibility and precision in those investigations which were carried out. The problems arising in diffractometry are now, however, beginning to be understood in detail and some recent results should prove reassuring. Thus, for example, Abrahams (1964) has shown that structure factors can be determined to a real accuracy of about 1% by high precision automatic X-ray diffractometry; Levy, Agron & Busing (1963) have demonstrated similar accuracy in neutron diffraction; data on protein crystals have been collected by means of analogue diffractometers at a rate of some 2000 reflexions per week (Blake & Phillips, 1962), obtaining an accuracy limited by counting statistics to about 6–9% in F (Blake, Fenn, North, Phillips & Poljak, 1962). Lonsdale (1963) has quoted similar measurements made by Phillips (unpublished) which have established that intensity measurements on proteins using many different crystals can be made to have a consistency at least as good as photographic measurements, with a great saving in time. Similar measurements on protein crystals have been made on the automatic digital diffractometer at Urbana, Illinois which is of the Buerger type (Buerger, 1960) (Dickerson, private communication). Small molecule X-ray structure determinations are being carried out routinely by automatic analogue diffractometers (Dunitz, private communication; Cooper, Saunderson & Watson, 1963) and on digital diffractometers (Macintyre, private communication); at least one structure determination has been reported (Geller & Katz, 1962) in which the data were collected on the interesting automatic diffractometer designed by Bond & Benedict (1955).

It is the object of the present paper to examine the considerations which determine the design of automatic X-ray, and to some extent neutron, diffractometers, and to discuss the relative merits of some of these different designs.

Basically, whatever the type of diffractometer adopted, it has to provide facilities for two separate functions, the setting of the crystal and of the radiation detector to the correct positions for each reflexion in turn and the measurement of the reflexions. The automatic measurement of a given Bragg reflexion consists in the recording of the integrated intensity as the crystal is swept through the reflecting range and of the true local background. The counts received during these parts of a measuring cycle can readily be recorded on a read-out scaler. The only variation in technique consists in keeping the crystal stationary during the measurement of the reflexion, integration being supplied by illuminating the crystal by convergent radiation from an extended uniform source. (Cochran, 1950; Furnas & Harker, 1955; Cowan, Macintyre & Werkema, 1963). It is in the setting of the crystal and detector that three basic approaches are possible. For any geometrical arrangement of the diffractometer, the crystal and the detector between them must be capable of rotational movement with three independent degrees of freedom while the crystal is constrained to remain in the incident beam and the detector to point at the crystal.

It is possible to change the crystal and detector settings in such a way that all possible combinations occur in turn, a special measuring procedure being invoked whenever a Bragg reflexion is detected. In a diffractometer of this type, no knowledge of the unit-cell parameters or even of the initial orientation of the crystal need be assumed since a search is made of the whole of reciprocal space for the Bragg reflexions. The only practical instrument of this type which has been constructed (Bond & Benedict, 1955) was designed as an equi-inclination instrument; it was set manually for each reciprocal lattice level in turn, the automatic search for reflexions being made within the levels. Even here it was found that the complete coverage of the levels was very uneconomical in time, and for this reason this type of instrument has not been developed further to date.

In the other two types of diffractometer it is necessary to solve the equations which define the angular positions of the crystal and detector axes for all those reflexions which it is desired to measure. These equations relate trigonometrical functions of the angles to the indices of the reflexions and the unit-cell parameters of the crystal; they can be solved in one of two ways. In the first method, the settings are computed by an external digital computer and recorded on punched paper tape or cards; these settings can then be read into the control circuitry of the diffractometer. If the diffractometer is directly linked to a digital computer, the intermediate storage media are of course dispensed with. With off- or on-line digital control, the digital shaft positioning itself is carried out by methods akin to those used in machine tool control.

Alternatively, an analogue computer can be used which conveniently forms part of the diffractometer itself. Once the crystal is aligned, only the unit-cell dimensions need be set up; the control circuit then permutes the indices automatically. The analogue computer can consist of a system of slides which represent the reciprocal lattice axes. A system of linkages converts the linear translations along these slides into rotations about the origin of the reciprocal lattice and maintains the crystal parallel to this

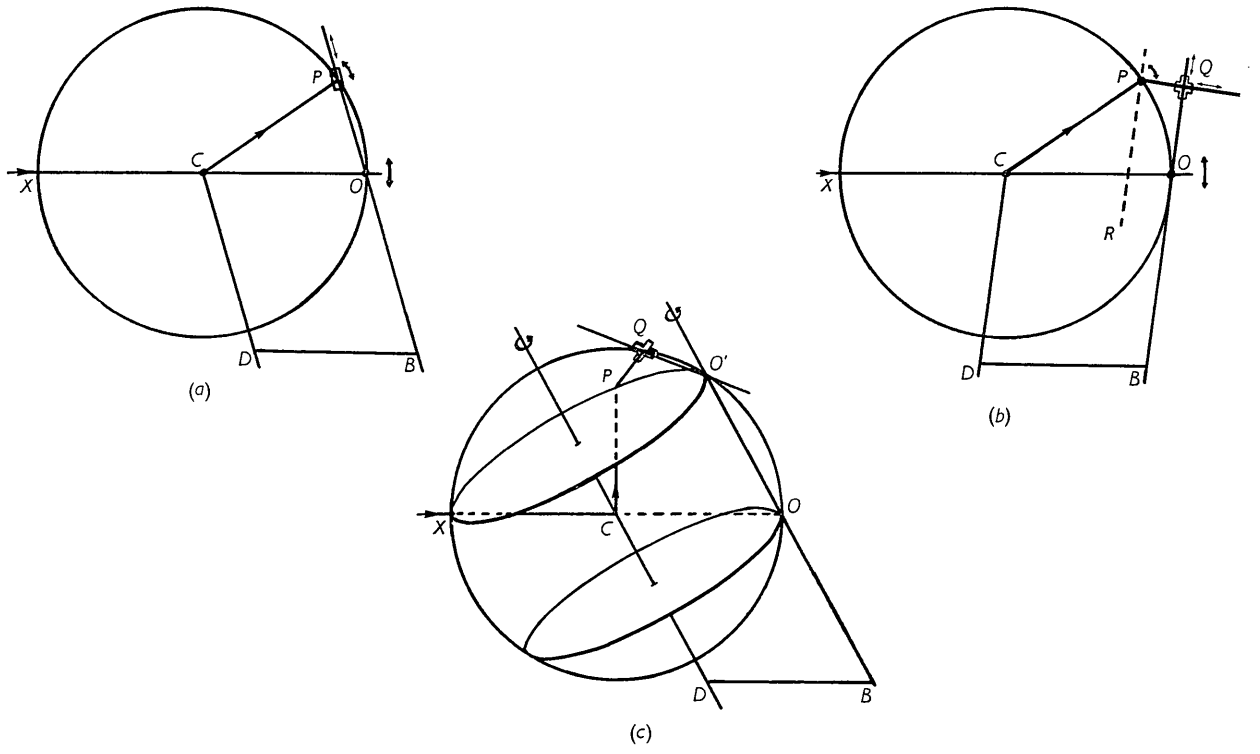

Fig. 1. Linkage system in a mechanical analogue linear diffractometer. (*a*) Scan of the central reciprocal lattice row $h00$ (POB): A reflexion occurs whenever $OP=ha^*$; the carriage P moves along POB; the counter arm CP is pivoted at C and on the carriage P; POB is pivoted at O; the crystal shaft CD is kept parallel to POB. (*b*) Scan of a non-central reciprocal lattice row $hk0$ (PR); $PQ=kb^*=$constant for the row as Q moves along QOB. (*c*) Measurement of an upper-level reflexion hkl (P) on the equi-inclination level XPO': $OO'=lc^*$; as $O'Q=ha^*$ and $QP=kb^*$ are changed P moves around the circle XPO'; CD is kept parallel to OO' and inclined at an angle $\sin^{-1} lc^*/2$ to the horizontal; the rotation about the axis CD is kept equal to the rotation about OO'.

analogue reciprocal lattice. A number of essentially similar linkage systems have been used by Mathieson (1958), Arndt & Phillips (1959, 1961), Ladell & Lowitzsch (1960), Potter (1962), Harris (1963) and Ladell & Cath (1963). Fig. 1 illustrates the way in which one such system is used to bring a reciprocal lattice point P on a central row (*a*), on a non-central row in the zero level (*b*), and on an upper level (*c*) into reflecting position.

It could be argued that there is no basic difference between the two types of diffractometer: if an analogue diffractometer on the one hand and a digitally controlled diffractometer linked to a digital computer on the other hand were enclosed in two black boxes, it would be impossible to differentiate between the two installations from the way in which the input information (lattice parameters and initial orientation) and output information (measured intensities) were communicated to and from the black boxes.

Nevertheless, the two methods of control, analogue and digital, produce two quite distinct types of diffractometer. An analogue-controlled instrument is essentially a fixed-program machine in which the automatic sequence, and especially the sequence in which the reflexions are measured, is determined by 'hardware', that is by mechanical or electronic sequence controllers. A digitally controlled machine, or more specifically, an instrument controlled by a punched tape or punched card program, permits a greater flexibility in use, in that the individual stages of the setting and measuring operations and their sequence can be varied more readily, simply by the type of program which is fed into the machine. In addition, the setting method, in general, determines the strategy of data collection. Once the necessary circuitry for reading in settings from punched tape, *etc.*, has been provided, it is very little more expensive or complicated to set three or more shafts than to set two: analogue 'hardware' has to be individually designed and constructed for each crystal or detector shaft. It is for this reason that in automatic analogue diffractometers, whether employing mechanical methods such as those mentioned above, or using an electrical analogue computer (Drenck, Diamant & Pepinsky, 1959), it is usual to measure reflexions level by level, the geometrical arrangement being like that of a Weissenberg camera so that only two shafts have to be set within each level, the third shaft in

some cases being set by hand when changing to the next level (Table 1; the nomenclature for the axes is that employed by Buerger, 1960).

Table 1. *Geometrical arrangements*

1. Weissenberg geometry

μ, ν	Constant within a level	
ω, Υ	vary for each point	
Normal-beam	$\mu = 90°$	$\nu = \sin^{-1}\zeta$
Equi-inclination	$\mu = -\nu$	$\nu = \sin^{-1}\zeta/2$
Flat-cone	$\mu = \sin^{-1}\zeta$	$\nu = 90°$

2. Normal-beam equatorial

	$\varphi, \chi, \omega, 2\theta$
Symmetrical	$\omega = \frac{1}{2}.2\theta$
Asymmetrical	$\omega \neq \theta$

Design factors

The suitability of a particular instrument for a given application must be judged mainly on the following points, not necessarily in the order given:

1. Accuracy of intensity measurement.
2. Speed of operation, *i.e.* number of reflexions measurable per hour.
3. Maximum angular range and number of accessible reflexions.
4. Amount of manual intervention needed.
5. Accessibility of specimen for the provision of high and low temperature attachments.
6. Availability of a digital computer for data processing and program tape preparation.
7. Versatility, *i.e.* adaptability for different types of measurement.
8. Reliability and ease of servicing.
9. Initial cost.

Some of these points will now be examined in detail.

Accuracy

Not all diffractometer users require the same degree of accuracy of intensity measurements, especially since the demands of accuracy necessarily conflict with the requirements of speed and convenience in use. For problems such as accurate electron distribution determinations, and in the use of anomalous dispersion techniques, the maximum possible accuracy is required: a reliability of 1–2% in the corrected structure factors is often demanded, though not as frequently realized today. In other problems, diffractometer methods are preferred to film techniques by virtue of greater speed and convenience: intensity measurements to 10% are then often sufficient.

Uncertainties in intensity measurements come under the following headings:

1. *Setting accuracy*

The crystal and detector must, of course, be set with sufficient precision to allow accurate intensity measurements to be made. The required precision is discussed elsewhere by Arndt & Phillips (1957) and Arndt (1963*a*) and on page 1190 of this issue by Abrahams (1964). The conclusions are that, if moving crystal methods are used in the measuring cycle, the various shafts need not, in general, be set to better than 0·1°: however, it is necessary to know the lattice parameters of the crystal to a high precision, and since it is desirable to measure them on the diffractometer it should be possible at least to set the shafts to 0·02° by hand while the lattice parameters are determined.

The convergent beam method demands a higher setting accuracy than do moving crystal methods; it may be necessary to resort to special peak-searching techniques when this method is applied to small crystals (Cowan, Macintyre & Werkema, 1963).

2. *Counting statistics*

A thorough analysis of the statistical problems of measuring an X-ray reflexion in the presence of a background has been given by Mack & Spielberg (1958). It should merely be noted here that attempts to achieve good statistics can lead to so long a time being spent on each measurement that the real accuracy of the results is actually reduced by virtue of drifts in X-ray output and detector and detector circuit performance. A preferable approach is to collect several complete sets of data, with somewhat relaxed statistical precision on each, such measurements being made on a number of different crystals.

3. *Stability of source and detectors*

Some of the errors due to these causes are discussed by Abrahams (1964). With suitable precautions they become negligible.

4. *Polychromatic radiation*

Methods of minimizing the effects of white radiation by the choice of the optimum scan and by appropriate filter technique are discussed in the following paper by Alexander & Smith (1964). A balanced filter procedure particularly suitable for counter diffractometers has been described by Young (1963). Ladell & Spielberg's analysis (1963) appears to argue the case for crystal-reflected radiation. The use of such radiation, however, entails the employment of awkward polarization corrections (Whittaker, 1953; Azaroff, 1955, 1956; Bond, 1959; Levy & Ellison, 1960), which, moreover, for work of the highest precision, depend upon the degree of perfection of the monochromator crystal. In addition, monochromators, especially plane crystal monochromators, are known to produce a non-uniform beam and their use for accurate work, therefore, demands a very precisely centred, spherical or at least cylindrical crystal. Where such samples are available, and where the unwanted background is due principally to

polychromatic radiation, crystal monochromators will be the first choice. With less regular samples, balanced filters are probably preferable; in cases where a high background is caused by crystal disorder, the small reduction in the background produced by eliminating the white radiation is not worth the loss of intensity due to monochromators or balanced filters.

5. *Absorption and extinction*

Furnas (1957, unpublished) has pointed out the possibility of making experimental corrections for absorption effects by azimuthal rotation of the crystal; Willis (1963) uses a similar rotation for extinction corrections in neutron diffraction. Such variation of the azimuth is possible only with diffractometers which have more than the minimum number of independent rotations (Willis, 1962*a*, *b*).

6. *Double reflexions*

Intensity errors can arise from multiple Bragg scattering (Renninger, 1937; Willis, 1963; Cole, Chambers & Dunn, 1962). Such double scattering always occurs in instruments with equi-inclination geometry (Yakel & Fankuchen, 1962). In this connexion, Fig. 2 shows that if a normal-beam equatorial diffractometer is operated in the usual mode in which the plane of the χ-circle bisects the angle between the incident and diffracted beam (Furnas & Harker, 1955), the reflexion P which is measured in the equatorial plane lies in fact on an equi-inclination layer whose trace on the equatorial plane is AP; with an orthogonal lattice a point Q on the zero layer whose trace is BO will also lie on the sphere of reflexion, where PQ is the normal to the parallel layers.

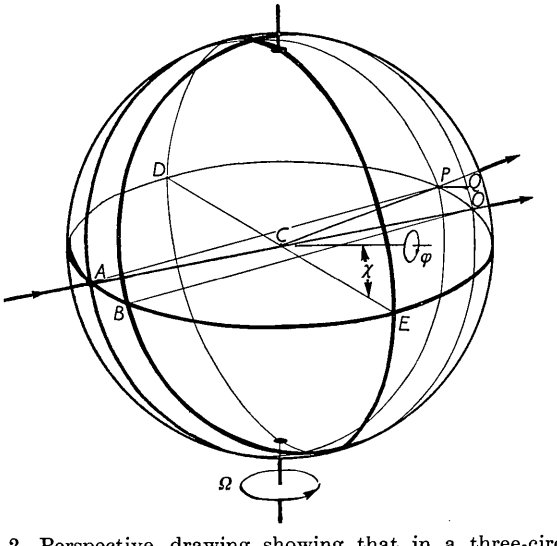

Fig. 2. Perspective drawing showing that in a three-circle diffractometer the φ axis is an equi-inclination axis. The equi-inclination and the zero levels intersect the equatorial plane in AP and BO respectively. The plane of the χ-circle intersects the equatorial plane in DE. The angles ACD and DCP are each equal to $90°-\theta_{hkl}$. For an orthogonal level P, hkl, and Q, $hk0$, are on the sphere of reflexion simultaneously, where PQ is parallel to the φ axis and perpendicular to the two levels.

This relationship is brought out more clearly by a stereographic projection on to the χ-plane (Fig. 3).

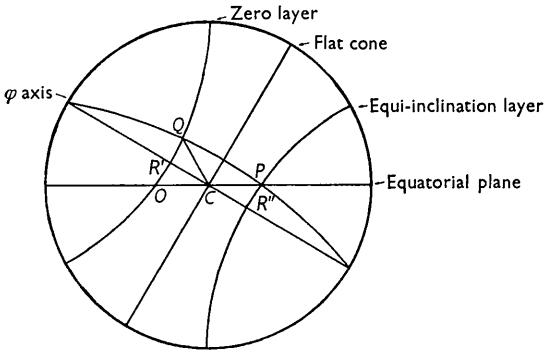

Fig. 3. Stereographic projection of Fig. 2 on to the χ-plane. If a point R on the flat-cone level which is projected at C lies on the sphere then points R' and R'' on adjacent levels lie approximately on the sphere if the lattice is orthogonal.

If it is essential to avoid double reflexions, as in high precision neutron diffraction work where their effect is more serious than in X-ray crystallography, it is necessary to depart from the symmetrical mode of operation, and thus from equi-inclination geometry. The angle which the χ-plane makes with the incident beam must then be chosen so as to secure values of the azimuth, that is of the angular rotation around the scattering vector, so that no other reciprocal lattice points lie on, or very near, the sphere of reflexion. Zocchi & Santoro (1963) and Santoro & Zocchi (1964) have examined this problem and developed computer programmes which determine the optimum value of the azimuth.

Speed of operation

The speed of operation of an automatic diffractometer is largely determined by the time spent on each reflexion in order to secure a statistical precision adequate for the problem in hand. The time required for setting the instrument between reflexions should be negligible compared with the counting time; this can be achieved only with relatively fast slewing speeds: in the writer's opinion this should be at least 10° of arc per second for multi-circle instruments and a comparable quantity for other types.

Some shaft-setting methods used for diffractometers are capable of prolonged and reliable operation only at speeds considerably slower than the above.

One way of increasing the output of a diffractometer consists in measuring more than one reflexion at a time by employing one of two methods suggested by Phillips (1964; see also Arndt, North & Phillips, 1964). Using the equiinclination geometry (Fig. 3) reflexions hkl and hkl' at P and Q, respectively, on levels symmetrically placed on either side of the flat-cone level

can always be measured simultaneously. In Fig. 3, these two levels are the equi-inclination and the zero levels, but this need not be the case. Alternatively, if a reflexion in the flat-cone level itself is brought into the reflecting position, corresponding points on adjacent levels are also on the sphere of reflexion

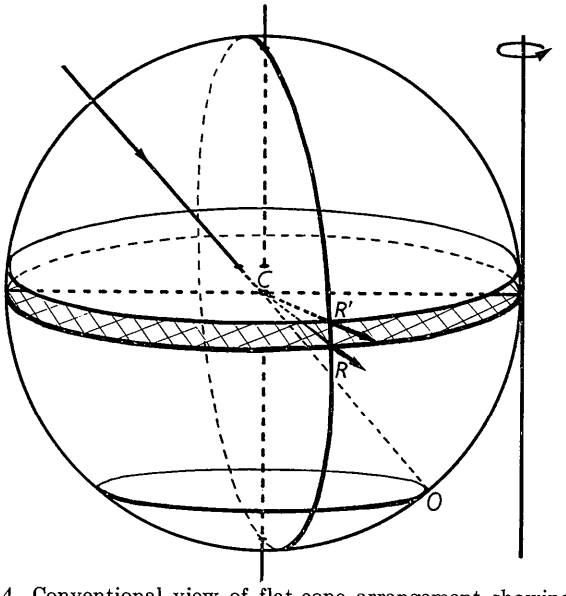

Fig. 4. Conventional view of flat-cone arrangement showing that the arc through R and R' is parallel to the oscillation axis. R' is here on a general level, while in Fig. 3 it was shown on the zero level.

at the same time to a close degree of approximation for large unit cells. This is illustrated in Fig. 4 which is a more conventional representation of the flat-cone geometry. Three-counter attachments for the linear diffractometer (Arndt & Phillips, 1961) are in successful operation at the Royal Institution, London, and double and multiple counter attachments for a four-circle diffractometer (Arndt, 1963*b*) are under construction (Arndt & Phillips, to be published).

Angular limitations

In instruments of the 'Weissenberg' type, reflexions which lie on or near the crystal oscillation axis cannot be measured by the usual measuring cycle. In addition, reflexions on upper levels near this axis cannot be brought into the Ewald sphere at all when normal-beam or flat-cone methods are used. Somewhat less serious are restrictions imposed on the maximum Bragg angle attainable (*e.g.* $\theta_{max}=32{\cdot}5°$ for our linear diffractometer, which is about equal to the maximum Bragg angle of the Buerger precession camera) since such limitations can be overcome, at least in principle, by the use of harder radiation.*

In normal-beam equatorial instruments angular limitations are usually much less severe and can be overcome to a very large extent by employing an asymmetrical setting (Willis, 1962*b*; Wooster & Wooster, 1962).

Amount of manual intervention required and versatility

Analogue instruments are usually fixed-program instruments in which the path through the reciprocal lattice is determined by hardware. Manual intervention is required in order to intersperse the record with measurements of reference reflexions, to change the crystal rocking range, or to insert or remove attenuating filters, or even to proceed to another reciprocal lattice level. All such actions can readily be brought under program control in a digitally controlled instrument.

The amount of manual intervention can be still further reduced by controlling the diffractometer directly by means of a computer, which can then make the decision as to when action is required. Such systems have been described and discussed by Bowden, Busing, Edwards, Hodgson & Mills (1963), Gerhard & Katz (1963), and Cole, Okaya & Chambers (1963).

It is possible that all automatic X-ray diffractometers of the future will be directly controlled by computers. However, at present, not enough experience has been obtained with off-line digitally controlled instruments to enable one to specify precisely what should be the contingencies upon which the computer should act and the full potentialities of an on-line installation are far from being realized. The main advantage of such a linkage at present is that it avoids the use of punched paper tape or cards for the two-way passage of information between computer and diffractometer. These media and the equipment needed to read and record the information on them are at present probably the greatest source of unreliability of the whole system.

It is possible so to design an automatic diffractometer installation that no modifications are necessary to convert from punched tape or punched card control to direct linkage to a computer. It is merely necessary to break down the different operations which the instrument is called upon to perform into the simplest steps and to provide an order or instruction for each step. Thus, for example, a measuring cycle consisting of two background measurements with stationary crystal and detector and an integrating scan with moving crystal requires three separate orders: after the completion of each step, the installation produces a standard signal which results in the next order being read in. The diffractometer installation thus has three channels for communication with peripheral equipment: an input channel along which it receives its orders, an output channel along which the numerical

* Restriction of the θ range is a function of the particular mechanical design. Other designs *e.g.* Mathieson (1958), Ladell & Lowitzsch (1960) permit a closer approach to $\theta=90°$. (Ed.).

output of data is passed and a signal channel in which completion of an operation is indicated. These three channels can readily be connected to the output, input and interrupt channels of a time-sharing digital computer.

Diffractometers with appropriate order codes have been described by Abrahams (1962), Cowan, Macintyre & Werkema (1963), Arndt & Willis (1963) and Arndt (1963*b*). Such instruments, even when used off-line, have a much greater versatility than fixed-program installations. This very versatility, however, makes such instruments more difficult to use and more experience is necessary to take full advantage of their potentialities. Where only routine data collection is required, the additional facilities of programmed diffractometers are often not needed.

Accessibility of specimen

Other factors being equal, the amount of space near the crystal for the mounting of high and low temperature attachments is obviously greater the simpler the movements required of the crystal (and thus, necessarily, the larger the angles through which the detector must move). It follows that normal-beam and flat-cone 'Weissenberg' arrangements are preferable when the mounting of such attachments is a prime consideration.

Availability of a computer

Any modern structure determination requires the availability of an electronic computer. If the amount of data collected is such as to warrant an automatic diffractometer at all, then a computer is virtually essential for checking and processing the experimental data. However, it is quite possible, if time on the computer is only available at intervals, to accumulate the results obtained on an analogue-controlled diffractometer before processing the data. Digitally controlled instruments require the use of a computer for the production of the setting program in addition to its employment for data processing. The best way of making use of the greater flexibility of such an installation is to make a preliminary run covering all reflexions. As a by-product of processing the results from this run, a new program tape is prepared which specifies longer measuring periods for weak reflexions or the insertion of filters for very strong reflexions. For this way of operating the instrument, daily access to a computer, albeit for quite short periods, is necessary.

Data processing programs have been discussed by Cetlin & Abrahams (1963) and by Blake, North & Phillips (1963) and North (1964).

I am glad to record my gratitude to all those who have helped me in the preparation of this account, by discussion, by demonstration of their instruments and by allowing me access to preprints and unpublished reports.

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Evaluation of Digital Automatic Diffractometer Systems

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The ideal automatic diffractometer system is a logical assembly that can measure, *without systematic error* and with little personal intervention, every structure amplitude in the sphere of reflection. Typical sources of error in existing digital systems are discussed and criteria given for their detection. A quantitative measure of departure from the ideal system may be obtained by application of these criteria. Some preliminary results are presented for one system.

Introduction

This paper has three main purposes. The first is to illustrate some of the problems associated with digital automatic diffractometer equipment, the second is to give some criteria for their detection, and the third is to present some typical results for one system. The need for such complex systems should perhaps be briefly justified. Structural crystallographers have long been aware of the difficulties associated with the visual estimation of photographically recorded intensities. There is now a consensus that higher accuracy is required for many physical problems than the photographic method is capable of providing. A parallel difficulty is the large amount of the crystallographer's own time this method consumes.

Attempts at improving accuracy have led to the development of modern counter techniques. While these techniques are inherently more accurate, they can use even more time than the photographic methods they replace. Fortunately, the reason for this disadvantage in manual counter techniques lies in those very repetitive elements which are required for successful automation. Modern automation design has hence been applied to single-crystal diffractometry in an attempt both to improve the accuracy *and* to decrease the time required in making integrated intensity measurements (Abrahams, 1963)*.

* An outline of some of the factors which led to automation is contained in a film *Automated X-Ray Diffractometry* which was used to introduce an Informal Discussion on Automatic Single-Crystal Diffractometry at the Sixth International Congress of the I.U.Cr., Rome, September, 1963. Prints of this film (16 mm in colour, with optical sound track) are available on request to the author. — Ed.

Sources of error

This paper has been restricted to the X-ray case, although it is in part also applicable to neutron diffractometry. Limitation of time must necessarily cause some selection among the topics to be considered.

The ideal automatic diffractometer system may be defined as a logical assembly of subsystems that can measure, with the minimum of personal intervention and without systematic error, every structure amplitude in the sphere of reflection. All such systems contain the five logical elements shown in Fig. 1. Manual diffractometers contain all except the automatic control subsystem, so that this discussion also applies to the manual case.

It is apparent that each of these five logical elements has a capacity for introducing error into the total system. A well designed system keeps the sum of all such error below the level required by the final overall accuracy. To control the introduction of error, we may consider each subsystem in Fig. 1 separately.

A. X-ray and crystal

Fluctuations, both long- and short-term, in the intensity and spectral distribution of the X-ray supply