International Tables for Crystallography (2010). Vol. B, Section 2.5.3.3.4, pp. 333–334.

Table 2.5.3.10. *Space-group sets indistinguishable by dynamical extinction lines*

(1) $P3$, ($P3_1$, $P3_2$)	(2) $P312$, ($P3_112$, $P3_212$)	(3) $P321$, ($P3_121$, $P3_221$)
(4) $P6$, ($P6_2$, $P6_4$)	(5) $P622$, ($P6_222$, $P6_422$)	(6) $P6_3$, ($P6_1$, $P6_5$)
(7) $P6_322$, ($P6_122$, $P6_522$)	(8) $P4$, $P4_2$	(9) ($P4_1$, $P4_3$)
(10) $P4/m$, $P4_2/m$	(11) $P4/n$, $P4_2/n$	(12) $P422$, $P4_222$
(13) $P42_12$, $P4_22_12$	(14) $I4$, $I4_1$	(15) $I422$, $I4_122$
(16) $I23$, $I2_13$	(17) $I222$, $I2_12_12_1$	(18) $P432$, $P4_232$
(19) ($P4_132$, $P4_332$)	(20) $I432$, $I4_132$	(21) $F432$, $F4_132$
(22) ($P4_122$, $P4_322$)	(23) ($P4_12_12$, $P4_32_12$)	

distinguishes the difference in the relative arrangements of twofold rotation axes and 2_1 screw axes along the [111] direction between the two space groups by examining the symmetry of intensity pairs appearing in the overlapping discs of a coherent [111] ZOLZ pattern. Saitoh, Tsuda *et al.* (2001) extended the method to distinguish the other ten indistinguishable space-group pairs. The method can distinguish between a space group which is composed of a principal rotation axis and a twofold rotation axis like $P321$ and a space group which is composed of a principal screw axis and a twofold rotation axis like $P3_121$ (or $P3_221$) by investigating the difference in the relative arrangements of the twofold rotation axis with respect to the principal axis. Table 2.5.3.11 shows the 12 space-group pairs which are distinguishable by applying the coherent CBED method.

The pairs in parentheses form left- and right-handed space groups. Handedness or chirality may occur in space groups that do not possess mirror and/or inversion symmetry. The handedness of space groups is identified in such a way that the senses of two crystal axes are determined with the aid of kinematical structure-factor calculations and the sense of the third axis is determined with the aid of dynamical calculations. This method was used for quartz by Goodman & Secomb (1977) and Goodman & Johnson (1977) and for MnSi by Tanaka *et al.* (1985). We also mention that Taftø & Spence (1982) developed a simple but clever method without computation for determining the absolute polarity of the sphalerite structure utilizing multiple-scattering effects on weak beams, which are almost independent of thickness. Because of the importance of structure in the field of semiconductor science, this method is conveniently used nowadays to determine polarity.

It is worth mentioning that space groups that are indistinguishable by CBED (Table 2.5.3.10) do not appear frequently in real inorganic materials. The crystal data collected by Nowacki (1967) on 5572 different inorganic materials shows that the number of materials belonging to space groups among sets (2), (3), (5), (7) and (11) in Table 2.5.3.10 is more than 15 but the number belonging to space groups among the other sets is less than ten. This implies that the probability of finding indistinguishable space groups is very low.

Table 2.5.3.11. *Space-group sets distinguishable by coherent CBED*

The space-group pairs in parentheses can not be distinguished by coherent CBED but can be distinguished by a handedness test. An asterisk (*) indicates the incidence at which the distinction is carried out by many-beam interference (Saitoh, Tsuda *et al.*, 2001).

Space-group set	Incidence
(2) $P312$, ($P3_112$, $P3_212$)	$[1\bar{1}01]$
(3) $P321$, ($P3_121$, $P3_221$)	$[11\bar{2}3]$
(5) $P622$, ($P6_222$, $P6_422$)	$[11\bar{2}3]$
(7) $P6_322$, ($P6_122$, $P6_522$)	$[11\bar{2}3]$
(12) $P422$, $P4_222$	[321], [211], [112]*
(13) $P42_12$, $P4_22_12$	[211]
(15) $I422$, $I4_122$	[111]
(16) $I23$, $I2_13$	[111]
(17) $I222$, $I2_12_12_1$	[111]
(18) $P432$, $P4_232$	[321], [211]*
(20) $I432$, $I4_132$	[111]
(21) $F432$, $F4_132$	[432]

2.5.3.3.4. Dynamical extinction in HOLZ reflections

Space-group determination as described in the previous sections is carried out using the extinction lines appearing in ZOLZ reflections. Vertical glide planes whose translation vectors are perpendicular to the specimen surface do not cause extinction lines in ZOLZ reflections but cause them in HOLZ reflections. (It is noted that the vertical glide planes with glide translations not parallel to the surface are not the symmetry elements of diperiodic plane figures.) Vertical glide planes whose translation vectors are parallel to the surface cause extinction lines in both ZOLZ and HOLZ reflections. Vertical screw axes are expected to form extinction lines in HOLZ reflections whose vectors are parallel to the screw axes. These reflections, however, cannot be observed by ordinary CBED. Thus, the extinction lines appearing in observable HOLZ reflections are used to identify not screw axes but glide planes. Examination of HOLZ extinction lines together with ZOLZ extinction lines is an efficient way to characterize the glide vectors and determine the space group.

The dynamical extinction lines appearing in HOLZ reflections caused by the glide planes whose glide vectors are not only parallel but also not parallel to the specimen surface were tabulated by Nagasawa (1983) for various incident-beam orientations of all the space groups that have glide planes. The tabulated results appear on pages 214–225 of the book by Tanaka *et al.* (1988). Table 2.5.3.12 shows the results. The meanings of the letters used in the table are explained in Fig. 2.5.3.13. We consider a vertical glide plane with a glide vector perpendicular to the surface as is shown in Fig. 2.5.3.13(*a*). Letter A is given for cases in which the Ewald sphere intersects two circled-cross reflections in the first Laue zone as seen in Fig. 2.5.3.13(*b*), where black circles and circled crosses denote allowed reflections and kinematically forbidden but dynamically allowed reflections due to the glide plane, respectively. A^* denotes cases in which the Ewald sphere intersects a circled-cross reflection on one side of the incident beam and a black-circled reflection on the other, as seen in Fig. 2.5.3.13(*c*). This case occurs only in space group $P2_1/a\bar{3}$. $A_{\rm h}$ denotes cases in which the Ewald sphere intersects a circled-cross reflection on one side but does not intersect on the other, owing to the asymmetric arrangement of reflections with respect to the incident beam.

The first column of Table 2.5.3.12 list the space groups and the following columns show the type of the extinction lines for possible incident-beam directions. In each pair of columns, the left-hand column gives the reflection indices of the extinction line and the symmetry elements causing the extinction and the right-hand column gives the type of extinction. The first suffix 1, 2 or 3 of a glide symbol distinguishes the first, the second or the third glide plane of a space group. The second suffixes 1 and 2, which appear in the tetragonal and cubic systems, distinguish two equivalent glide planes which lie in the x and y planes. The suffix o of a reflection index implies that the index is odd-order. Figs.

2.5.3.14(*a*) and (*b*) were taken for FeS_2, space group $P2_1/a\bar{3}$, with incident-beam directions of [100] and [110]. Inserts show enlarged HOLZ patterns for ease of viewing. Extinction lines of type *A* are seen in the h_ok0 HOLZ reflections in Fig. 2.5.3.14(*a*) due to the *b*-glide plane (equivalent to the *a*-glide plane in the space-group symbol) parallel to the (001) plane. An extinction line *A** is seen in an h_ok0 HOLZ reflection in Fig. 2.5.3.14(*b*) due to the same glide plane as that of Fig. 2.5.3.14(*a*). It should be noted that extinction lines in HOLZ reflections are better observed in thinner specimen areas than those suitable for the observation of the extinction lines in ZOLZ reflections, because the profiles of HOLZ reflections are concentrated into small areas of CBED discs in thicker specimens.

In summary, the use of not only ZOLZ, but also HOLZ extinction lines is recommended for space-group determination.

2.5.3.3.5. Symmetry elements observed by CBED

In the above sections, point-group and space-group determination methods were described following the theory of Buxton *et al*. (1976). They assumed that the observable symmetry elements are those of an infinitely extended parallel-sided specimen or of diperiodic plane figures. CBED patterns determine diffraction groups. Crystal point groups are identified by consulting Fig. 2.5.3.4, which gives the relations between diffraction groups and crystal point groups. When the assumption made by Buxton *et al*. (1976) is accepted in a strict sense, CBED symmetry m_2 caused by a twofold rotation axis oblique to the specimen surface, which is not a symmetry element of a diperiodic plane figure, ought not to be observed. However, the symmetry m_2 due to a twofold rotation axis in the [$1\bar{1}0$] direction of an Si film with [100] surface normal has been clearly observed at [111] electron incidence (Tanaka *et al*., 1988, p. 33). This indicates that crystal symmetry elements oblique to the specimen surface are observable when the specimen is tilted. An important condition for CBED is that the top and bottom surfaces be parallel over the specimen area illuminated by the incident beam. CBED observes the symmetry elements of a crystal to the extent that the boundary conditions at the specimen surface do not break the symmetries of the CBED patterns. Gjønnes & Gjønnes (1985) reported that the breaking of CBED symmetry due to a surface oblique to the incident beam is practically negligible.

In Section 2.5.3.3 on space-group determination, space-group symmetry elements of crystals which have glide and screw components parallel to the specimen surface were considered to act as space-group symmetry elements of diperiodic plane figures by mitigating the strict application of the assumption of diperiodic plane figures. In fact, vertical glide planes with a glide vector not parallel to the specimen surface, which were dealt in Section 2.5.3.3.4, are not the symmetry elements of diperiodic plane figures. Ishizuka (1982) showed theoretically that a vertical glide plane with a vertical glide vector produces dynamical extinction lines in HOLZ discs if the Laue zones are well separated. Tanaka *et al.* (1988, pp. 214–225) tabulated the extinction lines appearing in HOLZ discs caused by the vertical glide planes whose glide vectors are not only parallel but also not parallel to the specimen surface. Dynamical extinction lines caused by the glide planes with a glide vector not parallel to the surface have been demonstrated using FeS_2 and $MgAl_2O_4$ (Tanaka *et al*., 1988, pp. 51–61).

Vertical 2_1, 3_1, 3_2, $\ldots$, 6_5 screw axes, which are not symmetry elements of diperiodic plane figures, are expected to form dynamical extinction lines in kinematically forbidden reflections that are located in the direction of the screw axes or of the surface normal. The extinction lines, however, are difficult to observe in ordinary CBED. Thus, CBED does not observe all the symmetry elements of the crystal space groups but observes many more symmetry elements than those of the diperiodic plane figures. It is clear now that it makes no sense to construct space groups using actually observable symmetry elements because they do not form a complete set of groups. It is of no importance to give the relation between the 230 space groups of crystals and the 80 space groups of diperiodic plane figures. Buxton *et al.*'s theory, which determines crystal point groups with the help of diperiodic plane figures, is very beautiful and successful. However, it is not correct to state that CBED observes the symmetry elements of the diperiodic plane figures. The use of the groups of diperiodic plane figures should be recognized as a convention for the sake of convenience. As a further example, horizontal screw axes and horizontal glide planes must be located at the middle of a specimen to form symmetry elements of the diperiodic plane figures. However, those screw axes and glide planes which are not located at the middle of a specimen do produce CBED symmetries. Since we now know that CBED does not observe the symmetries of the diperiodic plane figures but observes those of a physical crystalline specimen, we can determine the corresponding infinite crystal symmetries more freely, by using our knowledge of the symmetries of the sample concerned, guided but not restricted by the beautiful theory of Buxton *et al.* (1976).

One point to note, for symmetry determination, is that one has to be aware of spurious symmetries that appear for crystals of certain structure types (Tanaka *et al*., 1988, pp. 20–32 and 42–45) and destroy the correct determination of the point and space groups. Another point for precise symmetry determination is that one has to be aware of how CBED symmetry is destroyed by a small breakdown of crystal symmetry (Tanaka *et al*., 1988 pp. 46–47).

2.5.3.3.6. Examples of space-group determination

A simple example of point-group determination has already been given for Si in Section 2.5.3.2.5. In this section, two examples of space-group determination for rutile and samarium selenide are described, in which the point-group determination still accounts for an important part. The examples look to be a little sophisticated but are a good exercise for those who want to acquire experience in CBED space-group determination. The present determination is carried out by assuming the lattice parameters to be known.

Rutile (TiO_2). The space group of rutile is well known to be $P4_2/mnm$. The lattice parameters are $a = b = 0.459$ nm and $c = 0.296$ nm. Fig. 2.5.3.15(*a*) shows a CBED pattern taken with the [001] incidence at an accelerating voltage of 80 kV. Since no fine HOLZ lines appear in all the discs, projection diffraction groups (column VI of Table 2.5.3.3) have to be applied to explain this pattern. The projection (proj.) WP shows symmetry 4*mm*. The projection diffraction group is found to be $4mm1_R$ from Table 2.5.3.3. Thus, possible diffraction groups are $4m_Rm_R$, 4*mm*, 4_Rmm_R and $4mm1_R$. Another CBED pattern at a second crystal orientation needs to be taken because Fig. 2.5.3.15(*a*) shows only projection symmetry. Figs. 2.5.3.15(*b*) and (*c*) show CBED patterns taken with the [101] incidence at an accelerating voltage of 100 kV. In Fig. 2.5.3.15(*b*), which is the central part of Fig. 2.5.3.15(*c*), no HOLZ lines are seen. The symmetries of the projection BP and projection WP are both 2*mm*. The projection diffraction group of the pattern is $2mm1_R$. The WP of Fig. 2.5.3.15(*c*) is seen to have one mirror symmetry *m*. The diffraction groups which satisfy symmetry *m* are *m*, $m1_R$ and 2_Rmm_R. Among these diffraction groups, the diffraction group whose projection becomes $2mm1_R$ is only diffraction group 2_Rmm_R. By consulting Fig. 2.5.3.4, diffraction group 2_Rmm_R obtained from Figs. 2.5.3.15(*b*) and (*c*) and one diffraction group $4mm1_R$ among diffraction groups $4m_Rm_R$, 4*mm*, 4_Rmm_R and $4mm1_R$ obtained from Fig. 2.5.3.15(*a*) commonly satisfy point group 4/*mmm*. Thus, the point group of rutile is determined to be 4/*mmm*.

Fig. 2.5.3.15(*d*) shows an ordinary diffraction pattern taken with the [001] incidence at an accelerating voltage of 80 kV. With the help of the lattice parameters and the camera length, the indices of the reflections are given as shown in the figure. There

references