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Measurement of refractive index in thin section using dispersion staining and oil immersion techniques

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Abstract

With the aid of a 10× dispersion staining objective adapted for apertural and central screening, and a liquid-grain interface produced with a cutting tool, the index of refraction of a crystal in thin section can be measured directly. Accuracy of ±0.001 is attained.

Introduction

Petrographers are commonly faced with the necessity or desirability of obtaining the index of refraction (n) of a specific crystal in a thin section, yet with no way of doing so without removing the grain from the thin section, which can be a laborious task, or by making a mineral separation from the rock sample. In the latter case there is no assurance that the separated grains are identical with the specific one of interest in the thin section. The dispersion staining method, described and evaluated here, offers a relatively rapid technique for determining at least one index of refraction for a selected grain in thin section. The procedure is particularly useful, for example, for obtaining the n of a member of an isomorphous series, which commonly allows chemical characterization of the mineral.

Petrographers are familiar with the theory and methodology of index of refraction determination using dispersion colors (Bloss, 1967). If the n of a grain is close to a match with that of the oil in which it is immersed, a dispersion of white light is observed which produces color fringes around the mineral. Observation of these colors can lead to a reasonably precise determination of n.

A 10× dispersion staining objective (W. C. McCrone Inc., Chicago, Illinois) can be used as an aid in making this determination. In cases where the n of a grain and liquid match at one wavelength, but not at others, it is possible with the 10× dispersion staining objective to make the grain to appear to have colored borders. The color observed is indicative of the wavelength for which a match between n of the grain and liquid has been obtained (Grabar and Principe, 1963). Thus, dispersion staining aids markedly in the rapid identification of minerals in grain mounts (Laskowski, 1965). Apertural screening and central screening with a 10× dispersion staining objective enables one to determine nD of crystals, not only in grain mounts, but also in petrographic thin sections.

Our purpose here is to report an evaluation of the method, based on the determination of refractive indices of albite, labradorite, quartz, and microcline in thin section, and to describe the technique in sufficient detail to encourage its use.

Theory

The theory of the dispersion staining objective has been described (Cherkasov, 1960; Brown *et al.*, 1963; Grabar and Principe, 1963). A standard 10× objective is fitted with two types of removable screens placed in the rear focal plane of the objective.

Apertural screening

The aperture screen is a diaphragm with an open space about 2 mm in diameter. This diaphragm is placed in the rear focal plane of the 10× objective (Fig. 1). To adjust the system for proper illumination, the following conditions are met:

1. Nicols uncrossed

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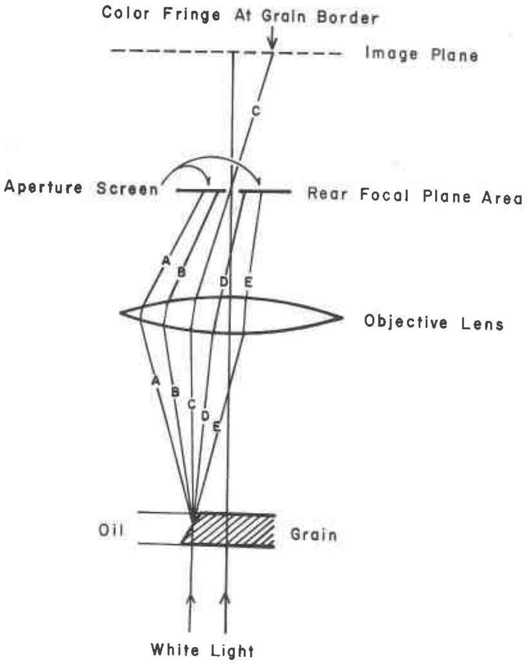

Fig. 1. Schematic diagram showing the effects of apertural screening on dispersed rays of white light as viewed in focus.

2. High-power condenser out
3. Light source at high intensity
4. Bertrand lens in
5. Aperture screen in
6. Substage diaphragm of the microscope closed so that the radius of light visible through the Bertrand lens is slightly smaller than the radius of, and located within, the aperture screen
7. Now take the Bertrand lens out
8. The system is now arranged for measurement with the aperture screen.

Figure 1 shows the system schematically. Parallel bundles of white light strike the liquid–grain boundary and are dispersed into various rays (A, B, C, D, E). Rays of wavelengths A, B, D, and E are deviated at the liquid–grain boundary, but wavelength C is not deviated. As light of wavelength C is not deviated, it is the wavelength or color of light for which the liquid and grain have the same refractive index. All the wavelengths (A, B, C, D, E) of dispersed light encounter the aperture screen as they pass through the rear focal plane of the objective. Only the wavelength which is not deviated at the liquid–grain boundary (wavelength C) is permitted to pass to the image plane. Thus the grain will appear to have colored borders, and the color indicates the wavelength for which the index of refraction of the liquid matches the index of refraction of the grain (Table 1).

Central screening

Central screening can be used to define further the results obtained in the apertural screening measurement. The central screen is an opaque dot (1–3 mm in diameter) which is mounted on a glass plate. This screen is also placed in the rear focal plane of the 10× objective (Fig. 2). To adjust the system for proper illumination, the following conditions are met:

1. Remove the aperture screen
2. Nicols uncrossed
3. High-power condenser out
4. Light intensity high
5. Bertrand lens in
6. Insert central screen so that the opaque dot is in the center of the crosshairs
7. Adjust the substage diaphragm of the microscope so that the radius of light visible through the Bertrand lens is equal to the radius of the opaque dot on the screen
8. Adjust the opaque dot so that it completely covers the radius of light created in step 7
9. Open the substage diaphragm ever so slightly to allow a faint rim of light to be barely visible around the periphery of the opaque dot
10. Now take the Bertrand lens out
11. The system is now arranged for measurement with the central screen.

Figure 2 shows the system schematically. Parallel bundles of white light strike the liquid–grain boundary and are dispersed into various rays (A, B, C, D, E). Wavelength C is not deviated at the liquid–grain boundary, thus wavelength C is the wavelength for which the index of refraction of the liquid and the index of refraction of the grain are equal. Wave-

Table 1. Colors observed in apertural screening and related wavelengths

Color	Wavelength of liquid-grain $\underline{n}$ match
Deep Violet to Black	Less than 4000 A°
Dark Blue violet	4000-4600 A°
Royal Blue	4600-4800 A°
Blue green	4800-5200 A°
Green	5200-5400 A°
Yellow green	5400-5700 A°
Yellow	5700-6000 A°
Yellow orange to Red orange	6000-6200 A°
Red orange to Red	6200-6500 A°
Red to Brown red	Greater than 6500 A°

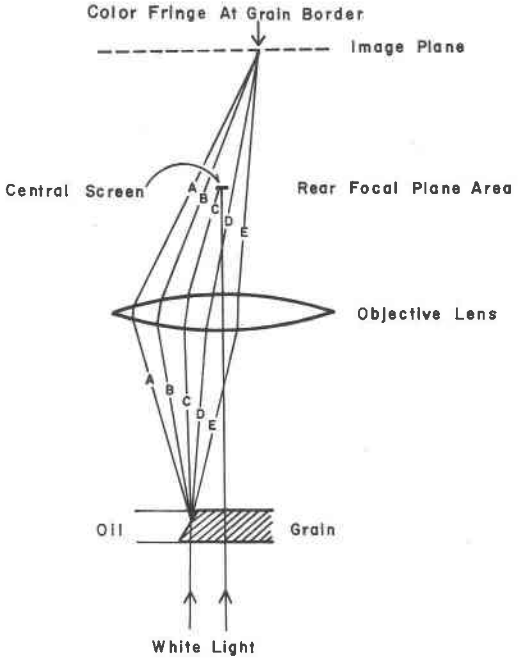

Fig. 2. Schematic diagram showing the effects of central screening on dispersed rays of white light as viewed in focus.

lengths A, B, D, and E are deviated at the liquid-grain boundary. As all of the wavelengths of dispersed light are collected and pass through the rear focal plane of the objective, they encounter the central screen. The central screen blocks out wavelength C and allows wavelengths A, B, D, and E to pass to the image plane. The grain borders of the crystal thus become illuminated and colored. When viewed in focus, the color at the grain border is related to the actual wavelength (as the complementary color to the wavelength) for which the index of refraction of the liquid and that of the grain are equal (Table 2). When de-focusing, two Becke lines are produced. The colors of these Becke lines are similarly related to the wavelength at which the liquid–grain index match is obtained (Table 2).

Table 2. Colors observed in central screening and related wavelengths

In focus	Becke lines	Wavelength of liquid-grain $\underline{n}$ match
Light Blue to White	Light Blue and White	Greater than 7000 A°
Greenish azure	Green	6500-7000 A°
Blue green	Blue and Green	6300-6500 A°
Royal Blue	Royal Blue	6100-6300 A°
Bluish violet	Blue and Red	5700-6100 A°
Crimson	Blue violet and Red orange	5200-5700 A°
Light Orange	Violet and Orange	5000-5200 A°
Golden yellow	Faint Violet and Gold	4400-5000 A°
Pale Yellow	Faint Yellow	Less than 4400 A°

Procedure for *n*D measurement in petrographic thin section

Measurement of an index of refraction of a specific mineral (in this case *n*D-beta of an albite will be measured) can be performed directly on the thin section. The following description of the determination of the *n*D for the beta index of plagioclase serves as an example:

1. It is necessary to work with a thin section without a coverslip. If the desired slide has a coverslip, carefully pry the coverslip off with a sharp razor. Use of safety glasses is advised.
2. Select the largest grain of a specific mineral species which is oriented for the particular index desired for measurement.
3. Place the thin section on the cutting apparatus (Fig. 3), and place the cutting apparatus on the microscope stage. Relocate the grain desired for measurement using a low-power objective.
4. Place the brass guide across the grain to be measured, and hold the guide firm with the magnets. Make sure the indentation on the brass guide is across the grain to be measured.
5. Take the cutting apparatus and attached slide

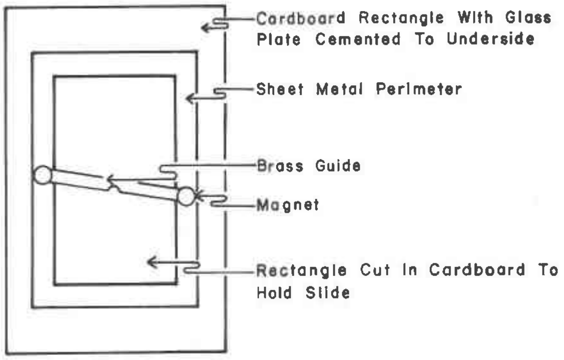

Fig. 3. Cutting apparatus. A guide which allows one to orient the cutting tool to a selected grain. A slide is placed in the central rectangle, and the assemblage is placed directly on the microscope stage. The slide is viewed with a low-power objective, and a grain for measurement is located. The brass guide is placed across the desired grain and held in place by two magnets. The entire assemblage is then removed from the stage of the microscope for cutting.

off the microscope stage and place on a flat table.

6. Apply three drops of acetone to the slide in the vicinity of the desired grain and rinse with water. Be careful not to disturb the brass guide.

7. Using a high-speed drilling tool fitted with a 1-inch-diameter carborundum disc, make a vertical cut along the brass guide across the grain to be measured. This cut must be executed in one downward motion and must penetrate through the rock section into the glass. The idea here is to create a trough or slit in the grain to be measured.

8. Remove the slide from the cutting apparatus and add one drop of acetone to the fresh cut with a small pipette. Allow this drop to evaporate partially, then wash the entire slide with water. Dry the slide.

9. Before proceeding, check to be sure that the cut produced is across the grain in question.

10. Place a drop of index liquid (Cargille liquids are preferred) on the cut, making sure that the temperature of the liquid is about 25°C. A liquid–grain boundary has now been established. Using successive mounts, find a liquid which produces discernable dispersion colors. Clean the cut on the slide with a drop of acetone each time liquids are changed. As the *n*D of a given liquid approaches the *n*D of the grain, dispersion colors will become visible at the cut-grain boundary in normal illumination with low intensity. Make sure the grain is in the desired optical orientation.

11. Insert the aperture screen and set the illumination as previously described.

12. Place the crosshairs at a point on the grain where color fringes are observed, and recheck the centering of the screen.

13. Record the color observed through the aperture screen, and refer to Table 1 for the wavelength for which the indices of refraction of the grain and liquid are equal. [The albite crystal described in Table 3 yielded green borders as viewed through the aperture screen when a liquid with *n*D = 1.532 at 25°C was used. This indicated (Table 1) a crystal–liquid index match at some wavelength between 5200 and 5400A.]

14. Remove the aperture screen and insert the central screen. Set the illumination for central screening as previously described. Perform step 14 without al-

Table 3. Results of dispersion staining measurements on known crystals

Material	$\underline{n}$	Type of mount	$\underline{n}$D liquid	Colors observed during screening (25°C) Apertural	Colors observed during screening (25°C) Central	λ of liquid-grain $\underline{n}$ match	$\underline{n}$D measured	Published $\underline{n}$D
Optical Glass	Iso.	Grain	1.508	Green	Violet+Orange	5200 A°	1.511	1.51. (Cargille)
			1.512	Yellow orange	Royal Blue	6100 A°		
Optical Glass	Iso.	Grain	1.608	Yellow green	Blue+Red	5700 A°	1.6095	1.61 (Cargille)
			1.612	Orange red	Blue+Green	6400 A°		
Ammonium Chloride	Iso.	Grain	1.632	Green	Violet+Orange	5200 A°	1.6396	1.6392 (Cargille)
			1.636	Yellow green	Bl.viol.+Red orng.	5550 A°		
Sodium Bromate	Iso.	Grain	1.608	Blue green	Violet+Orange	5100 A°	1.6167	1.6165 (Cargille)
			1.612	Green-Yel. grn.	Bl.viol.+Red orng.	5400 A°		
Low Quartz	ε	Grain	1.544	Royal Blue	Gold+Violet	4700 A°	1.5525	1.553 (Bloss, 1967)
			1.552	Green	Bl.viol.+Red orng.	5300 A°		
Low Quartz	ω	Grain	1.540	Green	Bl.viol+Red orng.	5300 A°	1.5446	1.544 (Bloss, 1967)
			1.544	Yellow	Blue+Red	5850 A°		
Low Quartz	ω	Thin Section	1.540	Blue green	Bl.viol.+Red orng.	5200 A°	1.5449	1.544
			1.544	Yellow green	Blue+Red	5700 A°		
Low Quartz	ε	Thin Section	1.548	Blue green	Bl.viol.+Red orng.	5200 A°	1.5539	1.553
			1.556	Yellow orange	Royal Blue	6150 A°		
Albite	β	Grain	1.532	Green	Violet+Orange	5200 A°	1.5352	1.531-1.537 (Kerr, 1959)
			1.536	Red orange	Blue	6200 A°		
Albite	β	Thin Section	1.532	Green	Violet+Orange	5200 A°	1.5355	1.531-1.537
			1.536	Orange yellow	Blue+Red	6050 A°		
Labradorite	β	Grain	1.556	Green	Violet+Orange	5200 A°	1.5592	1.558-1.569 (Kerr, 1959)
			1.560	Orange red	Blue	6200 A°		
Labradorite	β	Thin Section	1.556	Green-Blue grn.	Violet+Orange	5200 A°	1.5594	1.558-1.569
			1.560	Yellow orange	Blue	6150 A°		
High Ca Labradorite	β	Grain	1.564	Blue green	Violet+Orange	5100 A°	1.5677	1.558-1.569
			1.568	Yellow orange	Blue+Red	6050 A°		
High Ca Labradorite	β	Thin Section	1.564	Green	Violet+Orange	5200 A°	1.5679	1.558-1.569
			1.568	Yellow-Lt.Orange	Blue+Red	6000 A°		
Microcline	β	Thin Section	1.520	Yellow green	Bl.viol.+Red orng.	5550 A°	1.5226	1.522-1.526 (Bloss, 1967)
			1.524	Orange	Blue+Red	6100 A°		

tering the position of the slide on the microscope stage, or changing the liquid.

15. Observe the colors produced by central screening, and refer to Table 2 for the wavelength at which the indices of refraction of the grain and liquid are equal. This wavelength or range of possible wavelengths should coincide with the results obtained from apertural screening. [The albite crystal described in Table 3 yielded violet and orange color fringes viewed through the central screen when a liquid with *n*D = 1.532 at 25°C was used. This indicated (Table 2) a crystal–liquid index match at some wavelength between 5000 and 5200A.]

16. For the albite example, steps 13 and 15 yielded a crystal–liquid index match at some wavelength within the range of 5200–5400A (step 13) and within the range of 5000–5200A (step 15). These two wavelength ranges overlap at 5200A, thus 5200A was chosen as the wavelength for which the index of refraction of the crystal and the liquid (in this case *n*D = 1.532 at 25°C) are equal. In some cases the results attained from Tables 1 and 2 will overlap over a range of wavelengths. If this occurs, choose a midpoint within the overlapping wavelengths as the wavelength for which the *n* of the crystal and the liquid are equal.

17. Plot the liquid (at 25°C) on a graph of *n vs.* wavelength (Fig. 4). Plot the match wavelength, attained in step 16, on the liquid curve. For the albite example, the match wavelength attained in step 16 (5200A) was plotted at point *A* on Figure 4.

18. Clean the slide with a drop of acetone, rinse in water, and dry.

19. If a wavelength match of 5400A or less was attained above, choose a higher index liquid for the next measurement. If a wavelength match of greater than 5400A was attained above, choose a lower index liquid for the next measurement.

20. Repeat steps 11–18 with the new index liquid; be sure the crystal is in the same orientation as before. (With liquid *n*D = 1.536 at 25°C, 6050A was determined to be the wavelength for which the index of refraction of the albite in Table 3, and liquid *n*D = 1.536 at 25°C, are equal. This match wavelength is plotted at point *B* on Figure 4.)

21. Draw a line through the two match points as in Figure 4. This line is an accurate representation of the change in index of refraction of the crystal in the orientation present with respect to a change in wavelength. The point on the Y axis where the resulting line intersects the sodium D line is the *n*D of the grain, in the orientation tested (point *C* on Figure 4).

Discussion

Table 3 shows results of dispersion measurements made on various samples. All measurements were conducted at about 25°C. Indices of refraction of ammonium chloride, sodium bromate, and two optical glasses (all Cargille standards) were measured using grain mounts and the dispersion staining method previously described. Accuracies of equal to or better than ±0.001 were obtained in all cases. These results are comparable with accuracies obtained by Cherkasov (1960) and Grabar (1963).

The index of refraction for sodium D light of the epsilon ray and *n*D-omega for low quartz, *n*D-beta of albite, *n*D-beta of microcline, *n*D-beta of labradorite, and *n*D-beta of high-calcium labradorite were measured directly from petrographic thin sections. In each case, a specific grain in a specific orientation was chosen for measurement. The index of refraction for sodium D light of the omega ray and *n*D-epsilon values of 1.5449 and 1.5539 measured for low quartz compare favorably with the values given by Bloss (1967). The index of refraction for sodium D light of the beta ray for albite was measured in thin section to be 1.5355. A grain mount of the same sample gave an *n*D-beta of 1.5352. These values fall within a range of values (Kerr, 1959) for the albite field. The indices of refraction for sodium D light of the beta rays for low-calcium labradorite and high-calcium labradorite were measured in thin section to be 1.5594 and 1.5679 respectively. Grain mounts of the same samples gave *n*D-betas of 1.5592 and 1.5677 respectively. Kerr (1959) placed the labradorite field at *n*D-beta = 1.558–1.569. The index of refraction for sodium D light of the beta ray of microcline was determined to be 1.5226 from a thin section mount. Bloss (1967) placed the microcline field at *n*D-beta = 1.522 to 1.526.

Composition is related to *n*D-beta for the plagioclase solid solution series (Chayes, 1952). Using his graph for *n*D-beta *vs.* plagioclase composition, the albite, labradorite, and high-calcium labradorite in Table 3 are estimated to have compositions of 8 percent anorthite, 53 percent anorthite, and 68 percent anorthite respectively.

In cases discussed above, liquid–grain boundaries were created directly on thin section slides. Color fringes were produced. To interpret these color fringes (some of them faint) produced in thin section, the dispersion staining method is preferred over other standard methods because of the accuracy attained (±0.001), quality of colors produced (a color fringe which appears very faint in regular light is generally

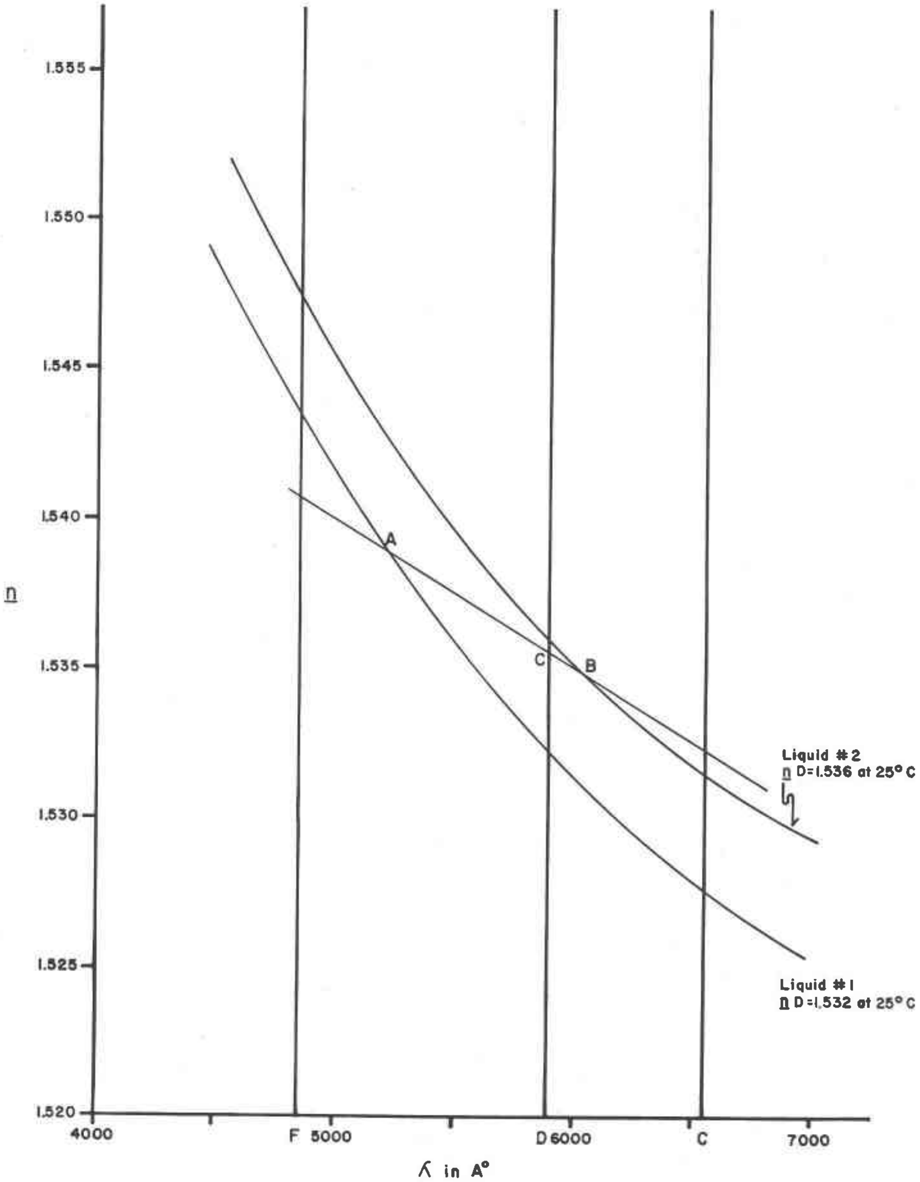

Fig. 4. Determination of *n*D of an albite. Liquids 1 and 2 are plotted, *n vs.* wavelength, according to values for *n*F, *n*D, and *n*C given for each liquid. Line *ACB* represents the change in index of refraction of the albite with respect to a change in wavelength. The crystal is oriented to measure the beta index. Point *C* is the *n*D-beta (1.5355) of the albite crystal.

quite clear when the dispersion staining lens is employed), and ease of interpretation of the colors produced.

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