

## REVISED DISPERSION METHOD FOR LOW PLAGIOCLASE

S. A. MORSE, *Franklin and Marshall College, Lancaster, Pennsylvania*

## ABSTRACT

Revised cleavage flake curves for low plagioclase determination are derived from the refractive indices of Smith (1960), which are applied to the indicatrix orientations of Tsuboi (1923) or Slemmons (1962). A new dispersion chart, based on the  $\alpha'_{001}$  curve, allows use of the much-neglected dispersion method, which affords good statistical information and is capable of a standard error ( $s/\sqrt{n}$ ) of  $\pm 0.1\%$  An under exceptional conditions.

## INTRODUCTION

Surprisingly little application has been made of the dispersion method since its development by Merwin more than 50 years ago (Merwin and Larsen, 1912).<sup>1</sup> The method has much to commend it in petrographic work. It is applicable to a wide variety of minerals. It is rapid and convenient, requiring no temperature control apparatus. Since work is done at ambient temperatures, there is a minimum of uncertainty about the true temperature of the mount or about the index of the immersion liquid, both of which may be affected to a small degree by differential evaporation at elevated temperatures (Fairbairn, 1952). Most important, the method is capable of high precision, and furnishes a suite of statistics which can lead to knowledge of refractive index to  $\pm 0.0001$  and to knowledge of compositional variability or homogeneity. Maximum, minimum, and mean values of refractive index or composition can be obtained for 20 mineral grains in as many minutes, and the operator can easily choose the number of observations required to achieve the desired confidence limits. The method has been applied in these laboratories to olivines, pyroxenes, and garnets, as well as to plagioclase.

The pinnacle in the application of the dispersion method was perhaps achieved by Tsuboi (1934) in his famous dispersion chart for plagioclase determination. This chart was based on his  $\alpha'_{001}$  cleavage flake curve published in 1923. It has been apparent since the work of Chayes (1952) and Smith (1958, 1960) that the nine original plagioclases of Tsuboi included some unusual values of refractive index (Fig. 1). These account for the somewhat uneven 1923 curves which cause overestimation of An content by about 3 mol percent in much of the region below An<sub>50</sub>. In studying a layered intrusion, the writer (Morse, ms 1961) generated a new set of cleavage flake curves and a dispersion chart by combining the original indicatrix orientations of Tsuboi with the refractive indices of Chayes (1952), and later Smith (1960). Subsequent experience with

<sup>1</sup> For historical development, see Tsuboi (1923, p. 108-109) and his refinements of 1930 and 1934.

these curves has led to the belief that they are valid, and can be used to great advantage both in reconnaissance work and in precise studies of plagioclase composition variation. For comparative studies of plagioclases from a single environment or genetic history, it is even probable that compositional variation can be determined with greater precision by the dispersion method than by any analytical means, possibly excepting the electron microprobe, which gives comparable results and statistics.

#### NUMERICAL

The paragenesis of Tsuboi's (1923) nine samples leads one to believe that they are mostly of low structural state. An examination of the prin-

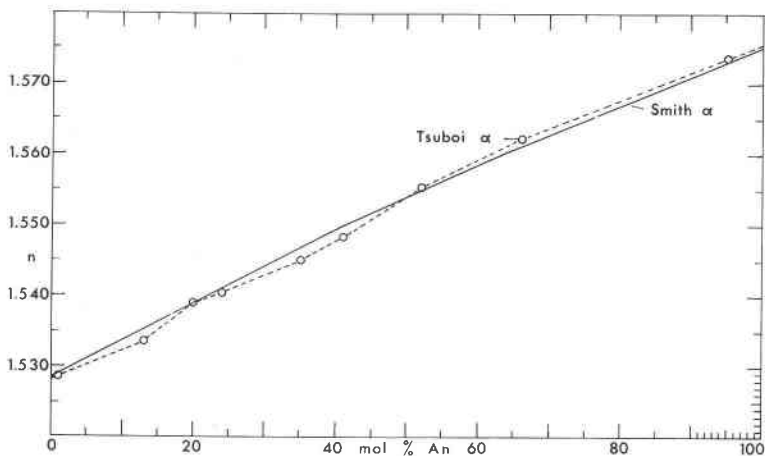


FIG. 1. Comparison of  $\alpha$  refractive indices of Smith (1960, solid curve) and Tsuboi (1923, dashed curve).

cipal refractive indices compared to modern curves (*e.g.*, Smith, 1960) suggests that the erratic nature of Tsuboi's cleavage flake curves is due almost wholly to abnormal indices and not to abnormal orientations. That these are valid orientations for low plagioclase appears to be confirmed by Slemmons' (1962, Table 3) universal stage data from a wider variety of samples. The curves for  $\alpha'_{001}$  from the data of Tsuboi and Slemmons differ but little; calculations of both are included here for comparison.

Smith (1958) has called attention to the reliability of  $\alpha$  as an estimator of composition independent of structural state except in the sodic region. His 1960 refractive indices appear to meet the criteria of sampling, analysis, precision, and internal consistency better than any others available, so these were chosen for the new cleavage flake curves.

TABLE 1. VALUES OF  $\alpha$  AND  $\gamma$  FOR THE NINE Tsuboi COMPOSITIONS, FROM SMITH'S DATA

Mole % An	$\alpha$	$\gamma$
1	1.5289	1.5390
13	1.5353	1.5442
20	1.5390	1.5472
24	1.5411	1.5488
35	1.5469	1.5539
41	1.5500	1.5570
52	1.5551	1.5626
66	1.5614	1.5701
95	1.5732	1.5859

To apply the indices of Smith to the orientations of Tsuboi, the Smith data were first plotted and connected with straight-line segments as follows:

For  $\alpha$ : An<sub>0</sub> 1.5284

An<sub>41</sub> 1.5500

An<sub>64</sub> 1.5607

An<sub>100</sub> 1.57525

For  $\gamma$ : An<sub>0</sub> 1.5386

An<sub>31</sub> 1.5518

An<sub>55</sub> 1.5642

An<sub>100</sub> 1.5886

The refractive index values for the nine Tsuboi compositions were then picked off the curves, with the results shown in Table 1. These values were introduced, along with Tsuboi's spherical coordinates, into Tsuboi's equations for the partial indices on (010) and (001) cleavage flakes. These equations are

$$\alpha'^2 = \frac{2\gamma^2\alpha^2}{(\gamma^2 + \alpha^2) + (\gamma^2 - \alpha^2) \cos(\psi - \psi')} \quad (1)$$

and

$$\gamma'^2 = \frac{2\gamma^2\alpha^2}{(\gamma^2 + \alpha^2) + (\gamma^2 - \alpha^2) \cos(\psi + \psi')} \quad (2)$$

where  $\psi$  and  $\psi'$  relate to the positions (in longitude) of optic axis  $A$  and optic axis  $B$ , respectively, with respect to either (010) or (001). The values for  $\cos(\psi - \psi')$  and  $\cos(\psi + \psi')$  calculated from Tsuboi's data are listed in Table 2.

The resulting values for refractive indices on cleavage flakes for the nine Tsuboi compositions are tabulated in Table 3. When plotted, the data can be fitted with straight-line segments defined by the values emphasized in Table 3. The final cleavage flake curves are shown in Figure 2.

TABLE 2. SUMMARY OF ORIENTATION PARAMETERS OF TSUBOI'S NINE PLAGIOCLASES, FOR EQUATIONS (1) AND (2)

Mole % An	For (010) flakes		For (001) flakes	
	$\cos(\psi - \psi')$	$\cos(\psi + \psi')$	$\cos(\psi - \psi')$	$\cos(\psi + \psi')$
1	0.9995	0.1080	0.9416	-0.9402
13	.9998	.0698	.9799	-.9750
20	.9994	-.0698	.9892	-.9946
24	.9992	-.1294	.9960	-.9982
35	.9994	-.1495	.9045	-.9768
41	.9963	-.1616	.8307	-.9657
52	.9354	-.0122	.7155	-.8047
66	.8752	-.1501	.6510	-.6619
95	.4131	-.4939	.3628	-.3481

To make the plagioclase dispersion chart (Fig. 3), values of  $\alpha'_{001}$  were picked off the curve of Figure 2 for every percent An; these became the *D*-line intercepts on the chart. Dispersion values were then interpolated from Tsuboi's determinations of dispersion in (001) sections of five different compositions; the slopes increase regularly with An content.

To derive an  $\alpha'_{001}$  curve from Slemmons' data, the optical elements from his Table 3 were plotted for every 10 percent An, along with Smith's (1958) axial angle values. The normal to the basal plane was used as the

TABLE 3. REFRACTIVE INDEX ON CLEAVAGE FLAKES FOR THE NINE TSUBOI COMPOSITIONS, USING SMITH'S REFRACTIVE INDEX DATA. VALUES USED IN CONSTRUCTING FIG. 2 ARE EMPHASIZED

Mole % An	$\alpha'$		$\gamma'$	
	(010) flakes	(001) flakes	(010) flakes	(001) flakes
0 (fictive)	<b>1.5284</b>	<b>1.5287</b>	<b>1.5330</b>	<b>1.5382</b>
1	1.5289	1.5292	1.5334	1.5387
13	1.5353	1.5354	1.5394	1.5441
20	1.5388	1.5391	1.5434	1.5472
24	1.5411	<b>1.5411</b>	1.5454	1.5488
35	1.5479	1.5472	1.5509	<b>1.5538</b>
41	<b>1.5500</b>	<b>1.5506</b>	<b>1.5541</b>	1.5569
52	1.5553	1.5562	1.5588	1.5619
66	<b>1.5619</b>	1.5629	<b>1.5651</b>	<b>1.5686</b>
95	1.5769	1.5772	1.5827	1.5817
100 (fictive)	<b>1.5795</b>	<b>1.5797</b>	<b>1.5856</b>	<b>1.5840</b>

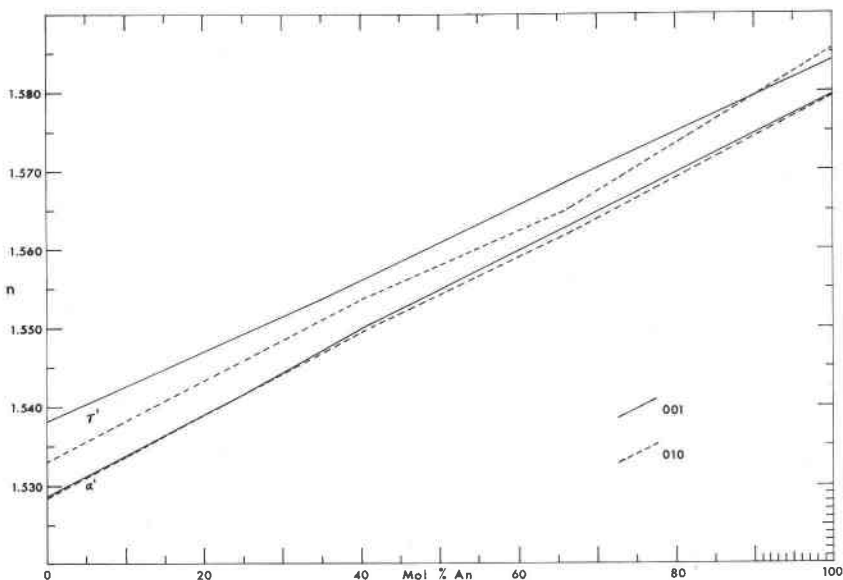


FIG. 2. Refractive indices on cleavage flakes of plagioclase derived from Smith's (1960) refractive indices and Tsuboi's (1923) orientations. Solid lines: (001) cleavage flakes; dashed lines: (010) cleavage flakes.

center of the net. Vibration directions were found by application of the Biot-Fresnel law. The extinction direction  $X'$  was then found from the intersection with the primitive of a prime ellipse which passes through  $Z$  and some point on the  $XY$  symmetry plane (say  $\beta'$ ). The partial index was then calculated from the principal indices taken from the Smith curves by solving the  $\alpha\beta$  ellipse for the value of  $\beta'$  and then solving the prime ( $\beta'$ ) ellipse for the value at  $X'$ . The resulting values of  $\alpha'_{001}$  (Smith + Slemmons) are tabulated for increments of 10 percent An in Table 4. These plot as a straight line from  $An_0$  (1.5290) to  $An_{84}$  (1.5722), and another to  $An_{100}$  (1.5797). The  $\alpha'$  curves derived from Tsuboi and Slemmons lie superimposed or very close to each other for much of their length. The maximum difference in index is 0.0004, near  $An_{40}$ ; this corresponds to a difference of 1 mol percent An over a short region of the curve. A difference corresponding to 0.5 percent An arises in the albite region.

At present there is little basis for deciding the relative merits of the two curves in the regions of disagreement; this will have to come with more analyses. The Tsuboi-derived curve, which is slightly higher at

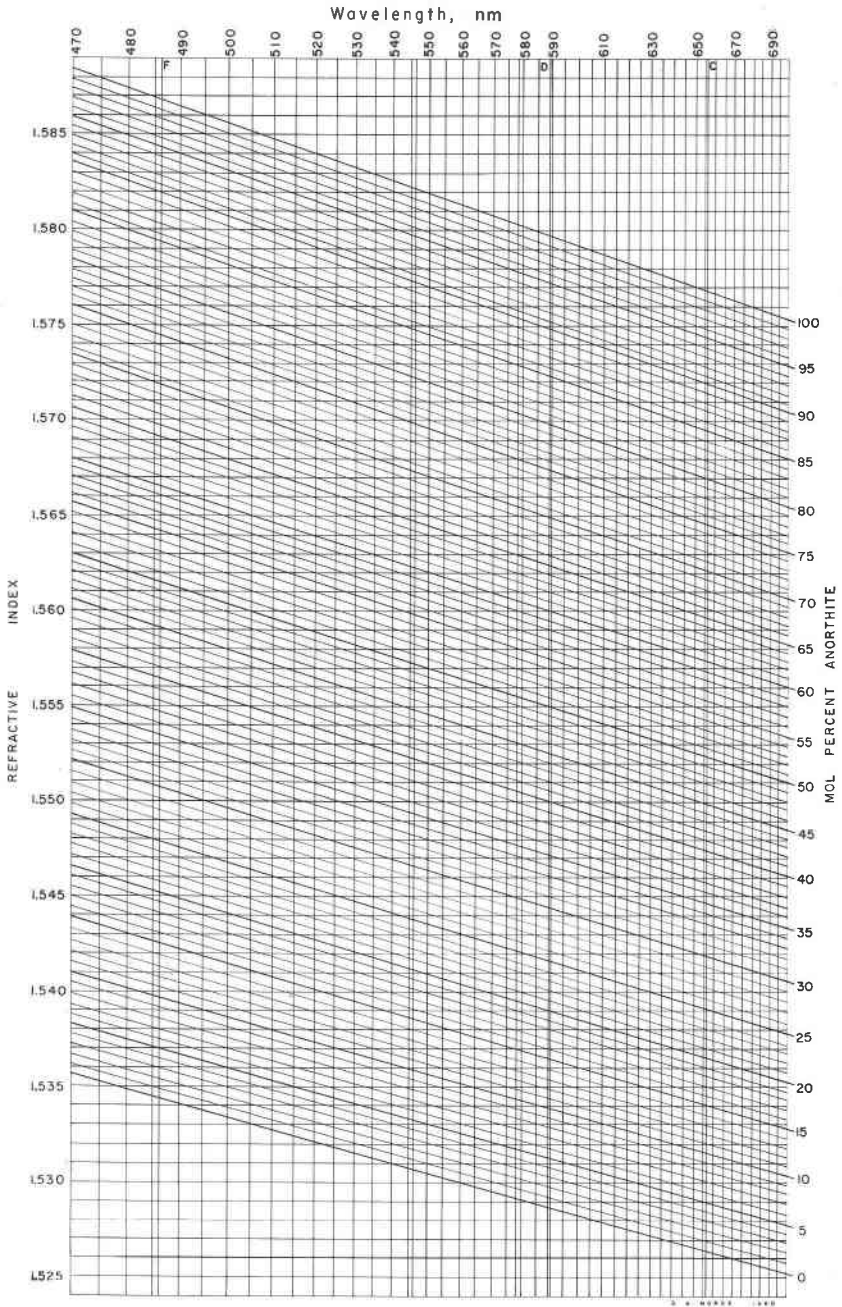


FIG. 3. Low-Plagioclase dispersion chart for  $\alpha'$  on 001 cleavage flakes (Smith-Tsuboi curve).

TABLE 4. VALUES OF  $\alpha'_{001}$  DERIVED FROM SMITH'S INDICES AND SLEMMONS' ORIENTATIONS

Composition	$\alpha'_{001}$
An 0	1.5290
10	1.5339
20	1.5390
30	1.5442
40	1.5496
50	1.5547
60	1.5597
70	1.5649
80	1.5701
90	1.5750
100	1.5797

An<sub>40</sub>, is retained in view of the high refractive index values found by Smith (1960) for a sample at this composition and in view of one of the writer's analysed samples whose  $\alpha'_{001}$  value lies nearer the Tsuboi-derived curve.

#### EXPERIMENTAL

*Source.* The source used for this work was a Leitz prism monochromator calibrated with sodium and mercury vapor lamps as sources, using the lines Na 467, 498, 515, 568, 589, and 616 nm and Hg 496, 546, 577, 623, and 691 nm. Entrance and exit slits were set at 0.02 mm for calibration purposes, at 0.04 mm and 0.02 mm respectively for minimum deviation spectrometry, and at 0.1 mm or rarely 0.2 mm for microscope illumination, depending on the spectral purity desired and the illumination needed near the limits of the wavelength range.

*Spectrometer.* A Model 113 Gaertner 20-inch spectrometer was used for refractive index determinations of immersion liquids at wavelengths of 490, 510, 550, 590, and 650 nm. An American Optical Company 60° optically flat hollow prism was used to contain the liquid. The prism angle,  $\alpha$ , was determined to  $\pm 0.002^\circ$  (mean deviation) by finding the minimum deviation angle,  $d$ , of demineralized water at a known temperature, and applying the relation  $\tan \alpha/2 = (\sin d/2)/(n \cos d/2)$ , where  $n$  refers to the refractive index of pure water at the temperature of observation, found in standard tables.

*Immersion liquids.* These were standard commercial liquids (Cargille) starting at 1.530 and continuing in intervals of 0.006 over the plagioclase

range. For more rapid work, liquids of higher dispersion may be used with the loss of some accuracy in determining the wavelength of match. The *D*-line refractive indices of the current liquids have remained stable within 0.0001 for the past eight years.

*Temperature measurement.* Temperatures in the hollow prism during minimum deviation measurements were measured with an iron-constantan thermocouple whose variable-temperature junction was immersed in the liquid under observation and whose standard junction was immersed in demineralized ice water. Both junctions were protected by a coating of epoxy to prevent electrolysis. Temperatures were read to the nearest 0.1° on a calibrated potentiometer. Both temperature and deviation angle were determined at least three times at a given wavelength setting.

The temperature of the mount on the microscope stage was determined to the nearest 0.1° by means of a mercury-in-glass thermometer whose bulb was suspended about 6–8 mm above the stage. Temperatures determined in this way were found to be virtually indistinguishable from those measured by a thermocouple whose junction lay on the cover glass. The use of standard glass powders (Sueno, 1933) would appear to be a satisfactory alternative to direct measurement of temperature. Direct measurement is made with some confidence, however, since oils, equipment, and samples are equilibrated at ambient temperature at all times.

*Overlay for liquids.* The refractive indices (corrected to 25°C) of the various liquids were plotted on a Hartmann net<sup>1</sup> for the five standard wavelengths mentioned above. A straight-line fit through each of the five points was usually obtained; for a few liquids, the point at 490 nm lay 0.0001 high, probably because of the low intensity of light at that wavelength with the fine slit settings used for minimum deviation. All the liquid dispersion curves were then transcribed on Mylar, along with the *D* line for reference, and this overlay was then used to find the intersection of a liquid dispersion curve with a plagioclase dispersion curve, the *D*-line value of some reference liquid being set at the value of *n* appropriate for the temperature of the observation. This value was read from a graph of  $dn/dT$ , the magnitude of which is  $-0.00041^{\circ}\text{C}$  for the liquids used.

<sup>1</sup> Abscissa (wavelength scale) defined by  $x = x_0 - 100,000/\lambda - 200$ , where  $\lambda$  is in nanometers (Tsuboi, 1930). Copies of the plain net and of the plagioclase dispersion chart may be obtained from the writer. Professor Tsuboi's dispersion net may be obtained from Ostrich Co., Ltd., Izumi-93, Komae-mati, Kitatama-gun, Tokyo, Japan.



*Immersion procedure.* About 10 plagioclase grains lying on (001) were observed for each sample in routine work, the number depending on the homogeneity of the sample and the precision desired. The identity of the cleavage makes no difference below  $An_{30}$ , where  $\alpha'$  is the same for both (001) and (010) cleavages. Grains were sought which showed sharp albite-law composition planes, to avoid those lying on stepped cleavages. Most cleavage flakes in samples sized 100–200 mesh appeared to have perfectly even cleavage surfaces, as discussed below under precision.

After setting  $X'$  to extinction, the wavelength was varied until the central illumination test indicated a match between crystal and liquid. The wavelength was then changed, first in one direction and then the other, till it was obvious that a match did not exist. If the limits of match, so defined, did not lie symmetrically about the proposed wavelength of match, further trials were made until a consensus about the match wavelength was reached. The final limits of match, in terms of wavelength, were then taken as half the mean distance to the limits of "obvious mismatch." These limits indirectly define the uncertainty attached to the refractive index or composition of a single grain. The wavelength of match, limits, and temperature were then recorded, and a new grain was sought. After sufficient data were collected, the mean wavelength and mean temperature were calculated, taking due account of the monochromator correction, and the results applied to the dispersion chart with its overlay of liquid dispersion slopes. If desired, composition range and statistical parameters can be read off at the same time. Use of the overlay with the dispersion chart is facilitated if data for a number of samples are accumulated on cards and then ordered by temperature to avoid unnecessary shifts of the overlay.

If the value of  $\alpha'$  rather than the  $An$  content is desired, this can be read by following the dispersion slope of the plagioclase to the  $D$  line.

#### SPEED AND PRECISION

In 1960 the writer determined by the dispersion method 124 samples of plagioclase in a  $5\frac{1}{2}$ -day working period, observing an average of 9 grains per sample. It is true that these were ideal samples, from fresh gabbroic rocks with little zoning, but the potential of the method emerges clearly nonetheless.

In order to test the hypothesis that errors may arise from grains not lying accurately on a cleavage, samples extracted from two homogeneous megacrysts of iridescent labradorite from anorthosite were examined. For the first of these, 13 grains were observed and wavelengths of match were determined within  $\pm 2$  nm for each grain. The results showed a range from  $An_{50.8}$  to  $An_{52.0}$ , a mean composition of  $An_{51.2}$  with a standard

deviation of 0.36 and a standard error of the mean of 0.1% An. Translated into terms of refractive index, the mean for  $\alpha'_{001}$  was 1.5557,  $s=0.00016$ , and standard error=0.00004. For the second sample, match wavelengths were found for 14 grains with an uncertainty of  $\pm 3$  nm. The composition range was An<sub>51.9</sub> to An<sub>53.4</sub>, mean 52.4,  $s=0.45$ , standard error=0.12% An. In terms of refractive index,  $\alpha'_{001}$  is 1.5563, with  $s=0.00026$  and the standard error=0.00007. Any method which can estimate plagioclase composition with a precision of tenths of percent An is not suffering badly from random errors such as might be induced by stepped cleavage. The data also suggest that it is possible with the dispersion method to distinguish differences in refractive index which are comparable to the error inherent in assigning an index to the liquid itself.

It should be emphasized that such low standard errors as quoted here will be achieved only in exceptional circumstances, and the magnitude of routine errors will have to be established by each laboratory for its particular quality of sample. Furthermore, it must be stressed that we speak here of precision, which is a necessary but not sufficient requirement for the accurate knowledge of a natural plagioclase composition.

#### DISCUSSION

There are some subtleties in the refractive index data of Smith which are not incorporated in the smoothed curves used in this study. The discontinuity in  $\alpha$  near An<sub>40</sub> is most notable among these, and direct comparisons of  $\alpha'_{001}$  with composition are not yet adequate to show the fine structure of a determinative curve in this region. Accordingly, there is some uncertainty at the <1% An level in determinations around An<sub>40</sub>, although this will be trivial for most petrographic purposes. The discontinuities found by Smith may, however, prove interesting with regard to plagioclase crystal chemistry and unmixing (see Laves and others, 1965, for example).

Because of its precision and statistical information, the dispersion method is a powerful tool in detailed studies of plagioclase composition and structural state. The writer (Morse, 1961 and in press) has been able to show real variations in structural state for identical plagioclase compositions within a single small volume of a layered intrusion by combining the dispersion method compositions with X-ray structural state. Similar studies on iridescent labradorite from the Nain anorthosite, Labrador, are in progress; these benefit greatly from the precision of relative composition of around 0.2% An with these homogeneous samples. Similarly, studies of local or regional composition variation within a given intrusion are enhanced by the dispersion method. The range of

zoning can be detected from the maximum and minimum An values found in immersion, and this is helpful in distinguishing adcumulate from orthocumulate rocks.

## REFERENCES

- CHAYES, FELIX (1952) Relation between composition and indices of refraction in natural plagioclase. *Amer. J. Sci.*, **Bowen Vol.**, 85-106.
- FAIRBAIRN, H. W. (1952) Notes on minimum deviation refractometry. *Amer. Mineral.* **37**, 37-47.
- LAVES, FRITZ, H.-U. NISSEN, AND W. BOLLMANN (1965) On schiller and submicroscopical lamellae of labradorite. *Naturwissenschaften* **52**, 427-28.
- MERWIN, H. E., AND E. S. LARSEN (1912) Mixtures of amorphous sulphur and selenium as immersion media for the determination of high refractive indices with the microscope. *Amer. J. Sci.* **34**, 42-47.
- MORSE, S. A. (1961) *The geology of the Kiglapait layered intrusion, coast of Labrador, Canada*. Ph.D. thesis, McGill University, Montreal.
- (in press) The Kiglapait layered intrusion, Labrador. *Geol. Soc. Amer. Mem.* **111**.
- SLEMMONS, D. B. (1962) Determination of volcanic and plutonic plagioclases using a three- or four-axis universal stage. *Geol. Soc. Amer. Spec. Pap.* **69**.
- SMITH, J. R. (1958) The optical properties of heated plagioclase. *Amer. Mineral.* **43**, 1179-1194.
- (1960) Optical properties of low-temperature plagioclase. In Hess, H. H., Stillwater igneous complex, Montana. *Geol. Soc. Amer. Mem.* **80**.
- SUENO, T. (1933) On the use of standard glass powders in refractive index determination. *Amer. Mineral.* **18**, 421-230.
- TSUBOI, SEITARÔ (1923) A dispersion method of determining the plagioclases in cleavage flakes. *Mineral. Mag.* **20**, 108-122.
- (1930) On the use of Hartmann's dispersion net in refractive index determinations with the microscope. *Geol. Soc. Tokyo. J.*, **37**, 39-45.
- (1934) A straight-line diagram for determining plagioclase by the dispersion method. *Jap. J. Geol. Geog.* **11**, 325-326.

*Manuscript received, March 13, 1967; accepted for publication, June 9, 1967.*