

Measurement of refractive indices and their dispersion

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Abstract

For transparent solids, two constants a_0 and a_1 summarize the dispersion curve for a principal refractive index by permitting this principal index to be calculated to within 0.0003 for any desired visible wavelength. To evaluate a_0 and a_1 from a set of data points (λ_i, n_i) —for example, those measured for a principal index by routine immersion methods plus wavelength variation to secure a grain-oil match—fit to the data by least squares the linearized dispersion equation

$$y = a_0 + a_1x$$

where y represents $(n_i^2 - 1)^{-1}$ and x represents λ_i^{-2} . This equation restates the single-term Sellmeier equation

$$n_i^2 = 1 + \frac{A\lambda_i^2}{\lambda_i^2 - \lambda_0^2}$$

in which λ_0 represents an absorption edge in the ultraviolet of strength proportional to A . Thus a_0 equals A^{-1} and a_1 equals $-\lambda_0^2 A^{-1}$ so that $(-a_1/a_0)^{1/2}$ represents λ_0 .

The inherently linear relationship between $(n_i^2 - 1)^{-1}$ and λ_i^{-2} in plots has been noted by Wemple and DiDomenico (1971) for over 100 widely different solids and liquids. The present writers, by simple linear regression, fit the linearized dispersion equation to (λ_i, n_i) data for 222 Cargille oils, to obtain r^2 values above 0.999 for 217 oils and above 0.997 for the remaining five. Similar regressions applied to Wolfe's (1976) measurements of the dispersion of principal indices α , β , and γ for 35 low plagioclases yielded an average r^2 value of 0.99 for the 105 linear regressions.

The efficacy of the curve-fitting technique in suppressing random errors was confirmed by applying the technique to three homogeneous optical glasses. The indices obtained were accurate to within 0.0003 for all visible wavelengths but low by 0.0016 at 1083 nm. For Corning glass 8269, the average of the refractive indices determined by four observers using our technique proved 0.00011 high at 435 nm, true at 546 nm, and 0.00017 low at 687 nm. This systematic error, whereby the true and empirical dispersion curves intersect at about 546 nm, perhaps occurs because a normal eye has peak sensitivity near 555 nm. This causes the wavelength of match λ_m to be overestimated if $\lambda_m > 555$ nm, but underestimated if $\lambda_m < 555$ nm. However, the effect is so slight as to be usually obscured by the random experimental errors.

Applied to a spindle-stage-oriented grain of anorthite from Mijakejima, Japan, the method yielded principal indices in close agreement with those measured by Leisen (1934) using the more technically demanding method of minimum deviation.

Introduction

Experimental (microscopic) techniques for accurately measuring the refractive indices of small grains are subject to both systematic and random errors. Rather than come to grips with them, many researchers in mineralogy simply report refractive indices to "±0.002." Rather surprisingly, first reports of new minerals may be thus marred. However, with proper care, as will be discussed, refractive indices to within 0.0003 are now routinely determinable with relatively unsophisticated and inexpensive equipment. Such increased accuracy should permit the optical parameters of minerals to be related more meaningfully to their crystal chemistry.

The accuracy of immersion methods for determining the refractive indices of transparent solids is limited by (1) the sensitivity of the criterion of match—and by how accurately one can determine: (2) the wavelength of match, (3) the temperature at match and (4) the immersion medium's refractive index for the wavelength and temperature of match. For anisotropic crystals the orientation was formerly a factor but, by using a spindle stage, errors from orientation are so minimized that they may be disregarded. Factors (1) to (4), however, remain a source of both random and systematic errors.

Reducing the effect of random errors

The effect of random errors upon the accuracy of the refractive indices ultimately reported for a crystal can be considerably reduced by matching the crystal's index to those (n_i) of several different immersion oils and for several different wavelengths (λ_i). To the several n_i, λ_i data-pairs that result, an accepted formula for dispersion in solids may be fitted by least-squares techniques. From the intercept and regression coefficients thereby determined for this equation, the crystal's refractive index can then be calculated for any visible wavelength. Moreover, they will not be as subject to random experimental error as were the input data.

Equations for dispersion

In the early stages of this work several equations for dispersion were tested. Among them was the Hartmann equation

$$n_i = A + C/(\lambda_i - B) \quad \text{Eq. 1}$$

where A , B , and C represent the Hartmann constants. The Hartmann equation primarily represents a curve whose flexibility permits it to be closely fitted to the data. This flexibility, however, proves disadvan-

tageous if Equation 1 is fitted to only five or six λ_i, n_i data-pairs. In such case, as noted by Bloss (in press), the resultant least-squares fit involves little smoothing of the data; instead the curve tends to thread through each point.

Except near an absorption band, experimental dispersion curves are well fit by the Sellmeier equation

$$n_i^2 - 1 = \sum_k \frac{A_k \lambda_i^2}{\lambda_i^2 - \lambda_k^2} \quad \text{Eq. 2}$$

where the summation is taken over k absorption bands, occurring at wavelengths λ_k , with strengths proportional to the constants A_k . For clear transparent substances with a strong absorption band in the ultraviolet, say at λ_0 , but none in the visible, the higher-order terms ($k > 1$) can be omitted. The resultant single-term Sellmeier equation

$$n_i^2 - 1 = \frac{A \lambda_i^2}{\lambda_i^2 - \lambda_0^2} \quad \text{Eq. 3}$$

holds well for the visible region. Upon inverting both sides of Equation 3, it becomes

$$y = a_0 + a_1 x \quad \text{Eq. 4}$$

where y represents $(n_i^2 - 1)^{-1}$, a_0 represents A^{-1} , a_1 represents $-\lambda_0^2 A^{-1}$, and x represents λ_i^{-2} . Thus, to a set of refractive-index dispersion data (λ_i, n_i) transformed to the set (x_i, y_i) for a solid or liquid, Equation 4 may be fit by a simple linear regression to determine the values of a_0 and a_1 . The value $(-a_1/a_0)^{1/2}$ then represents λ_0 . If desired, the photon energy E_0 in electron volts associated with λ_0 can be determined since, for any wavelength λ ,

$$E = \frac{hc}{\lambda \cdot 1.6021 \times 10^{-12} \text{ erg/eV}} \quad \text{Eq. 5}$$

where h (Planck's constant) equals 6.626×10^{-27} erg-sec, c (the velocity of light) equals 2.998×10^{10} cm/sec, and 1.6021×10^{-12} represents the number of ergs per electron volt. Thus Equation 5 becomes

$$E \text{ (in eV)} = \frac{12,399.19}{\lambda \text{ (in \AA)}} = \frac{1239.919}{\lambda \text{ (in nm)}} \quad \text{Eq. 6}$$

Although Wemple and DiDomenico (1971) had already observed graphically a linear relationship between $(n_i^2 - 1)^{-1}$ and λ_i^{-2} for over 100 widely different solids and liquids, a preliminary test of Equation 4 was now made using the refractive index values for wavelengths 4358, 4861, 5461, 5893, and 6563 Å kindly supplied by the manufacturer for each of 222 Cargille oils. To the five data-pairs for each oil, transformed to $(n_i^2 - 1)^{-1}$ and λ_i^{-2} , Equation 4 was fit by

least squares. The squared coefficients of linear correlation r^2 exceeded 0.999 for 217 cases and exceeded 0.997 for the remaining five. For the a_0 and a_1 values thus obtained, a_0 shows a regular trend relative to n_D (open circles, Fig. 1). By contrast, a similar plot of a_1 in the spindle stage's oil cell, and, *optionally*, (6) a heating device to change the oil's temperature so as to reduce the number of oil mounts required.

Equipment and supplies

The following equipment and supplies were used: (1) a polarizing microscope whose stage positions could be read to 0.1° by means of a vernier scale; (2) mounted on it, a detent spindle stage or occasionally a Supper spindle stage; (3) mounted substage over a tungsten light, a variable wavelength interference filter, or upon occasion a series of narrow-band-pass

interference filters with peak wavelengths at 5 nm intervals; (4) a standard set of certified Cargille immersion oils at 0.002 intervals; (5) a thermometer or thermocouple to monitor the temperature of the oil in the spindle stage's oil cell, and, *optionally*, (6) a heating device to change the oil's temperature so as to reduce the number of oil mounts required.

Method

Crystal orientation

Anisotropic crystals were oriented by means of the spindle-stage technique and computer program described by Bloss and Riess (1973). This eliminated any appreciable systematic error from misorientation during measurement of a principal refractive index, particularly for crystals in which $(\gamma - \alpha)$ is less than 0.070. For example, a 3° mis-setting of X toward Z or of Z toward X , which would be a relatively large

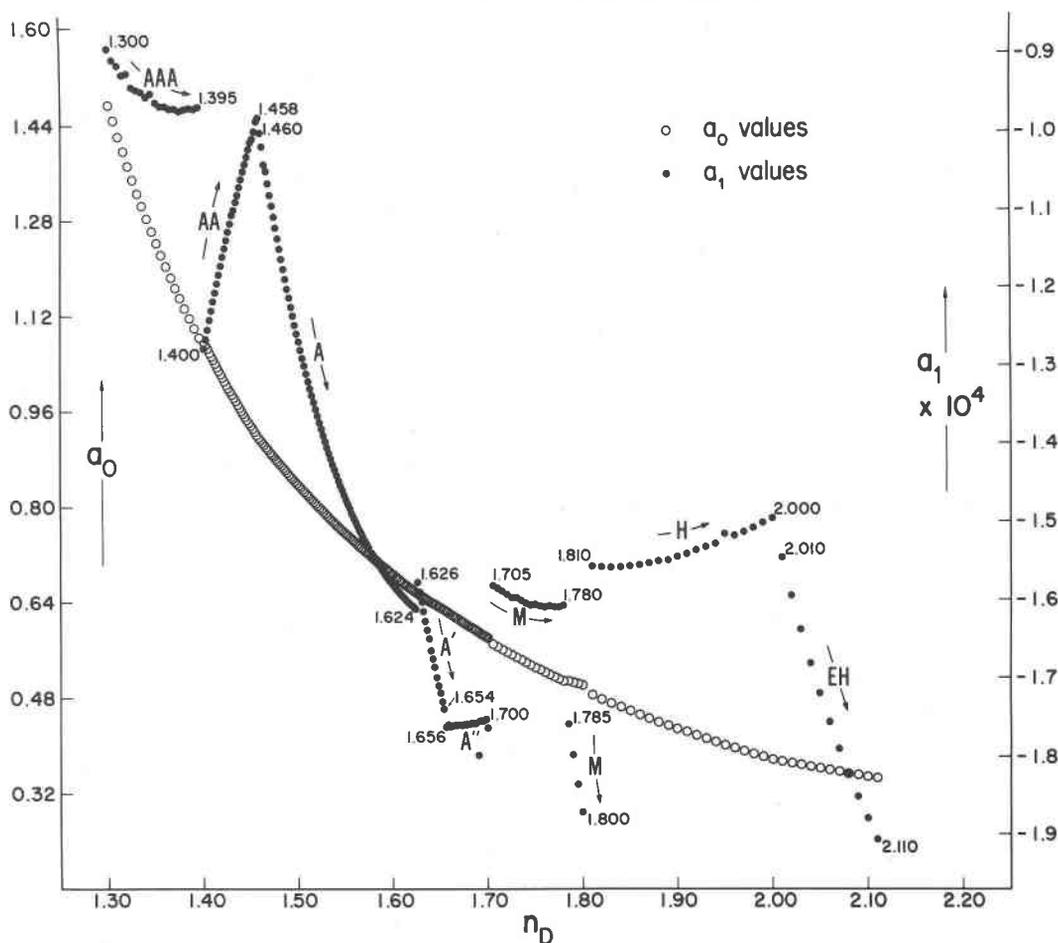


Fig. 1. The constants a_0 and a_1 have been plotted against n_D for each of 222 Cargille oils. The a_0 values are indicated by the open circles and left-hand scale. The a_1 values, plotted as black dots, are indicated on the right-hand scale provided these values are each multiplied by 10^4 (wavelengths in nanometers).

error by the Bloss-Riess technique, will introduce an error in the measured index of 0.0002 or less, if $(\gamma-\alpha)$ equals 0.070, and of about 0.0001, if $(\gamma-\alpha)$ equals 0.040. To the extent the mis-settings were not of X directly toward Z or of Z directly toward X , these errors would be even less than the values cited.

Control of wavelength of match

With the crystal correctly oriented for measurement of a principal index, a Schott wedge-interference filter (Leitz serial: 72035.14) was inserted until it isolated a wavelength band for which the refractive indices of grain and oil matched—this being the *dispersion method* introduced by Merwin and Larsen (1912). The resultant reading (in centimeters) on the calibrated scale engraved on the filter's housing, this value being called the *messort* number (symbol: m), was then converted to peak wavelength in nanometers ($= \lambda$). To do so, the data provided by the manufacturer for filter 72035.14 (Table 1, first two columns) were fit by least squares, using Program BMD05R of the Biomedical Computer Programs (Dixon, 1973), to a series of polynomials up to the fifth degree

$$\lambda = a + b_1m + b_2m^2 + b_3m^3 + b_4m^4 + b_5m^5$$

For filter 72035.14, as for five of our six other Schott-Leitz filters, a second degree polynomial proved superior to those of higher degree. For filter 72035.14, therefore, the equation

$$\lambda = 337.01 + 19.794 m + 0.197 m^2 \quad \text{Eq. 7}$$

permitted messort numbers m in centimeters to be converted to λ in nanometers with satisfactory accuracy (Table 1, col. 3).

Calculating the index of match

By inserting λ_i , the wavelength of match, as well as the previously determined constants a_0 and a_1 for the oil into Equation 4, the index of match n_i was calculated. If the oil's temperature T differed from 25°C during the match, the standard correction $(T - 25^\circ) dn/dT$ was applied to n_i to correct it to its value at temperature T . A thermocouple inserted into the oil cell (and near the grain) monitored the temperature of the oil.

In the double-variation method, the investigator varies the temperature of the oil and, for each new temperature, determines a new index and wavelength of match (n_i and λ_i) between oil and crystal. Usually the crystal's refractive indices are assumed to remain unchanged from one temperature to another. How-

Table 1. Calibration values supplied by the manufacture (cols. 1 and 2) for Schott-Leitz interference filter 72035.14 compared to peak wavelengths (col. 3) calculated from Equation 7.

Messort in cm	λ values in nm From manufacturer	Calculated*
(1)	(2)	(3)
3.1	400	400.26
5.38	450	449.20
7.67	500	500.41
9.83	550	550.60
11.88	600	599.90
13.86	650	649.16
15.86	700	700.45

*Calculated from Equation 7. These should be rounded off to nearest digit.

ever, if accuracy to the fourth decimal place is desired, this may not be true. For example, the γ_D index of anglesite (Table 2) would decrease by 0.0004 if the oil's temperature were increased from 25° to 35°C. The double-variation method, applied to anglesite, would thus report indices for 25°C likely to be systematically low to the extent the oils were heated above 25°C.

Recognizing a match

The variable interference filter was inserted until the last vestiges of the Becke line could be detected and then further inserted until they were detectable again. The average of these two messort numbers was then used to compute, using Equation 7, the wavelength of match. If the grain's shape prevented use of the Becke line test, a modified method of oblique illumination was used by partly inserting an opaque stop immediately below the microscope objective. Use of this stop in conjunction with the substage exit slit of the Leitz-Schott filter in effect approximated the oblique-illumination method suggested by Wright (1913): The method was improved by drilling a 1.5 to 3.0 mm diameter hole in the opaque stop and observing the border shadows on the grain through this hole. By this means, the shadow from the hole's edge could be conveniently caused to enter the field of view

Table 2. Average change in refractive index produced by a 10°C change in temperature within the 20–100°C range for four sulfates (from Kolb, 1911).

	Anhydrite CaSO ₄	Celestite SrSO ₄	Barite BaSO ₄	Anglesite PbSO ₄
α_D	0.00002	0.00017	0.00015	0.00028
β_D	0.00003	0.00013	0.00012	0.00023
γ_D	0.00007	0.00011	0.00020	0.00037

Table 3. Refractive indices determined for Corning glass 8269 by minimum deviation, and by four students using the present technique.

λ (nm)	Minimum Dev'n	Student Values				Average**	Error*** $\times 10^5$
		Janet	Art	George	Rob		
435	1.56302	1.56317	1.56323	1.56323	1.56290	1.56313(13)	+11
486	1.55627	1.55646	1.55635	1.55624	1.55631	1.55634(9)	+ 7
546	1.55076	1.55091	1.55070	1.55064	1.55077	1.55076(12)	0
587	1.54793	1.54804	1.54779	1.54781	1.54786	1.54788(11)	- 5
589	1.54783	1.54794	1.54768	1.54770	1.54775	1.54777(12)	- 6
632	1.54546	1.54552	1.54524	1.54536	1.54527	1.54535(13)	-11
687	1.54310	1.54310	1.54280	1.54302	1.54279	1.54293(16)	-17

*Although indices are cited to five decimal places, such accuracy is only claimed for the minimum deviation results. These were obtained using a specially designed spectrometer by Drs. C. J. Parker and Al Werner of Corning (personal communication).

**Average of the four student values plus its estimated standard deviation (in parentheses) given in terms of its least significant digits. Thus 1.56313(13) indicates an *esd* of 0.00013.

***Error represents the average student value minus the minimum deviation value.

from any desired direction until the direction was achieved that caused best development of border shadows on the grain. After these shadows were thus optimized, the wedge filter was inserted and two messort readings were taken, one just before and the other just after the shadows reversed. Their average value m , inserted into Equation 7, determined the wavelength of match λ_i .

Results

To assess its accuracy, our method was applied to three optical glasses of very high homogeneity, whose refractive indices had already been measured with great accuracy by the minimum-deviation method. The indices for Corning Glass 8269, as obtained by four students using our method and the Becke line disappearance as a test for match, agree closely with those obtained by minimum deviation (Table 3). Two entries show differences around 0.0003, but the average difference is 0.00012 with an *esd* of 0.00008. However, the column labelled "Error" in Table 3 indicates that a small systematic error is inherent to the method. At 435 nm the average index is reported too high by 0.00011; at 687 nm it is low by 0.00017. At about 546 nm the values are true. This systematic error may result because the human eye's sensitivity reaches a maximum at about 555 nm but decreases toward the shorter or longer wavelengths.¹ Consequently, the average of the two messort readings for

¹ However, to the extent brightness is reduced below normal, the eye's maximum sensitivity will shift from 555 to 515 nm and the long-wave cut-off decreases to 650 nm. This phenomenon, the *Purkinje effect*, means that, for low levels of brightness, sensitivity to red is reduced and that to blue is increased.

which the Becke line first disappears (m_1) and later reappears (m_2) will tend to be skewed toward longer wavelengths to the extent $\lambda > 555$ nm or toward shorter wavelengths to the extent $\lambda < 555$ nm. Reporting the wavelength of match as too long will cause the index of match to be reported too low. Reporting the wavelength of match too short will cause the index of match to be reported too high. One of us (S.J.L.) redetermined the refractive indices of Glass 8269, using a series of narrow-band interference filters spaced at 5 nm intervals across the visible spectrum. These results confirmed the slight systematic error illustrated in Table 3 and eliminated the possibility that the systematic error might stem from the Leitz-Schott filter.

Table 4A compares our results with those from minimum deviation for Corning Glass 8262. The dispersion equation calculated from our empirical measurements served to calculate refractive indices that were remarkably close to the indices measured by minimum deviation. The single exception is the index for 1083 nm, which is calculated too high by 0.0016. On the other hand, even this value seems close when one considers that the dispersion equation, based on index measurements made in the 400–700 nm range, was extrapolated 383 nm beyond 700 nm in calculating the index 1.51292. The "Error" column in Table 4A shows an irregular trend which is perhaps opposed to that in Table 3. The data are insufficient to establish this trend as valid.

A grain of Corning glass 8263 was also studied by our method, but this time using an interference microscope (Louisnathan *et al.*, 1976). Passage of the interference fringes, unshifted from oil to grain, was

Table 4a. Comparison of the refractive indices for Corning glass 8262 as measured by Parker and Werner (personal communication) using minimum deviation with those calculated using the a_0 and a_1 values obtained when Equation 4 was fit to various match data (λ_i , n_i pairs) here secured using the Becke line test as the criterion of match.

λ (nm)	Minimum Dev'n	This Work*	Error $\times 10^5$
435.8	1.53297	1.53290	- 7
480.0	1.52870	1.52858	- 12
546.1	1.52419	1.52405	- 14
587.6	1.52210	1.52202	- 8
643.8	1.51986	1.51985	- 1
667.8	1.51906	1.51910	+ 4
706.5	1.51791	1.51792	+ 1
365.0	1.54370	1.54368	- 2
1083.0	1.51132	1.51292	+160

*Calculated from: $(n^2-1)^{-1} = 0.782607 - 7929 \lambda^{-2}$
where λ is in nm.

taken as the criterion of match. The results (Table 4B) represent an accuracy (mean difference 0.00005) comparable to that (Table 4A) for the Becke line method (mean difference 0.00007). The interference-fringe method appears to eliminate any vestige of the wavelength-dependent systematic error evident in Table 3. This further supports the hypothesis that wavelengths of match λ_m , as determined by the normal human eye, will be overestimated if $\lambda_m > 555$ nm but underestimated if $\lambda_m < 555$ nm.

By permitting a highly accurate orientation of a principal vibration direction, a spindle stage permits the principal indices of biaxial crystals to be measured almost as easily as the indices of isotropic materials. Thus, the results obtained by the writers for Mijakejima anorthite (Table 5)—as based on 28 matches for α at various wavelengths, 34 for β , and 35 for γ —compare well with those measured by Leisen (1934) from minimum-deviation measurements on cut prisms. Discrepancies between our results and Leisen's may be attributed to (1) experimental errors by the writers or by Leisen, and/or (2) true differences between the anorthite from Mijakejima studied by Leisen and that studied here. For wavelength 687 nm, the value given by Leisen for β and γ appears to be in error. Thus the value for $2V_x$ at 687 nm, if calculated from Leisen's values, is 106.1° . This value for $2V_x$ seems markedly at variance with those determined from extinction measurements using the Bloss-Riess program as well as with the values for $2V$ calculated from Leisen's refractive index measurements at wavelengths 486 and 589.3 nm. Moreover, if

Table 4b. Comparison of the refractive indices for Corning glass 8263 determined by Parker and Werner (personal communication) using minimum deviation with those calculated using the a_0 and a_1 values obtained when Equation 4 was fit to various match data (λ_i , n_i pairs) where nonshift of interference fringes across the grain-oil boundary served as the criterion of match.

λ (nm)	Minimum Dev'n	This Work*	Error $\times 10^5$
435.8	1.52117	1.52129	+ 12
480.0	1.51707	1.51708	+ 1
546.1	1.51272	1.51266	- 6
587.6	1.51071	1.51065	- 6
643.8	1.50854	1.50853	- 1
667.8	1.50777	1.50779	+ 2
706.5	1.50666	1.50675	+ 9
365.0	1.53148	1.53178	+ 30
1083.0	1.50023	1.50182	+159

*Calculated from: $(n^2-1)^{-1} = 0.803404 - 8081 \lambda^{-2}$
where λ is in nm.

Leisen's values for γ at 486, 589.3, and 687 nm are plotted as $(\gamma^2 - 1)^{-1}$ versus λ^{-2} , the points are markedly non-collinear.

Wolfe (1976) measured the principal indices α , β , and γ at various wavelengths for 35 low plagioclases whose compositions ranged between albite and anorthite. Equation 4 fit his data with linear correlations (r^2) that averaged 0.99 for the 105 regressions performed. On the basis of the values of a_0 and a_1 determined by each regression, an absorption edge λ_0 appears to be located at around 100 nm ($E = 12.4$ eV) for each principal index and composition. For the Mijakejima anorthite, for example, on the basis of only two sets of measurements by one observer and one set by another, λ_0 in nanometers and its *esd* (in parentheses) was determined as 106.3 (3.4) for α , 104.8 (3.9) for β , and 101.5 (3.1) for γ .

Table 5. Comparison of the principal refractive indices measured for the Mijakejima anorthite in this work with those measured by Leisen (1934) using minimum deviation.

λ	486 nm		589.3 nm		687 nm	
	This	Leisen	This	Leisen	This	Leisen
α	1.5811	1.5808	1.5738	1.5741	1.5698	1.5700
β	1.5886	1.5886	1.5818	1.5821	1.5781	1.5777
γ	1.5936	1.5940	1.5871	1.5874	1.5835	1.5821*
$2V_{\text{calc}}$	101.9°	100.8°	102.1°	102.1°	102.6°	106.1°
$2V$ from extinction:**						
$2V_{550}$		101.4°		$2V_{589} = 102.3^\circ$		$2V_{660} = 102.7^\circ$

*Probably in error

**Determined with the method of Bloss and Riess (1973) as modified by Roberts (1975)

Conclusions

(1) For colorless materials with an absorption edge in the far ultraviolet, a linear relationship (Eq. 4) between $(n_i^2 - 1)^{-1}$ and λ^{-2} within the visible region is confirmed for 222 Cargille oils, the three principal indices of 35 low plagioclases, and three optical glasses.

(2) Optical parameters of increased accuracy and significance can now be routinely determined for minerals by fitting Equation 4 to the refractive-index dispersion data obtained for the mineral using the dispersion method. The resultant values for a_0 and a_1 should permit the refractive indices of the material to be calculated to 0.0003 for any desired visible wavelength. Also, for each principal index, the values of a_0 and a_1 permit calculation of λ_0 , the corresponding absorption edge in the ultraviolet.

(3) The Schott-Leitz wedge-interference filter can serve as a simple but adequate monochromator.

(4) The Cargille oils, as presently constituted, are sufficiently accurate and stable to permit determination of refractive indices to within 0.0003. Evidently the errors in their indices n_F , n_D , n_C etc. tend to be random so that, by the technique described, if several different oils are used, these errors tend to cancel each other.

(5) The spindle stage can orient an anisotropic crystal with such accuracy that any residual misorientation has a negligible effect on the fourth decimal place of the principal index involved.

(6) The human eye's peak sensitivity at 555 nm may cause the true and empirical dispersion curves to differ slightly in slope and to intersect at a wavelength near 555 nm.

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