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References

GRANT, D. F. (1973). *Acta Cryst.* A**29**, 217.
GROVE, W. E. (1966). *Brief Numerical Methods*, Chapter 6. New York: Prentice Hall.
HAMILTON, W. C. (1967). Abstract E6, 34, ACA Meeting, Minneapolis.
KILLEAN, R. C. G. (1969). *Acta Cryst.* B**25**, 977–978.
MACKENZIE, J. K. & WILLIAMS, E. J. (1973). *Acta Cryst.* A**29**, 201–204.
KILLEAN, R. C. G. (1972). *Acta Cryst.* A**28**, 657–658.
SHOEMAKER, D. P. (1968). *Acta Cryst.* A**24**, 136–142.
SHOEMAKER, D. P. & HAMILTON, W. C. (1972). *Acta Cryst.* A**28**, 408–411.
WERNER, S. A. (1972). *Acta Cryst.* A**28**, 143–151.
WILSON, A. J. C., THOMSEN, J. S. & YAP, F. V. (1965). *Appl. Phys. Lett.* **7**, 163–165.

Acta Cryst. (1973). A**29**, 217

Single-crystal diffractometer data: the on-line control of the precision of intensity measurement.

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A method is described for single-crystal data collection to a prestated counting-statistics precision. Procedures for dealing with weak and accidentally absent reflexions are included.

Four-circle diffractometer systems with on-line computer control provide an opportunity for measuring each reflexion in a data collection to a prestated (counting statistics) precision. The required precision will depend on the type of problem under investigation (and the precision of the results required) and could be the same for every reflexion or a function of θ and $|F_o|$. If, before each reflexion is measured, an estimate of its integrated intensity is found by measurement for a short trial time q, then the least time T needed to ensure the required precision can be calculated. The mode of integration is not considered here (Killean, 1973). There will, however, be a maximum time T_m that can reasonably be spent measuring any one reflexion and it will not be possible in this time to achieve the required precision for some weak reflexions. A predicted precision for such a reflexion, if measured for the maximum time, can be found and if the precision does not satisfy a criterion for the precision of weak reflexions, then the reflexion is deemed to be 'accidentally absent' and not measured.

If the fractional precision required for a given reflexion is k, then

$$k=\frac{\sigma(C)}{C}$$

where C is the net integrated counts. If I and B are the peak and background counts, B being measured for a time $1/n$th of that for I, then

$$k=\frac{(I+n^2B)^{1/2}}{(I-nB)}\,.$$

For the trial time q, the corresponding counts are I_q and B_q, $C_q=I_q-nB_q$ and $\sigma(C_q)=(I_q+n^2B_q)^{1/2}$. To ensure that a reflexion is almost certainly being measured to at least the precision of k,

$$C'_q=C_q-2\sigma(C_q)$$

is used instead of C_q to calculate T.

If $f=\dfrac{T}{q}$ then

$$k=\frac{\{f(I_q+n^2B_q)\}^{1/2}}{fC'_q} \qquad (1)$$

and

$$f=\frac{(I_q+n^2B_q)}{(kC'_q)^2}\,.$$

A further remeasurement of the reflexion for a time $q(f-1)$ when added to the trial measurement will give the required k. If $T<q$, then no further measurement is necessary for such a strong reflexion.

If $T>T_m$, a decision has to be taken whether or not the weak reflexion is to be measured. The predicted precision k', if the reflexion were measured for T_m, would be from (1):

$$k'=\frac{\{f_m(I_q+n^2B_q)\}^{1/2}}{f_mC'_q} \qquad (2)$$

where $T_m=f_mq$. A criterion for measuring this reflexion would be $k'<k_c$ where k_c is a prestated acceptable precision for weak reflexions. Almost all such reflexions satisfying this criterion when measured for T_m would have at least the precision k_c. Very weak reflexions for which $k'>k_c$ would not be acceptable in the data set, would not be further measured and would be considered to be accidentally absent. It is possible if q is made too small, that C'_q could become negative for some very weak reflexions. Then it would be preferable to use C_g in equation (2) for the weak-reflexions criterion. However, a better choice of q at the outset of the experiment can confine the negative C'_q to those very weak reflexions which would anyway be considered accidentally absent.

This method of data collection has the advantages (i) of economy in the use of diffractometer time by measuring each reflexion for the least time necessary to ensure a certain precision and (ii) of a further saving in time by measuring only those weak reflexions for which a satisfactory precision can be predicted (an important consideration when collecting data from crystals with high temperature factors).

This measuring routine has been incorporated into the on-line control by an IBM 1130 computer of a Siemens AED diffractometer. The preset parameters for a data collection include k, k_c, q and T_m. Typically q is made $\frac{1}{25}$th to $\frac{1}{50}$th of T_m which itself is chosen by considering k in relation to the intensity expected from weak reflexions and the time available for the experiment.

References

KILLEAN, R. C. G. (1973). *Acta Cryst.* A**29**, 216–217.