

#### IV. Concluding remarks

In their pioneer experiments, Borrmann & Lehmann (1963) obtained agreement between observed and calculated fringe spacings within 25% in the high-absorption case and 70% in the low-absorption case. The reason for the deviation is that in their simple theory they considered only the effect of the phase relationship of waves upon the fringe spacing, but neglected the effect of the absorption and the intensities of the waves. In this paper, we have considered all factors affecting the spacings of fringes. Therefore we have obtained good agreement, within 14%, between observed and calculated fringe spacings in the high- and low-absorption cases. The computer simulation patterns also fit the experimental patterns very well. Through our theory the BL fringes are physically clearer.

*Acta Cryst.* (1989). **A45**, 609–613

### The Two-Wavelength Technique in Crystal Structure Determination

BY E. A. KLOP, H. KRABBENDAM AND J. KROON

*Laboratorium voor Kristal- en Structuurchemie, Rijksuniversiteit, Padualaan 8, 3584 CH Utrecht, The Netherlands*

(Received 24 January 1989; accepted 23 March 1989)

#### Abstract

A generalization and extension is given of the two-wavelength ratio technique for phase determination [e.g. Cascarano, Giacovazzo, Peerdeman & Kroon (1982). *Acta Cryst.* **A38**, 710–720 and references therein]. It is shown that the phase-determining formula of the ratio technique for acentric reflections merely depends on Bijvoet ratios and dispersion terms even if there are no restrictions on these dispersion terms. The system of equations which forms the basis for both the ratio technique and the Bijvoet difference method of Singh & Ramaseshan [*Acta Cryst.* (1968), **B24**, 35–40] (S&R) permits the derivation of a relationship between the scale factors of data collected at different wavelengths for acentric reflections. If this relationship is used as scaling scheme, the S&R method is algebraically equivalent to the ratio technique. For centric reflections the two methods are equivalent provided that the same scaling is applied.

#### Introduction

The availability of intense tuneable synchrotron radiation has renewed interest in multi-wavelength methods for phase determination of protein structures by means of anomalous X-ray scattering. Two-

The authors thank Professor Shun Changde for helpful discussions, the British Council for financial support to MZ and Professor A. R. Lang FRS for guiding advice and topographic facilities at the University of Bristol.

#### References

- BORRMANN, G. & LEHMANN, K. (1963). *Crystallography and Crystal Perfection*, edited by G. N. RAMACHANDRAN, pp. 101–108. London, New York: Academic Press.
- KATO, N. (1968) *J. Appl. Phys.* **39**, 2225.
- LANG, A. R., KOWALSKI, G., MAKEPEACE, A. P. W. & MOORE, M. (1986). *Acta Cryst.* **A42**, 501–510.
- LEHMANN, K. & BORRMANN, G. (1967). *Z. Kristallogr.* **125**, 234–248.
- SAKA, T., KATAGAWA, T. & KATO, N. (1972). *Acta Cryst.* **A28**, 102–113, 113–120.

wavelength methods employ either sums (differences) or ratios of two intensities, called respectively Bijvoet sums (differences) or Bijvoet ratios if the two intensities correspond to Friedel equivalents (or, in general, to reflections which are related by Laue symmetry operations which do not belong to the crystal class).

Singh & Ramaseshan (1968) (S&R) presented an algebraic two-wavelength method using Bijvoet sums and differences which allows calculation of the structure-factor magnitudes of the anomalous scatterers and, after solving the heavy-atom structure, the structure-factor phases. A slightly modified S&R method was used by Klop, Krabbendam & Kroon (1989) together with a direct-methods phasing technique which does not require the solution of the heavy-atom structure.

Unangst, Müller, Müller & Kleinert (1967) proposed an alternative formalism for phase determination when using anomalous-dispersion data which are based on Bijvoet ratios. Their results were derived in a limited form, using an approximation. Geometrical constructions of the multi-wavelength Bijvoet-ratio procedure for phase determination were given by Hosaya (1975). Bartunik (1978) presented an exact two-wavelength Bijvoet-ratio method for phase determination in which the real and imaginary

components of the anomalous scattering factor at the two wavelengths obey certain conditions and provided that the resonant structure is known. His method is only applicable in neutron diffraction. Cascarano, Giacobozzo, Peerdeman & Kroon (1982) have calculated structure-factor amplitudes of the anomalous scatterers *via* the two-wavelength ratio method using an approximation. Woolfson (1984) treated the two-wavelength mono-anomalous case.

Several authors have stressed the advantages of multi-wavelength methods using Bijvoet ratios rather than Bijvoet differences (Unangst *et al.*, 1967; Bartunik, 1978). These authors pointed out that there is no need for scaling of intensities in methods using Bijvoet ratios whereas Bijvoet difference methods do require scaling. Furthermore, absorption effects are thought to be much more severe in the latter than in the former because Bijvoet ratios are to a very good approximation free of absorption whereas Bijvoet sums (differences) are not. Cascarano *et al.* (1982) remark that the S&R method is only valid if the two wavelengths are chosen on the high-frequency side of the absorption edge. According to Ramaseshan & Narayan (1981) valuable information (*i.e.* the absolute values of the radius vectors in the complex plane) is ignored by using only Bijvoet ratios. Helliwell (1984) concludes that it is too early to say which approach will prove to be the most appropriate. Apparently there is consent that Bijvoet difference methods do not coincide with ratio methods.

By looking for a connection between the Bijvoet difference method and the Bijvoet ratio method, we will examine whether or not this consent is justified. The Bijvoet difference method of S&R (1968) was derived without any approximations or restrictions on the dispersion terms and included both the determination of the heavy-atom amplitudes and phase determination. However, the contributions to the ratio method presented in the literature involve approximations, restrictions or implicit formulae and deal with either amplitude or phase determination. In order to compare the Bijvoet ratio method with the S&R method it is therefore necessary to formulate the ratio method without any approximations or restrictions. To this end we will start from the ratio method of Cascarano *et al.* (1982), incorporate phase determination into the method, and compare it with the S&R method.

### Definitions

The definitions of Klop *et al.* (1989) are used throughout, some of which are repeated here for convenience.

$F_{ij}^+$  Structure factor for reflection  $\mathbf{h}_i$  at wavelength  $\lambda_j$ , not necessarily on absolute scale. [ $F_{ij}^+ \equiv F(\mathbf{h}_i, \lambda_j)$ .]

$F_{ij}^-$  Complex conjugate of the structure factor for reflection  $-\mathbf{h}_i$  at wavelength  $\lambda_j$ , on the same scale as  $F_{ij}^+$ . [ $F_{ij}^- \equiv F^*(-\mathbf{h}_i, \lambda_j)$ .]

$F_i^N$  Contribution to  $F_{ij}^+$  based on the normal parts of the scattering factors of all atoms (including anomalously scattering atoms) on absolute scale.

Other quantities are defined in the text.

### The ratio technique

Define the geometrical structure factor  $G_i$  by

$$G_i = \sum_{k=1}^N n_k T_k \exp [2\pi i \mathbf{h}_i \cdot \mathbf{r}_k]$$

where " specifies that the summation is restricted to the anomalous scatterers,  $n_k$  is the occupation factor of atom  $k$  and  $T_k$  is its isotropic temperature factor. If  $c_{ij}^{1/2}$  is the scale factor to put  $|F_{ij}^+|$  and  $|F_{ij}^-|$  on an absolute scale, and  $f_j'$  and  $f_j''$  are the real and imaginary parts of the scattering factor of the heavy atoms at wavelength  $\lambda_j$ , we have, with the assumption that the anomalous scatterers are identical,

$$c_{ij}^{1/2} F_{ij}^\pm = F_i^N + (f_j' \pm i f_j'') G_i \quad (j=1, \dots, U) \quad (1)$$

$$G_i \neq 0$$

where  $z_i \equiv F_i^N / G_i$  and  $U$  is the number of wavelengths. We use (1) to obtain

$$\frac{|z_i + (f_j' + i f_j'')|}{|z_i + (f_j' - i f_j'')|} = \frac{|F_{ij}^+|}{|F_{ij}^-|} = r_{ij}^{1/2}. \quad (2)$$

Note that the scale factors  $c_{ij}^{1/2}$  cancel. From (2) it follows that the ratio of the distances of the point  $z$  in the complex plane to the points  $-(f_j' + i f_j'')$  and  $-(f_j' - i f_j'')$  equals the square root of the Bijvoet ratio  $r_{ij}$ . If  $r_{ij} \neq 1$ , (2) is the equation of the circle of Apollonius in the complex plane. With  $z \equiv a + ib$ , (1) is written as

$$c_{ij} |F_{ij}^\pm|^2 = [(a_i + f_j')^2 + (b_i \pm f_j'')^2] g_i^2 \quad (3a, 3b)$$

where  $g_i$  is the magnitude of  $G_i$  and  $G_i \neq 0$ . Equation (2) is written as

$$(a_i + f_j')^2 + b_i^2 - 2s_{ij} b_i + f_j''^2 = 0 \quad (4)$$

where

$$s_{ij} \equiv f_j''(r_{ij} + 1)/(r_{ij} - 1) \quad (5)$$

for  $r_{ij} \neq 1$ . In a two-wavelength experiment ( $U = 2$ ) two solutions,

$$z_{i,\pm} = a_{i,\pm} + i b_{i,\pm},$$

are obtained, corresponding to the points of intersection of the circles of Apollonius for the wavelengths  $\lambda_1$  and  $\lambda_2$ . These points determine the line

$$2a_i(f_1' - f_2') = 2b_i(s_{i1} - s_{i2}) - (f_1''^2 - f_2''^2) - (f_1''^2 - f_2''^2). \quad (6)$$

The expressions for  $b_i$  at the points of intersection are found from (4) and (6) and were denoted by  $Q_{\pm}$  in the paper by Cascarano *et al.* (1982):

$$Q_{\pm} = (v_i \pm w_i)/2u_i, \quad (7)$$

where

$$u_i = [(\Delta f')^2 + (s_{i1} - s_{i2})^2] \quad (8a)$$

$$v_i = (f_1''^2 - f_2''^2)(s_{i1} - s_{i2}) + (\Delta f')^2(s_{i1} + s_{i2}) \quad (8b)$$

$$w_i = \Delta f' \{ 4s_{i1}s_{i2}[f_1''^2 + f_2''^2 + (\Delta f')^2] - 4(s_{i1}^2 f_2''^2 + s_{i2}^2 f_1''^2) - [f_1''^2 - f_2''^2 - (\Delta f')^2]^2 - 4(\Delta f')^2 f_1''^2 \}^{1/2} \quad (8c)$$

and with  $\Delta f'$  defined by  $\Delta f' \equiv f_1' - f_2'$ . This quantity was denoted by  $\Delta$  and erroneously defined as  $|f_1' - f_2'|$  in the paper by Cascarano *et al.* If  $f_1' \neq f_2'$  then  $a_i$  follows from (6) and (7). If  $f_1' = f_2'$  intersection of the line

$$2b_i = (f_1''^2 - f_2''^2)/(s_{i1} - s_{i2})$$

with the circle (4) (for  $j=1$  or  $j=2$ ) leads to the solution for  $a_i$ .

Subtraction of the equations (3) yields

$$g_{i,\pm}^2 = c_{ij} \Delta I_{ij} / 4f_j'' b_{i,\pm} \quad j=1 \text{ or } 2 \quad (9)$$

with the Bijvoet difference  $\Delta I_{ij}$  defined by

$$\Delta I_{ij} \equiv |F_{ij}^+|^2 - |F_{ij}^-|^2.$$

For (pseudo-) centrosymmetrical reflections  $c_{ij}^{1/2} |F_{ij}^+| = c_{ij}^{1/2} |F_{ij}^-| \equiv F_{ij}$ , *i.e.*  $r_{ij} = 1$ , so (4) is not applicable. Let  $\varphi_i^N$  and  $\varphi_i^H$  be the phases of  $F_i^N$  and  $G_i$  respectively. For (pseudo-) centrosymmetrical reflections,  $\varphi_i^N = \varphi_i^H \pmod{\pi}$  so  $z$  is real, *i.e.*  $b = 0$ . Intersection of any of the four circles of Apollonius

$$\frac{|z_i + (f_1' \pm i f_1'')|}{|z_i + (f_2' \pm i f_2'')|} = \frac{F_{i1}}{F_{i2}} \quad (10)$$

with the line  $b = 0$  (*i.e.* the real axis in the  $ab$  plane) yields two solutions:  $z_{i,\pm} = a_{i,\pm}$  with  $a_{i,\pm} = T_{\pm} - f_1'$ ;  $T_{\pm}$  is defined in equation (8) of Cascarano *et al.* (1982). The geometric heavy-atom amplitude  $g_{i,\pm}$  can be calculated from (3):

$$g_{i,\pm}^2 = F_{ij}^2 / [(a_{i,\pm} + f_j')^2 + f_j''^2] \quad j=1 \text{ or } 2. \quad (11)$$

If the resonant structure is known, the phases  $\varphi_i^H$  can be calculated and the  $\pm$  ambiguity can be resolved. Structure factors  $F_i^N$  are readily obtained from the calculated values for  $a_i$ ,  $b_i$ ,  $g_i$  and  $\varphi_i^H$  via the identity

$$a_i + ib_i \equiv F_i^N / G_i, \quad (12)$$

so that

$$|F_i^N| = (a_i^2 + b_i^2)^{1/2} g_i \quad \varphi_i^N = \varphi_i^H + \arg(a_i + ib_i).$$

The subscripts ( $\pm$ ) have been omitted.

### Connection with the S&R method

Expression (9) differs from the Bijvoet difference expression for  $g_{i,\pm}^2$  [equation (6) of Klop *et al.* (1989)]. The latter expression contains an extra scale factor compared with the former. The results of a two-wavelength Bijvoet difference procedure are greatly influenced if the two sets of data are not on a common scale [*e.g.* Sakamaki, Hosaya & Fukamachi (1980)]. In the paper by S&R (1968) the intensities are assumed to be on an absolute scale. However, only relative scaling (inter-wavelength scaling) is required and equation (5) of Klop *et al.* (1989) can be used to eliminate the extra scale factor in the Bijvoet difference expression, after which this expression is expected to yield the same  $g$  value as (9). This can be checked by the following calculations.

Starting from (7) and (9) we have

$$g_{i,\pm}^2 = \frac{c_{i2} \Delta I_{i2}}{4f_2'' b_{i,\pm}} = \frac{\rho_i u_i (v_i \pm w_i)}{v_i^2 - w_i^2} \quad (13)$$

where

$$\rho_i \equiv c_{i2} \Delta I_{i2} / 2f_2'' \quad (14)$$

and

$$b_{i,+} = Q_-, \quad b_{i,-} = Q_+. \quad (15)$$

The following equation easily follows from (9):

$$c_{i1} \Delta I_{i1} / f_1'' = c_{i2} \Delta I_{i2} / f_2''; \quad (16)$$

this is the same as equation (5) of Klop *et al.* (1989). Next, if (16) is used together with (14) and the definition of  $s_i$ , (5), then

$$s_{i1} \pm s_{i2} = \frac{2f_1'' c_{i1} M_{i1} \pm 2f_2'' c_{i2} M_{i2}}{c_{i1} \Delta I_{i1} \pm c_{i2} \Delta I_{i2}} = (c_{i1} M_{i1} \pm c_{i2} M_{i2}) / \rho_i \quad (17)$$

where  $M_{ij} \equiv (|F_{ij}^+|^2 + |F_{ij}^-|^2) / 2$ . After some calculation  $v_i$  and  $w_i$ , defined in (8b) and (8c), can be expressed as

$$v_i = Q_i / \rho_i \quad (18)$$

$$w_i = (Q_i^2 - R_i P)^{1/2} / \rho_i \quad (19)$$

where  $P$ ,  $Q_i$  and  $R_i$  are

$$P = [(\Delta f')^2 - (f_1''^2 - f_2''^2)]^2 + 4(\Delta f')^2 f_1''^2 \quad (20a)$$

$$Q_i = (\Delta f')^2 (c_{i1} M_{i1} + c_{i2} M_{i2}) + (f_1''^2 - f_2''^2) (c_{i1} M_{i1} - c_{i2} M_{i2}) \quad (20b)$$

$$R_i = (c_{i1} M_{i1} - c_{i2} M_{i2})^2 + \rho_i^2 (\Delta f')^2. \quad (20c)$$

These parameters are identical to the S&R parameters defined in equation (7) of Klop *et al.* (1989).  $Q_i$  should not be confused with  $Q_{\pm}$ . Next, using (17)–(19) and (20c), we obtain

$$v_i^2 - w_i^2 = R_i P / \rho_i^2 = [(\Delta f')^2 + (s_{i1} - s_{i2})^2] P = u_i P, \quad (21)$$

and collecting the results we find

$$g_{i,\pm}^2 = c_{i1}\Delta I_{i1}/4f_1''b_{i,\pm} = [Q_i \pm (Q_i^2 - R_iP)^{1/2}]/P \quad (22)$$

if condition (16) holds. Hence we find equation (6) of Klop *et al.* (1989). We conclude that for acentric reflections the solutions for the heavy-atom magnitude  $g$  calculated by the Bijvoet ratio method are equal to those calculated by the S&R method provided that the data used in the S&R expression have been brought to a common scale *via* (16). Further, it is not difficult to show that the expressions (6), (7) and (15) yield the same  $a$  and  $b$  values as (9a) and (9b) of Klop *et al.* (1989) in the S&R method if (16) is used in the S&R expressions.

In the S&R method the same expressions can be used for both acentric and centric reflections. For the algebraically simple case of (pseudo-) centric reflections both the ratio expression (11) and the S&R expression (6) (Klop *et al.*, 1989) contain two scale factors and it can be shown that the same  $g$  values are obtained in both methods. Furthermore, the same values are found for  $a$  and  $b$  ( $b = 0$ ).

### Discussion

In the two-wavelength Bijvoet ratio method proposed by Bartunik (1978) the algebraic equations are simplified by using wavelengths such that  $f_1' = -f_2'$  and  $f_1'' = f_2''$ . These conditions can be fulfilled with resonant neutron scattering, but not with resonant X-ray scattering, for which  $f' < 0$  (except for a small wavelength range). Bartunik presents a formula (3) for the phase difference  $\varphi^N - \varphi^H$  valid for acentric reflections which depends only on relative structure factors and on the dispersion terms  $f'$  and  $f''$ . Dropping the restrictions imposed on the dispersion terms and using (12), we obtain an equation which holds for both X-ray and neutron scattering:

$$\tan(\varphi_i^N - \varphi_i^H) = b_i/a_i \quad (23)$$

or, by (6), if  $f_1' \neq f_2'$ ,

$$\tan(\varphi_i^N - \varphi_i^H) = 2b_i(f_1' - f_2')[2b_i(s_{i1} - s_{i2}) - (f_1'' - f_2'') - (f_1'^2 - f_2'^2)]^{-1}. \quad (24)$$

If the resonant structure is known, the  $\pm$  ambiguity is resolved by using the  $b$  value of the solution ( $a$ ,  $b$ ,  $g$ ) with the  $g$  value closest to the one calculated from the known resonant structure. The favourable characteristics of Bartunik's equation (3), as summarized by the latter author, also apply to equation (23), which has the extra advantage that the choice of wavelengths is not dictated by conditions imposed on the dispersion terms. Specifically, it turns out that for acentric reflections, even without restrictions on the dispersion terms, the phase difference merely depends on Bijvoet ratios and dispersion terms and hence is not, or is only to a small degree, affected by absorption effects or scaling errors.

If

$$(f_1'' - f_2'') = -(f_1'^2 - f_2'^2) \quad (25)$$

then

$$\tan(\varphi_i^N - \varphi_i^H) = (f_1' - f_2')/(s_{i1} - s_{i2}). \quad (26)$$

So if the restriction given in (25) is fulfilled, there is no ambiguity in the phase difference  $\varphi_i^N - \varphi_i^H$  because (26) is independent of  $b$ . This formula was first derived by Unangst *et al.* (1967) using an approximation instead of (25).

If  $f_1' = -f_2'$  and  $f_1'' = f_2'' \equiv f''$  then (26) reduces to

$$\tan(\varphi_i^N - \varphi_i^H) = \frac{f_1'}{f''} \left( \frac{M_{i1}}{\Delta I_{i1}} - \frac{M_{i2}}{\Delta I_{i2}} \right)^{-1} \quad (27)$$

which is equation (3) of Bartunik

$$[M_{ij} \equiv (|F_{ij}^+|^2 + |F_{ij}^-|^2)/2].$$

Using the Bijvoet ratio technique we derived an expression (9) for the heavy-atom amplitude, valid for acentric reflections, which contains only one scale factor. Therefore there is no need to bring the two data sets to a common scale; in other words, there is no need for relative scaling of the data. The corresponding S&R expression [equation (6) of Klop *et al.* (1989)], however, contains two scale factors, so relative scaling is required and if this scaling is done *via* (16) the resulting amplitudes are equal to those derived by the Bijvoet ratio technique. We conclude that application of the ratio technique to acentric reflections implies that relative scaling is performed implicitly according to (16). Application of the S&R method requires explicit relative scaling, *e.g.* *via* (16) or by averaging  $c_{i1}/c_{i2}$  over a number of reflections (see Sakamaki, Hosaya, Tagai, Ohsumi & Satow, 1984).

### Concluding remarks

In the present paper a generalization and extension of previous contributions to ratio methods has been formulated. It was shown that the phase difference  $\varphi^N - \varphi^H$  depends only on Bijvoet ratios and dispersion terms, without conditions imposed on these dispersion terms. The advantages of using Bijvoet ratios summarized by Bartunik (1978) apply therefore not only to neutron scattering but also to X-ray scattering.

We have also shown that for acentric reflections the same solutions ( $a$ ,  $b$ ,  $g$ ) are found in both the S&R method and the ratio technique, if the two-wavelength data are brought to a common scale *via* (16). For centric reflections the same holds true without the latter condition and the two methods coincide. For acentric reflections the two methods coincide if (16) is used in the S&R method, in which case the advantages associated with the use of Bijvoet ratios also apply to the S&R method.

In the light of the established connection between the ratio technique and the S&R method, the view of

Cascarano *et al.* (1982) that the S&R results only apply to an experiment carried out with both wavelengths at the high-frequency side of the absorption edge is erroneous.

For acentric reflections the S&R method is more flexible than the ratio technique since it offers the possibility of using relative scaling schemes other than (16).

#### References

- BARTUNIK, H. D. (1978). *Acta Cryst.* **A34**, 747-750.  
 CASCARANO, G., GIACOVAZZO, C., PEERDEMAN, A. F. & KROON, J. (1982). *Acta Cryst.* **A38**, 710-720.  
 HELLIWELL, J. R. (1984). *Rep. Prog. Phys.* **47**, 1403-1497.  
 HOSAYA, S. (1975). *Anomalous Scattering*, edited by S. RAMASESHAN & S. C. ABRAHAMS, pp. 275-287. Copenhagen: Munksgaard.  
 KLOP, E. A., KRABBENDAM, H. & KROON, J. (1989). *Acta Cryst.* **A45**, 203-208.  
 RAMASESHAN, S. & NARAYAN, R. (1981). *Structural Studies on Molecules of Biological Interest*, edited by G. G. DODSON, J. P. GLUSKER & D. SAYRE, pp. 233-245. Oxford: Clarendon Press.  
 SAKAMAKI, T., HOSAYA, S. & FUKAMACHI, T. (1980). *Acta Cryst.* **A36**, 183-189.  
 SAKAMAKI, T., HOSAYA, S., TAGAI, T., OHSUMI, K. & SATOW, Y. (1984). *J. Appl. Cryst.* **17**, 219-225.  
 SINGH, A. K. & RAMASESHAN, S. (1968). *Acta Cryst.* **B24**, 35-40.  
 UNANGST, D., MÜLLER, E., MÜLLER, J. & KEINERT, B. (1967). *Acta Cryst.* **23**, 898-901.  
 WOOLFSON, M. M. (1984). *Acta Cryst.* **A40**, 32-34.

*Acta Cryst.* (1989). **A45**, 613-620

## The Relation of Scan Range and Reflection Shape in Single-Crystal 1D Profile Measurement

BY A. MCL. MATHIESON

*Chemistry Department, La Trobe University, Bundoora, Victoria, Australia, 3083*

(Received 19 December 1988; accepted 29 March 1989)

#### Abstract

Changes in shape of 1D profiles of small-single-crystal Bragg reflections have been examined in terms of the shapes of the components which, convoluted together, generate the profile. In most practical cases, operational features require truncation of the angular scan range of measurement and the conventional linear formula for scan range,  $\omega = a + b \tan \theta$ , is then not strictly valid. A more appropriate relationship involves a combination of root mean square (RMS) and linear (LIN) forms,

$$\omega = [(p')^2 + (q' \tan \theta)^2]^{1/2} + (p'' + q'' \tan \theta)$$

where  $p'$  is associated with the leading and trailing edges of the distribution of the combined  $\theta$ -invariant components and  $p''$  with its plateau width while  $q'$  is associated with the leading and trailing edges of the distribution of the wavelength component and  $q''$  with the separation of its outer peaks if there are more than one. For operational purposes, this relationship can be substituted with adequate precision by  $\omega = [c^2 + (d \tan \theta)^2]^{1/2}$ , but the parameters  $c$  and  $d$  do not then have a simple relationship to the  $\theta$ -invariant and  $\theta$ -variant components. Use of a conventional linear formula when a RMS one is the relevant one can mean that, in the lower  $\theta$  range, the estimate of integrated intensity will be too high and, in the higher  $\theta$  range, it will be too low, so that, with increasing  $\theta$ , a positive then a negative systematic error is introduced and not merely a negative error as the conventional interpretation of truncation holds. The conclusions of the present analysis are tested against

experimental data where the conventional treatment for truncation failed [Eisenstein & Hirshfeld (1983). *Acta Cryst.* **B39**, 61-75]. For the estimation of integrated intensity, and hence of structure factors, which are consistent over the operational range of  $\theta$ , a RMS formula for the scan range is advisable.

#### Introduction

Diffraction of a small single crystal involves measurement of a large number of Bragg reflections distributed over a wide range of scattering angle,  $\theta$ . To ensure that these reflections are placed on a mutually consistent and therefore directly comparable basis, the measurements for each reflection should be carried out over an exactly equivalent region of diffraction space, determined by the angular ranges of the respective contributing components (Mathieson & Stevenson, 1985). If one wishes to ensure that this condition is truly satisfied, then 2D measurements in  $\Delta\omega$ ,  $\Delta 2\theta$  space have the advantage that the appropriate region can be defined readily.

Until position-sensitive detectors with an authentic resolution of (say) 50  $\mu\text{m}$  are commonly available to facilitate 2D data collection of individual Bragg reflections in diffraction space, it is likely that the majority of crystal structure studies will continue to be effected using 1D measurement of the counter profile varying  $\omega$ . As one moves from reflection to reflection, this involves adjusting the scan range [and the detector aperture, except in the case of the  $\omega/2\theta$  scan mode (Mathieson, 1983)] in some systematic