

epiped and for the other of which the unit cell is an acute parallelepiped.

In the former case the translations a_0 , b_0 and c_0 of the unit cell are the shortest possible. In the latter case, the translations a_0 , b_0 and c_0 are not necessarily the shortest possible, but they will usually be so, and the interaxial angles will usually be not greatly different from 90° . Moreover, they have a unique definition and are automatically derived by the application of the Delaunay reduction in reciprocal space.

The application of the above rules thus avoids the inconvenient values of lattice parameters obtained in many cases when the Delaunay reduction is carried out in one (direct or reciprocal) space only.

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The Laue goniometer and its use as a proportional diffractometer. By A. R. B. SKERTCHLY, *Textile Physics Research Laboratory, The University, Leeds 2, England*

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The Laue goniometer has long been used for the study of symmetry and lattice distortion, principally by metallurgists. Exact analysis is rendered difficult because two variables (wavelength and lattice spacing) are associated with each diffraction spot, and estimates of unit-cell size are generally only approximate. If the diffracted image were to be investigated by a proportional counter and multichannel pulse-height analyser then direct knowledge of the wavelengths and relative intensities associated with each spot could be obtained.

As the rate of arrival of X-ray quanta is governed by the Poisson distribution, and the effective integrated intensity (N) is a differential count between background (B) and $N+B$, then the variance associated with a count N is $N+2B$.

The coefficient of variation is $\{100/(N+2B)\}/N$, and if N is greater than B (as is usual except for very weak spots) we must have $N > 10^4$ if this coefficient is to be less than 1%.

Cochran (1950) reports a standard deviation of only 1–2% in I_{hkl} values for Geiger-counter measurements, compared with 12–18% for photographic recording.

Channel-selection considerations limit the maximum rate of arrival of quanta to 1600 per sec. (Hutchinson & Scarrott, 1951), which means that an intensity measurement with a coefficient of variation of 1% may be obtained in about 6 sec.

The pulse-height variance $(\sigma_p)^2$ for mono-energetic radiation absorbed in an argon-filled proportional counter can be expressed (Curran, Angus & Cockroft, 1949) by

$$\sigma_p^2 = \bar{p}^2(\alpha + \beta)/\bar{N},$$

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where

- \bar{p} = mean pulse height,
 \bar{N} = E/W_k = number of ion pairs initially produced,
 E = energy of incident quanta,
 W_k = energy required to produce one ion pair = 27 e.V. for argon (Valentine, 1952),
 $\alpha = \beta = \frac{2}{3}$.

Thus in the region of $\lambda = 1 \text{ \AA}$, $E = 12340 \text{ e.V.}$ and the coefficient of variation is 5.35%.

Lang (1952) assumes a Gaussian pulse-height distribution so that the standard error of the mean, expressed as a percentage of the mean, is

$$115/\sqrt{(\bar{N}N)}.$$

Thus if $\lambda = 1 \text{ \AA}$, and $N = 10^4$ the standard error of the mean is 0.05%.

The performance is limited by the stability of the grid potential in the amplitude analyser, which restricts the number of available channels to 120 and the maximum counting rate to 1600 counts per sec. (Titterton, 1953).

The preceding shows that a typical measurement of intensity and wavelength could be made in less than 10 sec. with the following degrees of accuracy:

- Intensity: coefficient of variation 1%;
 Wavelength: standard error 0.05% (expressed as a percentage fraction of the mean).

Furthermore, the harmonic content of the radiation may also be quantitatively measured if the pulse-height

analyser channel range coincides with the useful range of white radiation.

The success of the technique depends on the linearity of response to quanta of different energies which the proportional counter main gas constituent possesses. Curran *et al.* (1949) have shown that for argon up to an energy of 40 keV. linearity is preserved to within < 0.01%. The wavelength distribution of white radiation and absorption characteristics of the counter must be accurately known and maintained, the former demanding good X-ray tube stability such as is achieved in the system developed by Stone (1955); this maintains output constant to within 0.3% over long intervals, which is adequate for the purpose in view.

The lattice parameter a for cubic crystals may be found from a set of functions of the form

$$a = \frac{1}{m} \sum_1^m n\lambda \frac{\sqrt{(h^2+k^2+l^2)}}{2 \sin \theta},$$

where m is the number of observations.

The error (σ_a) associated with a has two main components due to wavelength and angular imprecision; thus

$$\sigma_a^2 = \sigma_\lambda^2 + \sigma_\theta^2.$$

Edmunds, Lipson & Steeple (1955) show that $\sigma_\theta < \sigma_\lambda$, so that $\sigma_a \approx \sigma_\lambda$.

If we have m Laue diffraction spots indexed, then the standard error of a , expressed as a percentage of the mean, is

$$\frac{100 \sigma_a}{\sqrt{m} a} = \frac{100 \sigma_\lambda}{\sqrt{m} \bar{\lambda}} = \frac{0.05}{\sqrt{m}} = 0.005\%, \quad \text{for } m = 100.$$

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On the use of central inclination Weissenberg photographs in studies of large unit cells. By

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The construction of most commercially available Weissenberg apparatus is unfortunately such that the reflections with low θ values obtained from crystals with very short reciprocal spacings are intercepted by the beam stop, and cannot be measured. There exist round-about ways to remedy this situation, e.g. additional oscillation photographs, substitution of F_c for F_o in later stages of refinement, and, of course, the use radiation of longer wavelength. A more practical procedure would appear to be the following:

A situation is created where the circle of reflection is smaller and the Weissenberg spots are therefore recorded more spread out on the film. This may be achieved by skewing the camera so that the zero layer is photographed at non-orthogonal incidence of the X-ray beam.

Thus in a particular case a could be computed accurately to 1 part in 10^4 , roughly an order of magnitude worse than in a precision analysis. We could then proceed with a structure analysis and refinement, which in many instances would provide data from more complicated sets of planes which are extremely sensitive to small shifts in atomic parameters.

Mention might also be made of the possible use of this technique in elucidating the spurious pole figure maxima which occur in orientation studies in metals (Geisler, 1954). It is felt that such procedures will be of considerable use to metallurgical crystallographers.

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