

The Measurement of Integrated Intensities

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Abstract

Expressions for the integrated intensities of the Laue and of the rotating-crystal methods are given, which take the spectral distribution of the incident radiation into account. The correlation between the integrated intensities of both methods is elucidated. A procedure is described for the measurement of integrated intensities on an absolute scale using radiation with a continuous spectral distribution superimposed by sharp (spectral) lines. It is shown that crystal-monochromatized radiation may also exhibit, besides sharp lines, continuous tails. Neglecting these tails causes errors up to 2% for observed integrated intensities.

Introduction

Measuring integrated intensities of MnF_2 , TiO_2 and VO_2 with graphite-monochromatized $\text{Ag K}\alpha$ radiation by the rotating-crystal method, the author noticed that for all strong and medium strong reflections – those with small Bragg angles excepted – the high-angle background intensity was systematically larger than the low-angle one. Integrated intensities, measured with a MnF_2 crystal, were shown to be normally distributed and the sample variances compared with hypothetical Poisson variances behaved well as expected from statistics (Alte de Veiga, Andrade & Gonschorek, 1982). Nevertheless, least-squares refinement led to a goodness-of-fit parameter far outside the range in which it could be accepted from a statistical point of view. These observations and the intention to measure integrated intensities on an absolute scale stimulated a more thorough examination of the definition and measurement of integrated intensities.

1. Integrated reflecting power

With the incident and scattered wave vectors \mathbf{k}_0 and \mathbf{k} , $k = k_0 = 1/\lambda$, the scattering vector \mathbf{s} is defined as

$$\mathbf{s} = \mathbf{k} - \mathbf{k}_0. \quad (1)$$

If \mathbf{k}_0 and \mathbf{k} include the angle 2θ , the modulus of \mathbf{s} is $s = 2 \sin \theta/\lambda$. The Laue equation $\mathbf{s} = \mathbf{h}$ leads, with $h = 1/d_{\mathbf{h}}$, to the Bragg equation $2d_{\mathbf{h}} \sin \theta_{\mathbf{h}} = \lambda$. For $\mathbf{s} = \mathbf{h}$ the vectors \mathbf{k}_0 and \mathbf{k} include the angles $\pi/2 + \theta_{\mathbf{h}}$ and $\pi/2 - \theta_{\mathbf{h}}$ with \mathbf{h} .

To define the integrated intensity, Zachariasen (1967) considers the incident-wave vector $\mathbf{k}_0 = \mathbf{k}_0(\varepsilon_1)$ as a function of a parameter ε_1 . This parameter is so chosen that $\mathbf{k}_0(0)$ satisfies the Laue equation, *i.e.* $\mathbf{k}_0(0)$ makes an angle of $\pi/2 + \theta_{\mathbf{h}}$ with \mathbf{h} and has the modulus $k_0(0) = 1/\lambda = (2d_{\mathbf{h}} \sin \theta_{\mathbf{h}})^{-1}$. It is assumed that a given $\mathbf{k}_0(\varepsilon_1)$ is associated with the incident intensity I_0 which is measured in the dimension energy/(area \times time). The wave vectors \mathbf{k} of scattering from lattice planes (\mathbf{h}) are oriented within a small solid angle and do not necessarily satisfy Laue's equation, so that the total amount of radiation energy, scattered per unit time, is obtained by integration of the diffracted intensity over an area. The ratio of the total amount of radiation energy of wavelength $\lambda = 1/k_0(\varepsilon_1)$ which is scattered per unit time at lattice planes \mathbf{h} to the incident intensity (of the same wavelength), is called the reflecting power $P_{\mathbf{h}}(\varepsilon_1)$. If ε_1 is varied systematically, the integrated reflecting power $P_{\mathbf{h}}$ is defined as

$$P_{\mathbf{h}} = \int P_{\mathbf{h}}(\varepsilon_1) d\varepsilon_1. \quad (2)$$

The function $P_{\mathbf{h}}(\varepsilon_1)$ and, if ε_1 is dimensionless, also the quantity $P_{\mathbf{h}}$ both have the dimension of area and they depend upon how ε_1 is varied. Here the Laue method and the rotating-crystal method will be considered.

Laue method

The orientation of $\mathbf{k}_0(\varepsilon_1)$ relative to the crystal is constant, only the modulus $k_0(\varepsilon_1)$ is varied. The parameter ε_1 can be chosen as

$$\lambda = \lambda_{\mathbf{h}}(1 + \varepsilon_1) \quad (3)$$

with $\lambda_{\mathbf{h}} = 2d_{\mathbf{h}} \sin \theta_{\mathbf{h}}$. The angle $\theta_{\mathbf{h}}$ is determined by the angle $\pi/2 + \theta_{\mathbf{h}}$, which $\mathbf{k}_0(\varepsilon_1)$ and \mathbf{h} make with each other.

Zachariasen (1967), on the basis of the kinematical theory, deduces two expressions for the integrated reflecting power P_h of the Laue method, both of which do not have the dimension of an area. Bouman (1957) avoids this discrepancy by redefining the incident intensity I_0 , so that this quantity in the Laue method has another meaning and another dimension than in the rotating-crystal and powder methods.

The expression for P_h with the correct dimension and with unaltered meaning of I_0 is obtained, if in Zachariasen's derivation the parameter ε_1 (Zachariasen, 1967, p. 105, equation [3.64]) is replaced by ε , as defined in (3). This gives the integrated reflecting power the form

$$P_h = Q_h(\theta_h) v \quad (4)$$

with

$$Q_h(\theta_h) = \left(\frac{a}{V}\right)^2 \frac{1 + \beta \cos^2 2\theta_h}{1 + \beta} \frac{|F_h|^2 \lambda_h^3}{2 \sin^2 \theta_h}. \quad (5)$$

Here v is the volume of the crystal and V that of the unit cell. The constants a and β depend upon the nature of the incident radiation: $a = 10$ fm for neutrons and $a = e^2/mc^2$ for X-rays; $\beta = 0$ for unpolarized neutrons, $\beta = 1$ for unpolarized X-rays and $0 < \beta < 1$ for most commonly used monochromatized X-rays.

Rotating-crystal method

The incident wave vector \mathbf{k}_0 ($k_0 = 1/\lambda$) is constant and the crystal is rotated (this is equivalent to a rotation of the incident beam relative to the crystal). For the equatorial geometry ($\chi = \Phi = 0$; $2\theta_h = \psi$ in equation [3.78] of Zachariasen (1967)), the expressions

$$P_h = Q_h(\lambda) v, \quad (6)$$

$$Q_h(\lambda) = \left(\frac{a}{V}\right)^2 \frac{1 + \beta \cos^2 2\theta_h}{1 + \beta} \frac{|F_h|^2 \lambda^3}{\sin 2\theta_h} \quad (7)$$

are found.

Here the angle θ_h for given $k_0 = 1/\lambda$ and $|\mathbf{h}| = 1/d_h$ is defined by $\sin \theta_h = \lambda/2d_h$. Both $Q_h(\theta_h)$ and $Q_h(\lambda)$ have the dimension $(\text{length})^{-1}$.

2. Integrated intensity

The spectral distribution of the incident radiation may be given by

$$I_0(\varepsilon) = \frac{dE}{d\varepsilon}. \quad (8)$$

With

$$\lambda = \lambda_1(1 + \varepsilon) \quad (9)$$

the differential dE denotes the radiation energy with wavelengths between $\lambda = \lambda_1(1 + \varepsilon)$ and $\lambda = \lambda_1(1 + \varepsilon + d\varepsilon)$, which crosses a unit area per unit time. The reference wavelength λ_1 can be chosen arbitrarily.

Laue method

Comparing (3) and (9) ε can be expressed as a function of ε_1 . The integrated intensity I_h with reference to (2) takes the form

$$I_h = \int P_h(\varepsilon_1) I_0[\varepsilon(\varepsilon_1)] \frac{\lambda_h}{\lambda_1} d\varepsilon_1 \quad (10)$$

and has the dimension energy per time. If $I_0[\varepsilon(\varepsilon_1)]$ is constant within the small region of ε_1 values, for which $P_h(\varepsilon_1)$ gives a contribution to P_h , (10) reduces to

$$I_h = Q_h(\theta_h) v I_0 \left(\frac{\lambda_h - \lambda_1}{\lambda_1}\right) \frac{\lambda_h}{\lambda_1}. \quad (11)$$

In the experiment, because of the finite size of the source of radiation and of the crystal, the direction of \mathbf{k}_0 and consequently the angles θ_h are variable for all \mathbf{h} , say within the limits $\pm\delta$. Therefore, in (11), $\lambda_h = 2d_h \sin \theta_h$ also is variable: $\Delta\lambda_h = 2d_h \cos \theta_h 2\delta$, and according to (9) the corresponding ε interval is $\Delta\varepsilon = \Delta\lambda_h/\lambda_1 = d_h \cos \theta_h 4\delta/\lambda_1$. Hence the range of ε values, which act as arguments of $I_0(\varepsilon)$ in (11), depends upon $d_h \cos \theta_h$. This disadvantage does not exist if the rotating-crystal method is applied.

Rotating-crystal method

Radiations of different wavelengths do not interfere with each other, so that according to (6) the integrated intensity of the rotating-crystal method has the form

$$I_h = v \int_{\varepsilon(1)}^{\varepsilon(2)} I_0(\varepsilon) Q_h[\lambda_1(1 + \varepsilon)] d\varepsilon. \quad (12)$$

The limits of integration are fixed by the angular range (θ_h^1, θ_h^2) through which the crystal is rotated during the measurement of the diffracted intensity. With (9), the Bragg equation yields:

$$\varepsilon(i) = 2d_h \sin \theta_h^i/\lambda_1 - 1, \quad i = 1, 2. \quad (13)$$

Equation (12) only is valid if $I_0(\varepsilon)$ vanishes at the values $\varepsilon(1)$, $\varepsilon(2)$ and in their neighbourhood. If the spectral distribution $I_0(\varepsilon)$ has the form of a δ function with $I_0 = \int I_0(\varepsilon) d\varepsilon$, (12) is replaced by

$$I_h = Q_h(\lambda) v I_0. \quad (14)$$

The fact that the direction of \mathbf{k}_0 is variable has no influence as long as \mathbf{k}_0 remains parallel to the equatorial plane. If \mathbf{k}_0 includes an angle with that plane of, for example, 0.5° , the numerical value of $Q_h(\lambda)$, according to Zachariasen (1967, p. 108, equation [3.78])

with $\chi = -\Phi = 0.5^\circ$, changes by 0.008%. This certainly can be neglected.

Equation (2) prescribes how the observed integrated intensity I_h^o is to be deduced from the observed intensity profile $I_h^o(\theta_h + \varepsilon_1)$. Theoretically, for a definite wavelength, the intensity profile has the form $I_h(\theta_h + \varepsilon_1) = I_0 P_h(\varepsilon_1)$ and application of (2) leads to

$$I_h^o = \int_{\theta_h^1}^{\theta_h^2} I_h^o(\theta_h + \varepsilon_1) d\varepsilon_1 = \int_{\tau_1}^{\tau_2} I_h^o(\theta_h + \varepsilon_1) \frac{d\varepsilon_1}{dt} dt. \quad (15)$$

3. Correlation between the Laue and the rotating-crystal methods

Suppose that the spectral distribution $I_0(\varepsilon)$ varies slowly with ε , so that in the Laue method (11) can be applied for a certain range of values $\varepsilon = (\lambda_h - \lambda_1)/\lambda_1$ (in the Laue method λ_h for given d_h is variable with θ_h)! Then the profile $I_h^o(\theta)$ which is observed, if the crystal is rotated through an interval (θ_h^1, θ_h^2) , is equivalent to a series of successive integrated intensities of the Laue method. Therefore, according to (11) the theoretical profile of a rotating-crystal experiment can be written as

$$I_h(\theta) = Q_h(\theta) v I_0 \left(\frac{2d_h \sin \theta - \lambda_1}{\lambda_1} \right) \frac{2d_h \sin \theta}{\lambda_1}. \quad (16)$$

With this relation, the distribution $I_0(\varepsilon)$ can be determined from the intensity profile $I_h(\theta)$ and inserted into (12):

$$I_h = \frac{\lambda_1}{2d_h} \int_{\varepsilon(1)}^{\varepsilon(2)} I_h(\theta) \frac{Q_h[\lambda_1(1 + \varepsilon)]}{Q_h(\theta) \sin \theta} d\varepsilon. \quad (17)$$

Here the variable ε is replaced by λ as in (9) and the expressions $Q_h(\lambda)$ and $Q_h(\theta)$ from (7) and (5) are inserted.

Finally, λ is replaced by θ with the aid of Bragg's equation. This yields an integral which corresponds to that in (15) and denotes the integrated intensity of the rotating-crystal method, as expected, because it was derived from the alternative form (12). This result will be used later on.

4. The wavelength dependency of integrated intensities of the rotating-crystal method

For X-rays from an X-ray tube there is the characteristic radiation $I_0^C(\varepsilon)$ and the *Bremsstrahlung* $I_0^B(\varepsilon)$ and one may write $I_0(\varepsilon) = I_0^C(\varepsilon) + I_0^B(\varepsilon)$. Usually the wavelengths of the characteristic radiation and the lattice spacings d_h are known with a high degree of accuracy. Therefore the λ -dependent part of $Q_h(\lambda)$, (7), can also be evaluated very accurately, if the spectral lines are

narrow enough so that the expression $Q_h(\lambda)$ can be considered as constant for each line. The ratio of the total amount of radiation energy of the $K\alpha_1$ line to that of the $K\alpha_2$ line is called p . With the integrals A_1 and A_2 , taken over the two lines, the ratio

$$p' = \frac{A_2}{A_1} = p \frac{Q_h(\lambda_2)}{Q_h(\lambda_1)} \quad (18)$$

and from this the ratio p is found. The integrated intensity according to (12), if $I_0^B(\varepsilon) = 0$ is assumed, takes the form

$$I_h = v I_0^C Q_h(\lambda_1, \lambda_2) \quad (19)$$

with

$$I_0^C = \int I_0^C(\varepsilon) d\varepsilon \quad (20)$$

and

$$Q_h(\lambda_1, \lambda_2) = (1 + p)^{-1} [Q_h(\lambda_1) + p Q_h(\lambda_2)]. \quad (21)$$

If $I_0^B(\varepsilon)$ has a form so that its contribution to the observed profiles $I_h^o(\theta)$ varies linearly with θ in the neighbourhood of the characteristic lines, this contribution together with the diffuse background scattering can be determined and subtracted from the observed intensity, (15). This situation is equivalent to the case $I_0^B(\varepsilon) = 0$ and is found if X-rays from an X-ray tube are used and if for the monochromatization a β filter is chosen with an absorption edge far enough from the wavelengths of the characteristic radiation; an example is given in Fig. 1. The commonly used crystal monochromators applied for radiation from an X-ray tube, however, do not seem to yield profiles $I_h^o(\theta)$ so

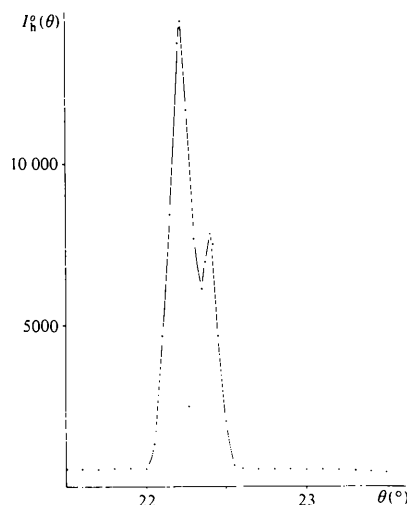


Fig. 1. Reflection 004 of rutile, Ag $K\alpha$ radiation, 50 kV, 20 mA, β filter: 50 μm palladium, $\theta_h = 22.22^\circ$, the coordinate numbers denote counts/12 s. Diffuse background scattering: 50 counts/12 s.

that the contribution of $I_0^B(\epsilon)$ can simply be subtracted, as already stressed by Hope (1975). As a typical example the profile of reflection $0\bar{5}1$ (interplanar spacing $d_{0\bar{5}1} = 0.886 \text{ \AA}$) from the monoclinic phase of VO_2 taken with graphite-monochromatized Ag $K\alpha$ radiation is shown in Fig. 2. This profile agrees well with what was found by Franklin (1951): 'In graphitic carbons ... the (001) lines, although narrow, are always unsymmetrical, the intensity falling off more slowly on the small-angle side'. From this it is clear

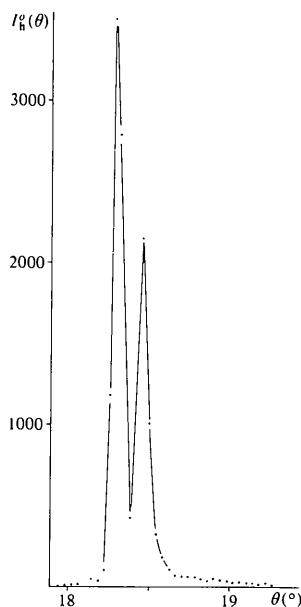


Fig. 2. Reflection $0\bar{5}1$ of monoclinic VO_2 , graphite monochromatized Ag $K\alpha$ radiation, 55 kV, 20 mA, $\theta_h = 18.31^\circ$, the coordinate numbers denote counts/4.8 s. Diffuse background scattering: 5 counts/4.8 s. The higher resolution of the two $K\alpha$ lines of this profile compared with that in Fig. 1 mainly arises because there the crystal needle was parallel, here it was perpendicular to the equatorial plane.

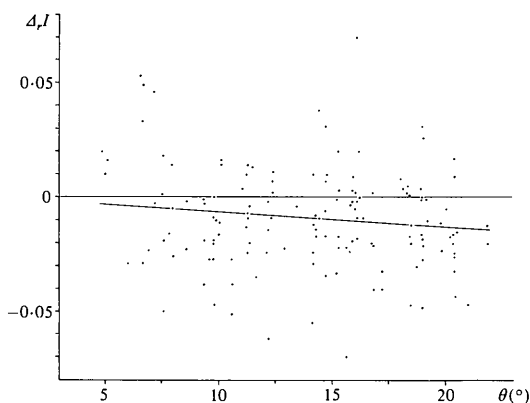


Fig. 3. Relative differences $\Delta_r I = (I_{h,1}^o - I_{h,2}^o)/I_{h,1}^o$ of observed integrated intensities of monoclinic VO_2 . The two sets of data $I_{h,1}^o$ and $I_{h,2}^o$ were measured with two different scan widths. Only the 160 strongest integrated intensities were taken into account.

why the high-angle background intensity is found larger than the low-angle one, and it is clear that in routine intensity measurements the observed integrated intensity for different Bragg angles comprises different portions of the primary intensity $I_0(\epsilon)$.

To obtain a quantitative measure of how much the observed integrated intensities I_h^o , (15), vary with varying scan widths (θ_h^1, θ_h^2), two complete data sets were measured with the same VO_2 crystal on a CAD-4 diffractometer. The scan widths were chosen according to $(\theta_h^2 - \theta_h^1) = D \tan \theta_h + E$, with $D = 0.45^\circ$, $E = 1.00^\circ$ and $D = 0.1^\circ$, $E = 1.5^\circ$ for the first and second data set. 'Background' scattering was measured on both sides of the profiles up to θ values 25% further from θ_h than θ_h^1 and θ_h^2 . The relative differences $\Delta_r I = (I_{h,1}^o - I_{h,2}^o)/I_{h,1}^o$ of the observed integrated intensities, background subtracted, of the two data sets are plotted against θ in Fig. 3. At $\theta = 20^\circ$ the difference amounts to about 1.6%. Refinements of the overall temperature factor $B = 8\pi^2 \bar{u}^2$ with the two data sets leads to a difference $\Delta \bar{u}^2 = 0.00027 \text{ \AA}^2$, the \bar{u}^2 value from the second data set being the smaller one. This is of the same order of magnitude as the differences of the U_{ik} values found for the atoms of rutile by Gonschorek (1982a) with two different data sets. One data set was measured by Shintani, Sato & Saito (1975) with graphite-monochromatized Ag $K\alpha$ radiation yielding larger U_{ik} 's, the other by Gonschorek (1982a) with β -filtered Ag $K\alpha$ radiation. Apparently, the large goodness-of-fit parameter for MnF_2 (Alte da Veiga *et al.*, 1982) is also caused mainly by this source of error.

The same problem arises for β -filtered radiation, if integrated intensities are to be determined on an absolute scale and if the primary intensity

$$I_0 = \int I_0(\epsilon) d\epsilon \quad (22)$$

directly is measured as described by Gonschorek (1982a). For X-rays from synchrotron radiation and for neutrons a variety of monochromators is in use and the spectral distribution $I_0(\epsilon)$ of the radiation striking the crystal depends upon the monochromator.

In the following a procedure will be described for the determination of that portion α^{-1} of I_0 which contributes to the observed integrated intensity I_h^o if finite scan intervals (θ_h^1, θ_h^2) are chosen.

First it is noticed that, because of the angular divergence of the primary beam, the limits $\epsilon(1)$ and $\epsilon(2)$ as introduced in (13) are not determined unequivocally. This question is discussed in Appendix C.* For the moment, with a given scan interval (θ_h^1, θ_h^2) and taking (13) into account, the contribution of the

* The discussion of the error arising from the fact that a scan interval (θ_h^1, θ_h^2) does not unequivocally determine an interval $[\epsilon(1), \epsilon(2)]$, as required in (12), has been deposited. See deposition footnote, § 6.

Bremsstrahlung $I_0^B(\varepsilon)$ to the observed integrated intensity is supposed to be given by

$$I_0^B = \int_{\varepsilon(1)}^{\varepsilon(2)} I_0^B(\varepsilon) d\varepsilon \quad (23)$$

and the ratio

$$\alpha = I_0/(I_0^B + I_0^C) \quad (24)$$

is defined. Now the observed integrated intensity I_h^o , (15), is multiplied by α and with I_h from (12) the equation

$$\alpha I_h^o = \frac{I_0}{I_0^B + I_0^C} I_h \quad (25)$$

can be used for the determination of $|F_h|^2$ on an absolute scale. The primary intensity I_0 , the ratio α and the integrated intensity I_h^o experimentally are determined, whereas the theoretical expression for I_h will be replaced by an approximate expression, so that the denominator in (25) is cancelled.

5. Determination of α

The quantities I_0 and $(I_0^B + I_0^C)$, as required in (24), are approximated by two integrals of the form

$$I_p = X \int I_h^o(\theta) p(\theta) d\theta \quad (26)$$

with

$$p(\theta) = \frac{(1 + \beta) \cos \theta}{(1 + \beta \cos^2 2\theta) \sin^2 \theta} \quad (27)$$

In (26) $I_h^o(\theta)$ is the observed profile of a reference reflection. The function $p(\theta)$ is equivalent to $1/Q_h(\lambda)$, if in (7) the wavelength λ is replaced by $2d_h \sin \theta$ and if the different constants in (7) are replaced by one constant X . For the approximation of I_0 the integration in (26) is extended to values where $I_h^o(\theta)$ vanishes, and the result is called I_p^{tot} . For the approximation of $(I_0^B + I_0^C)$, the result of which is called I_p^{red} , the limits of integration (θ_h^1, θ_h^2) in (26) are found from the scan interval $(\theta_{h'}^1, \theta_{h'}^2)$ with which the observed integrated intensity $I_{h'}^o$ of a reflection h' according to (15) is determined:

$$d_h \sin \theta_h^i = d_{h'} \sin \theta_{h'}^i, \quad i = 1, 2. \quad (28)$$

Now α , defined in (24), is approximated by

$$\alpha' = I_p^{\text{tot}}/I_p^{\text{red}} \quad (29)$$

and the constant X has no further influence.

The relative error $\Delta\alpha/\alpha = (\alpha - \alpha')/\alpha$, which must be reckoned with if α is determined *via* α' , is derived in

Appendix B.* The different error sources, contributing to the relative error $\Delta\alpha/\alpha$, are discussed in Appendix C.†

Examples of the determination of α' with three different reference reflections h , using Nb-filtered Mo $K\alpha$ radiation, are given by Gonschorek (1982*b*, Table 4). The agreement of α' values obtained with the aid of these reference reflections lies within 1–3‰.

6. Approximation of the integrated intensity I_h

Once in (25) the ratio α is determined, the integrated intensity

$$I_h = v \int_{\varepsilon(1)}^{\varepsilon(2)} I_0^B(\varepsilon) Q_h[\lambda_1(1 + \varepsilon)] d\varepsilon + v I_0^C Q_h(\lambda_1, \lambda_2) \quad (30)$$

derived from (12) for X-rays from an X-ray tube is considered. Here the first expression is replaced by (17) with $I_h(\theta)$ as that part of the reflection profile which corresponds to $I_0^B(\varepsilon)$. For β -filtered radiation this part is constant as in Fig. 1, if reflections h are chosen with large Bragg angles θ_h . Then $Q_h[\lambda_1(1 + \varepsilon)]$ is replaced by $Q_h(\lambda_1, \lambda_2)$ from (21) and (17) reduces to

$$I_h^B = \frac{1}{p_{1,2} \theta_h^i} \int_{\theta_h^1}^{\theta_h^2} I_h(\theta) p(\theta) d\theta \quad (31)$$

Here $1/p_{1,2}$ is the mean of $1/p(\theta_h)$ for $K\alpha_1$ and $K\alpha_2$ and $p(\theta)$ is defined in (27). Since in (31) $I_h(\theta)$ is constant, the integral can be exactly evaluated; this is done in Appendix A.‡§ Remembering the conclusion drawn after (17), there is no difference between I_h^B from (31) and the first expression in (30), if for $I_h(\theta) = \text{constant}$ the equation

$$p_{1,2}[\theta_h^2 - \theta_h^1] = \int_{\theta_h^1}^{\theta_h^2} p(\theta) d\theta \quad (32)$$

is valid. Then, with a scan interval (θ_h^1, θ_h^2) satisfying (32), (25) takes the form

$$\alpha' I_h^o = I_0 v Q_h(\lambda_1, \lambda_2) \quad (33)$$

with α' as defined in (29). From this equation, structure factors $|F_h|$ have been determined on an absolute scale by Gonschorek (1982*a*).

* The derivation of the relative error $\Delta\alpha/\alpha$ which arises, if α as defined in (24) is approximated by α' as defined in (29), has been deposited. See deposition footnote, § 6.

† A discussion of errors contributing to $\Delta\alpha/\alpha$ and of their correction has been deposited. See deposition footnote, § 6.

‡ The exact evaluation of the integral in (31) for $I_h(\theta) = \text{constant}$ has been deposited.

§ Appendices A, B and C have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38707 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

For graphite monochromatized X-rays, $I_h(\theta)$ corresponding to $I_0^B(\epsilon)$ is not constant and the integral in (31) cannot be evaluated. In spite of this it is common practice to consider $I_0 v/a'$ as constant and to use (33) for the determination of observed structure factors on a relative scale. The error arising by neglect of the first part in (30) is about 1% as was estimated from three reflection profiles with different Bragg angles. However, this error scarcely varies with the Bragg angle so that for relative measurements the effective error reduces to about 0.2%.

The error caused by considering a' as constant in (33) may rise to about 2% depending upon which scan intervals are chosen and which Bragg-angle range is covered during data collection.

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On the Equivalence of Point Configurations due to Euclidean Normalizers (Cheshire Groups) of Space Groups

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Dedicated to Professor M. J. Buerger on the occasion of his 80th birthday

Abstract

The Euclidean normalizers of space groups form the appropriate mathematical tool for several problems treated independently by crystallographers in the past, e.g. the comparison, the classification and the standardized description of crystal structures. Explicit tables are presented that enable the user to handle Euclidean normalizers in an easy way and, especially, to calculate all descriptions of a crystal structure compatible with a chosen space-group setting. The use of the tables is illustrated by different examples, and the role of Euclidean normalizers for crystal-structure determination is discussed.

1. Introduction

Depending on the position of a representative point, point configurations (*i.e.* sets of symmetrically equivalent

points) of a given space group in general differ with respect to their geometrical properties. For a study of these properties all different cases are covered if the coordinates of such a reference point are varied over the whole range of an asymmetric unit. In most space groups, however, it is possible to confine the parameter variation to a smaller region by taking into account the symmetry of the pattern of symmetry elements. In plane group *pgg*, for instance, Laves (1931) derived all possible plane partitions into Dirichlet domains by considering only the range $0 \leq x, y \leq \frac{1}{2}$ instead of the asymmetric unit $0 \leq x, y < \frac{1}{2}$ 'for reasons of symmetry'. Under the term 'reduced asymmetric units' this principle has been revived by Fischer (1968) in a paper on the packing of circles in a plane. Later it was applied in connection with sphere packings (Fischer, 1970, 1971, 1973, 1974) and Dirichlet partitions (Koch, 1972, 1973). For a classification of point configurations by means of symmetry, shortest dis-