A Multiple-Counter X-ray Diffractometer with Equatorial Geometry

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The design, construction and operation of a multiple-counter diffractometer are described. The instrument is used to measure up to five X-ray reflexions from single crystals quasi-simultaneously: rates of data collection from protein crystals of 250 reflexions per hour are achieved.

1. Introduction

Phillips (1964) showed that single-crystal X-ray diffractometers with either inclination or equatorial geometry may be modified to measure a number of reflexions quasi-simultaneously, thus significantly increasing the efficiency of data collection. Such a modification to a linear (inclination) diffractometer was described by Arndt, North & Phillips (1964), and instruments of this type have been operated successfully for a number of years. However, diffractometers with equatorial geometry offer greater accuracy and flexibility of operation.

The required modification to the conventional four-circle instrument is the addition of a fifth axis on the counter arm lying radially in the equatorial plane, and carrying a linear array of \(2n+1\) detectors, with an effective separation \(u\) (see §2), subtending an angle \(v\) at the crystal when the crystal to counter-aperture distance is \(L\). For a crystal mounted about a reciprocal axis with spacings \(a^*\), we must be able to set

\[
u/L = v = a^*.
\]

With proportional counters this is easiest to achieve by fixing the separation \(u\), and varying \(L\) for a given axis length.

The fifth axis is referred to as the \(\sigma\) axis, and the whole instrument is a five-circle diffractometer.

2. Counter mounting

The widest window on a commercially available proportional counter (Twentieth Century Electronics PX 28R/Xe) is 8 mm, and these counters have an external diameter of 19 mm. Such counters may be arranged in chevron arrangement behind a line of apertures, as shown by Arndt, North & Phillips (1964). These apertures must be circular as all settings of the \(\sigma\) axis are to be expected. The minimum distance between the counters, measured perpendicular to the X-ray beam, that leaves the full width of window accessible is 13.5 mm. In order to reduce the length of counter arm necessary for a given length of crystal axis, however, we have chosen to mount the counters 12.5 mm apart (at the expense of reducing the effective aperture width to 6 mm) and we have reduced the separation of apertures still further by rotating the outer counters about their axes so that their windows are brought closer to the centre. The separation \(u\) [equation (1)] of the circular apertures set in front of the counters is 9.5 mm.

A larger value of \(u\) and hence \(L\) will improve the signal-to-noise ratio because the background of scattered radiation falls off as \(1/L^2\), whereas the diffracted X-rays diverge much less quickly. An upper limit is placed on \(u\) and on \(L\), however, by the requirement that all the diffracted intensity should be collected by the counter.

Approximate expressions for the size of the diffracted beam \(D\) have been given by various authors (Burbank, 1964; Ladell & Spielberg, 1963; Alexander & Smith, 1962, 1964).

For \(\omega\) scans and small \(\theta\),

\[
D = C + \frac{L}{S} (C + F) + \max(2ML \sin \theta, 2L\delta \theta)
\]

where \(C\) is the crystal size, \(L\) is the crystal to detector distance, \(S\) is the source to crystal distance, \(F\) is the source size, \(M\) is the mosaic spread, \(\delta \theta\) is the dispersion on \(\theta\).

The mosaic spread and the dispersion terms are orthogonal, so only the larger need be considered: this will generally be the dispersion term in the worst case for a given data set, at the maximum \(\theta\).

The chosen aperture separation \(u\) of 9.5 mm is near the lower limit for these counters. In retrospect, an aperture separation of about 11 mm would probably have been better, since the increase of usable aperture to 8 mm more than compensates for the increased crystal to counter distance and diffracted beam size required. For the shorter crystal to counter distances, the maximum usable aperture is reduced by 0.5 mm because of parallax between the various parts of the aperture system. A 9.5 mm separation allows collection axis lengths of 41 to 163 Å for \(L\) in the range 25 to 100 cm (for Cu Kα radiation).
Table 1. Maximum crystal to detector distance, $L$, and the corresponding maximum collection axis length
for a counter separation of 9·5 mm, detector aperture 5·5 mm, mosaic spread less than 0·46°

<table>
<thead>
<tr>
<th>Source to crystal distance $S$ (mm)</th>
<th>Resolution $2\AA$</th>
<th>Resolution $3\AA$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Crystal size 0·5 mm</td>
<td>1·0 mm</td>
</tr>
<tr>
<td>200</td>
<td>$L_{\text{max}}$ (mm)</td>
<td>464</td>
</tr>
<tr>
<td></td>
<td>$d_{\text{max}}$ (Å)</td>
<td>75</td>
</tr>
<tr>
<td>250</td>
<td>$L_{\text{max}}$ (mm)</td>
<td>507</td>
</tr>
<tr>
<td></td>
<td>$d_{\text{max}}$ (Å)</td>
<td>82</td>
</tr>
<tr>
<td>300</td>
<td>$L_{\text{max}}$ (mm)</td>
<td>540</td>
</tr>
<tr>
<td></td>
<td>$d_{\text{max}}$ (Å)</td>
<td>88</td>
</tr>
</tbody>
</table>

Table 1 gives values of the maximum $L$ and the corresponding axial length that could be measured, for a 0·5 mm crystal, 0·4 mm source and 5·5 mm aperture at 2 and 3 Å resolutions ($\theta = 22·6$ and 14·9°), with $2\theta$ taken as 0·015 tan $\theta$ rad (Alexander & Smith, 1964). These values suggest that there are problems with crystals larger than 0·5 mm and axes longer than 80 Å at the higher resolutions. The calculation is unduly pessimistic, however, as all but a very small part of the diffracted beam is concentrated in a much smaller detector area, and the value for the dispersion term is perhaps rather large. In practice, the appropriate aperture size is determined empirically by measuring a few intensities with a range of apertures, and choosing the smallest available aperture which does not reduce the background-corrected intensity significantly. If the diffracted beam is too large for the counter window, the counters may be set at half the calculated distance from the crystal, to measure alternate reflexions along the collection axis.

### 3. Construction

Fig. 1 shows an overall view of the instrument, which is a modification of a Hilger & Watts Y230 four-circle diffractometer. The standard counter arm has been replaced by a beam with a level surface extending from 200 mm to 1 m from the crystal. The beam is supported by jockey-wheels which run on a circular track. The $\sigma$ circle is mounted on the beam and may be clamped at any distance from the crystal with its axis horizontal. Levelling screws are provided to adjust its height and tilt. The $\sigma$ circle carries a counter box with five proportional counters and five circular apertures as described in § 2. Each aperture has orthogonal half-slits to aid alignment, and the limiting apertures may vary from 2 to 5·5 mm. The $\sigma$ axis can be aligned optically to lie in the horizontal plane and to point at the common intersection of the other axes. The beam is sufficiently rigid for the vertical height of the $\sigma$ axis to vary by less than 0·2 mm between its extreme positions.

The proportional counters are xenon-filled wide-window counters (Twentieth Century Electronics PX 28R/Xe) with a stopping power of 80% for Cu $K\alpha$ X-rays. The counter electronics were purchased from J & P Ltd, Reading: they are of modular construction (NIMS) and include pulse shaping in the amplifier stage. The cables from the proportional counters to the preamplifiers are fed axially through the $\sigma$-circle mechanism. The 5·8 mm cables normally fitted to the counters have been replaced by 2·5 mm diameter ones to reduce drag on the $\sigma$ rotation. These cables are as short as possible (40 cm) consistent with a $\sigma$ rotation of 200°. The preamplifiers are hung from a slave arm, one end of which is pivoted above the $\theta$ axis. The other end is moved by a motor driving a small wheel on a circular track. A simple servo mechanism keeps the slave arm parallel to the counter arm. The cables to the preamplifier are fed along the slave arm.

A tube filled with helium is normally placed between the counters and the crystal. This is made of plastic water pipe, closed with 'Mylar' windows, and may be adjusted to any desired length.

### 4. Control system

The diffractometer is controlled through the basic unit of a 'Crystalign' control system, which is connected to a Ferranti Argus 500 computer. The micro-logic interface was built by Faul–Coradi (Scotland) Ltd, who also installed the $\sigma$-axis control electronics. The diffractometer may be operated from the manual console of the basic unit, or from a teletype linked to the control program in the computer. This program occupies about 4K 24-bit words of the Argus core store, and shares processor time with other programs. The on-line teletype is used for monitor output, and may be used for output of data to papertape: alternative output is to magnetic tape.

### 5. Setting geometry and scans

The diffractometer is designed to measure $2n+1$ ($n=0$, 1 or 2) reflexions in reciprocal lattice lines parallel to a reciprocal lattice axis (the collection axis)
as nearly simultaneously as possible. The operations needed to do this may be considered in three stages. First, the reciprocal lattice point corresponding to the central reflexion is made to lie on the Ewald sphere by choice of the crystal setting angles $\varphi$, $\chi$ and $\omega$; secondly, the line of reciprocal lattice points is set in an appropriate relation to the sphere of reflexion by rotation through an angle $\psi$ about the vector to the central point; and finally, to measure the reflexions, the crystal may be rotated about an axis which will usually, but not necessarily, be an instrumental axis.

Three ways of using the diffractometer will be described.

(a) Flat-cone geometry, $\varphi$ scan

This is the Weissenberg setting described by Phillips (1964). In this setting the line of reflexions to be measured lies tangential to the Ewald sphere. For a crystal mounted with the collection axis along the diffractometer $\varphi$ axis, this setting is given by $\varepsilon = \theta$ (where $\varepsilon = \omega - \theta$) i.e. $\omega = 2\theta$. Scanning about the $\varphi$ axis is then equivalent to use of the linear diffractometer in the flat-cone setting (Arndt, North & Phillips, 1964), and the same inaccessible or blind region exists close to the mounting axis.

The main disadvantage of this method is that the peak width on the scan varies sharply in reciprocal space. Since the rotation axis ($\varphi$) makes an angle $\varphi$ with the reciprocal lattice vector of the central reflexion, the peak width and hence the required scan width are approximately proportional to $1/\sin \varphi$. This can be allowed for in the way the measurements are made.

(b) Flat-cone geometry, $\omega$ scan

For the central reflexion, the optimum scanning axis is $\omega$, since this axis is perpendicular to the reciprocal lattice vector: $\Delta \omega$, the angle between the peak of the central reflexion and that of the outer reflexion, is always smaller than or equal to the corresponding $\Delta \varphi$ [$\Delta \omega \sim \Delta \varphi/d^* \ (\text{Phillips}, 1964)$]. Moreover, with $\omega$ scans the peak width is much more constant over reciprocal space than with $\varphi$ scans. However, reflexions symmetrically disposed about the central reflexion no longer pass through the sphere simultaneously, particularly at low values of $\varphi$.

It is not essential for the flat-cone setting that the $\varphi$ axis coincide with the collection axis, but the setting is simplest for this mounting, since $\omega = 2\theta$ and the problems of physical interference discussed in § (c) are avoided.

(c) Optimum setting

Although the $\omega$ scan seems preferable to the $\varphi$ scan, the flat-cone setting is not optimum in the sense of minimizing $\Delta \omega$. The line of reflexions to be measured is tangential to the sphere of reflexion, but when the crystal is rotated about the $\omega$ axis the outer reflexions do not cut the sphere at the same value of $\omega$. Their perpendicular distances to the sphere are the same, but in general their radii from the $\omega$ axis are different (Fig. 2). However, by rotation by an angle $\psi$ about the scattering vector for the central reflexion, one outer reflexion may be moved closer to the sphere and the other further away, such that when rotated about the $\omega$ axis they cut the sphere simultaneously. This reduces the $\omega$ rotation required to bring all the reflexions through the sphere, and is the optimum setting.

Expressions for $\psi$, which defines the setting, $\sigma$ (the setting angle for the fifth axis), $\Delta \omega$, $\Delta \gamma$ (the angular error in the detector setting) and the Lorentz factor are derived in Appendix I. The value of $\Delta \omega$ varies with $\theta$, and at low angles will be larger than the peak width of individual reflexions: in that case there is little ad-
vantage in measuring more than a single reflection at a
time. We check that \( \Delta \omega \) is less than some maximum
tolerable value, and make a similar check on \( \Delta \gamma \) to
ensure that the outer reflexions fall entirely in the
detector. \( \Delta \gamma \) is always very small, and may be approxi-
imated by

\[
\Delta \gamma \sim \frac{d^*}{2} \Delta \omega = \frac{1}{2} t^2 \tan \theta.
\]

A value of \( \psi \) defines a set of diffractometer angles \( \phi \),
\( \chi \) and \( \omega \), but this setting may not be usable, either
because the \( \chi \) circle hits the X-ray tube or the counter
arm, or because an X-ray beam is obstructed by part
of the \( \chi \) circle. The range of accessible \( \psi \) values is
smallest when \( \psi \) is near 90°, i.e. reflexions for which \( \chi \) is
near zero. This is no problem for a crystal with the
collection axis along the \( \phi \) axis, but a crystal not in this
special mounting will have a cone of reflexions close
to \( \phi = 90° \) which cannot be measured in the multi-
counter mode.

For reflexions close to the collection axis it is not
possible to find a setting which brings the outer re-
flexions to be measured through the sphere of reflexion
simultaneously. This region of reciprocal space is
given by

\[
\sin q' < \frac{d^*}{2} - t^2
\]

where \( t \) is the reciprocal spacing between the central
and outer reflexion, \( d^* \) is the reciprocal lattice vector
length for the central reflexion, and \( q' \) is the angle
between the collection axis and the reciprocal lattice
vector for the central reflexion (Fig. 3). This blind
region is very similar to that for the flat-cone setting,
which is given by \( \sin q' < d^*/2 \), since \( t \) is generally
small. Within this region it is still possible to find the
setting which minimizes \( \Delta \omega \). This is given by \( \psi = \pm 90° \),
i.e. the collection axis lies in the equatorial plane. Ex-
pressions for \( \Delta \omega \) and \( \Delta \gamma \) for this condition are
derived in Appendix II. The value of \( \Delta \omega \) must be cal-
culated for each reflexion in the line to be measured,
since the central reflexion is not necessarily the first to
cut the sphere of reflexion.

Fig. 4 shows the regions of reciprocal measurable
with \( \Delta \omega \leq 0.4° \) for reciprocal axial spacings of 0.01, 0.02
and 0.03 along the collection axis. Only a small volume
of reciprocal space is made accessible by relaxing the
criterion of simultaneous measurement of the outer
reflexions, and we have not tried to measure reflexions
in this part.

6. Strategy of data collection

Except in certain high-symmetry space groups, it is
necessary to measure the inaccessible reflexions close
to the mounting axis in another setting. This may be
done in several ways.

(a) The reflexions may be measured using a single
counter, in the conventional four-circle mode. This will
always be necessary for the lowest-angle reflexions.

(b) Another crystal may be mounted, or the same
crystal remounted about another axis.

(c) The crystal may be mounted on a right-angled
bracket to bring another axis along the \( \phi \) axis. Reflex-
ions difficult or impossible to measure on the first
mount are always those most easily measured on the
second.

(d) Since the derivation of the multi-counter setting
allows any mounting of the crystal, the missing re-
flexions can be measured about another collection axis
without remounting the crystal. The counter box has
to be moved to a new position. There should be no
problem in setting the angles, as settings corresponding
to a wide range of \( \psi \) values are physically accessible for
reflexions close to the mounting axis.

The data from the different mounts and from the
different counters must be scaled together. All the
scale factors may be determined together from overlap
between the sets (Hamilton, Rollett & Sparks, 1965),
but we prefer to measure the scale factors between
counters with a separate run at the end of the main
data collection, measuring overlapping sets of five
reflexions by stepping parallel to the collection axis.
The scale factors between data from different mounts may then be determined from the overlaps.

7. Method of intensity measurement

Possible methods of measurement are similar to those for a conventional four-circle instrument, but use can be made of the additional information from the five reflexions. Usually at least one of the five reflexions will be sufficiently strong to define the peak position, and the relative positions of the five peaks are given by the $\Delta \omega$. We have used a modification of the 'ordinate analysis' technique of Watson, Shotton, Cox & Muirhead (1970). The five peak scans are notionally shifted by $\Delta \omega$ to line up the peaks, and then the ordinates are summed. This combined profile is then scanned to find the peak centre, defined as the centre of the $n$ consecutive largest counts: $m$ counts on either side of the equivalent point in the individual profiles are taken as the peak, and the remainder as background. We usually use $m=\frac{1}{2}n$ because this method looks at the combined peaks, it suffers less from the overestimation of weak reflexions than does the single-counter method (Tickle, 1975). However, a modification of the centroid method (Tickle, 1975) to work on the combined profile might be an improvement on the 'ordinate analysis' method.

More recently, we have used a method which fits a learned profile function to the ordinates (French, 1975), in an attempt to extract the maximum information from the observed counts.

8. Number of counting channels

The proportion of reciprocal space that can be explored depends on the length of the collection axis and the maximum value of $\Delta \omega$ that can be tolerated. Table 2 shows the maximum number of counters $N$ that can be used for $\Delta \omega \leq 0.4^\circ$ for various axial lengths at different maximum values of $\sin \theta$.

It is clear that more than five channels could be usefully employed even with a collection axis length of 50 Å. This fact could perhaps be best exploited with a linear position-sensitive detector (see, for example, Faruqi, 1975) in place of the proportional counters. Such a system would also have the advantage of allowing a fixed crystal to detector distance and would make possible measurement of backgrounds between the diffracted beams.

Table 2. Maximum number of quasi-simultaneous reflexions for $\Delta \omega \leq 0.4^\circ$

<table>
<thead>
<tr>
<th>Resolution limit (Å)</th>
<th>50</th>
<th>75</th>
<th>100</th>
<th>150</th>
<th>200</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>3</td>
<td>7</td>
<td>9</td>
<td>13</td>
<td>15</td>
</tr>
<tr>
<td>3</td>
<td>5</td>
<td>9</td>
<td>13</td>
<td>17</td>
<td>21</td>
</tr>
<tr>
<td>2</td>
<td>7</td>
<td>11</td>
<td>15</td>
<td>21</td>
<td>27</td>
</tr>
</tbody>
</table>

9. Conclusions

The five-circle diffractometer is a powerful machine for measuring diffracted intensities, and has proved convenient to use. With five counters, the machine is useful for crystals with axial lengths of perhaps 50 to 130 Å and unit-cell volumes up to $10^6 \text{ Å}^3$. The data seem to compare well with data measured with a single counter on this and other diffractometers. We measure typically about 200–250 reflexions per hour, and, for example, the 20,000 or so reflexions for a 3 Å resolution data set for 3-phosphoglycerate kinase (Blake & Evans, 1974) take about four to five days to measure on one or two crystals.

We are indebted to the Medical Research Council for financial support.

APPENDIX I

Optimum setting for the five-circle diffractometer

1. Setting angles

Figs. 2 and 3 show the outermost reflexions $T$ and $W$, projected on the equatorial plane at $Q$ and $U$, passing simultaneously through the sphere of reflexion. $OS$ is the reciprocal lattice vector for the central reflexion lying in the equatorial plane: the lattice point corresponding to the central reflexion passes through the sphere at $C$, and when it is there, the reciprocal lattice points corresponding to the outer reflexions lie at $R$ and $V$ above and below the equatorial plane. The possible settings of the diffractometer may be characterized by the angle $\psi$, the rotation about the vector $OS$ (or $OC$) in a clockwise direction, so that in order to calculate the setting angles we need to find the value of $\psi$, which brings the outer reflexions $T$ and $W$ on to the sphere together. We also need to define the zero of $\psi$, and it is convenient to define it as zero when the collection vector $ST$ lies in the vertical plane through $OS$, i.e. $T$ is above $P$ in Fig. 3. The setting angle matrix for this $\psi = 0$ setting may be calculated in a way similar to that described by Busing & Levy (1967).

We may express $\psi$ in terms of $d^*$, $q^*$, and $t$: $d^*$ is the length of the vector to the central reflexion $OS$, $q^*$ is the angle between this vector $OS$ and the collection vector $ST$, and $t$ is the length of $ST$ (this may be considered negative for the other end of the collection vector, i.e. $|SW| = -t$). The components of $t$ in, and perpendicular to, the equatorial plane are respectively $p$ and $q$ (Fig. 3). $\delta$ is the projection of $q^*$ on the equatorial plane.

From Fig. 2,

$$AQ^2 = 1 - q^2$$

$$SQ = p \text{ but } t^2 = p^2 + q^2 \text{ therefore } AS^2 = 1 - t^2$$

$$OS = d^* = 2 \sin \theta \text{ (for central reflexion).}$$

Therefore
\[ \cos O\dot{S}A = \sin \delta = \frac{d^*t^2 - t^2}{2d^*\sqrt{1-t^2}} = m. \]  \hspace{1cm} (A1.1)

From Fig. 3,

\[
\sin \delta = \frac{PQ}{p} \\
PQ = PT \sin (-\psi) \\
PT = t \sin q' \\
p^2 = t^2 - q^2 \\
p = t \sin q' \cos \psi.
\]

Therefore

\[ \sin \delta = \sin q' \sin \psi / \sqrt{(1 - \sin^2 q' \cos^2 \psi)}. \]

Combining this with (A1.1)

\[ \sin^2 q' \sin^2 \psi = m^2 - m^2 \sin^2 q' \cos^2 \psi \]

i.e.

\[ \cos^2 \psi = (\sin^2 q' - m^2) / \sin^2 q'(1 - m^2). \]

With \( \psi = 0 \) defined as the collection vector in the vertical plane, we need only consider \( \psi \) in the range \(-90^\circ \leq \psi \leq +90^\circ\), since the sphere is symmetrical to reflexion in the equatorial plane. The sign ambiguity for \( \cos \psi \) may be resolved by considering the diagrams: the outer reflexion farther from \( O \) lies on the side of \( OS \) nearer \( A \). In the diagram, \( \psi \) is negative, but if \( q' > 90^\circ \), \( \psi \) must be positive, i.e. \( \psi \) has the opposite sign to \( \cos q' \).

If \( \sin q' < m \), \( \cos^2 \psi \) is negative, which means that for that reflexion there is no value of \( P \) which will make the two outer reflexions pass simultaneously through the sphere, because the value of \( \delta \) which would cause this to happen is greater than \( q' \). Such a reflexion is not accessible in this setting (see Appendix II).

From \( \psi \), the diffractometer angles \( \varphi, \chi \) and \( \omega \) may be calculated (Busing & Levy, 1967) (it should be noted that these authors use the symbol \( \omega \) for the angle here called \( \psi \)).

2. Separation of reflexions on \( \omega \)

The outer reflexions do not pass through the sphere at the same setting of the \( \omega \) axis as the central reflexion.

In Fig. 2,

\[ \Delta \omega = A\dot{O}C - A\dot{S} \]
\[ A\dot{O}C = 90^\circ - \theta \]
\[ \cos A\dot{O}S = (d^*t^2 + t^2) / 2d^*. \]

Therefore for small \( \Delta \omega \),

\[ \Delta \omega \sim (\cos A\dot{O}S - \sin \theta) / \cos \theta = t^2 / 4 \sin \theta \cos \theta. \]

Note that this is a function only of \( \theta \).

3. Counter settings

The diffracted beams for the central and outer reflexions lie along \( AC, AT \) and \( AW \), but it is a good approximation (see § 4) to set the counter apertures along \( AR, AC \) and \( AV \) (Fig. 2). These vectors lie in a plane, and the angle this plane makes with the perpendicular to the equatorial plane is the counter setting angle \( \sigma \), i.e. where \( \sigma = 0 \) the line of counter apertures is vertical. The most convenient way to calculate \( \sigma \) is to consider the rotation of the collection vector \( v_0 \) into the \( 2\theta \) frame of reference (Busing & Levy, 1967), i.e. by the notation of Busing & Levy

\[ v_{02\theta} = \Theta R v_{0\theta}, \]

where \( \Theta \) is the rotation matrix corresponding to the \( \theta \) axis, and \( R \) is the instrument angle matrix for the diffractometer angles \( \varphi, \chi, \) and \( \epsilon \). The value of \( \sigma \) may then be calculated from the two components of \( v_{02\theta} \) in the plane perpendicular to the diffracted beam.

Otherwise, \( \cos \sigma \) is given from Figs. 2 and 3:

\[ \sigma = 90^\circ - T \dot{S}Q, \text{ since } T \dot{S}A = 90^\circ, \]
\[ \cos \sigma = q / t = \sin q' \cos \psi. \]

However, we need \( \sin \sigma \) as well to get \( \sigma \) in the range of about \(-90^\circ \) to \(+90^\circ\).

With this value of \( \sigma \), equivalent pairs of reflexions of the type \( hkl \) and \( hkl \) (or say \( hkl \), if the collection axis is \( a^* \)) will be measured on different counters, except for those measured on the central counter. To ensure that these equivalent reflexions are measured on the same counter, we invert \( \sigma \) to \( \sigma - 180^\circ \) for one equivalent, and normally use \( \sigma \) in the range 0 to \(-180^\circ\).

4. Error in counter settings

If the detector for the outer reflexion is set along \( AR \), the angular error in its position is \( TAR \). Writing \( OQ \) as \( s \), and assuming \( \Delta \omega \) is small \( TAR \sim TR \cos ATO = \frac{1}{2} s \Delta \omega \), where \( r = OT \).

This angle is always very small, and the component perpendicular to the equatorial plane is even smaller, since \( R \) and \( T \) are at the same distance from the equatorial plane. As a cruder but adequate approximation, we may replace \( rs \) by \( d^*t^2 \), then

\[ \Delta \gamma \sim \frac{1}{2} (1 - q^2) TAR \sim \frac{d^*t^2}{2} \Delta \omega = \frac{1}{2} t^2 \tan \theta \]

where \( \Delta \gamma \) is the equatorial component of the counter setting error. The setting angle of the counter arm, \( \theta \), may be offset by \( \frac{1}{4} \Delta \gamma \) to minimize the effect of this error.

5. Lorentz factor

The inverse Lorentz factor for the outer reflexion \( T \) is the component along \( TA \) of its motion when rotated about the \( \omega \) axis through \( O \). This may be considered in its resolved components in and perpendicular to the equatorial plane.

\[ L^{-1} = OQ \sin A\dot{Q}O \cos TAQ \]
\[ = s \sqrt{(1 - q^2) \sin A\dot{Q}O} \]
\[ \cos A\dot{Q}O = \frac{s^2 - q^2}{2s} \frac{1}{(1 - q^2)}. \]

Therefore

\[ L^{-1} = \frac{1}{2} \left[ 4s^2 - (s^2 + q^2)^2 \right] = \frac{1}{2} \left( 4s^2 - r^2 \right). \]

where \( r = OT \).
\[ \Delta \omega = A \hat{\omega} R - A \hat{\omega} T \]
\[ \sim \frac{\cos A \hat{\omega} T - \cos A \hat{\omega} R}{\sin A \hat{\omega} T} \text{ for small } \Delta \omega \]
\[ \cos A \hat{\omega} T = r/2 \]
\[ \cos A \hat{\omega} R = \frac{1}{2r} \left[ r^2 - t^2 - 2t \sin (\theta - \phi') \right]. \]

Therefore
\[ \Delta \omega = \frac{t^2 + 2t \sin (\theta - \phi')}{r\sqrt{4 - r^2}}. \]

This must be evaluated for each reflection, since the central reflexion does not necessarily cut the sphere first.

2. Counter setting error
\[ \Delta \gamma \sim r \Delta \omega \cos A \hat{\omega} O \]
\[ = \frac{r^2}{2} \Delta \omega. \]

**APPENDIX II**

**Setting for reflexions inaccessible in the optimum setting**

For reflexions with \( \sin \phi' < m \), it is not possible to make the outer reflexions pass through the sphere simultaneously, but their separation can be minimized by maximizing \( \delta \). Clearly, \( \delta = \phi' \), and \( \psi = \pm 90^\circ \). By the same argument as before for the sign of \( \psi \),
\[ \psi = -90^\circ \text{ if } \cos \phi' < 0 \]
\[ \psi = +90^\circ \text{ if } \cos \phi' > 0. \]

All diffracted beams lie on the equatorial plane, so \( \sigma = \pm 90^\circ \).

1. *Separation of reflexions on \( \omega \)*

In Fig. 5,
\[ OR^2 = OT^2 = r^2 = d^2 + t^2 + 2d^*t \cos \phi' \]
\[ AR^2 = 1 + t^2 + 2t \sin (\theta - \phi') \]

**References**


