

An Immersion Method for Exact Determinations of Refractive Indices

The Glass Method

by

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Abstract

The glass method is an improvement of the ordinary immersion method involving the use of a monochromator (single variation). The refractive index of the immersion liquid is determined by simultaneous measurement of the refractive indices of grains of optical glass and of each mineral grain. Under favourable conditions the uncertainty will then be ± 0.0002 on the refractive index of the mineral grain. Furthermore the temperature of the microscope stage can be determined by means of the glass method, and the decomposition of the immersion liquid can be examined rather accurately (± 0.0001 on n_D).

1. Introduction.

During the past two years a number of optical investigations, especially mineral determinations, have been carried out in the Mineralogical Institute of the University of Copenhagen. The mineral determinations, the primary aim of the work, have been carried out in accordance with the ordinary immersion method involving the use of a monochromator (PAULY 1948, WHERRY 1918, and POSNJAK and MERWIN 1922). The following well-known mixtures of immersion liquids have been used: paraffin-oil and monobrome-naphthalene from $n = 1.48$ to 1.66 and monobrome-naphthalene and metyleniodide from 1.66 to 1.74 .

During the work we ascertained that the immersion method employed was rather defective on account of errors on the n_D -value of the immersion liquid and in the instruments. These errors turned out to be due to changes of temperature and composition of the liquid and due to errors in the correction of the monochromator. In order to eliminate all these errors the glass method was devised.

2. The principle of the Glass Method.

The purpose of the glass method is the simultaneous determination of the refractive indices—R.I.'s—of a mineral and of the liquid, in which it is immersed. This can be done by measuring the R.I. of a solid with known optical properties immediately before and after measuring the mineral grain in the same mount, the solid being placed close to the mineral grain.

For the solids with known optical properties a series, mainly consisting of minerals (Cargille Laboratories Inc., New York, U.S.A.), and a series

of different types of optical glass (Wagner & Müntz, München, Germany) were formerly used, but none of these series was supplied with the sufficient optical data. Measurements, therefore, based on these reference series cannot be very accurate, probably not better than ± 0.01 , which is poor as compared with the ordinary immersion method, which gives about ± 0.003 . (Standard deviation).

For the solids with known optical properties we have chosen optical glasses. Together with the glass, all desired information can be obtained from the manufactures with the necessary accuracy: n_D , dispersions between several spectral lines, and temperature coefficients.

By adjusting the monochromator one can find the wave-length at which the glass and the liquid have the same R. I.'s—i.e. the R.I. of the liquid can be determined at the moment of measuring. In the greater part of the visible spectrum this determination can be done with an accuracy of ± 0.0001 , which of course requires that the dispersion curves of the immersion liquids have been determined with sufficient accuracy.

3. Optical Glass.

In our laboratory we have so far used optical glass from "Chance Glass" Ltd., Smethwick, Birmingham, England.

Table 1 gives an example of the information supplied with each glass sample.

Table 1

Glass type: SBC 34889/s (SBC = special barium crown)
Laboratory reference: VB 81/44

$$n_d = 1.6910. \quad n_F - n_C = 0.01260.$$

$$\text{Reciprocal dispersive power: } v = 54.8 \left(v = \frac{n_d - 1}{n_F - n_C} \right)$$

Density = 4.11 g/ccm. Resistance to air = S 3.

Partial dispersions:

<i>b</i> - <i>C</i>	<i>C</i> - <i>d</i>	<i>d</i> - <i>e</i>	<i>e</i> - <i>F</i>	<i>F</i> - <i>g</i>	<i>g</i> - <i>h</i>
0.00218	0.00383	0.00300	0.00577	0.00688	0.00570

The letters correspond to the following spectral lines:

$$\begin{aligned} b &= 7065.2 \text{ \AA} - \text{He.} & e &= 5460.7 \text{ \AA} - \text{Hg.} & g &= 4358.3 \text{ \AA} - \text{Hg.} \\ C &= 6562.8 \text{ \AA} - \text{H.} & F &= 4861.3 \text{ \AA} - \text{H.} & h &= 4046.8 \text{ \AA} - \text{Hg.} \\ d &= 5875.6 \text{ \AA} - \text{He.} & & & & \end{aligned}$$

The n_d value is stated to be correct to within ± 0.0001 , and the uncertainty of the partial dispersions is app. ± 0.00003 .

"Chance Glass" Ltd. delivered the glass in pieces of about $1 \times 2 \times 2$ cm, one of the surfaces of 1×2 cm having been polished in such a way that the R. I. can be controlled with an accuracy of ± 0.0002 , i.e., the accuracy of our refractometer. For a purchase of at least five samples the price of each was £ 1.0.0. Refraction and dispersion data were given and on special request, information on some temperature coefficients as well.

For each type of glass supplied, "Chance Glass" Ltd. indicates one of six degrees of resistance of the glass surface to atmospheric air. These are classified "S 1" to "S 6", of which "S 6" is the least resistant. For more than one year we have kept a small amount of each of these glass samples as a powder in ordinary glass tubes

with cotton stoppers in our thermostatically regulated laboratory (20°.5 C), where the air is very humid, and so far there has been no tarnish of the glass. Neither have we observed any sign of tarnish of the glass in the immersion liquids, and even after heating for 80 minutes to about 110° C in "Clearax", there has been no effect on the "S 6" surface. Consequently, the glass seems to be sufficiently resistant to laboratory conditions. The reservation must be made, however, that some immersion liquids, for instance those containing arsenic tribromide, attack glass very strongly and would therefore attack our glass powders.

The catalogues of the glass-works mention both flint and crown glass, i.e., glass with high and low dispersion respectively. For the glass method the crown glass is the best, as it covers a greater range of R. I. since its less steep dispersion curve cuts a greater number of the dispersion curves of the liquids than does that of the flint glass.

At the moment we have glass with n_d from 1.65 to 1.75. We have received an offer for several more samples of glass with n_d values down to 1.48, but we have hesitated to purchase them, as we first wanted to see the results of the glass method. Furthermore the very small dispersion of our low index liquids renders the glass method almost inapplicable below $n = 1.55$. The reason is that it is difficult to match both mineral and glass with the same liquid.

For the time being it is difficult to get glass with an R. I. higher than 1.75. "Chance Glass" Ltd. has offered delivery of a series manufactured especially for us, but the price is very high. If the demand for such a series was greater, a more favourable agreement with the glass-works could certainly be made. Professor FRANS WICKMAN in Stockholm has suggested that the author should collect orders from laboratories interested in the method so that an agreement concerning a suitable series of glasses at a favourable price could be made. The author would of course be glad to make the necessary arrangements and looks forward to hearing from anyone interested in this matter.

4. The Procedure.

At first a rough determination of the refractive index—the R. I.—of the mineral is made on the basis of the ordinary immersion method—without glass—in order to select the type of glass and liquid to be used.

Then a powder mount of the mineral, the liquid, and the glass is made; if for example the n_y of the mineral lies between 1.68 and 1.70, liquid 1.6864 and glass 1.6910 can be employed.

The mount is placed on the microscope stage and when thermal equilibrium is established, the measurements can be carried out.

The wave-length at which the R. I.'s of glass and liquid are equal is determined by means of a corrected monochromator. This can be done with an accuracy of $\pm 10 \text{ \AA}$ throughout the greater part of the spectrum, corresponding to an accuracy of about ± 0.0001 of the R. I. of the liquid. During the calculations it is practical to use frequency instead of wave-length.

Similarly the frequency at which the R. I. of the liquid coincides with that of the mineral—e.g. n_y —is determined.

Finally a measurement of the glass grain is made to check that the R. I. of the liquid has not changed.

For the calculations we use the following terms: (refer Fig. 1)

f_G the frequency at which $n_{\text{glass}} = n_{\text{liquid}}$.

n_G the R. I. of the glass for the frequency of f_G . (I.e. n_G is equal to the R. I. of the liquid at f_G).

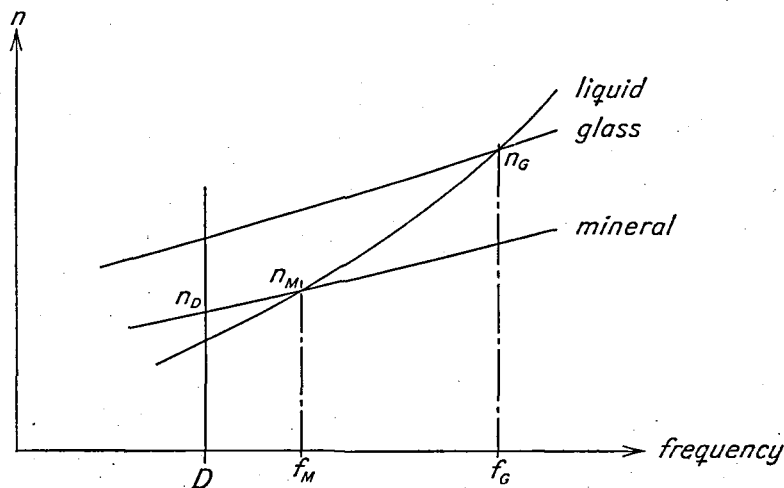


Fig. 1. The principle of the glass method. (See text page 180).

f_M the frequency at which $n_{\text{mineral}} = n_{\text{liquid}}$.

n_M the R. I. of the mineral at the frequency of f_M .

n_D the R. I. of the mineral at the frequency of the D -line of sodium light.

The calculations of n_M and n_D can be carried out in the following way (refer Fig. 1).

1) Point (f_G, n_G) on the dispersion curve of the glass fixes the dispersion curve of the liquid, as the latter necessarily passes through this point.

2) The dispersion curve of the liquid going through (f_G, n_G) is drawn by means of interpolation or parallel displacement.

3) At the frequency f_M the n_M -value is read on the dispersion curve of the liquid.

4) The n_D -value of the mineral can be determined by means of the dispersion curve of the mineral if this curve is established beforehand. If not, this dispersion curve should be determined by measuring the R. I. of the mineral at various frequencies. This can be done in two ways: By using other liquids, the R. I.'s of which are close to that of the first; or by changing the temperature of the microscope stage (the double variation method). The second method, which is the more accurate, is discussed in Chapter 10.

5) So far we have only measured one grain. In order to avoid errors and to account for variations in the R. I. of the mineral, several grains should be measured (refer Chapter 7).

5. The Monochromator Conversion Scale.

It may be seen in Chapter 6 that a monochromator often has an error which must be corrected. Furthermore it is practical to make use of frequency instead of wave-length, since the dispersion curves will then

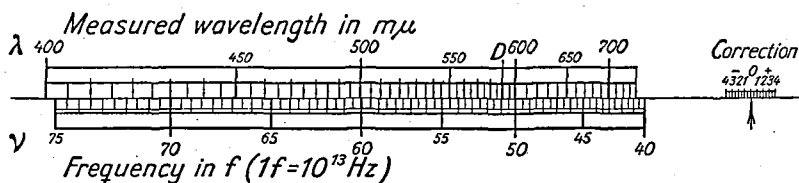


Fig. 2. A monochromator conversion scale made for the conversion of wave-length into frequency and for the correction of the error of the monochromator. This monochromator conversion scale is only valid for the particular Leitz monochromator used in our laboratory (see text).

be less curved. In order to facilitate these conversions a monochromator conversion scale could be constructed in the following way:

1) It should be noted that the monochromator handle has two scales: an equidistant angle scale and a wave-length scale. (Leitz: Grosser Monochromator in besonderer lichtstarker Ausführung).

2) The equidistant angle scale is plotted as an equidistant linear scale.

3) The angle values corresponding to the different wave-lengths are read on the monochromator, and a wave-length scale is drawn by plotting the read wave-lengths on the angle scale. This wave-length scale is not equidistant, and can be seen in the upper part of Figure 2.

4) A frequency scale corresponding to the above wave-length scale is now constructed. The frequency ν hz = $\frac{3 \times 10^{17} \text{ m}\mu/\text{sec}}{\lambda \text{ m}\mu}$, where λ is the wave-length. This scale is seen in the lower part of Figure 2. A small part of the angle scale is used for correction purpose and is shown in the extreme right of Fig. 2.

The error of our monochromator angle scale is not constant but varies according to the wave-length. The variation has been eliminated by means of minor corrections when drawing the wave-length scale. This is the explanation of the apparent discrepancy between the two scales of Fig. 2.

The monochromator conversion scale has the following properties:

1) When the frequency scale is displaced by an amount dependant upon the angular error of the D-line, the true frequency corresponding to any particular wave-length (which is read from the monochromator) can be read off directly from the conversion scale.

2) Since a) the dispersion curves obtained, when the R. I. is plotted against the rotation angle of the monochromator handle, are practically linear, and b) the rotation angle provides the basis for drawing the two parts of the conversion scale, it follows that a given linear distance on the scale corresponds not only to the same angle of rotation throughout the scale, but also to the same change of R. I. throughout the spectrum.

As, furthermore, the dispersion curves of glass and mineral are nearly parallel, a certain change of the R. I. of the liquid will cause nearly the same linear displacement on the conversion scale of the wave-lengths at which the R. I.'s of glass and mineral coincide with that of the liquid. Hence

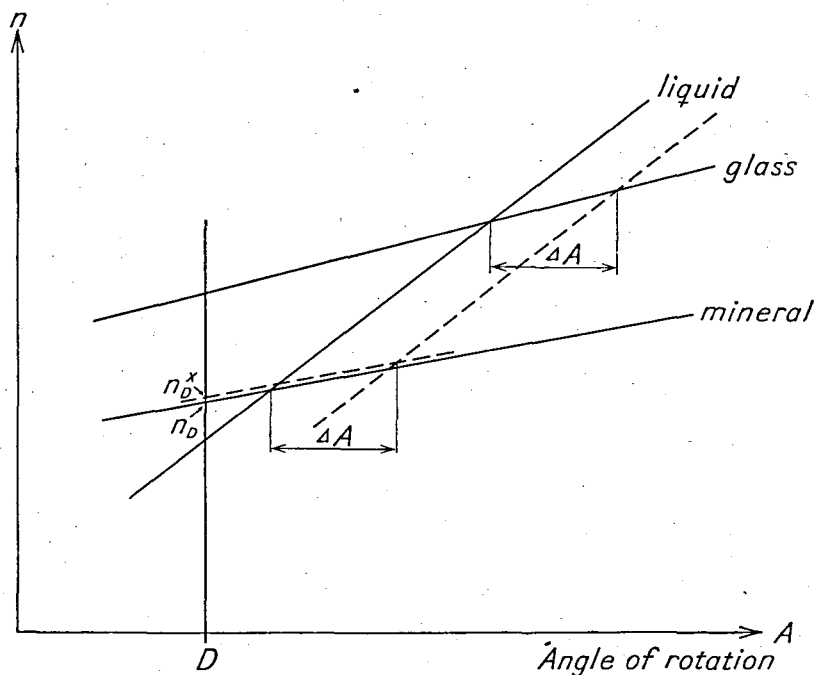


Fig. 3. The full curves correspond to an n_D -determination with a correct monochromator.

The broken curves and the resulting n_D^x correspond to the case in which the error of the monochromator is ΔA .

$n_D^x - n_D$ is very small because the dispersion curves of glass and mineral are nearly parallel.

it follows that the difference between the readings for glass and mineral on the monochromator angle scale (= the difference of the frequencies for glass and mineral on the conversion scale) is nearly constant (cf. Fig. 3), and therefore one pair of measurements of glass and mineral can be displaced on the conversion scale by moving both frequencies the same distance to the same side. This is very useful when several mineral grains are to be measured since, if all the glass measurements are moved to the same figure—e.g. to their mean value—and the mineral measurements are moved correspondingly, we obtain directly comparable results for the minerals. This will be illustrated in Chapter 7.

6. The Accuracy of the Glass Method.

This chapter will briefly discuss some errors and uncertainties common to all immersion methods as well as those particular to the glass method.

a. *The Monochromator and the Colour of Light.* It is necessary to apply some correction to the monochromator readings; the amount may vary in time and can also depend on the wave-length. Consequently, the

necessary corrections required for different spectral lines should be determined occasionally, and allowance should be made for this when constructing and using the monochromator conversion scale. (Refer Chapter 5).

The glass method, however, almost eliminates refraction errors due to wrong "correction" of the monochromator. Failure to correct exactly will influence the measurements of both glass and mineral to the same extent (as the dispersion curves of glass and mineral are nearly parallel) and is thus of almost no significance. This is shown on Fig. 3, where the ordinate— n —is the refractive index, and the abscissa— A —is the angle of rotation of the monochromator handle. This abscissa has been chosen in order to make the dispersion curves straight lines and with the purpose that the error due to wrong "correction" should be the same linear distance— ΔA —throughout the spectrum.

It should be remembered that the light coming from the monochromator consists of a certain range of wave-lengths, for which reason the slits of the monochromator should be as narrow as possible.

The colour of the light has a great influence on the accuracy of the measurements, because the sensibility of the eye is small near the limits of the visible spectrum. Measurements in the interval 5000–6000 Å (green and yellow) are the best. Wave-lengths from 4700–6400 Å (blue and orange) are still fairly good. The more the above limits are exceeded, the more uncertain and tiring the measurements will be. As it is generally easier to measure a grain of glass than a mineral grain, a greater part of the spectrum can be used for glass measurements than for mineral measurements.

b. *The Immersion Liquids.* Generally the n_D -values of the immersion liquids are determined with a refractometer at room temperature with an accuracy of ± 0.0002 . For the glass method an accuracy of ± 0.005 is sufficient.

During the work the temperature of the mount increases slowly. We have measured a rise in temperature of 3–4° C by means of glass grains and a stable liquid. (Refer Chap. 8). Of course the glass method automatically eliminates errors of this kind.

Part of the generally used immersion liquids are unstable when exposed to atmospheric air. The index of such liquids therefore changes in the mount during the work, especially near the border. (For particulars see Chapter 9). Also in this case the glass method eliminates the errors involved.

Errors originating from the dispersion curves can only be avoided by careful work with a good goniometer, by the use of spectral lines, and by temperature control (FAIRBAIRN 1952).

c. *Centering the Light.* If the light entering the microscope from the monochromator is badly centered, this may be a source of error. If a mat glass plate is inserted in the plane of the upper iris diaphragm, and the light is focused on the plate, poor centering cannot cause errors. If the mat

glass plate is not employed—e.g. in order to get more light—it is imperative to center the slit accurately and to place the grain in the middle of the beam. This is due to the fact that even a narrow beam represents a continuous range of wave-lengths.

d. *The Minerals.* Accurate determinations of refractive indices can only be made on minerals which are colourless—or nearly so—well-crystallized, and free of inclusions. Faces giving exactly the desired orientations should be common. If the mineral has but faint or oblique cleavage, the needed accuracy of orientation cannot be attained by interference figures alone, and a universal stage should be employed (EMMONS 1943). This is due to the fact that an accuracy of ± 0.0001 in the n_D -value requires the orientation to be correct within a few degrees.

e. *The Method of Measuring the Refractive Indices.* It is the author's impression that refractive index determinations by means of the Becke line and the Van der Kolk effect give the same results and are equally sensitive. Under favourable conditions both methods can give results with an accuracy of ± 0.0001 .

f. *The Glass.* Sources of error special to the glass method are the optical data for the glasses and the uncertainty of the determination of the wave-length at which glass and liquid have the same R. I.'s.

The accuracy of the optical data of the glass, mentioned in Chapter 3, is sufficient for refractive index determinations with an accuracy of ± 0.0001 . Better results cannot be obtained at present by the glass method.

The uncertainty of an R. I. determination of a glass grain is very small. The glass grains are ideal for measurements by the immersion method as they are pure, as their orientation has no influence, and as nearly all grains are suited for measuring due to their conchoidal fracture. It should be added that the glass grains must lie close to the mineral grains (refer Chapter 9), and that they should be of an adequate size. According to our experience, measurements can be carried out more accurately on large grains than on small ones. As, in general, the glass can be measured more accurately than the mineral, there will normally be no reason for using glass grains larger than the mineral grain.

g. *Conclusions.* The refractive index of pure, colourless, and coarsely crystalline minerals with good cleavage can often be measured with an accuracy of $\pm 0.0002-3$ by means of the glass method, while the accuracy of the very same measurement without the glass is but $\pm 0.002-3$. The time for measurement is approximately the same in both cases. In ideal cases an accuracy of ± 0.0001 can be obtained by the glass method.

7. Examples.

Two samples of hypersthene were used in the measurements to be discussed in this chapter. The optical properties of these samples are for

Table 2
Example 1 = Hypersthene G.G.U. No. 18202.1.

	1	2	3	4	5
Grain No.	Wave-length read for $n_{\text{glass}} = n_{\text{liquid}}$	Wave-length read for $n_z = n_{\text{liquid}}$	Frequency chosen for $n_{\text{glass}} = n_{\text{liquid}}$	Frequency calculated for $n_z = n_{\text{liquid}}$	Wave-length calculated for $n_z = n_{\text{liquid}}$
1	613 m μ	582 m μ	49.0 f	51.6 f	573 m μ
2	609 -	580 -	49.0 -	51.5 -	575 -
3	609 -	579 -	49.0 -	51.6 -	574 -
4	608 -	579 -	49.0 -	51.5 -	575 -
5	607 -	573 -	49.0 -	51.9 -	570 -
6	600 -	571 -	49.0 -	51.6 -	573 -
7	601 -	573 -	49.0 -	51.5 -	575 -
8	601 -	571 -	49.0 -	51.7 -	573 -
9	601 -	576 -	49.0 -	51.2 -	578 -
10	601 -	580 -	49.0 -	50.8 -	582 -
			Mean value: 51.6 \pm 0.1 f*)		
Standard deviation: \pm 4 m μ			\pm 0.2 f*)		\pm 2 m μ *)

*) Grain No. 10 has been omitted in this calculation as it deviates much from the mean.

all practical purposes identical, but the calculations have been made differently in the two cases. The samples were collected during the expeditions of the Geological Survey of Greenland (G. G. U. = Grønlands Geologiske Undersøgelse).

Example 1: Hypersthene from ultrabasic rock, Qugssug, Godthaabsfjord, West Greenland. G.G.U. No. 18202.1.

The first two columns of Table 2 give the uncorrected results, Column 1 giving the observed wave-lengths at which the R. I.'s of glass and liquid coincide, and Column 2 giving those at which n_z -hypersthene coincides with the R. I.-liquid. Ten different hypersthene grains have been measured.

These wave-lengths are now converted into frequency and corrected by means of the monochromator conversion scale in the manner described in Chapter 5. Each pair of results is displaced so that all those for glass have the same frequency; this frequency is usually chosen as a whole number near their mean value—in this case 49.0 f ($1 f = 10^{13}$ Hz). For this reason all figures of Column 3 are identical. The frequency 49.0 f can be regarded as the frequency at which the R. I. of the glass grains equals that of the liquid, presuming that the R. I.-liquid did not change, but remained constant at the mean value.

Column 4 gives the calculated frequencies at which $n_z = n_{\text{liquid}}$, corresponding to the glass frequency of Column 3.

The last column gives the wave-lengths corresponding to the frequencies of the fourth column. The wave-lengths are not used for the calculations and are only listed here in order to show that the standard deviation of the mineral measurements has diminished from \pm 4 m μ in Column 2

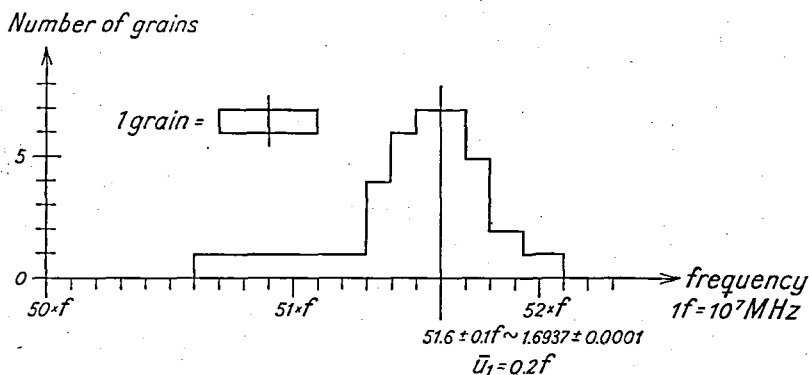


Fig. 4. The histogram shows the distribution of 10 refractive index determinations on hypersthene G.G.U. No. 18202.1. The abscissa gives the corrected frequencies at which the refractive index of each mineral grain is equal to that of the liquid. Each measurement is plotted as a rectangle with unit height and base = $0.4 f$. ($0.4 f = 2 \times 0.2 f$, the standard deviation of one measurement, \bar{u}_1 , being $0.2 f$).

to $\pm 2 \mu$ in Column 5. This is due to the fact that variations in the R. I. of the liquid are eliminated by the use of the glass.

The results in Table 2 can be statistically treated in several ways. For instance, the standard deviation can be calculated as in Table 2. Since in this way the distribution of the results cannot be seen easily, a histogram can be used with advantage (See Fig. 4). In this histogram each n_z -frequency is plotted as a rectangle with unit height and base equal to twice the standard deviation. Measurements made on badly orientated grains and therefore deviating significantly from the other ones can easily be detected on the histogram. It should be borne in mind that such deviations may be due to variations in chemical composition, variations which could perhaps be calculated from Fig. 4. Such calculations, however, should be based on a thorough knowledge of the ideal case—that is without variation in chemical composition of the mineral—and on a wide range of measurements (of at least 50 grains).

In Fig. 4 the distribution is not symmetrical, but has a tail towards low frequencies, i.e., towards low refraction. This tail is characteristic of nearly all the sets of n_z -hypersthene values measured. This is consistent with the fact that all directions, differing from the true z -direction, have lower R. I. than the n_z -value, and the tail is thus most probably due to poor orientation of some of the grains. Consequently, the symmetrical part of the histogram must show the frequencies corresponding to the true n_z -values. Hence it follows that the mean n_z -frequency is to be found at the top of the symmetrical part of the histogram, i.e., slightly to the right of the mean value.

As the n_z -value is the highest R. I. of the mineral, it is usually assumed that the highest R. I. measured will be the "true" n_z -value. Thus from Fig. 4 this n_z -value will be taken as $51.8 f$ corresponding to $n_z = 1.6940$. As a result of this practice a systematical error of $+0.0003$ is introduced.

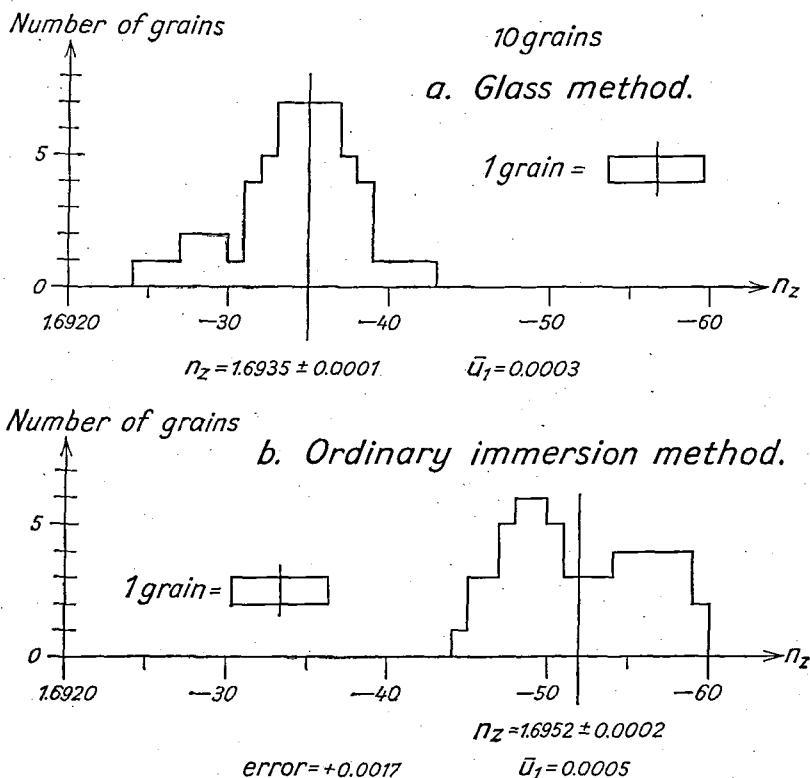


Fig. 5. The distribution of the results of measurements on hypersthene G.G.U. No. 18202.2. The upper part of the figure shows the results corrected for changes in the refractive index of the immersion liquid by means of the glass method. Such a correction has not been made in the lower part of the figure. The same experimental data have been used in both cases.

Example 2: Hypersthene from ultrabasic rock, Qugssuq, Godthaabsfjord, West Greenland. G.G.U. No. 18202.2.

In this case ten mineral grains were measured, and the R. I. has been calculated for each mineral grain individually in two ways: a) according to the glass method, and b) according to the ordinary immersion method. The same measurements have thus been used in both calculations, but in the first case the results have been corrected by means of the glass measurements. In the second case the R. I. of the liquid has been measured with a refractometer at room temperature. The results are given in Fig. 5a and b.

It will be seen from Fig. 5 that the ordinary immersion method gives considerably higher results than the glass method and that these results have a somewhat greater spread. If, as in Fig. 5a, the n_z -value is read from the top of the histogram, and as in Fig. 5b, from the mean value, the difference between the two n_z -determinations will be +0.0017. This

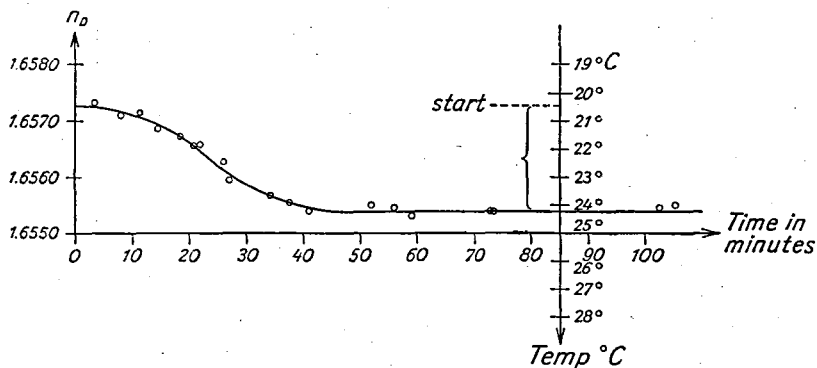


Fig. 6. The rise of temperature and the decrease in the refractive index of monobromenaphthalene as a function of the time during which the operator has worked with the microscope.

difference is caused by a rise in temperature of 3–4° C of the immersion liquid during the measurements (see Chapter 8). An approximate correction can be made for the temperature change, but it will be unsatisfactory since the R. I. of the immersion liquid alters continuously throughout the time of measurement. The greater spread in Fig. 5b is due to both rise in temperature and to change of the composition of the liquid (see Chapters 8 and 9).

If the n_D -value is assumed to the highest R. I. measured, the error will be greater, about +0.0023.

To conclude: The classical immersion method gives too high results and a great spread.

8. The Temperature of the Microscope Stage.

During the work the temperature of the microscope stage and the immersion liquid rises because of heat from the operator and from the microscope lamp. Nevertheless, the R. I. of the immersion liquid at room temperature has hitherto very often been used in the calculations of the R. I.'s of the minerals.

As small variations in temperature have practically no effect on the shape of the dispersion curves, the glass automatically corrects for these changes of temperature.

The temperature of a mount on the microscope stage can easily be determined by using the glass as a standard. For this purpose a mount was made of a stable liquid—monobromenaphthalene—and an adequate glass—1.6607. This was placed on the microscope stage for about one hour in order to reach room temperature. Under these conditions glass and liquid had the same R. I. at the monochromator value $535 \pm 1 \text{ m}\mu$ (without correction). This gives $n_D = 1.6573$ for the liquid corresponding to a room temperature of 20°.5 C. Then a number of glass grains were measured several times in order to determine the R. I. of the liquid as a function of the time during which the microscope was used. Fig. 6 shows that the temperature rises steadily during the first 40 minutes and then

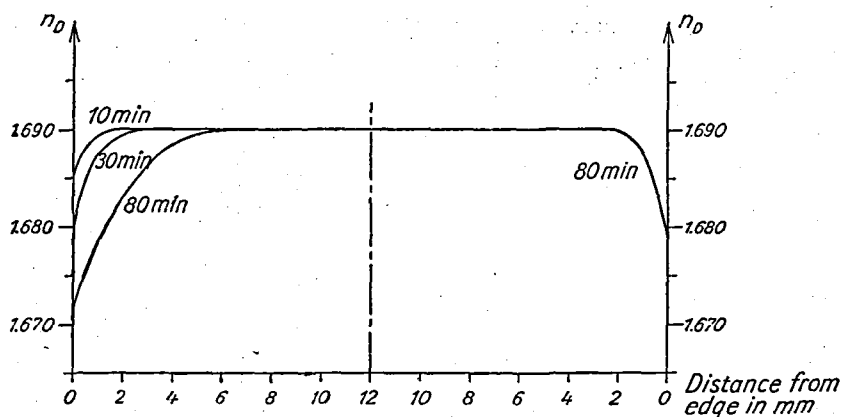


Fig. 7. The refractive index of an unstable liquid at various times measured along a line across the mount (width 24 mm).

remains constant at $24^{\circ}.2$ C, i.e. $3^{\circ}.7$ above room temperature. The $3^{\circ}.7$ correspond to a systematic error of $+0.0015-20$ on all measurements carried out by the ordinary immersion method.

The error can to some extent be diminished by determining the R. I. of the liquids at the final temperature of the mount.

9. Unstable Immersion Liquids.

Most immersion liquids are not pure, stable liquids, but unstable mixtures. The instability is due to oxidation of the components, absorption of water, and evaporation.

These changes need no further comments, but for one exception. If the changes do not add noticeable amounts of new compounds to the mixture, but only remove one component, the result will be a simultaneous change of all properties to the values of an unaltered liquid of a new composition. As an example could be mentioned that the series of monobromo-naphthalene and metyleniodide mixtures are unstable because of decomposition of the metyleniodide. Such a mixture with $n_D = 1.7100$ will therefore after some time have $n_D = 1.7000$, and according to our experience the dispersion curve is then identical with that of a newly mixed liquid with $n_D = 1.7000$. (The metyleniodide series is discussed by BRUUN and BARTH 1947). Small errors due to changes of this kind are automatically eliminated by the glass method.

If the decomposition of the liquid results in the addition of new components, the dispersions may be subject to uncontrollable changes. The liquid will then be unsuitable for accurate measurements, unless two different sorts of glass are measured simultaneously.

By means of the glass the changes of the immersion liquid may easily be controlled. Such a control measurement was carried out in a mount under a cover glass 24×24 mm square, the n_D of the glass being 1.6910, and the n_D of the liquid being 1.692. The liquid was a mixture of monobromo-naphthalene and metyleniodide. In this mount the R. I. of the

liquid was measured several times by means of grains of glass placed at different distances from the edge of the cover glass. Fig. 7 shows the results. The distances from the edge of the cover glass have been measured with an accuracy of ± 0.1 mm.

Fig. 7 shows a strong decrease of the R. I. of the liquid near the edge of the cover glass. The decomposition advances slowly through the mount, but does not reach the central part within 80 minutes. It is seen that the curve corresponding to 80 minutes is unsymmetrical. The decrease of refraction is by far the greatest at the left side, which may be due to an unequal thickness of the mount.

The conclusion is that grains lying near the edge of the mount should never be measured if the liquid is unstable—even if the glass method is employed.

Double Variation Combined with the Glass Method.

The double variation method using the universal stage (EMMONS 1943) is often the fastest and the most accurate of the known immersion methods, because one and the same mineral grain can be used at all wave-lengths for the determination of the dispersion curve of the mineral. But the classical double variation method has three weak points: 1) the determination of temperature is not very accurate 2) an apparatus in fixed arrangement is necessary on account of the temperature correction 3) unstable immersion liquids cannot be used because of the prolonged dispersion measurements.

The glass method solves the three problems when combined with the double variation method:

- 1) The temperature is automatically determined by means of the glass (see Chapter 8).
- 2) Apparatus in fixed arrangement is unnecessary as the glass method makes the temperature correction superfluous.
- 3) The glass method controls the changes of unstable liquids (and thus it allows the prolonged measurements).

We have succeeded in measuring dispersion curves for hypersthene with a reproducibility of ± 0.0001 from $470\text{ m}\mu$ to $630\text{ m}\mu$ without knowing the exact temperature of the heating stage.

Conclusion.

Hitherto the fourth decimal in refractive indices determined by the immersion method has in most cases been unreliable.

The method discussed in this paper is tenfold more accurate than the classical immersion method, and it does not take much more time to carry out.

Unfortunately it is not possible at present to take full advantage of the accuracy of the method due to lack of sufficiently accurate data on the minerals.

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DANSK RESUMÉ

Glasmetoden er en forbedring af den almindelige immersionsmetode (med monokromator) udarbejdet med det formål at få større målenøjagtighed. Dette opnås, idet man bestemmer immersionsvæskens lysbrydningskoefficient ved hjælp af nogle korn af optisk glas, hvis optiske data man kender nøje. Når man umiddelbart derefter måler et mineralkorn i forhold til væsken, kan man under gunstige omstændigheder opnå en nøjagtighed på ± 0.0002 for mineralkornets n_D , medens man med den almindelige immersionsmetode må nøjes med ± 0.002 . Den langt større nøjagtighed er betinget af, at man har præparatets temperatur og lysbrydningsvæskens sønderdeling under kontrol, samtidig med at glasset yderligere bevirker, at monokromatorens justeringsfejl ophæves. Glasmetoden er ikke væsentligt mere tidsrøvende end den almindelige immersionsmetode.

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