the close vicinity of the maximum of the reflection curve, where, however, the shape of the reflection curve is not dependent on the type of the deformation field of the defects.

The results obtained enable us to explain the contrast of the growth striations with microdefects in double-crystal topography. From the above results it is obvious that for all angles of incidence of the primary wave the intensity diffracted from the region with the microdefects is greater than that from the perfect crystal. Thus, in the case of diffraction which is not sensitive to the variation of the mean lattice parameter in the striations, the striations with microdefects have a black contrast.

References

DEDERICHS, P. H. (1971). *Phys. Rev.* B4, 1041–1050.
DEDERICHS, P. H. (1972). *Dynamical Diffraction Theory by Optical Potential Methods. Solid State Physics*, Vol. 27. New York and London: Academic Press.
ESHELBY, J. D. (1956). *The Continuum Theory of Lattice Defects. Solid State Physics*, Vol. 3. New York and London: Academic Press.
HOLÝ, V. (1982a). *Phys. Status Solidi B*, 111, 341–351.
HOLÝ, V. (1982b). *Phys. Status Solidi B*, 112, 161–169.
HOLÝ, V. (1983). *Acta Cryst.* A39, 642–646.
KATO, N. (1980). *Acta Cryst.* A36, 763–769, 770–778.
LARSON, B. C. & SCHMATZ, W. (1980). *Phys. Status Solidi B*, 99, 267–275.
OLEKHNOVICH, N. M. & OLEKHNOVICH, A. I. (1981). *Phys. Status Solidi A*, 67, 427–433.
TRINKAUS, H. (1972). *Phys. Status Solidi B*, 54, 209–218.

Printing Sets of Structure Factors for Coping with Orientation Ambiguities and Possible Twinning by Merohedry

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Abstract

Any crystal whose point group is a subgroup of index p of its lattice symmetry (merohedral crystals, $p = 2$, 4 or 8) can be referred to p non-equivalent possible settings of the cordinate axes. These possible settings are tabulated, for all 44 oriented point groups and for each setting s, as the triplet of indices $h_s k_s l_s$ into which the original hkl transforms, together with the relevant transformation operation. At least one such set of suitable observed structure factors should be published with every structure description for merohedral crystals so that the chosen setting can be identified. A list of the types of reflections unsuitable for orientation purposes is given. A unique orientation of the coordinate axes could be reached experimentally, without structural knowledge, by attributing to the largest I_{obs}, indices hkl in a specified asymmetric domain of the point-group symmetry of the lattice. The table also serves as a complete collection of possible twin laws in twinning by merohedry; each transformed symbol $h_s k_s l_s$ represents the reflection which, on the diffraction pattern of the twin, contributes its intensity to that of hkl.

Introduction

When measuring physical properties of crystals previously described in the literature, it is essential to identify, among the possible crystal settings, the one that was used for the structure solution. The crystallographer who collects diffraction intensities for a crystal of known structure is confronting the same problem. As long as the intensity data were tabulated in the original publication, the retrieval of the crystal setting was straightforward: the reciprocal axes were simply chosen in such a way that the intensities of the indexed reflections would match those of the published ones.

At the present time intensity data are no longer published, being instead deposited in manuscript form in some archival center. As a consequence, ambiguities in the identification of the axial setting arise in all crystals but those with the same point-group symmetry as their lattice symmetry (holosymmetric crystals) or with all its symmetry axes (holoaxial hemisymmetry, column 2 in Table 1) provided the latter are referred to a right-handed coordinate system, RHCS. This situation exists even if metric

conventions governing cell shape and size, such as those of *Crystal Data* (1972–78), which were recommended by the IUCr Commission on Crystallographic Data (Kennard, Speakman & Donnay, 1967) are used. If the adopted setting turns out to be different from the original one, the intensities of reflections with the same indices in both settings will no longer match, and the atomic coordinates in the new description will differ from those in the original one.

The present paper deals with the ambiguities that arise from symmetry in indexing reflections in reciprocal space, a point-group problem that does not involve any uncertainty of origin and thus limits the choices to those that are due to the orientation of the coordinate system with respect to the crystal. Other ambiguities resulting from fortuitous equalities of cell dimensions or special values of inter-edge angles not imposed by symmetry will not be considered. For example, the case of a monoclinic crystal with $\beta = 90°$, which leads to the possible settings hkl and $hk\bar{l}$ obtained by rotation $[100]_\pi$ is not treated here.

The problem of finding a unique setting and origin for crystal-structure description and structure-type identification has been solved for all space groups by Parthé & Gelato (1984), who made use of the results of Fischer & Koch (1983) on the equivalent point configurations due to Euclidean normalizers of space groups. However, their considerations apply to compilations of published structural results and do not tell the experimentalist how to orient the crystal at the start of the structure determination.

Cause of setting ambiguities

Ambiguities arise for any crystal whose point symmetry is a subgroup of its lattice point symmetry. We list the alternate symbol into which the triplet hkl transforms when one of the additional symmetry operations of the lattice is applied to the set of coordinate axes (Table 1). It follows that the number of such distinct triplets equals that of the non-equivalent settings or alternative orientations of the set of coordinate axes. The lattice-symmetry operation that is applied is shown (in Table 1) by the symbol of the corresponding symmetry element, *e.g.* [001] for a 180° rotation about the c axis, $[001]_{\pi/2}$ a 90° rotation, (010) for a reflection in (010), ($\bar{1}$) for an inversion in the center. Note that the symbols used in Table 1 apply to both direct and reciprocal lattices.

The case of observable anomalous dispersion

The anomalous dispersion will first be assumed sufficiently intense for all the 44 oriented subgroups of lattice point symmetries (Donnay, 1977) to be identified from the cell data and observed intensities. The number of possible settings (Table 1) is then

equal to the index of the subgroup under consideration: eight for 311 (the only ogdosymmetry), four for the three tetartosymmetries (eight subgroups in all), and two for the three hemisymmetries (which total 25 oriented subgroups). In every holoaxial group, for the non-enantiomorphic space groups, the number of possible settings can be halved if, by convention, only a RHCS is used. The case of enantiomorphic space groups is somewhat different: in a structure, described in an enantiomorphic space group with right-hand screws, $P3_112$ for instance, the number of possible settings is also halved, as only those referred to the RHCS need be considered. For the enantiomorph with left-hand screws the settings identified as LHCS in Table 1 are available for use in $P3_212$, where they will produce the same numerical values for the coordinates as a RHCS in $P3_112$. In such a case the publication should give the structure of the enantiomorph of the opposite hand (*i.e.* with right-hand screws) in $P3_112$ with RHCS, even though the material studied was left-handed (Donnay & Le Page, 1978).

Care has been taken to select the RHCS for alternate settings whenever there is a choice, namely for the non-centrosymmetric, non-holoaxial crystals (antimerosymmetries in columns 3 and 6, Table 1). For example, in $4mm$ the alternate-setting operations $(001)_z$, ($\bar{1}$), $[010]_\pi$ and $[110]_\pi$ lead to the triplets $hk\bar{l}$, $\bar{h}\bar{k}\bar{l}$, $hk\bar{l}$ and $kh\bar{l}$, which are equivalent in $4mm$ symmetry. We avoid (001) and ($\bar{1}$) because they lead to a LHCS; the choice between the two rotations is arbitrary; [110] is used in Table 1.

In order to permit easy retrieval of the original setting and spare the reader the trouble of ordering the list of deposited intensity data, authors of structure papers for compounds with more than one possible setting s could include, in the text to be published, a set of $h_s k_s l_s$ listed in order of decreasing observed structure factors or intensities. Only one reflection of the set, say the strongest, is needed to fix the choice of axes; the others will serve to check that the choice made is the correct one. The non-orienting types of reflections (NOR) are listed in Table 2 for all 44 oriented point groups. A RHCS is assumed in all cases. The holoaxial merosymmetries have then the same non-orienting reflections as the corresponding Laue symmetries and have been put together. The non-orienting types of reflections for Laue groups and paramerohedries are those for which the alternative triplets are crystal-symmetry equivalent to the initial triplet. Identity is of course a crystal-symmetry operation.

Unobserved anomalous dispersion

If the X-ray diffraction patterns indicate or, in the absence of anomalous dispersion, simulate a centrosymmetric crystal, only the Laue symmetries need

Table 1. *The possible distinct axial settings in the 44 oriented crystal point groups*

Holosymmetry = lattice symmetry.
Merosymmetry = any proper subgroup of the holosymmetry.
Hemi.: subgroup of index 2.
Tetarto.: subgroup of index 4.
Ogdo.: subgroup of index 8.
Holoaxial: axes only.
Para-: centrosymmetric.
Anti-: neither holoaxial nor paramerosymmetric.

	Holosymmetric Laue classes			Hemisymmetric Laue classes			Tetartosymmetric Laue classes	
	Holo.	Holoaxial hemi.	Antihemi.	Parahemi.	Holoaxial tetarto.	Antitetarto.	Paratetarto.	Ogdo.
a	1̄ hkl	1 hkl °h̄k̄l̄ (1̄)						
m	2/m . hkl	2 hkl °h̄k̄l̄ (1̄)	m hkl h̄kl̄ [010]					
o	2/m 2/m 2/m hkl	222 hkl °h̄k̄l̄ (1̄)	mm2 hkl h̄kl̄ [010] 2mm hkl h̄k̄l [001] m2m hkl hk̄l̄ [100]					
t	4/m 2/m 2/m hkl	422 hkl °h̄k̄l̄ (1̄) 4̄2m 4mm hkl khl̄ [110]	4̄m2 hkl h̄kl̄ [010]	4/m hkl h̄kl̄ [010]	4 hkl h̄kl̄ [010] °h̄k̄l̄ (1̄) °hk̄l (010)	4̄ hkl h̄kl̄ [010] khl [110] khl [001]$_{\pi/2}$		
r	3̄ 2/m hkl	32 hkl °h̄k̄l̄ (1̄)	3m hkl k̄h̄l̄ [11̄0]	3̄1 hkl k̄h̄l̄ [11̄0]	31 hkl k̄h̄l̄ [11̄0] °h̄k̄l̄ (1̄) °k̄h̄l (11̄0)	Rhombohedral axes		
	hkl	hkl °h̄k̄l̄ (1̄)	hkl kh̄l̄ [110]	hkl kh̄l̄ [110]	31 hkl kh̄l̄ [110] °h̄k̄l̄ (1̄) °k̄h̄l (11̄0)	Hexagonal axes		
h	6/m 2/m 2/m hkl	622 hkl °h̄k̄l̄ (1̄)	6mm hkl kh̄l̄ [110]	6/m hkl kh̄l̄ [110]	6 hkl kh̄l̄ [110] °h̄k̄l̄ (1̄) °k̄h̄l (110)	6̄ and 3m1 31m hkl h̄k̄l [001] kh̄l̄ [110] kh̄l̄ [1̄10]	3̄11 hkl h̄k̄l [001] kh̄l̄ [110] kh̄l̄ [1̄10]	311 hkl h̄k̄l [001] kh̄l̄ [1̄10] °h̄k̄l̄ (1̄) °hk̄l̄ (001) °k̄h̄l (110) °k̄h̄l (1̄10)
		6̄m2 6̄2m hkl h̄k̄l [001]	3̄2/m1 3̄12/m hkl h̄k̄l [001]	321 312 hkl h̄k̄l [001] °h̄k̄l̄ (1̄) °hk̄l̄ (001)				
c	4/m 3̄ 2/m hkl	432 hkl °h̄k̄l̄ (1̄)	4̄3m hkl khl̄ [110]	2/m 3̄* hkl khl̄ [110]	23 hkl khl̄ [110] °h̄k̄l̄ (1̄) °k̄h̄l̄ (110)			

Abbreviations
a anorthic (triclinic), m monoclinic, o orthorhombic, t tetragonal, r rhombohedral, h hexagonal, c cubic.
Transformation operation (= twin law): [...] half-turn rotation; (...) reflection in plane; (1̄) inversion in point.
° Left-hand coordinate system (LHCS).
(The dot stands for 'symmetry'.)

* In space group Pa3̄ the alternate setting is eliminated by the extinction criterion hkl: $k = 2n$ (but not $l = 2n$).

be considered. No ambiguity remains if the Laue symmetry of the crystal is the lattice symmetry (holosymmetric Laue classes, Table 1, columns 1, 2 and 3). If the Laue symmetry of the crystal possesses only half the number of operations in the lattice symmetry (hemisymmetric Laue classes, columns 4, 5 and 6), there exist two possible settings. In the tetartosymmetric Laue classes (columns 7 and 8), four settings are possible (reduced from eight in the ogdosymmetry). The $h_s k_s l_s$ reflections chosen for publication should be selected so as to cover a wide range of intensities.

Twinning by merohedry

Note that Table 1 can also be construed as a listing of twin laws for twinning by twin-lattice symmetry (Donnay & Donnay, 1974) with twin index $n = 1$,

Table 2. *Non-orienting and twin-proof reflections (NORs)*

The ± sign that precedes one index in the triplets of point group $2/m\,\bar{3}$ affects only that one index. The convention to use only the RHCS results in the NORs of the holoaxial subgroups being the same as those of the corresponding Laue symmetry. The indices of the h system refer to hexagonal axes of coordinates.

Laue symmetries and subgroups		NORs ± h, ± k, ± l; ± includes zero
a	$\bar{1}$	1	—
m	$2/m$	2	—
		m	0k0
o	$2/m\,2/m\,2/m$	222	—
		mm2	hk0
		m2m	h0l
		2mm	0kl
t	$4/m\,2/m\,2/m$	422	—
		4mm	hk0
		$\bar{4}m2$	,hk0; hhl, $\bar{h}$hl
		$\bar{4}2m$	hk0; h0l, 0kl
	$4/m$	4	h0l, 0kl; hhl, $\bar{h}$hl
		$\bar{4}$	hk0; and as in 4/m
r	$\bar{3}\,2/m$	32	—
		3m	—
		in r axes	h$\bar{h}$0 see $\bar{3}$1
		in h axes	hh0
	$\bar{3}1$	31	—
		in r axes	hhl, hkk, lkl; h$\bar{h}$0, 0kk, $\bar{l}$0l
		in h axes	h0l, 0kl, $\bar{h}$hl; hh0, h2$\bar{h}$0, 2$\bar{h}$h0
h	$6/m\,2/m\,2/m$	622	—
		6mm	hk0
		$\bar{6}2m$	h0l see $\bar{3}$1
		$\bar{6}m2$	hhl see $\bar{3}$11
	$6/m$	6	h0l see $\bar{3}$1
			hhl see $\bar{3}$11
		$\bar{6}$	as in 6/m
	$\bar{3}\,2/m\,1$	321	hk0; hhl see $\bar{3}$11
		3m1	as in $\bar{3}\,2/m\,1$
	$\bar{3}\,1\,2/m$	312	hk0; h0l see $\bar{3}$1
		31m	as in $\bar{3}\,1\,2/m$
	$\bar{3}11$	311	hk0; h0l see $\bar{3}$1; hhl, h2$\bar{h}$l, 2$\bar{h}$hl
c	$4/m\,\bar{3}\,2/m$	432	—
		$\bar{4}3m$	hk0, h0l, 0kl
	$2/m\,\bar{3}$	23	±hhl, h±kk, lk±l

which is twinning by merohedry in the Friedel classification. Every alternative setting then represents a twinned orientation, like that of 'crystal no. 2' in a dual twin. The transformed symbol (Table 1) represents the X-ray reflection that is superposed onto the original *hkl* on the diffraction pattern of a twin. The possibility of such penetration twinning must, of course, always be taken into account. The availability in the literature of the discriminating structure factors, presumably obtained on a single crystal, will allow one to know the extent of twinning in the sample he is examining. A way to estimate *a priori* the maximum fraction of crystal 2 is discussed under *Example*.

The 'twin-proof' reflections (TPR) are the same as the NORs in Table 2, namely those that receive symmetry-related intensity contributions from the constituent crystals of the twin. They are indistinguishable from single-crystal reflections. When more than one twin law is possible, in the tetarto- and ogdosymmetric point groups, care must be taken to identify the NORs (here to be thought of as TPRs) that are

associated with each individual twin operation. Although the NORs cannot be used to resolve the orientation problem, they are the most useful reflections when it comes to solving a crystal structure from twin data. If, for example, a specimen shows the pseudosymmetry $6/m\,2/m\,2/m$, and the crystal point group is known to be $\bar{3}\,2/m\,1$, then the Patterson projections on (00.1) and $(1\bar{1}.0)$ using the NORs $F^2(hk0)$ and $F^2(hhl)$, respectively (Table 2), will show only the interatomic peaks of a single crystal. These peaks will be of great help for distinguishing, on the three-dimensional Patterson synthesis, the peaks that arise from the second crystal in the alternative orientation.

Suggestion for unique crystal orientation

If two authors determine a new structure independently, it is desirable that they select the same crystal setting experimentally. This could be achieved provided both used the same kind of radiation (*i.e.* X-rays, neutrons or electrons): their lists of observed intensities, in decreasing order, will be similar with some interchanges due to statistics and to differences in scattering factors, temperatures or Lp ratios (diffractometer data are assumed). Nevertheless, the chances are good that the reflection at the top of their lists will be the same. After finding the strongest reflection, which can be used as an orienting reflection (Tables 2 and 4), this reflection will be given indices that place it within, or on, the boundaries of the asymmetric domain pertaining to the lattice symmetry (Table 3), so that a unique setting results.* The example given (Table 4) also illustrates how twinning by merohedry can be detected by considering intensity ratios of reflections related by the twin operation (the same as the alternate-setting operation).

For centrosymmetric and non-centrosymmetric point groups that contain symmetry operations of the second kind (columns 1, 3, 4, 6 and 7 in Table 1), the unique setting can always be obtained with a RHCS because all listed alternate settings were selected with a RHCS. For holoaxial point groups, if anomalous dispersion is observed for the orienting reflection, the unique setting above might correspond to either a RHCS or a LHCS. If a LHCS is obtained, all coordinate axes should be reversed and the Bijvoet-related reflection of the most intense one will be found in the desired asymmetric domain.

The orienting procedure described has the advantage that it leads to a unique choice of axial orientation at the start of the investigation without knowledge of the crystal structure and requires only a minimum of conventions, premeasurements or calculations.

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* For space group $Pa\bar{3}$, the systematic absences fix the orientation. The above procedure is therefore unnecessary.

Table 3. *Asymmetric domains of the seven lattice symmetries*

These are identical to those given by Gabe (1970); see also Le Page & Gabe (1979).

Lattice symmetry	Boundary planes through origin	Type of reflections	Extra conditions†
$\bar{1}$	(010)*	hkl	$\pm h, (k \geq 0), \pm l$
		h0l	$\pm h, (l \geq 0)$
		h00	$(h \geq 0)$
2/m	(010),(001)*	hkl	$\pm h, (k, l \geq 0)$
		hk0	$(h, k \geq 0)$
2/m 2/m 2/m	(100),(010),(001)	hkl	$(h, k, l \geq 0)$
4/m 2/m 2/m	(010),(1$\bar{1}$0),(001)	hkl	$h \geq k$
$\bar{3}$ 2/m	rhombohedral axes (111),(1$\bar{1}$0),(0$\bar{1}$1)	hkl	$\pm k, \pm l; h \geq k \geq l; h+k+l \geq 0$
		$-(k+l)kl$	$0 \geq k \geq l$
	hexagonal axes (001),($2\bar{1}$0),($1\bar{2}$0)‡	hk0	$h \geq k$
6/m 2/m 2/m	(001),(1$\bar{1}$0),($1\bar{2}$0)‡	hkl	$h \geq k$
4/m $\bar{3}$ 2/m	(001),(1$\bar{1}$0),(01$\bar{1}$)	hkl	$h \geq k \geq l$

* These planes are net planes in the reciprocal lattice only.
† Unless otherwise stated $h, k, l \geq 0$ is implied; ±values include zero; parentheses denote implied conditions stated for clarity.
‡ ($2\bar{1}$0) and ($1\bar{2}$0) correspond respectively to (100)* and (010)*.

Table 4. *Most intense X-ray reflections of cafarsite, $Ca_5Fe_6Ti_2 (AsO_4)_{12}.4H_2O$, from Italy; $a = 15\cdot945$ Å, space group Pn$\bar{3}$*

$2\theta(°)$ (Mo Kα graphite monochromator)	I (integrated net counts)	hkl (first choice of axes)*	$\dfrac{I_{hkl}}{I_{khl}} > 1$ (first choice)	hkl second choice of axes
14·45	168 837	440; NOR	1	440
20.50	72 143	800; NOR	1	080
14·90	60 079	530; OR	1·6	350
25·16	40 790	844; NOR	1	484
24.35	39 849	574	3·9	754
14.90	37 967	350	—	530
23·23	31 704	190	13·7	910
13·02	30 967	341	2·3	431
13·02	13 427	4$\dot{3}$1	$\dot{-}$	3$\dot{4}$1
24·35	10 243	5$\dot{5}$4	$\dot{-}$	5$\dot{7}$4
23·23	2·307	9$\dot{1}$0	$\dot{-}$	1$\dot{9}$0
⋮	⋮	⋮	⋮	⋮

* This choice of axial orientation is also the one in the literature (Edenharter *et al.*, 1977).

Example

An actual example will illustrate the method of orienting a crystal and also of coping with twinning. Cafarsite, an arsenate mineral, belongs to point group 2/m $\bar{3}$, space group Pn$\bar{3}$. Its structure was determined by Edenharter, Nowacki & Weibel (1977). One of us (YLP) was asked to refine it and therefore needed to recover the previous setting. Table 4 (column 2) lists the intensities measured, indexed with respect to the first choice of setting which had a 50% probability of being the one in the literature. The only available alternate setting, in this case, is obtained (Table 1) by a 180° rotation about [110]. Indices hkl transform in the alternate setting to kh$\bar{l}$, which by reflection in (001) is equivalent to khl, the indices given here (Table 4, column 4). The strongest two reflections are NORs

(Table 2). The third strongest, however, is 530, which is not a NOR (Table 2) and thus becomes the orienting reflection; it obeys the conditions $\bar{h} \geq k \geq l$, which corresponds to the lattice symmetry 4/m $\bar{3}$ 2/m (Table 3), and so is to be given as orienting reflection in the publication. The first choice of setting eventually turned out to have been the literature setting.

For the purpose of putting on record an arbitrary choice of axes, in a paper to be published, by giving one hkl and its $h_sk_sl_s$, reflections 530 and 350 are not the best ones to use, because their intensity ratio is only 1.6 (Table 4, column 6). A pair of reflections differing much in intensity, such as 190 and 910 with intensity ratio 13·7, serves the purpose better: it leaves no doubt as to the chosen setting, even with a poor sample.

All NORs show I_{hkl}/I_{khl} ratio equal to unity, as is to be expected from twin-proof reflections. Other twin-related alternative-setting ratios can reveal the absence of twinning by merohedry when their values differ greatly from unity. Our cafarsite data include the intensity ratio $I_{821}/I_{281} \sim 100$, which enables the maximum volume fraction of 'crystal no. 2' (the smaller one) to be estimated at 0·01 in the twin (if the specimen *is* a twin), on the assumption that, in a single crystal, I_{281} would in the worst case be zero. If it were not zero, the volume fraction of crystal no. 2 would be even smaller. On refinement our specimen turned out to be monocrystalline.

It should be clear that an hkl and its alternate triplet, one of which is a reflection of high intensity whereas the other is structurally absent, are ideal for listing as crystal-orientation guide, as well as for disproving twinning. If the intensity ratios are found to be near unity for all the observed reflections, the probability of twinning being present in the specimen is also close to unity, with volume fractions of the constituents nearly equal. Should twinning be very intimate and repeated, these volume fractions may indeed be equal, in which case the twin (point-group) symmetry will simulate a crystal (point-group) symmetry. This phenomenon, however, will not be further considered here.

References

Crystal Data (1972-78). 3rd ed., vols. 1-4. Swarthmore, PA: JCPDS, International Center for Diffraction Data.
DONNAY, G, & DONNAY, J. D. H. (1974). *Can. Mineral.* **12**, 422-425.

DONNAY, J. D. H. (1977). *Acta Cryst.* A33, 979–984.
DONNAY, J. D. H. & LE PAGE, Y. (1978). *Acta Cryst.* A34, 584–594.
EDENHARTER, A., NOWACKI, W. & WEIBEL, M. (1977). *Schweiz. Mineral. Petrogr. Mitt.* 57, 1–16.
FISCHER, W. & KOCH, E. (1983). *Acta Cryst.* A39, 907–915.

GABE, E. J. (1970). *Crystallographic Computing*, edited by F. R. AHMED, pp. 331–335. Munksgaard. Copenhagen.
KENNARD, O., SPEAKMAN, J. C. & DONNAY, J. D. H. (1967). *Acta Cryst.* 22, 445–449.
LE PAGE, Y. & GABE, E. J. (1979). *J. Appl. Cryst.* 12, 464–466.
PARTHÉ, E. & GELATO, L. (1984). *Acta Cryst.* A40, 169–183.

Experimental Procedures for the Determination of Invariant Phases of Centrosymmetric Crystals

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Abstract

The [222] n-beam pattern of germanium has been recorded using Cu $K\alpha_1$ radiation and newly developed instrumentation. The asymmetric unit of the pattern contains 17 interaction maxima. At least one invariant triplet phase may be derived from each maximum. Two of the latter are considered unsuitable for phase determination owing to overlap with adjacent peaks. The phases of the remaining 15 were determined. Relevant experimental data are presented and from these the validity of the authors' phase assignments can be readily checked. Techniques for indexing n-beam patterns and for improving the visibility of triplet phase interactions are discussed. It is shown that procedures for improving the visibility of *n-beam interactions* do not necessarily lead to increased visibility of *phase indications*.

1. Introduction

One of the authors (J. Ladell) has recently completed construction of a novel four-circle single-crystal diffractometer for use in diffraction experiments. The (biaxial) diffractometer utilizes inclination geometry, features two mutually orthogonal crystal rotation axes, and provides for moving the detector on a spherical surface. It is used in conjunction with a highly collimated and monochromatic incident beam, and provides the capability of controlling specimen orientation and detector placement with high precision. It has been used for the experimental determination of the invariant phases of perfect crystals of germanium and mosaic crystals of lead molybdate, zinc tungstate and other specimens.

In this paper we will discuss the use of this new equipment for the experimental determination of the phases of germanium. We will also discuss general aspects of n-beam diffraction, which, to the best of our knowledge, have not been dealt with previously.

The selection of germanium for this investigation requires some explanation. Its crystal structure is simple; all atomic positions are fixed by symmetry. The phases of all but a few very weak reflections can be easily calculated. It is, however, largely because of that simplicity that we have chosen to demonstrate the phase procedure by applying it to germanium. Relevant experimental data are given so that the reader can readily check the validity of our phase assignments by calculating the phases independently, and comparing the results with the experimental evidence and with the authors' phase assignments.

2. Experimental

Techniques for generating n-beam diffraction systematically and for calculating the azimuthal angles at which n-beam interactions occur have been discussed by Cole, Chambers & Dunn (1962) and Post (1975). Additional details, with emphasis on the application of n-beam diffraction to experimental phase determination, are given by Gong & Post (1983) and Post (1983).