Orientation of Calcium Molybdate (CaMoO₄) and Other Single Crystals Having the Scheelite Structure*

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Values of interplanar angles useful in distinguishing the [100] from the [110] direction in CaMoO₄ are given to facilitate orientation by analysis of back-reflection Laue patterns. The distinction between the [001] and [00$\bar{1}$] directions in the scheelite structure is discussed and examples of physical properties dependent upon this distinction are given. The [001] direction is defined in terms of the crystal structure and a procedure for identifying [001] using Laue patterns is described. The same distinction applies to certain nonpolar directions of all crystals belonging to the following nonenantiomorphous point groups: $\bar{1}$, m, 2/m, mmm, $\bar{3}$, 3m, $\bar{3}$m, $\bar{4}$, 4/m, 4/mmm, $\bar{6}$, 6/m, 6/mmm, m3 and m3m.

Key Words: Calcium molybdate, crystal orientation, crystallographic groups, interplanar angles, physical properties, scheelite structure, single crystal, x-ray back-reflection.

1. Introduction

A large number of phases having the scheelite structure are known [1].[1] The ability of these crystals to accept trivalent ions into solid solution and their optical suitability as laser ion hosts [2] has stimulated recent interest in this family of crystals. The growth of scheelite itself, CaWO₄, has been studied [3, 4, 5] and an etch pitting technique for the observation of dislocations [6, 7] has been developed. Substitution of trivalent rare-earth ions has been studied [8, 9] as has the effect of vacuum reduction and photolytic coloring of doped crystals [10, 11]. Work has been published on a number of physical properties including electron spin resonance [12, 13], plastic deformation [14], and sound velocity [15]; it appears likely that more physical properties data on scheelite structure crystals will soon be published. It is important that these data be referred to the proper crystal axes; the correct and complete crystal orientation requires that some fine distinctions be made. The structure of CaWO₄ belonging to space group I4₁/a–C₄ₕ⁶ with Laue symmetry 4/m has been carefully refined both by x-ray diffraction [16, 17] and by neutron diffraction [18]; Zalkin and Templeton [16] point out that there are two distinct ways in which the axes may be oriented with respect to the structure and that the distinction depends upon differences in the intensity of symmetry unrelated pairs of diffracted beams of the distinct types (hkl) and (khl). The metal atoms in special positions, as will be elaborated in section 4 below, do not contribute to these differences which are for many reflection pairs quite small. Previous authors, however, do not discuss the specific process

of orientation by Laue pattern analysis. We shall show that this distinction can be made from visual inspection of a back-reflection Laue pattern provided it is taken on a surface parallel to (001). Some interplanar angles useful in Laue pattern analysis have been given by Nassau [19] and by Frounfelker and Hirthe [20], but the distinction between [001] and [00$\bar{1}$] is not discussed. These angles are useful in distinguishing between [100] and [110] but the distinction depends upon small angular differences; additional angles are given in the present paper to make identification more certain. We shall illustrate the orientation procedure with angles and Laue patterns for CaMoO₄. The results should be applicable to any of the scheelite-structure materials. They should also serve as a general illustration of the technique for determining the sense of axes in those crystals where different senses exist and are manifested in a certain cyclic sequence of intensities of diffracted beams.

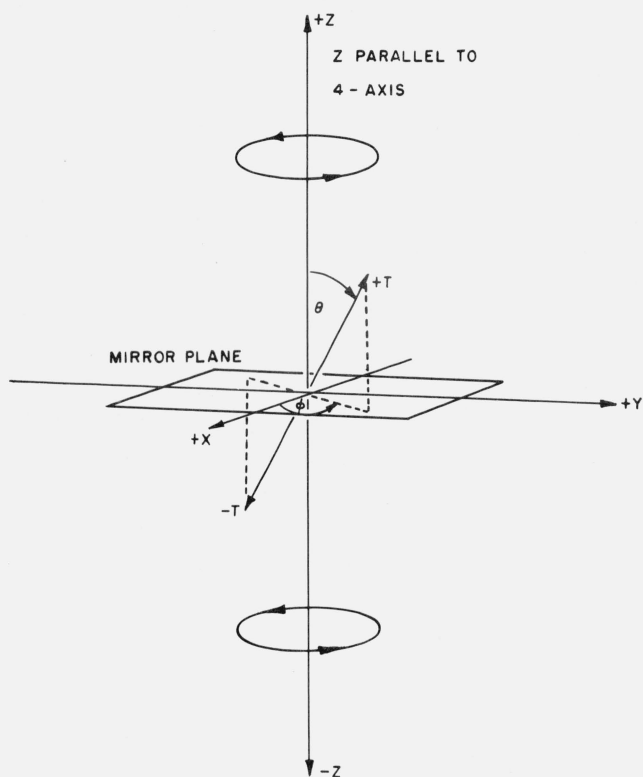## 2. Relation of Axes to Point Group and Space Group Symmetry

The point group of scheelite is 4/m (C₄ₕ) and the space group is I4₁/a. The crystallographic c axis is conventionally [21, 22] taken parallel to the 4-axis, and the Z axis of the set of right-handed rectangular XYZ axes (to which physical properties are usually referred) may be taken in either direction along the 4-axis with different results for the sign of certain physical properties. On first thought this statement may appear incorrect; if +Z is arbitrarily chosen parallel to the 4-axis, the perpendicular mirror plane makes −Z equivalent. Clearly the Z axis is not of the type variously termed vector, polar, or uniterminal by dif-

[1] Italicized figures in brackets indicate the literature references at the end of this paper.

+Z

Z PARALLEL TO
4 - AXIS

+T

θ

MIRROR PLANE

+Y

+X

φ

-T

-Z

FIGURE 1. *Distinction between +Z and −Z in point group 4/m.*

TABLE 1. *Angles between planes in crystals of* CaMoO₄ c/a = 2.187

{$h_1k_1l_1$}	{$h_2k_2l_2$}	Values of angles between {$h_1k_1l_1$} and {$h_2k_2l_2$}
001	011	65.4°
	023	55.5°
	012	47.6°
	013	36.1°
	014	28.6°
	015	23.1°
	016	19.5°
	017	16.9°
001	111	72.1°
	223	64.1°
	112	57.1°
	113	45.9°
	114	37.7°
	115	31.4°
	116	26.9°
	117	23.6°
	118	20.6°
	1·1·10	16.5°
010	012	42.4°
	023	34.4°
	034	31.4°
	011	24.5°
	043	18.9°
	032	16.9°
	021	12.8°
	031	8.6°
	041	6.5°
110	112	32.9°
	223	25.9°
	334	23.3°
	111	17.9°
	443	13.6°
	332	12.1°
	221	9.1°
	331	6.1°
	441	4.6°
100	410	14.0
	310	18.4
	210	26.5
	110	45.0

ferent writers [23, 24, 25]. The +Z and −Z axes are equivalent in the sense that they are related by one or more symmetry operations of the crystal; they are, nevertheless, distinguishable as shown in figure 1. Consider any feature of the crystal (such as a particular sequence of atoms) which has a clockwise sense when viewed from the origin looking along +Z. The mirror plane requires the presence of an equivalent sequence around −Z which will have counterclockwise sense when viewed from the origin. The space group of scheelite has a glide plane instead of a mirror plane so that two equivalent sequences would be displaced perpendicular to [001] as well as reflected but the conclusion that the sequences show opposite senses of rotation when viewed from an intermediate point remains valid. To specify an arbitrary direction, T, in the usual spherical polar coordinate system of figure 1 thus requires a range of $0 \leq \theta \leq 180°$ and $0 \leq \Phi < 90°$. If measurements are made from −T to −Z to give values of θ' and Φ' (with Φ' measured in the same sense about −Z as Φ about +Z) the conversion is $\theta = \theta'$, $\Phi = 90° − \Phi'$. The choice of X fixes Y once +Z has been chosen and the stipulation made that XYZ form a right-handed rectangular system; the X axis is taken parallel to either of the two equal dimensions of the smallest unit cell [22].

3. Distinction Between [100] and [110] Using Angles Determined From a Laue Pattern

The usual back-reflection Laue camera [26] using 3 cm specimen-to-film distance gives a pattern corresponding to a spherical segment subtending about 60°. Poles lying outside this segment can be constructed from hyperbolas on the film but the angular error increases so that it is best to base identification on angles between poles corresponding to spots on the film. The center of the sector may lie anywhere in the spherical triangle with corners (001), (100), (110); angles in any of the other octants are the same so we can momentarily disregard the distinction between [001] and [00$\bar{1}$]. Evidently at least one of the poles (001), (102), (100), (113), and (110), must fall within the sector. Angles between poles lying near these reference poles are given in table 1. It should be noted that the distinction between the direction of [100] and [110] depends on differences such as 28.7 deg from (001) to (104) compared to 26.9 deg from (001) to (116) so that some care is needed. These angles are based on $c/a = 2.187$ of Swanson, Gilfrich, and Cook [27] for CaMoO₄; other scheelite structures having different c/a ratios will have somewhat different values. The tables of Frounfelker and Hirthe

[20] give values of angles for several c/a values; these tables can be used as a practical guide in making small corrections to our angular values for the variations in c/a encountered for other scheelite structures. Angles between 100 and $hk0$ are independent of c/a; angles between 100 and hkl are given by arc cot $\sqrt{2}\,(c/a)(h/l)$.

4. Distinction Between [001] and [00$\bar{1}$] Using Intensity of Spots on Laue Pattern

The pair of diffracted beams $\{hkl;\ khl\}$ would have equal intensities were it not for the contribution from the oxygen atoms in general positions. The unequal intensities of such pairs can be used to define a sense of rotation about Z. The result can be derived from the structure factor tables [21] for $I4_1/a$ that the intensity of a diffracted beam, $I(h,\ k,\ l)$, should obey

$$I(h,\ k,\ l) = I(\bar{k},\ h,\ l) = I(\bar{h},\ \bar{k},\ l) = I(k,\ \bar{h},\ l)$$

$$= I(\bar{h},\ \bar{k},\ \bar{l}) = I(k,\ \bar{h},\ \bar{l}) = I(h,\ k,\ \bar{l}) = I(\bar{k},\ h,\ \bar{l}).$$

These are different from the set

$$I(k,\ h,\ l) = I(\bar{h},\ k,\ l) = I(\bar{k},\ \bar{h},\ l) = I(h,\ \bar{k},\ l)$$

$$= I(\bar{k}\ \bar{h},\ \bar{l}) = I(h,\ \bar{k},\ \bar{l}) = I(k,\ h,\ \bar{l}) = I(\bar{h},\ k,\ \bar{l}).$$

The relevant structure-factor equations are quoted in the International Tables [21].

For distinguishing [001] from [00$\bar{1}$] it is required that there is at least one pair of reflections $\{hkl;\ khl\}$ for which $I(hkl)/I(khl)$ differs from one by as large a factor as possible. Intensity ratios differing greatly from one will be associated with reflections for which the metal ions' contribution to the structure factor—equal for hkl and khl—is small. This condition assures the relative independence of intensity from the chemical identity of the ions, as for example a change from W to Mo ions.

Using 20 to 25 kV peak Ag x-radiation with the characteristic radiation repressed, good back-reflection pictures were obtained. Back-reflection pictures of $CaMoO_4$, figure 2, revealed several pairs of planes with intensity differences between hkl and khl easily recognizable. The following four pairs were used in the orientation determination: $\{2,1,11;\ 1,2,11\}$, $\{3,1,18;\ 1,3,18\}$, $\{2,1,13;\ 1,2,13\}$, and $\{3,2,15;\ 2,3,15\}$. Their corresponding intensity ratios, measured on a densitometer comparator, were 5, 0.7, 4, and 0.3.

In back-reflection pictures of $PbMoO_4$, the differences in intensities of hkl and khl were not as easily recognized because of the large contribution to the intensity by the Pb ion. Two sets of planes $\{2,1,11;\ 1,2,11\}$ and $\{2,1,13;\ 1,2,13\}$ could still be recognized as exhibiting intensity differences. The measured intensity ratios were 2 for both sets of planes. Upon examination of the structure factor equations it was found that the metal ion contribution was the smallest relative to the oxygen contribution in these two sets of planes $\{2,1,11;\ 1,2,11\}$ and $\{2,1,13;\ 1,2,13\}$ for both $CaMoO_4$ and $PbMoO_4$.
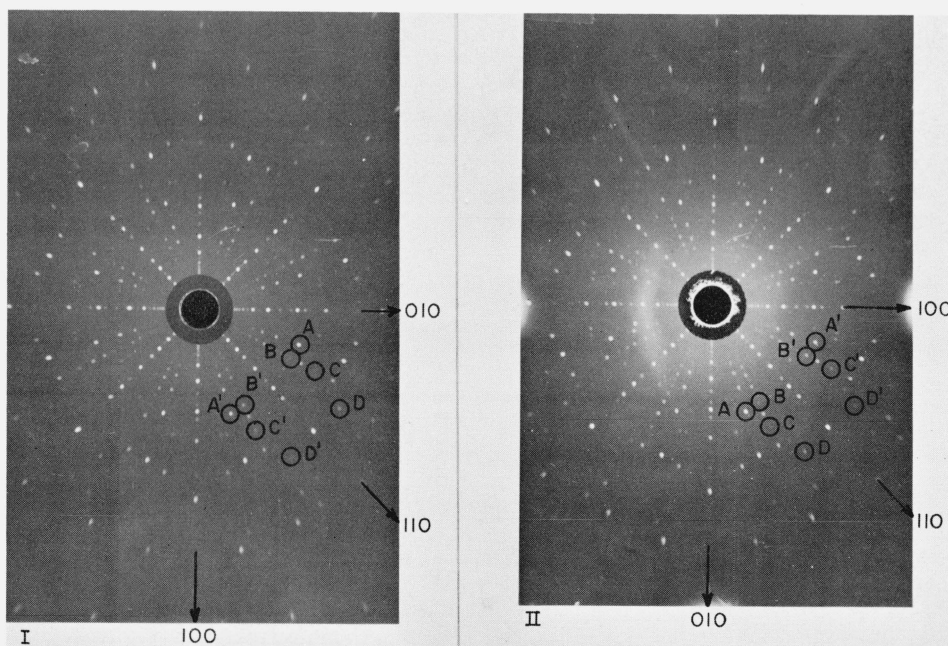

FIGURE 2. *Laue back-reflection pictures of* $CaMoO_4$ *showing the (001) and (00$\bar{1}$) orientations.*

Furthermore, when comparing intensities for pairs of reflections {hkl; khl} for purposes of orientation it is desirable to pick a pair for which the contribution to the intensities from the oxygen in the general position is not a sensitive function of the x, y, and z parameters of the oxygen general position. These oxygen parameters are variable for different members of the crystal-chemical group of scheelite phases. Appreciable variability with solid-solution effects must be expected. Evaluation of the structure factor equations for the different pairs of planes over a range of x, y, and z's indicated that the two pairs of planes {2,1,11; 1,2,11} and {2,1,13; 1,2,13} best satisfied this condition. Thus these two pairs of planes are quite useful in determining the sense of [001] in scheelites, even in the case of the heavy cation Pb in $PbMoO_4$.

To determine the correct setting of the axes with respect to the crystal using the Laue method the ratio of the reflected intensities of each of the previously mentioned 4 pairs of reflecting planes was calculated for both $CaMoO_4$ and $PbMoO_4$.

Using the oxygen parameters specified by Sillén and Nylander [28] for $CaMoO_4$ ($x = 0.25$, $y = 0.15$, $z = 0.075$) the calculated ratios for $CaMoO_4$ were not consistent with the measured ratios in that the calculated ratio $I(3,2,15)/I(2,3,15) \approx 1$. However, when the calculations were done using the data of Zalkin and Templeton [16] for the oxygen parameters ($x = 0.2415$, $y = 0.1504$, $z = 0.0861$) of $CaWO_4$, the calculated intensity ratios were consistent with the measured ones. From the few data points examined here, a structure refinement for $CaMoO_4$ is indicated. Using the $CaWO_4$ oxygen parameters for the $CaMoO_4$ calculations it was found that $I(2,1,11) > I(1,2,11)$; $I(3,1,18) < I(1,3,18)$, $I(2,1,13) > I(1,2,13)$ and $I(3,2,15) < I(2,3,15)$. With this information the axes of $CaMoO_4$ can then be chosen to agree with the setting of Zalkin and Templeton, and to satisfy the above inequalities.

The same procedure was carried out with $PbMoO_4$. Using the data of Leciejewicz's refinement of $PbMoO_4$ [29], the intensity ratios of the two observable pairs of planes were $I(2,1,11) > I(1,2,11)$ and $I(2,1,13) > I(1,2,13)$. Thus these two sets of planes can be used to chose the correct setting of the axes consistent with the intensity ratios and the sense of [001].

Figure 2 shows two Laue patterns of $CaMoO_4$ taken with the [001] and [00$\bar{1}$] orientations respectively. In figure 2(I) (the $+c$ direction out of the plane of the picture) the spots are labeled as follows: $A' = (3,1,18)$, $A = (1,3,18)$, $B' = (2,1,13)$, $B = (1,2,13)$, $C' = (2,1,11)$, $C = (1,2,11)$, $D' = (3,2,15)$ and $D = (2,3,15)$. In figure 2(II) ($-c$ direction out of the plane of the picture) the spots are labeled as follows: $A' = (3,1,\overline{18})$, $A = (1,3,\overline{18})$, $B' = (2,1,\overline{13})$, $B = (1,2,\overline{13})$, $C' = (2,1,\overline{11})$, $C = (1,2,\overline{11})$, $D' = (3,2,\overline{15})$, and $D = (2,3,\overline{15})$. It may be noted here that the differences in the intensities are somewhat diminished in making a print from the x-ray film; the intensity differences are more easily observed on the film itself. One can now choose $+Z$ from simple inspection of these patterns. It is, however, necessary to have a flat ground parallel to (001) within a few degrees and to have the beam incident perpendicular to (001) within a few degrees. Otherwise differences in absorption corrections applicable to the non-equivalent reflections constituting a pair {h, k, l; k, h, l} could distort the intensity relationships to such an extent that the inequalities can no longer be recognized at sight on the Laue pattern. The absorption, Lorentz, and polarization corrections (for crystals intercepting the entire beam) are the same for hkl and khl when the incident x-ray beam is parallel to [001] and the surface is (001). One can first determine the orientation, apart from the distinction between $+Z$ and $-Z$, from a Laue pattern taken at random orientation and then grind a small flat parallel to (001) for a second Laue pattern.

5. Physical Properties Dependent on the Distinction Between $+Z$ and $-Z$

Scalar, first rank, and symmetric second rank tensor properties do not depend on the distinction between $+Z$ and $-Z$ as can be seen from the fact that the matrices for these properties are the same as for the highest symmetry tetragonal point group, 4/mmm, for which the distinction between $+Z$ and $-Z$ vanishes. The center of symmetry possessed by 4/m makes scheelite nonpiezoelectric so that this property need not be considered. The sign of the elastic compliance [30]s_{16} ($s_{16} = s_{61}$) does, however, depend on the choice of $+Z$; the Standards on Piezoelectric Crystals [22] apparently overlooks this fact. A tensile stress along X in a crystal of point group 4/m will cause an elastic deformation in which both X and Y must remain perpendicular to Z, but the angle between X and Y need not remain a right angle. Thus, in general, application of σ_{11} will cause a nonvanishing value of e_{12} and give a nonzero value of $s_{61} = 4s_{1211}$. Suppose the value of s_{61} is positive for our choice of $+Z$ and a particular choice of X. If we rotate our coordinate system 90° about Z so that $X' = Y$ and $Y' = -X$ we have a tensile stress $\sigma'_{22} = \sigma_{11}$ and a shear strain $e'_{12} = -e_{12}$ giving an elastic compliance $s'_{62} = 4s'_{1222} = -s_{61}$. This is consistent with the requirement for crystals of point group 4/m that $s_{62} = -s_{61}$. Thus no change in the numerical values of the elastic constants results from a rotation of the coordinate system 90° about Z. If, however, we reverse the direction of Z and choose a right-handed system such that $Z'' = -Z$, $X'' = X$, $Y'' = -Y$ the result is $\sigma''_{11} = \sigma_{11}$, $e''_{12} = -e_{12}$ and $s''_{61} = 4s''_{1222} = -s_{61}$ so that the sign of s_{61} is changed by interchanging $+Z$ and $-Z$ and retaining a right-handed system.

A number of tensor properties are listed by Nye [30] (see pp. 290–291) and some appear to be dependent on the choice of $+Z$. Thus it appears that some elements in the matrix of the piezo-optical tensor will depend on this choice. The situation should be considered carefully for any property more complex than a symmetrical second rank tensor and the decision made as to whether the additional trouble of determining $+Z$ is required for correct determination of all matrix elements.

6. General Occurrence of Symmetry Related Directions Which Are Distinguishable

The effect here discussed is clearly not limited to crystals having the scheelite structure. It does not depend, for example, on the existence of a screw axis 4_1 or 4_3 parallel to z, but would apply to crystals with structures belonging to any space group in the $4/m$ class. It is interesting to inquire which directions in which point groups have the property of being symmetry related but distinguishable by the right or left handedness of their surroundings when viewed from a common origin. We shall term these enantiomorphous nonpolar directions keeping in mind that the concept of an enantiomorphous crystal (one whose mirror image cannot be brought into coincidence with the crystal itself by any sequence of proper rotations) is different. A nonpolar direction cannot have this enantiomorphous character if it lies in a mirror plane or if it is perpendicular to an evenfold axis. Other symmetry elements occurring in crystallographic point groups do not destroy this enantiomorphous character. Thus, for example, a general direction in any centrosymmetric point group is an enantiomorphous nonpolar direction. The general direction $[u\,v\,w]$ in m3m is equivalent to $[\bar{u}\,\bar{v}\,\bar{w}]$ in the sense that the two are symmetry related but they are nevertheless distinguishable by handedness of environment; we would require a left-handed coordinate system to write $[\bar{u}\,\bar{v}\,\bar{w}]$ for $[u\,v\,w]$. In noncentrosymmetric point groups a general direction is always polar. The special directions for both centrosymmetric and noncentrosymmetric classes require detailed examination; the results are summarized for centrosymmetric classes in table 2. All enantiomorphous nonpolar directions in centrosymmetric classes except general directions are found to be parallel to symmetry axes of order two or greater. Only four of the noncentrosymmetric point groups are found to contain enantiomorphous nonpolar directions as listed in table 3.

TABLE 3. *The enantiomorphous nonpolar directions in noncentrosymmetric crystals*

Point group	Nonpolar directions with $[uvw]$ distinguishable from $[u\,\bar{v}\,w]$
m	[010]
3m	[100]; [010]; [110]
$\bar{4}$	[001]
6	[001]

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7. References

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TABLE 2. *The enantiomorphous nonpolar directions, other than the general direction, in centrosymmetric crystals*

Centro-symmetric class	$[uvw]$ Indistinguishable opposites	$[uvw]$ Distinguishable opposites parallel to axial symmetry (order ≥ 2)
$\bar{1}$	None	None
2/m	$v=0$	[010]
mmm	u, v or $w=0$	None
$\bar{3}$	None	[001]
3m	$v=2u; v=\frac{1}{2}u; v=-u$	[100]; [010]; [110]
4/m	$w=0$	[001]
4/mmm	$u, v,$ or $w=0, u=\pm v$	None
6/m	$w=0$	[001]
6/mmm	$u, v,$ or $w=0, u=\pm v, 2v,$ or $\frac{1}{2}v$	None
m3	$u, v,$ or $w=0$	$[\pm 1 \pm 11]$
m3m	$u, v,$ or $w=0, u=\pm v; v=\pm w; w=\pm u$	None

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