

THE DARK-FIELD COLOR IMMERSION METHOD

NELSON B. DODGE, *Bausch and Lomb Optical Co.,
Rochester, New York.*

ABSTRACT

With dark-field illumination, color criteria provide an alternative to the usual methods for comparing index of crushed grains with immersion media. Since organic immersion liquids have steeper dispersion curves than inorganic solids, spectrum colors are produced from white light by refraction at interfaces of grains and liquid. With ordinary illumination, when the dispersion curves intersect in the yellow, the oblique illumination test produces colored grain borders as explained by F. E. Wright and others. Dark-field colors appear in bright contrast to a dark background, affect grains everywhere in the microscope field, and require no changing of focus.

Most microscopes can be adapted for dark-field illumination, which requires a hollow cone of light from the condenser, focused on the preparation, having a greater aperture than the objective. The field of view is dark, except where refraction and reflection in the preparation send light to the eye. When solid and liquid indices differ widely, grains appear white against a dark background. When indices differ by a few units in the second decimal place, or less, grains are yellow if higher and blue if lower in index than liquid. At equality of sodium index, $\pm .001$, grains appear purplish blue with a scattering of deep red borders.

The colors can be explained as a result of the Christiansen effect. They are due to the subtraction of the lost transmitted wavelengths from white light.

INTRODUCTION

The dark-field color immersion method may be used for obtaining refractive indices of crushed minerals. The essential feature of this method is the employment of low-grade dark-field illumination with white light to produce colors indicative of relative sodium index of grains and immersion liquid. Many possibilities and difficulties remain to be explored, but it is evident that in many cases this method may be used with advantage to supplement the standard procedures in current use, namely the Becke line—central illumination method and the oblique illumination method (2, 4).

In both of these standard methods, when used with white light, colors are seen, due to the Christiansen effect, when the refractive indices of mineral grains and surrounding immersion liquid are near equality. Most familiar is probably the appearance of blue and orange-red on opposite sides of grains in the oblique illumination method. Less used but often seen are the differences in color between borders and interiors with central illumination. These phenomena have been known since the 1870's, when both the current methods were discovered. F. E. Wright in 1911 summarized earlier work and evaluated the methods based on color from white light due to relative dispersion, finding that an accuracy of better than $\pm .002$ is not to be expected of them (5). A serious disadvantage of

such methods is that colors appear over a comparatively short range of index difference between solid and liquid.

This appearance of color in normally colorless mineral fragments results from the difference in dispersion of minerals and the liquids used for immersion media. In the great majority of cases, organic immersion liquids have steeper dispersion curves than inorganic solids lying in the same general index range. Differential refraction of light crossing liquid-solid interfaces at other than right angles produces spectral colors. The range of index difference over which colors appear varies with the amount by which the dispersion of the liquid exceeds that of the solid material. These same fundamental causes are believed to underlie the colors observed in dark-field illumination. In dark-field, however, the colors are much more brilliant, and are useful as criteria over a greater range of index difference between liquid and solid.

The use of dark-field colors to determine refractive index was suggested by Mr. G. C. Crossmon of the Bausch and Lomb Chemical Laboratory (1).

DARK-FIELD ILLUMINATION

Dark-field conditions may be produced by placing an opaque central

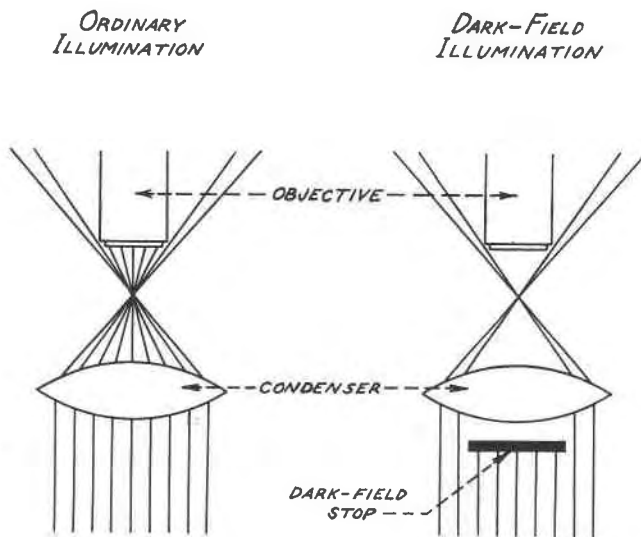


FIG. 1

stop below the substage condenser. This results in a hollow cone of light which is focused on the object slide. Biological microscopes are usually

designed to permit dark-field work. Petrographic microscopes ordinarily require some adaptation, as explained in a later section. The essential condition is that the inside angle of the cone of light must slightly exceed the angular aperture of the objective. With nothing on the stage, all light is lost, and the field of view is dark (Fig. 1).

When a preparation of grains in an immersion liquid is placed on the stage, the grains reflect and refract light into the aperture of the objective. This causes the grains to appear luminous against a dark background. If solid and liquid are far apart in index the grains appear white. When the sodium indices of solid and liquid differ by a few units in the second decimal place, or less, the grains appear colored.

COLOR PHENOMENA IN DARK-FIELD

With reference to crushed grains in immersion fluids, Fig. 2, top row, shows dispersion curves for solid and liquid. Three cases are shown:

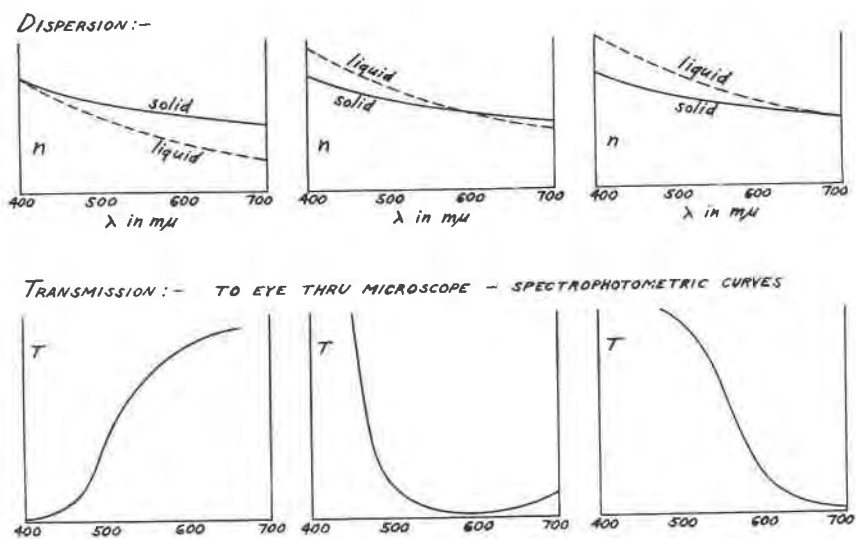


FIG. 2

On the left, solid is greater than liquid in index of refraction for sodium light ($589.3\text{ m}\mu$); in the middle they are equal; and on the right the liquid is greater in n_D . The corresponding transmission curves (Fig. 2, bottom row) were determined by spectrophotometric measurement of the colored light transmitted through the microscope, using crushed glass in three different liquids. The transmission scale is relative and differs from one

figure to another. These spectrophotometric curves were determined by Mr. W. G. Kirchgessner of this Laboratory, assisted by the writer.

With intersection of the dispersion curves at the violet end of the visible spectrum, the mineral will appear yellow against a dark background. The yellow color is at first faint, mixed with much white; with increasing index of the liquid the color intensifies. The origin of the yellow color is as follows: Where the dispersion curves intersect, liquid and solid are equal in index; light of that wave-length is not deviated by the grains, so it passes straight through the preparation, misses the objective, and is lost. On either side of this wave-length, where the dispersion curves are close together, light is not deviated enough to enter the objective. Toward the red end, the separation of curves is greater, and light of these longer wave-lengths is refracted enough to reach the eye through the objective (Fig. 2, top, left). Thus, in terms of the transmission curve (Fig. 2, bottom, left), a band of some width with center at the intersection is subtracted from the original white light. This results in a transmission to the eye which would register as yellow.

If the liquid index is raised further, the intersection of dispersion curves moves to the right, and with it goes the band of subtracted wave-lengths. The result of this changing subtraction is that first some violet comes in along with the yellow; the yellow changes gradually to orange and then red; while the violet becomes more bluish and relatively more intense. Red and blue-violet are seen about equally when the liquid is a few points in the third decimal place below the solid in n_D , when using the more common immersion fluids.

Figure 2, middle, top and bottom, shows the situation when grains and liquid are equal in index for sodium light. Grains will now appear mostly blue, or bluish violet, with scattered touches of red here and there. Many grains will show no red.

At the right of Fig. 2 the dispersion curves intersect in the red. The transmission curve indicates a sensation of blue. Blue is the only color shown when solid n_D is less than liquid n_D .

Some of the color criteria are illustrated on the accompanying color plate, from Kodachrome.

The color sequence as described holds good for various mixtures of such organic liquids as Government oil, butyl carbitol, α -chlornaphthalene (or Halowax oil), α -bromnaphthalene, and methylene iodide. If cinnamic aldehyde, having an unusually high relative dispersion, is used as one component, extremely brilliant colors are produced and they extend over a much longer range of index difference, but the detection of the match point for sodium index is rendered less exact. In choosing liquids one must keep in mind that accuracy of match will vary inversely

with total range of criteria. Solids tested include corundum, quartz, halite, topaz, sucrose, and a variety of optical glasses of difference indices and dispersions.*

Media for making permanent mounts such as balsam, Aroclor 4465, and naphthalene formaldehyde resin also show the characteristic colors with minerals of appropriate index. However, such media usually require purification as they are likely to be full of extraneous suspended matter of widely differing indices which becomes all too visible in dark-field illumination.

The fact that a match of liquid and solid indices for sodium light is

TABLE 1. SUMMARY OF COLOR CRITERIA FOR RELATIVE INDEX (n_D) OF GRAINS AND LIQUID IN DARK-FIELD

<i>Grains Greater</i>	<i>Grains = Liquid</i>	<i>Liquid Greater</i>
Yellow	Blue or blue-violet with	Blue
Yellow and violet	very little red or none.	
Orange and violet		
Red and blue-violet		

indicated by the vanishing point of the red color and the transition of violet to blue has so far not been explained as anything but a fortunate coincidence resulting from the constancy of the relative shapes of the solid and liquid dispersion curves.

EQUIPMENT

The colors described above were obtained with a research type petrographic microscope using a standard $10\times$ (16 mm.) 0.25 N.A. strain-free achromatic objective, and a standard 0.28 N.A. condenser (upper element swung out). The opaque stop used is 14 mm. in diameter and mounted so as to swing in and out readily between the polarizer and the bottom of the condenser. It is quickly and easily centered with one hand; centering is observed by inserting the Bertrand lens. The best illumination is obtained with the condenser slightly lowered. Owing to the small diameter of the standard calcite polarizer, light was not passed around the stop to make the hollow cone; this was remedied by replacing the calcite with polaroid mounted in an interchangeable tube (a standard item from a student model instrument can be used). There seems no objection to having polaroid in the substage and calcite in the analyzer.

* For the highly unusual case in which the dispersion of the solid is greater than that of the liquid of the same index, the color sequence is reversed. The only such case so far actually observed is sylvite in a mixture of Government oil and Nevillite oil.

The same set-up is quite satisfactory for the 6× (22.7 mm.) and the 4× (32 mm.) objectives.

A simpler arrangement, also for use with the 10×, 6×, and 4× objectives, is to use a standard 13 mm. dark-field stop, with a condenser N.A. of 0.28. For higher powers, such as the 20× and 45× objectives, the standard petrographic type condenser may be too small in diameter to pass light around the larger opaque stop which is needed. In such cases a different condenser can be substituted.

Directions for setting up dark-field illumination by simple modifications which the user can perform himself without instrument machine shop facilities are so specific for different makes and models of microscopes that it is suggested that the manufacturer be consulted for detailed instructions. These arrangements need not interfere with a quick return to ordinary bright-field illumination whenever desired. Ordinarily only minor items of additional equipment are needed.

When no petrographic microscope is available a great deal of useful work may be done with a biological microscope (1).

Strong illumination is needed for dark-field, such as can be obtained from a 100-watt lamp. Suitable filters are needed to cut down glare and to reduce excess of longer wave-length light from the tungsten filament. The above-described colors were obtained while using a daylight blue glass, polished on both sides. A neutral ground glass or a second daylight blue may be added to regulate the light. Obviously the exact nature of the colors will be affected by differences in filters used. A condensing lens on the lamp was focused to produce nearly parallel light. A diaphragm on the lamp is useful to help in darkening the background.

When working with the 4 mm. objective the illumination becomes decidedly more critical. Satisfactory results will be obtained by using the 6-volt 108-watt ribbon filament lamp, with a Corning Daylite blue filter,

- Upper left:* Sample of commercial talc for use as filler. Talc (blue), lower index, and tremolite (yellow), higher index, in liquid having $n_D=1.588$. White grains: Unidentified impurity.
- Upper right:* Crushed topaz (white and yellow), higher index, and quartz (blue), lower index, in liquid having $n_D=1.556$.
- Lower left:* Crushed topaz in pure cinnamaldehyde ($n_D=1.619$). Grains equal to or greater than liquid in index, depending on their orientation with respect to polarizer.
- Lower right:* Crushed quartz (yellow) and chalcedony (blue-violet with red) in liquid having $n_D=1.536$. n_D of chalcedony = 1.539.

All 100×, using petrographic microscope with polaroid in substage. 14 mm. dark-field stop placed just below condenser of N.A.=0.28. Liquids except lower left, are mixtures of α -chlornaphthalene and butyl carbitol.



polished on both sides (no ground glass). Careful adjustment of the illuminator, the mirror, centering of the dark-field stop, and position of the substage makes a great difference in the intensity of the colors produced.

Scrupulously clean slides and cover glasses, free from scratches, cracks, or other defects must be used, and the immersion liquids should not contain suspended matter, as all such things show up in brilliant white in dark-field. In addition to obscuring the view of grains under observation, they scatter white light, reducing contrast by lightening the background. Tiny spots on the cover glass, being out of focus, blur out into larger white circular patches of light.

ACCURACY

The dark-field color method with white light has been checked many times against the Becke line—central illumination method with the sodium arc, both with and without a temperature-controlled stage cell. An accuracy of $\pm .001$ may be attained with the dark-field method under favorable conditions, such as by using a set of liquids with an index interval of .002 in an air-conditioned room or where temperature changes are slow enough that correction curves can safely be applied. With an index interval between liquids in the set of .004 or .005, the accuracy would about $\pm .002$. With index variation methods involving changing of temperature or varying of composition of liquid, the dark-field method can be made practically exact in the third decimal place.

With monochromatic light, the Becke line—central illumination method, the oblique illumination method, and the double diaphragm method* of Saylor (3) (the method of two-fold oblique illumination of Wright, 6) are all inherently more exact than the dark-field color method. Therefore it is not suggested as a substitute for them in such fundamental studies as the determination of the indices of the end-members of systems, or other analyzed materials where the highest accuracy is desired. However, for most ordinary work, such as identification, the dark-field method can be used quite safely.

For materials of high birefringence, the dark-field method will probably suffer a loss in accuracy.** This has not been investigated, but seems to follow inescapably from the calculations of F. E. Wright (7) and the experimental work of C. P. Saylor (3), on other methods involving oblique incidence of light. Due to the symmetrical character of the illumination in the present case, it is possible that this error may be somewhat less

* Discovered by F. E. Wright, this method was improved by C. P. Saylor who placed the upper diaphragm just above the upper element of the objective.

** As pointed out by Dr. Horace Winchell and Dr. Chester B. Slawson during discussion of this paper.

than in those methods involving unilateral oblique illumination. In any event, from Saylor's work it seems apparent that the error is relatively small with low aperture objectives (32 mm. and 16 mm.).

SPECIAL APPLICATIONS

One of the most obvious and striking uses of the dark-field color method is for detecting impurities in various powdered materials. By selecting an appropriate immersion liquid the substance to be checked can be shown in color, while impurities will appear white or a different color. The impurities will almost always contrast strongly with the desired material, so that a glance at a field gives an immediate estimate of the situation, and grain counts and measurements are facilitated.

When grains are heavily loaded with inclusions, the outer Becke line may be obscured by many internal Becke lines due to the inclusions. Even though inclusions are more conspicuous and more clearly observed in dark-field, the color criteria for the host mineral are unaffected. The inclusions are seen in brilliant white in colored grains.

If routine testing work is to be done by semi-skilled technicians this method has obvious advantages.

For photographic record purposes this method, with color photography, is not subject to the possible confusion of other methods, such as uncertainty as to whether a Becke line was photographed above or below focus, or whether the proper orientation was maintained with oblique illumination.

For very fine-grained material, requiring high-power objectives, dark-field illumination permits more reliable observations than ordinary illumination, as pointed out by F. E. Wright (8). In such cases, more accurate determinations may be made by half-shadowing the field, using monochromatic light, as advised by Wright (9), but sufficient accuracy for identification work may be had by observing the dark-field colors with white light, as proposed by Crossmon (1) and in the present paper.

SUMMARY

A microscopic immersion method for refractive index, based on dark-field illumination, has been presented in the hope that petrographers may find it useful. Some special applications in which it affords advantages have been pointed out. Necessary optical conditions have been described; these are subject to improvement and simplification particularly for high power objectives. Accuracy about equal to that of methods in current use is attainable for most work. The color criteria are so striking and clear cut that this method is really a pleasure to use.

REFERENCES

1. CROSSMON, G. C., (a) Optical staining of tissue: *Jour. Opt. Soc. Am.*, **38**, 417 (1948).
(b) Microscopical distinction of corundum among its natural and artificial associates: Analytical Chemistry, in press.
2. LARSEN, E. S., AND BERMAN, H., The microscopic determination of the nonopaque minerals, 2nd Ed.: *U. S. Geol. Surv., Bull.* **848** (1934).
3. SAYLOR, C. P., Accuracy of microscopical methods for determining refractive index by immersion: *J. Research NBS*, **15**, 277-294 (1935).
4. WRIGHT, F. E., The methods of petrographic-microscopic research: *Carnegie Institution of Washington, Publication No.* **158**, 92-98 (1911).
5. WRIGHT, F. E., *Op. cit.*, 85-87 (1911).
6. WRIGHT, F. E., The determination of the relative refringence of mineral grains under the petrographic microscope: *Jour. Wash. Ac. Sc.*, **4**, 389-392 (1914).
7. WRIGHT, F. E., The accurate measurement of the refractive indices of minute crystal grains under the petrographic microscope: *Jour. Wash. Ac. Sc.*, **5**, 101-107 (1915).
8. WRIGHT, F. E., The methods of petrographic-microscopic research: *Carnegie Institution of Washington, Publication No.* **158**, 53, 94-95 (1911).
9. WRIGHT, F. E., *Op. cit.*, 87 (1911).