Mineralogical applications of a two-crystal Weissenberg X-ray goniometer

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Introduction.

THE study of minerals, using X-ray goniometers in place of optical instruments, has proceeded far in recent years. It is now a common practice among mineralogists and petrologists to confirm the nature of a given specimen by X-ray methods when there is some doubt about the identification by the older methods. The occurrence of pseudomorphs sometimes leads to misidentifications even in crystals of large size. If the material is cryptocrystalline it may be almost impossible to make an identification by optical methods. In such cases X-ray photographs may be of great assistance. When the atomic arrangement is simple, giving a powder photograph that has only a few lines, the identification can be carried out by using standard indexes of powder photographs or, often more simply, by comparing the photograph of the unknown mineral with those of likely known minerals. If the mineral to be identified occurs in single crystals it is usually better not to take powder, but rather single-crystal, photographs. From such photographs the cell dimensions, axial ratios and angles, and most of the symmetry elements may be found. Many minerals are solid solutions and the cell dimensions change with the composition. A measurement of the cell dimensions may enable the composition to be determined. For such measurements, however, high accuracy is required; special types of goniometer and methods of using them have been developed for the purpose.

The distribution of atoms within the unit cells of many minerals is now well known and much research is directed to completing this branch of mineralogy. For such studies a number of experimental techniques have been devised, mostly depending at some stage on accurate information concerning the intensity of X-ray reflections from particular atomic planes. For this purpose oscillation or Weissenberg photographs have been extensively used, though ionization- or counter-spectrometers have also proved of value. The measurement of the intensity of a particular X-ray reflection is beset with two difficulties.

One concerns the crystal itself and the other the recording apparatus. In order that the measurement shall have significance the conditions under which the reflection occurs must be strictly defined. For example, according to one method, the reflecting surface must be flat and large enough to intercept the whole X-ray beam. Alternatively the crystal must be so small that it is completely bathed in the X-ray beam and, at the same time, it must be so weakly absorbing that the X-rays can pass through it with less than about 30 % reduction in intensity. The recording apparatus must satisfy the requirement that all X-ray reflections from planes on a given crystal must have the same chance of registering their intensity. For this purpose the X-ray beam must be constant in intensity and the photographic film must be equally sensitive for each crystal reflection. These requirements are not easily met and it is especially difficult when the reflections which have to be compared come from different crystals. It was in connexion with this problem that the present instrument was originally devised. (Wooster, 1948.)

The chemical analysis of minerals has been carried out for many years, but constant efforts are made to reduce the labour and the time required for performing such analyses. Spectrographic and other techniques can be employed to determine the presence or amount of certain elements with greater ease than the older methods. A method, as yet little used, based on the differences of absorption of X-rays by the chemical elements can also be employed. This is unlikely to be used except for a limited number of elements, but as these include many of the common metals the method may prove important. The instrument described below is well suited for this kind of determination of X-ray absorption coefficient.

General description of the apparatus.

The Weissenberg type of X-ray goniometer provides for simultaneous rotation of the crystal and translation of the camera. The X-ray beam, usually horizontal, falls on a crystal oscillating about a horizontal axis perpendicular, or nearly perpendicular, to the X-ray beam. The cylindrical camera is coaxial with the axis of oscillation of the crystal and moves in a direction parallel to that axis. In fig. 1 there is a general view of the apparatus. The X-ray beam enters from the back through a horizontal cylindrical aperture, and after striking the crystal falls upon the objective of the tele-microscope.[1] The horizontal axis on which both crystals are mounted runs from left to right and intersects the axis

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[1] A microscope that can be used as a telescope.

of the telescope. The two goniometer heads can just be seen pointing towards one another in fig. 1. When every adjustment has been made the two crystals are 3 mm. apart. The cylindrical camera is constructed from two hemicylinders since it is not possible to have two crystals mounted 3 mm. apart and place around them a whole cylinder. In fig. 1 the lower half of the camera can be seen in its usual place on the instrument and the upper half is lying in front of the base. The camera is

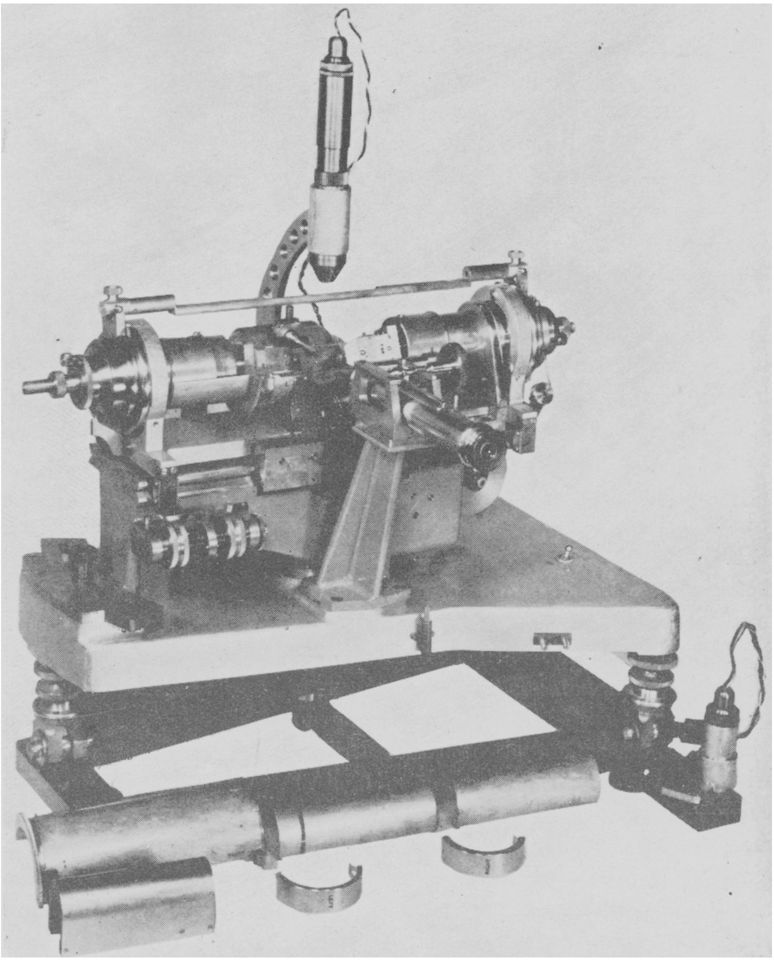

FIG. 1. Genera view of the two-crystal Weissenberg goniometer.

mounted on a carriage similar to that of the moving roller on many typewriters. This carriage has two V-grooves which are opposite corresponding V-grooves on the track. Ball-bearings between these V-grooves enable the carriage to move freely from left to right and back again. Under the track there is visible in fig. 1 a box which contains the synchronous driving motor. The motor rotates a worm long enough to move the camera through 10 cm. by means of a nut riding on the screw thread. A steel tape is attached to one end of the camera carriage and passes over a small pulley to a cylindrical drum on the right-hand end of the crystal axis. This tape is kept taut by a second tape attached to the same drum at one end and to a spring-box at the other. In this way a uniform movement of the camera and the crystal can be obtained and while the crystal turns through 200° the camera moves along 100 mm. At each end of the sideways movement of the camera the motor is reversed, simply by stopping it. As the motor is synchronous it can rotate equally well in either direction and if stopped from going in one direction it reverses and rotates in the opposite direction (Wooster, 1948). The left-hand goniometer head is rotated by means of a bar which can be rigidly clamped to both goniometer heads. This is shown at the top of the instrument in fig. 1. The goniometer heads are mounted on a frame which, like the camera carriage, can move along the track on ball-bearings; the extent of its movement never exceeds 5 mm. and between its resting positions is a distance of 3 mm. A toothed-wheel mechanism (see fig. 2) is automatically operated every time the camera carriage reaches the right-hand side and this exchanges the crystal which is in the X-ray beam with the one which previously was 3 mm. to one side of the beam. The box under the track is pivoted about a vertical axis running through the base of the instrument, so that, without altering the position of the collimator relative to the X-ray tube, equi-inclination Weissenberg photographs may be taken. The maximum possible angle of rotation of the base is ± 20°. Since it is essential in taking Weissenberg photographs that only one layer line should be recorded on any photograph, all unwanted reflections are excluded by cylindrical screens with circumferential slots. These are necessarily in the form of hemi-cylinders which, when placed in position on the instrument, form cylinders coaxial with the camera. The lower halves are shown in place on the instrument in fig. 1 and the upper halves are lying in front of the base. The right-hand screens are attached to a cylinder which does not move when the goniometer heads move sideways. The left-hand screens move with the goniometer heads, sliding outside the right-hand screens. Thus it can be arranged that when the right-hand crystal is in the beam, X-rays which have been reflected through both the upper and lower hemi-cylindrical screens can reach the film, whereas when the left-hand crystal is in the beam, the lower or the upper opening in the screen is closed while the other one is open. The crystals are accurately set by taking an oscillation photograph. For this purpose the cylindrical screens are moved sideways from the centre, leaving a free space. Only the upper half of the camera is used, and this is mounted by means of two special spacing-rings on the screens so that it is co-axial with the axis of oscillation of the crystal. The base of the instrument has three levelling toes which rest in a frame provided with the usual conical depression, groove, and plane for the three toes. This frame is mounted on three wheels which enable the instrument to be moved towards, or away from, the X-ray tube with ease. The wheels run on a T-shaped frame and by means of a clamp the instrument may be fixed in any desired position relative to this frame.

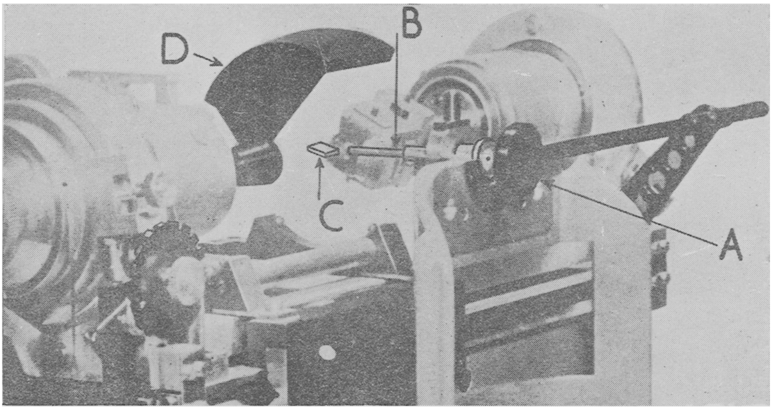

FIG. 2. Photograph of two-crystal Weissenberg goniometer arranged for the measurement of absorption coefficients. A, holder for absorbers; B, collimator; C, monochromatizing crystal; D, camera.

Applications of the two-crystal method.

1. *The comparison of the intensities of reflection of particular planes in one crystal with those of particular planes of a standard crystal.*

It is usual in the determination of a crystal structure to require a knowledge of the absolute structure amplitudes, F_{hkl}, of at least a certain number of reflecting planes. These structure amplitudes are connected with the atomic parameters of each of N atoms by the formula

$$F_{hkl} = \sum_{n=1}^{n=N} f_n \cos 2\pi(hx_n+ky_n+lz_n)$$

when a centre of symmetry is at the origin of the co-ordinate system. In the absence of such a centre of symmetry the formula is more complicated but the physical principles involved are the same. In this formula

N = number of atoms per unit cell,
f_n = atomic structure factor for the nth atom,
x_n, y_n, z_n = co-ordinates of the nth atom,
h, k, l = indices of the reflecting plane.

The simplest application of this method arises when $N = 2$. Then,

$$F_{hkl} = 2f \cos 2\pi(hx+ky+lz),$$

and from three values of F_{hkl}, obtained by the study of only three reflections, x, y, z can be determined. In principle, therefore, an absolute measurement of F_{hkl} leads to a direct determination of the atomic co-ordinates, when the structure is simple. When the structure is more complicated various combinations of experimental observations are employed to determine x_n, y_n, z_n for all values of n. Some of these methods depend on a determination of *relative* values of F_{hkl}, which can most conveniently be obtained from a Weissenberg photograph, while others require *absolute* values of F_{hkl}. The absolute value of F_{hkl} can be obtained by finding the ratio of the intensity of the reflection from the particular plane *hkl* to the intensity of the direct beam. Account must, of course, be taken of the fact that the crystal reflects over a certain range of angle and not only at a particular setting. It is difficult to make such an absolute measurement because the direct beam is usually not strictly monochromatic and any direct beam will produce a much greater effect on a photographic film than corresponds to its monochromatic component. To overcome this difficulty it is usual to calibrate the intensity of the direct beam by reflecting it from a crystal of known reflecting power. This method, however, introduces its own difficulties. If the X-ray beam is reflected from the standard crystal at a different time from that during which it is reflected from the crystal under examination, the intensity of the incident beam may not be the same during the two reflections. The mains voltage and frequency are always fluctuating, so that the maintenance of an X-ray beam of sufficient constancy is almost impossible over periods during which X-ray photographs are taken. It therefore becomes necessary to interchange the two crystals in the X-ray beam a large number of times during the exposure if a sufficiently accurate comparison is to be made.

A further difficulty arises in registering the intensity of two reflected beams with equal sensitivity. If the records are made on separate pieces of photographic film, it is difficult to avoid errors due to differences in development and processing generally. These difficulties can be overcome only by recording the two beams, one from the standard crystal and the other from the crystal under examination, on the same piece of film. Thus the two essentials of a satisfactory instrument for the determination of absolute values of F_{hkl} are (1) a frequent exchange in the X-ray beam of the two crystals and (2) the record of both reflections shall be made on the same film, in such a way that they are clearly distinguished from one another. The instrument described here satisfies both these conditions. Two experiments are described below, both illustrating applications of the two-crystal goniometer to the problem of comparing the intensities of reflection from a standard crystal and another crystal.

1.1 *Comparison of the integrated reflections of the h00 reflections of calcite and rock-salt.* As mentioned above, in one type of measurement a plate of crystal is used which intercepts the whole X-ray beam. The integrated reflection, obtained by recording all the radiation reflected as the crystal rotates at a uniform speed through the reflecting setting, is, under this experimental condition, easily related to F_{hkl}. In the following experiment a cleavage plate of rock-salt was mounted on one goniometer head and a cleavage plate of calcite on the other goniometer head. By manually controlling the movement of the toothed wheel the ratio of the number of times that the rock-salt and calcite crystals came into the beam was 1 : 6, 1 : 5, 1 : 4 in the three photographs of fig. 3 (reading left to right). The strongest reflections are 200 for rock-salt and 100 for calcite (unit cell of same shape as the cleavage rhombohedron). It will be seen that these spots are most nearly equal for the middle photograph where the ratio of exposures was 1 : 5. Thus it is possible to see directly that for these particular crystals of rock-salt and calcite the ratio of the integrated reflections was 5 : 1. A more accurate comparison could be made by using a micro-photometer to determine the total blackening of the spots. This method could be applied to the comparison of any series of reflections arising from the first, second, &c., orders of reflection from a plane parallel to the surface of the crystalline plate. The second experiment is directed to showing how the instrument may be used for determining the absolute values of a series of reflections from planes having indices such as *hko*, *okl*, or *hol*.

1.2. *The determination of the integrated reflections for a number of planes having a common zone axis.* Most minerals have crystal structures with many parameters and a number of methods have been devised for finding the atomic distribution. One of these depends on a knowledge of the absolute values of F_{hko}, F_{okl}, and F_{hol}, i.e. on the structure amplitudes of planes parallel respectively to the crystallographic z, x, and y axes. A Weissenberg photograph of one crystal set up with the appropriate orientation can provide *relative* values of F_{hko}, but to obtain *absolute* values it is necessary to calibrate the intensity of the incident beam by using two crystals. It is always best to use a standard crystal of somewhat similar chemical and physical character to the crystal under examination. Quartz is therefore a suitable standard for silicate minerals, and in the photograph of fig. 4 quartz is the standard with which natrolite is compared. The crystals used for obtaining the photographs of fig. 4 were needles approximately 0·3 mm. in diameter and 3 mm. long. For accurate work the crystals must be carefully measured to determine the effect of absorption of X-rays in the specimen and also the effect of the actual shape in favouring certain reflections at the expense of others. The lower half of the photograph records the reflections from the natrolite only while the upper half has these reflections and those of the quartz crystal as well. With care it is easy to distinguish

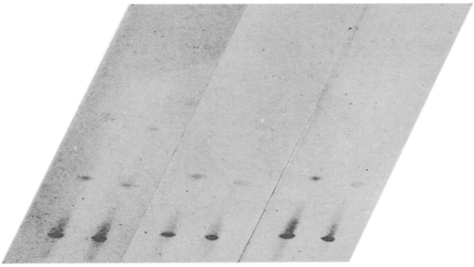

Fig. 3. Portions of three two-crystal Weissenberg photographs comparing the intensities of reflection of rock-salt (left) and calcite (right) in each photograph.

Left-hand photograph	Exposure of rock-salt 1 ,, ,, calcite 6
Middle photograph	,, ,, rock-salt 1 ,, ,, calcite 5
Right-hand photograph	,, ,, rock-salt 1 ,, ,, calcite 4.

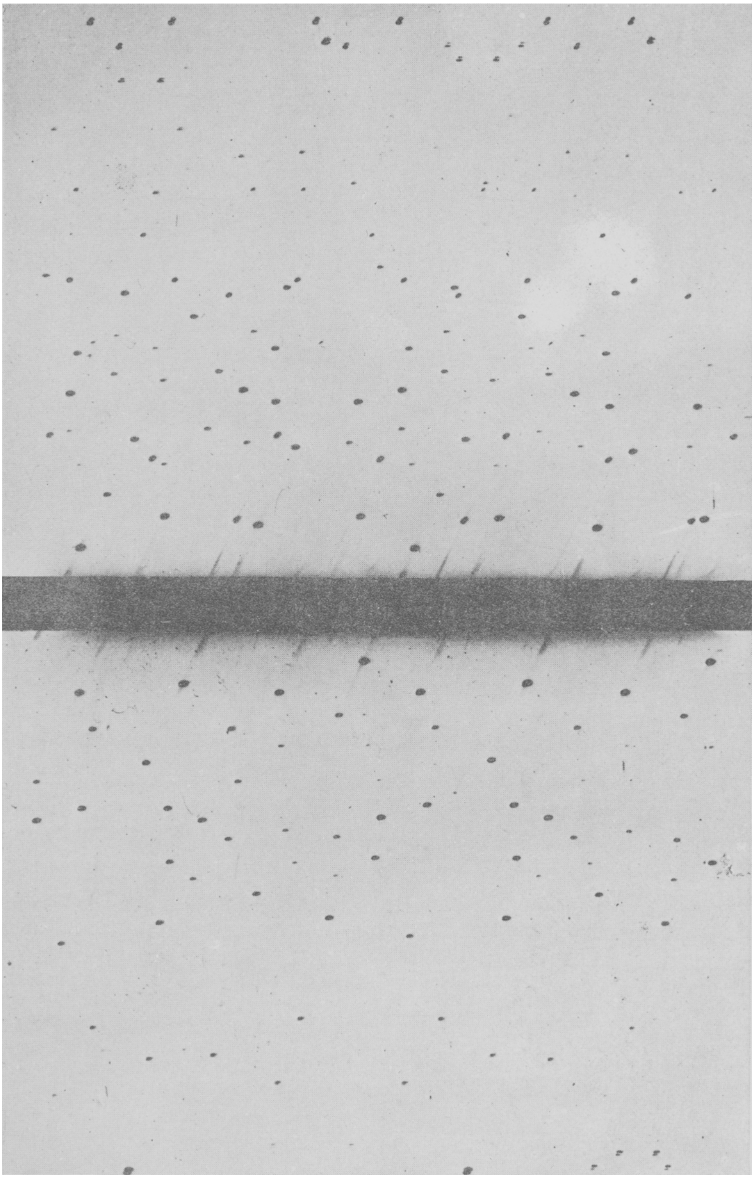

FIG. 4. Two-crystal Weissenberg photograph using Cu-$K\alpha$ and radiation filtered through nickel. *Upper half*: quartz rotation axis [001], superimposed on natrolite rotation axis [001]. *Lower half*: natrolite alone.

between the reflections from the two crystals because the position of the natrolite spots in the upper half can be derived from the position of the corresponding spots in the lower half. In taking this photograph each crystal remained in the X-ray beam for an equal length of time and, during the several hours' exposure, each crystal was moved into and out of the beam some 30 times. Thus any variations in the intensity of the X-ray beam are likely to be of small influence on the accuracy of the comparison between the intensities of reflection from the two crystals. Much valuable work can be done by simple visual comparison of the spots produced by the natrolite with those produced by the quartz, but for accurate work it is necessary to determine the total blackening of the spots with an integrating micro-photometer. The particular crystal of quartz which is used as a standard can be directly calibrated on an ionization- or counter-spectrometer. Any or all of the reflections recorded on the photograph can be registered by a counter spectrometer from the same small crystal using strictly monochromatic radiation. The intensity of the direct beam can be measured immediately before and after the measurement of each integrated reflection. In this way no assumptions need be made about the perfection, or absorption of the standard crystal.

2. *An X-ray method of determining the amount of a particular element in the composition of a mineral.*

2.1. *General principles.* When X-rays pass through any crystal the absorption is determined, except under certain special circumstances, by the number and kinds of atoms passed through and not at all by their arrangement. The special circumstances referred to arise only when a strictly parallel monochromatic beam is employed and an appreciable fraction of the beam is reflected by a set of atomic planes. Usually if in passing through a thickness, t, of a crystal the intensity of the X-rays is reduced from I_0 to I, then

$$I = I_0\, e^{-\mu t}, \tag{1}$$

where μ is the linear absorption coefficient.

An absorption coefficient which is of importance in connexion with minerals is the gram-atomic absorption coefficient, μ_g, since this is readily applicable when the composition is expressed in percentages of metallic oxides. The relation between μ and μ_g is defined by the expression

$$\mu_g = W\mu/\rho \quad \text{or} \quad \mu = \rho\mu_g/W,$$

where W = atomic weight, ρ = specific gravity.

In a compound containing m types of atoms, each formula weight containing n % of atoms of each type, the value of μ is given by

$$\mu = \rho \sum^{m} n\mu_g \Big/ \sum^{m} nW. \tag{2}$$

This formula is valid because, as mentioned above, the absorption effects of different atoms are additive.

The variation of μ_g for Cu-$K\alpha$ radiation (λ = 1·54 Å.) with atomic number of the absorbing element is large, as is shown in Fig. 5. The maximum value for the characteristic $K\alpha$ radiation of copper is that of cobalt which is 20,900. Iron is close to this, being 18,100. Among the elements commonly occurring in minerals calcium and potassium are next in order, having coefficients of 6,890 and 5,590 respectively. The elements Si, Al, Mg, Na, F, and O have small absorption coefficients, all being less than 2,000. Thus in minerals containing one of the transition metals between titanium and cobalt the effect of x % of the transition metal is approximately the same as that of $10x$ % of the elements Na, Mg, Al, and Si. Thus the fraction of the mineral consisting of iron exercises a preponderating influence in determining the absorption

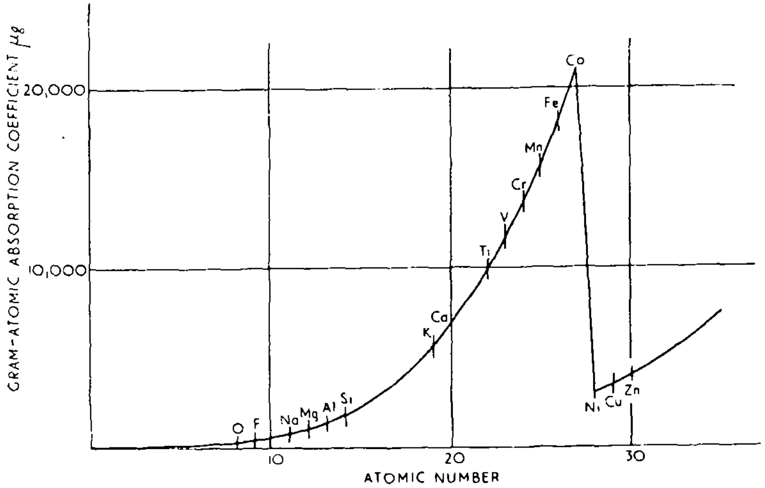

FIG. 5. Diagram showing variation of gram-atomic absorption coefficient with atomic number.

coefficient. If the characteristic radiation of nickel ($\lambda = 1{\cdot}66$ Å.) were used, iron would have the greatest absorption coefficient. Similarly with cobalt radiation ($\lambda = 1{\cdot}79$ Å.), the corresponding element is manganese and with chromium radiation ($\lambda = 2{\cdot}29$ Å.) the most heavily absorbing element is titanium. Thus to each characteristic radiation there corresponds a small group of elements which have a preponderating influence on the absorption coefficient. In the following section this general relation between absorption coefficients and chemical composition is applied to an isomorphous series of iron-containing minerals, namely, the micas.

2.2. *Relation between linear absorption coefficient and chemical composition in the micas.* The micas form a series of minerals in which the iron content ranges from very little to almost 30 %. The variation of the absorption coefficient for copper radiation over the whole series is, therefore, large. To illustrate the method of calculating the absorption coefficient, the biotites listed on p. 630 of Dana's 'Descriptive Mineralogy' (1892) and a few other micas are here chosen. The percentage composition of no.1 on p.630 is given as follows: SiO_2 40·00, Al_2O_3 17·28, Fe_2O_3 0·72, FeO 4·88, MgO 23·91, K_2O 8·57, Na_2O 1·47, H_2O 1·37, F 1·57. The gram-atomic absorption coefficients, μ_g, of the oxides can be readily obtained from the atomic weights and mass absorption coefficients (International Tables for Crystal Structure Determination (1944)). Thus for SiO_2 we have

$$\mu_g = 1690 + 2 \times 203 = 2{,}096$$

and the corresponding figures for the other oxides are Al_2O_3 3,229, Fe_2O_3 36,809, FeO 18,303, MgO 1,191, K_2O 11,383, Na_2O 1,625, H_2O 203, F 332. To apply formula (2) the percentage composition is multiplied by the gram-atomic absorption coefficient for each oxide and the sum of these products taken ($\sum^{m} n\mu_g$). The percentage composition is multiplied by the molar weight of each oxide in turn and the sum taken ($\sum^{m} nW$). The ratio of these two quantities, multiplied by the density, gives the linear absorption coefficient μ. Thus, in this example, for which the density is 2·87, μ is 169 cm.$^{-1}$[*] This computation was carried

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[*] $\frac{\mu}{2{\cdot}87} = (40{\cdot}00 \times 2096 + 17{\cdot}28 \times 3229 + 0{\cdot}72 \times 36{,}809 + 4{\cdot}88 \times 18{,}303 + 23{\cdot}91 \times 1{,}191$
$+ 8{\cdot}57 \times 11{,}383 + 1{\cdot}47 \times 1{,}625 + 1{\cdot}37 \times 203 + 1{\cdot}57 \times 332)/(40{\cdot}00 \times 60 + 17{\cdot}28 \times 102 +$
$+ 0{\cdot}72 \times 160 + 4{\cdot}88 \times 72 + 23{\cdot}91 \times 40 + 8{\cdot}57 \times 94 + 1{\cdot}47 \times 62 + 1{\cdot}37 \times 18 + 1{\cdot}57 \times 19)$

or, finally $\mu = 169$ cm.$^{-1}$.

out for each member of the series of micas on p. 630 of Dana's 'Descriptive Mineralogy' for which the density was given and some phlogopites and muscovites as well. To compare the absorption coefficient with the percentage of iron in the mineral it is necessary to bring Fe_2O_3 and FeO on to a comparable basis. The gram-atomic absorption coefficient for Fe_2O_3, 36,809, is almost twice as great as that of FeO, 18,303, and we may therefore regard one molar weight of Fe_2O_3 as twice as absorbing as one molar weight of FeO. Thus, in the graph of fig. 6, the linear absorption coefficient μ, has been plotted against ($2 \times \%\ Fe_2O_3 + \%\ FeO$). A straight line has been drawn through the points corresponding to the various micas. It is significant that the line is straight, indicating that the iron content alone determines the change in absorption coefficient.

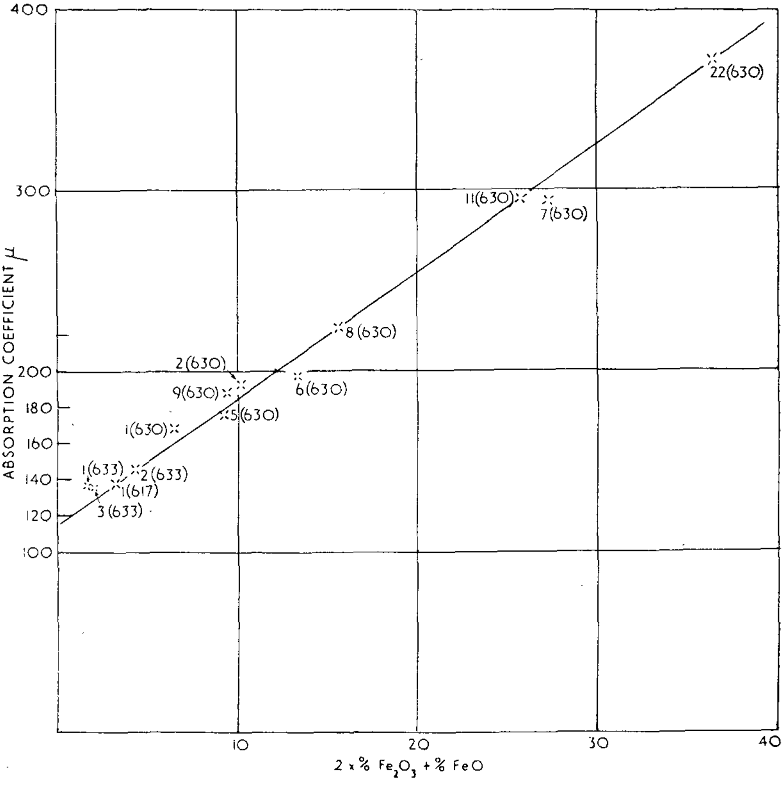

FIG. 6. Diagram showing the variation of linear absorption coefficient with percentage of iron in various micas.

It is also important that the maximum horizontal deviation of the points from the straight line corresponds to just over 1 % of FeO. The accuracy of analysis of the iron-content is unknown, but it is clear that the absorption coefficient could be used in conjunction with this graph to determine the percentage of iron oxide in any of the micas listed here with an accuracy of about ±1 %.

From this example it is possible to see which mineral groups lend themselves to partial chemical analysis in this way. The necessary conditions are as follows: (*a*) a concentration of FeO amounting to a few per cent., (*b*) the absence of a large amount of titanium, calcium, or potassium, unless a constant percentage of these elements is present throughout the series. If the percentage composition of titanium is being studied, then similar conditions can be laid down. Bearing these limitations in mind, the following mineral series are probably suitable for study: pyroxene, amphibole, garnet, chrysolite, tephroite (for manganese), humite, tourmaline, staurolite, mica, and serpentine. In each series it is to be expected that if a measurement of the absorption coefficient can be made accurate to x %, then the percentage composition of the iron or titanium or manganese can be found with an accuracy of $(x+y)$ %, where y depends on the deviations from the straight line in the graph of absorption coefficient plotted against percentage composition of the heavily absorbing element. In the case of the micas y appears to be about ±1.

2.3. *The experimental measurement of the linear absorption coefficient using the two-crystal Weissenberg goniometer.* The measurement of a linear absorption coefficient involves a determination of the thickness, t, of the material traversed by the X-rays and the ratio of the intensity of the X-rays after passing through, I, to that of the X-rays at incidence, I_0. The equation (1) may be written

$$\mu = \frac{1}{t}\log_e\frac{I_0}{I}.$$

It is essential that the radiation should be strictly monochromatic and only crystal-reflected radiation is adequate for the purpose. This is due to the rapid variation of the absorption coefficient with wave-length. In fig. 2 the two-crystal goniometer is shown set up for making these measurements. The goniometer has been turned at right angles to its position in fig. 1, and the additional parts used in this measurement are shown darker. The X-rays pass through the absorbing plate which is mounted on the small disk A. This disk is carried on a rod attached to the frame supporting the goniometer heads. When the frame moves along the horizontal track, as it would do normally in order to interchange the positions of the two crystals, the disk A is moved sideways through 3 mm. By covering the opening in the centre of the disk with two absorbing layers placed side by side, one of a standard substance, e.g. nickel, and the other of the mineral under examination, it is possible to arrange that the X-rays pass alternately through the two absorbers. The thicknesses of these absorbers are adjusted so that the absorption for them both is approximately the same. After passing through the absorber, the X-rays enter the collimator B, which permits a beam $\frac{1}{2}$–1 mm. in diameter to pass through it. This beam then falls on a stationary crystal plate C so orientated that it reflects the characteristic copper radiation. By omitting the bar which normally connects the two goniometer heads together the left-hand crystal can remain stationary in any desired orientation. The crystal C is long enough to reflect the X-ray beam equally well in both positions of the goniometer head. Two areas of the crystal 3 mm. apart reflect the X-ray beams emerging through the two absorbers, but it is easy to test the equality of the reflecting power of these adjacent points on the crystal surface simply by repeating the experiment with the absorbers interchanged. After reflection from the crystal C, the X-ray beam strikes the photographic film which is mounted in the quarter-cylindrical camera D. This camera is mounted on the right-hand goniometer head and moves sideways when the absorbers are interchanged and rotates through a certain angle during the time that the X-rays are passing through the specimens. This process is achieved by the instrument in the following way. Suppose initially that the left-hand absorber is in the X-ray beam and the motor is in operation. The camera is rotating and the monochromatic part of the radiation is being steadily reflected on to the film. In this way a straight vertical line is obtained on the left-hand side, fig. 7. After the screw-driven carriage has moved from the right to the left and back again the right-hand absorber comes into the X-ray beam and the reflected X-rays trace out the right-hand vertical line of fig. 7. The exposure may last several hours, during which time the absorbers are interchanged many times. After exposure for a sufficient time the film is developed and the

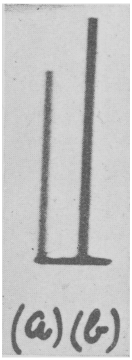

FIG. 7. Two traces produced by the crystal-reflected radiation after passing through absorbing plates of (a) biotite, (b) nickel.

blackening of the streaks is compared either visually or, better, by a micro-photometer. The simplest procedure is to repeat the experiment with different thicknesses of nickel until that thickness is found which has the same absorption as the specimen under test.

There are two important considerations which affect the conditions of the measurement. The first is concerned with the accuracy of measurement of the thickness and the second with the filtering action of the absorber, *if the radiation is not entirely monochromatic.* The first requirement is that the thickness shall be great enough to enable it to be determined with a specified accuracy, say 1 %. A micrometer screw gauge normally measures with an accuracy of 0·0002 in. or 0·0005 cm. To obtain an accuracy of 1% in the measurement of the thickness it is necessary to use a plate 0·05 cm. thick. Such a plate will reduce the intensity to 1/22,000 of its incident value if the absorption coefficient is taken as 200 cm.$^{-1}$ The length of exposure required, with such a reduction of intensity by the absorber, is some 6 hours. This is a long, but not unduly long, exposure. The second requirement for a satisfactory measurement is that the radiation shall be truly monochromatic. This is largely achieved by reflecting the radiation from a crystal but, with calcite at least, there is the possibility that radiation of wave-length 0·77 Å., i.e. half the wave-length of the characteristic $K\alpha$ radiation, will be reflected along with the $K\alpha$ radiation. Under normal operating conditions not only is $\lambda/2$ radiation part of the white radiation of the X-ray tube, but so also are $\lambda/3$, $\lambda/4$, and $\lambda/5$. To be quite certain that no sub-harmonic of the $K\alpha$ radiation is produced by the tube, the applied high tension must be kept below about 16 kV for copper anodes. If a crystal such as fluorspar or diamond is used as a monochromator the $\lambda/2$ radiation cannot be reflected for structural reasons and so the high tension can be raised to 24 kV, which is just not high enough to excite $\lambda/3$ radiation. The importance of the purity of the reflected beam arises from the difference in the absorption coefficients for λ and $\lambda/2$. For elements of atomic weight less than that of nickel the absorption coefficient for $\lambda/2$ radiation is about one-eighth of that for λ radiation. For elements of atomic weights above nickel and in the range within which Cu–$K\alpha$ radiation can be applied, there is little difference in absorption coefficient. When a large reduction in the intensity of the X-rays is introduced, by the necessity of using a specimen the thickness of which can be measured with sufficient precision, the disturbance introduced by the $\lambda/2$ radiation is serious. To indicate how free from $\lambda/2$ radiation the beam must be we shall suppose that in the beam emerging from the absorber the intensity of the $\lambda/2$ component is 1 % of that of the λ. If the reduction factor for the λ radiation is 22,000, then that for the $\lambda/2$ component is approximately 5 for elements of atomic number less than that of nickel, and about 20,000 for nickel and elements of higher atomic number. Thus the $\lambda/2$ component is enhanced about 4,000 times by passage through the absorber for elements of low atomic number and not enhanced appreciably for nickel and elements of higher atomic number. Thus, in the range of atomic numbers within which the method described here is applicable, the intensity of the $\lambda/2$ component must be less than 1/400,000th part of the λ radiation in order to achieve a result accurate to 1 %. There is, of course, no difficulty in doing this by keeping the high tension below the appropriate value.

To test the applicability of this method of finding the absorption coefficient, cleavage flakes of biotite and phlogopite were balanced in turn against nickel. Biotite of thickness 0·012 inch and phlogopite of thickness 0·0165 inch had the same absorption, judged by visual inspection of the films, as 0·006 inch of nickel. The linear absorption, coefficient of nickel for Cu-$K\alpha$ radiation is 438 cm.$^{-1}$, and hence the values for biotite and phlogopite are 220 cm.$^{-1}$ and 160 cm.$^{-1}$ respectively. On referring to fig. 6 it will be seen that the values thus found experimentally correspond with those derived by calculation from the chemical composition. However, the accuracy of the measurement of thickness in this experiment was low and the chemical analysis of the specimen was not available, so that these tests can only be regarded as illustrating the method.

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Summary.

The main features of a two-crystal Weissenberg goniometer of the Wooster-Martin type are briefly described. The applications to two types of mineralogical problem are considered. (*a*) The determination of the intensities of reflection of a series of orders of a given plane or of the reflections from all planes having a common zone axis. Special attention is given to the measurement of absolute as well as relative intensities of reflection. (*b*) The determination of the X-ray absorption coefficients for certain iron-containing series of minerals with a view to a physical measurement of the percentage composition of iron in the mineral.

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