A spindle stage study of the optical properties of a topaz

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Abstract. -2V and refractive indices have been determined for fragments of a topaz crystal, using the spindle stage techniques of Bloss. Dispersion of these optical properties has also been measured for wave lengths from 400 to 900 nm.

 $2V_z$ at 589 nm is 65.7° ± 0.05°, and the optic axes are dispersed 2.9° (v < r). Refractive indices at 589 nm are $N_x = 1.610$ 9, $N_y = 1.613$ 7, $N_z = 1.620$ 9 all with error limits of about 0.000 3. Dispersion measurements suggest an absorption edge at about 100 nm. Both single and double variation methods were used to determine dispersion curves for refractive indices. The two methods yielded comparable results under the same conditions of measurement, but it is concluded that the double variation method can be significantly more precise and accurate than these results, whereas the single variation method cannot.

The composition of the topaz was determined directly by electron microprobe analysis and calculated from the optical parameters and unit cell dimensions and volume, using the Ribbe-Rosenberg equations. The results are internally consistent and fit previously determined optical versus compositional relations. The fluorine content determined by microprobe is 19.3 ($\sigma = 0.3$) wt. % and by optical and X-ray means 19.7 (± 0.5) wt. %.

Key words : Topaz, optical properties, dispersion, spindle stage.

Étude des propriétés optiques d'une topaze au moyen d'une platine à aiguille.

Résumé. — On a déterminé l'angle 2V et les indices de réfraction de fragments d'un cristal de topaze au moyen des techniques de platine à aiguille de Bloss. On a aussi mesuré la dispersion de ces propriétés optiques pour les longueurs d'onde de 400 à 900 nm.

A 589 nm, $2V_z$ vaut 65,7° ± 0,05° et la dispersion des axes optiques est 2,9° (v < r). Les indices de réfraction à 589 nm sont $N_x = 1,610.9$, $N_y = 1,613.7$, $N_z = 1,620.9 \pm 0,000.3$. Les mesures de dispersion suggèrent la présente d'une arête d'absorption aux environs de 100 nm.

Les méthodes de simple variation et de double variation ont été toutes deux utilisées pour déterminer les courbes de dispersion des indices de réfraction. Les deux méthodes conduisent à des résultats comparables pour les mêmes conditions de mesure, mais la méthode de double variation peut donner des mesures beaucoup plus précises et certaines que ces résultats, ce qui n'est pas le cas pour la méthode de simple variation.

La composition de la topaze a été déterminée directement par microsonde électronique et calculée à partir des paramètres optiques et les dimensions de la maille en utilisant les équations de Ribbe-Rosenberg. Les résultats sont cohérents et sont en bon accord avec les relations préalablement déterminées entre composition et propriétés optiques. La teneur en fluorine déterminée par microsonde est 19,3 ($\sigma = 0,3$)% en poids tandis qu'elle est de 19,7 \pm 0,5 % en poids par les méthodes optiques et de rayons X.

Mots clés : topaze, propriétés optiques, dispersion, platine à aiguille.

The optical properties of topaz have been studied rather extensively over a period of many years (e.g., Penfield and Minor, 1894; Rosicky, 1916; Rinne, 1926; Kôzu and Ueda, 1929; Pardee et al., 1937; Ödman, 1950; Fairbairn and Podolsky, 1951; Rosenberg, 1967; Ribbe and Rosenberg, 1971; Akizuke et al., 1979; Crovisier, 1980) and they are relatively well known for light of wave length 589 nm and for variations in composition and crystal structure (Ribbe and Gibbs, 1971; Akizuki et al., 1979). In the process of learning to use and testing the spindle stage techniques of Bloss (Bloss and Reiss, 1973; Bloss, 1978; Bloss, in press) the refractive indices, 2V and composition of a topaz have been measured to a relatively high degree of precision, and their dispersion determined over a range of wave lengths from 400 nm to 900 nm.

The material used in this work was fragments of the crystal from which a topaz sphere was made by Fairbairn and Podolsky (1951) for their study of 2V measurement with the universal stage. The locality of origin and chemical analysis of the crystal were not given by those authors as their only concern was evaluation of the precision and accuracy of the universal stage in measuring 2V.

In the present study the chemical composition of the material was determined by electron microprobe, X-ray and optical methods (see later discussions) to be very fluorine-rich, containing 19.7 (\pm 0.5) wt. % F. All fragments appear optically to be homogeneous. None of the anomalies such as compositional zoning, sector zoning, and variation of 2V and extinction angles as described by Akizuki *et al.* (1979) for some Brazilian topazes were found. To make this determination nine fragments ranging in size from 0.2 mm to \$.7 mm were examined optically, viewing basal sections on the flat stage.

INSTRUMENTAL METHODS

The techniques used were essentially those developed by Joel and Garaycochea (1957) and Wilcox (1959) as expanded, refined and adapted for computer analysis by Bloss (1978) and his colleagues (Louisnathan *et al.*, 1978) at the Virginia Polytechnic Institute and State University (VPI).

Refractive index determination and extinction angle measurements were done both at the University of Houston and at VPI. Dispersion measurements were made with Dr. Bloss' equipment at VPI (c.f. Louisnathan et al., 1978, p. 396).

The procedure can be outlined in brief as follows :

A. Determination of 2V.

1. Measure extinction angles of the fragment in monochromatic light at a regular interval of rotation on the axis of a spindle stage.

2. From the extinction angles calculate the extinction curves, 2V, and the location of the principal optical directions X, Y, Z and optic axes using a computer program (Excalibr of Bloss, in press).

3. Repeat the procedure with a range of wave lengths of monochromatic light to determine dispersion of the principal optical directions and optic axial angle.

B. Determination of refractive indices.

1. From computed positions of X, Y, Z, orient each of these successively parallel to the vibration direction of the microscope polarizer and in the plane of the microscope stage.

2. In each parallel position determine a match between calibrated immersion liquids and the mineral index at several wave lengths, using single or double variation methods, and thereby establishing dispersion curves for N_x , N_y , N_z .

3. With dispersion curves, or equations derived for them, determine principle refractive indices at a common wave length, such as that of Na_{D} light.

The methods are outlined in some detail because they are not all standard procedures as yet and part of the purpose of this report is to illustrate the degrees of precision and accuracy to be expected from these procedures and what is necessary to achieve such degrees. The work done in the two different aboratories serves as a good test of the reliability of he results.

Extinction Angle Measurement.

The extinction angles measured are the angles etween the vibration directions (X' and Z') in the lane of the microscope stage and the spindle axis. There is a pair of such angles for each position of rotation about the spindle axis. The poles of the vibration direction so determined plotted on a sphere define the extinction curves for a given grain in a given orientation on the spindle (Joel and Garaycochea, 1957).

Extinction angles were measured with a Supper spindle stage which accepts standard X-ray goniometer heads and allows 360° of rotation of the spindle. For each grain the angles were measured with a full 360° rotation of the grain at 10° intervals on the spindle. For comparison of different grains extinction angles were measured at a wave length of 589 nm using a narrow-band-pass optical filter and a Macé de Lepinay plate and Wright eyepiece. The angles were read to 0.05° and measured six times (the average standard deviation for each angle was 0.07° and the maximum was 0.15°).

For dispersion of 2V at different wave lengths filters of 400, 500, 666 and 900 nm were used to monochromatize the light. Extinction angles were measured at the minimum reception of a photomultiplier tube that was installed over the ocular of the microscope. Angles were read to 0.1 degrees with an estimated standard deviation of all readings totaling 0.04°.

Three fragments (Top 1, 2, 3 UH) were measured at the University of Houston with a Zeiss standard GFL Pol microscope with the 589 filter and Macé de Lepinay plate. Top 3 UH was remounted (Top. 3 VPI) and measured at VPI on a Leitz Ortholux microscope, using the same spindle stage, Macé de Lepinay plate, and 589 nm filter used in Houston. The spindle stage and fragment were then transferred to another Leitz Ortholux microscope for determing dispersion of 2V, and refractive indices, using the photomultiplier tube for extinction angle measurement.

Refractive Index Measurement.

For all work standard Cargille immersion oils series A of 0.002 index interval were calibrated with Zeiss Abbé refractometers.

Top. 2 was measured in Houston utilizing the single variation method with a Bausch and Lomb high intensity grating monochromator. Matching was achieved by observation of disappearing Becke line and is the largest single source of error in all index measurements. Matches were repeated six to twelve times, depending on ease of distinguishing the Becke The precision of determination ranged line. from ± 1 nm to ± 5 nm, with a standard deviation of 2.37 for 11 sets of readings on which statistics were kept. Matching indices with several oils allowed the plotting of the dispersion curve for each principal vibration direction on a Hartmann net, and the consequent determination of the indices at a wave length of 589 nm. All measurements were made at room temperature (20° to 21 °C) which varied over a range of no more than 0.5 °C for any set of measurements. Temperature readings were made at each sequence of readings using that oil. Corrections were made for temperature variation and all data

were corrected to a temperature of 20.5 °C, assuming no change of index with temperature for the mineral.

Top. 3 was measured at VPI employing the double variation technique reported by Louisnathan et al. (1978). The method uses the Sellmeier equation relating refractive index and light wave length to determine the constants $(a_0 \text{ and } a_1)$ that summarize dispersion curves of the immersion oils and mineral fragment (Louisnathan et al., 1978, p. 395, Eq. 4). For mineral refractive indices matches with oil were repeated four to six times and the precision of λ determination ranged from ± 0 to ± 7 nm, with a standard deviation of 3.28 for 30 sets of readings. For calibration of oils and mineral index measurements all values were corrected to a temperature of 25 °C. The equations for dispersion curves of the indices of the topaz were used to calculate the values of those indices at 589 nm.

In practice the double variation procedure used for Top. 3 at VPI is more rapid than the single variation method with the Hartmann net and it provides many more data points for the dispersion curves of the mineral over a greater interval of wave length. It is clearly superior, but does take a more elaborate instrumental setup. The results obtained by the two methods are very close to each other, and well within the estimated limits of error for each method. Of course the single variation data can also be analyzed using the least squares fit of the Sellmeier equation, as illustrated in the appendix and in table IV with results essentially the same as obtained from the Hartmann net.

RESULTS

2V.

The optic axial angles $2V_z$ as determined for fragments 1, 2 and 3 are given in table I. For the values calculated by Excalibr two modifications were used. Excalibr was originally designed to use

	Sample	Measurement Method	2VZ	Standard Deviation ^C
Topaz	sphere	Optic angle goniometer	65.17 ^a 65.18 ^a	.01 .02
Topaz	sphere	Universal stage, average from 5 stages, using Na _D light	65.27 ⁸	.31
Top. 1	fragment	Extinction angles	65.71 ^b	.05
Top. 2	fragment	Extinction angles	65.71 ^b	.06
Top. 3	fragment UH	Extinction angles	65.65 ^b	.05
Гор. 3	fragment VPI	Extinction angles	65.89 ^b	.21

Fairbairn and Podolsky (1951), Table 5.

Calculated to the minimum of the least squares residual in program EXCALIBR, see text.

For topaz sphere this value is the "error of the mean" or "standard error" of Fairbairn and Podolsky (1951, p. 825). For the fragments used in the extinction angle measurements this value is the estimated standard deviation as calculated by the EXCALIBR program (Bloss, in press, Ch. 6). extinction angles measured only through 180° rotation on the spindle axis. For this work the angles for the full 360° rotation were used in order to compensate for such problems as :

(1) observational errors in reading angles;

(2) errors in centering the grain;

(3) variation of extinction angle across the lens;

(4) oil and grain do not have precisely the same indices of refraction;

(5) the grain is an irregular shape.

All of these problems, as pointed out by Fisher (1962), have distinct effects on extinction angle measurement that are to a large extent compensated by measuring pairs of extinction angles 180° apart on the spindle axis. To adapt the angles measured through 360° to program Excalibr the formula

$$\frac{(2MR - MS_{180-350}) + MS_{0-170}}{2}$$
 Eq. 1

(Bloss, in press) was used to combine extinction angles 180° apart to a single angle for program input, thus deriving a set of 18 angles from the 36 measured. In the formula MR is the microscope stage reading when the spindle axis is precisely aligned with one of the polarizer vibration directions, and MS₀, MS₁₈₀, etc. are microscope stage readings at extinction for spindle axis settings of 0°, 180°, etc. (Bloss, 1978).

The second modification was to calculate 2V at a truly minimum least squares residual (LSR) derived from Joel-type equations (Bloss and Reiss, 1973, p. 1060-61; and Bloss, in press ch. 6). Excalibr computes 2V and the corresponding LSR stepwise at incremental values of MR and, depending on the geometry of grain orienation, the difference in calculated 2V over each interval may be as great as 0.2°. The best value of 2V is taken as that one for which LSR is the smallest. The smallest LSR may fall at an MR value between the steps of Excalibr. Therefore, a small correction has been made by calculating 2V at the point within an Excalibr increment where LSR is smallest. This was done by fitting a quadratic function to the variation of LSR with different values of MR and minimizing that function. 2V is then obtained by linear interpolation between incremental Excalibr values of 2V.

The difference in $2V_z$ between Top. 3 UH and Top. 3 VPI is attributed chiefly to the fact that the VPI orientation of the crystal was not as favorable as that of UH (Bloss and Reiss, 1973, p. 1059). Stereographic projections (Figure 1) demonstrate that while all orientations have optic axes at large angles to the spindle, one axis of Top. 3 VPI has the smallest at 43.8° (Figure 1d). Moreover, Top. 3 VPI has the X vibration direction at the smallest angle (38.2°) to the spindle of any of the major optical directions in any of the grains, giving the smallest polar extinction curve of the four cases. The stereographic plots of Top. 1, 2 and 3 UH reveal that in all cases both extinction curves cross the circles located 40° from the spindle axis (Figures 1a-c), thus fulfilling conditions for a most favorable orientation of the grain wherein the effect of errors in measurement of extinction positions is



FIG. 1. – Stereographic projections of extinction curves for the three grains studied. Top. 1, 2, 3 UH are different grains, all measured at the University of Houston. Top. 3 VPI is the same grain as 3 UH, but measured at Virginia Polytechnical Institute. All figures rotated about the spindle axis to portray optical directions in similar positions with the X vibration direction in the vertical E-W plane. Angles from X and A to the spindle axis calculated by Excalibr program. Spindle axis oriented east-west.

	A1		A ₂		x		Y		Z	
	s	E	s	E	s	E	s	Ē	s	E
400	21.73 (.08)	83.18 (.05)	161.63 (.09)	43.99 (.06)	65.83 (.09)	37.49 (.05)	107.82	119.68	5.37 (.07)	110.7
500	21.86 (.06)	82.65 (.04)	160.74 (.07)	43.71 (.04)	65.82 (.07)	37.58 (.04)	107.61 (.06)	119.84 (.05)	5.13 (.06)	110.64
666	22.08 (.08)	82.11 (.05)	160.20 (.09)	43.41 (.06)	65.86 (.08)	37.52 (.05)	107.52 (.08)	119.8 4 (.06)	5.10 (.07)	110.56 (.02)
00	22.23 (.09)	81.88 (.05)	159.89 (.11)	43.10 (.07)	65.74 (.09)	37.50 (.06)	107.55 (.09)	119.76 (.07)	5.12	110.63

Figures	in	parentheses	аге	estimated	standard	deviations	calculated t	vc	EXCALIBR	(Bloss,	in	press.
Ch. 6).								-				

TABLE II. – Dispersion data for indicatrix orientation of Top. 3 VPI. Excalibr, Calculated Spindle Stage Coordinate Angles.

Sample	LSR (x10 ⁻⁶)	Estimated Std. Dev.	λ (nm)	
Top. 1	12.6	.05	589	Testingtion combo
Top. 2	24.3	.06	589	measured using Mace
Top. 3 UH	10.6	.05	589	de Lepinay quartz wedge
Top. 3 VPI	256.8	.21	589	
Top. 3 VPI	55.9	.10	400	D
Top. 3 VPI	32.3	.07	500	measured using
Top. 3 VPI	57.3	.10	666	photomultiplier tube
Top. 3 VPI	73.2	.11	900	

a Both calculated by EXCALIBR program (Bloss, in press, Ch. 6).

TABLE III. – Least squares residuals and estimated standard deviation of $2V_{Z}^{a}$.

minimized (Wright, 1966). The equatorial extinction curve of Top. 3 VPI (Figure 1d), on the other hand, does not intersect the 40° curve, and it has significantly less character (strong curvature) than those of Top. 1, 2, 3 UH. A second factor casting doubt on the value of $2V_z$ for Top. 3 VPI is a rather high LSR (Table III). The residual for Top. 3 VPI at 589 nm is an order of magnitude greater than that of Top. 1, 2, and 3 UH. This suggests that the extinction angles were not quite as precisely located at VPI as at Houston. The net result of these considerations appears to be a $2V_z$ value for Top. 3 VPI at λ 589 nm that is a bit too, high. It has a larger standard deviation than those of other grains, but one which would bring $2V_z$ within the range of those for Top. 1, 2, 3 UH.

Dispersion of 2V.

Figure 2 shows the values of $2V_z$ for Top. 3 VPI calculated from extinction angles measured at wave lengths of 400, 500, 589, 666 and 900 nm. The points



FIG. 2. – Dispersion of $2V_Z$. • – Top. 3, VPI; \bigcirc – Top. 3 UH; I – estimated standard deviation calculated by Excalibr (Bloss in press, Ch. 6).

lie close to a smooth curve; however, the value at 589 may be a bit high, as just discussed, and therefore $2V_{7}$ Top. 3 UH is also shown. Table III demonstrates that for Top. 3 VPI the least squares residuals for extinction angles and estimated standard deviations of $2V_z$ for measurements using the photomultiplier tube are all less than the measurement at 589 nm, using the Macé de Lepinay plate. This suggests that the extinction measurements were better with the photomultiplier tube and that the $2V_z$ values are more accurate. The orientation of the grain when measured with the photomultiplier was the same as with the Macé de Lepinay plate at VPI. This less desirable orientation, discussed in the previous section, appears to be reflected in the consistently higher least squares residuals of extinction angles and estimated standard deviations of $2V_z$ for $\lambda 400$ -900 nm, as compared with Top. 1, 2 and 3 UH (Table III).

It is concluded that $2V_z$ values for wave lengths other than 589 nm are reasonably close to the true

value, but that the value for 589 should be closer to 65.7 (UH) than the value of 65.9 (VPI). This change makes a smoother curve.

Table II gives the calculated spindle stage coordinates for Top. 3 VPI dispersion measurements. Optic axis dispersion is obvious, and Disper, the subroutine of Excalibr which statistically analyzes the probability of the data representing actual dispersion in the mineral (Bloss, 1978; Bloss, in press), shows that the null hypothesis (no dispersion) can be rejected for all 2V data. For the major axes of the indicatrix the case is more ambiguous and though slight dispersion of Y and Z is a possibility the data do not warrant a firm conclusion.

Refractive Indices.

Measurement of the principal refractive indices N_x , N_y and N_z by the single variation method in Houston and the double variation method at VPI produced closely similar results for a wave length of 589 nm as shown in table IV. The values for each principal index

and Sample	Sellmeie	r Constants	Predicted ind	lices for Fra	unhofer lin	es
Double Variation Least Squares Sellmeier	BO	^a l	C: 656.1 nm	<u>D: 589.3 n</u>	<u>m</u>	<u>F: 486.1 n</u>
Top. 3 VPI						
NX	.64919	-7726.7	1.6075	1.6109 (.0009) ^a	(.924) ^b	1.6193
NY	.64399	-7089.9	1.6105	1.6136 (.0005) ^a	(.983) ^b	1.6213
NZ	.63340	-6586.1	1.6180	1.6210 (.0003) ^a	(.984) ^b	1.6283
Single Variation Least Squares Sellmeier						
Top. 2						
NX	.64282	-5509.4	1.6085	1.6109 (.0000) ^a	(.999) ^b	1.6168
NY	.63838	-5190.3	1.6114	1.6137 (.0004) ^a	(.972) ^b	1.6193
NZ	.62869	-4897.5	1.6186	1.6208 (.0002) ^a	(.990) ^b	1.6263
Single Variation Hartmann Plot						
Top. 2						
NX			1.6084	1.6109 (.0003) ^e		1.6169
NY			1.6116	1.6138 (.0004) ^c		1.6194
NZ			1.6188	1.6209 (.0002) ^e		1.6261
Standard error of	the estima	te.				
r ² from analysis o	of variance.					
Limits of error e	estimated b	y plotting on	through centers	viation of d	ata points	and locating

TABLE IV. – Dispersion of refractive indices.

fall within a range of 0.000 2. All are well within the probable limits of error for each type of measurement.

For Top. 2 a comparison was made between the plotting method, using the Hartmann relation, and a mathematical treatment, using the Sellmeier relation and least squares treatment of the data. With the least squares method a standard error can be calculated for comparison with the visually estimated error limits of the Hartmann plot and the calculated estimated error of the double variation method. The estimates of error for N_x and N_y in the double variation method are a bit higher and their r^2 values somewhat low. This is attributed to a small drift in the temperature of the ice-water bath used to calibrate the thermocouple of the temperature control system. This was corrected in the measurement of N_z and its estimate of error and r^2 values are better. The single variation estimates of error and r^2 values are a bit misleading and do not necessarily reflect a greater precision, because they are based on fewer data points.

In view of the strong dependence of refractive index of topaz on the relative F versus OH content, it seems reasonable to assume that the similarity of refractive indices between the two different fragments attests to chemical homogeneity of the material used in this work.

Dispersion of Refractive Indices.

The methods used allow convenient determination of the variation of principal refractive indices with wave length. Table IV gives the results for Top. 2 and Top. 3 VPI.

Using the relation

$$\lambda_0 = \left(\frac{-a_1}{a_0}\right)^{1/2}$$
 Eq. 2

derived from equations 1 and 2 of Bloss (1978), the absorption edge λ_0 can be calculated for each principal vibration direction. For Top. 3 VPI these turn out to be 109, 105 and 102 nm respectively for X, Y and Z, while the respective values are 92, 90 and 88 for Top. 2. Although the extrapolation is extreme and the values must be considered only approximate they are quite in keeping with what is to be expected with colorless transparent silicates of compositions such as that of topaz (Strens and Freer, 1978, p. 21).

Composition of Topaz.

Table V summarizes the results of all compositional determinations made on the topaz used in this study. Electron microprobe analysis was done on separate fragments from those measured optically. The consistency of optical properties between grains strongly indicates homogeneity of the large crystal from which these fragments were obtained and it is assumed therefore that the fragments used for microprobe and X-ray analyses have compositions essentially the same as the grains spindled for optical measurements.

Electron microprobe analysis shows the topaz to be very fluorine rich (19.3 \pm 0.5 Wt. % F) (*). X-ray diffraction analysis with unit cell refinement for the b

Electron Microprobe (Average of 10 spots) Cations on the basis of 24 (O, OH, F) Si - 3.963 4.00 SiO2 - 32.23 (+ .3, .15)(1) Al2O3 - 56.63 (+ .6, .33) A1(4) _ .037 F - 19.34 (+ .5, .32) A1(6)_ 8.172 108.20 F - 7.521 0 = F - 8.14 Σ - 19.693 100.06 Numbers in parentheses give the limits of the range of values measured and one standard deviation, respectively, for the ten spots measured. Calculated from unit cell values (x-ray powder diffraction) b axis - 8.7949Å - F = 20.0 (+ .3)(2) (2) Averages from four diffractograms. Numbers in parentheses give the limits of the range of values for the four measurements. Calculated from optics 1. Refractive indices at λ 589 (values in Table 4). $N_{Y} - F = 20.2$ $N_{Y} - F = 20.2$ (All values of each index give same result to first decimal) $N_{Z} - F = 20.2$ 2. 2VZ (values in Table 1) Goniometer - F = 19.6 Universal stage - F = 19.6 Extinction angles - F = 19.7 (All 4 values give same result)

TABLE V. - Topaz composition.

axis gives a slightly higher result (20.2 Wt % F) using the equation of Ribbe and Rosenberg (1971, p. 1816). Ribbe and Rosenberg (1971, p. 1818) observe that unit cell volume is not a good estimator of fluorine content because of the poor correlation between a and cdimensions and fluorine content, thus the equation for volume was not used. The polynomial regression equations for refractive index published in Ribbe and Rosenberg (1971, p. 1816) give spurious results because they are shown only to a whole number and the decimal values are needed. Accordingly, the eightsample data set used by Ribbe and Rosenberg (1971, p. 1815) was used to calculate a new group of least squares polynomial regression equations for refractive index determination curves (**). The new equations are as follows :

The results given by these equations are the same for all three indices (20.2 Wt % F). Finally, the fluorine content was calculated using the $2V_z$ equation of Ribbe and Rosenberg (1971, table 2). All of the $2V_z$ values obtained in this work give the same result (19.7 Wt % F), while all those obtained by Fairbairn and Podolsky (1951) with the optical goniometer and universal stage are a bit lower (19.6 Wt % F).

^(*) Analyses were performed by R. Brown in the Electron Microprobe Laboratory, Geochemistry branch, Johnson Space Center, Houston, Texas. Analytical conditions were : acceleration current 15 kV, sample current 0.02 microamperes on CPX, CP19. Spot size about 10 microns. Standards were kyanite for Si and Al and high fluorine topaz for F.

^(**) The program used for these calculations is BMDP5R-Polynomial Regression, Health Sciences Computing Facility, University of California, Los Angeles. Copyright (c) 1979, Regents of University of California.

Taking the simple average of all X-ray and optical determinations in table V yields a value of 19.9 ($\sigma = 0.27$) weight percent fluorine. This suggests that the microprobe results are a little low. The best estimate for the composition is judged to be 19.7 \pm 0.5 weight percent fluorine.

Conclusions.

These results indicate that 2V can be determined routinely to an accuracy of about 0.1 degrees by Bloss' extinction angle method. A major improvement might be achieved if a cylinder or sphere were fashioned of the grains to be measured (Bloss, 1978, p. 441). Clearly relatively slight dispersion of 2V is easily detectable and is shown for the topaz studied. There is not dispersion of the X vibration direction but there may be very slight dispersion of Y and Z, although the author considers it dubious.

In the present work the two methods for determination of refractive indices appear to be of comparable levels of accuracy and precision; however, it is felt that the single variation method is at its limits while the double variation method can be rendered more precise with the investment of only a modest increment of time. For Top. 3 VPI one day sufficed for the refractive index dispersion measurements by the double variation method, whereas the single variation method required 3 days to make fewer measurement sets on Top. 2.

The chief limiting factor for precision of refractive index measurements is the ability of the eye to detect the Becke line disappearance or the reversal of shadows in oblique illumination in making the match with an oil. This limits the precision of determining the wave length of match. Grain shape is important here and possibly spherical grains would improve the measurements.

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APPENDIX

For those who may wish to test the procedures used in deriving the Sellmeier constants for the refractive indices of the mineral from the method of least squares there is here provided the example of Top. 2.

Vibration Direction	λ of match between oil and mineral (nm)	Observed N of oil at λ of match (corrected for temp.)	Predicted N	Difference	
х	533	1.613 7	1.613 7	0.000 0	0.623 44
	604	1.610 3	1.610 3	0.000 0	0.627 72
	640	1.609 0	1.609 0	0.000 0	0.629 37

Sellmeier constants a_0 and a_1 in the relation :

$\frac{1}{N^2 1} = a_0 + a_1 \frac{1}{\lambda^2}$

Derived from Eqs. 1 and 2 of Bloss (1978).

$$a_0 = 0.642 \ 82$$

$$a_1 = -5 \ 509.41$$

$$r = 0.999 \ 9$$

$$r^2 = 0.999 \ 9$$

standard error of estimate = 0.0000

Y

Z

of of commute				
$ \begin{array}{r} \operatorname{At} \lambda \\ 1/N^2 - 1 \\ N \end{array} $	486.1 0.619 50 1.616 85	589.3 0.626 96 1.610 90	656.1 0.630 03 1.608 49	
	$\lambda_0(\mathbf{X}) = \left(\frac{-a_1}{a_0}\right)^{1/2} =$	= 92.58		
λ	N _{obs}	N_{pred}	Δ	$1/N^2 - 1$
507 572 595 649	1.618 0 1.614 0 1.613 8 1.611 9	1.617 9 1.614 4 1.613 5 1.611 6	$\begin{array}{r} + \ 0.000 \ 1 \\ - \ 0.000 \ 4 \\ + \ 0.000 \ 3 \\ + \ 0.000 \ 3 \end{array}$	0.618 08 0.623 06 0.623 31 0.625 70
	$a_0 = 0.638 \ 38$ $a_1 = -5 \ 190.$ $r = 0.986 \ 3$ $r^2 = 0.972 \ 8$ SEE = 0.000 4	28		
$ \begin{array}{c} \operatorname{At} \lambda \\ 1/N^2 - 1 \\ N \end{array} $	486.1 0.616 41 1.619 35	589.3 0.623 43 1.613 70	656.1 0.626 33 1.611 40	
	$\lambda_0 = 90.17$			
λ	N _{obs}	N_{pred}	Δ	$1/N^2 - 1$
502 525 556 607	1.625 3 1.623 6 1.622 5 1.620 1	1.625 2 1.623 8 1.622 3 1.620 2	$\begin{array}{r} + \ 0.000 \ 1 \\ - \ 0.000 \ 2 \\ + \ 0.000 \ 2 \\ - \ 0.000 \ 1 \end{array}$	0.609 16 0.611 22 0.612 56 0.615 49
	$a_0 = 0.628 \ 69$ $a_1 = -4 \ 897.5$ $r = 0.995 \ 5$ $r^2 = 0.991 \ 0$ SEE = 0.000 2	55		
$\frac{\operatorname{At}\lambda}{1/N^2-1}$ N	486.1 0.607 96 1.626 29	589.3 0.614 59 1.620 83	656.1 0.617 32 1.618 61	

 $\lambda_0 = 88.26$

It is understood that the data points are too few for the statistics to be greatly meaningful. However, each λ of match was read many times and only the averages of each reading are given here to show the method.