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NASA CR-75

NASA CR-75

N64-30992
(ACCESSION NUMBER)
295
(PAGES)
(NASA CR OR TMX OR AD NUMBER)
(THRU)
1
(CODE)
25
(CATEGORY)
FACILITY FORM 302

LASER MATERIALS: A TUTORIAL REVIEW WITH BIBLIOGRAPHY

by James E. Bradley

Prepared under Grant No. NsG-74-60 *by*
OHIO STATE UNIVERSITY
Columbus, Ohio
for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • OCTOBER 1964

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For sale by the Office of Technical Services, Department of Commerce,
Washington, D.C. 20230 -- Price $4.00

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I. INTRODUCTION

This paper has a twofold purpose: (1) to discuss basically the nature, properties, and production of laser materials, and (2) to present an extensive bibliography of the literature that contains additional information on possible laser material and their properties.

So far, laser action has been reported in gases, crystalline solids, and glasses. This leaves only the liquid state as nonproductive. The close similarities between the "structure" of liquids and glasses would lead one to believe that laser action in some liquids might also be possible. (In the last year, while this paper was in preparation, worker at Hughes observed laser action in seven organic liquids.) In this paper, we will discuss only the crystalline and glassy states. The approach will be from the point of view of material science, the material being the important thing, not the possible laser action.

1.1 Requirements of a Laser Material

A laser material consists of a laser ion contained in a host lattice (crystal) or carrier material (glass).

There are requirements for both the ion and its carrier. The requirements for the ion are that it has incomplete shells, (a transition element), and have the proper energy levels, relaxation time, and narrow line width. These requirements are met, for example, by the rare-earth elements.

The requirements for a crystal lattice are that it be able to accept the desired ion and that it have good transmittance in that part of the spectra where the laser is to work. It must be possible to grow single crystals that are large enough to use and are of good quality. The crystal must be chemically and thermally stable. Also, the crystal should ideally be isotropic, or if anisotropic, uniaxial.

The requirements for a glass laser are not well known, but it must have good transmittance in that part of the spectra where it is to operate.

1.2 Organization of This Paper

This paper has five tutorial review sections in addition to the introduction. In the first four sections, matters basic to crystalline or glass laser materials will be discussed. The fifth section deals with the nature of laser ions, in particular, rare earths. All these will be considered in a basic way, but the bibliography presents sources where details on any of the subjects covered, as well as others, can be found.

Crystallography is discussed in Section II. This is done because of the effect that the internal arrangement of crystals has on laser action, and also the effects produced on the laser ion by its crystal field. In Section III, crystal chemistry will be discussed; this is presented because it explains into which structures one might be able to "dope" a desired ion.

Section IV deals with methods of crystal growth and the types of crystals they may produce. In Section V the structure, chemistry, and spectral properties of glass are discussed in order to provide a general background in glass, which may become an important laser material. Finally Section VI is concerned with the general nature of the rare earths and actinide ions, because of their interesting spectral character.

The bibliography is divided into two parts. Part A contains some of the better or more recent reference books, review articles, and bibliographies, on some of the subjects related to laser materials. Also listed are abstracts and related materials where additional information on these and other subjects may be found. It is not the intention of this portion of the bibliography to give a complete list of references on the subjects covered.

Part B is a partially annotated bibliography of specific references on a group of subjects related to lasers and laser materials. The contents, sources of references, and degree of coverage of each section are given at its beginning. For the most part the abbreviations used for journals are those adopted by Chemical Abstracts.

Though every attempt was made to remove all mistakes from the bibliography, this was almost an impossible task for one person on a compliation of more than 2,000 references. Any mistakes are those of the author, and for these he apologizes.

II. CRYSTALLOGRAPHY

Crystallography is the study of the external geometry and internal structure of crystalline solids. We are interested in crystallography because of the effect that internal structure has on the laser properties of a crystal. The information that we want from crystallography is thus, the nature of the internal arrangement. The information that is usually presented in the literature is based on a system of crystallographic notation; it is this system of notation that we will discuss in this chapter. The sources of crystallographic information are given in the bibilography.

2.1 Crystal System Notation

In order to study the internal structure of crystals a brief look will be taken at the external development of faces and the classification of crystals. To define the position of faces occurring, on crystals, straight lines are assumed to pass through the ideal center of each crystal. These intersecting lines are the crystallographic axes, and are used to divide all crystalline matter, on the basis of symmetry, into six, or according to some, seven groups called crystal systems. The axes also serve the purpose of coordinates when geometrical drawings of crystals are made. All crystals except those of the hexagonal system are referred to three axes, and in the hexagonal four axes are used. If the lengths of the axes needed to describe the crystal are the same, each axis is referred to by the letter a. If the axes are not alike, the one extending from front to rear is designated the a axis, the axis from right to left, the b axis and the vertical one is termed the c axis. The opposite ends of the axes are made positive and negative as shown in Figure 2.1a.

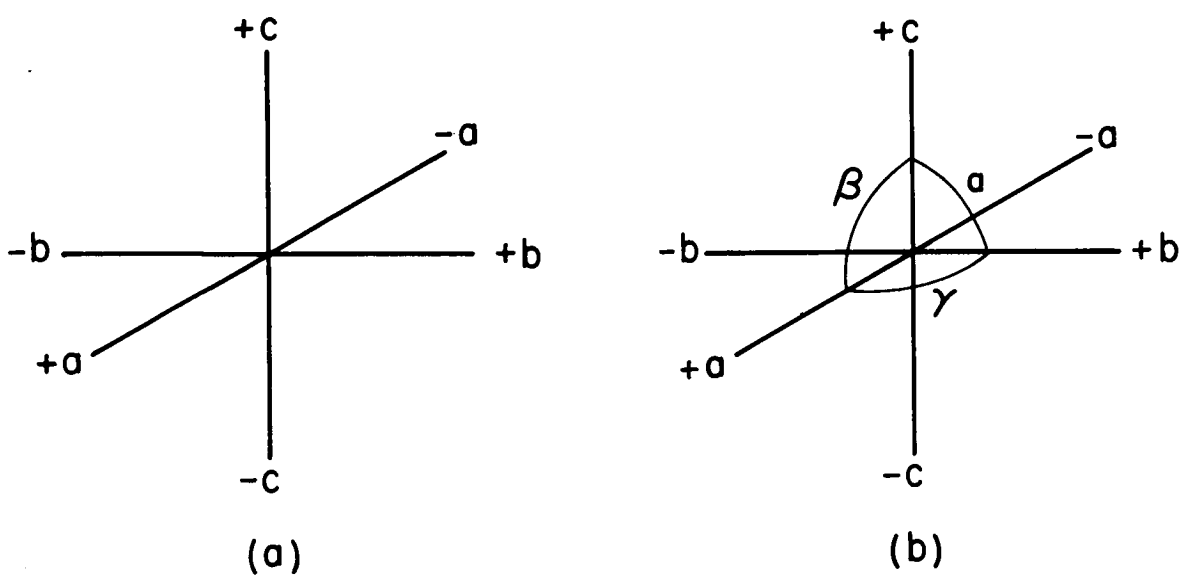


Fig. 2.1. The axial cross

Also used in division into systems are the angles of intersection of the axes. The angles between the various axes are designated as shown in Figure 2.1 b. The systems may be classified by means of the axes and angles as follows:

Cubic or Isometric System

Three identical axes intersecting at right angles. Axes are designated a_1, a_2, a_3 (See Figure 2.2 a).

Hexagonal System

Four axes, three of which are identical and lie in the same horizontal plane, and intersect each other at 120°. The fourth axes is perpendicular to the plane of the other three and is either longer or shorter. The three horizontal axes are designated a_1, a_2, a_3, and the other is designated c (Figure 2.2 c). Some subdivide the hexagonal into hexagonal and trigonal or rhombohedral.

Tetragonal System

Three axes, two of which are identical and in horizontal plane and at right angles to each other. The third is vertical and at right angles to the others. Horizontal axes are a_1 and a_2, and the vertical is c. (Figure 2.2 b)

Orthorhombic System

Three unequal axes intersecting at right angles. The axes are designated as shown in Figure 2.2 d.

Monoclinic System

Three unequal axes, two of which (a,c) intersect at an oblique angle β, the third axis is perpendicular to the other two.

Triclinic System

Three unequal axes intersecting at three angles other than 90°. It is thus possible to place all crystalline materials into one of these divisions or systems.

2.2 Weiss Parameters and Miller Indices

Crystal faces are defined by rational multiples of their intercepts on the crystallographic axes. A face may intercept all the axes, intercept two and be parallel to the third or intercept one and be parallel to the others. The relative distance of intersection in terms of the lattice constants (i.e. the characteristic length a, b, c along the crystal axes) on each axis are the Weiss parameters of the face.

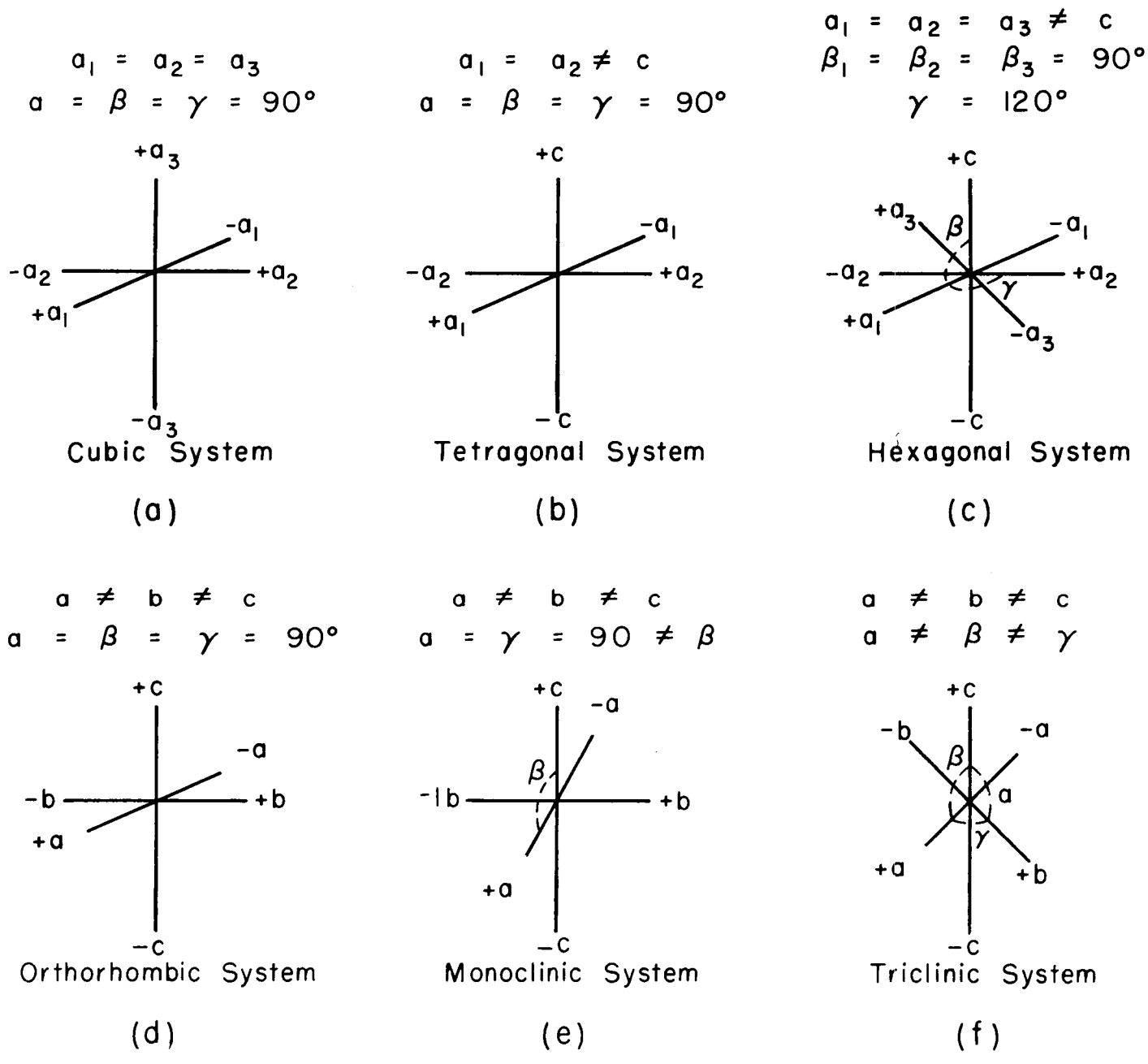


Fig. 2.2 The six crystal systems

The reciprocals of the intercepts are the Miller indices, the most universally employed method of describing planes or faces of crystals. The Miller indices are cleared of fractions and written in order hkl (these being the intercepts on the a, b, and c axes) without ratio sign, even though both the Weiss parameters and Miller indices are ratios.

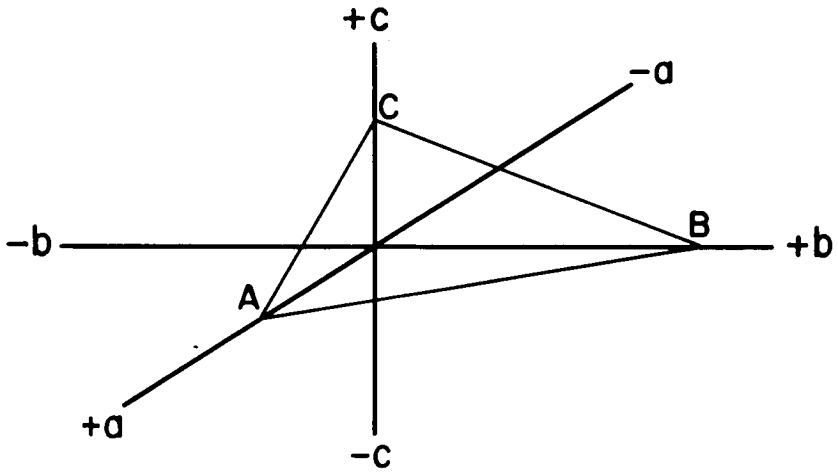
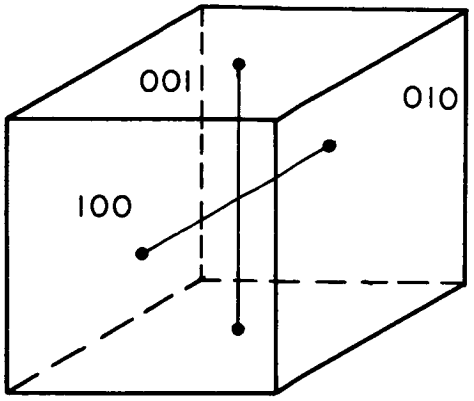


Fig. 2.3. Axial intercepts

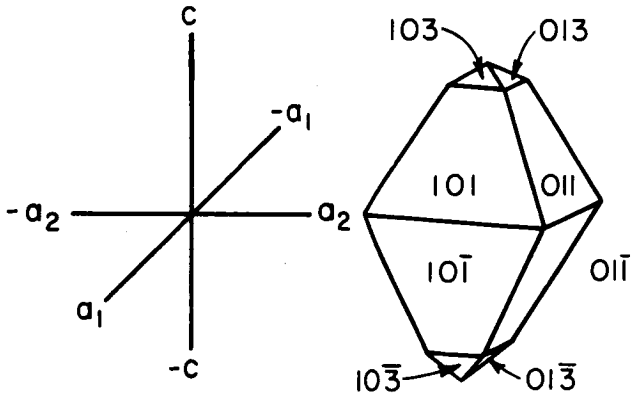
For example in Figure 2.3 the plane ABC (crystal plane or face hkl) has the intercepts 1 on the a, 3 on the b, 1 on the c, the Miller indices are $\frac{1}{1}$: $\frac{1}{3}$: $\frac{1}{1}$ or clearing 313. If the plane had intersected the negative ends of the axes the indices would have been written $\bar{3}\bar{1}\bar{3}$.

The Miller indices are usually small numbers or if the faces are parallel to the axes the intercept is ∞, the Miller index is zero, as in 001 where the plane is parallel to a and b and intercepts only c.

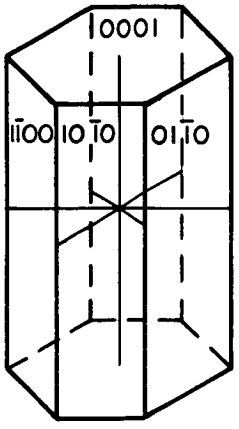
Well-developed faces are commonly parallel to planes of the highest point density (planes that contain the greatest number of lattice points) in the structure these planes also usually have simple indices. Miller indices are thus the reciprocals of lattice parameters cleared of fractions. Figure 2.4 show some crystals and the Miller indices for the faces.



Cubic



Tetragonal



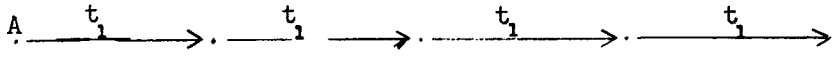
Hexagonal

Fig. 2.4 Crystals and crystal faces

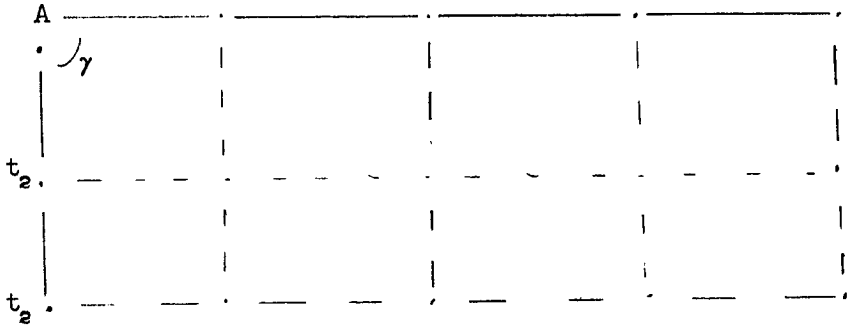
2.3 The Lattice

A crystalline substance is made up of atoms and ions arranged in some orderly manner. The basis of the order is repetition of the atoms or ions into a three dimensional periodic arrangement. This periodic arrangement can be described by means of a basis and a lattice as follows: A lattice is by definition a parallel net-like arrangement of points in which the environment about any point is the same in every way as that about all other points. If we consider the basis as the smallest collection or unit of atoms in crystals having the same arrangement orientation and composition repeated throughout the crystals, it is then possible to form a crystal structure by associating with each lattice point a basis. This is illustrated in Figure 2.5.

We will now consider the nature and the development of different lattices. If we first consider one dimensional repetition and to point A apply successive



translations t_1 we generate a row of points. If we now move to two dimension and add an additional translation t_2 we get a planar net built up.



If we consider the two translations t_1 and t_2 and the included angle γ, we find that there are only five possible planar nets, consistent with crystal structures, and these are shown in Figure 2.6.

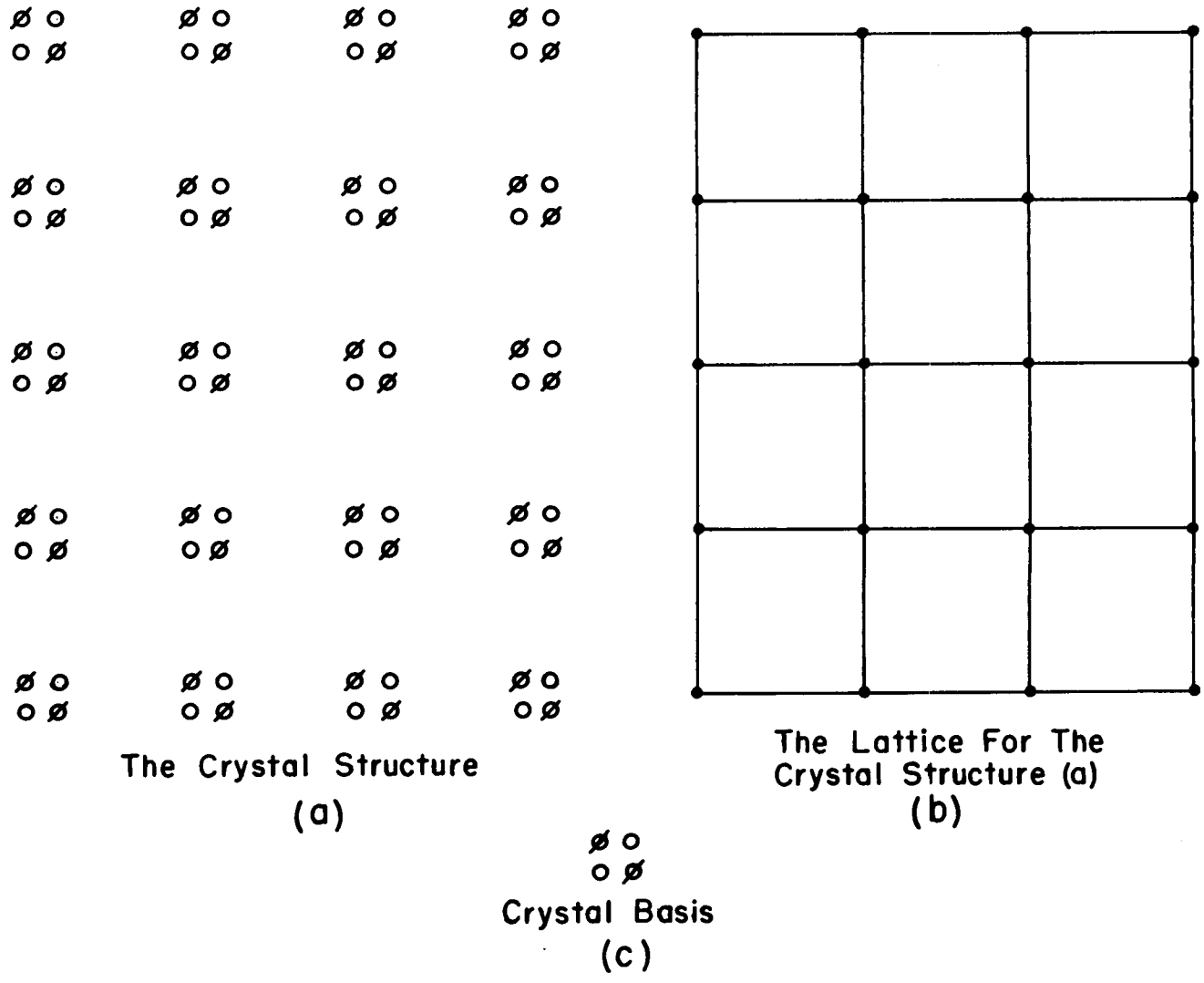


Fig. 2.5 Crystal structure, its lattice, and its basis

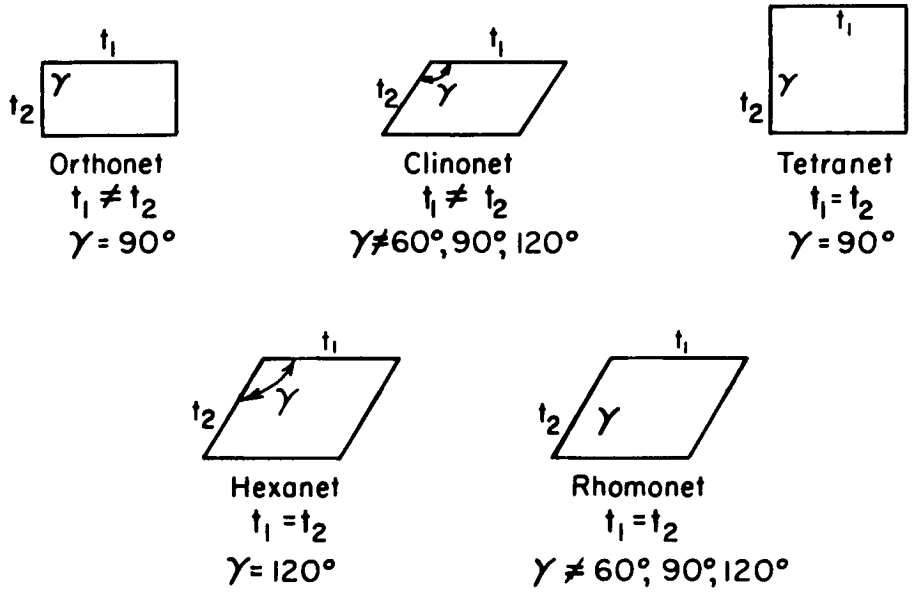


Fig. 2.6 Possible planar nets

The addition of a third non-coplanar translation t_3 generates a three-dimensional array of points referred to as the space lattice or just the lattice. Bravais in 1850 showed that there are only 14 ways of filling space with points, thus the space lattices are sometimes referred to as Bravais lattices; these are shown in Figure 2.7.

The space lattice is built up of three-dimensional units whose sides are t_1, t_2, and t_3. This unit or parallelepiped is repeated throughout space (the crystal), with the environment about every point being identical with that about all other points. The simplest parallelepiped of a lattice with the edges parallel to the crystal axes is called the "unit cell." If lattice points are only at the corners of this cell, it is called primitive, and contains only one lattice point. If, however, the unit cell has additional points, not at the corners, it is a multiple cell. There are five different types of cells that are chosen in crystallography. They are as follows:

Primitive cell. Points only at the corners of cells and designated by P. (1 point per cell).

Body centered cell. Points at corners plus a point at cell center designated by I. (2 points per cell).

Fully faced centered cell. Points at corners plus points at centers of all faces designated by F. (4 points per cell).

Face centered or C centered cell. Points at corners plus points in center of opposite faces designated by C. (2 points per cell).

Rhombohedral cell. Points only at corners of rhombohedral cell designated by R. (1 point per cell). See Figure 2.7.

The 14 space lattices and their distribution among the six-crystal system are shown in Figure 2.7.

2.4 Symmetry Elements in Crystals

Symmetry is a correspondence of parts. Crystals have both external and internal symmetry, the external symmetry being a result of the internal symmetry. When a pattern (internal and external) is repeated, some basic geometrical element that produces the repetition is required. The elements can be considered as a few simple geometrical operations and are called the elements of symmetry. The elements of symmetry are translation, rotation, reflection, and inversion. The operators that produce these are the rotation axes, plane of symmetry, and the inversion center. The symmetry operations are shown in Figure 2.8.

Plane of Symmetry. A plane of symmetry is said to exist when for each point (interior and exterior), face, edge, or solid angle on one side of the plane there exists a like element on the opposite side of the plane. (The symbol m is used for a plane.)

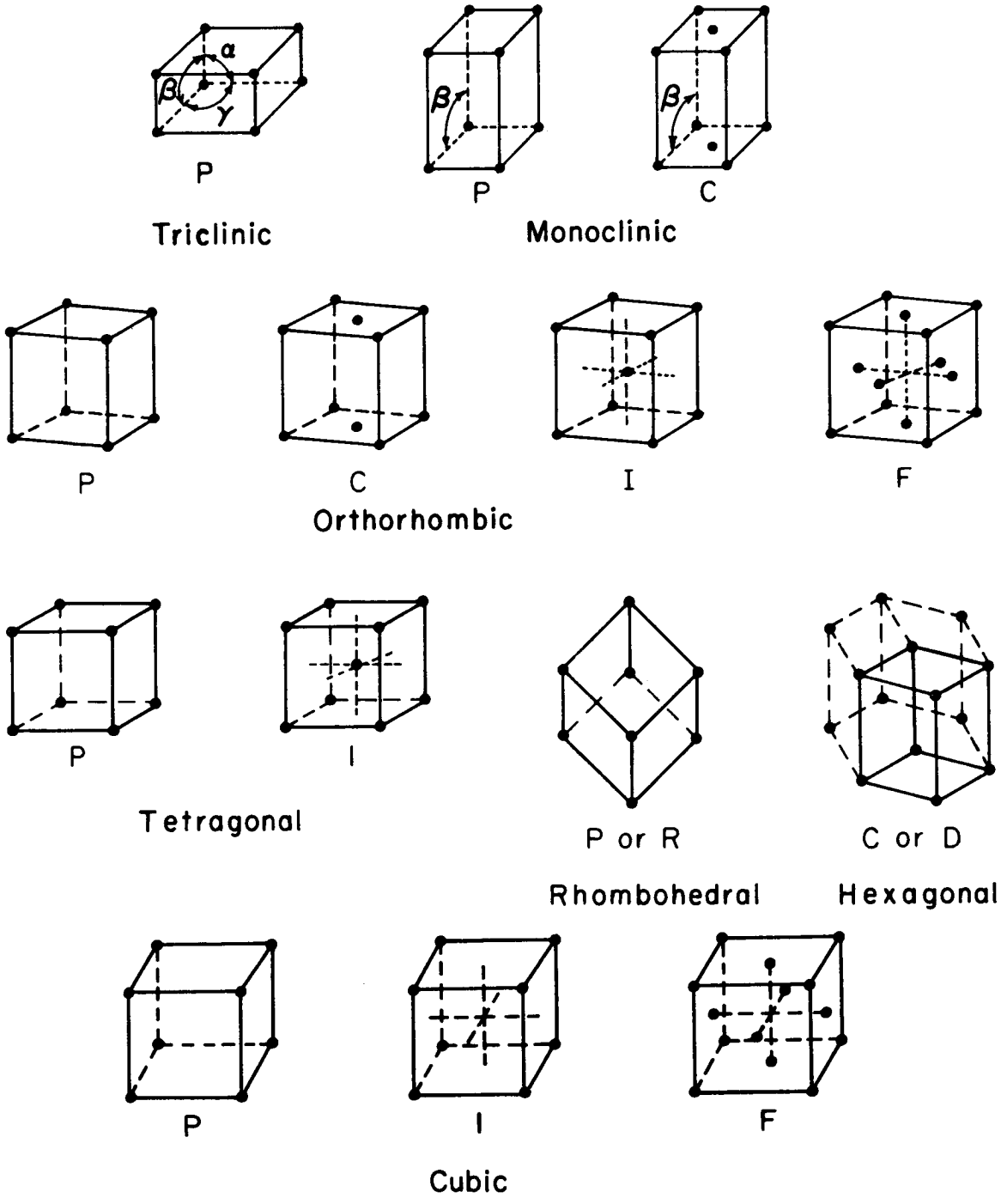


Fig. 2.7 Bravais space lattices

Axis of Symmetry. If a solid can be revolved through a certain number of degrees, about a line as an axis, with the result that it again repeats its position in space, that axis is an axis of symmetry. The axis is defined according to the number of times it repeats a face in a rotation of 360°. For example, a four-fold axis means that the solid is symmetrical with respect to rotations of 90° about the axis. Buerger (1956, pp. 31-34) shows that in crystallographic considerations only 1, 2, 3, 4, and 6 fold axes are possible.

Inversion center. A crystal has a center of symmetry if an imaginary line passed from a point on its surface through the center emerges at a similar point, at equal distance from the center, on the opposite side. (A combination of rotation and inversion, the axis of rotary inversion is given the usual fold number with a bar such as $\bar{1}$, $\bar{2}$, $\bar{3}$, $\bar{4}$, and $\bar{6}$).

Translation repeats objects, atoms or ions, moved equal distances in some fixed direction and is applied only to internal symmetry.

Symmetry and symmetry notation will now be discussed with respect to the concepts of point groups and space group.

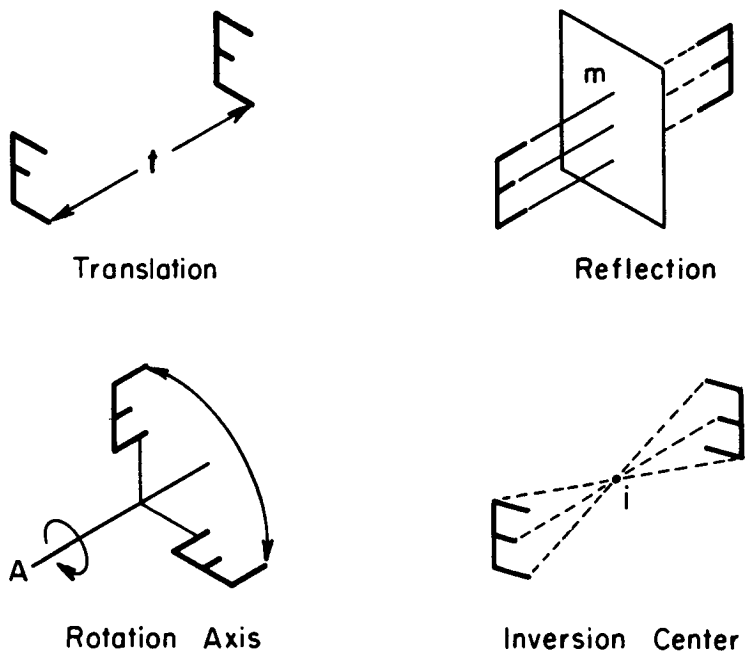


Fig. 2.8. Symmetry operations

2.5 Point Group Symmetry of Crystals

If you consider all the non-redundant non-translational symmetry elements that a crystal has, this set of symmetry elements can be used to describe the symmetry of the crystal. It can be shown that there are only 32 possible combinations of the various symmetry elements (see Buerger, 1956, Chap. 6) giving rise to the 32 point groups or crystal classes which are shown in Table 2.1. Notice that the symmetry elements, or geometrical operators, of a crystal class, or point group, are always regarded as passing through a single point, which is the origin of the crystallographic axes. This point is thus not repeated by the symmetry operations. This leaving of one point unmoved excludes the presence of translational operations. It has already been mentioned that we are limited to axes of one, two, three, four, and six fold rotation and inversion. These alone or in combination with mirror planes give rise to the 32 possible crystallographic point groups, or those groups of symmetry operators which can operate on an infinite three-dimensional lattice so as to leave one point unmoved. Therefore, the point symmetry of any particular crystal can be classified by specifying the point group to which it belongs. Since the crystals are also classified by the six (or seven) crystal systems, it is evident that more than one point group belongs to the same crystal system. This is illustrated in Table 2.1. In Table 2.1 it can be seen that the symmetry of the groups increases from top to bottom and that within a group the bottom-most member has the full symmetry possible for that crystal system.

The following paragraph describes the characteristics of several point groups:

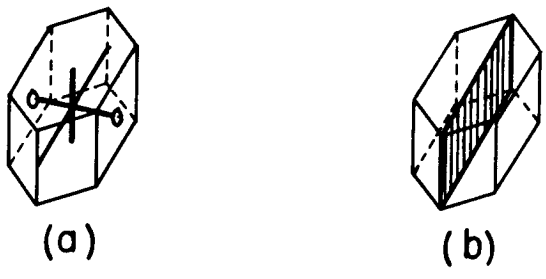


Fig. 2.9. Symmetry of the monoclinic system

Table 2.1 Point Groups and Point Groups Symmetry and Notation

Crystal System	Crystal Class	Full Symmetry	Hermann-Mauguin Symbols	SCH Symbols
Triclinic	Pedial	None	1	C_1
	* Pinacoidal	C	$\bar{1}$	C_i
Monoclinic	Domatic	1P	m	C_s
	Sphenoidal	$1A_2$	2	C_2
	* Prismatic	C, $1A_2$, 1P	2/m	C_{2h}
Orthorhombic	*Rhombic - pyramidal	$1A_2$, 2P	mm2	C_{2v}
	Rhombic - disphenoidal	$3A_2$	222	D_2
	*Rhombic - dipyramidal	C, $3A_2$, 3P	2/m 2/m 2/m	D_{2h}
Tetragonal	Tetragonal - disphenoidal		$\bar{4}$	S_4
	Tetragonal - pyramidal	$1A_4$	4	C_4
	Tetragonal - dipyramidal	C, $1A_4$, 1P	4/m	C_{4h}
	*Tetragonal - scalenohedral	$3A_2$, 2P	$\bar{4}$ 2m	D_{2d}
	Ditetragonal - pyramidal	$1A_4$, 4P	4mm	C_{4v}
	Tetragonal - trapezohedeal	$1A_4$, $4A_2$	422	D_4
	*Ditetragonal - dipyramidal	C, $1A_4$, $4A_2$, 5P	4/m 2/m 2/m	D_{4h}
Hexagonal (Rhombohedral) (Trigonal	Trigonal - pyramidal	$1A_3$	3	C_3
	Rhombohedral	C, $1A_3$	$\bar{3}$	C_{3i}
	*Ditrigonal - pyramidal	$1A_3$, 3P	3m	C_{3v}
	*Trigonal - trapezohedral	$1A_3$, $3A_2$	32	D_3
	*Hexagonal - scalenohedral	C, $1A_3$, $3A_2$, 3P	$\bar{3}$ 2/m	D_{3d}
Hexagonal (Hexagonal)	Trigonal - dipyramidal	$1A_3$, 1P	$\bar{6}$	C_{3h}
	Hexagonal - pyramidal	$1A_6$	6	C_6
	*Hexagonal - dipyramidal	c, $1A_6$, 1P	6/m	C_{6h}
	Ditrigonal - dipyramidal	$1A_3$, $3A_2$, 4P	$\bar{6}$ m_2	D_{3h}
	*Dihexagonal - pyramidal	$1A_6$, 6P	6 mm	C_{6v}
	Hexagonal - trapezoheoral	$1A_6$, $6A_2$	622	D_6
	*Dihexagonal - dipyramidal	C, $1A_6$, $6A_2$, 7P	6/m 2/m 2/m	D_{6h}
Isometric	Tetartoidal	$3A_2$, $4A_3$	23	T
	*Diploidal	C, $3A_2$, $4A_3$, 3P	2/m $\bar{3}$	Th
	*Hexterahedral	$3A_2$, $4A_3$, 6P	$\bar{4}$ 3m	Td
	Gyroidal	$3A_4$, $4A_3$, $6A_2$	432	O
	*Hexoctahedral	C, $3A_4$, $4A_3$, $6A_2$, 9P	4/m $\bar{3}$ 2/m	O_h

* Most common

C = center
A = axis
P = plane

Figure 2.9 shows the symmetry elements of the monoclinic systems; Figure 2.9 (a) shows the single two fold axis of the sphenoidal class, and Figure 2.9 (b) the lone plane of the domatic class. If we were to imagine both of these symmetry elements as occurring in the same crystal, we would have the symmetry of the prismatic class, noting that a center is introduced by the combination of a two fold axis at right angles to a symmetry plane.

2.6 Point Group Notation

By international agreement, crystallographers have adopted the Hermann-Manguin system of point group notation, but the Schoenflies notation is used in spectroscopy and other branches of science so both are presented in Table 1. The derivations of these are given by Buerger (1956, Chap. 6) and so this will not be undertaken here, instead only the method of notation will be considered.

Schoenflies notation

Cn (Cyclic)	An n fold rotation axis
Dn (Dihedral)	An n fold rotation axis and n two-fold axes ⊥ to it
Sn	An n fold rotation-reflection axis
T (Tetrahedral)	Four 3 fold axes directed toward the vertices of a tetrahedron
O (Octahedral)	Three mutually ⊥ 4 fold axes

Subscripts

v	Vertical reflection plane containing the symmetry axis.
h	Horizontal reflection plane ⊥ to the axis.
i	Inversion
s	Reflection plane
d	Dihedral reflection plane bisecting the angle between the two fold axes.

Thus, the notation C_{2h} indicates a crystal class that has as its symmetry a two-fold rotation axis ⊥ to a plane of symmetry. This is the symmetry produced by combining Figure 2.9a and 2.9b.

Herman-Manguin notation

1, 2, 3, 4, and 6	Various axes of rotation
$\bar{1}$, $\bar{3}$, $\bar{4}$, and $\bar{6}$	Various axes of rotary inversion
m	Plane of symmetry
2/m	Combination of m and 2 (2 fold axis ⊥ to a mirror plane)

The number and order of the notation varies from system to system as follows:

Isometric or Cubic system usually has three parts. The first part refers to the axial directions and indicates whether the a axes are twofold or fourfold. The second part refers to the three-fold axis and tells if it is 3 or $\bar{3}$. The third part, if present, refers to the diagonal planes and two-fold axes.

Hexagonal. The first symbol refers to the c or principle axis and maybe $\bar{6}$, 6, 3 or $\bar{3}$, the last two indicating the trigonal subdivision of the hexagonal. The second symbol indicates the a axes. The third part gives the symmetry of the intermediate axial direction.

Tetragonal, is like those of the hexagonal, except that the c or principle axis is fourfold in nature.

Orthorhombic. The three symbols refers to the a, b, and c direction in that order.

Monoclinic. The single symbol refers to the nature of the b axis.

Triclinic. In this system there are only two classes, one with only a center $\bar{1}$ and the other class has no symmetry, except one-fold axes, and it is called 1.

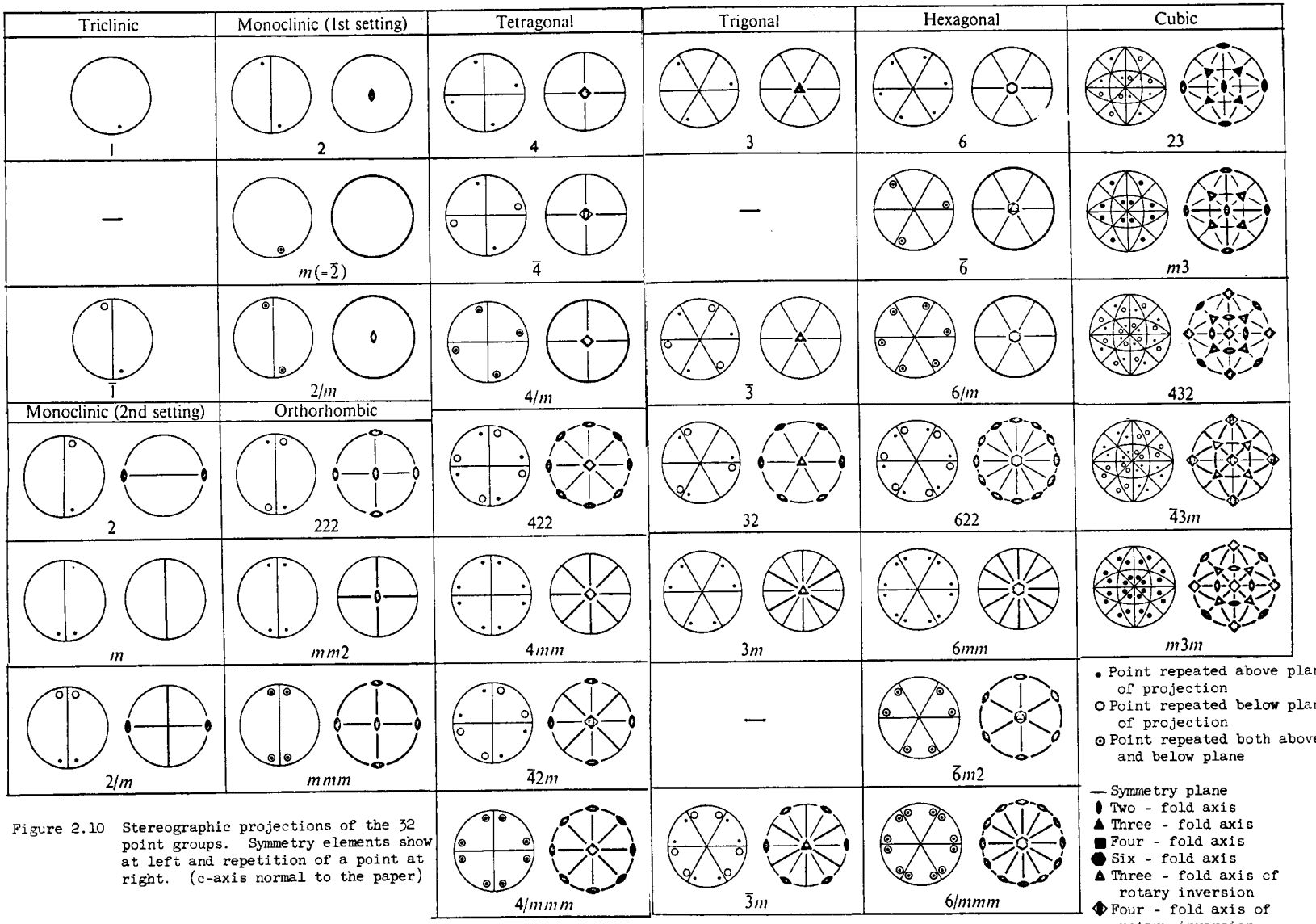
None of these systems gives the full symmetry as given in Table 1, but the full symmetry is present in notation even if not expressed. For example in the notation $\frac{2}{m}$, a two fold axis $\perp$ to a plane of symmetry, it was shown that this makes necessary the presence of a center of symmetry.

Or in the more complicated $\frac{4}{m}\frac{2}{m}\frac{2}{m}$, with a full symmetry of C, $1A_4$, $4A_2$, 5P given in the notation without producing a center, two more two-fold axes, and two more planes. The repetition of a point produced by the action of the symmetry elements of each class is shown in Figure 2.10 by the use of the stereographic projection.

2.7 Space Groups

Space groups are obtained from the application to point groups of screw axes and glide planes which produce translational symmetry not allowed in point groups. The action of translational elements on the 32 point groups produced 230 possible nonredundant space groups. The symmetry of the crystal as a whole is completely specified by the symmetry of its space group. The systems and point groups are rougher classifications of the symmetry of the entire crystal.

A point group can have only rotation axes and reflection planes, because all symmetry elements are associated with a single point. In a space group, however, the result of a repeated symmetry operation need not repeat the original array and a translation may be required to repeat



• Point repeated above plane of projection
○ Point repeated below plane of projection
⊙ Point repeated both above and below plane

— Symmetry plane
Two - fold axis
Three - fold axis
Four - fold axis
Six - fold axis
Three - fold axis of rotary inversion
Four - fold axis of rotary inversion
Six - fold axis of rotary inversion

Figure 2.10 Stereographic projections of the 32 point groups. Symmetry elements show at left and repetition of a point at right. (c-axis normal to the paper)

the original array. The screw axis and glide plane supply the needed translation. The characteristics and notation of the space group can be described as follows: A screw axis combines a rotation with a translation parallel to the axes. It has already been stated that rotation axes are limited to 1, 2, 3, 4, and 6 fold rotations; these same restrictions apply to screw axes. A screw axes combines a rotation with a translation parallel to the axis. The translation, or pitch (T), of a screw axis is required, after n rotations through an angle, to have a cumulative translation distance equal to some multiple of lattice translation m t. In other words

$$nT = mt$$

where n and m are integers. Thus, the possible values of the translation, or pitch, are

$$T = \frac{m}{n} t$$

where n is the fold of the axis 1, 2, 3, 4, or 6 and m = 0, 1, 2, 3, . . . Table 2.2 lists the only unique screw axes and their symbols. In this symbol the large numbers indicate the nature of rotation and subscript is the value of m in the above equation.

Table 2.2 Possible Screw Axes

Symbol	Rotation Angle	Translation or Pitch
2_1	π	$\frac{1}{2}$ t
3_1	$\frac{2\pi}{3}$	$\frac{1}{3}$ t
3_2	$\frac{2\pi}{3}$	$\frac{2}{3}$ t = - $\frac{1}{3}$ t
4_1	$\frac{\pi}{2}$	$\frac{1}{4}$ t
4_2	$\frac{\pi}{2}$	$\frac{2}{4}$ t
4_3	$\frac{\pi}{2}$	$\frac{3}{4}$ t = - $\frac{1}{4}$ t
6_1	$\frac{\pi}{3}$	$\frac{1}{6}$ t
6_2	$\frac{\pi}{3}$	$\frac{2}{6}$ t
6_3	$\frac{\pi}{3}$	$\frac{3}{6}$ t
6_4	$\frac{\pi}{3}$	$\frac{4}{6}$ t = - $\frac{2}{6}$ t
6_5	$\frac{\pi}{3}$	$\frac{5}{6}$ t = - $\frac{1}{6}$ t

It should be noted that such pairs of axes as 3_1 and 3_2, and 6_2 and 6_4 are enantiomorphous, that is, they differ from each other only in the direction of motion and are therefore commonly called left or right handed. Figure 2.11 A and 2.12 A shows the effect produced by a screw axis.

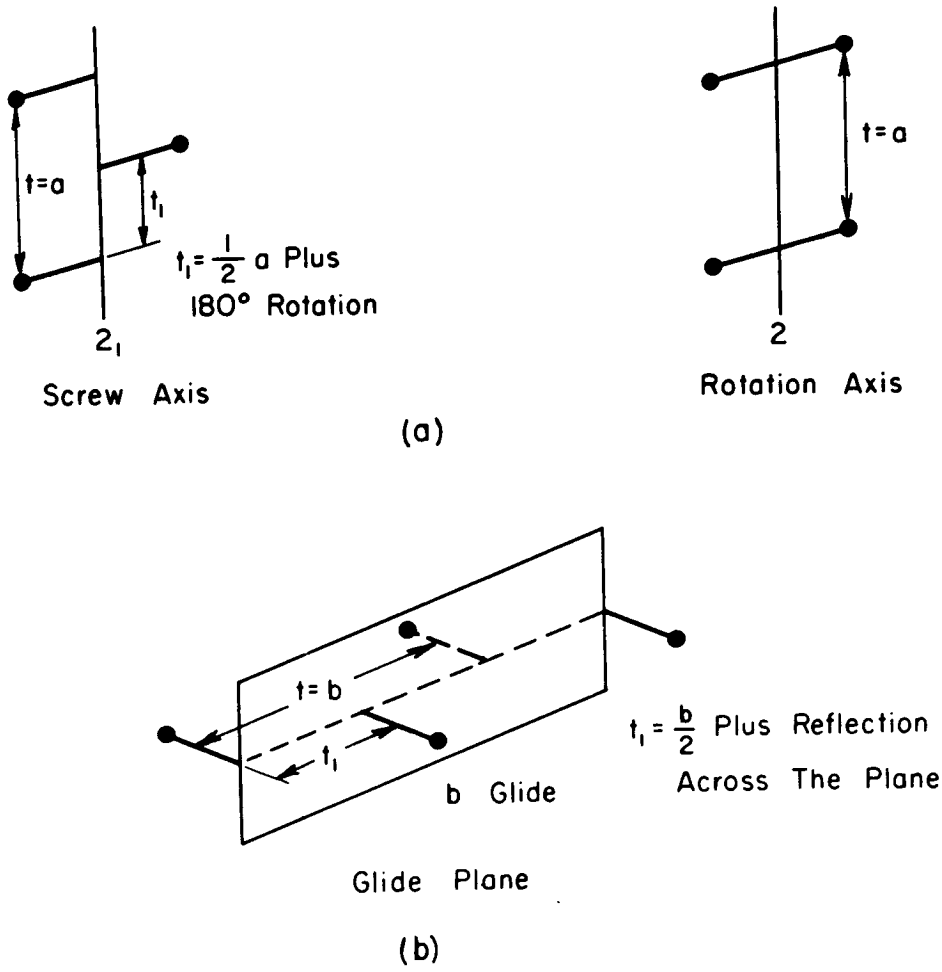


Fig. 2.11. Screw axis and glide plane

A glide plane involves the operation of reflection across a plane combined with a shift in position parallel to the plane (a translation). There are six possible glide planes which are shown in Table 2.3.

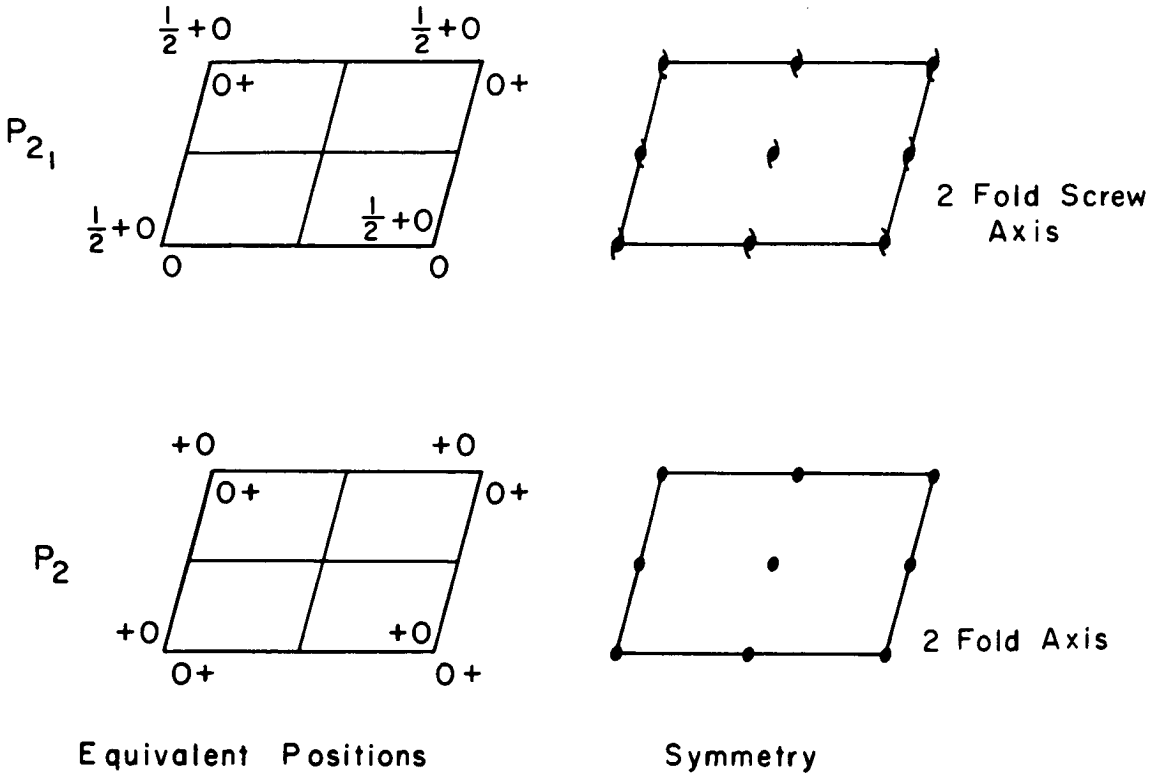


Fig. 2.12 a Equivalent positions, symmetry and the effect produced by a screw axis

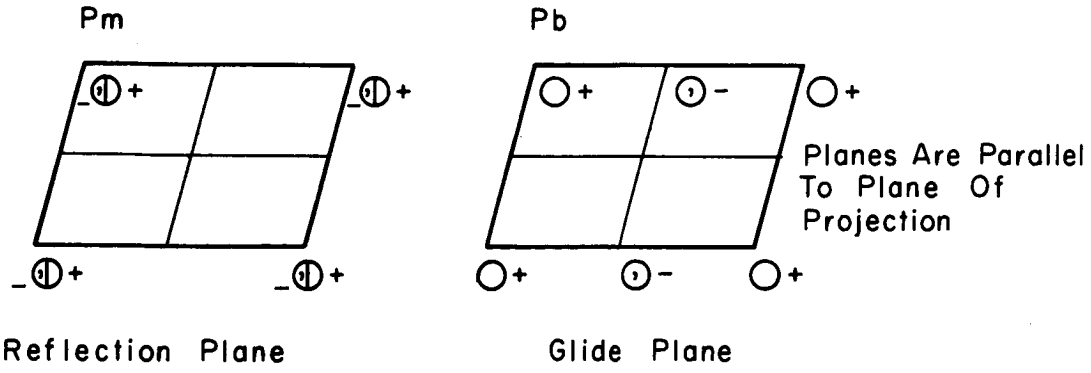


Fig. 2.12 b Equivalent positions and effects produced by a glide plane

Table 2.3 Possible Glide Planes

Symbol	Significance	Translation
a		$\frac{a}{2}$
b	axial glide	$\frac{b}{2}$
c		$\frac{c}{2}$
n	diagonal glide	$\frac{a}{2} + \frac{b}{2}$, $\frac{a}{2} + \frac{c}{2}$ or $\frac{b}{2} + \frac{c}{2}$
d	"diamond" glide	$\frac{a}{4} + \frac{b}{4}$, $\frac{a}{4} + \frac{c}{4}$ or $\frac{b}{4} + \frac{c}{4}$
m	mirror	0

Figure 2.11 B and 2.12 B shows the effect produced by an a axial glide. The crystal dimension a is not changed but a new set of points at $\frac{1}{2}$ a are produced.

2.8 Space Group Notation

The first term in the notation is the lattice type, followed by the proper point group notation to which modifications produced by the presence of glide planes and screw axes are added. For example, $P4_1 32$ would mean a primitive lattice in the point group 432, cubic, with the four-fold axis being a screw axes. $P \frac{4}{n} \frac{2_1}{c} \frac{2}{c}$ would mean a primitive lattice in the point group $\frac{4}{M} \frac{2}{M} \frac{2}{M}$, tetragonal with the four-fold axis being $\perp$ to an n glide, the a axis being a two fold 2_1 screw axis $\perp$ to a c glide and the intermediate axis is a two fold axis $\perp$ to a c glide.

The development of space group theory was in the late 19th century, this was long before the development of x-rays which demonstrates that the atomic arrangements in crystals actually conforms to these space groups.

A more detailed coverage of any of the subjects covered in this section can be found by consulting the bibliography.

Table 2.4 shows the 230 space groups and the point groups from which they are generated by the screw axes and glide planes.

Table 2.4 Space Groups

Point-group	*Hermann–Mauguin symbols*†		*Schoenflies symbols*
Triclinic			
1	$P1$		C_1^1
$\bar{1}$	$P\bar{1}$		C_i^1, S_2^1
Monoclinic‡			
	1*st setting* (*z-axis unique*)	2*nd setting* (*y-axis unique*)	
2	$P2$	$P2$	C_2^1
	$P2_1$	$P2_1$	C_2^2
	$B2$. $A2$, $I2$	$C2$. $A2$, $I2$	C_2^3
m	Pm	Pm	C_s^1
	Pb. Pa, Pn	Pc. Pa, Pn	C_s^2
	Bm. Am, Im	Cm. Am, Im	C_s^3
	Bb. Aa, Ia	Cc. Aa, Ia	C_s^4
$2/m$	$P2/m$	$P2/m$	C_{2h}^1
	$P2_1/m$	$P2_1/m$	C_{2h}^2
	$B2/m$. $A2/m$, $I2/m$	$C2/m$. $A2/m$, $I2/m$	C_{2h}^3
	$P2/b$. $P2/a$, $P2/n$	$P2/c$. $P2/a$, $P2/n$	C_{2h}^4
	$P2_1/b$. $P2_1/a$, $P2_1/n$	$P2_1/c$. $P2_1/a$, $P2_1/n$	C_{2h}^5
	$B2/b$. $A2/a$, $I2/a$	$C2/c$. $A2/a$, $I2/a$	C_{2h}^6
Orthorhombic			
	Normal setting	*Other settings*	
222	$P222$		D_2^1, V^1
	$P222_1$	$P2_122$, $P22_12$	D_2^2, V^2
	$P2_12_12$	$P22_12_1$, $P2_122_1$	D_2^3, V^3
	$P2_12_12_1$		D_2^4, V^4
	$C222_1$	$A2_122$, $B22_12$	D_2^5, V^5
	$C222$	$A222$, $B222$	D_2^6, V^6
	$F222$		D_2^7, V^7
	$I222$		D_2^8, V^8
	$I2_12_12_1$		D_2^9, V^9
$mm2$	$Pmm2$ (Pmm)	$P2mm$, $Pm2m$	C_{2v}^1
	$Pmc2_1$ (Pmc)	$P2_1ma$, $Pb2_1m$, $Pm2_1b$, $Pcm2_1$, $P2_1am$	C_{2v}^2
	$Pcc2$ (Pcc)	$P2aa$, $Pb2b$, $P2aa$	C_{2v}^3
	$Pma2$ (Pma)	$P2mb$, $Pc2m$, $Pm2a$, $Pbm2$, $P2cm$	C_{2v}^4
	$Pca2_1$ (Pca)	$P2_1ab$, $Pc2_1b$, $Pb2_1a$, $Pbc2_1$, $P2_1ca$	C_{2v}^5
	$Pnc2$ (Pnc)	$P2na$, $Pb2n$, $Pn2b$, $Pcn2$, $P2an$	C_{2v}^6
	$Pmn2_1$ (Pmn)	$P2_1mn$, $Pn2_1m$, $Pm2_1n$, $Pnm2_1$, $P2_1nm$	C_{2v}^7
	$Pba2$ (Pba)	$P2cb$, $Pc2a$, $Pba2$, $P2cb$	C_{2v}^8
	$Pna2_1$ (Pna)	$P2_1nb$, $Pc2_1n$, $Pn2_1a$, $Pbn2_1$, $P2_1cn$	C_{2v}^9
	$Pnn2$ (Pnn)	$P2nn$, $Pn2n$	C_{2v}^{10}
	$Cmm2$ (Cmm)	$A2mm$, $Bm2m$	C_{2v}^{11}
	$Cmc2_1$ (Cmc)	$A2_1ma$, $Bb2_1m$, $Bm2_1b$, $Ccm2_1$, $A2_1am$	C_{2v}^{12}
	$Ccc2$ (Ccc)	$A2aa$, $Bb2b$	C_{2v}^{13}
	$Amm2$ (Amm)	$B2mm$, $Cm2m$, $Am2m$, $Bmm2$, $C2mm$	C_{2v}^{14}
	$Abm2$ (Abm)	$B2cm$, $Cm2a$, $Ac2m$, $Bma2$, $C2mb$	C_{2v}^{15}
	$Ama2$ (Ama)	$B2mb$, $Cc2m$, $Am2a$, $Bbm2$, $C2cm$	C_{2v}^{16}
	$Aba2$ (Aba)	$B2cb$, $Cc2a$, $Ac2a$, $Bba2$, $C2cb$	C_{2v}^{17}
	$Fmm2$ (Fmm)	$F2mm$, $Fm2m$	C_{2v}^{18}
	$Fdd2$ (Fdd)	$F2dd$, $Fd2d$	C_{2v}^{19}
	$Imm2$ (Imm)	$I2mm$, $Im2m$	C_{2v}^{20}
	$Iba2$ (Iba)	$I2cb$, $Ic2a$	C_{2v}^{21}
	$Ima2$ (Ima)	$I2mb$, $Ic2m$, $Im2a$, $Ibm2$, $I2cm$	C_{2v}^{22}

Table 2.4 (continued)

Point-group	Hermann–Mauguin symbols		Schoenflies symbols
	Normal	Other settings	
mmm	*Pmmm*		D_{2h}^{1}, V_h^1
	Pnnn		D_{2h}^{2}, V_h^2
	Pccm	*Pmma, Pbmb*	D_{2h}^{3}, V_h^3
	Pban	*Pncb, Pcna*	D_{2h}^{4}, V_h^4
	Pmma	*Pbmm, Pmcm, Pmam, Pmmb, Pcmm*	D_{2h}^{5}, V_h^5
	Pnna	*Pbnn, Pncn, Pnan, Pnnb, Pcnn*	D_{2h}^{6}, V_h^6
	Pmna	*Pbmn, Pncm, Pman, Pnmb, Pcnm*	D_{2h}^{7}, V_h^7
	Pcca	*Pbaa, Pbcb, Pbab, Pccb*	D_{2h}^{8}, V_h^8
	Pbam	*Pmcb, Pcma*	D_{2h}^{9}, V_h^9
	Pccn	*Pnaa, Pbnb*	D_{2h}^{10}, V_h^{10}
	Pbcm	*Pmca, Pbma, Pcmb, Pcam, Pmab*	D_{2h}^{11}, V_h^{11}
	Pnnm	*Pmnn, Pnmn*	D_{2h}^{12}, V_h^{12}
	Pmnn	*Pnmm, Pmnm*	D_{2h}^{13}, V_h^{13}
	Pbcn	*Pnca, Pbna, Pcnb, Pcan, Pnab*	D_{2h}^{14}, V_h^{14}
	Pbca	*Pcab*	D_{2h}^{15}, V_h^{15}
	Pnma	*Pbnm, Pmcn, Pnam, Pmnb, Pcmn*	D_{2h}^{16}, V_h^{16}
	Cmcm	*Amma, Bbmm, Bmmb, Ccmm, Amam*	D_{2h}^{17}, V_h^{17}
	Cmca	*Abma, Bbcm, Bmab, Ccmb, Acam*	D_{2h}^{18}, V_h^{18}
	Cmmm	*Ammm, Bmmm*	D_{2h}^{19}, V_h^{19}
	Cccm	*Amaa, Bbmb*	D_{2h}^{20}, V_h^{20}
	Cmma	*Abmm, Bmcm, Bmam, Cmmb, Acmm*	D_{2h}^{21}, V_h^{21}
	Ccca	*Abaa, Bbcb, Bbab, Cccb, Acaa*	D_{2h}^{22}, V_h^{22}
	Fmmm		D_{2h}^{23}, V_h^{23}
	Fddd		D_{2h}^{24}, V_h^{24}
	Immm		D_{2h}^{25}, V_h^{25}
	Ibam	*Imcb, Icma*	D_{2h}^{26}, V_h^{26}
	Ibca	*Icab*	D_{2h}^{27}, V_h^{27}
	Imma	*Ibnm, Incm, Imam, Immb, Icmm*	D_{2h}^{28}, V_h^{28}
Tetragonal	Normal	Larger cell	
4	$P4$	$C4$	C_4^1
	$P4_1$	$C4_1$	C_4^2
	$P4_2$	$C4_2$	C_4^3
	$P4_3$	$C4_3$	C_4^4
	$I4$	$F4$	C_4^5
	$I4_1$	$F4_1$	C_4^6
$\bar{4}$	$P\bar{4}$	$C\bar{4}$	S_4^1
	$I\bar{4}$	$F\bar{4}$	S_4^2
$4/m$	$P4/m$	$C4/m$	C_{4h}^1
	$P4_2/m$	$C4_2/m$	C_{4h}^2
	$P4/n$	$C4/n$	C_{4h}^3
	$P4_2/n$	$C4_2/n$	C_{4h}^4
	$I4/m$	$F4/m$	C_{4h}^5
	$I4_1/a$	$F4_1/a$	C_{4h}^6
422	$P422$	$C422$	D_4^1
	$P42_12$	$C422_1$	D_4^2
	$P4_122$	$C4_122$	D_4^3
	$P4_12_12$	$C4_122_1$	D_4^4
	$P4_222$	$C4_222$	D_4^5
	$P4_22_12$	$C4_222_1$	D_4^6
	$P4_322$	$C4_322$	D_4^7
	$P4_32_12$	$C4_322_1$	D_4^8
	$I422$	$F422$	D_4^9
	$I4_122$	$F4_122$	D_4^{10}

Table 2.4 (continued)

Point-group	*Hermann–Mauguin symbols*		*Schoenflies symbols*
	Normal	*Larger cell*	
4mm	$P4mm$	$C4mm$	C_{4v}^{1}
	$P4bm$	$C4mb$	C_{4v}^{2}
	$P4_2cm$ ($P4cm$)	$C4_2mc$ ($C4mc$)	C_{4v}^{3}
	$P4_2nm$ ($P4nm$)	$C4_2mn$ ($C4mn$)	C_{4v}^{4}
	$P4cc$	$C4cc$	C_{4v}^{5}
	$P4nc$	$C4cn$	C_{4v}^{6}
	$P4_2mc$ ($P4mc$)	$C4_2cm$ ($C4cm$)	C_{4v}^{7}
	$P4_2bc$ ($P4bc$)	$C4_2cb$ ($C4cb$)	C_{4v}^{8}
	$I4mm$	$F4mm$	C_{4v}^{9}
	$I4cm$	$F4mc$	C_{4v}^{10}
	$I4_1md$ ($I4md$)	$F4_1dm$ ($I4dm$)	C_{4v}^{11}
	$I4_1cd$ ($I4cd$)	$F4_1dc$ ($I4dc$)	C_{4v}^{12}
$\bar{4}2m$	$P\bar{4}2m$	$C\bar{4}2m$	D_{2d}^{1}, V_d^{1}
	$P\bar{4}2c$	$C\bar{4}c2$	D_{2d}^{2}, V_d^{2}
	$P\bar{4}2_1m$	$C\bar{4}m2_1$	D_{2d}^{3}, V_d^{3}
	$P\bar{4}2_1c$	$C\bar{4}c2_1$	D_{2d}^{4}, V_d^{4}
	$P\bar{4}m2$	$C\bar{4}2m$	D_{2d}^{5}, V_d^{5}
	$P\bar{4}c2$	$C\bar{4}2c$	D_{2d}^{6}, V_d^{6}
	$P\bar{4}b2$	$C\bar{4}2b$	D_{2d}^{7}, V_d^{7}
	$P\bar{4}n2$	$C\bar{4}2n$	D_{2d}^{8}, V_d^{8}
	$I\bar{4}m2$	$F\bar{4}2m$	D_{2d}^{9}, V_d^{9}
	$I\bar{4}c2$	$F\bar{4}2c$	D_{2d}^{10}, V_d^{10}
	$I\bar{4}2m$	$F\bar{4}m2$	D_{2d}^{11}, V_d^{11}
	$I\bar{4}2d$	$F\bar{4}d2$	D_{2d}^{12}, V_d^{12}
4/mmm	$P4/mmm$	$C4/mmm$	D_{4h}^{1}
	$P4/mcc$	$C4/mcc$	D_{4h}^{2}
	$P4/nbm$	$C4/amb$	D_{4h}^{3}
	$P4/nnc$	$C4/acn$	D_{4h}^{4}
	$P4/mbm$	$C4/mmb$	D_{4h}^{5}
	$P4/mnc$	$C4/mcn$	D_{4h}^{6}
	$P4/nmm$	$C4/amm$	D_{4h}^{7}
	$P4/ncc$	$C4/acc$	D_{4h}^{8}
	$P4_2/mmc$ ($P4/mmc$)	$C4_2/mcm$ ($C4mcm$)	D_{4h}^{9}
	$P4_2/mcm$ ($P4/mcm$)	$C4_2/mmc$ ($C4/mmc$)	D_{4h}^{10}
	$P4_2/nbc$ ($P4/nbc$)	$C4_2/acb$	D_{4h}^{11}
	$P4_2/nnm$ ($P4/nnm$)	$C4_2/amn$	D_{4h}^{12}
	$P4_2/mbc$ ($P4/mbc$)	$C4_2/mcb$ ($C4/mcb$)	D_{4h}^{13}
	$P4_2/mnm$ ($P4/mnm$)	$C4_2/mmn$ ($C4/mmn$)	D_{4h}^{14}
	$P4_2/nmc$ ($P4/nmc$)	$C4_2/acm$	D_{4h}^{15}
	$P4_2/ncm$ ($P4/ncm$)	$C4_2/amc$	D_{4h}^{16}
	$I4/nmm$	$F4/mmm$	D_{4h}^{17}
	$I4/mcm$	$F4/mmc$	D_{4h}^{18}
	$I4_1/amd$ ($I4/amd$)	$F4_1/ddm$	D_{4h}^{19}
	$I4_1/acd$ ($I4/acd$)	$F4_1/ddc$	D_{4h}^{20}
Trigonal and Hexagonal			
3	$P3$ ($C3$)	($H3$)	C_3^{1}
	$P3_1$ ($C3_1$)	($H3_1$)	C_3^{2}
	$P3_2$ ($C3_2$)	($H3_2$)	C_3^{3}
	$R3$		C_3^{4}
$\bar{3}$	$P\bar{3}$ ($C\bar{3}$)	($H\bar{3}$)	C_{3i}^{1}, S_6^{1}
	$R\bar{3}$		C_{3i}^{2}, S_6^{2}

Table 2.4 (continued)

Point-group	*Hermann–Mauguin symbols*		*Schoenflies symbols*
	Normal	*Larger cell*	
32	$P312$ ($C312$)	($H32$)	D_3^1
	$P321$ ($C32$)	($H312$)	D_3^2
	$P3_112$ ($C3_112$)	($H3_12$)	D_3^3
	$P3_121$ ($C3_12$)	($H3_112$)	D_3^4
	$P3_212$ ($C3_212$)	($H3_22$)	D_3^5
	$P3_221$ ($C3_22$)	($H3_212$)	D_3^6
	$R32$		D_3^7
3*m*	$P3m1$ ($C3m$)	($H31m$)	C_{3v}^1
	$P31m$ ($C31m$)	($H3m$)	C_{3v}^2
	$P3c1$ ($C3c$)	($H31c$)	C_{3v}^3
	$P31c$ ($C31c$)	($H3c$)	C_{3v}^4
	$R3m$		C_{3v}^5
	$R3c$		C_{3v}^6
$\bar{3}m$	$P\bar{3}1m$ ($C\bar{3}1m$)	($H\bar{3}m$)	D_{3d}^1
	$P\bar{3}1c$ ($C\bar{3}1c$)	($H\bar{3}c$)	D_{3d}^2
	$P\bar{3}m1$ ($C\bar{3}m$)	($H\bar{3}1m$)	D_{3d}^3
	$P\bar{3}c1$ ($C\bar{3}c$)	($H\bar{3}1c$)	D_{3d}^4
	$R\bar{3}m$		D_{3d}^5
	$R\bar{3}c$		D_{3d}^6
6	$P6$ ($C6$)	($H6$)	C_6^1
	$P6_1$ ($C6_1$)	($H6_1$)	C_6^2
	$P6_5$ ($C6_5$)	($H6_5$)	C_6^3
	$P6_2$ ($C6_2$)	($H6_2$)	C_6^4
	$P6_4$ ($C6_4$)	($H6_4$)	C_6^5
	$P6_3$ ($C6_3$)	($H6_3$)	C_6^6
$\bar{6}$	$P\bar{6}$ ($C\bar{6}$)	($H\bar{6}$)	C_{3h}^1
$6/m$	$P6/m$ ($C6/m$)	($H6/m$)	C_{6h}^1
	$P6_3/m$ ($C6_3/m$)	($H6_3/m$)	C_{6h}^2
622	$P622$ ($C62$)	($H62$)	D_6^1
	$P6_122$ ($C6_12$)	($H6_12$)	D_6^2
	$P6_522$ ($C6_52$)	($H6_52$)	D_6^3
	$P6_222$ ($C6_22$)	($H6_22$)	D_6^4
	$P6_422$ ($C6_42$)	($H6_42$)	D_6^5
	$P6_322$ ($C6_32$)	($H6_32$)	D_6^6
6mm	$P6mm$ ($C6mm$)	($H6mm$)	C_{6v}^1
	$P6cc$ ($C6cc$)	($H6cc$)	C_{6v}^2
	$P6_3cm$ ($C6_3cm$)	($H6_3mc$)	C_{6v}^3
	$P6_3mc$ ($C6_3mc$)	($H6_3cm$)	C_{6v}^4
$\bar{6}m2$	$P\bar{6}m2$ ($C\bar{6}m$)	($H\bar{6}2m$)	D_{3h}^1
	$P\bar{6}c2$ ($C\bar{6}c$)	($H\bar{6}2c$)	D_{3h}^2
	$P\bar{6}2m$ ($C\bar{6}2m$)	($H\bar{6}m$)	D_{3h}^3
	$P\bar{6}2c$ ($C\bar{6}2c$)	($H\bar{6}c$)	D_{3h}^4
6/mmm	$P6/mmm$ ($C6/mmm$)	($H6/mmm$)	D_{6h}^1
	$P6/mcc$ ($C6/mcc$)	($H6/mcc$)	D_{6h}^2
	$P6_3/mcm$ ($C6/mcm$)	($H6/mmc$)	D_{6h}^3
	$P6_3/mmc$ ($C6/mmc$)	($H6/mcm$)	D_{6h}^4

Table 2.4 (continued)

Point-group	*Hermann–Mauguin symbol*	*Schoenflies symbol*
Cubic		
23	$P23$	T^1
	$F23$	T^2
	$I23$	T^3
	$P2_13$	T^4
	$I2_13$	T^5
$m3$	$Pm3$	T_h^1
	$Pn3$	T_h^2
	$Fm3$	T_h^3
	$Fd3$	T_h^4
	$Im3$	T_h^5
	$Pa3$	T_h^6
	$Ia3$	T_h^7
432	$P432$ ($P43$)	O^1
	$P4_232$ ($P4_23$)	O^2
	$F432$ ($F43$)	O^3
	$F4_132$ ($F4_13$)	O^4
	$I432$ ($I43$)	O^5
	$P4_332$ ($P4_33$)	O^6
	$P4_132$ ($P4_13$)	O^7
	$I4_132$ ($I4_13$)	O^8
$\bar{4}3m$	$P\bar{4}3m$	T_d^1
	$F\bar{4}3m$	T_d^2
	$I\bar{4}3m$	T_d^3
	$P\bar{4}3n$	T_d^4
	$F\bar{4}3c$	T_d^5
	$I\bar{4}3d$	T_d^6
$m3m$	$Pm3m$	O_h^1
	$Pn3n$	O_h^2
	$Pm3n$	O_h^3
	$Pn3m$	O_h^4
	$Fm3m$	O_h^5
	$Fm3c$	O_h^6
	$Fd3m$	O_h^7
	$Fd3c$	O_h^8
	$Im3m$	O_h^9
	$Ia3d$	O_h^{10}

Philips (1946) in speaking about crystalline materials stated: "It seems that we may place the present total at approaching 20,000 (of which about 2,000 have been found to occur in nature as minerals and many of these are of extreme rarity). Of this vast number: 50 per cent belong to the monoclinic system, a further 25 per cent to the orthorhombic, and about 15 per cent to the triclinic. Thus these three systems account for about 90 per cent of the crystal kingdom, leaving only a few per cent in each of the remaining more symmetrical system. The order of decreasing numerical importance of these seem to be cubic, tetragonal, trigonal, and hexagonal." Within each system the greater proportion of its representatives belong to the highest symmetry class."

If as previously stated the laser lattice should be optically either isotropic (restricted to cubic crystals), or uniaxial (restricted to tetragonal, trigonal, and hexagonal crystals), then only about 10% of all known crystalline material qualify.

We will next take a look at the chemical nature of crystals.

III. CRYSTAL CHEMISTRY

Crystal chemistry is the study of the relations between the chemical and structural features of a crystalline material. The discovery of x-ray diffraction was the birth of crystal chemistry for it supplied the tool to study the arrangement of atoms or ions in crystals. The real growth of crystal chemistry started in 1920 with the observations of W. L. Bragg on the interatomic distance in a set of closely related compounds. The field has grown rapidly in the last 25 years, aided by the growth of solid-state research. This is because crystal chemistry gives an explanation of why crystals have the properties they do, and helps in the attempt to produce crystals with a particular set of properties. This is the importance of crystal chemistry in laser materials, the production of crystalline materials with desired composition and properties. The crystal chemistry approach is the study of the basic particles of crystals, atoms and ions, and the geometry and way in which they are bonded together. Our first consideration will be the nature of this bonding.

3.1 Bonding in Crystals

The electrical fields associated with atoms act in such a way as to bring atoms close enough together that interaction between outer shells may occur. These interactions produce the chemical bonds that bind crystals together and belong to one of four bond types; ionic, covalent, metallic, and van der Waal's. In real crystals, pure bond types are rare. The unusual bond is a mixture of types. Crystals in which all bonds are the same kinds are called homodesmic; those which contain two or more bond types of different character and strength are called heterodesmic crystals.

The importance of the bonding in a crystal is many fold, for the type and strength of the bond controls the properties a crystal will have. The hardness, melting point, cleavage, coefficient of thermal expansion, electrical and thermal conductivity all depend on bonding. And most important of all the energy levels in laser crystals depends upon the bonding of the active ion to its surroundings.

When atoms or ions approach each other, they gain stability by attaining noble gas configurations, ones in which the outer shell of electrons is completely filled. In the metallic bond this is accomplished by the sharing of electrons from incomplete shells between a number of atoms. In this case the shared electrons form an "electron cloud" with great mobility which is responsible for the good electrical and thermal conductivity of metals. The covalent or homopolar bond is accomplished by a sharing of electrons in the incomplete shells in such a way that the shared electrons fill the outer shells of each.

Van der Waals bonds exist between atoms with completely filled outer shells because of temporary unbalance of charge due to shifts of electrons. This is sometimes referred to as the stray field bond.

Among natural minerals the metallic bond is present in native metals and in a few sulfides and arsenides. The purely covalent bond which is common in organic compounds is rare in minerals. The very weak van der Waals bond which is present in inert gases is rare in mineral, its only real importance is in some layered structures such as micas and clays.

The most common bond type in inorganic compounds and minerals is ionic. Almost all minerals except native metals, sulfides, and sulfosalts are chiefly ionic bonded. Of the 2,000 mineral species it has been estimated that about 90% or 1,800 can be considered as ionic, for the purpose of calculation without making serious mistakes.

Because of the importance of the ionic bond in crystal chemistry it will be discussed in more detail than the other bond types. An atom can achieve a completely filled outer shell of electrons by gaining or losing sufficient electrons to reach the nearest inert gas configuration. In this manner sodium loses an electron and becomes a positively charged ion with the neon configuration. Chlorine adds an electron and becomes a negatively charged ion with the argon configuration.

Ions of opposite sign, once formed, are attracted to each other by coulombic forces. There are also repulsive forces that at some distance will balance the attractive forces. The distance at which these repulsive forces balance the attractive forces is the interionic distance characteristic of these ions. The interionic distance between a pair of ions of opposite charge is characteristic of that pair of ions. This is the basis for the ionic radius of an ion. The interionic distance is regarded as the sum of the radii of two ions in contact. Thus if we assume the radius of one ion, the other can be found. In this way tables of ionic radii are obtained. The tables are usually based, in part, on a radius of 1.32 Å for oxygen. The actual considerations of ionic radii are more complicated and numerous corrective factors are usually employed in preparing a table of ionic radii. We will now consider some of the factors that effect the size of ions.

3.2 Size of Ions

The nature of the forces are simplest for ionic solids and the binding energies are thus more easily calculated than for other crystalline solids. The ionic attraction can be closely approximated by the electrostatic forces between a pair of ions treated as point charges of positive and negative electricity. The Coulomb attraction between two point charges Z_1e and Z_2e at a distance r apart is given by,

$$u = \frac{Z_1 Z_2 e^2}{r}$$

At close approach, repulsive forces act on the ion. Born and Lande expressed this repulsive force in the following expression for the potential energy of a pair of ions.

$$u = Z_1 Z_2 \frac{e^2}{r} + \frac{be^2}{r^n}$$

Where the positive term is an empirical expression of the repulsive force, b is a constant, and n is the power of the distance to which r must be raised to give the correct repulsive force. The value of n can be determined from compressibility measurements of crystals and has the approximate values of 5 for H^- or He, 7 for Ne, 9 for Ar or Cu^+, 10 for Kr or Ag^+ and 12 for Xe and Au^+ and average values for crystals of mixed types.

In a crystal there are present repulsive forces which are also inversely proportional to some power of the separation. The potential energy of the crystal can therefore be written,

$$u = A \frac{Z_1 Z_2 e^2}{r} + B \frac{1}{r^n} \qquad \text{(a)}$$

where A is (the modeling constant) a numerical constant of the order of unity, which sums the effect of all the ions in the structure and is thus constant for each structure type, and B is a constant yet to be determined. For the simplest case of the univalent alkali halides (a) reduces to,

$$u = \frac{-Ae^2}{r} + \frac{B}{r^n} \qquad \text{(b)}$$

At the equilibrum distance r_o potential energy is a minimum and the first derivative of b will vanish,

$$\left(\frac{du}{dr}\right)_{r=r_o} = \frac{Ae^2}{r^2} - \frac{nB}{r^{n+1}} = 0$$

it follows that

$$B = \frac{Ae^2 r_o^{\,n-1}}{n}$$

so that

$$u = \frac{-Ae^2}{r_o}\left(1 - \frac{1}{n}\right).$$

Born and Mayer found the repulsive term to be incorrect because the van der Waals forces were not taken into account. They gave the following relation for potential energy which took the van der Waals forces into consideration:

$$u = \frac{Ae^2}{r} + Be^{-r/n} - \frac{c}{r^6} + \delta$$

where c is a constant and δ is a small number which expresses the energy of the structure at absolute zero. In the above, the positive term is the repulsive force term and the negative is the attractive force term. The general effect of these are the repulsive forces tend to increase the size of the ion, and the attractive forces tend to decrease it.

The value of the above equation is that it makes it possible, within limits, to predict the structure that a group of atoms will assume. This will be the most stable structure or the one with the lowest potential energy (u).

3.3 Estimation of Bond Type

As previously pointed out the mixed covalent-ionic bond is the most common. The commonness of this mixed bond type makes it desirable to be able to determine the per cent of each in a mixed bond. Pauling has introduced a plot of electronegativity difference which expresses the amount of ionic-covalent character a bond in a compound has. Pauling gave each element a numerical value of electronegativity, which he describes as the power of an atom in a molecule to attract electrons to itself.

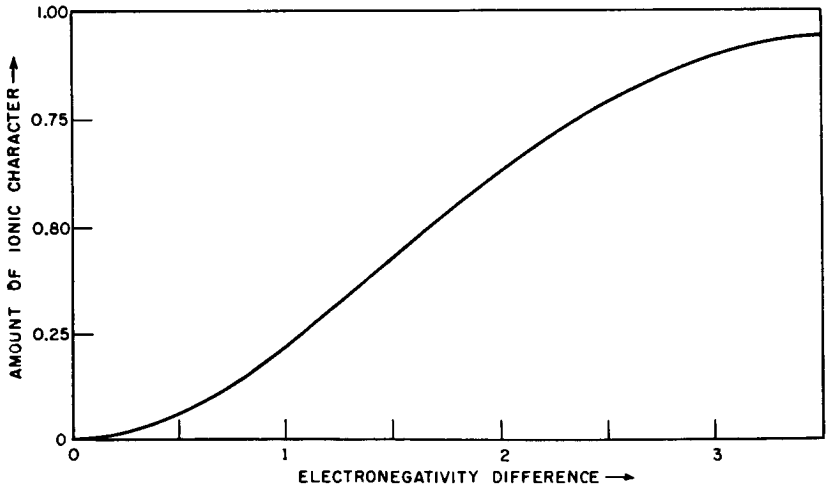


Fig. 3.1. Curve relating amount of ionic character and electronegativity (after Pauling 1960)

Pauling (1960, p. 93) gives a rather complete list of electronegativity values for elements in their common oxidation states and Gordy and Thomas (1956) for the less common oxidation states. Figure 3.1 gives Pauling's plot and Table 3.1 gives the electronegativity values for some of the common ions and for the rare earths.

For example, the very common Si-O bond, 3.5 - 1.8 = 1.7, is found to be 50 per cent ionic and 50 per cent covalent. It is easily seen that most common rare earth elements will have a greater ionic than covalent character, and that the bond between the rare earth ion and F in CaF_2 will be very highly ionic.

Table 3.1 Electronegativity Values

LA ↑		Sb	1.8	K	0.8
Ce		As	2.0	Na	0.9
Pr		Te	2.1	Ca	1.0
Nd		P	2.1	Li	1.0
Pm		Se	2.4	Mg	1.2
Sm	1.1	I	2.5	Al	1.5
Eu	to	S	2.5	Si	1.8
Gd		Br	2.8	C	2.5
Tb	1.2	Cl	3.0	O	3.5
Dy		N	3.0		
Ho		F	4.0		
Er					
Tm					
Yb					
Lu ↓					

3.4 Pauling's Rules

Pauling in 1929, proposed on the basis of observation of packing arrangements, a set of principles that determined the structure of ionic crystals.

Rule 1. "A coordinated polyhedron of anions is formed about each cation, the cation-anion distance being determined by the radius sum and the coordination number of the cation by the radius ratio."

The anion is usually oxygen, and the radii of the anions is usually considerably larger than the radii of the cations. Because of the electrostatic nature of the binding forces each cation tends to surround itself with as many anions as possible. The number that can be grouped about each cation depends on the relative sizes of the cation and anion. The number of anions that surround each cation is known as the coordination number of the cation. (It is also some times referred

to by the nature of the coordination polyhedron produced as cubic, tetrahedral, etc.). Relative size of ions is expressed by the radius ratio, which is the ratio of the radius of the cation to the radius of the anion.

Rule 2. "In a stable coordination structure the total strength of the valency bonds which reach an anion from all the neighboring cations is equal to the charge of the anion." This rule is called the Electrostatic Valency Principle. In its applications, the valence of the cation is divided equally among the number of bonds to the anion, this number depending on the coordination number

$$\frac{\text{Charge cation}}{\text{Coordination number of the anion}} = \text{Electrostatic valency (E.V.)}$$

Rule 3. "The existence of edges, and particularly of faces common to the two anion polyhedra in a coordinated structure decreases its stability; this effect is large for cations with high valency and small coordination number, and is especially large when the radius ratio approaches the lower limit of stability of the polyhedron."

The sharing of elements of tetrahedra are shown in Figure 3.2.

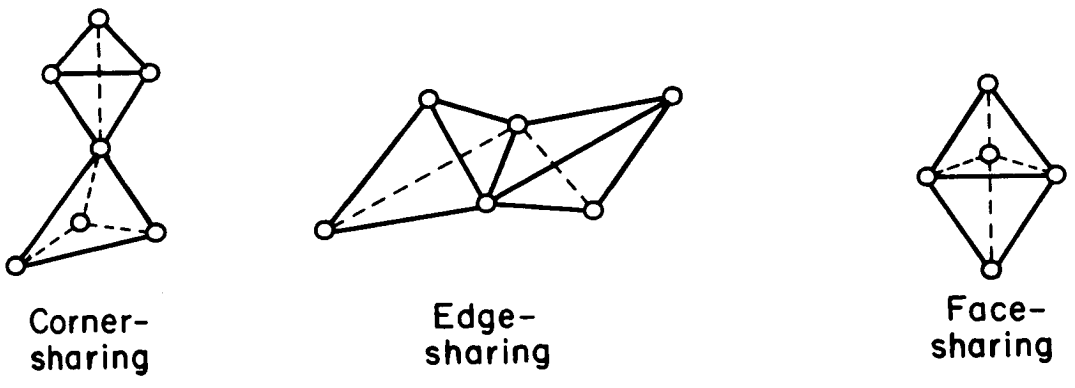


Fig. 3.2. Sharing of polyhedral elements in tetrahedra

This principle is explained by the fact that highly charged cations tend to maintain as large a separation as possible. The closer together cations become, the larger the crystal energy and the lower the stability. The closeness is increased in the order, corner sharing, edge sharing, face sharing. The crystal energy increases in the same order and the stability decreases in this order.

Rule 4. In a crystal containing different cations those of high valency and small coordination number tend not to share polyhedron elements with each other. Explanation is the same as above (under 3).

Rule 5. The number of essentially different kinds of constituents in a crystal tends to be small.

This refers to crystallographic configuration and arises from the tendency in nature for substances to attain forms of lowest potential energy and thus of simplest structure.

We will now discuss some of Pauling's rules in more detail and the effect they have in crystal chemistry.

3.5 Coordination Polyhedra

If the coordinated ions forming polyhedra and the coordinating ion at the center of polyhedra are the same size, the radius ratio is one. There are two possible three-dimensional arrangements which permit each sphere to form intimate contact with the largest number of neighbors. These are the so-called hexagonal and cubic closest packing. In both cases the central sphere is surrounded by 12 nearest neighbors, and in both the hexagonal and cubic closest packing the central sphere is in contact with six spheres in its own layer. The difference in the two close packed arrangements is the way in which spheres in the layers above and below the center sphere are arranged. We will not discuss these differences for this type of arrangement is important only in metals and micas. Anyone interested in the nature of these arrangements is referred to Azaroff (1960, p. 58). This is referred to as 12-fold coordination or as having coordination number (C.N.) 12.

The most frequent coordination encountered in actual crystals are 3, 4, 6, 8, and 12. Of these 4, 6, and 8 are common and 4 and 6 are the most common. Three-fold is common only in radicals such as CO_3, NO_3, and BO_3 and as previously stated 12-fold is common only in metals and micas, 2-fold is rare. The other coordinations 5-, 7-, 9-, and 10-fold coordination, are very rare and require special arrangement in very complex structures. It has been found in considering radius ratio for the prediction of coordination, of noncomplex structures that 5-, 7-, 9-, and 10-fold coordination can be omitted without producing difficulty.

We will thus, consider 8-fold coordination next. If the size of the coordinating cation is slightly smaller, 8-fold coordination is

stable. The centers of the anions lie at the eight corners of a cube and this is sometimes called cubic coordination. A consideration of the geometry of this arrangement makes it possible to determine the limiting conditions under which this coordination will be stable. If we let the radius of the anion be equal to unity we find the lower limit for cubic coordination is in radius ratio of 0.732.

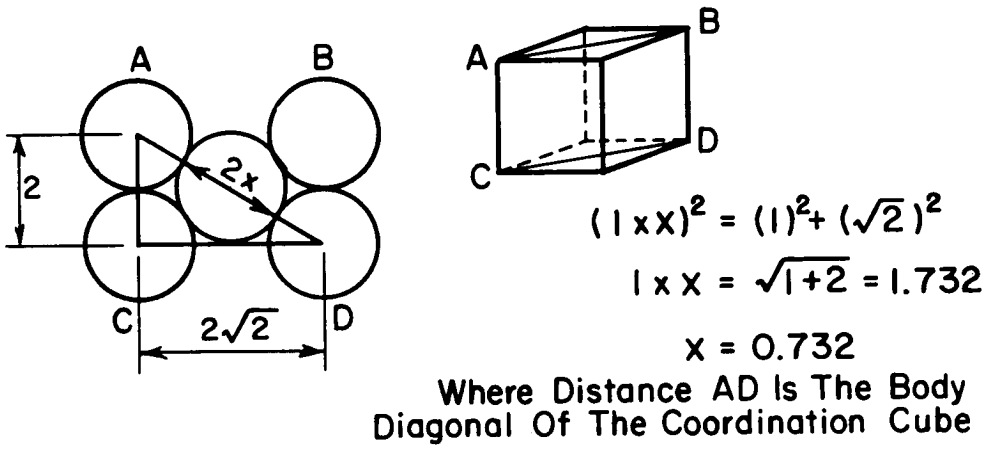


Fig. 3.3. Cubic coordination

This cubic coordination has maximum stability for values of radius ratio between 0.73 and 1.00.

It is possible by simple geometric consideration of the type shown above to establish the range of radius ratios for which a given coordination is stable. Table 3.2 records this information.

Table 3.2 Radius Ratio and Coordination

Range of stable radius ratio cation/anion	CN number of nearest anion negatron	Anion polyhedra formed
Less than .155	2	
.155 - .255	3	planar equilateral triangle
.255 - .414	4	tetrahedron
.414 - 732	6	octahedran
.732 - 1.00	8	cube
Greater than 1.00	12	cubic or hexagonal close packed arrangement

In some cases the radius ratio fails to predict the coordination that is experimentally found by x-ray structure studies. In most cases this is due to polarization, that is, a distortion of the ion that makes it act as if it were an ion of different size. The polarization is determined by the coordination, polarizability and polarizing power of the ions concerned. The polarizability of an ion depends on size and looseness of binding and is thus pronounced in large anions. The polarizing power of an ion depends on its electric field and is greatest for small highly charged ions, chiefly cations. Thus care must be used when applying the radius ratio concept to predict coordination in structures that have small highly charged cations and large feebly charged anions. Also the radius ratio concept only applies to materials that are ionically bonded and thus would fail if applied to materials in which the chief bonding is covalent.

3.6 Electrostatic Valency

The relative strength of the ionic bond linking ions is a function of valence and coordination. This is because the strength of all the bonds reaching an ion must equal the valence of that ion and these bonds are supplied by the nearest neighbors.

The electrostatic valency is a measure of the relative strength of any bond reaching the coordinating ion. It is computed by dividing the valence of the coordinating ion by the number of nearest neighbors. If, for example the cation has a valence of 4 and it is in 8-fold coordination, the electrostatic valence (ev) = $\frac{4}{8}$ or $\frac{1}{2}$. The coordination ion gives up $\frac{1}{2}$ a charge unit to each of its neighbors. Crystals like this in which all bonds are of equal strength are termed isodesmic. If the strength of the bonds within a radical such as CO_3 or NO_3 is calculated, it is always found to be greater than one-half the total charge on the anion.

For example, CO_3 in $CaCO_3$, the radius ratio indicates 3-fold coordination. The valence is 4, thus the ev = $\frac{4}{3}$ = $1\frac{1}{3}$, which is greater than one-half the (-2) charge on the oxygen. Crystals in which radicals are formed are termed anisodesmic. This seems simple but consideration should be given the fact that the compound $BaTiO_3$ is often called barium titanate, implying the presence of titanate radicals. But when we consider the electrostatic valency (e.v.),

$$\frac{Ti}{O} \quad \frac{.76A}{1.32A} = .50 = \text{6-fold coordination}$$

$$(e.v.) = \frac{4}{6} = \frac{2}{3}$$

which is less than one-half the charge on the oxygen, $BaTiO_3$ is thus a double salt and not a titanate and anyone studying such a material should realize this.

If the same type calculation is performed and the strength of the bonds is found to equal exactly half the charge of the anion, the crystal is called mesodesmic.

Many of the properties of crystals are controlled by the bond strength. The bond strength is inversely proportional to the interionic distance and directly proportional to charge. The greater the bond strength, the more energy required to break bonds and the higher the melting point and hardness, and the lower the coefficient of thermal expansion. Other physical properties, such as cleavage, etc., and chemical properties, such as solubility, etc., are also dependent, at least in part, on bond strength.

3.7 Substitution

The major consideration in the "doping" of a crystal is what controls this "doping" or substitution. In nature substitution of one ion for another is the rule rather than the exception. This substitution is usually referred to as substitutional solid solution or ionic substitution. The extent to which substitution takes place is determined by the size and charge on the ions involved, nature of the structure, and the temperature of formation. The most important single factor is the size of the ion, which must fit without distortion. Goldschmidt, based on empirical data gathered from the substitution in ionic crystals, came to the conclusion that ions of two elements can readily substitute for one another if their ionic radii differ by no more than 15 per cent. If the radii difference is between 15 and 30 per cent, substitution is difficult, and if the difference is greater than 30 per cent, substitution is not likely.

It is not essential that the substituting ions have the same charge; a difference of one can be tolerated, as long as the electrical

neutrality of the structure is maintained. The neutrality of the structure is maintained by what is called coupled substitution. The substitution of 3+ rare earth ions for Ca^{++} in crystals of CaF_2 or $CaWO_4$ are examples of this kind of substitution. The size of the Ca^{++} and RE^{+++} ions are very near, but in order to keep the structure neutral it is necessary to put in enough Na^+ also nearly the same size, to balance the excess positive charge caused by the rare earth ions. This is the usual way in which the charge is balanced, but it can also be done by "holes" or vacant ion sites in the structure.

The effect of the other factors that control substitution are:

1. Coordination number - the lower the C.N., the more rigid is the size requirements.

2. Polarization - the higher the polarizing power of an ion the harder it is to replace.

3. Speed of growth - the more rapid, the greater the amount of substitution.

4. Temperature of growth - the higher the temperature the increased chance of substitution.

In the above, 3 and 4 also have a tendency to produce a distorted crystal, so where lattice perfection is required, it should not be used.

It is also possible in an open structure such as sphalerite to introduce large amounts of material which don't go into the lattice sites but rather take up interstitial sites.

Because of the importance of size, and to a lesser extent charge, if substitution in a structure is to take place a "foreign ion" of the proper size must exist. In some cases this is not true and some ions show almost no substitution. This is somewhat the case with Si and is the reason why natural quartz is so pure, and why it is not possible to dope a foreign ion into the quartz structure. It is, however, possible in quartz because of its open structure to put minor amounts of material in interstitial sites without disrupting the structure.

Figure 3.4 is a plot of ionic charge against ionic size for most of the common ions and the ions of interest in laser materials, and gives an indication where substitution might be expected. It should again be noted here that tables of ionic radii are calculated on the basis of 6-fold coordination and if the coordination is not 6 fold the size of the ion is changed by a small factor.

If crystals of high "doping" are required, the best way to produce these are to use mixed crystals, or isostructural crystals. These are crystals with the same structure, having a common anion and usually displaying extensive ionic substitution.

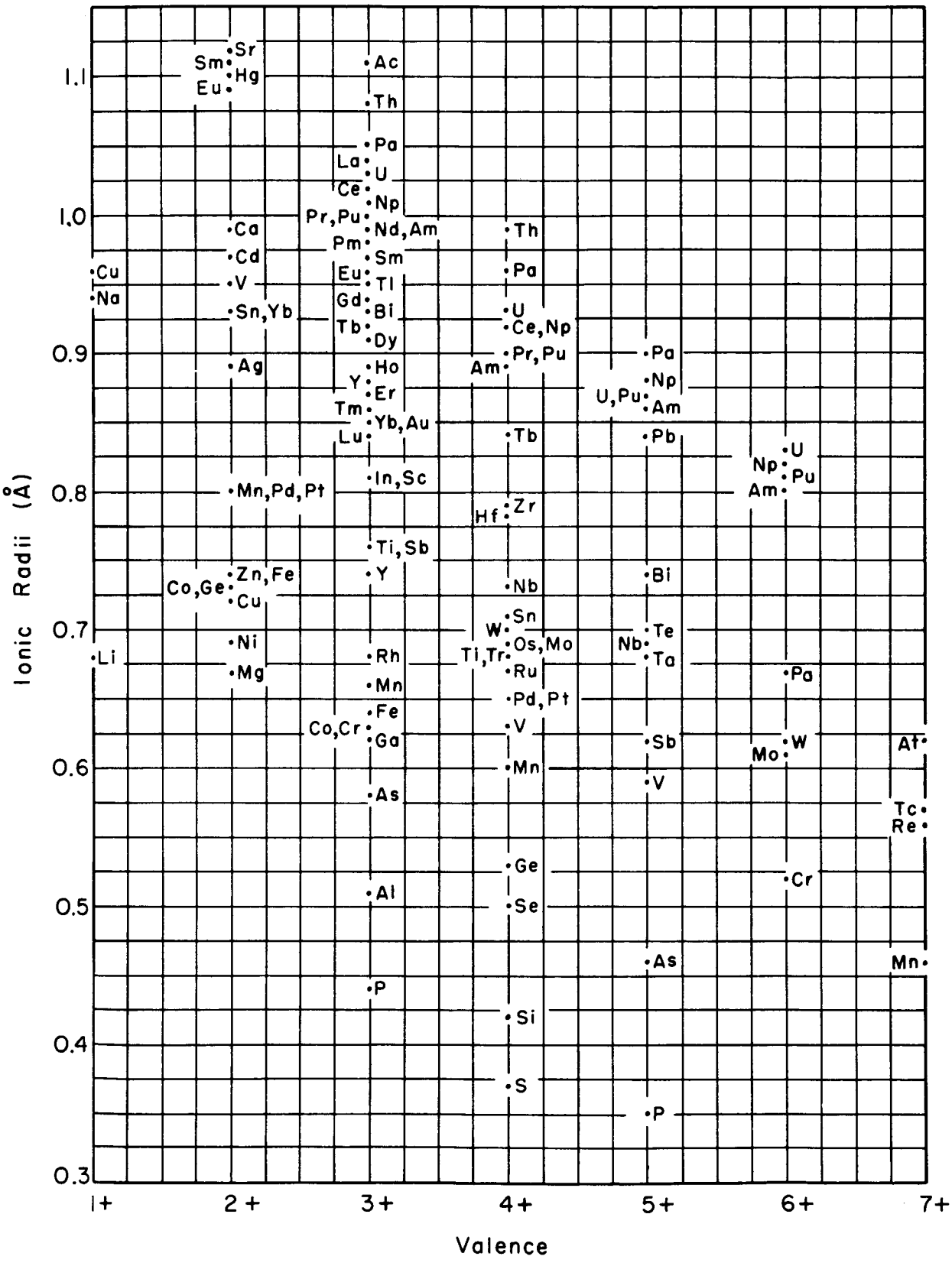


Fig. 3.4 Plot relating ionic radius and ionic charge

3.8 Polymorphisn

Some crystalline solids having the same chemical composition can exist in two or more crystalline forms depending on the temperature, pressure, and the presence of minor impurities. This phenomenon termed polymorphisn or allotropy is of importance in a consideration of laser materials. The crystal may be subject to liquid gas temperatures perhaps causing an inversion from one state to another.

For any given crystalline substance there is just one thermodynamically stable structure for a given range of temperature and pressure. For a crystal at absolute zero the most stable structure is the one with the least lattice energy. An increase in temperature causes increased atomic vibrations and produces strain on the crystal bonds. In adjustment to this strain the high temperature form, if one exists, has the lower C.N. If the temperature is further increased, the strain becomes great enough to break bonds and the crystal melts. In some substances, however, there exists at some temperature an alternate structure capable of resisting the thermal agitation better than the existing one and a polymorphic inversion, or transformation, to the new stable structure occurs. The new structure will persist until another inversion takes place or until it melts.

Another consideration of possible importance in producing "doped" laser crystals is related to polymorphism. The low temperature modification generally has the higher specific gravity and thus the least open structure. Substitutional solid solution can take place only when an opening exists of the proper size to accept the ion to be substituted.

The less compact, usually the higher temperature form, would have greater chance of accepting an impurity ion. Also if the high temperature modification were the one with favorable laser character it might be possible to extend its range of stability indefinitely by additions at high temperature of large numbers of impurity ions.

One of the best known and most studied examples of polymorphism, is the polymorphism of SiO_2 which has at least eight polymorphs.

3.9 Structure Type

A concept that has great importance in crystal chemistry is the idea of structure type. If a substance is said to have the rutile structure type, it means that the ions in its structure have a similar relationship to each other as the ions in rutile. Rutile has a 6-3 coordination scheme, which means that around each Ti are 6 O's and around each O is 3 Ti's. It should be noted that this meets the requirements that there be twice the number of Ti as O ions as indicated by the chemical formula TiO_2. All substances having the rutile structure type have six anions around each cation and three cations around each anion. It should also be noted that it is the rutile structure type and not the TiO_2 structure

type for TiO_2 has two other polymorphic forms, brookite and anatase which have structures different from rutile.

The structure type concept makes it possible to predict the properties a substance of known structure type will have. This is due to the effect on properties produced by bond strength as has already been pointed out. Predictions of this type can only be made within a structure type and for compounds in which the bonding is chiefly ionic. If within a structure type two compounds exist, the one with lower charge will have the lower melting point and hardness; or if the charge on the ions is the same, the one with the greater interionic distance will have the lower melting point and hardness. The effect produced by charge is greater than that produced by interionic distance. One of the objectives of crystal chemistry is to aid in the synthesis of crystalline materials having desired properties and the above will aid in doing this.

We have considered the nature of crystalline materials and now we will turn our attention to the methods used in the synthesis of crystalline materials.

IV. CRYSTAL GROWTH

Crystal growth is a process that nature has carried out for billions of years. Man's entry into the field of crystal growth shows a much shorter and, in general, less successful history. The early attempts at crystal growth were usually in conjunction with experiments in chemistry, whose chief function was the separation of material for classification or purification.

The earliest uses of natural crystals were as gemstones. Since prehistoric times, man has valued crystals for their ornamental value. Thus, it's little surprise that one of the earliest successes in the large-scale production of single crystals was Verneuil's growth of synthetic gemstones. The development of crystal growth has followed this pattern ever since, that is, the production of crystals to fill a need. The second great advance in crystal growth resulted from a need for large good quality crystals because of their spectral and piezoelectric properties. The most recent and perhaps most demanding need has been for single crystal semiconductors and now the need of high quality single crystals for masers and other solid-state devices.

The presentation here will be limited to a brief discussion of crystal growth techniques and the materials that they provide. Such topics as velocity of ideal growth, theories of growth, mechanism of growth, and dislocations, though important, are beyond the scope of this report and anyone interested in any of these subjects is referred to the bibliography for further reading.

On the subject of available literature, it is large but scattered, only six references are at all complete. These are the 1944 Volume of the Faraday Society, the first two Russian congresses on crystal growth (translated into English), the outstanding work of Buckley (still the best overall book on crystal growth), and the more recent works of Mullins and Van Hook. There exists a real need for a book on crystal growth that could take into consideration the great advances made in this field in recent years. The recent books, of Van Hook and Mullin, though well done do not completely fill the existing need in the field of recent work on the growth of large single crystals.

As previously stated, nature has produced crystals for billions of years. The processes used to produce crystals in the laboratory are similar to those that operate in nature. The attempts in the laboratory to produce the conditions of crystallization that have existed in nature, have to some extent been successful. Some of the factors that in the past have produced difficulty, are high temperatures and pressures. Recently, to some extent, these have been overcome, but the time factor seems the unconquerable factor in duplicating natural processes.

Methods of Crystal Growth

There are three general ways in which crystals have been formed in nature and these have all been duplicated in the laboratory. We will divide our discussion into these three types of growth. They are growth from the solution, growth from the melt, and growth from the vapor.

4.1 Growth from Solution

Growth from a saturated solution is the cheapest and oldest of the methods for single crystal growth. The growing is performed at low to moderate or often room temperature. These low temperatures mean that the apparatus can be simple and inexpensive. The chief demand is for a constant and controlled temperature and a dust-free work area. The choice of the solvent is of great importance; in fact, the use of this method depends on being able to find a suitable solvent. The most common solvent is water but many other things have been used, especially organic solvents and, to a lesser extent, melts of inorganic compounds and metals. A good solvent should have low viscosity, to facilitate transport of solute to the growing crystal, and a solubility in excess of 5 to 7 mole per cent. In this method, a solute is added to an appropriate solvent and then the solution is made supersaturated by one of several procedures to be discussed later. In the saturated solution, small crystals start to grow. One or more of these are chosen as seeds and are suspended in the solution and allowed to grow. Growth takes place by the diffusion, through the solution, to the seed of solute which is added to the seed.

Buckley has comprehensively traced the methods and apparatus for the preparation of single crystals from solution. The methods differ chiefly only in the way in which saturation is obtained in the solution. To obtain saturation, three different techniques are employed: (1) withdrawal of solvent by evaporation, (2) a change in temperature, and (3) addition of solute.

Changing Temperature

The solubility of most solutes in a solvent is a function of temperature. Figure 4.1 shows the plot of solubility and supersolubility as curves. The slopes of these curves may be either positive or negative. If the temperature of a solution is changed in the appropriate direction, the solution will become saturated. If this temperature change is continued, the solution will become supersaturated and the growth of seed crystals or the addition to an existing seed will occur. (Temperature changes from A to B, Figure 4.1). It is best if a system can be chosen in which both the temperature coefficient and solubility are large. Some of the apparatus used is shown by Buckley. For the most part, it is simple and differs only in the way in which the seed is held, the solution stirred, and the temperature controlled. Stirring is necessary in all methods of growth from solution to prevent concentration differences.

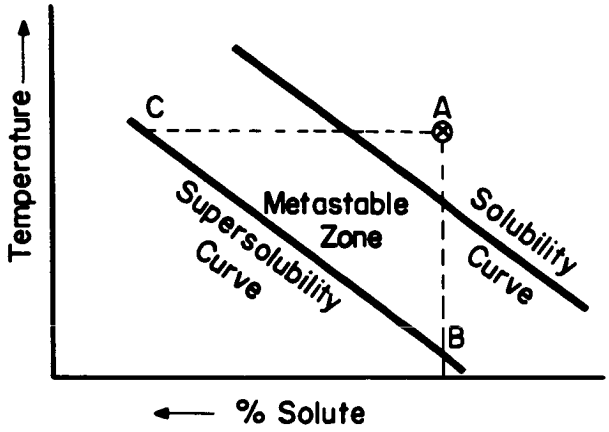


Fig. 4.1. Solubility and supersolubility

Evaporation of Solvent

This was probably the first method used for crystal growth, as it was employed in the production of salt. This method causes a change in the solution that would follow a line like A to C (Figure 4.1). It can be used in systems with a low temperature coefficient of solubility.

The apparatus differs from that used in the changing temperature method only in attempting to encourage evaporation instead of changing temperature. Both methods use the same types of seed mountings and solution-stirring devices. A vacuum is sometimes used to encourage evaporation.

Addition of Solute

In this method more solute is added to the solution as it is used up. The concentration of the solution is thus held almost constant. In Figure 4.1, the concentration would be held at some point near the supersaturation curve; the requirement of the temperature coefficient of solubility and total solubility are somewhat reduced.

The apparatus again uses the rotation and mounting of crystals as in the other methods, but differs in over-all design. Various types are used but all basically consist of two containers, one for growing crystals and another to supply new saturated solution to the first. The method of circulation of solution varies greatly.

The rate at which high quality crystals can be grown from solutions is much slower than from the melt. If the rate of growth is too rapid, inclusions of liquid are apt to be trapped in the crystal. The slow growth rate is one of the chief advantages of the growth from the melt over that from solution. In general, the rate from the melt is as fast per minute as the per day rate from solution, a matter of a few millimeters.

In evaluating the three methods for growth from the solution, evaporation is the poorest to produce quality crystals, changing temperature the best, and addition of solution a close second which sometimes works in cases where the other two cannot be used. These methods are used in the production of large single crystals of salts as ADP, EDT, Rochelle salt, and alkali-halides, in quantity.

4.2 Hydrothermal Method

The hydrothermal method is a variation on the growth from the solution method. Materials which have negligible solubility in aqueous solution at room temperatures have been shown to have some solubility at temperatures near the critical temperature of water, 374.2°C. The hydrothermal method permits variation of the pressure at which growth takes place, and makes it possible to increase the growth temperature to near the critical temperature of water. The change in growth temperature results in a change in solubility and nucleation and allows crystals to be grown that cannot be grown by any other method.

In operation, growth is carried out in a pressure vessel, usually called a bomb or autoclave (Figure 4.2). A temperature difference is established between the top and bottom of the autoclave. In the lower hotter part, the nutrient is placed and in the cooler upper part, a seed crystal. The hot solution with the nutrient is transported by convection to the upper cooler part containing the seed. Upon reaching the cool upper part, the solution becomes supersaturated with respect to the seed and it supplies nutrient to the growing crystal. The solution cools and is depleted of its nutrient and by convection is returned to the lower zone to again dissolve more nutrient. The apparatus differs somewhat in general detail but all hydrothermal apparatus requires a pressure vessel capable of containing the corrosive solvent, and a means of producing, controlling, and recording the temperature.

This method has been used to produce large quartz crystals of high quality. The growth rate of this quartz has been as much as $1\frac{1}{2}$ grams per day. It has the advantage of being able to grow materials not stable at the melting point (α-quartz) and also substances with high vapor pressure (ZnO), and substances that decompose at low temperatures but cannot be grown from standard solutions. The chief disadvantages deal with the matter of techniques and the hazards of high pressure.

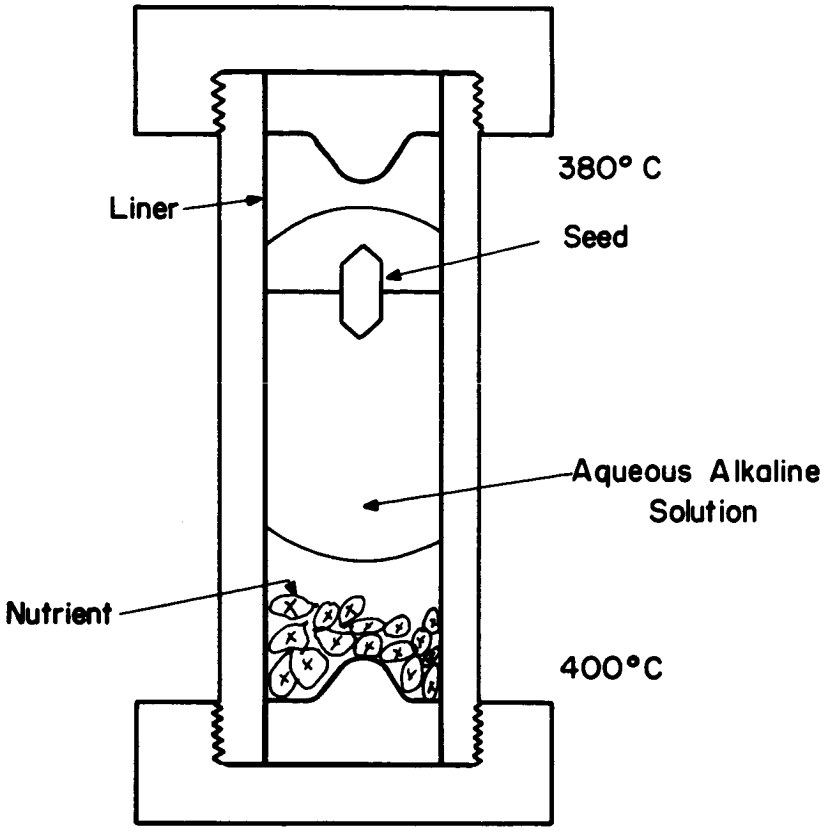


Fig. 4.2. A hydrothermal autoclave

4.3 Growth from the Melt

When it is not possible to obtain the desired single crystals by the simple and inexpensive solution method some other method must be found. The method that has been used most is growth from the melt. The growth of crystals from anhydrous melts can be accomplished by any of several methods. All these methods fall into one of three categories:

(a) Crystal pulling or withdrawal method (Kyropoulas-Czochralski)
(b) Crucible method (Stockbarger, Bridgman, and Stober)
(c) The flame fusion (Verneuil)

Withdrawal Method

In this method a crystal is withdrawn from a melt held in an open crucible. A small single crystal seed is dipped into the surface of the melt and then slowly withdrawn. The disadvantage of crucible restraint is eliminated if the seed is lowered into the top of the melt near the center and is then withdrawn rapidly enough that the growing crystal is

not permitted to touch the crucible but slowly enough that the crystal-liquid contact is not broken. The melt is kept just above the melting temperature of the material and the seed is kept slightly cooler than the melt so that material will solidify as it is withdrawn. Actually the crystal is raised rapidly so that the freezing interface is actually above the normal surface of the melt. The freezing interface is supported above the melt surface by the surface tension between the crystal face and the melt.

Figure 4.3 is a schematic diagram of a crystal puller. The polycrystalline raw material in crucible A is melted by furnace B. The seed C is attached to shaft D which is cooled and can be rotated as it is withdrawn. The rotation serves two purposes, that of stirring the melt and also any spurious nucleation taking place on the surface of the melt is swirled away from the growing crystal. The secondary furnace E is to reduce the strain on the crystal caused by too rapid a thermal gradient. The whole apparatus is enclosed in container F so that the crystal can be pulled in an inert atmosphere at atmospheric or higher pressures or in a vacuum.

Apparatus requirements are very critical. Some of the more important of these are:

(a) Little or no mechanical fluctuation in the pulling and rotation mechanism.
(b) A crucible able to withstand the temperature required.
(c) Thermal fluctuations of 0.5°C or less are desirable and in some cases required.
(d) It must be able to maintain a good thermal gradient.

Pulling rates vary with the material and apparatus but are of the magnitudes of 0.1 to 5.0 cm per hour. Slower pulling rates have a tendency to produce more perfect crystals. If all apparatus requirements are met and the starting material is pure, the pulling proceeds without trouble. The only other factor that will affect the quality of the final crystal is the quality of the seed crystal.

The shape of the final crystal is controlled by the surface tension between melt and crystal, pulling rate, and temperature gradient, as well as by the lattice constants of the material. It is possible within limits to control the shape of the final crystal. The orientation can be controlled by the use of a properly oriented seed.

Crucible Method

The melt is contained in a crucible and is moved through a thermal gradient so that an isothermal freezing plane or interface will pass from one end of the crucible to the other. Many different crucible designs have been used but the main objective of all is nucleation in which one nucleus is favored over all others and grows to fill the crucible with a single crystal. Figure 4.4 a, b, c, and d shows some of the methods used to insure that only one crystal is formed. They all depend on the

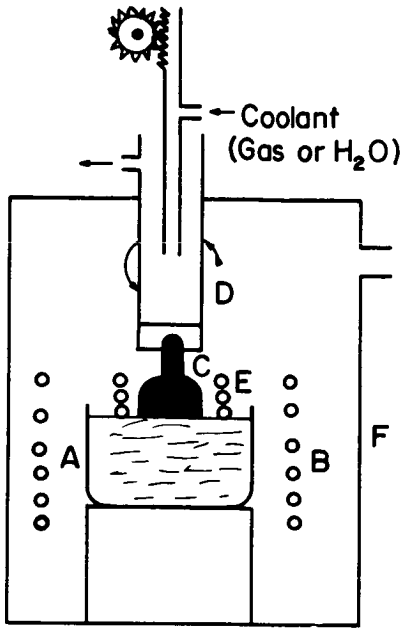


Fig. 4.3 Schematic drawing of a crystal puller

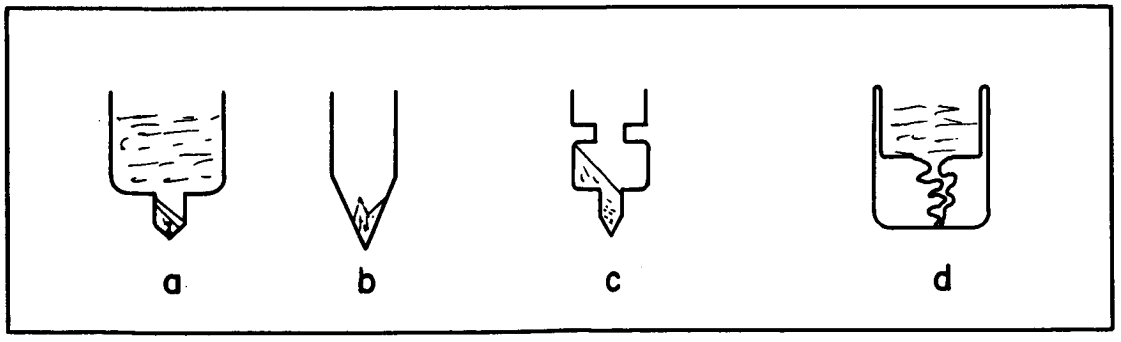


Fig. 4.4 Methods used to select a single nucleus

geometry at the lower end, favoring the selection of a single nucleus. An oriented seed can be provided if a special orientation is desired.

Figure 4.5 shows the method, as used by Stockbarger, to grow calcium fluoride. Two furnaces are placed one above the other, the upper one is kept at a temperature of 50 to 80°C above, and the lower one at 50 to 80°C below the melting point of the material being grown. The two furnaces are separated by a radiation shield to lower radiation along the furnace interior and to accentuate the temperature gradient. The hotter furnace, being at the top, reduces convection currents. The material in the crucible is melted in the upper furnace and then slowly lowered into the lower furnace. As the special tip passes through the isotherm corresponding to the liquidus of the melt, nucleation occurs. As described before, one nucleus is preferred and, as the crucible is lowered until completely in the lower furnace, a single crystal is grown that fills the crucible. The size and shape of the final product is controlled by the size and shape of the crucible used. The only temperature limitations of this method are those of the furnace and the crucible. Melts unstable in air can be grown in gas atmosphere or in a vacuum. The apparatus should be without thermal or mechanical fluctuations and the crystal melt interface must be planar (as in pulling).

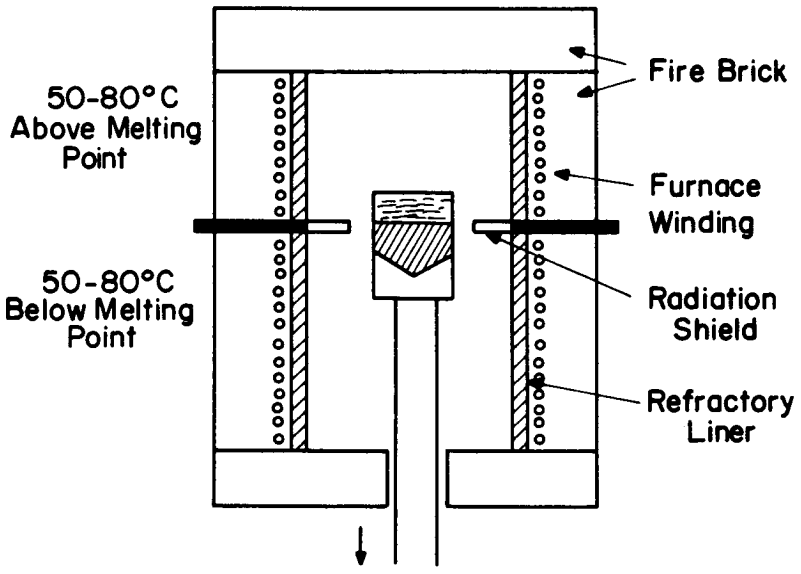


Fig. 4.5. The stockbarger furnace

The method of Stöber (Figure 4.6) is a slight variation of the above method. The furnace C is used to melt the material in the crucible. The heater D is used to set up a thermal gradient that is independent of the furnace. As the power to the furnace is decreased, the gradient will pass from the bottom to the top, and the crystal A will grow as the melt B solidifies.

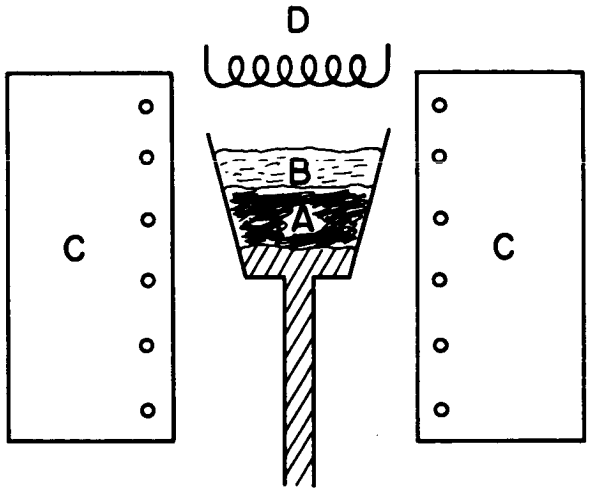


Fig. 4.6. The Stöber method

Flux Melt Method

This is a technique that has recently become very popular and has been applied to many materials. It is a simple, fast, and inexpensive method that uses standard equipment. It is very similar to growth from an aqueous solution, but here the solvent is made liquid by melting. Some of the materials that have been used as fluxes (solvents) are B_2O_3, V_2O_5, NaCl, KF, PbF_2, KCl, BiF_3, PbO, and Bi_2O_3.

Enough of the solvent to form a homogeneous melt is placed in a platinum crucible with the material to be grown, or its constituents. The material is melted and brought to a temperature of about 1000°C. This can be done easily in a conventional low-cost laboratory furnace. The melt is then cooled, the solution becomes supersaturated with respect to the high melting material, the one to be grown, and crystallization takes place. If cooling is slow, only a few nuclei will develop or a seed crystal maybe used. Crystallization is continued until all the solute is used up. This method has been used to grow beryl, ruby, ZnS, $BaTiO_3$, and ferrite garnets.

There are many modifications of the cruible growth method but the Stockbarger, Stöber, and flux method outline the main features.

4.4 Growth Without a Crucible

Flame Fusion

The flame fusion method was first developed by Verneuil in 1891, for the growth of synthetic gem corundum (ruby and sapphire). For about 50 years this process was limited to the production of corundum, and spinel. It has now been extended to other materials and in addition to corundum and spinel, rutile, mullite, and scheelite have been grown by this method.

The process consists of a feed device, gas control, properly housed burner, and a lowering device. A schematic drawing of a commonly used furnace is shown in Figure 4.7. In operation it consists of an inner tube carrying oxygen and the raw material, and an outer tube which carries hydrogen. Finely ground material is tapped at regular intervals, from a gauze or perforated bottomed powder container, into the oxygen stream and carried to the tip of burning oxyhydrogen torch. The tip of the oxyhydrogen flame is directed onto a refractory pedestal. The melted raw material forms a cone-shaped mound of sintered material on the pedestal. This is then melted in the flame. A single crystal is formed at the tip of the polycrystalline mass. The pedestal is slowly lowered and by careful control of the lowering rate, raw material feed, and temperature a single crystal boule will be produced. If a particular orientation is desired, a seed crystal is placed on the end of the pedestal and introduction into the flame and material is added as it is withdrawn.

The feed process is very critical and produces the chief difficulty. There seems to be more art than science in the Verneuil method. The technique is limited to materials that are not easily reduced and is chiefly applied to refractory materials with melting points between 1700 and 2500°C.

A recent improvement has been the addition of induction and arc image heating.

Zone Melting

The zone-melting method was introduced by Pfann in 1952, and is in wide use for the removal of impurities and the controlled addition of desired "doping" to a crystal. The technique can also be used for the production of a single crystal from polycrystalline raw material.

In the zone-melting method a small molten zone travels slowly through a solid charge, and, in traveling, redistributes the solutes of the charge. There are two liquid-solid interfaces, a melting interface and a freezing interface. The redistributions take place at the freezing interface. In operation (Figure 4.8), a single crystal seed is placed at one end of a quartz boat containing a polycrystalline charge. A small portion of the seed is melted in the zone, then the zone is moved toward the polycrystalline material and the seed is allowed to grow. If optimum conditions are maintained, little mechanical and thermal fluctuations, a planar crystal-melt interface, and a slow growth rate, a single crystal should be formed

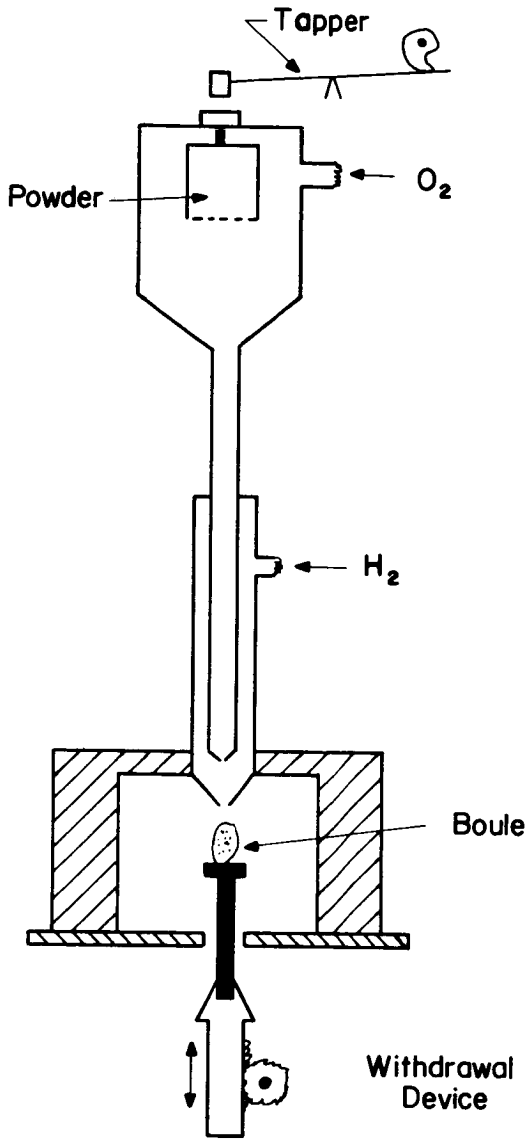


Fig. 4.7 Schematic of a Verneuil flame fusion furnace

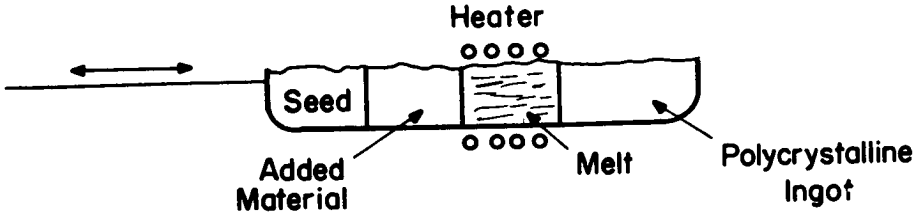


Fig. 4.8. Schematic of the zone metting method

the length of the boat. The number, size, travel direction, and the speed of the zone can be varied to produce the desired results. With close regulation of the variables it is possible to grow crystals of high perfection and uniform concentration. Rate of growth is usually in the range 0.1 to 2 millimeters per second. This method has been used in the growth of high quality crystals of germanium and other semiconductors.

Floating Zone

Zone melting can be carried out with a polycrystalline vertical ingot that acts as its own container. A polycrystalline rod (Figure 4.9) is supported vertically from above so as to touch a seed crystal rod that is supported vertically upward. The polycrystalline rod is melted onto the seed by moving a heating zone downward. The direction of the zone is reversed and the seed grows as the zone moves upward along the rod. The molten zone is held in place by its own surface tension between the two rods. The molten zone is obtained chiefly by induction heating although other methods have been used. This growth method is good for materials such as molten silicon which is highly reactive with crucibles. It also has been used for rhenium, aluminum, and tungsten.

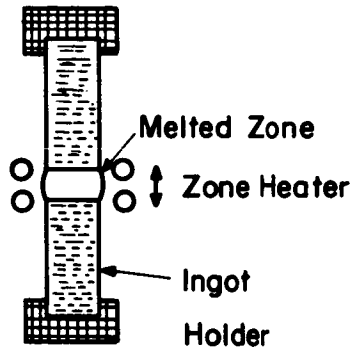


Fig. 4.9. The floating zone method

4.5 Growth from the Vapor

This method is applicable to many elements and compounds. The only requirement is that the substance have a high enough vapor pressure. Growth from the vapor is useful in growing sulfides, tellurides, and selinides and halides that have a high vapor pressure at temperatures below their melting points, and also for oxides that have melting points above 2500°C.

The method has not been used as much as the others discussed chiefly because the crystals produced are usually small. Its ability to produce crystals that cannot be grown by other methods have caused increased interest in this method. This interest and proper research should bring about improvement in the method that will make the growth of larger crystals possible.

The method consists of vaporization of a material and condensation in a thermal gradient into single crystal. Figure 4.10 shows a generalized diagram of the apparatus. The raw material is heated until

it vaporizes, and a carrier gas then carries it to the cooler part of the container where it is deposited. The two most important variables are the temperature of the vapor source and the temperature of the area when the crystal will grow. The first controls the vapor supply, and the temperature difference between the two controls the supersaturation at the growth area. The source of vapor is either from the volatilization of a pure substance or as a product of a chemical reaction in the gaseous state. An example of the latter is the reaction of Zn vapor with air to grow ZnO crystals.

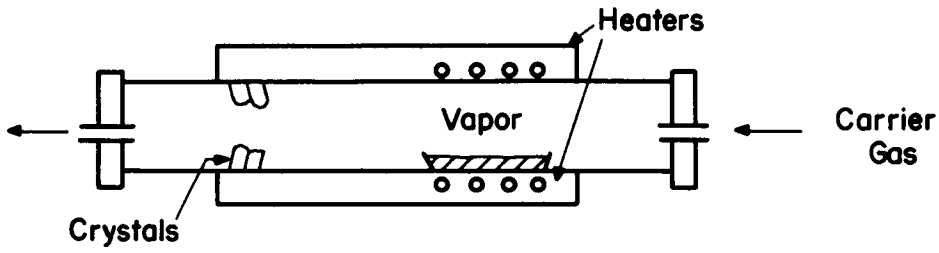


Fig. 4.10. Apparatus for growth from vapor

The rate of growth is intermediate between the more rapid growth from the melt and the slower growth from solution. Growth from the vapor has been used to grow crystals of ZnO, CdS, Mo, Zr, W, CdSe, CdTe, and whiskers of Fe, Cu, Ni, and Co.

We have considered the crystalline state and the growth of crystals and will now turn our attention to the glassy state.

V. GLASS

Glass is a very versatile material. It can be made to have many different properties. It can be colored or colorless, strong as steel or as fragile as an egg shell, lighter than a cork or as heavy as metal. In recent years, new and important uses have been made of glass, one of the newest has been as a laser material.

Glass is one of the oldest manufactured goods dating back to 3000 or 4000 B.C., yet the internal structure of glass is still not completely known and, in fact, there is still much controversy over details. It should be said, in all truthfulness, that glass making depends more on art than science. This is in part due to the fact that the "glassy state" has not received as much study as that of the solid state. The research has been more along the practical rather than the basic, and has been restricted to a few companies with little university or government interest.

Glassy, vitreous , and amorphous are terms used to describe this non-crystalline condition. In crystalline material, if the temperature is increased, a point is reached where the energy becomes great enough to break bonds and the material melts; the temperature remaining constant until all the solid becomes liquid. In cooling the melt, these phase changes occur in reverse, liquid to solid. In glass the behavior is quite different; as the temperature is increased, a point is reached where the rigid glass will start to soften and will continue to soften over a range of temperature until it becomes a viscous liquid. When a glass melt is cooled slowly, it passes through a temperature range where the liquid becomes an unstable phase and a crystalline phase or phases appear. The appearance of the crystalline phase is at the liquidus temperature, at which time the melt is quite viscous, that is, the atoms have low mobility. If the tendency toward crystallization is great enough to overcome the low mobility and a crystalline material is produced, this is crystallized or devitrified glass. If, however, the melt is cooled rapidly to below the liquidus temperature, the low mobility of the atoms overcomes the tendency to crystallize, devitrification is not possible, and a glass is produced.

X-ray photographs of glasses show that the atomic arrangement exhibit the same randomness in the solid state as in the liquid state. Glasses are thus sometimes called "supercooled liquids," because the structure of the glass is only slightly changed from that of the liquid.

5.1 Glass Structure

The foundation of our present understanding of the structure of glass was proposed by Zachariasen in 1932. He wrote a single, short paper (12 pages) on the subject. He suggested that glass is composed of three-dimensional random networks of polyhedra (tetrahedra or triangles) of oxygen ions, at the center of which are ions such as Si^{4+}, P^{5+}, B^{3+}, or V^{5+}. These ions and the oxygen form the network and are thus called network formers. The oxygen ions are of two types, those which belong to two

polyhedra and are called bridging oxygens, and the non-bridging oxygens, each of which belong to a single polyhedron. This forms a very open structure which has an excess negative charge which is balanced by ions such as Ca, Pb, Na, located in the interstices of the network and called "network modifiers." Actually, the presence of these ions modify, to some extent, the form that the network assumes. Figure 5.1 is a two-dimensional representation of glass structure as Zachariasen saw it.

The cohesion of the network depends on how open the structure is, thus on the percentage of bridging oxygen compared to the non-bridging oxygen. Even if the cohesion is good, the network lacks periodicity (at least long range periodicity). Zachariasen felt that glass softens rather than melts because of its asymmetry , i.e., all the bonds are of slightly different strength.

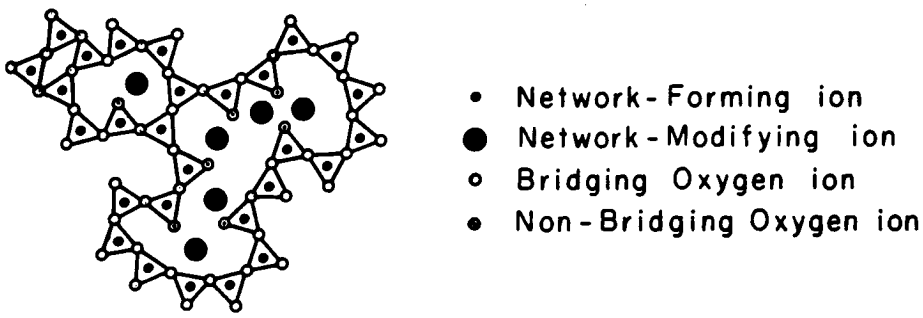


Fig. 5.1. Internal structure of glass

Before Zachariasen's work, glass experts generally regarded glass as a supercooled solution of various silicate molecules in silica. In 1921, A. A. Libedev had proposed the theory that glass might consist of a mass of very small crystals called crystallites. The crystallite theory of glass structure still receives strong support from some Russian investigators. Much attention is being given to the structure of glass by the Russians. All-Union Conferences on the Glassy State were held in 1953 and 1959. These meetings produced two large volumes of the proceedings of the meetings which have been translated into English. At both of these conferences, Lebedev spoke in favor of his theory and received strong support from quite a few other Russian workers. Porai-Koshits in his paper at the Leningrad Symposium in 1953, states that the controversy

is purely a terminological argument and is useless. And he feels that the term "polymer-crystallite structure" effectively conveys the significance of a unified viewpoint. This does take into consideration the work of Zachariasen, for when he proposed that glass was composed of three-dimensional random networks of alternate atoms of Si and O joined in long and cross-linked chains, he was proposing that glass was an inorganic polymer.

Zachariasen also gave a set of successful rules for predicting whether or not a substance would form a glass, much like Pauling's rule for ionic crystals. Zachariasen discovered that the general formulation of the characteristics which the structure of an oxide must possess in order to be able to form a glass is reduced to a geometric problem. This is complicated since it must be treated in three dimensions, but he was able to propose the following rules.

1. Every oxygen ion must be bound to not more than two positive ions, which must be highly charged and small.

2. The number of oxygen ions which surround such positive ions may not be too large or too small (3 or 4).

3. The ion groups adjacent to each other may have common carriers but no common edges or faces.

4. There must be ion groups which have at least three oxygen ions in common with neighboring ion groups.

Every oxide which satisfies these rules is able to form a glass. But more recently inorganic systems have been found which do not obey these rules and occur in glassy state. This failure and other shortcomings of Zachariasen's theory has caused it to undergo almost constant modification and improvements. Weyl and others have shown the importance of coordination and the deformation of the anions by the cations. Another effect of coordination can be studied by the "competition principle," this is a measure of the bonding strength and coordinations and is based on the "field strength" (F.S.).

$$F.S. = \frac{Z}{a^2}$$

Z = charge on ion

a = distance between the centers of adjacent ions

When different ions occur in a glass, those with the highest $\frac{Z}{a^2}$ value will be the network-forming ions (smallest coordination number) and those with the lowest $\frac{Z}{a^2}$ value will have preference for the network modifying positions (largest coordination number). This points up the fact that the network formers will be small highly charged ions.

Another modification that Zachariasen's theory has undergone is the consideration of the effect of bond nature. Zachariasen never considered the nature of the bond and regarded the polyhedrons to be invariable. Smekal

and other investigators have pointed out the importance of bond type to the structure of the glassy state. More will be said about nature of the bond later.

Zachariasen thought that all positive ions could be divided into two sharply defined groups, network formers and network modifiers. More recent studies have shown that three groups exist, network formers, intermediates, and network modifiers.

Table 5.1 The Classification of Some Oxides Used in Glasses

Formers	Intermediates	Modifiers
B_2O_3	AL_2O_3	MgO
SiO_2	Sb_2O_3	Li_2O
GeO_2	ZrO_2	BaO
P_2O_5	TiO_2	CaO
V_2O_5	PbO	SrO
As_2O_3	BeO	Na_2O
	ZnO	K_2O

The intermediates approach network-forming proportion but do not quite make it. But no sharp line exists between classes, some intermediates do on occasion act as network formers in some compositions and as a modifier in others. For example, in some silicate glasses that contain large amounts of Al_2O_3, at least in part, it acts as a network former.

Table 5.2 shows the electronegativity difference and ions character of bonds common to glasses. It will be seen that the network formers have low electronegativity differences and low ionic character. Modifying ions have large values for both and the intermediates have value in between. Thus glass formers show a high degree of covalent character but there seems to be a lower limit, for if the C-O is considered, a value of 20 per cent ionic bonding is found, but this is not a network former.

As useful as Zachariasen's theory and its modification have been, some questions still exist and controversy is still present about the structure of glass. It is for this reason that glassmaking is still as much art as science. For a general discussion of the present status of the structure of glass, the comprehensive review by Porai-Koshits and Andreev (1959) or the first chapter of Mackenzie (1960) should be consulted.

5.2 Chemistry of Glass

Glass of various compositions can be made, in fact many thousands of different glass compositions have been used, but only a few are common or important. These are outlined below.

Silica Glass is called fused silica, vitreous silica, or just silica glass, has low thermal expansion, therefore, high resistance to breakage from

Table 5.2 The Nature of Common Glass Bonds

Bond	Electronegativity Difference	Approximate % Ionic Character
B-O	1.5	42
As-O	1.5	42
Si-O	1.7	50
Sb-O	1.7	50
Ge-O	1.8	55
Sn-O	1.8	55
Ti-O	1.9	56
Zr-O	1.9	56
Be-O	2.0	60
Al-O	2.0	60
Y-O	2.2	65
Se-O	2.2	65
Mg-O	2.3	70
Ca-O	2.5	75
Sr-O	2.5	75
Li-O	2.5	75
Ba-O	2.6	80
Na-O	2.6	80
Rb-O	2.7	81
K-O	2.7	81
Cs-O	2.7	82

thermal shock. It resists deformation to temperatures of 1000°C and has good chemical durability but is hard to fabricate. It is a good transmitter of visible and ultraviolet radiation but shows strong absorption in the infrared.

Soda-Lime-Silica Glass is composed of approximately 75% SiO_2, 15% alkaline oxide and 10% CaO or similar oxide. The alkaline oxides are Li_2O, K_2O, and Na_2O or a combination, the other oxides are CaO, MgO, BaO, and ZnO or a combination. These are called lime, soda-lime or soda-lime-silica glasses. They are used chiefly for sheet glass.

Lead-Alkali Silicate Glasses. In soda-lime-silica glasses when the CaO content exceeds about 15 per cent the glass is difficult to melt and devitrifies easily. If PbO replaces the CaO this is overcome and a new series of glasses are produced. These glasses have very favorable optical and electrical properties. They have high index of refraction and high dispersion and are used as radiation shields.

Aluminosilicate Glasses. Silica is still the major oxide of these glasses, (about 60 per cent) but they contain about 20 percent Al_2O_3 and lesser amounts of the glass-forming P_2O_5 or B_2O_3. They have a low coefficient of expansion and thus good thermal shock resistance as well as high deformation temperatures.

Borosilicate Glasses. The best known member of this family of glasses is Pyrex. Silica is still the major oxide of these glasses but they contain about 15-30 per cent B_2O_3 and only small amounts of alkali and Al_2O_3. In these glasses B_2O_3 serves as both a glass-former and as a flux for the SiO_2. These glasses have great resistance to thermal shock, are chemically stable, and have good electrical properties.

Borate Glasses contains little or no SiO_2, about 40 per cent B_2O_3, and 20 to 30 per cent BaO plus a combination of other oxides. For example, for use in sodium vapor lamps, in addition to the above, the glasses also contains about 30 per cent Al_2O_3 and about 10 per cent MgO. Another borate glass containing large amounts of lanthanum will be described later, under the heading Optical Glasses.

Phosphate Glasses. A group of glasses in which P_2O_5 is the chief or only glass-forming oxide.

Colored Glasses. These are normal glasses which contain colored ions (transition group elements). A comprehensive test on the chemistry and properties of colored glasses is offered by Weyl. Some of the ionic colors produced in glasses are listed in Table 5.3.

Optical Glasses. These are glasses which have very good homogeneity, accurately defined and uniform values of dispersion and index of refraction. Index variation is usually about $\pm$ 0.001 to $\pm$ 0.003. They also usually have maximum transparency, chemical stability, and controlled coefficient of expansion. Many have special properties such as radiation resistance, infrared transmission, etc.

Of possible interest in laser research are the glasses developed by G. W. Morey which contain large amounts of lanthanum oxide, with substantial quantities of boron, thorium, tantalum, tungsten and zirconium oxides but little or no silica. Eastman Kodak manufactures these rare element glasses (see Eastman Kodak, 1958).

For a detailed discussion of optical glasses see Morey (1954).

Table 5.3 Ionic Color in Glass

Ion	Color
Fe^{+++}	green
Mn^{+++}	purple
Co^{++}	blue and pink
Ni^{++}	red
Cu^{++}	greenish blue
Cr^{+++}	green
U^{6+}	green
U^{4+}	green
V^{+++}	green
W^{6+}	blue
Mo^{6+}	green
Nd^{+++}	purple
Pr^{+++}	green
Ti^{4+}	yellow
Ce^{+++}	brown

5.3 Optical Properties of Glass

The optical properties of glass have been thoroughly studied and hundreds of glasses are available, both clear and colored. It is possible to vary the optical properties, and many glasses exist that have very special optical properties. General references would include Morey, 1954, and Shand, 1958, and the literature of the glass-producing companies.

Refractive Index.--Ranges from 1.458 for silica glass to over 2.00 for very dense optical glasses. The related dielectric constant varies from about 3.7 for silica glass, to a maximum of 16.0 or more for high lead glasses.

Absorption and Transmission. Absorption varies greatly from one wavelength to another; in colored glasses the selective absorption is responsible for the color. Good colorless glasses show small but quite uniform absorption for all wavelengths between 4000 and 75,000 Å. The best optical glasses have a 99 per cent transparency throughout the visible spectrum, as a result of the great care taken to exclude impurities, especially iron. Common window glass has a transparency of from 85 to 90 per cent in the visible range.

Most of the absorption of glasses in the infrared region is related to vibrations of the constituent elements in the lattice. All silicate glasses, colored or colorless, begin to show strong absorption between four and five microns and transmit little beyond this. An absorption edge not far beyond three microns, is attributed to certain vibrations involving silicon and oxygen. A treatment of these vibrations is to be found in a chapter by Simon in Mackenzie's (1960) book. FeO also shows strong absorption in the infrared about one micron.

In general, the absorption bands of potassium glasses are sharper than sodium glasses and phosphate glasses show sharper bands than silicate glasses. Rare earth ions in all show the usual sharp absorption bands characteristic of them.

Fluorescence. Weyl in his book, devotes several complete chapters to fluorescence in glasses. The fluorescent ions discussed include: Pr, Nd, Eu, Sm, Ce, Mn, U in great detail, Tl, Sn, Pb, and V.

A maximum of fluorescence usually accompanies additions below one per cent, further additions causing interference between the fluorescing systems themselves. For example, silicate glasses containing more than 10 per cent Nd^{+++} oxide do not fluoresce. Small amounts of iron also frequently "quench" fluorescence.

We have considered the nature of solid materials that might contain laser ions, both crystalline and non-crystalline. Next, we will consider briefly the nature of some of the laser ions.

VI. IONS OF INTEREST

We have taken a brief look at the nature of crystals and glasses which act as carriers of laser ions, but what about the nature of these ions? A basic discussion will be given of rare earth elements and to a lesser extent the actinide elements. This will be done because in general, these groups are less well known than other transition groups and also because as laser ions the rare earths are the most interesting.

6.1 Rare Earth Elements

What is the rare earth series? This is not a simple question, because a problem in nomenclature exists. The most widely accepted use of the term rare earth series, or lanthanide series, is for the elements lanthanum to lutetium (57-71). Some chemists include yttrium (39) or both yttrium and scandium (21) because of their close chemical similarity to other members of the series. Vickery has proposed the use of lanthanons for elements (57-71) and of rare earths for elements No. 57-71 plus 21 and 39. We shall consider the rare earths as La (57) plus the elements where the 4f orbitals are being filled Ce (58) to Lu (71).

The rare earths are a group of elements that in the periodic table are usually set off by themselves because they interfere with the regular nature of the sixth period. The elements are included as a group occupying the position of a single element in Group III of the sixth period. This sort of arrangement is made so that elements that are similar, such as zirconium and hafnium, will fall under each other in the periodic table. They are actually a transition group occurring at the start of the third transition group. Besides this uniqueness of placement in the periodic chart, the rare earths are also unique in their electronic configuration (except for the Actinides). This leads to some very interesting chemical and spectral properties.

Electronic Configuration

As shown in Table 6.1, the idealized configuration of the rare earths is: $(1s)^2(2s)^2 \ldots(4f)^x (5s)^2 (5p)^6 5d (6s)^2$ where x is 0 to 14.

The electrons are added to the well shielded 4f shell. There are seven 4f orbitals, each with a capacity of two electrons. The probable configuration is also presented in Table 6.1. It shows that the single 5d electron has a tendency to drop to the 4f level. If this is true and we limit the term rare earth to those atoms whose configurations have an incomplete 4f shell, lanthanum and lutetium would be excluded. However, because of the small chemical difference caused by the energy difference between a 4f and 5d electron and the close chemical similarities of these and the other rare earth elements, it is best to consider elements 57 to 71 as a group.

Oxidation State and Ionic Size

The characteristic oxidation state of the rare earth elements is 3+, having lost (if present) 5d, and the two 6s electrons and certain 4f

Table 6.1 Some Properties of the Rare Earths

Element	Symbol	Atomic Number	Electronic Configration						Oxidation States	The Trivalent Ion				Ionic Radii (Å)		
			Idealized			Probable				Unpaired Electrons	Magnetic Properties	Ground State	Color	Valence state		
														2+	3+	4+
Lanthanum	La	57		$5d^1$	$6s^2$		$5d^1$	$6s^2$	3	0	Dia-	$^1S_0$	Colorless		1.04	
Cerium	Ce	58	$4f^1$	$5d^1$	$6s^2$	$4f^2$		$6s^2$	3,4	1	Para-	$^2F_{5/2}$	Colorless		1.02	0.92
Praseodymium	Pr	59	$4f^2$	$5d^1$	$6s^2$	$4f^3$		$6s^2$	3,4	2	Para-	$^3H_4$	Green		1.00	0.90
Neodymium	Nd	60	$4f^3$	$5d^1$	$6s^2$	$4f^4$		$6s^2$	3	3	Para-	$^4I_{9/2}$	Violet Red		0.99	
Promethium	Pm	61	$4f^4$	$5d^1$	$6s^2$	$4f^5$		$6s^2$	3	4	Para-	$^5I_4$	Pink; Yellow		0.98	
Samarium	Sm	62	$4f^5$	$5d^1$	$6s^2$	$4f^6$		$6s^2$	2,3	5	Para-	$^6H_{5/2}$	Yellow	1.11	0.97	
Europium	Eu	63	$4f^6$	$5d^1$	$6s^2$	$4f^7$		$6s^2$	2,3	6	Para-	$^7F_0$	Almost Colorless (Pink)	1.09	0.96	
Gadolinium	Gd	64	$4f^7$	$5d^1$	$6s^2$	$4f^7$	$5d^1$	$6s^2$	3	7	Para-	$^8S_{7/2}$	Colorless		0.94	
Terbium	Tb	65	$4f^8$	$5d^1$	$6s^2$	$4f^9$		$6s^2$	3,4	6	Para-	$^7F_6$	Almost Colorless (Pink)		0.92	0.84
Dysprosium	Dy	66	$4f^9$	$5d^1$	$6s^2$	$4f^{10}$		$6s^2$	3	5	Para-	$^6H_{15/2}$	Yellow		0.91	
Holmium	Ho	67	$4f^{10}$	$5d^1$	$6s^2$	$4f^{11}$		$6s^2$	3	4	Para-	$^5I_8$	Pink; Yellow		0.89	
Erbium	Er	68	$4f^{11}$	$5d^1$	$6s^2$	$4f^{12}$		$6s^2$	3	3	Para-	$^4I_{15/2}$	Rose		0.87	
Thulium	Tm	69	$4f^{12}$	$5d^1$	$6s^2$	$4f^{13}$		$6s^2$	2,3	2	Para-	$^3H_6$	Green	0.93	0.86	
Ytterbium	Yb	70	$4f^{13}$	$5d^1$	$6s^2$	$4f^{14}$		$6s^2$	2,3	1	Para-	$^2F_{7/2}$	Colorless		0.85	
Lutetium	Lu	71	$4f^{14}$	$5d^1$	$6s^2$	$4f^{14}$	$5d^1$	$6s^2$	3	0	Dia-	$^1S_0$	Colorless		0.84	

electrons. Thus, as Table 6.1 shows, the 3+ ions do show a regular addition of an electron to the 4f shell between element 57 and 71. As Table 6.1 indicates, both 2+ and 4+ ions are known, but these are not as stable and are less common. Rare earth ions of the valence of both 2+ and 3+ have shown laser actions.

Within a normal period of the periodic table an increase in atomic number is paralleled by an increase in atomic size. This is because the addition of electrons produces an enlarging effect greater than the contracting effect of the increased nuclear charge. In the rare earths the added 4f electrons are so effectively masked, by the overlying higher energy subshells, that it cannot balance the increase in nuclear charge, and the size of the atom and therefore the 3+ ion decreases in going from La to Lu. This decrease is called the lanthanide contraction, and it has considerable effect in determining the ability of elements to substitute for one another. It causes such anomalies in size as Zr (atomic weight 91) having almost the same radius as Hf (at. wt. 178.6) and the fact that in nature these two substitute for one another freely. The size of the 3+ ion is rather similar throughout the rare earth series, Table 6.1 (after Zachariasen, 1954), about a 15 per cent variation, and this is the reason for the fact that all rare earths form similar compounds. It was also this fact that made separation of rare earths so hard and made pure rare earth compounds expensive and rare for so long.

A consideration of ionic size would seem to indicate that Y^{+3} (.92) and Bi^{+3} (.93) would be the most promising for which a rare earth might substitute. The most common substitution in nature is for Ca^{++} (.99) this is probably, at least in part, due to the fact that Ca is very abundant in nature. But another possibility is Sr^{++} (1.12), with Ca being best for the smaller rare earth and Sr for the larger, but in both cases something must be done to balance the charge. Another place that substitution can and does take place is for the actinides and this will be discussed in more detail later.

Spectra

The 4f filling which produced such a small chemical effect in the rare earths, plays a large part in determining energy states and thus the spectra. Characteristic of most rare earth ions is the color that it has in liquids, solids, or glasses. The characteristic colors of all the 3+ rare earth ions are given in Table 6.1 and those in other oxidation states in Table 6.2

Table 6.2 Color of Nontripositive Ion

Ce^{++++}	Orange-red
Pr^{++++}	Black (in Solid PrO_2)
Sm^{++}	Deep red-brown
Eu^{++}	Pale yellow
Tb^{++++}	Brown (in Solid TbO_2)
Yb^{++}	Green

Absorption Spectra

Table 6.3 shows the significant absorption of various 3+ valent rare earth ions. It is interesting to note that in the rare earths absorption spectra shifts toward the ultraviolet until gadolinum is reached, then the trend is reversed.

Table 6.3 Significant Absorption Bands in the Range 2,000 - 10,000 Å

3+ Ion	Absorption	Wavelength, Å
La	None	
Ce	Ultraviolet	2105, 2220, 2380, 2520
Pr	Visible	4445, 4690, 4822, 5885
Nd	Ultraviolet-visible-near infrared	3540, 5218, 5745, 7395, 7420, 7975, 8030, 8680
Pm	Visible-near infrared	5485, 5680, 7025, 7355
Sm	Ultraviolet-visible-near infrared	3625, 3745, 4020, 9550
Eu	Ultraviolet	3755, 3941
Gd	Ultraviolet	2729, 2733, 2754, 2756
Tb	Ultraviolet-visible	3694, 3780, 4875
Dy	Ultraviolet-near infrared	3504, 3650, 9100
Ho	Ultraviolet-visible	2870, 3611, 4508, 5370, 6404
Er	Ultraviolet-visible-near infrared	3642, 3792, 4870, 5228, 6525, 6820, 7800, 9725
Tm	Ultraviolet-visible-near infrared	3600, 6825, 7800
Yb	Near-infrared	9750
Lu	None	

The absorption spectra of the rare earths is characterized by a narrow sharp well-defined absorption band. This is in contrast to the usual transition metal ion spectra, which show broad absorption bands. The only other ions with such linelike absorption bands are the actinides.

As for the nontrivalent ions, Sm^{++}, Eu^{++}, Yb^{++}, all show intense absorption in the ultraviolet, Ce^{++++} shows broad absorption in the blue and ultraviolet, and Pr^{++++} and Tb^{++++} show strong absorption in the visible and ultraviolet. The divalent rare earth ions show very strong absorption bands above the fluorescent metastable transition and are very suitable for laser materials.

The complexity and sharpness of the absorption bands arise from pure electronic transitions involving the 4f subshell and the fact that since the 5s and 5p outer shells protect the 4f electronic energy states from

major perturbation by ionic fields outside the atomic system. This shielding is sufficient to reduce the crystalline stark splitting of the free ion terms to hundreds of cm^{-1} from the thousands of cm^{-1} common in other transition groups. Within the group, the stark splitting is an inverse function of (x) dimirishing from about $600cm^{-1}$ for Ce^{+++} (x-1) to about $100cm^{-1}$ for Yb^{+++} (x=13).

The absorption and also fluorescence spectra of rare earths contain lines of four main types (Yost, Russel, Garner, 1947).

1. Intense, sharp lines in widely separated groups.
2. Weak, but still sharp lines, usually occurring as satellites on the violet side of lines of the first type.
3. Weak, diffuse usually lying on the violet side of the first type also.
4. Strong, difiuse bands in the ultraviolet that show no structure.

Prandt and Scheiner (1934) gave a description of the spectra over the range 2000 to 8000 Å. Moeller and Brantley extended the range to 10,000 Å, and in a more recent study Stewart and Kato (1958) restudied the visible range. Some of the most recent studies have been those of Dieke and his fellow workers at John Hopkins and Van Uitert and his fellow workers at Bell Laboratories.

Fluorescence Spectra

The fluorescence of materials containing rare earths has long been known and used. But it was not until the work of Tomaschek and Deutschbein (1933) that the fluorescence of pure salts of rare earths and their aqueous solutions were discovered. Much of the information about the fluoresence spectra of rare earths is to be found in the works of Tomaschek and Gobrecht. The same papers also contain much of the early work on the energy levels of rare earths. An interesting fact about the fluorescence of pure salt is that it takes place only in Sm, Eu, Gd, Tb, and Dy. Here again the work of Dieke and his coworkers and Van Uitert and his coworkers should be noted.

The fluorescence yield of materials containing rare earths, glasses, crystals, liquid solutions, and solid solutions are high because of the protection against perturbation of the 4f electron. The yield approaches 100% even in liquid solutions.

Kröger (1948) contains tables of the colors of luminescence produced by rare earths in various materials with references to the orginal work. This is briefly summarized in Table 6.4 without regard to material.

Optical Emission

The emission spectra of the rare earths are highly complex with thousands of lines of nearly the same intensity and lacking the characteristics intensity of lines found in the emission spectra of other elements. Only

Table 6.4 Color of Luminescence
(Assorted Media)

Ce^{3+}	violet - ultraviolet
Er^{3+}	blue + green, green
Eu^{3+}	orange + infrared, red
Eu^{2+}	blue, green
Nd^{3+}	blue + green, blue + green + infrared, green
Pr^{3+}	green + red, green - infrared
Sm^{3+}	orange, orange + infrared
Sm^{2+}	red
Tb^{3+}	ultraviolet - red, green + red, blue green - infrared
Tu^{2+}	red
Tu^{3+}	blue, blue - infrared, ultraviolet - infrared
Yb^{2+}	yellow greenish, infrared, orange - red

La, Eu, and Tm have simple spectra. The reason for the usual emission spectra is again due to the 4f filling. Table 6.5 presents the most persistent lines in the emission spectra of each rare earth. Norris (1961) gives a complete listing of the emission lines of the rare earths.

Table 6.6 contains a list of rare earth minerals and other minerals that contain rare earths in lesser amounts.

6.2 Actinide Elements

The actinide elements are a second rare earth series, in which the 5f electron shell is filled, beginning with actinium (89) and going through element 103. This group includes the three natural elements thorium, protactinum, uranium, and the synthetic transuranium elements.

The oxidation states of the actinides are listed in Table 6.7. The greater number of oxidation state, than in the rare earths, is due to the weaker binding of the 5f than the 4f electrons.

The absorption spectra of the actinides is much like that of the rare earth elements, containing a complex spectra with narrow bands in the visible, near ultraviolet and near infrared regions. The narrow bands, as in the rare earths, is attributed to electronic transitions in an inner shell, the 5f for the actinides. The spectra is actually ten times more intense than that of the rare earths because of the reduced shielding of the 5f electrons. Table 6.8 shows the common ionic types and their color in aqueous solutions; a blank means the state does not exist in aqueous solution.

Table 6.5 Most Persistent Lines of Rare Earth Emission Spectra (in Å)

Ce	3942	Y	3242	Tm	3362
	4012		3327		3425
	4040		3600		3462
	4133		3710		3717
	4186		3774		3795
	4572		4102		3848
Sm	3568	Pr	3908	Lu	2615
	3592		4100		2911
	3609		4179		3077
	3634		4222		3359
	3885		4225		3397
	4424		4408		3507
Tb	3324	Eu	2727	La	3337
	3509		3819		3790
	3561		3930		3794
	3585		3971		3949
	3676		4129		3988
	3874		4205		4086
Er	3372	Dy	3407	Nd	4012
	3499		3531		4061
	3692		3645		4109
	3906		3944		4156
	4007		4000		4303
	4419		4211		4325
6d	3350	Ho	3398	Yb	3107
	3362		3456		3289
	3422		3796		3694
	3646		3891		3987
	3671		4053		5356
	3768		4103		7699

Table 6.6 Minerals that Contain Rare Earths

Mineral	Formula
Parisite	$Ca(Ce,La)_2(CO_3)_3F_2$
Cordylite	$Ba_2(Ce,La)_3(CO_3)_5F_3$
Bastnaesite	$(La,Ce)FCO_3$
Cerite	$(Ce,Ca)_2SiO_5$
Lanthanite	$(La,Ce)_2(CO_3)_3 \cdot 9H_2O$
Allanite	$(Ca,Fe^{++})_3(R^*,Al,Fe^{+++})_4Si_4O_{16}(OH)_2$
Beckelite	$Ca_3(Ce,La)_4Si_3O_{15}$
Chevkinite	$(Fe^{++},Ca)(Ce,La)_2(Si,Ti)_3O_{10}$
Hellandite	$Ca_2(R^*,Al,Fe,Mn)_6Si_4O_{19}$
Monazite	$(Ce,La,Y,Th)PO_4$
Aeschynite	$(Ce,Th)(Cb,Ti)_2(O,OH)_6$
Gadolinite	$Be_2Fe(YO)_2(SiO_4)_2$
Yttrialite	$(Yt,Gd)_2Si_2O_7 + ThO_2$
Rowlandite	$Yt_4Si_3O_{12}$
Thalenite	$Yt_2Si_2O_7$
Keilhanite	$(Ca,Ti,Al_2Fe_2R_2^*)SiO_5$
Yttrocrasite	$Yt_2Ti_4O_{11}$
Delorenzite	$Fe_2YtUTi_{24}O_{58}$
Xenotime	YPO_4
Yttrofluorite	$CaF_2 - YF_3$
Yttrocerite	$CaF_2 - (Y,Ce)F_3$
Samarskite	$(Yt,U,Fe,Th)(Cb,Ta)_2O_6$
Fergusonite	$(Yt,Er)(Cb,Ta)O_4$
Euxenite	$(Yt,Er,Ce,La,U)(Cb,Ti,Ta)_2(O,OH)_6$

Minerals that Contain Appreciable Amounts of Rare Earths	
Baddeleyite	ZrO_2
Pitchblende	UO_2
Columbite	$(Fe,Mn)Nb_2O_6$
Ilmenite	$FeTiO_3$
Rutile	TiO_2
Tantalite	$(Fe,Mn)Ta_2O_6$
Thorite	$ThSiO_4$
Thorianite	ThO_2
Titanite	$CaTiSiO_5$
Zircon	$ZrSiO_4$

(R^* = Rare Earths)

Table 6.6 (Continued)

Mineral	Formula
Calcite	$CaCO_3$
Apatite	$Ca_4(CaF)(PO_4)_3$
Fluorspar	CaF_2
Cassiterite	SnO_2
Scheelite	$CaWO_4$
Wolframite	$(Fe,Mn)WO_4$
Powellite	$CaMoO_4$
Zirkelite	$(Ca,Fe)(Zr,Ti)_2O_5$

Table 6.7 Oxidation States of Actinides
(see Asprey and Cunningham, 1960)

Atom Number	Element	Oxidation States
89	Actinium	3
90	Thorium	(2) 3 4
91	Protactinium	3 4 5
92	Uranium	3 4 5 6
93	Neptunium	3 4 5 6
94	Plutonium	3 4 5 6
95	Americium	(2) 3 4 5 6
96	Curium	3 4
97	Berkelium	3 4
98	Californium	3
99	Einsteinium	3
100	Fermium	3

Table 6.8 Color of Actinidic Ions
(from Seaborg, 1958)

	A^{+++}	A^{++++}	AO_2^{+}	AO_2^{++}
Ac	--	--	--	--
Th	--	colorless	--	--
Pa	--	colorless	colorless	--
U	red	green	?	yellow
Np	blue-purple	yellow-green	green	pink to red
Pu	blue-violet	orange-brown	purple	yellow to orange
Am	pink	--	yellow	tan
Cm	colorless	--	--	--

Fluorescence in actinide compounds can generally be observed if properly excited, and the fluorescence of uranium is well known and much used.

The chemical properties of the actinides and their compounds are similar to those of the rare earths. Some of the actinide compounds are presented in Table 6.9. It is also interesting to note that an actinide contraction also exists. (Table 6.10) An effect of this contraction is that, in general, the size of the actinide ions are near those of the rare earths and may substitute for them and the elements for which they substitute. (See Table 6.11)

Table 6.9 Properties and Crystal Structure Data for Some Important Actinide Compounds (after Seaborg, 1958)

Compound	Color	Symmetry	Space Group or Structure Type
ThH_2	Black	Tetragonal	ThD_2
Th_4H_{15}	Black	Cubic	
PaH_3	Black	Cubic	
β-UH_3	Black	Cubic	
Np_4H_{15}	Black		
PuH_2	Black	Cubic	Fm_3m
PuH_3	Black	Hexagonal	$P6_3/mmc$
AmH_2	Black	Cubic	
Am_4H_{15}	Black	Cubic	
Ac_2O_3	White	Hexagonal	La_2O_3
Pu_2O_3		Hexagonal	La_2O_3
Am_2O_3	Tan	Hexagonal	La_2O_3
Cm_2O_3	White	Cubic	
ThO_2	White	Cubic	Fluorite
PaO_2	Black	Cubic	Fluorite
UO_2	Brown to black	Cubic	Fluorite
NpO_2	Apple green	Cubic	Fluorite
PuO_2	Yellow-green to brown	Cubic	Fluorite
AmO_2	Black	Cubic	Fluorite
CmO_2	Black	Cubic	Fluorite
AcF_3	White	Hexagonal	LaF_3
UF_3	Black	Hexagonal	LaF_3
NpF_3	Purple or black	Hexagonal	LaF_3
PuF_3	Purple	Hexagonal	LaF_3
AmF_3	Pink	Hexagonal	LaF_3
CmF_3		Hexagonal	LaF_3
$AcCl_3$	White	Hexagonal	UCl_3
UCl_3	Red	Hexagonal	$C6_3/m$
$NpCl_3$	White	Hexagonal	$C6_3/m$
$PuCl_3$	Emerald green	Hexagonal	$C6_3/m$
$AmCl_3$	Pink	Hexagonal	$C6_3/m$
$AcBr_3$	White	Hexagonal	$C6_3/m$
UBr_3	Red	Hexagonal	$C6_3/m$
α-$NpBr_3$	Green	Hexagonal	$C6_3/m$
β-$NpBr_3$	Green	Orthorhombic	Ccmm
$PuBr_3$	Green	Orthorhombic	Ccmm
$AmBr_3$	White	Orthorhombic	Ccmm
ThF_4	White	Monoclinic	UF_4
PaF_4	Reddish brown	Monoclinic	UF_4
UF_4	Green	Monoclinic	C2/c
NpF_4	Green	Monoclinic	UF_4
PuF_4	Brown	Monoclinic	UF_4
AmF_4	Tan	Monoclinic	UF_4

Table 6.9 (Cont.)

Compound	Color	Symmetry	Space Group or Structure Type
CmF_4		Monoclinic	UF_4
$ThCl_4$	White	Tetragonal	I4/amd
$PaCl_4$	Greenish yellow	Tetragonal	I4/amd
UCl_4	Green	Tetragonal	I4/amd
$NpCl_4$	Red-brown	Tetragonal	I4/amd
UF_6	White	Orthorhombic	Pnma
NpF_6	Brown	Orthorhombic	Pnma
PuF_6	Reddish brown	Orthorhombic	

Table 6.10 Ionic Radii of the Actinidies (after Zachariasen, 1954)

Atomic Number	Element	3+	4+	5+	6+
89	Ac	1.11			
90	Th	1.08	0.99		
91	Pa	1.05	0.96	0.90	
92	U	1.03	0.93	0.87	0.83
93	Np	1.01	0.92	0.88	0.82
94	Pu	1.00	0.90	0.87	0.81
95	Am	0.99	0.89	0.86	0.80

Table 6.11 Some Isostructural Compounds of Actinide and Lanthanide Elements (after Makarov)

Compound type	Structure type	Actinide compounds	Lanthanide compounds
Halides	ZrF_4	ThF_4; UF_4; NpF_4; PuF_4	CeF_4
	LaF_3	AcF_3; UF_3; NpF_3; PuF_3; AmF_3	LaF_3; CeF_3; PrF_3 NdF_3; SmF_3
	UCl_3	UCl_3; $AsCl_3$; $NpCl_3$; $PuCl_3$; $AmCl_3$; UBr_3; $AsBr_3$; $NpBr_3$	$LaCl_3$; $CeCl_3$; $PrCl_3$; $NdCl_3$; $LaBr_3$; $CeBr_3$; $PrBr_3$
Hydrides	ThD	ThH_2; ThD_2	
Oxides, sulfides selenides and tellurides	NaCl	UO; PaO; NpO; PuO; AmO; ThS; US; PuS; ThSe; USe; UTe; PuTe	CeS; EuS; EuSe; EuTe; YbSe; YbTe
	La_2O_3	Pu_2O_3; Ac_2O_3	La_2O_3; Ce_2O_3; Pr_2O_3; Nd_2O_3
	CaF_2	PaO_2; NpO_2; PuO_2; AmO_2; ThO_2; UO_2	CeO_2; PrO_2
	γ-Nb_2O_5	γ-U_2O_5; Pa_2O_5; α-U_3O_8	
	Th_3P_4	Pu_2S_3--Pu_3S_4	Ce_2S_3--Ce_3S_4
Nitrides, phosphides, arsenides, etc.	NaCl	ThN; UN; PuN; NpN; $ThPo_{,75}$; UP; PuP; ThAs; UAs; PuAs; USb; UBi; PuBi	LaN; CeN; PrN; NdN; GeN; LaP; CeP; PrP; NdP; LaAs; CeAs; PrAs; NdAs; LaSb; LaBi; CeBi; CeSb; PrSb; PrBi
Carbides, silicides, etc.	NaCl	ThC; UC; PuC; NpC	
	CaC_2	UC_2	LaC_2; CeC_2; PrC_2; NdC_2; SmC_2
	FeB	USi; PuSi	
	α-$ThSi_2$	α-$ThSi_2$; α-USi_2; α-$PuSi_2$; $NpSi_2$; $PuGe_2$	$LaSi_2$; $CeSi_2$; $PrSi_2$; $NdSi_2$; $SmSi_2$; $PrGe_2$
	$CaPb_3$	USi_3; UGe_3; USn_3; UPb_3; $ThPb_3$; $PuGe_3$; $PuSn_3$; $PuPb_3$	$CePb_3$; $CeSn_3$; $LaPb_3$; $LaSn_3$; $PrPb_3$; $PrSn_3$
Borides, aluminides, etc.	AlB_2	UB_2; UGa_2; $ThAl_2$	$LaGa_2$; $CeGa_2$; $PrGa_2$
	UB_4	ThB_4; UB_4	CeB_4
	CaB_6	ThB_6	LaB_6; CeB_6; NbB_6; etc.
	$MgCu_2$	UAl_2; $PuAl_2$; $NpAl_2$	$CeAl_2$; $LaAl_2$
	$BaAl_4$	UAl_4	$LaAl_4$; $CeAl_4$

Table 6.11 (Cont.)

Compound type	Structure type	Actinide compounds	Lanthanide compounds
Intermetallides of VIIa and VII subgroups	$NaZn_{13}$	$ThBe_{13}$; UBe_{13}; $PuBe_{13}$; $NpBe_{13}$	$CeBe_{13}$
	AlB_2	$ThZn_2$; UHg_2	
	$ThMn_{12}$	$ThMn_{12}$	
	$MgZn_2$	$ThMn_2$; $PuOs_2$; UNi_2	
	$MgCu_2$	UMn_2; $PuMn_2$; $PuFe_2$ UFe_2; UOs_2; $PuCo_2$; UCo_2; $PuNi_2$; UIr_2	$CeCo_2$; $CeNi_2$; $LaNi_2$; $PrNi_2$; $PrNi_2$; $CeFe_2$; $CePt_2$
	$CaZn_5$	$ThFe_5$; $ThCo_5$; $ThNi_5$; $PuNi_5$	$LaNi_5$; $CeCo_5$; $CeNi_5$; $PrNi_5$

In general, it is not easy to grow single crystals of strongly radioactive substances. This is because the radioactive material causes destruction of the crystal lattice in the vicinity of its location. There is also a health hazard.

6.3 Other Ions of Interest

In a paper of this length, it would be impossible to discuss the 30 elements of the other three transition groups, even in the short and elementary manner that was used for the rare earth and actinides. This is due to their number and the fact that the close relations that exist within the rare earth and actinide groups are not present in the other groups. There exists for some of these elements books dealing with their chemistry and compounds, and these are listed in the bibliography. If interest in a given element or group arises, a check of a geochemistry book such as Rankama and Sahama would give some idea of its occurrence in nature and Lang's Handbock of Chemistry would give most of the common compounds. A consideration of the ionic radius and charge should make it possible to predict substitution for other ions in a given crystal structure. Spectral information will probably require a search of the literature.

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8.2. Minerals

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8.2b. Mineral Fluorescence

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Spencer, L. J., Fluorescence of minerals in ultraviolet rays, Nat. Hist. Mag. 1, 291-8; Am. Mineral. 14, 33-7 (1929).

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Autunite (Ca uranyl phosphate) is discussed.

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Absorption and fluorescent lines of autunite and potassium uranyl sulfate at temperatures were studied.

Venkateswaran, C. S., The fluorescence of ruby, sapphire and emerald, Proc. Indian Acad. Sci. 2 A, 459-65 (1935).

Emerald strong lines at 6806 and 6837 and weak bands at 6946, 6908, 6736, 6633, 6578 Å. Sapphire strong lines at 6927 and 6942 and bands at 7130, 7060, 6992, 6802, 6753, 6690, 6592 Å.

Ruby strong lines at 7027, 6992, 6945, 6927, 6814, 6791, bands at 7266, 7222, 7164, 7125, 7089, 6753, 6690, 6650, 6592, 6495, 6430, an unresolved spectrum between 6992 and 6945 and a continuous spectrum between 6100 and 6900 Å.

8.3. Rare Earth Elements

The general section here includes articles dealing with the preparation of certain compounds and also some of their properties. This is not a complete bibliography on these subjects, but is to supplement the bibliography on Rare Earths given under Rare Earth in Part A. The section on spectra is very complete and up to date as of early 1963. If the article deals with both absorption and fluorescence the article was placed in the spectra section. Both the spectra and fluorescence sections contain information on energy levels. The rare earth sections are chiefly from Chemical Abstracts, but some references were also obtained from Nuclear Science Abstracts.

8.3a. Rare Earth, General

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Al'tshuler, S. A., The theory of paramagnetic resonance in rare earth metals, Zhur. Eksptl. i Teoret. Fiz. 26, 439-46 (1954).

The metals with cubic, hexagonal and tetragonal lattices are considered separately. Factors affecting the spin-lattice relaxation time are taken into consideration.

Axelrod, J. M., Rare earth carbonates, (Univ. of Maryland, College Park) Univ. Microfilms, (Ann Arbor, Michigan) L. C. Card, 59-3008, 85 pp., Dissertation Abstr. 20, 876-7 (1959).

Ayant, Y. and Belorizky, Élie, Hyperfine structure of praseodymium in solids, J. Phys. Radium 22, 460-2 (1961).

Pr^{+++} and Pr^{++++} in a pure cubic state.

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Baker, J. M., Hayes, W. and Jones, D. A., Paramagnetic resonance of impurties in CaF_2, (Clarendon Lab., Oxford, England) Proc. Phys. Soc. (London) 73, 942-5 (1959).

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Beck, G. and Nowacki, W., Preparation and crystal structure of EuS and EuF_2, Naturwissenschaften 26, 495 (1938).

Becquerel, J., Determination of paramagnetic susceptibility of crystals of salts of rare earths by determining paramagnetic rotatory power, Compt. rend. 201, 1112-15 (1935).

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Bellucci, I. and Grassi, L., A markedly ceritic fluoapatite from Latium, Gazz. chim. ital 49, II, 232-46 (1919).

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La, Ce, Pr, Nd, Sm, Gd, Dy, Er, Yb, and Y, oxyselenides are hexagonal, space group D^3_{3d} - C $\bar{3}$ m.

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CeB_6, NdB_6, GdB_6, YbB_6, and LaB_6.

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M is $^{+++}$ rare earth, is body-centered, cubic with a = 12.40 ± .1.

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$(mC_6H_4NO_2SO_3)_3Y + 7\ H_2O$ and $(mC_6H_4NO_2SO_3)_3$ [illegible] $+ 7\ H_2O$

Monoclinic
(ratio 1.8667:1:2.4925) $\beta = 93^\circ 8'$ optical +

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CeO_2 - Y_2O_3	ThO_2 - Y_2O_3
" - Nd_2O_3	" - La_2O_3
" - Sm_2O_3	" - Ce_2O_3
" - Gd_2O_3	" - Nd_2O_3
" - Dy_2O_3	" - Sm_2O_3
CeO_2 - Yb_2O_3	ThO_2 - Gd_2O_3

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$M_2(CrO_4)_3 \cdot 8\ H_2O$

Solubility decrease with temperature (all are yellowish).

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$R^{+++}{}_2(CO_3)_3 3(CN_3H_6)_2CO_3 \cdot 4\ H_2O$

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uranyl sulfites
$(UO_2)_5 - (SO_3)_3R_2^{+++}\ 15\ H_2O$
R = La, Ce, Nd, Pr.

rare earth bisulfite cuprosulfites of Ce, La, Pe, Nd
$X\ Cu(SO_3)_2 \cdot 8\ H_2O$

cuprothiosulfates of Ce, La, Pe, Nd
$X\ Cu_2(S_2O_3)_3 \cdot 3\ OH_2O$

method of preparation given.

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Ce surrounded by 8 O at corners of a distorted Archimedes antiprism

Ce - O · 2.33 Å

iodate group has trigonal symmetry.

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$Pr(SCN)_3 2C_6H_{12}N_4 \cdot 8H_2O$, light green, soluable in H_2O.

$Pr(SCN)_3 \cdot 6C_{11}H_{12}N_2O$, light green, soluable in H_2O.

$Pr_2(S_2O_6)_3 \cdot 6C_6H_{12}N_4$, green crystals.

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$X^{++}O \cdot Al_2O_3 \cdot 2SiO_2$ (anorthite type),

where X = Li, K, Ca, Na, Rb and Al substituted for by La, Nd, Pr, Ga, Y, Sm, Er, or Sc.

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$$M_4^+[X^{++}(NO_2)_6]$$

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Isometric T_h^3 Fm_3 bounding between NO_2 and coordinating ions is ionic.

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$^{+++}$Nd, Pr and Lu

D_{3d}^{5} - R $\bar{3}$m

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III. Gadolinium
IV. Thulium

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SmP	5.760
SmAs	5.921
SmSb	6.271
SmBi	6.362
SmS	5.970
SmSe	6.200
SmTe	6.594
NdBi	6.424 au

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a, b, c, α, β, γ, and Z also given in C. A. Vol. 45, 8841 g for

$La_2(SO_4)_3 \cdot 9H_2O$ hex $C^2_{6h} - C^6_{\frac{3}{m}}$, $Pr_2(SO_4)_3 \cdot 8H_2O$ mon $C^6_{2h} - C^2_{\frac{c}{c}}$

$Nd_2(SO_4)_3 \cdot 8H_2O$ mon $C^6_{2h} - C^2_{\frac{c}{c}}$, $Pr_2(SO_4)_3 \cdot 5H_2O$ mon $C^6_{2h} - A^2_{\frac{a}{a}}$

$Sm_2(SO_4)_3 \cdot 8H_2O$ mon $C^6_{2h} - C^2_c$, $La(NO_3)_3 \cdot 6H_2O$ tric C^1_i - Pt

$NdCl_3 \cdot 6H_2O$ mon C^2_s - Pn, $SmCl_3 \cdot 6H_2O$ mon C^2_s - Pn

$LaCl_3 \cdot nH_2O$ tric C^1_i - Pt, $PrCl_3 \cdot nH_2O$ tric C^1_i - Pt

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$$M(BrO_3)_3 \cdot 9H_2O$$

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$CePO_4$ hex $C3_121(D_3^6)$
$NdPO_4$

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$Pr_2(SO_4)_3 \cdot 8H_2O$
$Pr_2Mg_3(NO_3)_{12} \cdot 24H_2O$
$Nd_2(SO_4)_3 \cdot 8H_2O$
$Nd_2Mg_3(NO_3)_{12} \cdot 24H_2O$
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La_2S_3	yellowish white
Nd_2S_3	yellowish green
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$Eu_2(SO_4)_3 \cdot 8H_2O$	mon.-prismatic
$Dy_2(SO_4)_3 \cdot 8H_2O$	mon.-prismatic
$Eu(C_2H_5SO_4)_3 \cdot 9H_2O$	dihex.-bipyramidal
$Eu(BrO_3)_3 \cdot 9H_2O$	hex.-bipyramidal
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MOCl

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Westbrook, J. H., Carter, R. E. and De Vries, R. C., A survey of oxygen binary systems. I. Rare earth metal-oxygen system, G. E. Co. Research Lab., Schenectady, N. Y., Jan. 31, 1961, 43 pp.

Whittemore, C. F. and James, C., The sebacates and cacodylates of the rare earths, J. Am. Chem. Soc. 35, 127-32

sebacate

$[C_8H_{16}(COO)_2]_3M_2 \cdot XH_2O$

cacodylate

$[(CH_3)_2AsO_2]M_2 \cdot XH_2O$

Wilke-Dörfurt, E. and Schliephake, O., Contributions to the study of the rare earths, Z. anorg. allgem. Chem. 170, 129-44 (1928).

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$NdCl_3 \cdot 6H_2O$ in H_2O
$SaCl_3 \cdot$ " " "
$YtCl_3 \cdot 6H_2O$ in H_2O

10-50°C

Wold, A., Post, B. and Banks, E., Rare earth nickel oxides, J. Am. Chem. Soc. 79, 4911-13 (1957).

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D_{2h}^{16} - Pb n m

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Zambonini, F., Atomic structure according to Bohr and isomorphogenism of metals of the rare earths with those of the calcium group, Atti accad. Lincei [v] 33, ii, 16-20 (1924).

Zambonini, F. and Carobbi, G., The double sulfates of the rare earth metals and of the alkaline metals, Atti. accad. Lincei [6] 2, 374-7 (1925).

$Nd_2(SO_4)_3Na_2SO_4 \cdot 2H_2O$
$4Nd_2(SO_4)_3 5Na_2SO_4 \cdot 8H_2O$
$3Nd_2(SO_4)_3 4Na_2SO_4 \cdot 6H_2O$
$2Nd_2(SO_4)_3 3Na_2SO_4 \cdot 5H_2O$

Zambonini, F. and Levi, R. G., The isomorphism of the molybdates of the rare earth metals with those of calcium, strontium, barium and lead, Atti. accad. Lincei [6] 2, 149-52, 225-30, 303-5 (1925).

$CaMoO_4$, $SrMoO_4$, $BaMoO_4$ and $PbMoO_4$ all have the same structure. They are isomorphous with Ce group molybdates. The rare earth can substitute in $CaMoO_4$.

Zambonini, F. and Restaino, S., The double sulfates of the rare earth metals and of the alkali metals. V. Cerous ammonium sulfate, Atti. accad. Lincei [6] 3, 178-83 (1926).

$Ce_2(SO_4)_3 \cdot (NH_4)_2SO_4 \cdot 8H_2O$ and $Ce_2(SO_4)_3 \cdot 5(NH_4)_2SO_4$ are described crystallographically.

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Zandy, H. F., The M_{IV} and M_V adsorption edges of Pr^{59}, Nd^{60}, Sm^{62} and Eu^{63}, Proc. Phys. Soc. (London) 65 A, 1015-22 (1952).

Close attention has been paid to the fine structure (white lines) on the long wave side of the edge. The white lines are thought to arise from transitions from $M_{IV,V}$ groups to the incomplete 4f ($N_{VI,VII}$) groups.

Methods of fractional crystallization of double Mg nitrates of rare earths, Virts. Zhur. Anal. Khim. 9, 299-303 (1954).

Compounds of the Cu_5Ca type crystal structure, Nachr. Akad. Wiss. Gottingen, Math. Physik Klasse, IIa Math.-physik.-chem. Abst. No. 1, 6 pp. (1950).

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All are Hex.

a_0 4.864 - 5.664
c_0 3.940 - 4.636

Phase diagram of the system $NdCl_3$-$CaCl_3$-NaCl, Zhur. Neorg. Khim. 2, 1639-42 (1957).

8.3b. Rare Earth Spectra

Agrawal, R. M., Asundi, R. K., Naik, R. C., Ramakrishnan, D. and Singh, S., $^1I_6$ state of Pr^{+++} in $LaCl_3$ at 77°K, Proc. Indian Acad. Sci. Sect. A 55, 106-12 (1962).

Spectra was recorded at 4200-5400 Å.

Albertson, W., Arc spectrum of samarium and gadolinium; normal electron configurations of the rare earths, Phys. Rev. 47, 370 (1935).

Azcona, Juan Manuel López de, Spectrochemical study of the rare earths, Bol. inst. geol. y. minero. España [3] 15, 270-433 (1941).

Concentrations of 1%, 0.1%, and 0.01% as octahydrate sulfate were arcked. Tables of about 250-400 lines from 2480 to 4930 Å are given, as well as other tables tabulating previous work.

Bailey, P. and Goldman, A., Magneto-optical effects in ferrimagnetic oxides, U.S. Dept. Com., Office Tech. Serv., P.B. Rept. 149, 572 75 pp. (1960).

The absorption and Faraday rotation of single crystal rare earth garnets were studied.

Banks, C. V. and Klingman, Donn. W., Spectrophotometric determination of rare earth mixtures, Anal. Chim. Acta 15, 356-63 (1956).

Nd, Pr, Sm, Eu, Gd, Dy, Ho, Er, Tm and Yb in the range 220-1000 mμ. (Oxides dissolved in perchloric acid. Spectra in graphic form.)

Banks, C. V., Heusinkveld, M. R. and O'Laughlin, J. W., Absorption spectra of the lanthanides in fused lithium chloride-potassium chloride eutectic, Anal. Chem. 33, 1235-40 (1961).

A detailed study of the spectra of the lanthanides.

Batsanova, L. R. and Grigor'eva, G. N., The optical properties of fluorides of the rare earth metals of the cerium group, Izv. Sibirsk. Otd. Akad. Nauk S.S.S.R. No. 2, 115-18 (1962).

La, Ce, Pr, Nd, Sm and Eu.

Becquerel, J. and Onnes, H. K., The absorption spectra of the compounds of the rare earths at the temperature obtainable with liquid hydrogen, Proc. Acad. Amsterdam 10, 592-601

Becquerel, J. and Onnes, H. K., Absorption spectra of the compounds of the rare earths at the temperature obtainable with liquid hydrogen and their change by magnetic field, K. Akad. Westenschappen, Amsterdam 10, 592-603

Bethe, H. A. and Spedding, F. H., Absorption spectrum of $Tm_2(SO_4)_3 8H_2O$, Phys. Rev. 52, 454 (1937).

Boulanger, F., The absorption spectra by reflection of some solid rare earth compounds, Compt. rend. 233, 650-1 (1951).

$Nd(OH)_3$
$Al_2O_3 \cdot Nd_2O_3$
Nd_2O_3
$Pr(OH)_3$
$Al_2O_3 Pr_2O_3$

Boulanger, F., The absorption spectra of rare earths, Ann. Chim. 7, 732-71 (1952).

Various solutions and crystalline compounds of Pr, Nd, and Tb were prepared and their visible absorption spectra was studied.

Bovey, L. and Steers, E. B., Optical spectra of some rare earth and transuranic elements in the 1-3 micron region. I. Apparatus, At. Energy Research Estab. (Gt. Brit.) C/R 2827, 10 pp. (1959).

Bovey, L. and Steers, E. B. M., Optical spectra of some rare earth and transuranic elements in the 1-3 μ region. IV. Spectrum of Np. V. Spectrum of Lu, At. Energy Research Estab. (Gt. Brit.) R 3118, 5 pp. (1959), and R 3225, 8 pp. (1959).

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Trivalent Pr, Tm, Ho, Tb, Eu, Nd and Sm are discussed.

Brunetti, R., Polychroism and orientation of ions in crystals of the rare earths, Atti. accad. Lincei [6] 7, 238-44 (1928).

Absorption spectra of $Pr_2(SO_4)_3 \cdot 5H_2O$ and $Na(BrO_3)_3$ are given.

Butement, F. D. S., Absorption and fluorescence spectra of bivalent samarium, europium, and ytterbium, Trans. Faraday Soc. 44, 617-26 (1948).

Absorption spectra from 2000 to 9000 Å of $SmCl_2$ in water and in solid NaCl, $SrCl_2$ and $BaCl_2$; $EuCl_2$ in H_2O and $SrCl_2$ and $BaCl_2$; and $YbCl_2$ in H_2O. Fluorescence spectra include $SmCl_2$ in $SrCl_2$ and $BaCl_2$; EuO_2 in $SrCl_2$ and in $BaCl_2$; $YbCl_2$ in $SrCl_2$ and $BaCl_2$.

Butement, F. D. S. and Terry, H., The absorption spectra of divalent samarium, J. Chem. Soc. (Lond.) 1112 (1937).

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Chow, Y. K., Absorption spectrum of neodymium fluoride NdF_3, Z. Physik. 124, 52-67 (1947).

NdF_3 crystal at liquid air temperature visible and near infrared with high dispersion Zeeman effect observed with magnetic field of 40,000 gausses.

Conway, J. G., Gruber, J., Hulet, E. K., Morrow, R. J. and Gutmacher, R. G., Absorption and self-luminescence spectra of $Cf^{3+}(5f^9)$, J. Chem. Phys. 36, 189-90 (1962).

3000 to 8500 Å region of $LaCl_3$.

Crosby, G. A., Whan, R. E. and Alire, R. E., Intramolecular energy transfer in rare earth chelates role of the triplet state, J. Chem. Phys. 34, 743-8 (1961).

Optical properties of rare earth ions in chelates and in inorganic salts are compared.

Crosswhite, H. M. and Dieke, G. H., Spectrum and magnetic properties of hexagonal $DyCl_3$, J. Chem. Phys. 35, 1535-48 (1961).

Absorption and fluroescence spectra and energy levels are given.

Crozier, M. H. and Runciman, W. A., Analysis of the spectra of trivalent promethium and holmium, J. Chem. Phys. 35, 1392-409 (1961).

Energy levels obtained.

Deutschbein, O., The structure of electrolytic solutions of the rare earths (europium), Z. ges. Naturw. 4, 237-8 (1938).

Spectra of different solutions are compared, also fluorescence spectrum of aqueous $Eu_2(SO_4)_3$ solution and solid $Eu_2(SO_4)_3 \cdot 8H_2O$ are compared.

Dieke, G. H. and Heroux, L., Absorption spectra, Zeeman effects, and magnetic properties of neodymium salts, Phys. Rev. 103, 1227-39 (1956).

$Nd_2Mg_3(NO_3)_{12} \cdot 24H_2O$
$Nd_2Zn_3(NO_3)_{12} \cdot 24H_2O$
$Nd(EtSO_4)_3 \cdot 9H_2O$
$Nd(BrO_3)_3 \cdot 9H_2O$
$NdCl_3 \cdot 6H_2O$
$(La + Nd)Cl_3 \cdot 7H_2O$
$Nd_2(SO_4)_3 \cdot 8H_2O$
$(La + Nd)_2(SO_4)_3 \cdot 9H_2O$
$Nd(OAc)_3 \cdot H_2O$

Dieke, G. H. and Leopold, L., Absorption, fluorescence and magnetic properties of gadolinium chloride ($GdCl_3 \cdot 6H_2O$), J. Opt. Soc. Am. 47, 944-54 (1957).

Dieke, G. H. and Singh, S., Absorption and fluorescence spectra with magnetic properties of $ErCl_3$, J. Chem. Phys. 35, 555-63 (1961).

Eder, J. M. and Valenta, E., Wave-length measurements in the visible part of the arc spectrum. II. Dy, Tr, Er, Gd, Nd, Y, Zr, La and Sm, Sitz. Akad. Wiss. Wien 119. 32 pp. (1910).

Eisenstein, J. C., Analysis of the absorption spectrum of $YbCl_3 \cdot 6H_2O$, J. Chem. Phys. 35, 2097-100 (1961).

Entire spectrum considered.

Elliott, J. P., Judd, B. R. and Runciman, W. A., Energy levels in rare-earth ions, Proc. Roy. Soc. A 240, 509-23 (1957).

The Coulomb interaction, spin coupling, and the influence of an external electrostatic field on a rare earth ion in a crystal lattice, are discussed.

Ewald, H., Transition probabilities in the absorption spectra of the rare earths depend upon temperature, Z. Physik. 110, 428-35 (1938).

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Absorption and luminescence spectra of Sm^{++} in Ca, Sr, and Ba fluoride single crystals. Energy level diagrams 0-18,000 cm^{-1} are given in C.A. 57, 6753 b.

Fog, J. and Osnes, E., Calibration of the wave-length scale on spectrophotometers by samarium and neodymium chlorides, Analyst 87, 760-1 (1962).

The visible absorption spectra of $SmCl_3$ and $NdCl_3$ solutions were measured.

Forrester, P. A. and Hempstead, C. F., Paramagnetic resonance of Tb^{+++} ions in $CaWO_4$ and CaF_2, Phys. Rev. 126, 923-30 (1962).

Freed, S., Spectra of ions in fields of various symmetry in crystals and solutions, Rev. Modern Phys. 14, 105-11 (1942).

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Freed, S. and Hochanadel, C. J., Solutions of salts fluid at the temperature of liquid nitrogen; spectra of solutions of rare earths, J. Chem. Phys. 17, 664-5 (1949).

The hexahydrate bromides and nitrates of Nd, Sm, Pr and Tm were studied.

Freed, S. and Hochanadel, C. J., Spectra of rare earths in solutions fluid at the temperature of liquid nitrogen, J. Chem. Phys. 18, 780-2 (1950).

Freed, S. and Katcoff, S., The absorption and fluorescence spectra of divalent europium ion in crystals, Physica 14, 17 (1948).

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In solutions.

Friederich, A., Hellwege, K. H. and Lämmermann, H., Further analysis of the spectra of trivalent samarium in single crystals of various symmetries, Z. Physik 155, 251-60 (1960).

Sm ethyl sulfate, $SmCl_3$ and Sm nitrate.

Gatter, A., Junkes, J., et al., Atlas of persistent spectra, Separate from Astrophys, Lab. of the Vatican Observatory, Vatican City (1949).

I. Spectra of 30 chemical elements - includes among other Co, Cr, Cu, Mg, Mn, Mo, Ni, Ti, V, Zn and Pt. 53 pp. and 28 charts.

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Gobrecht, H., A new energy change of an atom through absorption of light, Z. ges. Naturw. 3, 351-2 (1937).

The absorption and fluorescence spectra of $Yb_2(SO_4)_3 \cdot 8H_2O$ in the infrared was measured. (Ho^{+++}, Er^{+++}, and Tu^{+++} also showed infrared absorption.).

Gobrecht, H. and Tomaschek, R., Analytical detection of rare earths by absorption and fluorescence, Ann. Physik 29, 324 (1937).

Gobrecht, H., The spectra of trivalent thulium, Ann. Physik 31, 600-8 (1938).

The absorption and emission spectra.

Gobrecht, H., Screening constants and transitions between the multiplet terms of the ground states of the rare earth ions, Ann. Physik 31, 755-60 (1938).

Infrared absorption of trivalent, Nd, Eu, Dy, Ho, Er, Tu and Yb are given, also some fluorescence spectra.

Gobrecht, H., The absorption spectra and the ground terms of the ions of the rare earths, Ann. Physik 31, 755-67 (1938).

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Pure and with Eu.

Gramberg, G., Absorption spectrum and zeeman effect of crystalline dyprosium salts at low temperature, Z. Physik 159, 125-48 (1960).

(18,000 - 27,000 cm^{-1}) Dy ethyl-sulfate, chloride and nitrate.

Grishina, T. I., Determination of small quantities of rare earth elements in praseodymium, neodymium, samarium, terbium, dysprosium, holmium, erbrium, and thulium preparations, Zhur. Anal. Khim. 14, 427-30 (1959).

Spectral lines in range 3000-4000 Å were studied.

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Visible absorption spectra in ethylsulfate, nitrate, and chloride.

Guy, J. S. and Jones, H. C., A quantitative study of absorption spectra by means of the radiomicrometer, Am. Chem. J. 50, 257-308.

Infrared absorption of solutions of Nd chloride (0.65 μ to 1 μ), Nd nitrate (0.65 μ to 0.95 μ), Nd acetate (0.65 μ to 0.95 μ), Pr chloride and nitrate (0.55 μ to 0.7 μ), and several Ni and Co salts (0.5 μ to 1.18 μ).

Hagiwara, Z., Separation of the rare earths and their accompanying elements. XI. Absorption spectra of light lanthanons in the ultraviolet and visible range, Technol. Repts. Tohoku Univ. 20, 77-90 (1955).

$Ce_2(SO_4)_3$ in H_2SO_4 showed broad absorption maxima at 3160 Å and no absorption above 4800 Å. $SmCl_3$ shown from 3500 Å to 8500 Å, the most intense band is at 4020 Å. $Pr(NO_3)_3$ showed sharp maxima 4440, 4690, 4820, and 5920 Å. $NdCl_3$ showed minima at 4190, 4270, 4330, 4630, 4700, 4760, 5130, 5200, 5230, 5780, 6240, 6840, 7450, and 8010 Å.

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Hellwege, K. H., The electron vibration spectrum of neodymium salt crystals, Z. Physik 113, 192-202 (1939).

Infrared spectrum of Zn-Nd nitrate was measured in the region 25-58 μ.

Hellwege, K. H., Analysis of the spectra of crystalline europium salts, Nachr. Akad. Wiss. Göttingen, Math.-physik. Klasse, Math.-physik.-chem. Abst. No. 2, 58-66 (1947).

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Hellwege, K. H., Horstik, G., Huefner, S. and Laemmermann, H., Spectrum, Zeeman effect, and electronic levels of crystalline dysprosium salts in the photographic infrared, Z. Physik 165, 253-8 (1961).

Absorption spectrum of $Dy(C_2H_5SO_4)_3 \cdot 9H_2O$; $Dy(NO_3)_3 \cdot 6H_2O$.

Hellwege, K. H., Huefner, S. and Kahle, H. G., Spectrum, Zeeman effect, and electronic levels of trivalent erbium in crystalline salts. I. Erbium tris (ethyl sulfate) $Er(EtSO_4)_3 \cdot 9H_2O$. II. Erbium chloride $ErCl_3 \cdot 6H_2O$ and erbium nitrate $Er(NO_3)_2 \cdot 6H_2O$, Z. Physik 160, 149-70 (1960).

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Holleck, L. and Hartinger, L., Molar extinction-coefficients of the rare-earths and their analytical use, Angew. Chem. 67, 648-651 (1955).

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Holleck, L., Liebold, G. and Weidmann, G., Spectrophotometric determination of cerium, Angew. Chem. 68, 552 (1956).

Hoogschagen, J. and Gorter, C. J., Absorption of light in solutions of the rare earth salts, Physica 14, 197-206 (1948), (in English).

Aqueous solutions of Er, Tm, and Yb.

Jaffe, P. M. and Banks, E., Oxidation states of europium in the alkaline earth oxide and sulfide phosphorus, J. Electrochem. Soc. 102, 518-23 (1955).

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Jelley, E. E., Pleochroism of crystals of rare earth salts as evidence of the non-rotation of certain electronic orbits, Nature 136, 335-6 (1935).

Absorption spectra of Nd sulfate are shown.

Jones, H. C. and Anderson, J. A., The absorption spectra of a number of salts in water, in certain non-aqueous solvents, and in mixtures of these solvents with water, Am. Chem. J. 41, 163-208, 276-326 (1908).

The chlorides of Co, Ni, Cu, Fe, Cr, Nd, and Pr; and the bromide and nitrate of Nd in solvent.

Jones, H. C. and Anderson, J., The absorption spectra of solutions of certain salts of Co, Ni, Cu, Fe, Cr, Nd, Pr, and Er in water, methyl alcohol, ethyl alcohol and acetone, and in mixtures of water and other solvents, Carnegie Inst., Washington, D. C., (1909).

Joos, G. and Ewald, H., The riddle of supernumerary lines in the absorption spectrum of rare earth salts, Naturwissenschaften 25, 636-7 (1937).

Spectra of

$Nd_2Zn_3(NO_3)_{12} \cdot 24H_2O$
" Mg_3 " " "
$Nd_2Mn_3(NO_3)_{12} \cdot 24H_2O$

Joos, G. and Hellwege, K. H., True line width and oscillator strength of europium sulfate, Ann. Physik 39, 25-9 (1941).

$Eu_2(SO_4)_3 \cdot 8H_2O$ at -190°C.

Jones, H. C. and Strong, W. W., The absorption spectra of solutions of comparatively rare salts, including those of gadolinium, dysprosium and samarium, Carnegie Inst., Washington, D. C., 112 pp. (1912).

Judd, B. R., Optical absorption intensities of rare earth ions, Phys. Rev. 127, 750-61 (1962).

Kingsley, J. D. and Prener, J. S., Spectroscopy and luminescence properties of CdF_2:Eu, Phys. Rev. 126, 458-65 (1962).

Possible use as an optical maser is discussed.

Jørgensen, Chr. K., Absorption spectra of lanthanide and actinide ions, J. Chem. Phys. 23, 399-400 (1955).

Jørgensen, Chr. K. and Videnskab, Kgl. D., Studies of absorption spectra. VII. Systems with three or more f-electrons, Selskab. Mat.-fys. Medd. 29, No. 11, 29 pp. (1955).

The atomic energy levels causing the narrow bands of lanthanide and actinide elements are investigated.

Jørgensen, C. K. and Videnskab, Kgl. D., Variation of the parameters of electrostatic interaction F_k derived from absorption spectra of lanthanide complexes, Selskab. Mat. Fys. Medd. 30, No. 22, 38 pp. (1956), (in English).

The absorption spectra of trivalent Pr, Nd, Sm and Gd in complexes such as ethylenediamine-tetraacetate, nitrogen triacetate, tartrate, and citrate. Absorption data is given both in tabulated and graphical form. 97 references.

Jørgensen, C. K., Absorption spectra of dysprosium (III), holmium (III) and erbium (III) aquo ions, Acta. Chem. Scand. 11, 981-9 (1957), (in English).

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Joye, P.,Reflection spectra of praseodymium compounds, Arch. sci. phys. nat. 36, 431-8 (1913).

Spectra of Pr, oxide, hydroxide, chloride, sulfide, sulfate, nitrate ($6H_2O$), and the oxalate ($10H_2O$) over range 4400 to 5900 Å.

Joye, P., Reflection spectra of neodymium compounds, Arch. sci. phys. nat. 36, 41-58 (1914).

$2Nd_2O \cdot 6H_2O$, $2Nd_2O \cdot 3H_2O$, Nd_2O_3, Nd_2S_3, $Nd_2(SO_4)_3$, $NdCl_3$, $NdBr_3$, $Nd_2(CO_3)_3$, $Nd_2(C_2O_4)_3 + 10H_2O$, $Nd(N_2O_3)_3 \cdot 6H_2O$ were investigated.

All Nd compounds are characterized by a group of lines at 4200 to 5400, 5080 to 5480, 5700 to 6000, and in the region 6200 to 6800 Å. Spectra not observed beyond 6900 Å.

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Spectra

monoclinic

$GdCl_3 \cdot 6H_2O$ $GdBr_3 \cdot 6H_2O$ $Gd_2(SO_4)_3 \cdot 8H_2O$ $Gd_2(SeO_4)_3 \cdot 8H_2O$

hexagonal

$Gd(OOCH)_3$ $Gd(BrO_3)_3 \cdot 9H_2O$ $Gd(EtSO_4)_3 \cdot 9H_2O$
$Gd(EtCOO)_3 \cdot 2H_2O$ $Gd(PrCOO)_3 \cdot 2H_2O$

triclinic

$Gd(OAc)_3 \cdot 4H_2O$ $Gd(CCl_3COO)_3 \cdot 3H_2O$

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8.3c. Rare Earth Fluorescence

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Carlson, E. and Dieke, G. H., Fluroescence spectrum and low levels of neodymium chloride, J. Chem. Phys. 29, 229-30 (1958).

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Golrecht, H., Fluroescence of rare earths, Ann. Physik 31, 181-6 (1938).

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Haberlandt, H., Karlik, B. and Przibram, K., Fluorescence of fluorite. IV. The detection of uranium in fluorites and the low temperature fluorescence, Sitzber. Akad Wien., Math.-naturw. Klasse, Abt. lla 144, 135-40 (1935).

V. The fluorescence of europium dichloride and of alkali halide-europium fluorescence. Prizibram, K., Ibid 141-9.

Pure $EuCl_2$ fluoresces bright purple, and shows a narrow band at 690 mμ and a stronger one at 630 mμ.

Haitinger, O. M., Fluorescence phenomena of cerium and other rare earths Sitzber. Akad. Wiss. Wien, Math.-naturw. Klasse, Abt. IIa, 142, 339-42 (1933).

Ce, Eu, Sm, Gd, Tb, Dy, Tm and Ho preparations were found to show fluorescence in the visible; La, Pr, Nd, Er, and Yb did not.

Hellwege, K. H., Fluorescence and the coupling between electron terms and crystal lattice in hydrated salts of the rare earths, Ann. Physik. 40, 529-42 (1941).

Only the rare earths from Sm to Dy fluoresce because in the others the coupling is so strong that the excitation energy is quickly transferred to the lattice.

Iwase, E., Cathodoluminescence spectrum due to the presence of samarium in solid calcium compounds, Sci. Papers Inst. Phys. Chem. Research (Tokoyo) 34, 487-503 (1938).

Ca compounds containing Sm: silicate, sulfate, phosphate, tungstate, molybdate, fluoride, oxide, aluminate and borate.

Judd, B. R., Analysis of the fluorescence spectrum of neodymium chloride, Proc. Roy. Soc. (London) A 251, 134-42 (1959).

Kaplyanskiĭ, A. A., Moskvin, N. A. and Pryhevuskiĭ, A. K., Band splitting in the luminescence spectra of anisotropic centers in LiF and CaF_2: Eu crystal subjected to oriented deformation, Optics and Spectroscopy (USSR), (English Trans.) 10 [3], 185-88 (1961).

Karlik, B. and Przibram, K., The fluorescence of divalent rare earths, S.B. Akad. Wiss. Wien., IIa, 146, 209 (1937).

Eu, blue; Yb, yellowish green; Sm, red; and Tu, red.

Keller, S. P., Fluorescence spectra, term assignments, and crystal field splittings of rare earth activated phosphors, J. Chem. Phys. 29, 180-7 (1958).

The visible fluorescent emission, excited by 280 mμ radiation, of SrS activated with rare earths were studied.

Keller, S. P., Variation of valence state of europium in strontium sulfide phosphors, J. Chem. Phys. 30, 556-60 (1959).

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Optical study results of transmission, excitation, fluorescence phosphorescence, stimulation emission spectra of SrS, SrS:Ce,Sm and SrS:Eu,Sm.

Keller, S. P. and Pettit, G. D., Quenching, stimulation and exhaustion studies on some infrared stimulatable phosphors, Phys. Rev. 111, 1533-9 (1958).

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Keller, S. P. and Pettit, G. D., Low-temperature fluorescence spectra and crystal-field splittings of rare-earth activated strontium sulfide phosphors, J. Chem. Phys. 30, 434-41 (1959).

Activated with trivalent Ce, Pr, Nd, Sm, Er, Gd, Tb, Dy, Ho, Tm, Yb or Lu, and Eu^{++}.

Kondrat'eva, E. V. and Ustinov, V. B., Luminescence afterglow of terbium salt solutions, Vestnik Leningrad Univ. 14, No. 4, Ser. Fiz. i. Klim. No. 1, 5-10 (1959).

Bands at 5890, 5450, and 4890 Å of $Tb_2(SO_4)_3$ in concentrated H_2SO_4 solution.

Kremensky, N., Larionov, J. and Seidel, A., The structure of the bands in the fluorescence spectrum of aqueous solutions of terbium salts, Acta physchim. U.S.S.R. 6, 481 (1937).

Kroeger, F. A. and Bakker, J., Luminescence of cerium compounds, Physica 8, 628 (1941).

Larionov, Ya. I., Photoluminescence spectra of solutions of rare earth salts, Vestnik Leningrad Univ. No. 7, 18-28 (1947).

Tb salts show luminescence spectrum with bands at 681, 670, 648, 621, 589, 545, and 489 mμ. Width of the bands are 10-20 mμ. Other rare earths are also discussed.

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Larionov, I. and Seidel, A., Fluorescence of the salts of trivalent europium in aqueous solutions, Compt. rend. acad. sci. U.R.S.S. [N.S.], 3, 115-18 (1936).

Eu sulfate; 3 narrow bands at 5935, 6165 and 6965 Å, a narrow band at 3108 Å, a broad band at 3215 to 4070 and at about 4550 Å.

Levskin, V. L., et al., Alkaline earth phosphors, characterized by a high sensitivity to infrared rays, Zhur. Eksptl. Teoret. Fiz. 17, 944-63 (1947).

CaS and SrS with rare earths, emission spectra are given.

MacDonald, R. E., Fluorescence of europium tungstate, IBM J. Res. Develop. 6, 363-4 (1962).

$Eu_2(WO_4)_3$ strongest emission is in the 6150 Å region.

Mendenhall, C. E. and Wood, R. W., The effect of electric and magnetic fields on the emission lines of solids, Phil. Mag. 30, 316-20 (1915).

Crystals studied were fluorite, ruby, monazite, $Pr_2(SO_4)_3$, $Nd_2(SO_4)_3$ and $UO_2(NO_3)_2$.

Mukherji, P. C., Investigation of the diffuse fluorescence spectrum of ions of the rare earths in solution and in the crystal, Z. Physik 109, 573-85 (1938).

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Nichols, E. L. and Hawes, H. L., Note on the rare earths as activators of luminescence, J. Opt. Soc. Am. 13, 573 (1926).

Peattie, C. G. and Rogers, L. B., Analytical studies of fluorescence of Sm in calcium sulfate, Spectrochim. Acta 9 [4], 307-22 (1957).

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Przibram, K., The red fluorescence band of bivalent samarium, Anz. Akad. Wiss. Wien, Math-naturw. Klasse 73, 235-6 (1936).

Przibram, K., Coloration and luminescence produced by Becquerel rays. IV. The fluorescence of bivalent rare earths, Z. Physik. 107, 709-12 (1937).

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Przibram, K., Fluorescence of bivalent rare earths, Nature 139, 329 (1937).

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Przibram, K., The fluorescence of fluorite and bivalent rare earths, Compt. Rend. Acad. Sci. U.R.S.S. 56, 31-33 (1947).

Eu, Yb, Sm, and Lu.

Przibram, K., The light emission of europium (in part the experiments of Weger), Anz. Öst. Akad. Wiss., 2 December 1948.

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Seidel, A., Larionov, J. and Filipov, A., On the fluorescence of aqueous solutions of rare earth salts, J. Phys. U.S.S.R. I, 67 (1939).

Servigne, M., Infrared emission of luminescence of some rare elements, Compt. rend. 207, 905-7 (1938).

The IR. emission spectra of solid solutions of Sm, Eu, and Dy in $CaWO_4$ was determined and is shown graphically. Also compared to the same elements in solid solution with $CaSO_4$.

Stanevich, A. E. and Yaroslavskiĭ, N. G., Some infrared radiation sources within the 20-110 μ range, Inzhener-Fiz. Zhur., Akad. Nauk Belorus S.S.R. 1, No. 7, 49-53 (1958).

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Terol, S. and Ward, R., Preliminary-studies of the perovskite-type ternary oxide as luminophors, J. Electrochem. Soc. 102, 524-8 (1955).

Pr^{+++}, Sm^{+++} and Eu^{+++} as impurities. (Emission spectra are given.).

Tomaschek, R., The phosphoresence spectra as an aid in the investigation of the structures of solid and liquid substances, Reichsanet Wirtschaftsaubau, Chem. Ber. Prüf. Nr. 15, (PB 52016), 517-43 (1942).

Fluorescence spectra of rare earths in various crystals are given.

Tomaschek, R., Structure phenomena in solids and fluids by means of fluorescent line spectra, Ergebn. d. Exakten. Naturwissenschaften 20, 268 (1942).

Tomaschek, R. and Deutschbein, O., Fluorescence of pure salts of the rare earths, Nature 131, 473 (1933).

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Tomaschek, R. and Mehnert, E., Fluorescence of gadolinium salts and their solutions, Ann. Physik 29, 306-10 (1937).

Urbain, G., Cathodic phosphorescence of the rare earths, Ann. Chim. Phys. 18, 222-288, 288-376

Complete wave length tables and many charts of spectra are given.

Uzumasa, Y., Cathodic luminescence spectra of rare earths extracted from samarskite, ishikawaite and monazite of Ishikawa, Iwaki province (Japan), Japan J. Chem. 4, 7-9 (1929).

Luminescence spectra are given.

Van Uitert, L. G., Factors influencing the luminescent emission states of the rare earths, J. Electrochem. Soc. 107, 803-6 (1960).

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Van Uitert, L. G., Factors controlling the intensity of emission of Eu^{+++} and Tb^{+++} in scheelites, J. Chem. Phys. 37, 981-5 (1962).

Van Uitert, L. G. and Iida, S., Quenching interactions between rare earth ions, J. Chem. Phys. 37, 986-92 (1962).

Quenching of Eu^{+++} by Tb^{+++} was studied.

Van Uitert, L. G. and Soden, R. R., Emission spectra of trivalent terbium, J. Chem. Phys. 32, 1161-4 (1960).

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Van Uitert, L. G. and Soden, R. R., Influence of ion size upon the intensity of Eu^{+++} fluorescence in the tungstates, J. Chem. Phys. 36, 517-19 (1962).

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In alkali metal rare earth tungstates.

Van Uitert, L. G. and Soden, R. R., Effects of rare earth ion substitution upon the fluorescence of terbium hexa-antipyrene tri-iodide and sodium europium tungstate, J. Chem. Phys. 36, 1797-1800 (1962).

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Varsángi, F. and Dieke, G. H., Monochromatically excited fluorescence in rare earth salts, J. Chem. Phys. 31, 1066-70 (1959).

This is a very sensitive method for studying the coupling of the electronic levels with lattice vibrations. If a crystal contains several rare earth ions, their fluorescence spectra are excited independently of each other.

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Weissman, S. I., Intramolecular energy transfer. The fluorescence of complexes of europium, J. Chem. Phys. 10, 214 (1942).

Whan, R. E. and Crosby, G. A., Luminescence studies of rare earth complexes: benzoylacetonate and dibenzoylmethide chelates, Mol. Spectry. 8, 315-27 (1962).

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Wild, G., Spectroanalytical investigation of fluorite, Sitzber. Akad. Wiss. Wien. Math.-naturw. Klasse Abt. II a, 146, 479-92 (1937).

A study of fluorescence as related to rare-earth content, especially Yb and Eu.

Witzmann, H. and Müller-Buschbaum, H. K., Emission behavior of cerium-samarium-activated borate phosphors, Z. physik Chem. 210, 72-85 (1959).

Sm or Ce activated $CaO:\beta_2O_3$.

Witzmann, H. and Müller-Buschbaum, H. K., Fluorescence of rare earths, Naturwissenschaften 49, 180 (1962).

Zaĭdel, A. N., Iazeeva, G. S., Ostrovskaya, G. V. and Yakimova, P. P., Luminescence of gadolinium salts, Izv. Akad. Nauk. S.S.S.R., Ser. Fiz. 26, 74-80 (1962).

In solutions.

Zaĭdel, A. N., Larionov, Ya. I. and Filippov, A. N., Fluorescence of the ions of rare earths in solutions, Bull. Acad. sci. U.R.S.S., Classe sci. math. nat., Ser. phys. (1938), 333-4 (in English, 334-5).

Zaĭdel, A. N., Larionov, Ya. I. and Filippov, A. N., Fluorescence of the aqueous solutions of rare earth salts, I., J. Exptl. Theoret. Phys. (U.S.S.R.) 9, 17-24 (1939).

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$Gd(SO_4)_3$, $Gd(SO_4)_3 \cdot 8H_2O$, and $GdCl_3 \cdot 6H_2O$ spectra studied.

8.4. Transition Elements

This section covers all the transition elements except the rare earths. The references presented deal with absorption spectra and fluorescence, and are a set of selected references picked from those that appeared before early 1963. The chief source of these references was Chemical Abstracts but Nuclear Science Abstracts were also used. Many of the references are annotated. When both absorption spectra and fluorescence are discussed in one article the entry is made only in the Absorption Spectra section. Information on energy levels is to be found in both sections.

8.4a. Transition Elements Spectra

Ackerman, R. J., Thorn, R. J. and Winslow, G. H., Visible and U.V. absorption properties of uranium dioxide films, J. Opt. Soc. Am. 49 [11], 1107-12 (1959).

Addison, C. C. and Gatehouse, B. M., The infrared spectra of anhydrous transition metal nitrates, Chem. and Ind. (London), 464 (1958).

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3800 - 700 cm^{-1}

Ami El Sayed, M. F. and Sheline, R. K., Infrared spectrum and structure of the $Ni(CN)_4^{-4}$ ion, J. Am. Chem. Soc. 80, 2047 (1958).

Antipova-Karataeva, I. I., Investigation and interpretation of absorption band spectra of crystals colored with chromium ions, Optika i Spectroskopiya 7, 263-5 (1959).

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Antunes, M. T., The energy values of the $3d^8 4p$ electronic configuration of cobalt, Phys. Rev. 62, 362-8 (1942).

Appel, J., Electric and optical properties of silver telluride, Z. Naturforsch. 10a, 530 (1955).

Armstrong, A. M., Brearley, A. and Warren, I. H., Uranium antimonides, arsenides, phosphides, selenides, sulfides, and tellurides - an annotated bibliography, Can. Uranium Res. Found. Bibliog., No. B-4, 25 pp. (1961).

Spectral information included.

Arneth, R., Infrared absorption of ZnO crystals, Naturwissenschaften 45, 282 (1958).

1 to 12 μ. High transition in visible and near IR.

Arneth, R., Absorption of ZnO single crystals in the infrared, Z. Physik 155, 595 (1959).

Pure and doped ZnO crystals 10,000 and 700 cm^{-1}.

Asprey, L. B. and Keenan, T. K., Quadrivalent americium and cerium; the absorption spectra of the tri- and tetrafluorides of these elements, J. Inorg. and Nuclear Chem. 7, 27-31 (1958).

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Bader, W. and Westland, A. D., The electronic spectra of $MoCl_5$ and $NbCl_5$, Can. J. Chem. 39, 2306-15 (1961).

Visible spectra of $MoCl_5$ and $NbCl_5$ in solution.

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Binc, R. and Hadzi, D., Infrared spectra and hydrogen bonding in nickel dimethylglyoxime and related complexes, J. Chem. Soc., 4536 (1959).

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Bloss, F. D., Relationship between light absorption and composition in a solid solution series, Am. Mineralogist 40 [516], 371-97 (1955).

Series $Ni(NH_4)_2(SO_4)_2 \cdot 6H_2O$ and $Mg(NH_4)_2(SO_4)_2 \cdot 6H_2O$.

Bonino, G. B. and Fabbri, G., The infrared absorption spectrum of potassium ferrocyanide, Atti. accad. nozl. Lincei, Rend., Classe sci. fis. mat. e nat. 19, 386 (1955).

$K_4Fe(CN)_6$ and its trihydrate at 416-3333 cm^{-1}.

Bonino, G. B. and Fabbri, G., Infrared absorption spectrum of potassium molybdenum octacyanide, Atti. accad. nozl. Lincei, Rend., Classe sci., fis., mat. e nat. 20, 566 (1956).

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Brame, E. G., Jr., Johnson, F. A., Larsen, E. M. and Meloche, V. W., Infrared spectra of inorganic solids. III. Octacyano complexes of tungsten (IV), (-V) and Molybdenum (IV), (-V) dehydrate and and anhydrous, J. Inorg. and Nuclear Chem. 6, 99 (1958).

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Brame, E. G., Jr., Margrave, J. L. and Meloche, V. W., Infrared spectra of inorganic solids, J. Inorg. and Nuclear Chem. 5, 48 (1957).

667-5000 cm^{-1}

Li_3N, Cu_3N, BN, AlN, Mg_3N_2, Zn_3N_2, B_2O_3, Cu_2O_3, Ga_2O_3, Al_2O_3, B_4C, SiC, Mo_2B, CrN, ZrB_2 and TiB_2

Brazdzhyunas, P. P. and Vishchakas, Yu. K., Optical properties of polycrystalline layers of cadmium selenide, Lietuvos. T.S.R. Mokslu Akad. Darbai Ser. B., No. 4, 21 (1956).

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3000 to 1300 cm^{-1}
Bivalent hexammine complexes of Fe, Cd, Mn, Zn, Co, Ni and Cu; and bivalent tetrammine complexes of Cd and Cu. Trivalent hexammine complexes of Cr, Fe and Co.

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Chatt, J., Dunconson, L. A. and Gatehouse, B. M., et al., Infrared spectra and structure of some simple and bridged nitro complexes of platinum, palladium, and cobalt, J. Chem. Soc., 4073-80 (1959).

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$KPu(SO_4)_2 \cdot 5H_2O$	$PuO_2(ClO_4)_2 \cdot x\ H_2O$
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$CaWO_4$ and Zn_2SiO_4

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Van Gool, W., Fluorescence and photoconduction of silver-activated cadmium sulfide, Philip Research Repts. 13, 157-66 (1958).

Fluorescence of Ag activated CdS phosphors, with Ga or Cl show 2 bands with maxima at 6200 and 7300 A.

Van Gool, W., Fluorescence centers in ZnS, Philips Research Repts. No. 3 (Suppl.), (1961), 119 pp.

ZnS and CdS containing Ag, Cu, Au, Al, Sc, Ga, and In were studied.

Vitovskiĭ, B. V. and Anikin, I. I., Luminescence of synthetic sodium chloride and potassium chloride crystals with additions of different activators, Trudy. Inst. Krist. Akad. Nauk S.S.S.R. No. 11, 200-5 (1955).

By thermal electrodiffusion variable amounts $MnCl_2$ and $CuCl_2$ were introduced into NaCl and KCl monocrystals.

The luminescence colors for λ 365, 313, 280 and 250 mμ in the exciting light are tabulated and spectral curves are given.

The results are compared with thermally introduced Tl, Pb, Ag and Pt in NaCl and KCl.

White, C. E., Ho, M. and Weimer, E. A., Fluorescence spectra of metal chelates, Spectrochim Acta 16, 236-7 (1960).

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Al fluoride complex with Group II elements fluorescence was studied.

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KCl with Tl^{+}, Pb^{++} and Br^{+++} in range 2000 - 6000 Å.

8.5. Glass

This section is divided into three parts: 1. Rare Earths - all types of references on rare earths in glasses, especially spectra and fluorescence; 2. Absorption Spectra and Fluorescence - the general spectra of glasses of various types, and the fluorescence in glasses of ions other than rare earths; and 3. Structure - a collection of most of the references on glass structure, especially those that are based on spectra (and references dealing with structure, based of spectral information is included in this part and not in 2). The chief source of these references has been Ceramic Abstracts up to and including the March, 1963, issue. This was supplemented by the use of Chemical Abstracts.

8.5a. Rare Earth Glasses

Büttner, H., The application of rare earths to ceramics, Keram Rundschau 44, 267 (1936).

Čtyroký, V., Glasses colored with Nd_2O_3 and V_2O_5, Glastech. Ber. 18 [1], 1-7 (1940).

Transmission coefficients were calculated.

Čtyroký, V. and Fanderlik, M., Glasses colored with Nd_2O_3 and Cr_2O_3, Sklářské Rozhledy 20 [7], 1211-25 (1943).

Cr_2O_3 addition to Nd_2O_3 glass shifts the color toward green.

Deberg, A., The possible use of salts of rare earths in glass, Verre Silicates Ind. 6, 100 (1935).

Deutschbein, O., The fluorescence of the rare earths in glasses, Z. Physik 102, 772-80 (1936).

Sm, Pr, Nd, Dy, Eu, Tb, and Er in glasses were studied.

Dodd, C. X. and West, T. W., Spectral transmittance properties of rare-earth glasses, J. Opt. Soc. Am. 51 [8], 915-16 (1961).

Visible at 4°, 34° and 64°C.

(5% Dy, Er, Ho, Nd, Pr, and Sm.)

Erametsa, O. and Kolehmainen, A., Modification of the ion characteristic to the glass theory and the glasses colored with lanthanous, Acta Polytech. Scand. Chem. Met. Ser No. 12, 21 pp. (1960).

As lanthanides enter the region of intermediate oxides, lanthanide glasses were made from very pure base glasses with colored lanthanides, except thulium. Their absorption spectra were measured. With increasing lanthanide amount, the color increases slowly at first, but after a certain point it increases rapidly strictly following the Lambert-Beer law. This is explained by the theory that, owing to their intermediate character, the lanthanide first becomes fixed in the silicate skeleton and only then do they replace cations.

Foëx, M., Rare-earth glasses: Absorbing glasses; (1) neodymium and praseodymium (2) cerium, Verres et refractaires, Aug., 1947, pp. 3-18.

108 references

Foëx, M., Rare earth glasses, Verres et refract., Oct., 1947, pp. 19-27.

Rare earth oxides in various glasses show very intense fluorescence.

Hampel, C. A., Rare earths, I., Glass Ind. 41 [1], 15-16, 48-51 (1960); II. Cerium [2], 82-86, 109-13; III. Rare earths in the glass industry [3], 148-53, 174.

Properties of rare earth radiation absorption spectra.

Kapnicky, J. A. and Koehler, W. A., Effects of cerium oxide on the spectral transmission of some experimental glasses, J. Am. Ceram. Soc. 31 [11], 321-22 (1948).

Several soda-lime, borosilicate and flint glasses were used. The spectral transmission of each was determined over the spectral range 250 - 1000 mμ.

Krishnaiyar, N. C., Fluorescence of didymium in glass, Proc. Indian Assoc. for Cultivation of Science 8, 217-9 (1923).

Bands recorded at 475-504, 548-560, 588-612, and 622-645 mμ.

Levine, S., Rare earths. I. Glass and ceramics, Ceram. Age 70 [2], 28-31 (1957).

Effects of pure rare earths in glass are described.

Löffler, J., Rare earths and how they are used for coloring and decolorization of glass, Glashütte 86, 63 (1936).

Pringsheim, P. and Schlivitch, S., The fluorescence of praseodymium and neodymium glass, Z. Physik 61, 297-306 (1930).

Rosenthal, G., Absorption behavior of neoydmium in glasses, Glastech. Ber. 18 [6], 155-58 (1940).

5-15% (Nd_2O_3) in silica, borosilicate, borate, and phosphate glasses were examined.

Transmission curves are presented.

Stair, R. and Faick, C. A., Infrared absorption spectra of some experimental glasses containing rare-earth and other oxides, Jour. Research Nat. Bur. Standards 38 [1], 95-101 (1947), RP 1716.

Spectral transmission data are given on soda-lime glass containing rare-earth (Y, Pr, Nd, Sm, Gd, and Er) and other coloring oxides (V, Cr, Mn, Co, Ni, and U) in the spectral region of 0.7 to 4.5 microns.

Tashiro, M., Soga, N. and Sakka, S., Study of the absorption spectra of cerium in glass, Yogyo Kyokai Shi 68 [773], 132-37 (1960).

Vickery, R. C. and Sedlach, R., Absorption spectra of rare earths in glasses, Nature 181 [4601], 39-40 (1957).

Weidert, N., Rare earth elements in glass, Natl. Glass Budget 45, No. 16, 14 (1929).

A review.

8.5b. Spectra of Glasses

Adams, R. V. and Douglass, R. W., Infrared studies on various samples of fused silica with special reference to the bands due to water, J. Soc. Glass Technol. 43, 147T-158T (1959).

Aglan, M. A. and Moore, H., Colors of cobalt in glass, J. Soc. Glass Technol. 39, 351 (1955).

Visible absorption spectrum of cobalt in various glasses is given. Cobalt as the divalent ion, is pink colored glass if in sixfold coordination with oxygen, and blue color when in fourfold.

Anderson, S., Bohon, R. L. and Kimpton, D. D., Infrared spectra and atomic arrangement in fused boron oxide and soda borate glass, J. Am. Ceram. Soc. 38, 370-377 (1955).

Bachman, G. S., Effect of coloring oxide additions on the spectral transmission of a soda-lime-silica glass, Glass Ind. 28 [12], 630-34, 652 (1947).

Visible and infrared transmissions of coloring oxides Va, Cr, Cu, Fe, Co, Ni, Nd, Pr, Sm, and U in a base glass were investigated.

Bachman, G. S., Infrared study of the structure of glasses prepared by melting crystals, Glass Ind. 29 [8], 435-37 and 466 (1948).

Bailey, A. C. and Woodrow, J. W., The phosphoresence of fused quartz, Phil. Mag. 6, 1104 (1928).

Bamford, C. R., Study of the magnetic properties of iron in relation to its coloring action in glass I and II, Phys. and Chem. Glasses 1, 159-64 and 165-69 (1960).

Good coverage Fe^{+2} and $^{+3}$. A complementary study was the optical transmission properties in the visible and near IR.

Bamford, C. R., Study of the magnetic properties of iron in relation to its coloring action in glass: III, Iron in sodium silicate glasses and sulfur-amber coloration, Phys. Chem. Glasses 2, 163-68 (1961).

Visible and near infrared spectra studied.

Bates, T. and Douglass, R. W., Absorption bands of Cr^{+3} ion in solutions, crystals and glasses, J. Soc. Glass Technol. 43 [213], 289-307T (1959).

Brewster, G. F. and Kreidl, N. J., Color of iron-containing glasses of varying composition, J. Soc. Glass Technol. 35 [167], 332-406T (1951).

Color changes caused by composition variations in iron containing silicate and silica-free glasses in terms of spectral transmission.

Bruckner, R. and Scholze, H., Infrared bands of freshly prepared and weathered B_2O_3 glass, Glastechn. Ber. 31, 417-22 (1958).

Brüninghaus, L., The red fluorescence of glass, J. phys. radium [7], 2, 398-402 (1931).

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Bues, W. and Gehrke, H. W., Raman and IR absorption spectra of molten glassy and crystalline high-polymer phosphates, Z. anorg. u allgem. Chem. 288 [5-6], 307-23 (1956).

Bues, W. and Gehrke, H. W., Vibrational spectra of metals, glasses and crystals of sodium di-, tri-, and tetra-phosphates, Z. anorg. u. allgem. chem. 288, 291 (1956).

$Na_4P_2O_7$
$Na_5P_3O_{10}$ glass and $Na_6P_4O_{13}$ glass

Buzhinskii, I. M. and Bodrova, N. I., Spectral study of CdS and CdS-CdSe colored glasses, Optika i Spektroskopiya 12, 387-95 (1962).

Canina, V. G., Optical absorption of energy levels in vitreous GeO_2, Compt. rend. 248, 1488-91 (1959).

Castner, T., Newell, G. S., Holton, W. C. and Slichter, C. P., Note on the paramagnetic resonance of iron in glass, J. Chem. Phys. 32 [3], 668-73 (1960).

Cleek, G. W. and Scuderi, T. G., Effect of fluorides on IR transmittance of certain silicate glasses, J. Am. Cer. Soc. 42 [12], 599-603 (1959).

Fluorides reduce the absorption at 2.75 μ water band, thus improving as IR material.

Cleek, G. W., Villa, J. J. and Hahner, C. H., Refractive indexes and transmittances of several optical glasses in the infrared, J. Opt. Soc. Am. 49, 1090 (1959).

10,000 - 2000 cm^{-1}
13 optical glasses (8 flint)

Cohn, B. E., Luminescence and crystalline structure, J. Am. Chem. Soc. 55, 953-7 (1933).

Zn borate glasses containing Mn show fluorescence when exposed to ultraviolet light. The spectrum shows a band from about 5670-6750 AU with a maxima at 6100 AU.

Colbert, W. and Kreidl, N. J., Unusual colors produced by uranium in glasses, Jour. Optical Soc. Amer. 35 [11], 731-35 (1945).

Describes different fluorescence observed.

Day, E. and Rindone, G. E., Properties of soda aluminosilicate glasses: I. Refractive index, density, molar refractivity, and infrared absorption spectra, J. Am. Ceram. Soc. 45 [10], 489-496 (1962).

Day, R. K., Glass Research Methods, Industrial Publications, Chicago (1953).

Useful optical and light transmission information are given.

De Jong, J., Influence of different elements on the color and magnetic susceptibility of glass, J. Soc. Glass Technol. 38 [181], 57-83T (1954).

I. Iron absorption in near infrared at 1.2 μ due to ferrous ions.
II. Titanium and chromium.

Dewulf, G., Infrared-transmitting glasses, Rev. opt. 33, 513-18 (1954).

As_2Se, As_2S_5 { 1 mm thickness, 50% transmission in range 0.6 to 13μ and .2 to 15μ

Douglass, R. W., Infrared observations of glass structures, in Progress in Ceramics, ed. Burke, John Wiley and Sons, New York, 210-221 (1961).

Florence, J. M., Allshouse, C. C., Glaze, F. W. and Hahner, C. H., Absorption of near-infrared energy by certain glasses, J. Research Nat. Bur. Standards 45 [2], 121-28 (1950), RP 2118.

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Most silicate glasses have a region of strong absorption at wave length of 2.7 μ. This is caused by the pressure of H_2O and CO_2 in the glass.

Florence, J. M., Glaze, F. W. and Black, M. N., Infrared transmittance of some calcium aluminate and germanate glasses, J. Research Natl. Bur. Standards 55, 231 (Research Paper 2625) (1955).

Florence, J. M., Glaze, F. W., Hahner, C. H. and Stair, R., Transmittance of near-infrared energy by binary glasses, J. Am. Ceram. Soc. 31 [12], 323-31 (1948).

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Florinskaya, V. A., Infrared reflection spectra of sodium silicate glasses and their relation to structure, "Structure of Glass," Proc. All-Union Conf. Glassy State, 3rd, Leningrad 1959, 154-68 (Pub. 1960).

Florinskaya, V. A. and Pechenkina, R. S., Reflection and transmission spectra of potassium silicate glasses in the infrared, Doklady Akad. Nauk S.S.S.R. 91, 59 (1953).

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Foëx, M., The infrared transparency of silica glasses prepared in the solar oven under varying atmosphere, Bull. soc. chim. France, 767 (1954).

3636 cm^{-1}

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Kumar, S., Optical absorption of glasses containing ions with partially filled 3d-orbitals: I. Absorption bands, Central Glass and Ceram. Research Inst. Bull. (India) 6 [3], 99-107 (1959).

Ultraviolet, visible and near infrared regions on alkali silicate and aluminoborophosphate glasses of Ti^{+3}, V^{+3}, Cr^{+3}, Mn^{+3}, Mn^{+2}, Fe^{+2}, Fe^{+3}, Cu^{+2}, Co^{+2} and Ni^{+2}.

The color is due to the presence of absorption bands in or around the visible region of the spectrum.

S state ions (Mn^{2+} and Fe^{3+}) show weak absorption bands. The d state ions (Ti^{3+}, Mn^{3+}, Fe^{2+} and Cu^{2+}) show one broad absorption band and the f-state ions (V^{3+}, Cr^{3+}, Co^{+2} and Ni^{+2}) show two or more absorption bands.

II. Energy levels, pp. 108-16. From the number and position of the observed absorption band due to Ti^{3+}, V^{3+}, Cr^{3+}, Mn^{3+}, Fe^{2+} and Cu^{2+} in alkali silicate glasses. The ions are probably in octahedral coordination with O^{2-}. In high B_2O_3 glasses Co^{2+} and Ni^{2+} ions are present in hexacoordination state. Soda silicate glasses Co^{2+} is present in tetrahedron coordination. Energy levels corresponding to most of the absorption bands are assigned when possible.

III. Coordination and structure, pp. 117-26.

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II. Detailed study of the absorption curves given by Ni in glasses and certain sintered masses. 250-86T.

III. Studies of the absorption spectra of Ni in various solutions and in certain organic compounds. Also the magnetic properties of Ni in relation to its state in the glass. 287-313T.

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8.5c. Structure of Glass

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8.6. Organic Absorption Spectra and Fluorescence

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8.7. Semiconductors, Optical Spectra and Related Optical Properties

This list of references were not collected for this bibliography but instead is a list that the author had made for another purpose, and is therefore not complete or up to date. It is being presented because of the laser action that has been reported in 8.1 (see Laser Section - Holonyak and Bevacqua, Nathan, et. al., and Hall, et. al.). It is based on a survey of Chemical Abstracts for the period 1950-1961 but is not complete even for these years. A few more recent articles are also included.

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8.8. Crystal Growth

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