

AN ABSTRACT OF THE THESIS OF

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Title: DESIGN AND CONSTRUCTION OF AN X-RAY DIFFRACTION  
TEACHING DEVICE

Abstract approved:

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A design for an x-ray diffraction teaching device has been presented. From the design a diffraction device has been constructed.

Based on the principles of the original work by Max von Laue, a diffraction camera has been constructed using a diagnostic x-ray machine. The diffraction camera provides a fast, safe, easy to operate means of obtaining useable Laue diffraction patterns.

The analysis of the diffraction pattern is carried out using the gnomonic projection. The diffraction spots are identifiable in the gnomonic projection. Though the patterns lack perfection, they provide visible proof to the student that x-ray diffraction has taken place.

The safety of the diffraction device is assured through proper shielding. The leakage rates do not exceed the limits set by the National Council on Radiation Protection and Measurement.

Design and Construction of an X-ray  
Diffraction Teaching Device

by

Donald Robert Elle

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Typed by Marion F. Palmateer for Donald Robert Elle

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Thanks to my wife for her willing hands and tireless patience.

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# DESIGN AND CONSTRUCTION OF AN X-RAY DIFFRACTION TEACHING DEVICE

## INTRODUCTION

X-ray diffraction is a tool employed in the investigation of the fine structure of matter. It had its beginnings with von Laue's discovery in 1912 that crystals diffract x-rays, the manner of diffraction revealing the structure of the crystal. In the beginning x-ray diffraction was used only for the determination of crystal structure. In the course of time other uses have been developed. The method is now applied not only to structure determination but to such problems as chemical analysis and stress measurement, the study of phase equilibria, the measurement of particle size, and the determination of the orientation of one crystal or the many orientations of a polycrystalline material.

Since World War II the numerous and varied applications of x-ray diffraction have established it as one of the most fundamental and important research tools. Both academic and industrial laboratories include x-ray diffraction as a common tool in their research areas and in process control. The development of excellent and economical commercial diffraction equipment makes the availability of the tool even greater. The frequent mention of x-ray diffraction in journals and papers renders the technique more widely useable to all.

Because x-ray diffraction is such a useful tool and basic concept, it can be assumed that in the course of a scientifically oriented education the student will, at some time, come across x-ray diffraction either in his reading or in course work. The introduction to the student of the principles of x-ray diffraction is very often made through lectures. The theory is presented and the uses may be mentioned, yet the utilization of it may remain as just that, the mention in a text or lecture unless the student goes onto more advanced courses. It is felt that a need exists for a device which can illustrate the principles of x-ray diffraction. What is required is an x-ray diffraction teaching device. The basic techniques and theory of x-ray diffraction and crystallography should be presented and the analysis of the derived results should be demonstrated to satisfy the student that x-ray diffraction is being observed, as was theorized.

In the X-ray Science and Engineering Laboratory at Oregon State University there is a course open to those of all disciplines with a minimal background in physics. The subject matter entails a general survey of x-ray theory, machines, and applications and is open to students with at least one year of physics. The course X-ray Applications, GS-363, is a lecture-laboratory course in which the applications of x-rays are discussed and put to use in practical experience. In such a course it would be desirable to be able to discuss and to demonstrate x-ray diffraction.

With this in mind, a thesis project was planned to develop an x-ray diffraction teaching device, one that is of an elementary nature and simple in its operation. The history and theory of x-ray diffraction are to be presented with the goal of developing the principles of x-ray diffraction for the student. An elementary device utilizing the original aspects of x-ray diffraction is to be designed which will demonstrate x-ray diffraction in its most elementary forms.

## HISTORICAL DEVELOPMENT OF X-RAY DIFFRACTION

On November 8, 1895 Wilhelm C. Roentgen, professor of physics at the University of Wurzburg, Bavaria, was experimenting with a cathode-ray tube when he discovered a startling phenomenon (18, p. 3-18, 29, p. 1-15, 50, p. 1-5). He observed that a barium platinocyanide screen lying near the tube gave off a fluorescent light. After several weeks of investigation he published his findings in his famous First Communication, which was presented before the Wurzburg Scientific Society. He had ascertained that the cause of the fluorescence was of the nature of a radiation. This radiation originated where the electron beam struck the walls of the tube or the anode of the tube and was of a highly penetrating nature. These rays he called x-rays. Roentgen also studied the various electrical effects produced, in particular the ability to ionize gases, and discharge charged bodies. He attempted to refract x-rays by prisms of various materials, and to obtain diffraction of x-rays through slits, but never achieved satisfactory or conclusive results.

After Roentgen's discovery and subsequent work, the question arose as to the nature of x-rays. Whether they were a corpuscular emanation or a wave phenomenon could not conclusively be stated. Very few fundamental facts were uncovered in the next 15 years. There was constant technical development of the methods of x-ray

production in response to the demands of the medical profession, but of experiments disclosing the nature of x-rays, there were only four of importance.

That x-rays were scattered had been shown by Roentgen (29, p. 11). Utilizing this fact Barkla (29, p. 11-15) in 1905 demonstrated that x-rays were polarized. He also found (29, p. 11-15, 39, p. 2-7) in 1909 that scattered x-rays were of two kinds. One kind were x-rays of the same nature as the incident x-rays; the second kind were softer and homogeneous, which he called characteristic x-rays, since they depended on the material being irradiated. The third experiment (29, p. 11-12) was based on the photoelectric effect, first noted in 1887 by H. Hertz and subsequently used in predicting the wavelength of x-rays and interpreting the ionization of gases by x-rays. The last of these four experiments was reported by Roentgen (29, p. 14) in his First Communication in 1895 as an inconclusive experiment to produce x-ray diffraction using a fine slit. Faint fringes were reported by Walter (29, p. 15) in 1909 but could not conclusively be shown to be diffraction effects. So it was in 1912. X-rays demonstrated corpuscular properties as well as wave properties. The discussion among scientists of the period continued along these lines, trying to find evidence that would lead to a conclusive statement about the nature of x-rays.

What about the knowledge of matter in 1912? The comparative

uniformity of the behavior of gases had been one of the discoveries of experimental philosophy and by the end of the nineteenth century the laws of gaseous behavior were known. Of liquids less was known. The general properties of solids had been studied, such as elasticity, fatigue, annealing and breaking strength. These were studied as properties but how they took place was not yet clear. The chemists knew about the behavior of atoms and molecules and the part they played in the behavior of materials, but the observations were primarily confined to gases and liquids. The solid state was a mystery.

Crystallography did exist as a science but was limited to an external or geometrical study of material. The properties of crystals, cleavage planes, glide planes, directional properties (electrical conductivity, thermal conductivity, thermal expansion, refractive index, and diamagnetic susceptibility all vary with direction in non-cubic crystals), were studied from the macroscopic point of view. These properties seemed to indicate that there was something in crystals which was regularly arranged in space. But a microscope did not illuminate the details to a great enough extent. This was reasonable since with light in the visible region an atom or molecule is too small to be resolved in a microscope of even the highest power, the limits of resolution being about  $1300 \text{ \AA}$  while the atomic distances are about  $2 \text{ \AA}$ . Such was the state of knowledge concerning matter in 1912.

From its earliest history the University of Munich prided

itself upon having the chairs filled by eminent professors. In 1912 W. C. Roentgen was the head of an Institute for Experimental Physics and A. Sommerfeld was head of the Theoretical Physics Institute (29, p. 30-39). Roentgen's Institute was the larger of the two and was very demanding in its requirements for all possible precaution against errors and wrong interpretation. Consequently, only the devoted and serious students graduated under Roentgen. Of those who graduated P. P. Kock, P. Knipping, J. Brentano and W. Friedrich were a few. Roentgen, who held the chair from 1900-1922 was always interested in work to clarify the nature of x-rays. Sommerfeld's smaller Institute was an academic novelty. Dealing with theoretical physics, he requested and got experimental equipment for supporting the line of theoretical work. In 1911 Sommerfeld appointed W. Friedrich, who graduated from Roentgen's institute, as second assistant. He dealt primarily with x-rays. The first assistant was P. Debye, an outstanding physicist and mathematician. In 1909 Max von Laue, a student of Planck, joined Sommerfeld's group after he had completed his post-doctoral work. His interest covered all of physics and he wrote a monograph on the Special Theory of Relativity and in 1911 published work on wave optics.

In 1910 a student of Sommerfeld, Paul Ewald, was working on a problem for his doctorate on the optical properties of an anisotropic (different properties in different directions) arrangement of

isotropic (same properties in the same directions) resonators (29, p. 37-41). Before completion of the thesis Ewald was discussing his conclusions and asking for helpful criticism from Laue, for Laue had a strong leaning toward fundamental physical issues. A discussion of a theory of crystal structure arose which illuminated the idea that crystals were arranged in a lattice array, the distances between points being about  $1/500$  to  $1/1000$  of the wavelength of visible light. Subsequent to the discussion Ewald submitted his thesis and was awarded a Doctorate in March of 1912. Some time later Ewald read of Laue's discovery of x-ray diffraction.

From the discussion with Ewald, Laue had begun to consider what would happen to short wavelengths in a crystal. If the wavelength was of the same order as the atomic distances, then, reasoned Laue, some sort of diffraction effect would be expected to occur. Despite objections by Sommerfeld, W. Friedrich and P. Knipping were assigned the task of trying to verify Laue's hypothesis (29, p. 40-47, 39, p. 7-13). With Friedrich's experience in x-rays, an experimental arrangement was arrived at consisting of a lead lined box which contained a photographic plate, a crystal specimen and a hole through which x-rays could enter and strike the crystal. The source of x-rays, a gas tube, was not of high intensity which necessitated long exposures. In the initial attempt the film was placed between the crystal and the x-ray source which produced no diffraction

pattern. The second attempt had the film behind the sample, a copper sulfate crystal, which yielded positive results in the way of a distorted diffraction pattern. A schematic diagram of the apparatus is shown in Figure 1.

Later experiments with a more symmetric crystal and more advanced experimental equipment led to better results. Laue was able to express the theory of x-ray diffraction by applying the equation of the theory of diffraction from an optical grating twice over to get diffraction from a lattice. This theory will be discussed in detail later.

The work of Laue, Friedrich, and Knipping (29, p. 47-56) was reported to the Bavarian Academy of Sciences on June 8, and July 6, 1912 by Sommerfeld. At the same time Laue reported his work to his former co-workers in Berlin on June 8, 1912.

Laue's discovery was followed in the same year by the work of W. H. and W. L. Bragg (29, p. 57-73, 39, p. 13-23). W. H. Bragg, the father, worked at Cambridge while his son W. L. Bragg worked at Leeds, both in England. They attempted to explain Laue's discovery in more concise terms and concluded that x-ray diffraction could be explained by considering the x-rays to be reflected from the lattice planes of a crystal. W. L. Bragg, (29, p. 62) takes credit for the conception of the reflection theory and the application in general of the optical principles of diffraction. He also was the crystal

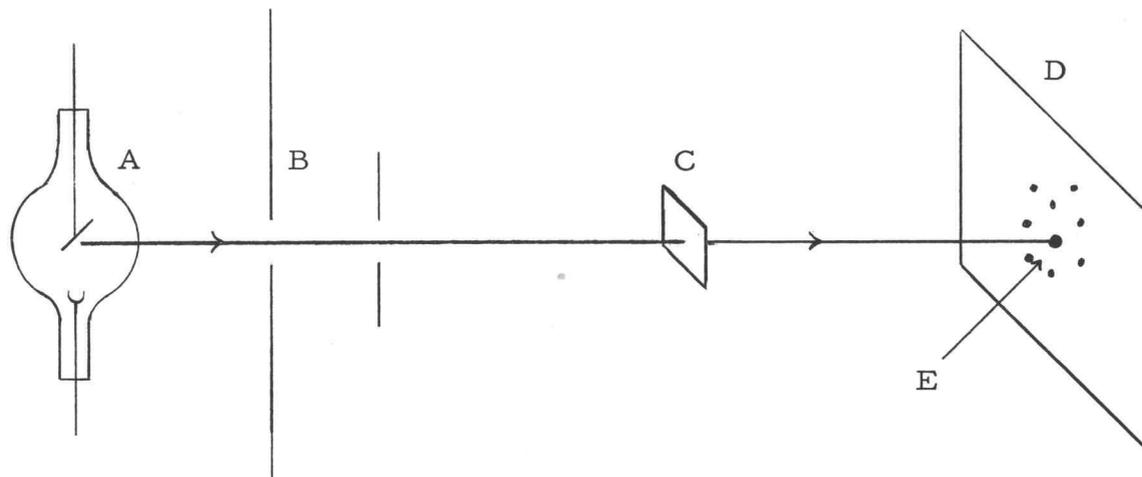


Figure 1. Schematic diagram of the Laue apparatus showing the x-ray source A, the shield and collimator B, the crystal specimen C, and the photographic film D with the diffraction spots E.

structure analyst of the two. W. H. Bragg (29, p. 61) was more interested in x-rays than crystals. He devised an instrument known as an x-ray spectrometer, utilizing an ionization chamber to measure the intensity of the reflected x-rays from a crystal face. With this instrument he found that each metal used as a target in the x-ray tube gave a characteristic spectra of definite x-ray wavelengths, much as elements give spectra in the optical region.

The x-ray spectrometer proved to be a powerful tool. One could examine the various faces of a crystal, noting the angles at which and the intensity with which they reflect x-rays. The structure of the crystal could be deduced from this. The emission of characteristic x-rays, later to be elaborated upon by Moseley (40, p. 83) and Siegbahn (49) was noted. This instrument was a powerful tool for chemical analysis and is in extensive use today in various forms.

These discoveries by the Braggs were published in a series of letters and papers starting in October 1912 and running until World War I started in August 1917 (4, 5, 6, 7, 8, 9, 10, 11). The subjects covered ranged from crystal structure to x-ray reflection.

Other workers in England who contributed much to the early development of x-ray diffraction were Moseley (29, p. 74) and C. G. Darwin (24). The work on characteristic x-rays by Moseley was of much importance. C. G. Darwin, in 1914, published two fundamental papers, one on x-ray reflection and the other on a dynamical theory

of x-ray diffraction.

In France, the early work on x-ray spectroscopy was done by Maurice de Broglie (29, p. 75) and in November 1913 a paper describing the first rotation camera was published. The method of rotation photographs, note Figure 2, which utilizes monochromatic x-rays impinging on a rotating single crystal, was first applied specifically to the analysis of crystals by E. Schiebold (25, p. 210) and described in 1919. It was further developed by Schiebold and Polanyi and Weissenberg (25, p. 210) in 1921 at which time the recording film was displaced simultaneously with the crystal rotation, note Figure 3. This method is known as the Weissenberg method and has been used extensively in crystal analysis since 1924.

The last primary method of x-ray diffraction is known as the powder method. In rotating single-crystal methods, the motion of the crystal brings various planes into proper orientation for diffraction. In the powder method, the orientations are simultaneously present because the monochromatic beam is directed onto a finely divided, randomly oriented powder, note Figure 4. The powdered sample is usually rotated to ensure random orientation, however. Not long after Laue's initial discovery the first halos of the powder type were observed by Fredrich (29, p. 77) when x-rays were passed through various waxes and by Keene (29, p. 333) when x-rays passed through thin metal foils. Neither of these two arrived at a proper

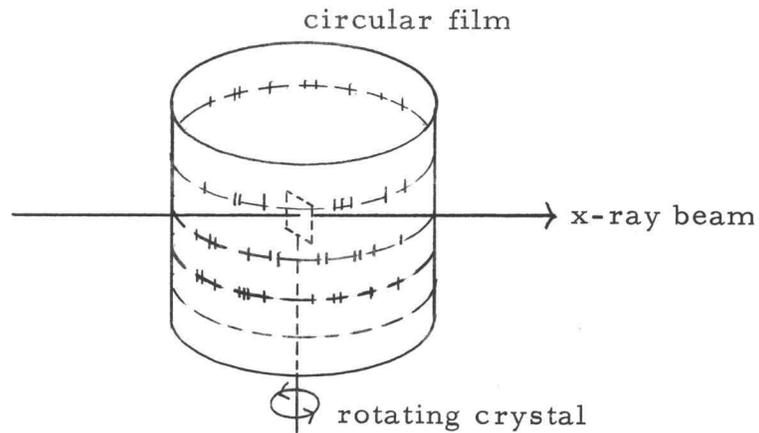


Figure 2. Schematic representation of relationship of beam-crystal-film in the rotation method. One crystal axis is parallel to the rotation axis yielding a pattern of lines.

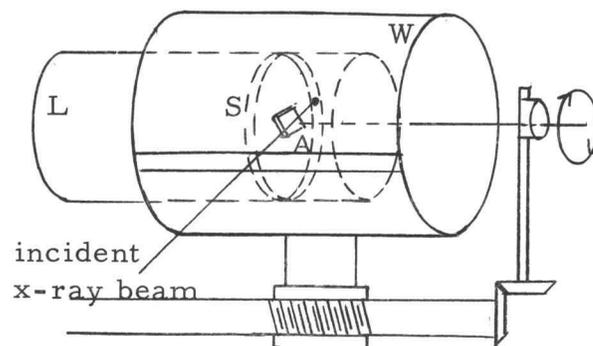


Figure 3. Schematic diagram of Weissenberg apparatus. A is a rotating crystal inside the diaphragm L with slit S through which one layer line passes. The film holder W is displaced in a horizontal direction synchronously with the rotating crystal.

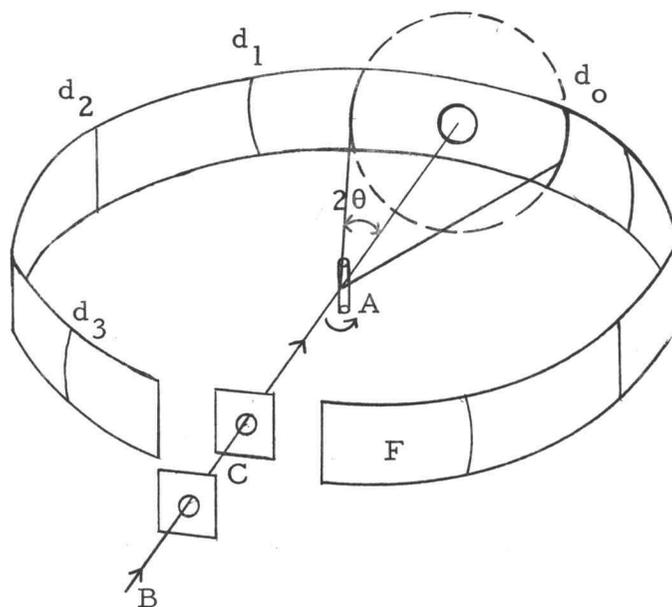


Figure 4. Schematic diagram of the powder method. A is the cylindrical shaped rotating powder sample which x-ray beam B strikes after passing through the collimators C. The sections of the various diffracted cones  $d_n$  are recorded on the film F.

interpretation of the results, however. In Japan the halos were noted by Nishikawa (29, p. 77), who early in the history of x-ray diffraction was working on diffraction effects in partially ordered material, and by Hupka (29, p. 77), but these two did not hit upon the powder method. This difficulty can be explained by the fact that none of these men had available strong sources of monochromatic x-rays. The powder spectrograph was originated by Debye and Scherrer (50, p. 341) in Germany in 1916 and independently by A. W. Hull (34) of the General Electric Company in the United States in 1917. These two had the insight and understanding to recognize the significance of the halo effect and to interpret it correctly. The method came into general use some six or eight years before the method of rotation analysis.

With these developments the first period in the history of x-ray diffraction ended. This period began with Laue's discovery in 1912 and ended in 1921. It included the First World War period during which little research could be done. This period laid a foundation on which all subsequent work was based. The basic discoveries had been made, it remained for them to be implemented, utilized, and elaborated upon.

Broken into decades, the development of the science of x-ray diffraction can be followed with relative ease, but with little detail (29, p. 120-135).

The time from the discovery of x-ray diffraction in 1912 to

1920 has already been mentioned. Let it be said again that it was during this time that the foundation of the art and science of x-ray diffraction was laid. It was during this period that initial structure analysis was carried out on simpler materials.

The next decade 1920-30 witnessed x-ray diffraction analysis taking form as a quantitative science with analysis being extended to more complex substances, particularly inorganic crystals. Darwin's theory made it possible to measure absolute intensities of diffracted rays, which aided in the analysis of complex materials. In 1928 Pauling published his paper on the "Coordination Theory of the Structure of Ionic Crystals" which stated the fundamental principles of inorganic crystal structure. The effective use of Fourier representation was accomplished in one dimension, and two-dimensional analysis was begun.

The decade from 1930-1940 saw the Fourier series develop into a standard method of analysis. It added geometrical precision to the molecules of organic chemistry. The powder method was also developed to a high degree in the analysis of alloys.

Excluding a five year period of the Second World War during which little research could be performed, the next decade, 1945-1955, was characterized by the so-called perfection of the trial and error methods of x-ray analysis and the development of techniques and equipment to a high degree of perfection.

From 1955 to the present great strides have been made. Trial and error methods are no longer satisfactory for the solution of such complex biological structures as the spiral structure of the polypeptide chain, and proteins. With the use of computers, which can handle massive amounts of data, the solution becomes one of interpretation of the answers produced.

It should not be forgotten that one of the greatest contributions from which all fields of x-ray diffraction benefited was that of William D. Coolidge (21) of the General Electric Research Laboratories. He developed the hot cathode x-ray tube which utilizes high vacuum techniques. This development allowed constant production of x-rays over a long period of time and facilitated the great advances made in x-ray diffraction.

## CRYSTALLOGRAPHY

X-ray diffraction is a basic scientific tool which can supply the researcher with fundamental data and information unattainable by other methods. Complete realization of its potentialities requires an elementary knowledge of crystallography.

One can define crystals (1, p. 1-70, 23, p. 1-10, 39, p. 50-52) in any of several ways. A crystal is the regular polyhedral form, bounded by smooth surfaces, which a chemical compound under the influence of its interatomic forces takes when passing from the liquid or gaseous state into a solid state. A crystal is a homogeneous solid possessing long range three dimensional internal order. The most fundamental fact about crystals and crystalline matter revealed by x-rays, is that the particles of which it is composed, atoms or molecules, are arranged in a regular and repeated pattern. A crystal can be pictured as being built up of a large number of small units arranged in a repetitive three dimensional array.

A consequence of this is that on a microscopic level crystals are heterogeneous or anisotropic. They are not generally the same in non-parallel directions. Certain properties of crystals will show variation with directions. These are known as directional or vectorial properties. Some of these properties are hardness, conductivity of heat or electricity, thermal expansion, growth rate,

symmetry, and diffraction of x-rays, electron, and neutrons.

The diffraction property is an example of a discontinuous vectorial property of a crystal.

### Elements of Crystallography

The properties and characteristics of crystals and crystal geometry are discussed in complete detail by several authors in texts and various publications (15, p. 11-60, 16, p. 1-140, 37, p. 1-150, 47, p. 34-60, 53, p. 21-50, 55, p. 1-14, 58, p. 21-50, 59, p. 1-70). What is presented here is a general survey and summation with little detail and few examples of what has been related in these references.

One of the first characteristics that is noticed about a crystal is the fact that the arrangement of its components is such that certain operations can be performed on the crystal which will bring it into coincidence with itself. These are termed symmetry operations. There are in effect, three microscopic symmetry operations, rotation, reflection, and inversion. In the first, the crystal possesses an axis of symmetry. If rotated around an imaginary line through its center it presents the same appearance more than once during a complete rotation. The line is the symmetry axis of rotation. It can be shown that there are but four possible axes. These are twofold, if the structure is repeated at  $180^\circ$ , threefold, if it is repeated at  $120^\circ$ ,

fourfold, if it is repeated at  $90^\circ$ , and sixfold if it is repeated at  $60^\circ$ . A rotation of  $360^\circ$  is called the identity operation for obvious reasons. In the second, a plane about which a crystal is symmetrical is called a symmetry plane. A symmetry plane so divides a crystal that a face on one side is matched by a face on the other side as its mirror image or reflection. The maximum number of symmetry planes is nine. This occurs when each face is matched by a parallel face in the antipodal position. In the third case it is said that a body has an inversion center if corresponding parts of the body are located at equal distances from the center of a line drawn through the center.

In discussing crystals it is necessary to possess a means of designating the various faces without ambiguity. This is done with the use of coordinate axes of reference. Any three noncoplanar edges of the crystal or directions in the crystal may be chosen for the crystallographic axes. A set of axes is chosen which bears as close a relationship to the general symmetry as is possible. The origin is thought of as being at the center of the ideal crystal. The unit lengths are fixed by the symmetry assuming some face to be the unit face.

By convention, a right-handed set of axes is used with the  $x$  axis toward the observer, positive  $y$  to the right and positive  $z$  vertical. In Figure 5 is a representation of the crystallographic axes. After selecting the axes, some plane intersecting all three axes is

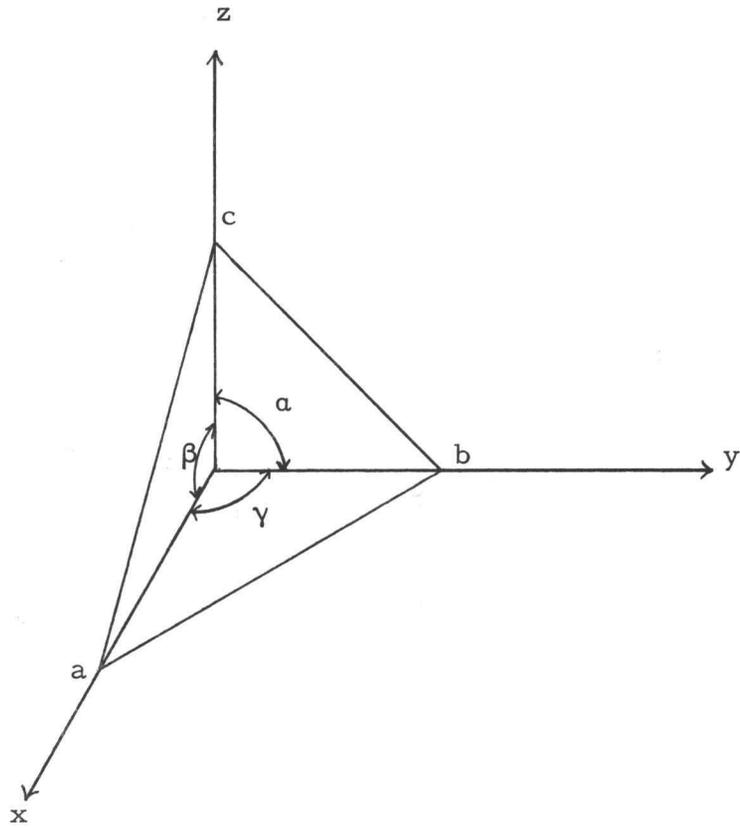


Figure 5. The crystallographic axes.

chosen as the unit plane. The points of intersection are often called  $a$ ,  $b$ , and  $c$ . The angles between the axes are defined as  $\alpha$ ,  $\beta$  and  $\gamma$ . In practice the correct selection of the crystallographic axes identifies the crystal as belonging to one of seven large symmetry groups known as the seven crystal systems. The names of these systems and their characteristics are listed in Table 1.

Once the choice of a unit plane has been made it is possible to express the intercepts of all other planes as  $m a$ ,  $n b$ , and  $p c$ , where  $m$ ,  $n$ , and  $p$  are small integers or infinity. This is always possible since a face may be moved parallel to itself without changing the characteristics of it. These numbers could be used as indices to define the location of a plane, but it is more convenient to use as indices numbers proportional to the reciprocals of  $m$ ,  $n$ , and  $p$ . These are called Miller indices and are derived from the parameters by their inversion and subsequent clearing of fractions. They are expressed as integers without a common divisor. The Miller indices are written as  $(hkl)$  and negative indices as  $(\bar{h} \bar{k} \bar{l})$ .

Another term of interest in crystallography is form. Form in its most familiar meaning is used to indicate general outward appearance. However, external shape is denoted as habit in crystallography and form consists of a group of crystal faces all of which have the same relation to the elements of symmetry and display the same chemical and physical properties because all are underlain by

Table 1. Characteristics of the seven crystal systems.

Crystal System	Relative Axial lengths Axial angles	Bravais Lattice
Cubic	$a = b = c$ $\alpha = \beta = \gamma = 90^\circ$	Primitive Body-centered Face-centered
Tetragonal	$a = b \neq c$ $\alpha = \beta = \gamma = 90^\circ$	Primitive Body-centered
Orthorhombic	$a \neq b \neq c$ $\alpha = \beta = \gamma = 90^\circ$	Primitive Body-centered Base-centered Face-centered
Rhombohedral	$a = b = c$ $\alpha = \beta = \gamma \neq 90^\circ$	Primitive
Hexagonal	$a = b \neq c$ $\alpha = \beta = 90^\circ \quad \gamma = 120^\circ$	Primitive
Monoclinic	$a \neq b \neq c$ $\alpha = \gamma = 90^\circ \neq \beta$	Primitive Base-centered
Triclinic	$a \neq b \neq c$ $\alpha \neq \beta \neq \gamma \neq 90^\circ$	Primitive

the same atoms in the same geometrical arrangement.

Crystal planes and faces are often observed to be arranged in belts around the crystal, with all faces in a given belt parallel to an imaginary line or direction passing through the center of the crystal. The belts are called zones, and the line to which they and their intersections are parallel is the zone axis. The notable thing about a zone is the direction of its zone axis, expressed as  $[uvw]$ . This may be obtained from the indices of two planes in the zone,  $(hkl)$  and  $(h'k'l')$ , from the relations

$$u = kl' - lk'$$

$$v = lh' - hl'$$

$$w = hk' - kh'.$$

Up to now topics from the field of geometrical crystallography have been discussed and nothing has been said about actual crystals and the atoms of which they are composed. The crystal must be looked on as an orderly arrangement of points which represent atoms or molecules. This arrangement would make a three dimensional network called a space lattice. The space lattice can be defined as a regular and unlimited distribution of identical points in space, such that a straight line drawn through two adjacent points of the network and continued will intersect at equal intervals the same succession of points. The environment about any particular point is the same as that about any other point.

By joining the points of a space lattice, a series of parallel sided unit cells are produced, each of which contains a complete unit of the crystal pattern. The whole structure is obtained by packing these side by side in space. The space lattice is completely defined by stating the distances between neighboring points along the three directions and the angle between these directions. These distances are referred to as the primitive or unit translations of the lattice. The measured edge lengths of the unit are commonly designated  $a_0$ ,  $b_0$ , and  $c_0$ .

In dividing space by the set of three axes, it is possible to produce unit cells of various shapes which can be called point lattices, which correspond to the seven crystal systems mentioned previously. There are, however, other arrangements of points which satisfy the requirements of the point lattice, namely that each point have identical surroundings. Bravais (12), the French crystallographer, showed in 1848 that there are 14 possible point lattices and no more. These 14 are called Bravais lattices and are listed in Table 1.

Having discussed symmetry operations earlier, it can now be noted that there are various combinations of symmetry operations that may be present in the 14 Bravais lattices. In fact there are 32 possible ways of combining symmetry elements about a point and each is called a point group or crystal class. If a crystal is well formed and has enough faces, optical examination can place it in one

of the 32 classes by defining the symmetry and obtaining the angular relationships between faces. So it is seen that the symmetry of a finite body is that of a point group.

A further elucidation of the crystal structure is given by space groups. A space group is built up from point arrays by considering translatory movements which distribute the latter in space in a manner consistent with the symmetry of the various point groups. X-ray diffraction provides the evidence to show the nature of space symmetry, the operations of which are screw axes, a combination of rotation and translation, and glide planes, a combination of reflection and translation. These elements of space symmetry applied to the 32 crystal classes give a maximum of 230 possible combinations. Each one is called a space group.

In both optical and x-ray crystallography the characteristics of crystals are conveniently discussed in terms of planes, as has been explained. Lattice points can be included in plane surfaces drawn through them. A large number of planes with different orientations can be drawn. Parallel to any plane there is a set of planes which can be generated by lattice translations. Knowing this, and using the principles of analytic geometry, the important relations between planes can be derived. These are the equation of a plane and the interplanar distance. The equations of a plane can be determined through the use of the Miller indices. The interplanar spacing can

be determined from analytic geometry and the Miller indices and is denoted by  $d_{hkl}$ . The value of  $d_{hkl}$  will vary with each crystal system, becoming more complex as the crystal system becomes less symmetrical. In Table 2 are the seven equations for the seven systems.

### Crystallographic Projections

Since the sizes and shapes of the faces of crystals are merely incidental, their variations serve only to obscure the true symmetry relationships. Crystal drawings made in perspective or in the form of plane and elevation are not suitable for displaying the angular relationship between lattice planes and directions. A discussion of such relationships is carried out most conveniently in terms of some representation of the crystal in which each face or plane is represented by a dot, which has neither size nor shape but has the particular angular relationships with respect to other dots in the pattern. This method is generally called a crystal projection (2, p. 1-46).

If, from an origin within the crystal, normals to the faces are drawn, the normals will radiate from the origin in directions depending on the interfacial angles, which correspond to the angles between lattice planes. If a sphere is drawn around the crystal and the intersection of sphere and normal are represented by dots, the pole of the corresponding face, we have a spherical projection (47, p. 18-25)

Table 2. Formulas for calculating values of  $d_{hkl}$  for the seven crystal systems.

Crystal System	a:b:c	$d_{hkl}$
Cubic	1:1:1	$\frac{a_o}{\sqrt{h^2 + k^2 + l^2}}$
Tetragonal	1:1:c	$\frac{a_o}{\sqrt{h^2 + k^2 + (l/c)^2}}$
Orthorhombic	a:1:c	$\frac{b_o}{\sqrt{(h/a)^2 + k^2 + (l/c)^2}}$
Rhombohedral	1:1:c	$\frac{a_o \sqrt{1 + 2 \cos^3 a - 3 \cos^2 a}}{\sqrt{(h^2 + k^2 + l^2) \sin^2 a + 2(hk + hl + kl)(\cos^2 a - \cos a)}}$
Hexagonal	1:1:1	$\frac{a_o}{\sqrt{4/3(h^2 + hk + k^2) + (l/c)^2}}$
Monoclinic	a:1:c	$\frac{b_o}{\sqrt{\frac{(h/a)^2 + (l/c)^2 - \frac{2hl}{ac} \cos \beta}{\sin^2 \beta} + k^2}}$

Table 2. Continued

Crystal System	a:b:c	$d_{hkl}$
Triclinic	a:l:c	$\frac{b_o}{\sqrt{\begin{vmatrix} h/a & \cos\gamma & \cos\beta \\ k & 1 & \cos\alpha \\ 1/c & \cos\alpha & 1 \end{vmatrix} + k \begin{vmatrix} 1 & h/a & \cos\beta \\ \cos\alpha & k & \cos\alpha \\ \cos\beta & 1/c & 1 \end{vmatrix} + 1/c \begin{vmatrix} 1 & \cos\gamma & h/a \\ \cos\gamma & 1 & k \\ \cos\beta & \cos\alpha & 1/c \end{vmatrix}}}$

of the crystal. Such a projection is difficult to visualize since it is three dimensional.

There are in use today several methods of projection in one dimension based on the spherical projection. The first and most widely used is the stereographic projection (22, p. 60-77). The second, used in more elementary cases and to be used in this work, is the gnomonic projection (3, p. 88-99, 25, p. 65-78, 45, 57, p. 124-144).

The primary use for gnomonic projection arises in the analysis of Laue photographs, the production and theory of which are discussed in a later chapter. The spots in a Laue transmission photograph lie on ellipses that pass through the central point, all spots on any ellipse being reflections from planes of one zone. The most convenient method of interpreting a Laue photograph is to employ a projection that transforms these ellipses into straight lines. The gnomonic projection does this and is widely used for this purpose.

In the gnomonic projection, face-normals are drawn from the center of the crystal until they meet a plane, the points so obtained being the gnomonic poles. As an example consider Figure 6 in which the path of x-rays producing a diffraction spot is  $OCC'$  and the diffracted ray  $CA$  is reflected by a plane with a trace  $DB$ , perpendicular to the plane of the paper. In Figure 7 a gnomonic projection of the previous figure is constructed. The x-rays strike a crystal

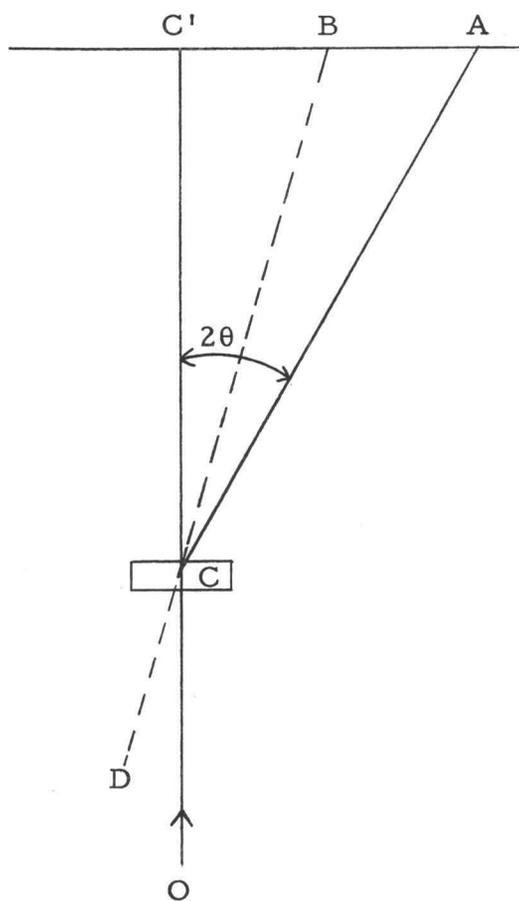


Figure 6. The diagrammatic representation of the path of x-rays  $OCC'$  producing a Laue spot  $A$  from the crystal plane  $DB$ .

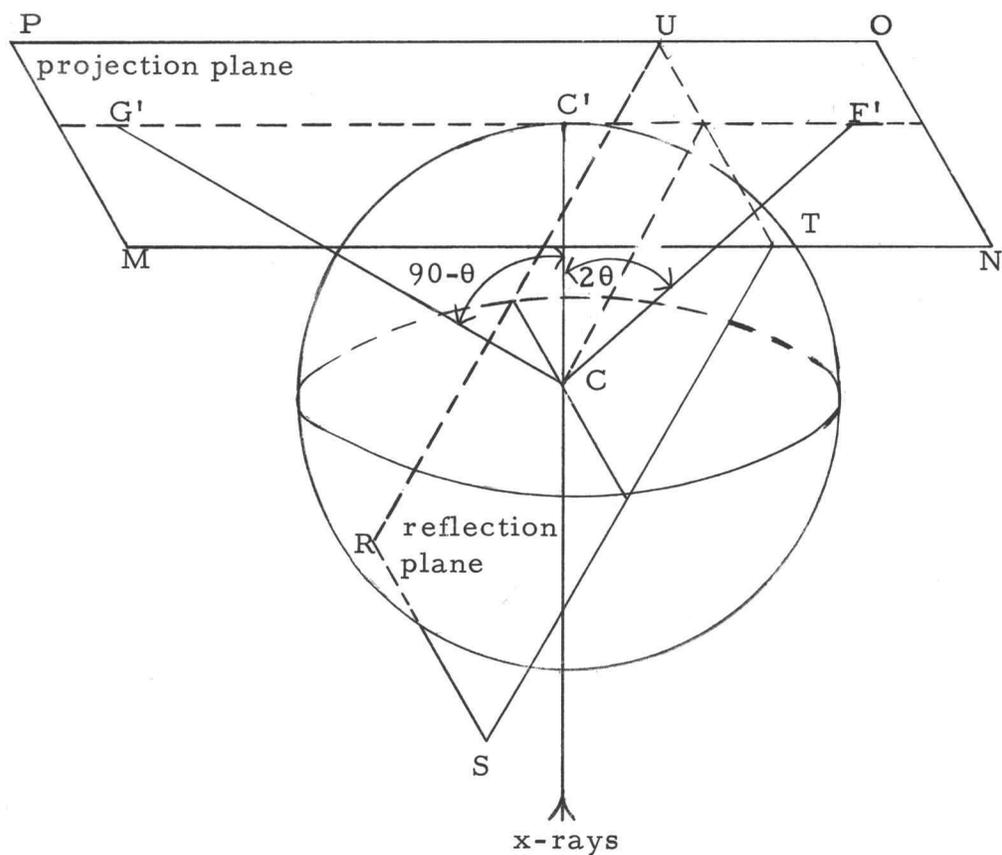


Figure 7. The gnomonic projection of a Laue spot. The x-rays incident at right angles to the projection and photographic plane  $MNOP$  are reflected by the crystal plane  $RSTU$  producing the Laue spot  $F'$ . The gnomonic projection of the plane  $RSTU$  is at  $G'$ .

at C and are reflected by a plane RSTU, a diffraction spot being located at the intersection of MNOP and the ray at F'. The gnomonic projection of this plane upon MNOP, the plane of projection, is G'. The planes G'C and CC' and CF' are coplanar. The diffracted spot and its gnomonic projection are related by the following equations (58, p. 126-133)

$$G'C' = CC' \cot \theta$$

$$F'C' = CC' \tan 2\theta.$$

In crystallographic use, it is customary to describe the normal to a face by stating its longitude, and polar distance from an arbitrary zero position. This information allows a plot of the normal to be constructed. However, using the equations above, a plot can be made of the projected pattern. Complete discussion of the use of this projection in the analysis of diffraction patterns is to be found in the next chapter.

## X-RAY DIFFRACTION

To use a tool such as x-ray diffraction it is not always necessary to understand what is happening. Our goal, however, is to understand x-ray diffraction and explain and illustrate it for the student. Consequently it is necessary to know some of the properties of x-rays (18, p. 64-180, 50, p. 20-170, 51, p. 20-130), and diffraction (26, p. 162-236, 42, p. 279-359), and to combine the two to get x-ray diffraction (22, p. 68-100, 31, p. 23-43, 36, p. 111-125, 42, p. 279-359, 54, p. 6-24, 59, p. 83-100). The theory of x-ray diffraction according to Laue (50, p. 320-323) and the simpler theory of Bragg (10) should also be known, as well as the methods of production and analysis of Laue diffraction patterns.

### X-rays

X-rays are a form of electromagnetic radiation. Electromagnetic radiations consist of oscillating electric and magnetic fields which vibrate at right angles to one another, as in Figure 8. Electromagnetic waves are most easily characterized by their wavelength or frequency. The wavelength  $\lambda$ , is the distance from a point on the wave to the corresponding point on the next wave, as in Figure 8. The wavelength of electromagnetic radiation covers a broad spectrum between  $10^2 \text{ \AA}$  to  $10^{-2} \text{ \AA}$ . All electromagnetic waves travel with the

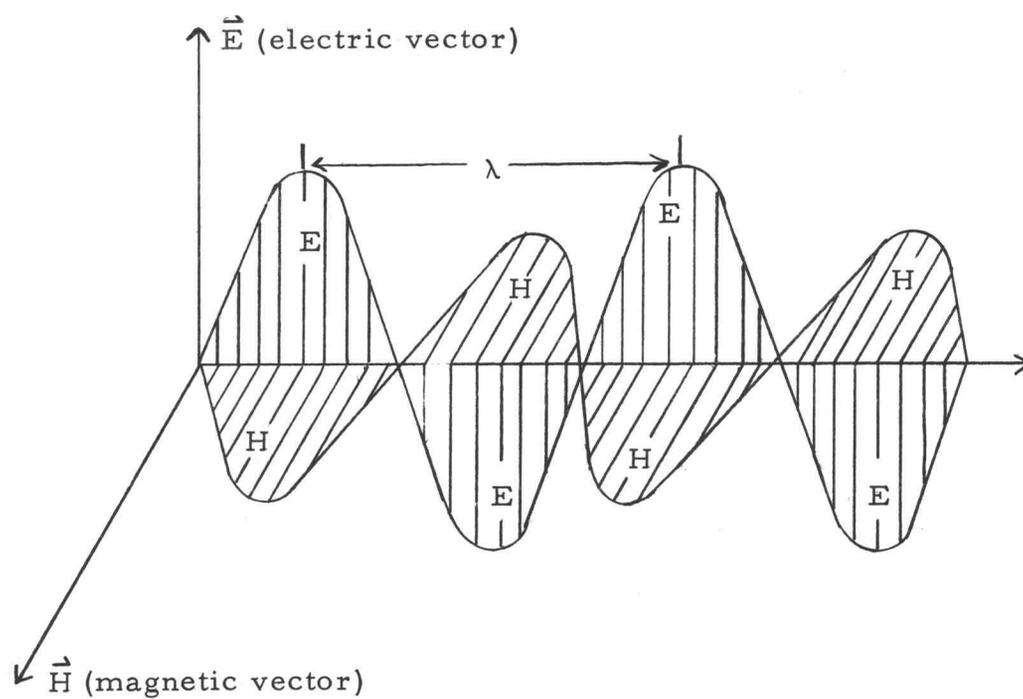


Figure 8. A diagrammatic representation of a plane polarized electromagnetic wave.

same speed in free space. The frequency  $\nu$ , of the vibration is the number of vibrations occurring in one second. The velocity can be then expressed as

$$c = \nu \lambda$$

where  $c$  is the speed of light in free space,  $3 \times 10^{10}$  cm/sec.

When electromagnetic radiation is emitted from an object, it is emitted discontinuously, not as a steady stream but as a large number of wave packets. These wave packets are known as quanta or photons of radiation. When electromagnetic radiation is emitted, a large number of quanta are emitted and each one of these quanta carries a specific amount of energy.  $E$ , given by

$$E = h\nu = \frac{hc}{\lambda}$$

where  $h$  = Planck's constant. One could conceivably consider a photon as a particle but not in the ordinary sense. It can travel at one speed only and if it is stopped by an interaction with an atom, then the whole of the quantum disappears and reappears either as energy absorbed by the atom or as other quanta of radiation.

X-rays are a form of electromagnetic radiation that can be produced artificially. The production requires that electrons be accelerated by some means and then strike a target. The interaction between the electrons and the atoms of the target material, in which the electrons are rapidly decelerated, results in the emission of

Bremsstrahlung or x-rays. The modern x-ray tube has a filament, or cathode, which provides a source of electrons when it is heated, and a target, or anode, all enclosed in an evacuated enclosure. A high voltage is applied between the filament and the target to accelerate the electrons which strike the target, producing x-rays.

When the x-rays produced are analyzed, it is found that a continuous spectrum of energies is produced. This is due to the fact that the electrons in slowing down do not all lose their energy in the same manner. Some may lose their kinetic energy in one interaction while others may lose it in several interactions. This spectrum of wavelengths can be represented by a curve such as that shown in Figure 9. The smooth curve represents continuous or white radiation and is due to electrons losing their energy in different manners. The maximum energy of an electron is given by

$$E = eV \text{ joules}$$

where  $e$  = charge on electron and  $V$  = accelerating voltage. The energy of the x-ray quanta will be  $h\nu$  so we have

$$h\nu_{\max} = eV.$$

There is, therefore, an upper limit of frequency possible for a given voltage  $V$ , which means there is a minimum value of  $\lambda$ , which is

$$\lambda_{\min} = \frac{hc}{eV} = \frac{12396}{V} \text{ with } \lambda \text{ in } \text{\AA} \text{ and } V \text{ in volts.}$$

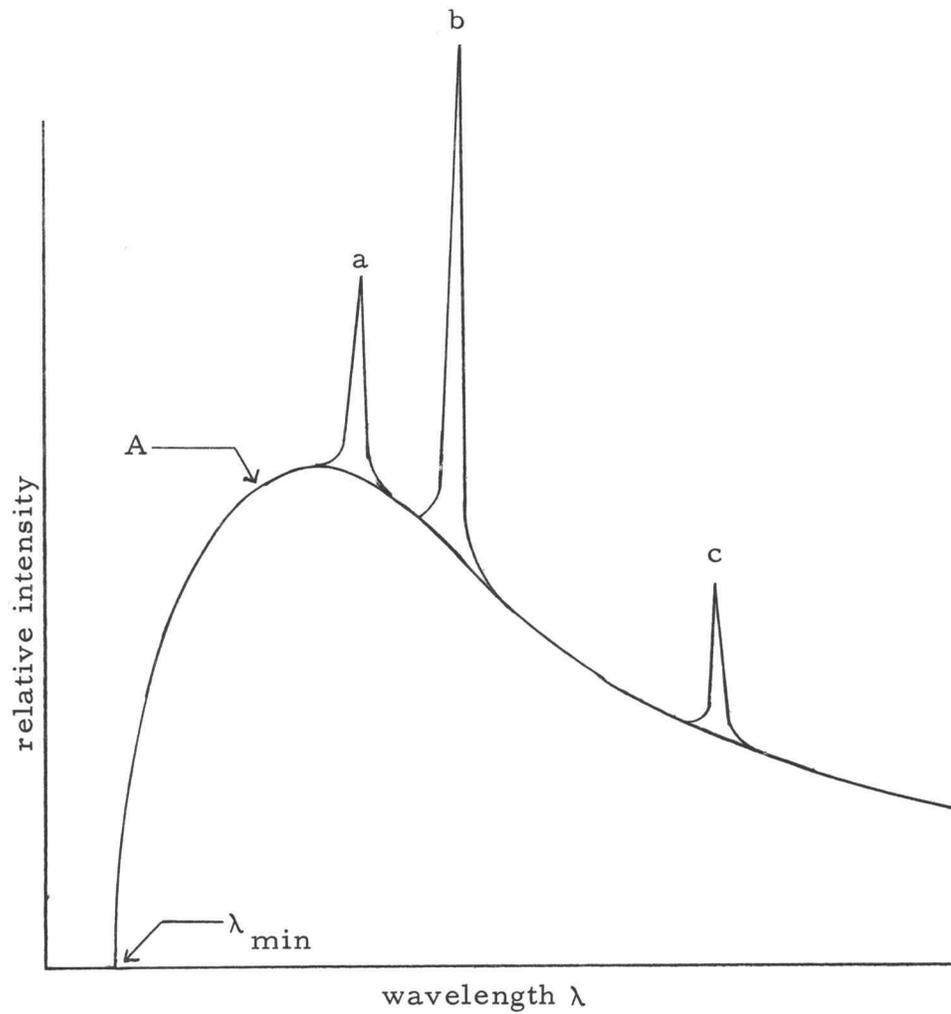


Figure 9. The energy spectrum produced by an x-ray tube. The continuous radiation A, with the characteristic x-ray peaks a, b, and c superimposed on it are shown.

That is why there is a sharp cut off in the spectrum of Figure 9.

When the voltage of an x-ray tube is raised above a certain critical value, characteristic of the target metal, sharp peaks in intensity appear at certain wavelengths superimposed on the continuous spectrum, as in Figure 9. These are called characteristic x-rays, and fall into several sets, referred to as K, L, M, etc. The origin of these lines lies in the atoms of the target material itself. They arise when an incident electron has enough energy to knock an electron out of an orbital shell, leaving the atom in an excited state. One of the outer electrons falls into the vacancy emitting energy in the process. The emitted energy is of a definite wavelength and is the characteristic x-ray. This is why there is a critical excitation voltage, enough kinetic energy must be present to remove the orbital electron. In the case of a tungsten target, characteristic peaks arise at  $0.18 \text{ \AA}$  and  $0.21 \text{ \AA}$ , produced at voltages near 60 kV.

Ability of x-rays to penetrate opaque substances is their most striking property, yet they are absorbed to a certain extent in passing through matter. The equation

$$I = I_0 e^{-ux}$$

where  $I$  = intensity after absorption,  $I_0$  = original intensity,  $u$  = absorption coefficient, and  $x$  = thickness of absorber, expresses the manner of absorption.  $I$  and  $I_0$  are measurable quantities

while  $u$  is a constant for a given wavelength in a material. A more useful form of the equation is

$$I = I_0 e^{-u/\rho (\rho x)}$$

where  $\rho$  = density of material and  $u/\rho = u_m$  = mass absorption coefficient. The mass absorption coefficient is particularly useful because for x-rays it is constant for a substance at a given wavelength. This equation can be used to calculate the thickness of material that will reduce the intensity of the x-rays to undetectable levels in the case of diffraction or to safe levels in the case of protection.

The detection of x-rays is an important aspect of x-ray diffraction, in that the diffracted rays are not intense. There are three principle means of detection; fluorescent screens (18, p. 83-88), photographic film (36, p. 106-110), and ionization devices. We will be concerned with the first two. Fluorescent screens are made of some chemical element that emits light when x-rays are absorbed in them. For viewing purposes these screens have on some inert base a coating of zinc sulfide with a trace of nickel. This emits yellow light and in a darkened room is visible to the eye. Intensifying screens, used in conjunction with x-ray film to increase the exposure to the film, also have an active coating on an inert backing. The coating is usually of calcium tungstate which emits blue light when x-rays are absorbed in it. X-ray film is more sensitive to blue light than it is to yellow. When the film is placed in contact with the

screen, the film is not only blackened by the x-rays but also by the visible light emitted by the intensifying screens at the same point.

X-ray film is affected by x-rays much the same way as regular film is by light. The x-ray causes a change in the silver atom of a silver bromide crystal in the emulsion of the film. After exposure the film is developed by a process which causes the silver to be deposited in the area of exposure. The silver deposit causes a blackening of the film. The degree of blackening or density is defined in terms of the amount of light it will transmit. A density of one allows  $1/10$  of a light beam to be transmitted.

X-ray film differs from ordinary film in that it has a double coating of emulsion, one on each side of the film. This increases the probability of interaction with x-rays. There are numerous types of x-ray film just as there are numerous types of regular film. Variations in speed and contrast are obtainable as well as variations in detail.

In a discussion of x-rays some mention must be made of safety precautions. A hazard exists since x-rays can destroy human tissue, a fact utilized in x-ray therapy. The biological effects of x-rays include burns, radiation sickness, and genetic mutations. Since x-rays are not visible it is necessary that equipment be properly designed and shielded to prevent needless exposure to the radiation. Limits of exposure have been established below which it is safe to work (44).

## Diffraction

When a beam of light passes close to the edge of an opaque obstacle, propagation is no longer truly rectilinear. It is possible to observe fringes near the edge of the geometrical shadow, and even some light can be seen in the shadow. The name diffraction is given to all departures from rectilinear propagation of light. The most obvious diffraction effects are produced by opaque obstacles. Diffraction is produced by any arrangement which causes a change of amplitude or phase which is not the same over the whole area of the wavefront. Diffraction thus occurs when there is any limitation of the width of a beam of light.

To observe diffraction effects, which result from variations of resultant amplitude and hence of the relative energy and the illumination, from point to point in the field of view, it is necessary to have coherent waves. The variations just mentioned are due to varying path differences so that the waves reinforce each other at some points and oppose each other elsewhere. For two beams to be coherent, the phase difference between the waves by which they are represented must be constant during the period normally covered by observations. Two waves are said to be noncoherent when the phase difference changes many times and in an irregular way during the short period of observation. Since light is electromagnetic radiation, the

foregoing discussion can be applied to x-rays.

Having previously discussed crystallography and the physics of x-rays, we can fit the two together and see how x-ray diffraction could arise. Historically this is what happened. Laue put the two ideas together and arrived at x-ray diffraction. It is easy today to say that this is not a surprising development since the interatomic distances of crystals and the wavelength of x-rays are close to being equivalent and the interaction of these two would produce some sort of diffraction effect. Yet at that time it was a remarkable display of intellectual achievement.

### X-ray Diffraction

To attain the elementary understanding of x-ray diffraction needed in using the teaching device, there are two theories that can be utilized. The first is the theory that Laue (50, p. 320-324) used to explain his discovery. The second and easier to visualize is that of W. L. Bragg (10).

Laue's explanation of diffraction is based on the interaction of scattered x-rays from rows of atoms. Since in three dimensions there are three rows, the result is three sets of diffraction cones, each set containing a system of coaxial cones. The intersection of these sets of cones results in constructive interference in one direction, producing the diffracted beam, and destructive interference in

other directions. In actual fact, what Laue envisioned was a three dimensional optical diffraction grating and his theory is based on just that.

If an x-ray beam, monochromatic in this case, is directed at a row of equally spaced atoms, each atom acts as a source of scattered waves. Then reinforcement can occur along certain directions to produce the diffracted beam. The conditions for reinforcement are seen in Figure 10.

If the incident beam makes an angle  $\alpha_0$  with the row and the diffracted beam leaves at  $\alpha$ , the path difference is

$$BC - AD = a(\cos\alpha - \cos\alpha_0) = z.$$

The path difference must be an integral number of wavelengths if the scattered waves are to be in phase. The phase difference for a wavelength  $\lambda$  and a wavelength of constant  $k$  is

$$\delta_\lambda = \frac{2\pi z}{\lambda}.$$

The waves are in phase when  $z = m\lambda$  or  $\delta_\lambda = 2m\pi$  where  $m$  is an integer. So we have that  $a(\cos\alpha - \cos\alpha_0) = m\lambda$  must be satisfied if the waves are to be in phase and to constructively interfere. This equation will be satisfied by all the generators of a cone that are concentric with the line of atoms and that have a semiapex angle  $\alpha$ . Thus for any given angle of incidence there will be a series of concentric cones surrounding the row of atoms, each cone being made



up of one order of diffracted rays.

If there is a two dimensional network of atoms with spacing  $a$  in one dimension and  $b$  in another, there will be two simultaneous equations to be fulfilled for constructive interference. They are

$$a(\cos\alpha - \cos\alpha_0) = m\lambda$$

$$b(\cos\beta - \cos\beta_0) = r\lambda$$

where  $\alpha$  and  $\beta$  are diffracted angles,  $\alpha_0$  and  $\beta_0$  are incident angles, and  $m$  and  $r$  are integers. These equations correspond to two sets of cones, a set around the  $a$  axis and another concentric with the  $b$  axis. The diffracted beam will occur along the intersection of these two sets.

Following the same arguments in three dimensions, as in a crystalline structure, there are accordingly three conditions to be satisfied simultaneously for diffraction. They are

$$a(\cos\alpha - \cos\alpha_0) = m\lambda$$

$$b(\cos\beta - \cos\beta_0) = r\lambda$$

$$c(\cos\gamma - \cos\gamma_0) = \rho\lambda.$$

These are known as the Laue equations. The first two have the same significance as before and the third relates to the periodicity in the third dimension, the  $c$  axis of the crystal. There are then three sets of cones with a common line of intersection if all three equations

are satisfied simultaneously. For a simultaneous solution to the Laue equations it is necessary that the directions defined by each of the three equations be the same. This will not happen in general so the number of diffracted beams is limited. The angles between the incident beam and the lattice rows can be varied however. It is possible to choose them so that the three equations define an identical direction. If the three cones happen to intersect in a single line, then all the atoms in the lattice will diffract rays in this direction at the same time. There will be constructive interference not only between rays from all atoms in a plane, but between rays from all such planes in the family of planes. By reasoning in this way Laue accounted for the diffracted beams observed photographically.

In the development of Laue's equations the treatment has been one of a formal problem in diffraction in that the diffraction from a lattice array is the cooperative diffraction from three non-coplanar rows of atoms. This method is hard to visualize as well as being cumbersome to work with. It can be shown (10) that diffraction of this sort is geometrically equivalent to a reflection of the x-ray beam by the plane referred to by (mnp).

This was the theory expressed by W. L. Bragg. The lattice planes of the crystal are considered to be reflecting planes. X-rays will be reflected with a reflection angle equal to the angle of incidence. Since x-rays penetrate the crystal and are reflected by each

successive layer, the path length is  $2d' \sin\theta$  greater for that layer than for the previous one, as in Figure 11. If this path difference is an integral number of wavelengths, the waves from each layer interfere constructively. Thus we have  $n\lambda = 2d'\sin\theta$  where  $d'$  is the interplanar spacing. The equation just stated is the familiar Bragg's law. It can be simplified by considering the equation

$$\lambda = 2\frac{d'}{n}\sin\theta$$

Since the coefficient of  $\lambda$  is unity, it is possible to consider a reflection of any order as a first-order reflection from planes spaced at  $1/n$  of the previous spacing. So, if we allow  $d = d'/n$ , we have

$$\lambda = 2d\sin\theta.$$

The value of  $d$  varies with the particular crystal system and the particular values of  $(hkl)$  and is better represented as  $d_{hkl}$ . We now have

$$\lambda = 2d_{hkl}\sin\theta,$$

Bragg's law in its most useful and proper form.

In the discussion of the Laue equations, it was concluded that a diffracted beam should be expected in a direction making angles  $\alpha$ ,  $\beta$ , and  $\gamma$  with the  $a$ ,  $b$ , and  $c$  axes of the crystal when the equations were simultaneously satisfied. Under these conditions the  $a$  axis rows are diffracting in the  $m$ th order, the  $b$  axis rows in the  $r$ th order and the  $c$  axis rows in the  $p$ th order.

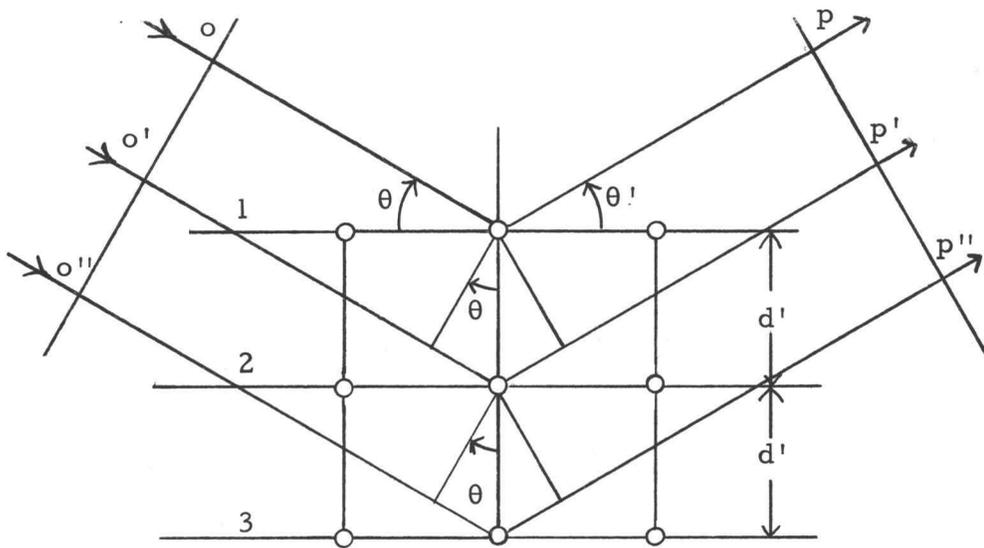


Figure 11. The conditions for Bragg diffraction. The path difference between  $op$  and  $o'p'$  is  $2d' \sin \theta$ .

In discussing the Bragg equation, a diffracted beam was expected in a direction coplanar with the incident beam and the normal to the (hkl) planes and departing from the crystal in a direction making a grazing angle  $\theta$  with these planes when the law is satisfied. This occurs for any given wavelength  $\lambda$  only at certain angles  $\theta$  for any given set of planes (hkl).

It can be shown that the two results are equivalent. Laue's equations, when simultaneously satisfied for given directions and given  $\lambda$ , represent Bragg's law diffraction of the nth order for the (hkl) planes when

$$m = nh$$

$$r = nk$$

$$p = nl.$$

The same principle axes are used in both methods.

The Bragg point of view makes the mechanism of x-ray diffraction in a crystal much easier to visualize than does the Laue analysis. Both predict the same results for a given set of conditions. To predict the diffraction direction for a particular set of planes, we can use the Bragg law in its simplified form and substitute for  $d_{hkl}$  the formula for the particular crystal system. These values of  $d_{hkl}$  are listed in Table 2. The result is an equation in which  $\theta$ , (hkl), and  $\lambda$  can vary. The directions in which a beam of given wavelength is diffracted from a given plane is determined by the crystal system and

its lattice parameters. Diffraction directions are governed by the shape and size of the unit cell. Conversely, all you can determine about the unit cell of a crystal from its diffraction directions is its size and shape.

To summarize, diffraction is essentially a scattering phenomenon in which large numbers of atoms cooperate. Since the lattice is a periodic arrangement of atoms, the scattered rays have a definite phase relationship. As a result of these phase relations, destructive interference occurs in most directions but constructive interference can occur in certain predictable directions and diffracted beams are formed.

### The Laue Method

For a more complete understanding of x-ray diffraction, the production and analysis of Laue photographs (3, p. 88-100, 22, p. 137-150, 25, p. 54-86, 58, p. 124-151) will serve as an elementary example of the methods available. The Laue method was the first diffraction method ever used and is a reproduction of von Laue's original work. In this method the wavelength of x-rays is variable and the value of  $\theta$  is fixed. Recalling the Bragg equation, we see that the Bragg angle  $\theta$  is fixed for every set of planes in the crystal, and each set picks out and diffracts that particular wavelength which satisfies the Bragg law for the particular values of

$d_{hkl}$  and  $\theta$  involved. We have a beam of white or polychromatic radiation, the continuous spectrum from an x-ray tube, incident on a fixed single crystal, each diffracted beam thus having a different wavelength.

The Laue camera consists of a pinhole system which collimates the beam into a narrow pencil of x-rays, a goniometer head or other device to support and orient the crystal, and a flat film in a light tight envelope placed to receive the diffracted x-rays. Schematically this is seen in Figure 1. There are two variations of this method depending on the relative position of the crystal, film, and x-ray source. The film is flat and perpendicular to the path of x-rays in both cases. In one case, the transmission method, the film is placed behind the crystal to receive the x-rays diffracted in a forward direction. This method requires a crystal of such density or size that the x-rays are not appreciably absorbed in passing through it. In the second case the film is placed between the crystal and x-ray source, the so-called back-reflection method. The diffraction pattern is in a backward direction and is recorded on the film. This method does not limit the crystal specifications but does necessitate a hole in the film for x-rays to pass through.

Each reflection plane in the crystal reflects a portion of the x-ray beam. To get the maximum continuous spectrum, thus the widest variation in  $\lambda$ , from an x-ray source, the voltage on the tube

must be below the excitation energy of the characteristic radiation. In the case of a tungsten tube this is less than 60 kV. The diffraction pattern produced is a pattern of spots that can be visualized as a pattern made by reflection from a number of mirrors inclined at different angles. Laue spots from planes with a common zone axis lie on ellipses, for if the x-ray beam is at an angle  $A$  to a zone axis, the reflected rays must also be at the same angle to each plane and therefore to the zone axis. The rays thus lie on the surface of a cone of semivertical angle  $A$ . The trace on a photographic film of the cone is an ellipse with one end of its major axis at the center spot of the pattern.

Since the positions of the internal planes depend upon the symmetry of the crystal, the arrangement of the diffraction spots on the photographic film must possess a corresponding symmetry. Owing to the fact that the face  $(hkl)$  and the parallel face  $(\bar{h}\bar{k}\bar{l})$  reflect to the same spot and cannot be distinguished, all crystals seem to have a center of inversion. For Laue patterns it is possible to distinguish only 11 different classes of symmetry instead of the entire 32 classes.

The shapes of the spots in a Laue pattern are determined by crystal imperfection and by geometrical conditions, such as the nature of the convergence or divergence of the primary beam of x-rays. If the beam is a divergent circular cone of rays and the crystal covers the entire beam, then the spots will be elliptical in form with the

minor axis radial to the center spot.

The usefulness of this historically first method is limited but it is still employed in conjunction with other methods today. The main uses for it are the determination of crystal orientation, the assessment of crystal perfection, and the determination of crystal symmetry.

The most convenient method of interpretation (32, p. 17-24) is to employ a projection that transforms the ellipses of the pattern into straight lines, at the same time allowing the assignment of indices to the spots. The gnomonic projection allows this and is easily obtained. The general theory of the gnomonic projection was discussed earlier and we recall that two equations relating the Laue spot and its gnomonic projection were obtained. Referring to Figures 6 and 7, the equations were

$$F'C' = CC' \tan 2\theta$$

$$G'C' = CC' \cot \theta.$$

In general,  $CC'$  is taken to be five centimeters. The actual plotting of the gnomonic projection could be carried out using these equations, but it is done more rapidly with a ruler. The left side of this ruler measures the distance  $F'C'$  of a Laue spot to the center spot. The right side is graduated in accordance with the equations to give the location of the projected Laue spot. In making the projection, the ruler is mounted at  $C'$  on a pin through the common center of the

ruler and the Laue pattern. Then, since the diffraction spot and projection lie on a line through  $C'$ , the projection of any spot is located on the opposite side of the ruler at a corresponding graduation. Such a ruler is shown in Figure 12. In Table 3 are the values from the two equations which make up the right and left side of the ruler for a 5 cm  $CC'$  distance (33).

Table 3. Data for preparing the gnomonic ruler with  $CC' = 5$  cm.

Left side of ruler cm	Right side of ruler cm	Left side of ruler cm	Right side of ruler cm
1.10	46.0	3.40	16.24
1.20	42.3	3.50	15.86
1.30	39.1	3.60	15.50
1.40	36.4	3.70	15.16
1.50	34.06	3.80	14.84
1.60	32.02	3.90	14.54
1.70	30.24	4.00	14.26
1.80	28.65	4.10	13.98
1.90	27.23	4.20	13.73
2.00	25.96	4.30	13.48
2.10	24.82	4.40	13.25
2.20	23.78	4.50	13.02
2.30	22.84	4.60	12.82
2.40	21.97	4.70	12.62
2.50	21.18	4.80	12.43
2.60	20.45	4.90	12.25
2.70	19.78	5.00	12.07
2.80	19.16	5.10	11.90
2.90	18.58	5.20	11.74
3.00	18.05	5.30	11.59
3.10	17.55	5.40	11.44
3.20	17.09	5.50	11.30
3.30	16.65	5.60	11.17

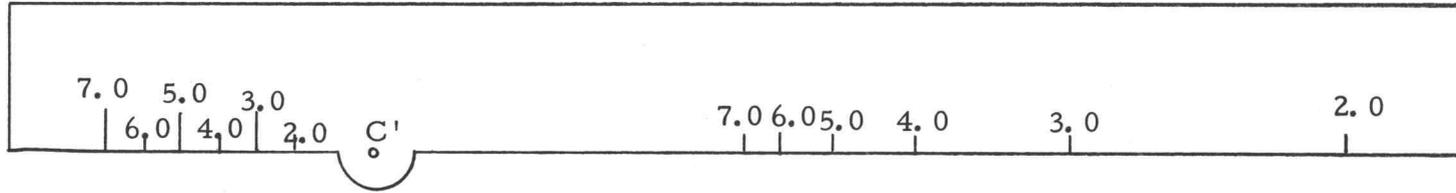


Figure 12. The gnomonic ruler.

The gnomonic projection arranges the spots in easily indexed rows and columns. The pattern into which the spots fall is recognizable and when the lines connecting the spots are drawn the indexing becomes obvious.

When the  $a$ ,  $b$ , or  $c$  axis of a crystal is normal to the plane of projection, the projection is made up of parallel lines in a symmetrical pattern. With the beam parallel to one axis, the index of this axis is unity, the other two indices being variable. In a cubic crystal, for example, the zone lines form a coordinate network of squares. The indices of any spot can be read from this network by reading the two coordinates from the plot assuming the third to be unity then clearing fractions. The result is a value for  $(hkl)$ . When the crystal axes parallel to the projection are of unequal length, the network will be rectangular in shape. Inclined axes project into parallelograms. Noting the axial ratios and the axial angles as listed in Table 1, the network expected from the projection of various crystals is easily determined.

Converging lines replace parallel lines if a crystal is not oriented with the incident beam exactly along an axis. It is possible to determine by inspection how much the crystal should be rotated to bring it into alignment. After the adjustment, the projection should be parallel.

In summary, the Laue method uses a single stationary crystal

and employs an x-ray beam containing all possible wavelengths, from those barely able to pass through the crystal to the quantum limit imposed by the voltage across the x-ray tube. The pattern obtained is interpreted by means of a gnomonic projection to give the Miller indices of the planes which diffract the x-rays. The data thus obtained gives some idea of the orientation of the crystal. It can also be used to aid in the elimination of some symmetry structures through the use of space-group theory.

## THE TEACHING DEVICE

In designing an x-ray diffraction teaching device for an undergraduate class in which only the basic fundamentals are to be considered, there are several requirements that must be met before a satisfactory design can be established. The primary requisite is that the device be elemental in its construction and operation. It must, however, be capable of demonstrating that the concepts learned in discussing x-ray diffraction are actually being applied. It should have as few "black boxes" as possible. The possible adjustments should be minimized so that the student is not likely to damage or misadjust the equipment. The student should be able to get the result, a diffraction pattern, and observe the effects of the x-rays striking the crystal.

The device should be rapid in its operation. The results should be obtainable in a one hour period in order that the student, in the course of a laboratory session of three hours, has ample time in which to correct errors and to analyze his results. The duty cycle of the x-ray equipment should be such that an adequate exposure will result.

The result, in order for the learning process to take place, should lend itself to understandable analytical procedures. From the diffraction pattern the student should see that what has happened has

truly been x-ray diffraction.

The last requirement is that it be safe for student operation. Since x-rays are an ionizing radiation and as such can represent a hazard, the device must be properly shielded. Standards of protection have been set down for educational institutions (44) and since this is a device to be used in such a place, it must conform to these limits. The primary one being that no more than 0.01 rem should be received by any student under 18 years of age during any one experiment.

### The Design

The most elemental of the several diffraction devices in existence is that based on the original piece of equipment developed by von Laue in 1912, known as the Laue diffraction camera. This device consists of a source of x-rays, a protective shield into which a collimator is mounted, a device on which to mount the crystal specimen, and a film holder, are all mounted on a table.

A portable diagnostic x-ray unit was available for use. It was composed of two parts, the x-ray tube head, containing the x-ray tube and high-voltage transformer immersed in oil, and the control unit, containing the controls, timer, and x-ray contactor. The device was constructed making use of these components.

A partial radiation shield was designed into which the front of the tube head would fit, encasing the face of the tube head, from which the protective cover had been removed. With the x-ray

machine properly shielded, a collimator through which the beam of x-rays emerges was attached to the shield.

The collimated beam of x-rays travels in a straight line perpendicular to the tube-head face to be incident on a crystal. The diffracted beams continue in the same direction in the transmission method. This set of conditions necessitates that the film holder, crystal, and collimator remain perpendicular to the beam and move parallel to each other. To satisfy this requirement an optical bench was used on which the three components could be mounted.

The crystal specimen was supported in a carriage constructed for the optical bench and could be moved vertically and horizontally.

The x-ray film and intensifying screens were contained in a cassette so the film holder was designed to support the cassette, permitting horizontal and vertical movement.

A device was needed to time the x-ray exposure periods and to record the elapsed exposure time. Using a discarded electric clock, a cam was constructed which results in a fractionated exposure over a period of time. Extended periods of operation result in a repetition of the fraction of time, allowing time for the x-ray tube head to dissipate heat between exposures. The total time was recorded on the face of another electric clock.

All these components were put together and mounted securely on a portable table. The table had a large working area on which the

x-ray tube head, shield and collimator, and optical bench were mounted. The control unit and timing device were mounted behind and somewhat below the working area, to allow easy access to the x-ray tube head. The various components of the teaching device are shown in Figures 13, 14, and 15. In Figure 16 the assembled components are shown and in Figure 17 there is a block diagram of the assembled components.

### Materials and Methods

The previous discussion is a general outline of the x-ray diffraction device. The following is a detailed description of the various components and their makeup.

The x-ray machine, shown in Figure 13, is one produced by the H. G. Fischer Company. It is a mobile diagnostic machine, model TC-30, rated at a maximum of 30 mA and 90 kVp. The x-ray tube is Eureka model 7-70 HRH and has a copper anode with a tungsten target. The heat storage capacity of the tube is 60,000 HU<sup>1</sup> and the cooling rate in oil is 34,000 HU per minute. The focal spot size is 2.8 mm and is four cm from the face of the x-ray head.

In using the machine for Laue diffraction, it is necessary to obtain the maximum continuous energy spectrum of x-rays, without

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<sup>1</sup>HU = heat unit = (kVp)(mA)(time in seconds).

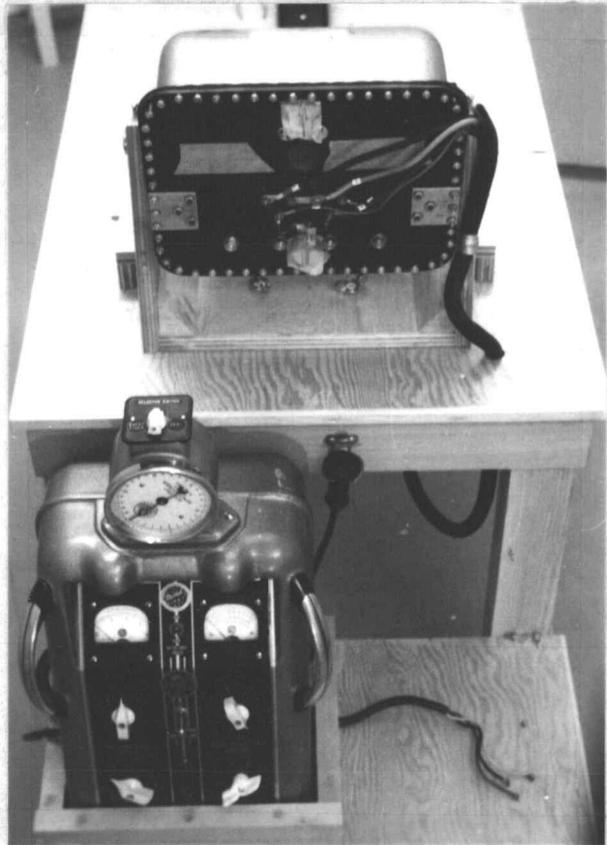
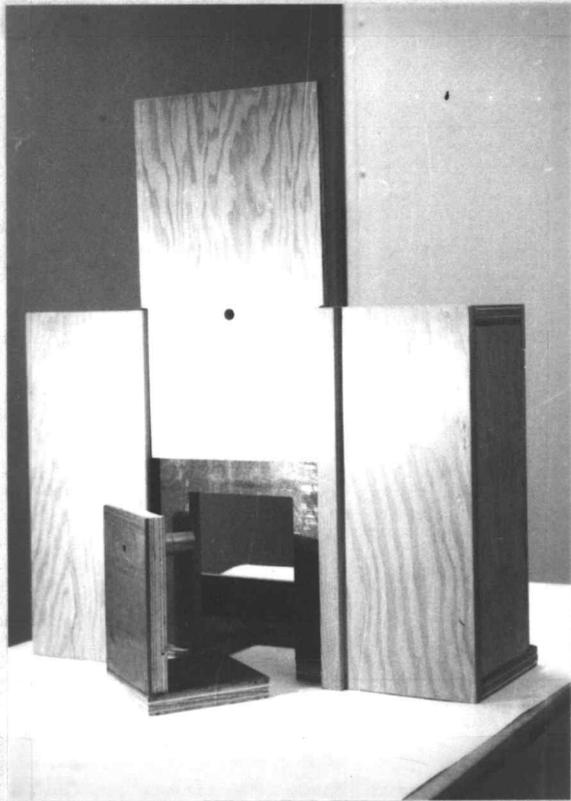
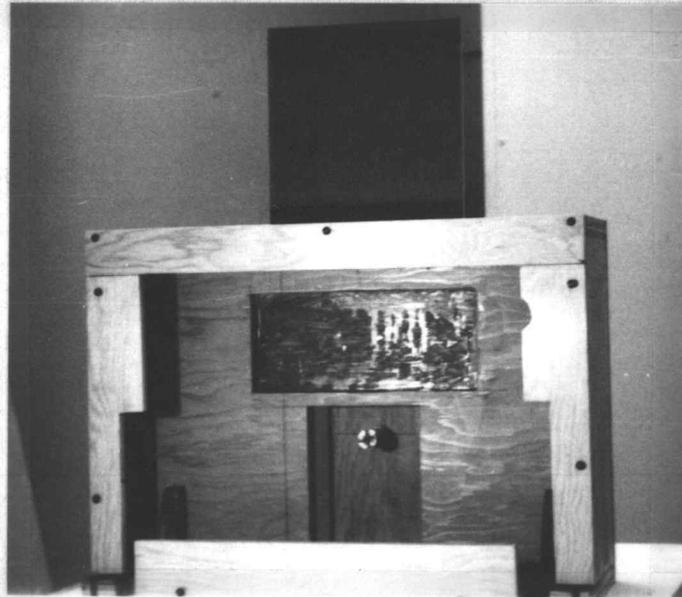


Figure 13. Two views of the x-ray tube head and control unit of the Fischer TC-30 x-ray machine mounted on the table.

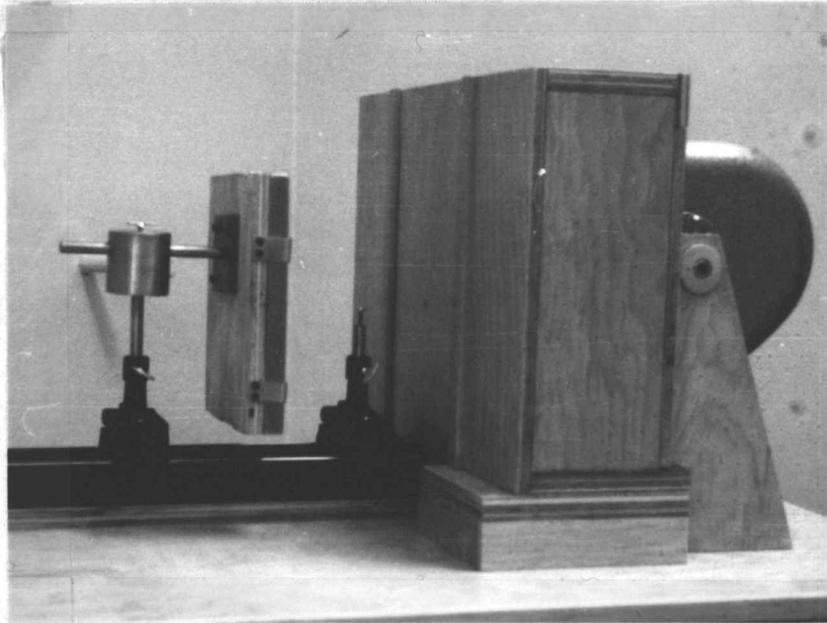


A

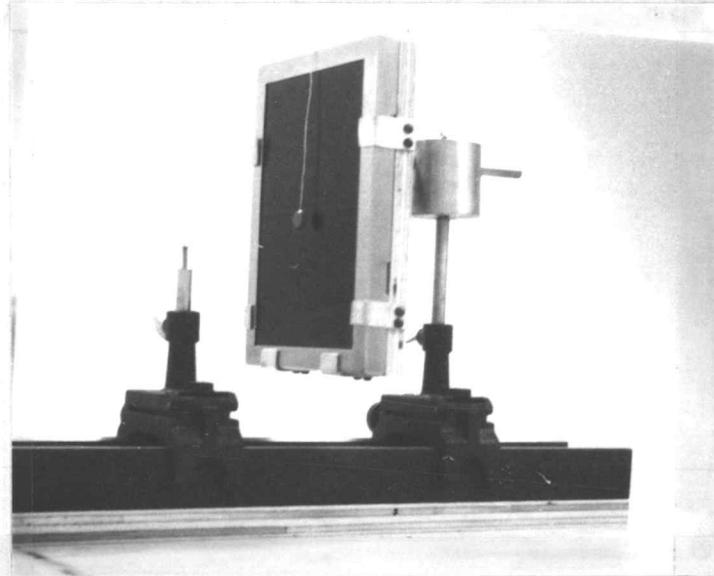


B

Figure 14. The shield and collimator. In A is a front view showing the protective door in the open position and the collimator removed. In B is a back view showing the attached supplementary shields.



A

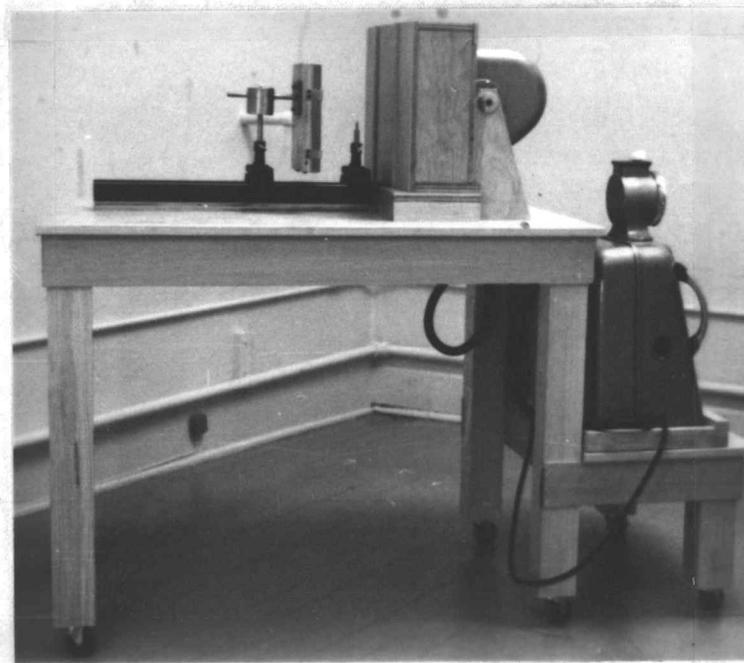


B

Figure 15. Two views of the optical bench, crystal support and film holder. In A the components are assembled in front of the shield and collimator. In B the components are alone.



A



B

Figure 16. Two views of the x-ray diffraction teaching device. In A the x-ray head, x-ray control unit and the exposure timer console are seen. In B the optical bench, film holder, crystal support, shield and collimator, and x-ray head and control unit are seen.

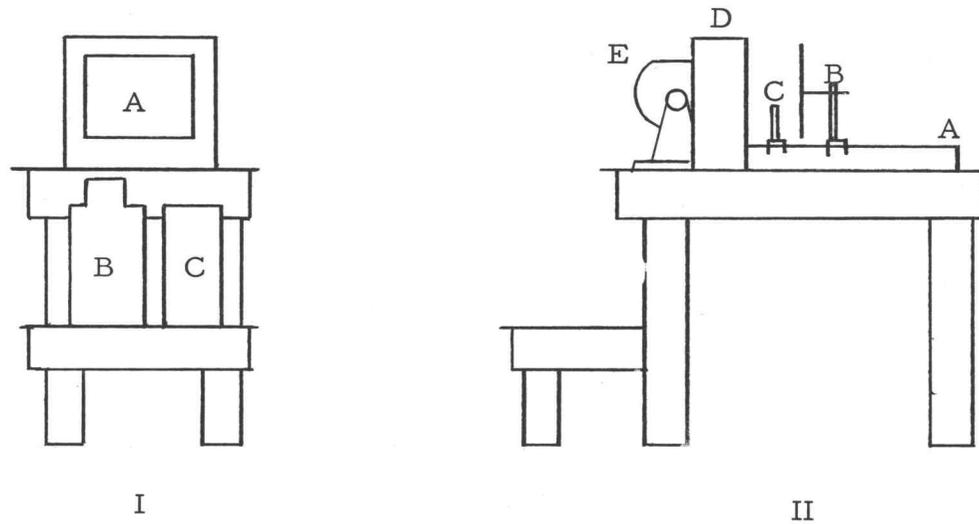


Figure 17. A block diagram of the assembled apparatus. In I A is the x-ray head in the shield, B is the x-ray control unit and C is the exposure timer. In II A is the optical bench, B the film holder, C the crystal mount, D the shield and collimator and E the x-ray head.

the characteristic lines. Because the characteristic lines of tungsten appear near 60 kV, an operating voltage of 55 kVp was used to obtain the maximum continuous energy spectrum of x-rays. To describe the x-ray beam, the first and second half value layers and the homogeneity coefficient were determined (52). For this machine at 55 kVp and 20 mA the unique first half-value layer is 0.76 mm Al and the unique second half-value layer is 1.86 mm Al. The homogeneity coefficient is 0.41. This indicates that the x-ray beam is made up of a wide spectrum of energies, which is good with respect to diffraction by the Laue method. To satisfy the requirement of speed, the intensity of the x-ray beam needs to be high and 20 mA is found to be the maximum feasible operating current. This machine has no oil circulator in the head, so the heat that can be dissipated is limited. Consequently, short exposure times are needed. The combination of 10 seconds on 50 seconds off is the maximum exposure at 20 mA and 55 kVp. It can be operated under these conditions for periods of up to 40 minutes without overheating.

With these facts at hand, a timer is needed to produce a sequence of 10 seconds on and 50 seconds off. A clock motor, which rotates at two revolutions per minute, drives a cam adjusted to close two microswitches for 10 seconds each minute. The microswitches are connected in parallel. One closes the x-ray contactor through the foot switch plug and the other starts the clock which records the

elapsed time. Both of these can be removed from the circuit by a switch. This allows adjustment of the x-ray machine without recourse to the delay caused by the 10 second intervals of the timer. The exposure timer console is shown in Figure 18.

The design of the protective shield is based on the criteria set forth by the National Council on Radiation Protection and Measurement (NCRP) (43). The shield is shown in Figure 14 and the design in Figure 19. It is constructed of 1/8 inch lead on 1/2 inch plywood. In addition there is a 1/16 inch lead on 1/4 inch plywood piece on the front which covers the cracks around the collimator support. Several pieces of 1/16 inch lead on 1/4 inch plywood are placed on the back of the shield to eliminate scatter in that direction. It was found in a radiation survey of the x-ray machine that the leakage from the casing of the head under operating conditions was undetectable, consequently a full shield was not needed. However, the leaded plywood pieces were added on the back of the shield to reduce the scatter around the head.

The collimator was constructed utilizing the specifications set forth by several authors (17, 19, 22, 1. 138-150, 49). An equation relating the size of the collimating hole, the focal spot size, and the distance between the holes is

$$h = d\left(\frac{2V}{u} - 1\right)$$

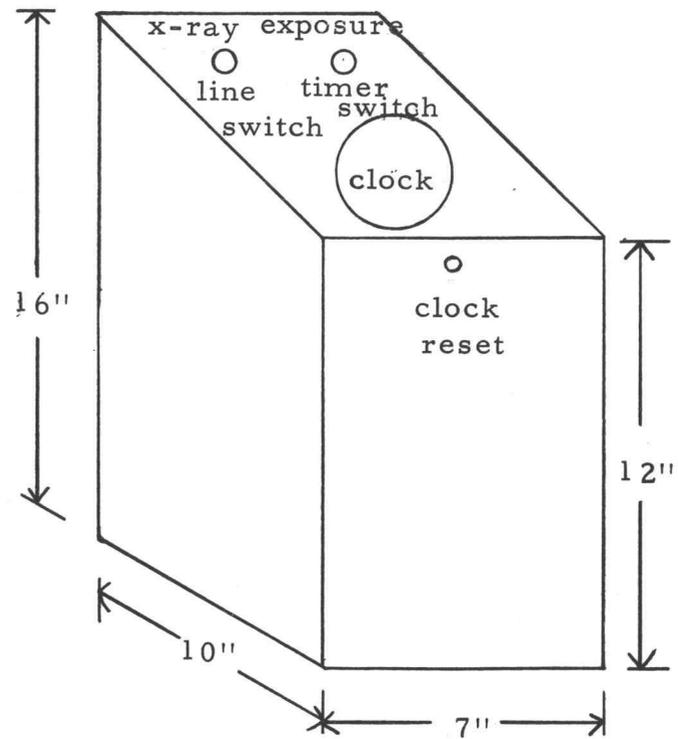


Figure 18. The exposure timer console.

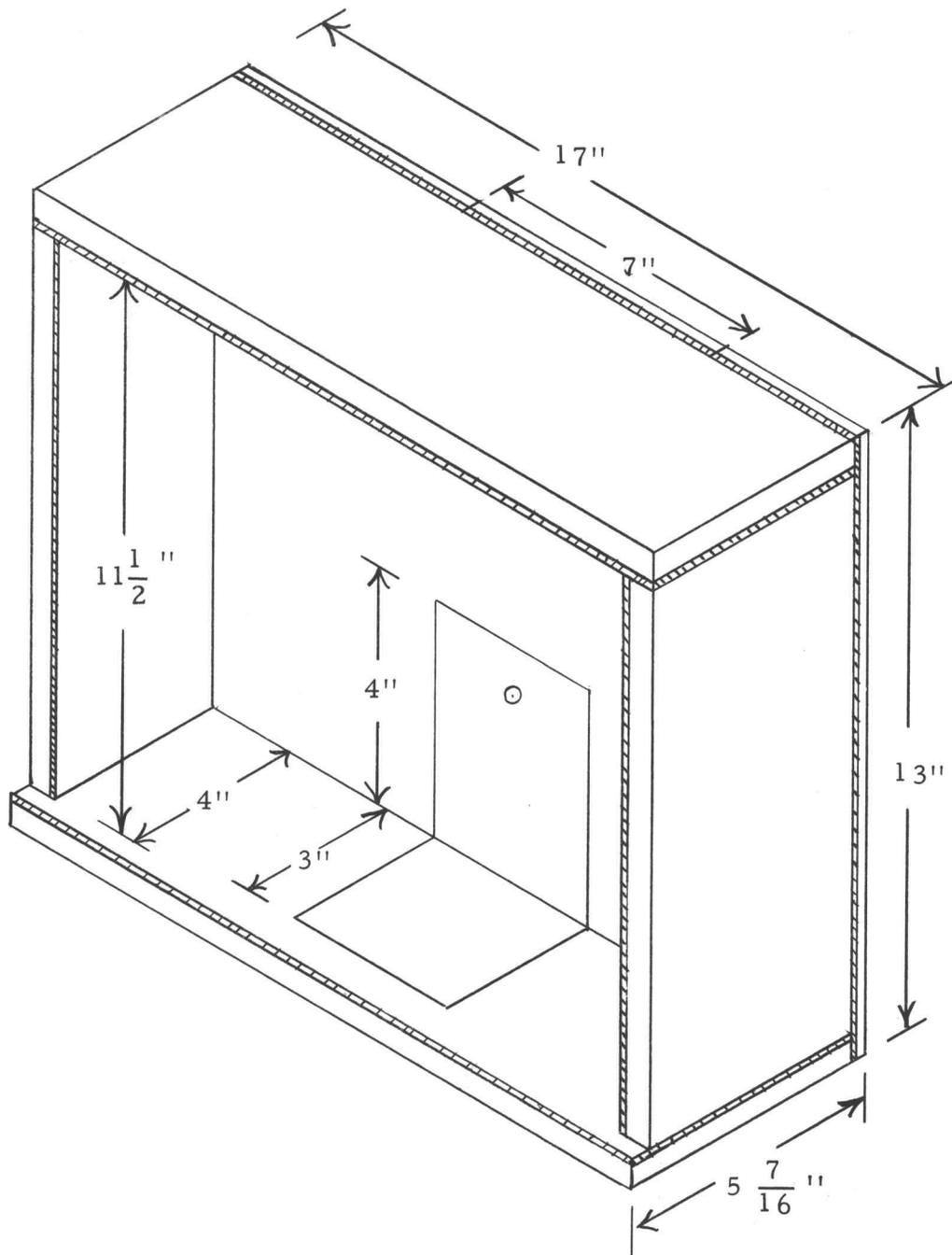


Figure 19. The design specifications for the radiation shield. The cross hatched areas indicate the location of the lead shielding.

where  $h$  = focal spot height,  $d$  = diameter of the hole,  $v$  = distance between exit hole and focal spot, and  $u$  = distance between pinholes. In Figure 20 is a sketch of the collimator geometry. Note that this is a guarded pinhole arrangement in that outside the exit hole is another hole to remove scatter from the exit hole. The result for our case is a collimator in which the distance  $u$  is 4.7 cm and the overall length is 5.3 cm. The 1/16 inch lead plugs are held in place by soldering the lead to the metal tube. The result is a collimator which produces a slightly divergent beam of x-rays.

The optical bench and the two hinged carriages are from the Cenco Company. The optical bench is 25 inches in length, number 85810 in the Cenco catalog. The hinged carriages are Cenco number 85802.

The crystal holder consists of an 8-32 machine screw threaded into a 1/4 inch rod which fits in the hinged carriage on the optical bench. The crystal is held in a slot in the head of the bolt with melted wax. This allows vertical and rotational movement of the crystal as well as horizontal movement, achieved by moving the optical bench sideways.

The film holder is designed to take a five by seven inch cassette which contains the film and intensifying screens. Support brackets attached to a five by seven inch piece of wood, which is attached to the hinged carriage on the optical bench, hold the cassette

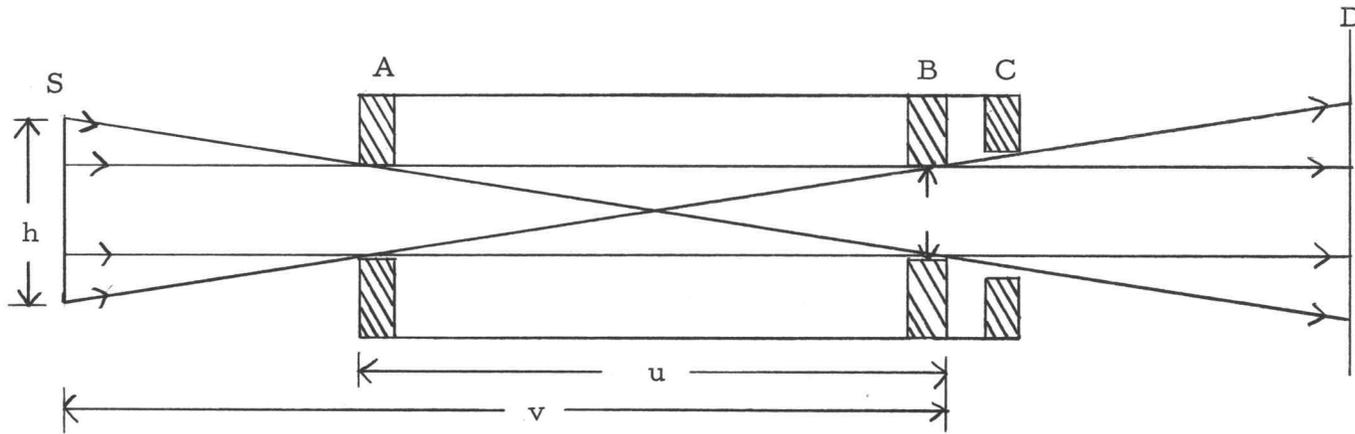
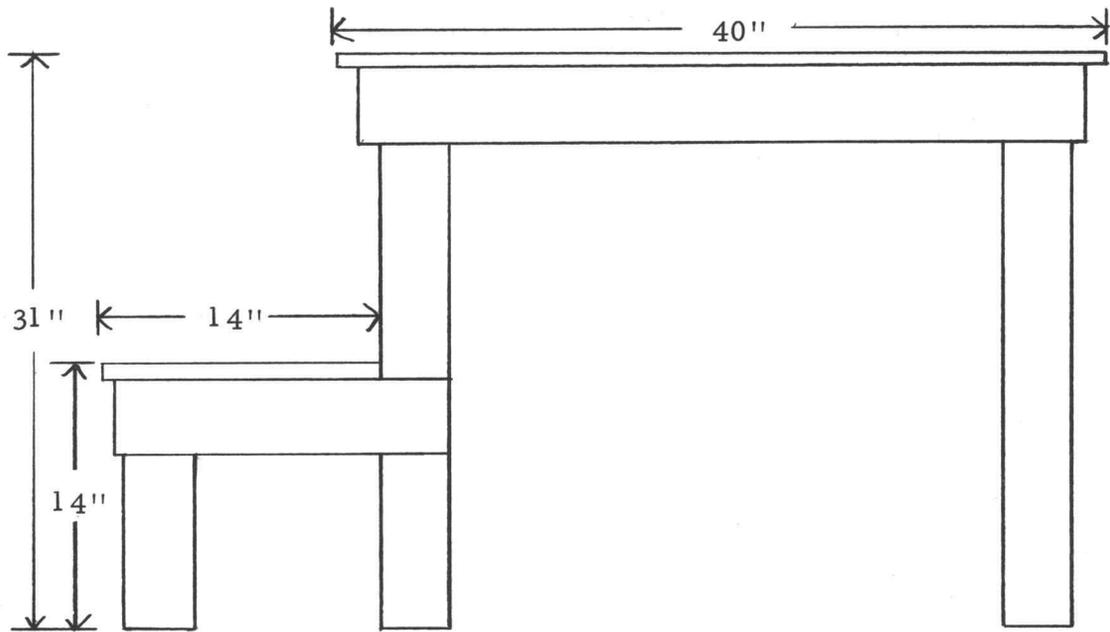


Figure 20. The geometry of a guarded pinhole collimator. A, B and C are of lead, C is the guard pinhole, S is the x-ray source and D is the crystal specimen.

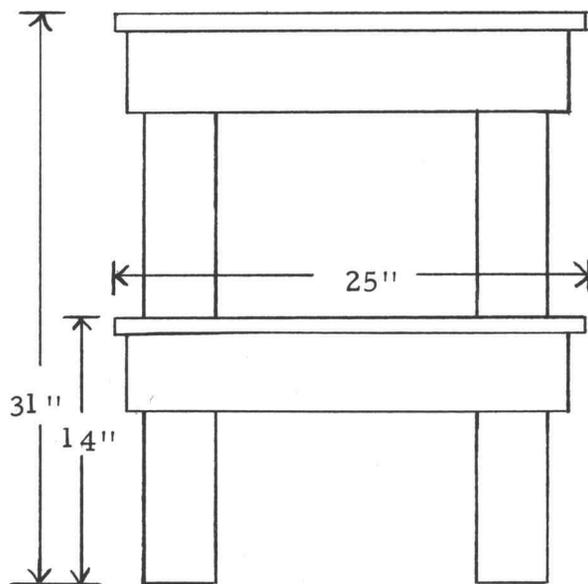
perpendicular to the x-ray beam.

It has been reported (58, p. 128-148) that an exposure of approximately 10 mA-hours is needed to get a good Laue pattern with film only. Recognizing that this is too long a period of time even at 20 mA, it is necessary to reduce the exposure time. One method of doing this is to use intensifying screens. There were available for use five by seven inch Dupont Par-Speed intensifying screens mounted in cassettes which fit the already designed film holder. As a result the exposure time was reduced to a few hundreds of seconds. This is attainable with the use of the exposure timer in less than a half-hour. It was necessary to add a piece of 1/8 inch lead to the center of the film holder to stop the undiffracted primary beam from reaching the film during all but a fraction of a second of the exposure time. This was done to prevent excessive fogging of the film in the area of the primary beam. The small exposure of the film to the primary beam gives a reference spot, the center of the diffraction pattern. In Figure 15 the film holder and sample holder are shown on the optical bench.

These various components are assembled on the portable table, the design and dimensions of which are shown in Figure 21. The working surfaces are constructed of 3/4 inch plywood. The table is made portable by wheels on the six legs. The x-ray tube head is supported by a 3/4 inch plywood support held to the table by two



side view



end view

Figure 21. The portable table design and dimensions.

removable wing bolts. The shield is supported on two fixed platforms. The optical bench is fixed on the table by two removable wing bolts, allowing horizontal movement. The control console is on the lower table and is held in place by a two-inch fence around it. The exposure timer console is constructed of 1/4 inch plywood and held to the lower table by screws on the inside. In Figure 16 the assembled apparatus is shown and in Figure 17 is a block diagram of the apparatus.

The x-ray film which is used in this device is matched to the Par-Speed screens to give the fastest combination possible. The film used is Kodak Royal Blue. It is processed in an Eastman Kodak X-Omat processor, which takes less than ten minutes to complete the developing process. Manual processing is possible though it takes much longer to complete.

Several crystal specimens can be used to produce useful diffraction patterns. The only requirement for a good pattern is that they be single crystals. They can be purchased from various suppliers of chemical materials or can be grown in the laboratory. Both methods have been used in this project. Four samples of crystal have been purchased from Semi-Elements Inc. The four are aluminum (Al), nickel sulfate hexahydrate ( $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ ), calcite ( $\text{CaCO}_3$ ), and rochelle salt ( $\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$ ). These are representative of the various symmetry classes. The author has grown crystals of rock salt (NaCl), and ammonium alum ( $\text{NH}_4\text{Al}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ ).

These samples are of the same order in density and so the exposure times are not too different from each crystal.

The length of time from adjustment of the crystal to viewing of the developed diffraction pattern using the automatic processor is no more than 40 minutes. The device is safe from the shielding point of view. There is no more than 0.5 mR in one hour at six inches from the surface of the device except for the primary beam. It goes without saying that care must be exercised when operating the device not to put one's hand in the beam.

## EXPERIMENTAL RESULTS

The visible results of this project are the diffraction patterns obtained. The possible number of substances on which the method will work are many. The six chosen represent four of the seven crystal systems and as such will give an idea of the different diffraction patterns possible. The four vary in the complexity of the diffraction patterns produced.

The exposure times varied between 100 seconds and 200 seconds depending on the detail desired in the diffraction pattern. The longer exposures make less intense spots visible, adding to the complexity of the diffraction pattern. The physical arrangement of the components was the same in all cases. A 5 cm crystal-to-film distance and a 3 cm exit hole-to-crystal distance was used for each diffraction pattern.

In analyzing these diffraction patterns, the gnomonic projection method was used. The gnomonic ruler (33, 58, p. 126-132) using the 5 cm measurements, was used to develop the projection. From the projected pattern the diffraction spots were indexed and the characteristics of the crystal visible in the gnomonic projection were observed. These, we recall, were the symmetry and orientation of the crystal. It will be noted in all of these results that the symmetry of the pattern is not exact and the gnomonic projection is therefore not

perfectly symmetrical. Converging lines have replaced parallel lines. This is a result of the inability to exactly orient a crystal axis along the x-ray beam.

In Figures 22, 24, 26, 27, 29, 30, 32 and 34 are seen the photographic reproductions of the diffraction patterns obtained. The exposure times are recorded with each one. In Figures 23, 25, 28, 31, 33 and 35 are the photographic reproductions of the gnomonic projection of the diffraction pattern just preceding.

In Figure 22 is the diffraction pattern of aluminum. Aluminum is a cubic crystal and the gnomonic projection is expected to be a pattern of squares. In Figure 23 the gnomonic projection of aluminum is not a pattern of squares, it is a pattern of converging lines, which means that the crystal was not properly oriented. The pattern does appear, however, to be a system of skewed squares, which would be expected from the orientation of the crystal.

In Figure 24 is the diffraction pattern of a laboratory grown rock salt crystal. This also is a cubic crystal but the gnomonic projection in Figure 25 shows the pattern of skewed squares, which indicates that the crystal was not properly oriented.

In Figure 26 is a diffraction pattern from the hexagonal crystal calcite with the x-ray beam along the four-fold axis. This isn't perfectly symmetrical indicating the misalignment. In Figure 27 is a calcite diffraction pattern with the beam perpendicular to the cleavage

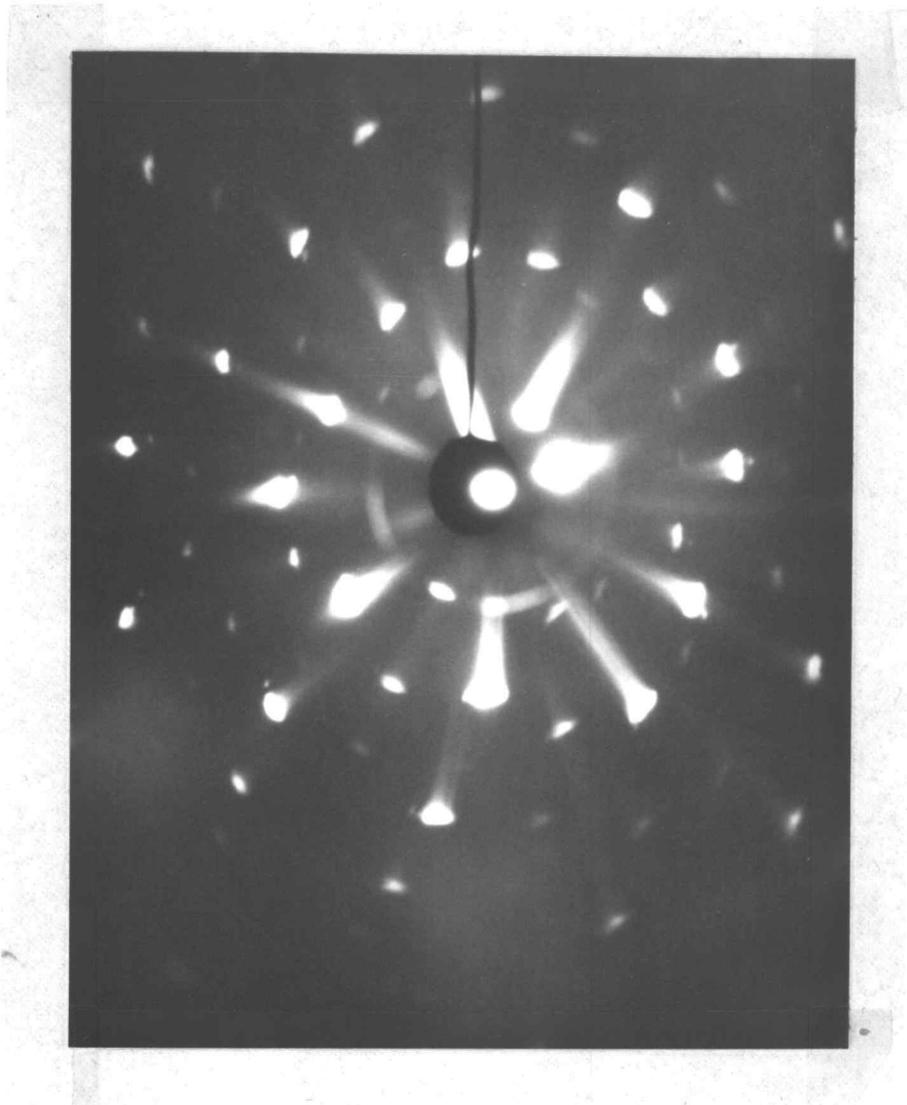


Figure 22. Diffraction pattern of an aluminum crystal, 150 second exposure.

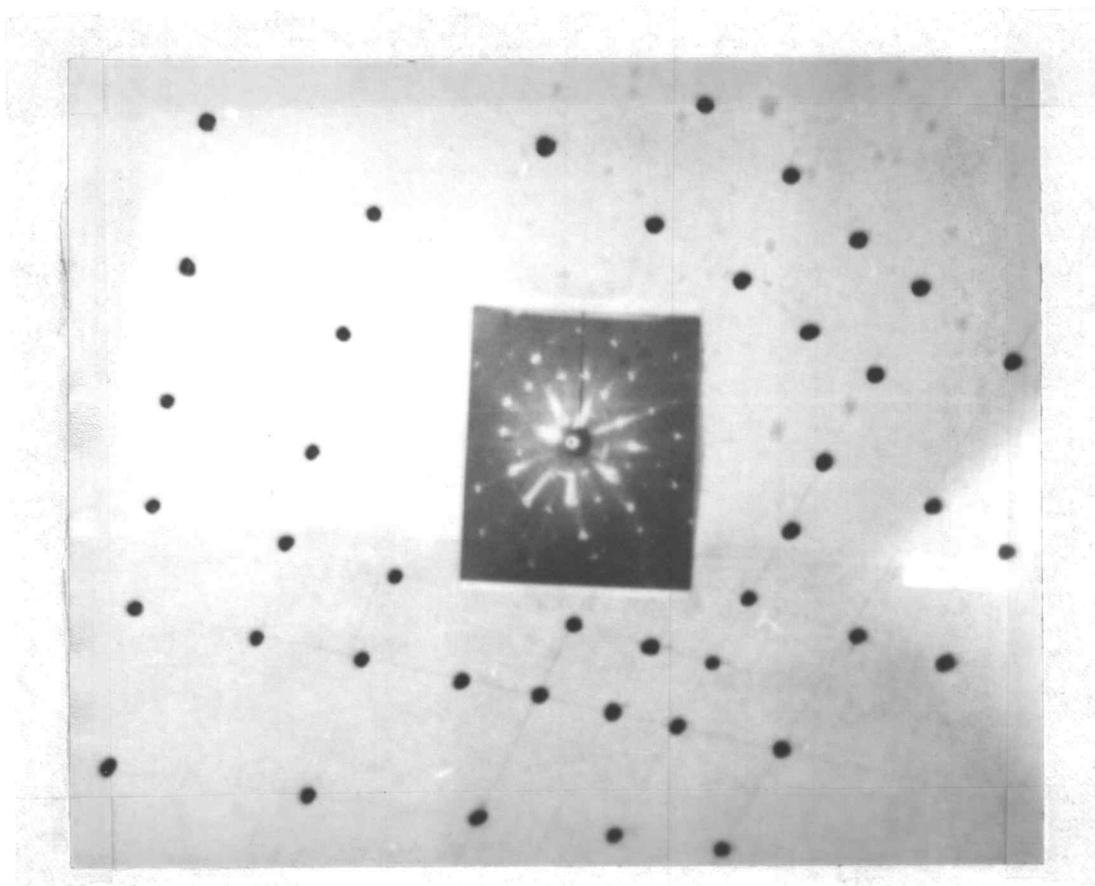


Figure 23. The gnomonic projection of the aluminum diffraction pattern.

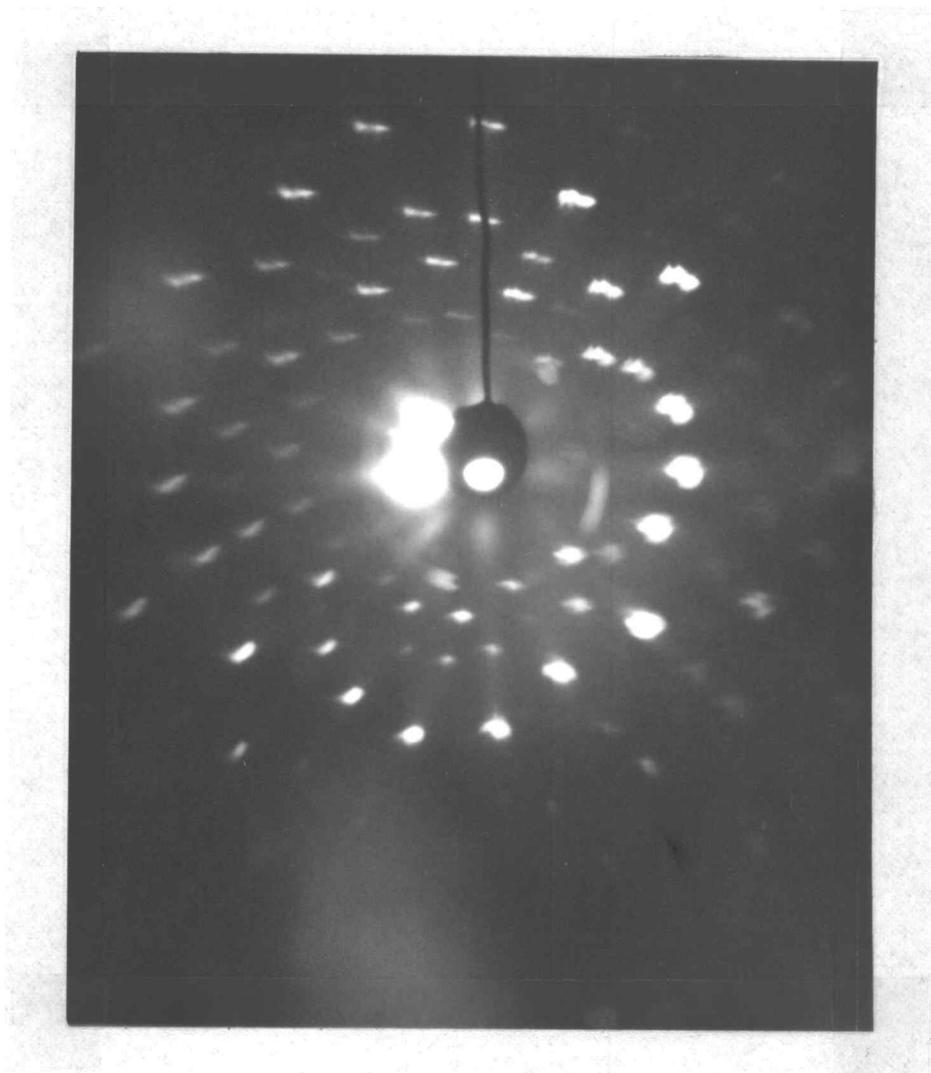


Figure 24. Diffraction pattern of a rock salt crystal with a 160 second exposure.

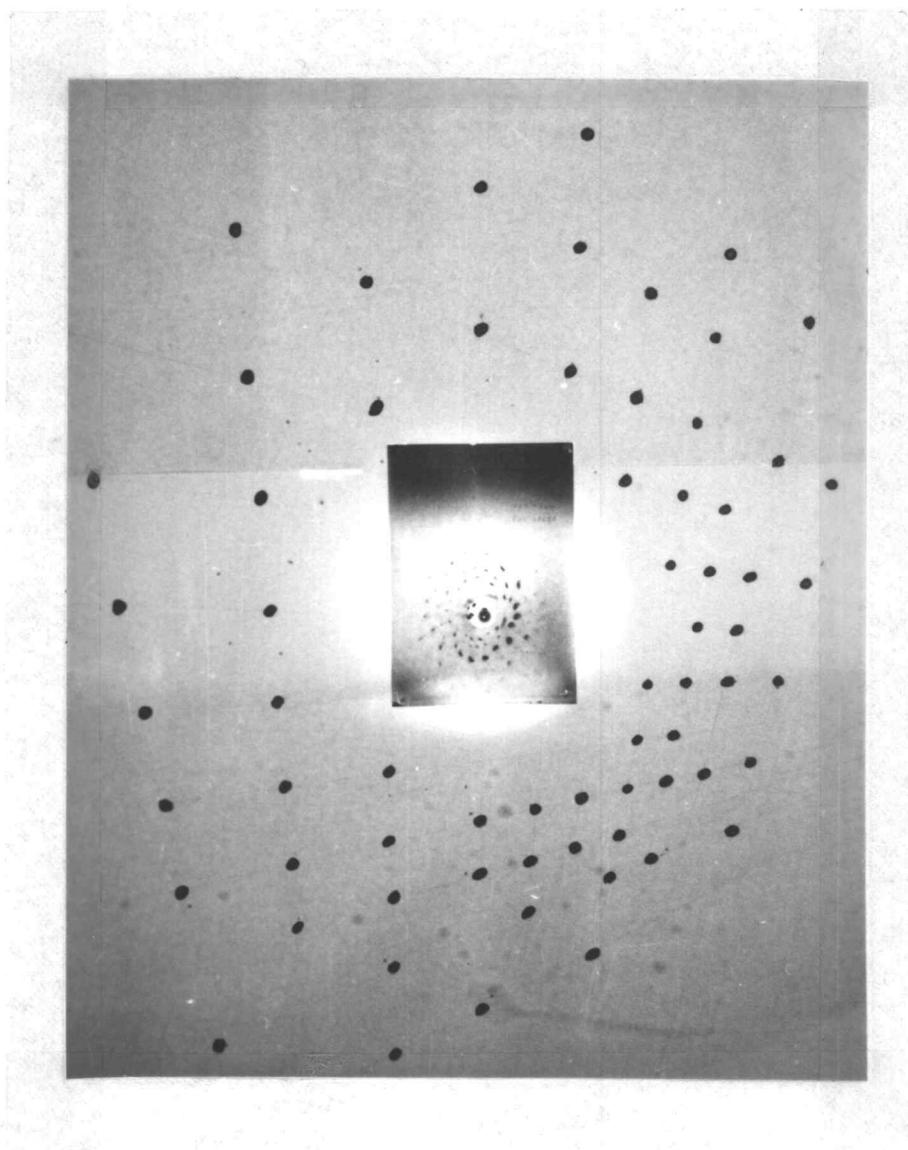


Figure 25. The gnomonic projection of the rock salt diffraction pattern.

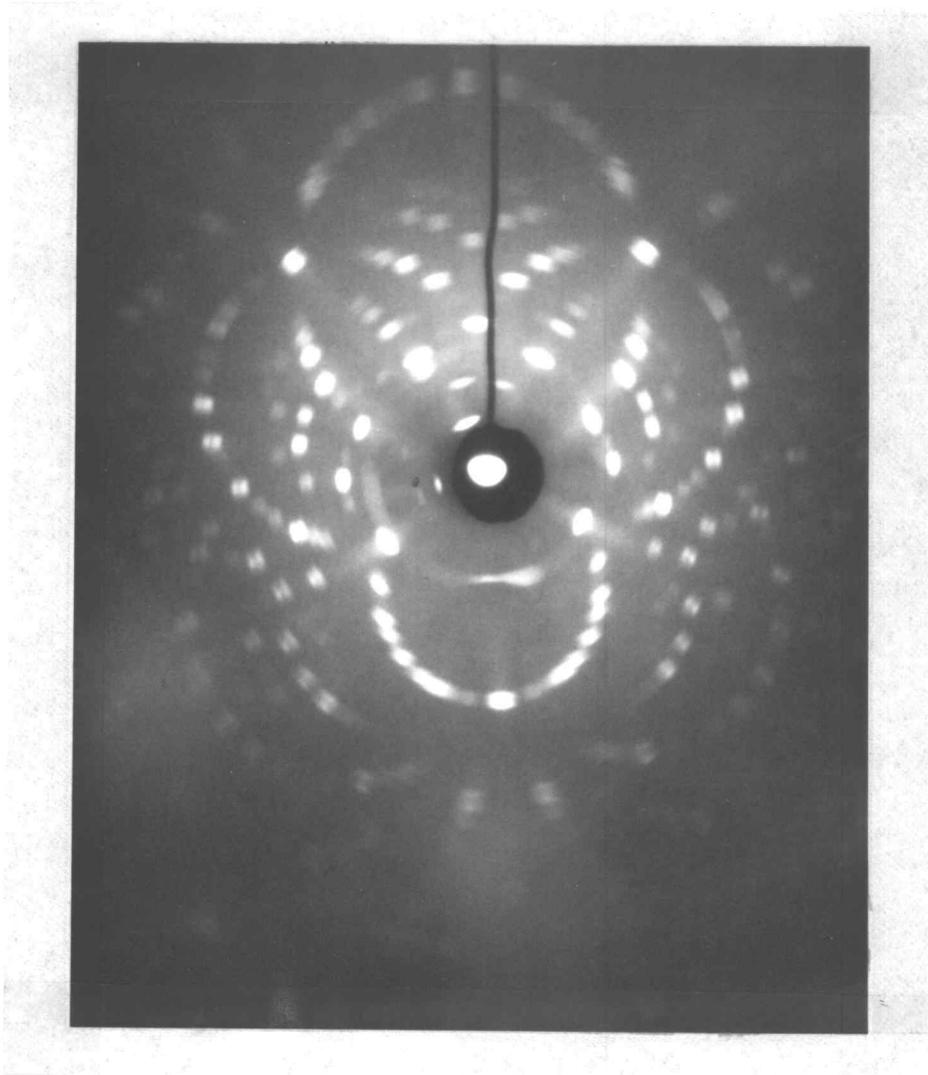


Figure 26. Diffraction pattern of a calcite crystal with a 200 second exposure. The x-ray beam is perpendicular to a crystal face.

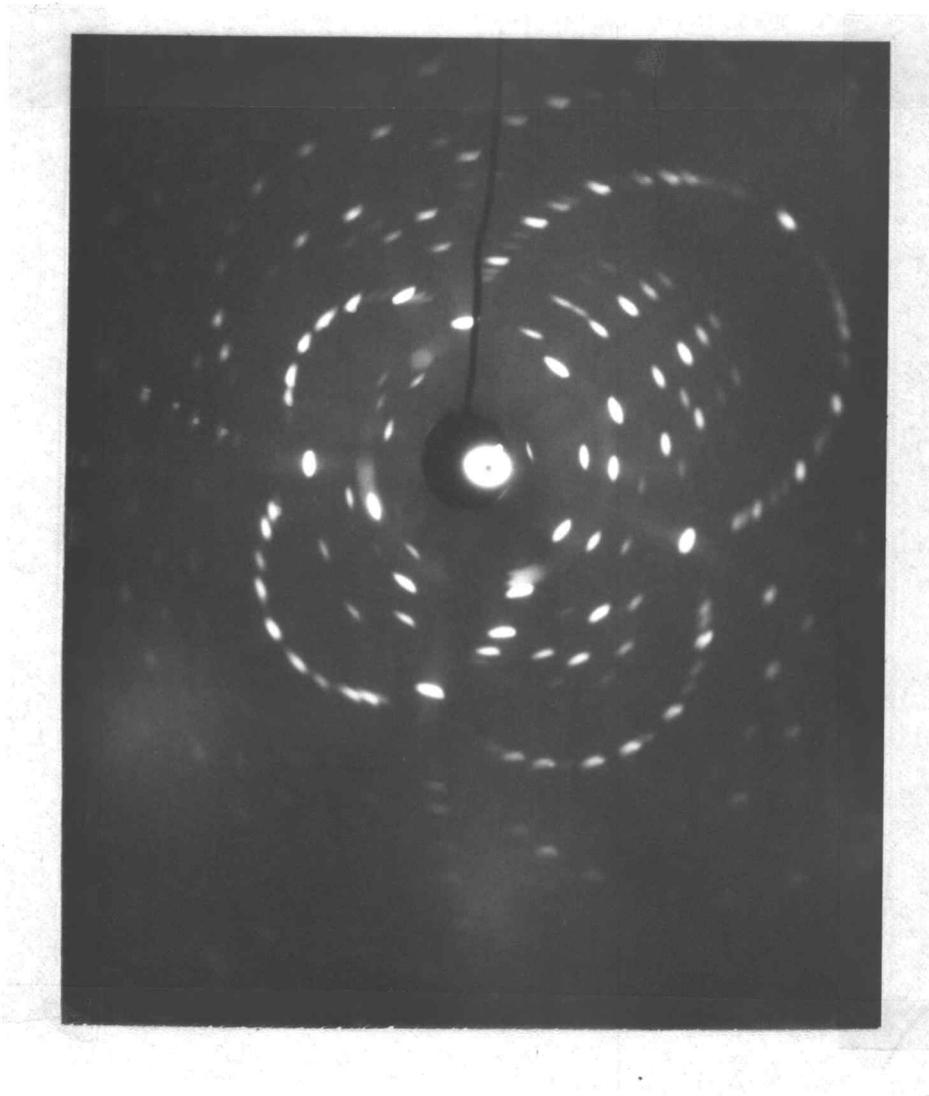


Figure 27. Diffraction pattern of a calcite crystal with a 200 second exposure. The x-ray beam is perpendicular to a cleavage plane.

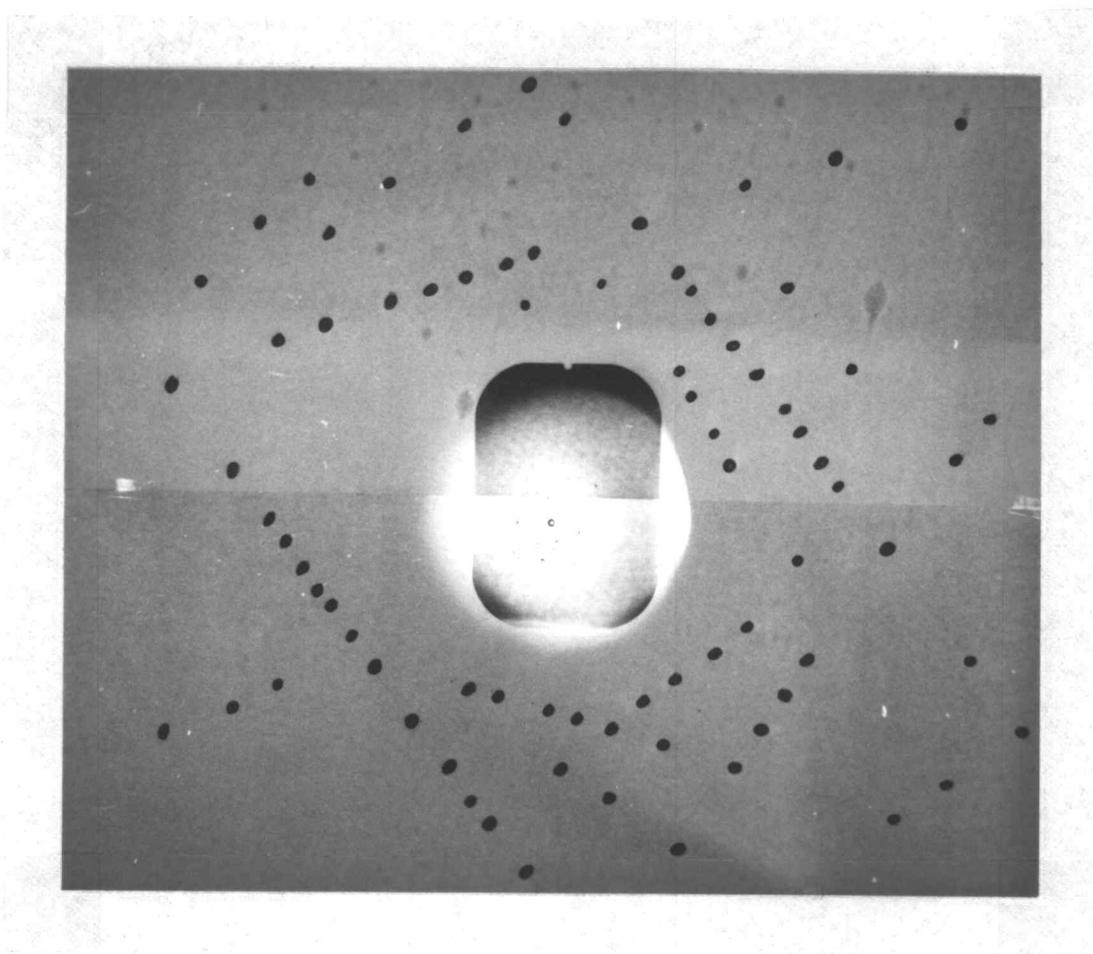


Figure 28. . Gnomonic projection of the calcite diffraction pattern.

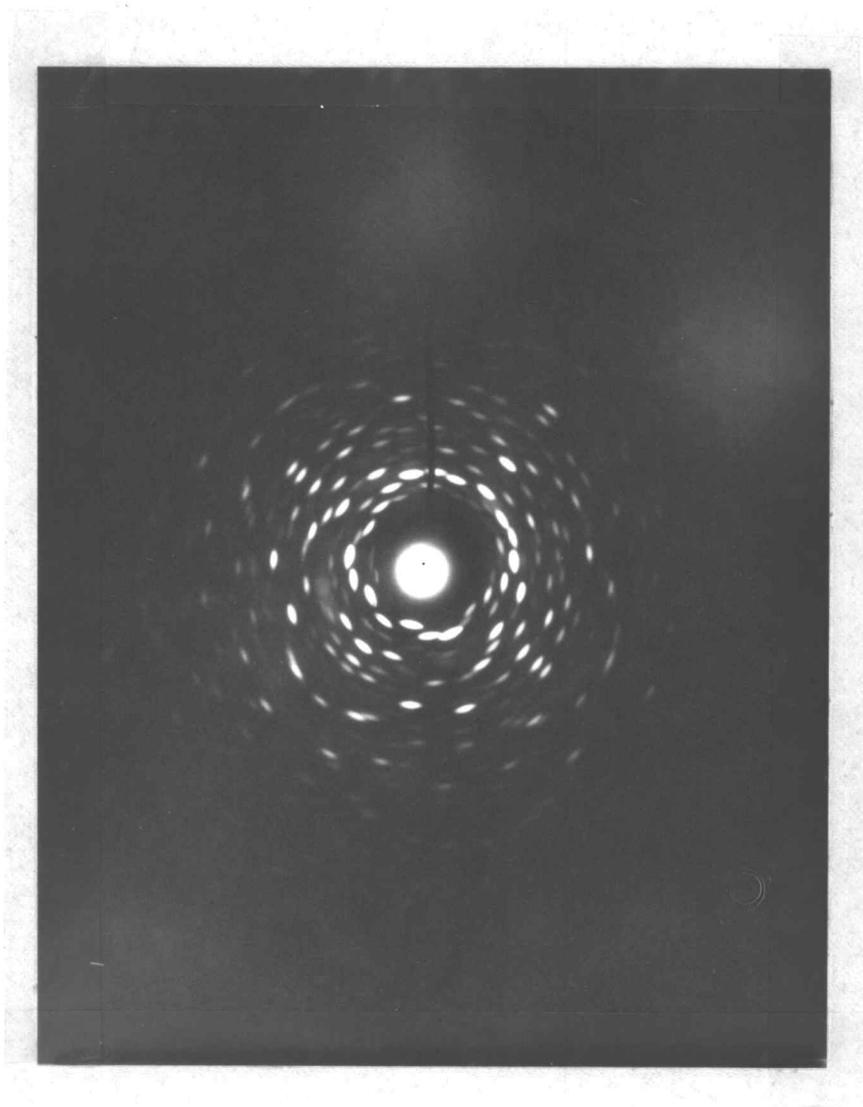


Figure 29. Diffraction pattern of a nickel sulfate crystal with a 100 second exposure.

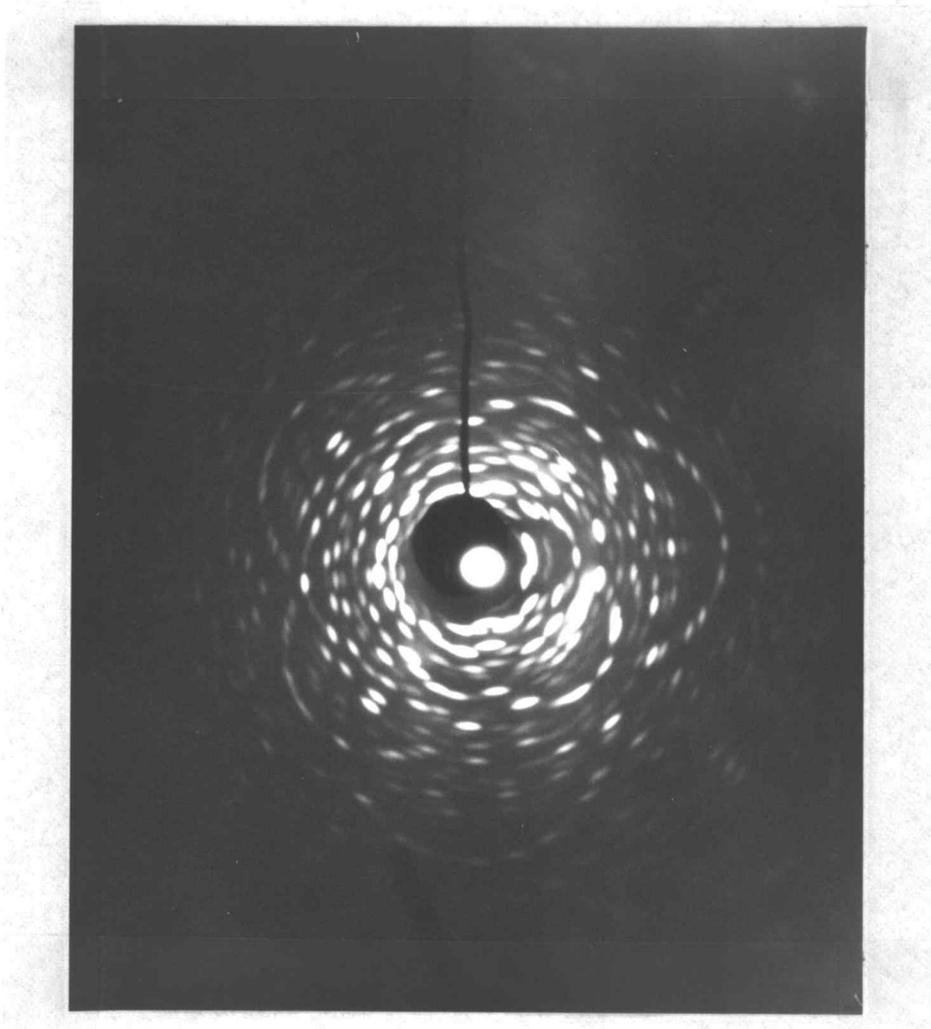


Figure 30. Diffraction pattern of a nickel sulfate crystal with a 200 second exposure.

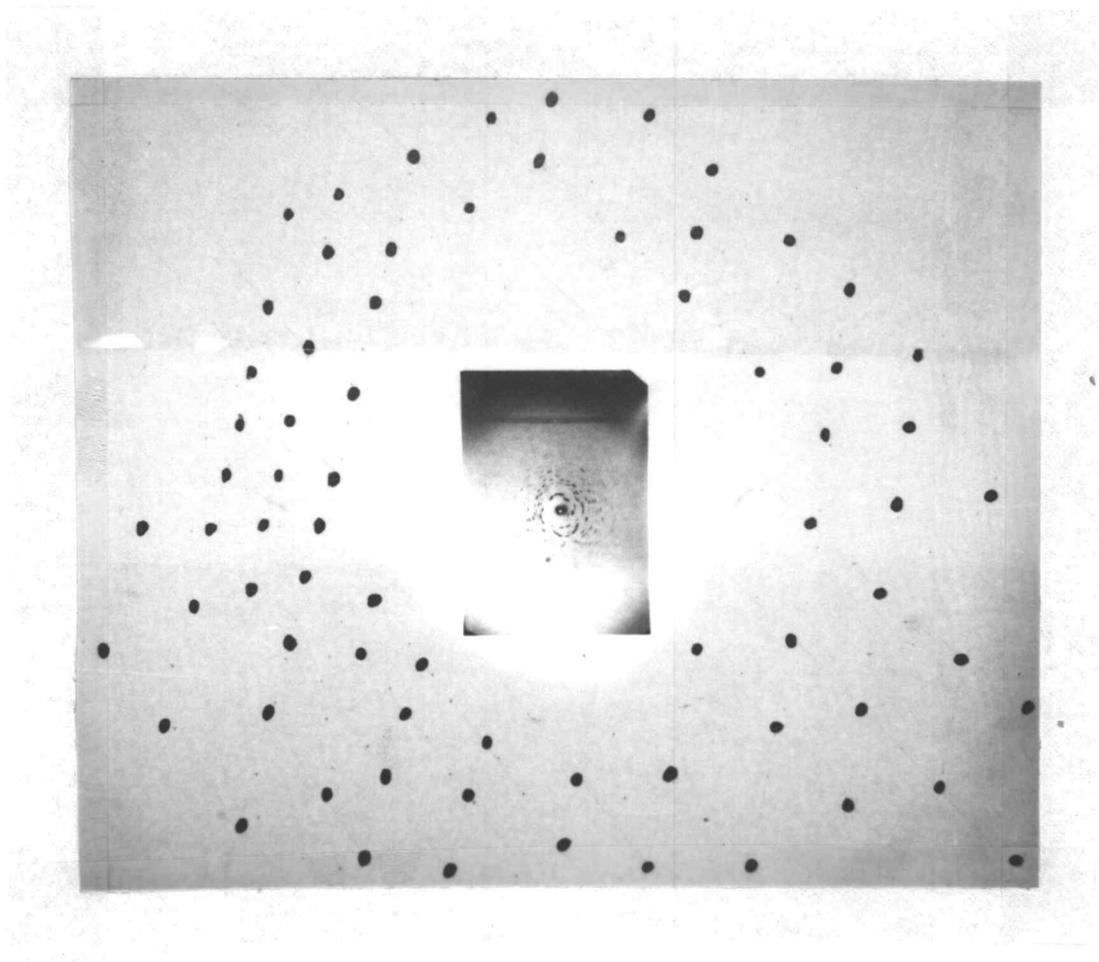


Figure 31. The gnomonic projection of the nickel sulfate diffraction pattern.

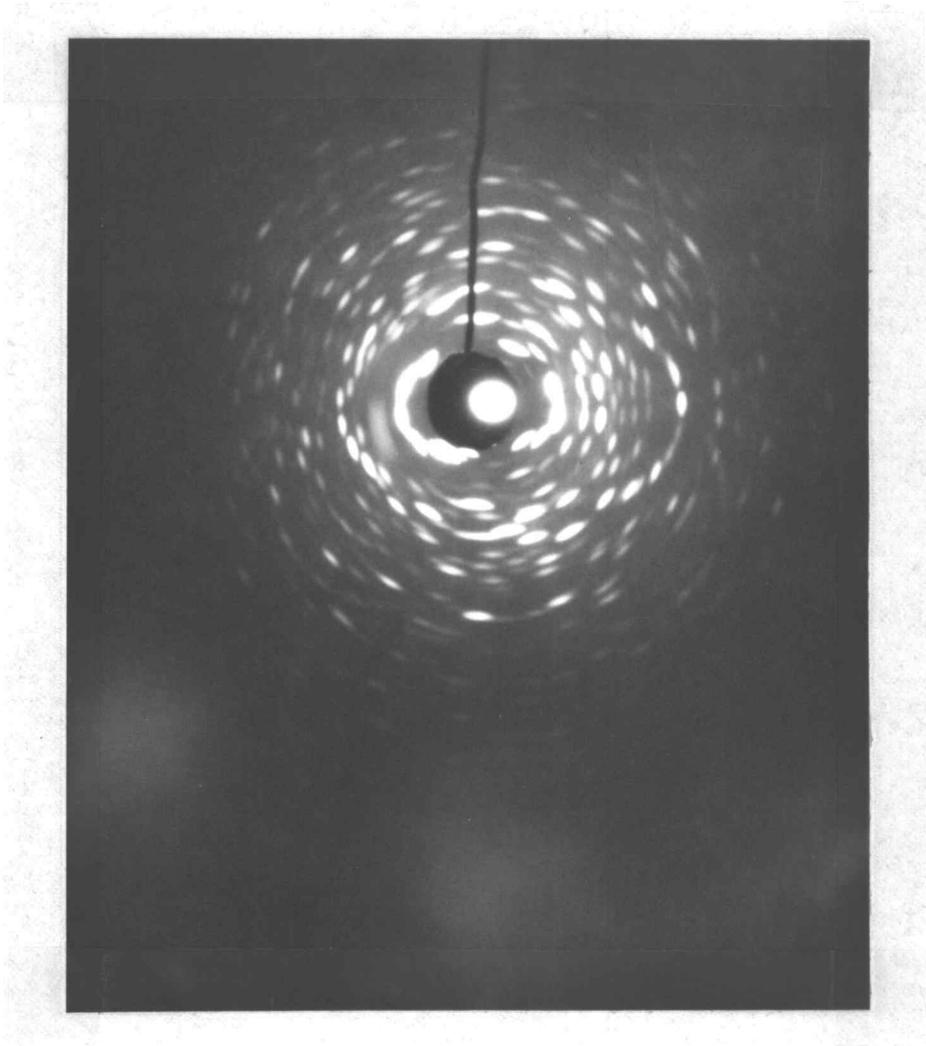


Figure 32. Diffraction pattern of an alum crystal with a 135 second exposure.

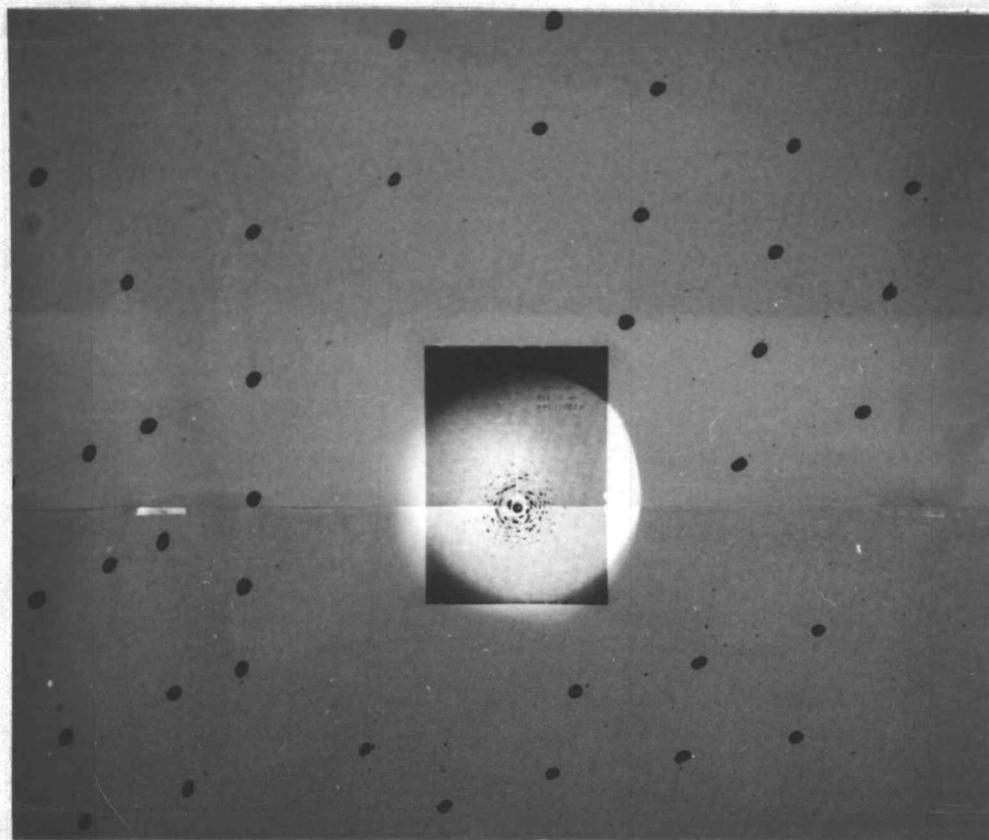


Figure 33. Gnomonic projection of the alum diffraction pattern.

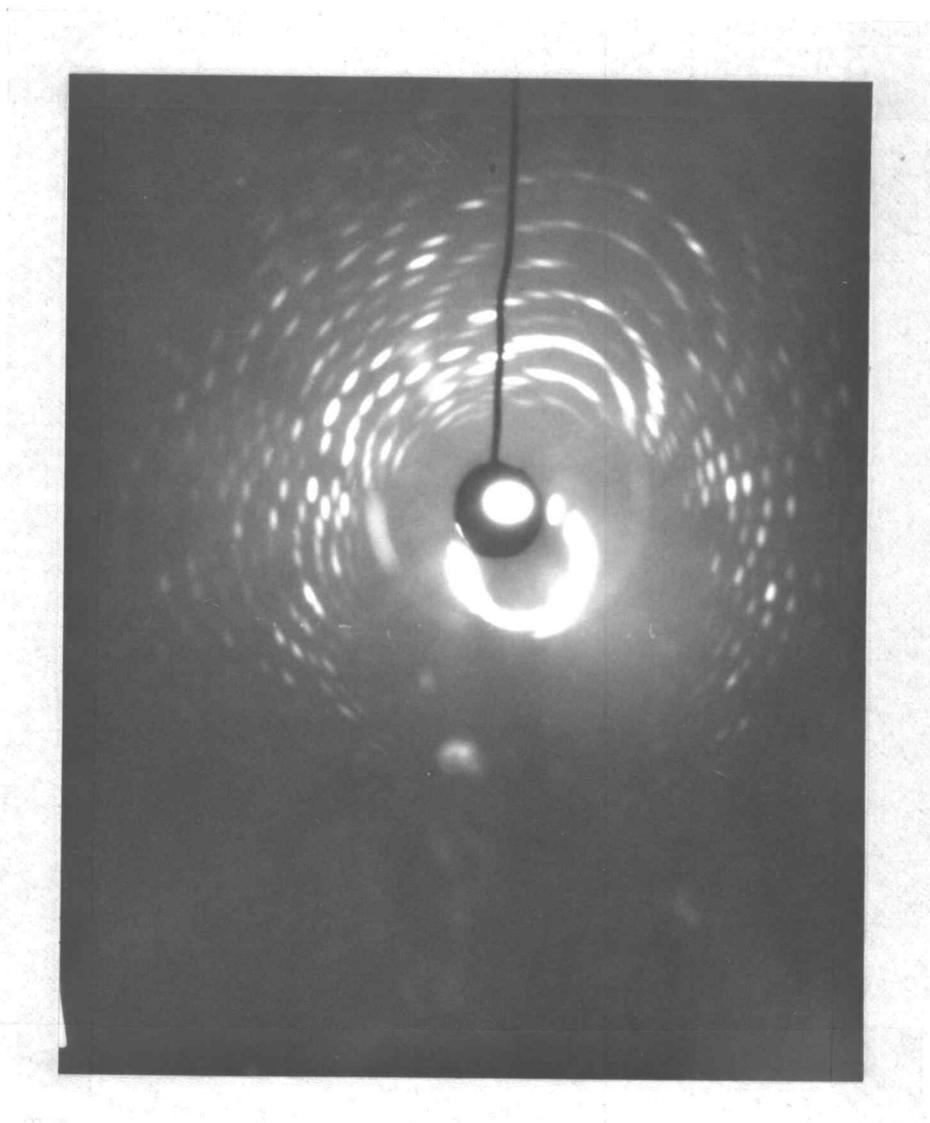


Figure 34. Diffraction pattern of a rochelle salt crystal with a 180 second exposure.

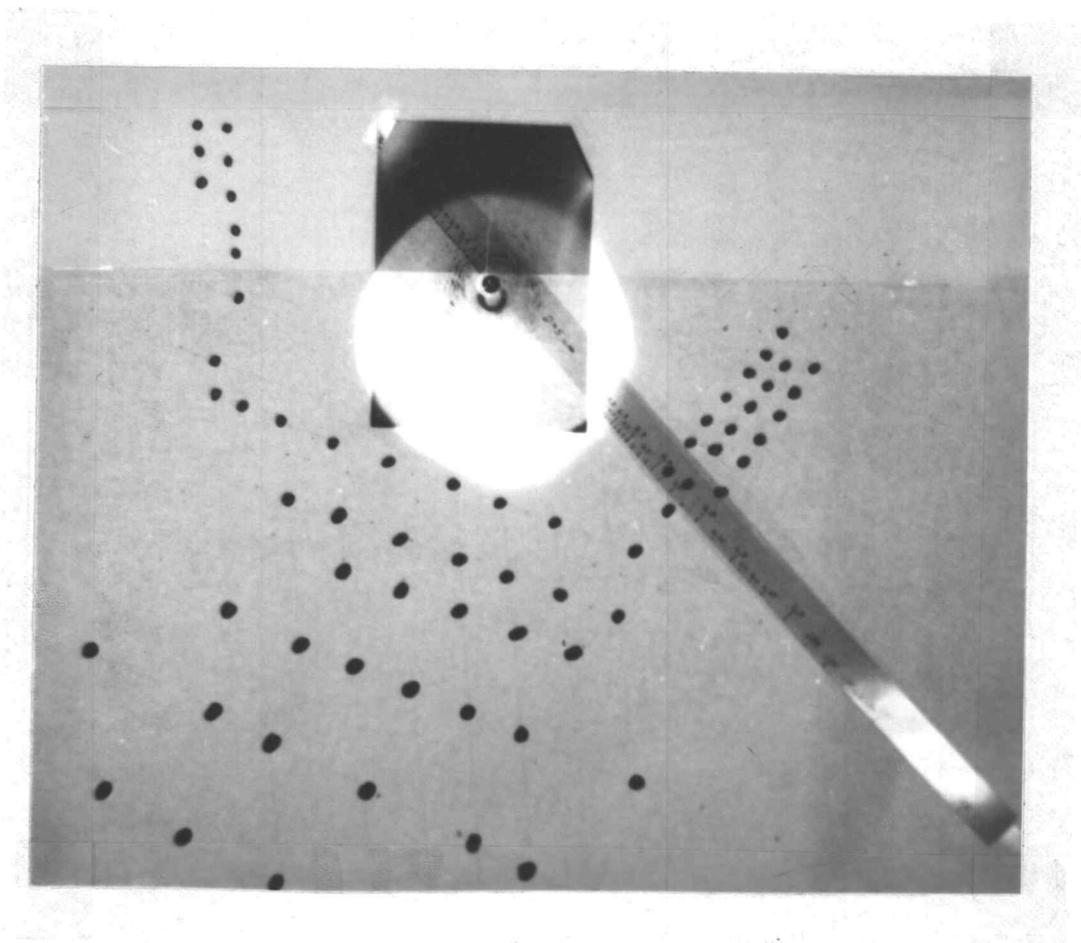


Figure 35. The partial gnomonic projection of rochelle salt with the ruler in place.

plane. It shows the misalignment which is more evident in the gnomonic projection of the diffraction pattern in Figure 28. The projection does have the characteristic appearance of an hexagonal crystal.

In Figure 29 is a 100 second exposure diffraction pattern of nickel sulfate, and Figure 30 is a 200 second exposure of the same crystal, showing the relationship of diffraction pattern to exposure times. In Figure 31 is the gnomonic projection of the tetragonal nickel sulfate hexahydrate crystal. The pattern is again of converging lines due to misalignment, but it shows the characteristic pattern of a tetragonal crystal, a pattern of squares.

In Figure 32 is the diffraction pattern of the laboratory grown ammonium alum crystal. It is a cubic crystal and the projection of the pattern in Figure 33 is of this shape but again skewed, due to misalignment.

In Figure 34 is a diffraction pattern of rochelle salt, a crystal with a rhombohedral structure. The incomplete gnomonic projection is shown in Figure 35 with the ruler in position to demonstrate the operation of making the gnomonic projection.

## SUMMARY AND CONCLUSIONS

The objective of this thesis was to present a design and a completed device for teaching x-ray diffraction principles. In achieving this objective, the device has been constructed so that it is fast, safe and easy to operate yet produces a Laue diffraction pattern of a quality sufficient for a simple instructional demonstration.

For better understanding of the development of x-ray diffraction and the people involved, an historical treatment of x-ray diffraction has been briefly presented.

The subject of crystallography has been superficially presented to acquaint the reader with basic concepts of crystal structure.

The phenomenon of x-ray diffraction has been discussed with respect to two theories, that of M. von Laue and W. L. Bragg. X-rays have been briefly discussed as has diffraction. The production of Laue diffraction patterns has been described and the analysis of these patterns by means of the gnomonic projection has been elucidated.

From all this preliminary work has been taken the nucleus of the design for the x-ray diffraction teaching device. Based on the work of Laue, a simple diffraction camera has been designed and constructed. The results are diffraction patterns on five by seven inch films, of some typical crystalline substances. The analysis of

the diffraction pattern is carried out using the gnomonic projection. Though the patterns obtained and thus the projections are not perfectly symmetrical, the results yield the same kind of information as they would if perfectly symmetrical. The only difference being one of orientation of the crystal in the diffraction camera.

The teaching device is designed in such a way that the radiation exposure to personnel does not exceed permissible levels. The leakage rate at six inches from the tube housing is less than 0.5 mR in an hour. The only hazard is the primary beam, which is stopped at the film plane. Effectively then, only an 8 cm area exists in which the primary beam is exposed. The student is cautioned against this in the operating procedures. Scatter from this beam is minimal. While in operation, the student should remain behind the x-ray machine, thus eliminating all hazard.

In conclusion then, an x-ray diffraction teaching device has been presented which will, with proper application, demonstrate the principles of x-ray diffraction. The device is not of a complicated nature and can be operated with a minimum of supervision.

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## APPENDICES

## APPENDIX I

## THE OPERATING PROCEDURE

To obtain the diffraction pattern the following procedure should be followed.

I. Turn on the x-ray machine.

Adjust the x-ray machine to 20 mA and 55 kVp. This is done by setting the SELECTOR SWITCH on TEST, using the hand switch, and adjusting the mA controls and the kVp controls to the proper settings.

II. Turn off the x-ray machine.

1. Align the x-ray head.

With the shielding removed, by removing the 1/16 inch lead shields on the back and pulling the remaining shield forward, the x-ray head is made perpendicular with respect to the support platform on the table top on which the shield rests. The shield is replaced and made perpendicular to the x-ray head with respect to the table top. When these two adjustments have been made, the collimator should be in line with the focal spot of the x-ray tube and produce an intense circular beam of x-rays. This may be checked by using a piece of film and the appropriate exposure.

2. Align the crystal specimen.

The crystal specimen, mounted in the rod in the hinged

carriage, is placed on the optical bench. The height is adjusted to cover the exit hole, visible through the hole in the protective door. The horizontal position is adjusted by loosening the wing nuts under the table top and moving the optical bench to the right or left until the crystal specimen covers the exit hole of the collimator.

3. Position the film holder.

The film holder, with the loaded Par-Speed cassette, is positioned on the optical bench to a spot 5 cm behind the crystal specimen. The 5 cm distance is measured to the film, not to the face of the cassette. The 1/2 inch diameter, 1/8 inch thick lead beam stop is positioned on the face of the cassette in front of the primary beam.

4. Set the exposure timer.

Plug the exposure timer outlet into the foot switch receptacle. Set the SELECTOR SWITCH on TEST. Reset the minute hand of the clock using the reset knob on the front of the timer console, seen in Figure 18. The exposure timer is now ready for operation. The on-off switch on the console will set in motion the clock motor initiating the 10 second on-50 second off sequence of exposures.

III. Obtain the diffraction pattern.

1. Make the x-ray exposure.

Once all the components are aligned, the exposure can begin. This is initiated by turning on the exposure timer and the x-ray machine and allowing the proper amount of time to pass.

2. Remove the lead beam stop and make the center spot.

When the proper exposure time has elapsed, the exposure timer is turned off. The lead beam stop is then removed and the SELECTOR SWITCH is set on TIMER and  $1/4$  of a second is selected. The hand switch is depressed, producing x-rays to make the center spot of the diffraction pattern.

3. Process the film.

After the exposure is made the film is developed using the automatic processor, the instructions for which are in the darkroom. The films can also be processed manually, following the usual procedure.

4. Analyze the diffraction pattern.

With the diffraction pattern available, the gnomonic ruler is used to obtain the gnomonic projection on a large piece of paper. The pattern is indexed and the results can be observed in their final form.

#### IV. Technic to be used.

The technic to be used is as follows: 55 kVp, 20 mA, collimator exit hole-to-crystal distance of 3 cm, crystal-to-film distance of 5 cm as in Figure 36, and an exposure time of 150 seconds at 10 seconds on-50 seconds off.

#### V. Summary.

In summary, the x-ray machine is adjusted to the proper

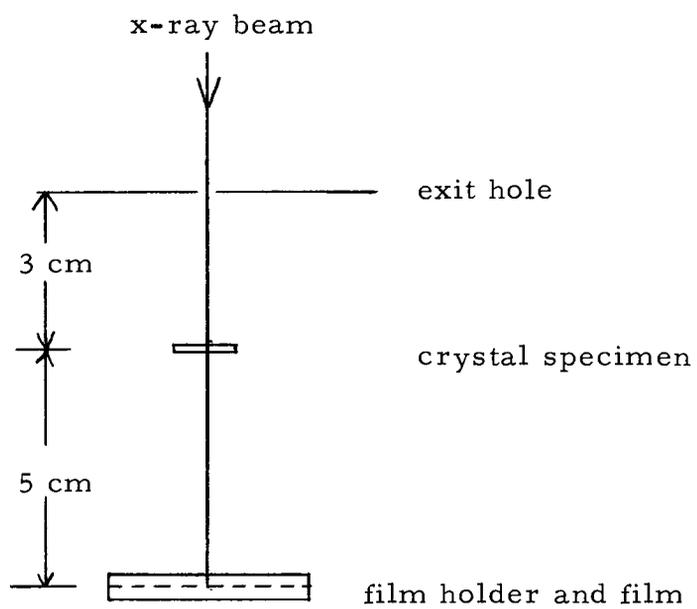


Figure 36. A schematic diagram of the distances involved in obtaining the diffraction pattern.

technic and the exposure timer is adjusted. With the x-ray machine off, the x-ray head and collimator can be aligned. The crystal and film holder are then aligned. The diffraction pattern is then made. The film is then developed and the resultant pattern is analyzed.

If the diffraction pattern is not good, the probable cause is in the alignment of the x-ray beam and the crystal. This should be re-examined and adjusted, using the developed film as a reference.

## APPENDIX II

## LABORATORY EXPERIMENT IN X-RAY DIFFRACTION

Science is a way of thinking about reality. The primary purpose of a laboratory such as this is to provide an opportunity to practice this kind of thinking in a situation of contrived simplicity. Even the simplest experimental arrangement has far more complexity within itself than is contemplated in the theory. One of the benefits to be gained is a first hand experience with the phenomenon of x-ray diffraction, something discussed in lecture but not part of ordinary experience.

The Apparatus and Its Use

The x-ray diffraction unit is based on the principles set forth in 1912 by M. von Laue. It utilizes a single crystal as a diffraction grating in three dimensions. The collimated beam of x-rays produces a diffraction pattern from the crystalline specimen which is recorded on x-ray film. The diffraction pattern can be analyzed to give some information about the crystal structure.

Theory

Diffraction of x-rays from a crystal obeys Bragg's Law. The

law states

$$n\lambda = 2d \sin\theta$$

where  $\lambda$  is the wavelength of x-rays,  $d$  is the distance between lattice planes,  $2\theta$  is the angle at which the x-rays leave the crystal as measured from the direction in which the x-rays would have continued had the crystal not been there, and  $n$  is the so-called order of diffraction.

The diffraction pattern can be analyzed through the use of the gnomonic projection. A convenient tool is the gnomonic ruler, the operation of which is related geometrically to the diffraction plane. The theory of gnomonic projections in relation to the Laue pattern is explained at length in previous pages.

### What to Do

The primary objective of this laboratory is to obtain a diffraction pattern. The operating instructions of the Laue camera tell how to get this pattern. Followed carefully a diffraction pattern will be produced. Caution, x-rays are harmful! When the diffraction camera is operating, remain behind the control console. Can you explain the origin of the various diffraction spots?

When the diffraction pattern has been obtained, make a gnomonic projection of it using the ruler provided for this purpose. Since the theory of a gnomonic projection is known to you, can you

interpret your diffraction pattern? Can the spots be indexed as to their respective diffraction plane?

What can be said about the orientation of the crystal with respect to the x-ray beam?

The answers to the above questions can be found in the body of this paper. Further detail can be found in several references (3, 18, 25, 32, 39, 50, 57).

## APPENDIX III

## SURVEY DATA OF THE TEACHING DEVICE

To determine the exposure rates from the shielded x-ray diffraction teaching device, a survey was performed using a Victoreen 440 survey meter. The results are shown in the polar plots of the device with the beam blocked, Figure 37, and with the crystal and film in place and the beam open, Figure 38. Notice that at a distance of four inches from all components that the exposure rate is less than 0.5 mR in one hour. This more than satisfies the standards set for conditions in educational institutions (44). Consequently this is a safe device for student operation.

In Figure 37 the dashed line around the shielded tube head represents a boundary outside of which the exposure rate is less than 0.4 mR in one hour. The distance from the focal spot to the boundary is on the average ten inches while from the shield to the boundary is but four inches.

In Figure 38 the dashed line around the open beam, sample and film holder represents a boundary outside of which the exposure rate is less than 0.4 mR in one hour. The distance from the components is four inches. In direct line with the primary beam, the distance is eight inches from the film plane.

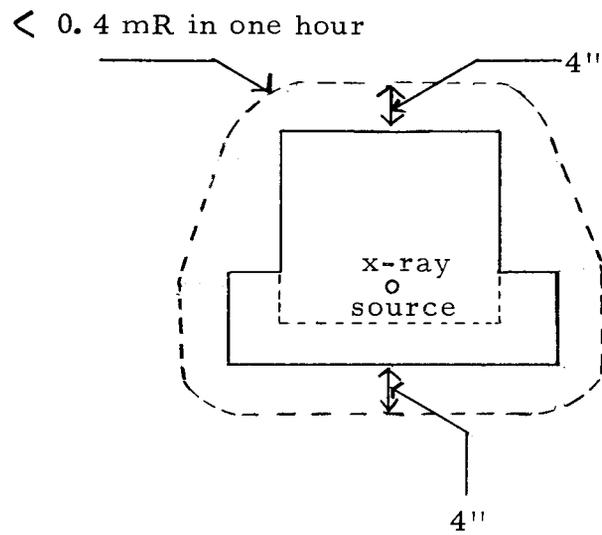


Figure 37. A polar plot of leakage exposure rate from the shielded x-ray tube unit with the beam blocked.

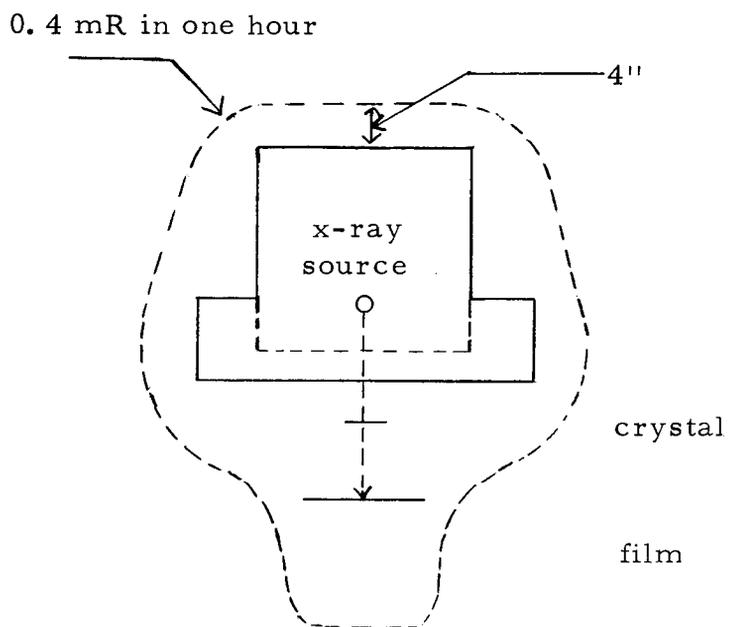


Figure 38. A polar plot of exposure rate around the components of the teaching device with the beam open and the crystal and film in the beam.