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In a previous paper the writer described an immersion procedure for refractive index determinations on glass fragments, in which dispersion effects were utilized as a guide to a proper end-point adjustment.[1] Specifically, a procedure was described in which advantage was taken of the so-called Christiansen effect, wherein dispersion differences between solid and liquid result in variable colored halos as the Nd of the immersion fluid approaches that of the solid specimen.

The "thermal dispersion" of a substance may be defined as the change in value of Nd for a given change in temperature. In the usual organic immersion liquids, this value is approximately 0.0005 per degree Centigrade over the temperature ranges generally employed. It is regarded as of negligible value in glass and mineral fragments.

Chromatic dispersion represents the difference between the refractive indices of a given substance for light of different wave lengths. The value may be expressed in various ways, but is most often defined as the numerical difference between the refractive index for the blue line of 486 mμ (Nf) and the red line of 656 mμ (Nc). The immersion liquids usually used have an Nf–Nc value which exceeds that of glass fragments commonly encountered. Thus, a typical immersion fluid might have a dispersion value of 0.0175 as compared with a glass fragment at perhaps 0.009.

The value for the chromatic dispersion of a material depends upon temperature and the nature of the substance. In the absence of special equipment it is not a simple matter to accurately determine the dispersion of a substance; it is still more difficult to determine the change in value with a change in temperature. Available data indicate, however, that the change is in the neighborhood of 1×10^{-5} per degree Centigrade in the liquids employed. In some liquids the dispersion increases with a rise in temperature (although Nd, Nf and Nc will decrease), whereas in others it decreases with a temperature rise. Figures available for glass samples show an increase in Nf–Nc for a rise in temperature, the change in value per degree being apparently less than that of immersion liquids utilized.

So far as the simpler Nd determinations are concerned, in experiments conducted thus far no anomalous result has been obtained which could be attributed to a specific dispersion value or characteristic of the materials employed. As stated previously,

however, color halos will vary slightly between specimens at true Nd end points, presumably depending upon the dispersion ratio of liquid and glass, and an interpretation of color alone will not permit recognition of the Nd end point with as great a precision as will the Becke line in sodium light.

Although temperature changes will affect the color halo (due to changes in dispersion within the system), tests indicate that work performed at "room temperature" in a non-temperature-controlled cell is as satisfactory as that performed under constant temperature conditions. A temperature variation of one degree Centigrade will cause a change in Nd of the immersion liquid of approximately 0.0005, with a correspondingly smaller modification in the dispersion.

It would be convenient to have a high Nd liquid and a low Nd liquid, each with the same dispersion, for use as immersion liquids, but lacking that it is necessary to select the liquids primarily on the basis of Nd value. Accordingly, the dispersion values of suitable liquids are found to be at variance. Thus, in any mixture we find that the final dispersion of the liquid system depends far more upon the relative volumes of the two fluids employed than upon any temperature variation likely to be introduced. It is generally this variation (in conjunction with the dispersion of the glass) rather than any temperature change, which accounts for the noticeable color differences of "end-point halos." The successful application of the color halo method to date, however, indicates that even though the relative liquid-glass dispersion ratios vary over quite a range, colors can give a close approximation of the true Nd end point.

With the method previously described, it is not possible to alter the Nd of the immersion fluid without altering the dispersion at the same time. Nor can the dispersion be modified without changing the Nd value. Such changes may be made, however, by using more than two liquids, or by utilizing temperature variation techniques which will require a volume mix modification even with only two liquids.

Although a number of temperature-variation devices have been described in the literature on immersion techniques, it appears that they are not too generally utilized in criminalistics laboratories. Since preparation of the earlier paper on this subject, the writer has constructed a unit for this work which may be of interest. It is designed for use in connection with the procedure previously described, and will be found a most useful accessory to anyone doing refractive index work by that (or any other) method. A description of this device will be found at the end of the present article.

In working with the temperature-control unit it was found that at warmer temperatures, nitrobenzene was objectionable as an immersion fluid due to its odor and toxicity. Accordingly, monobromobenzene was substituted. The latter liquid has a somewhat lower dispersion than nitrobenzene, but a slightly higher Nd value, and has proved equally suitable.

The device has permitted use of the immersion liquid at temperatures ranging from 18°C. to around 45°C. The most immediate practical use for the unit has been in permitting the rapid adjustment of a liquid to a proper end point, or for varying it within the "color range" of the glass. Since the Nd of the liquid changes at the rate of approximately 0.0005 per degree, a 10-degree change in temperature will cause the glass to cover a nearly complete range of color changes, permitting a closer observa-

tion and appreciation of those changes than is possible with volume mix adjustment. While the temperature changes are being made over a range, it has been found more convenient not to stir the cell contents than to do so, whereby uninterrupted observations may be made. The fluid will be seen to flow of its own accord due to thermal variations, but no unequal change within the area of the central cell depression has been observed. Mixing may be advisable from time to time if a constant temperature is maintained.

Experiments with this device indicate that glass samples varying by 0.001 in Nd may be distinguished without difficulty by the Becke line in sodium light, and that differences of perhaps half this value should be detectable. This has been concluded as a result of temperature variation techniques wherein fragments of two glasses, known to vary by 0.001, have been compared directly, and wherein the thermal dispersion of the liquid is utilized to verify the magnitude of that difference and to measure the change in Nd necessary to cause a change in the Becke line. If one records the temperature at which the Becke line just finally disappears at all points on the fragment, and then the temperature at which it first reappears at any point under the conditions of magnification and illumination previously described, it is noted that approximately two degrees are required to effect this change. Thus, the Becke line is actually invisible at all points for an Nd variation of approximately 0.001 in the liquid. It appears safe to conclude that if the Becke line (sodium) appears to move out on an up-focus with one specimen, and in at the same time on another specimen, their Nd value differs by at least 0.001. Should one specimen be completely invisible at which time the other is either just becoming invisible (or just becoming visible), their difference would be perhaps half of that.

Since the particle is "invisible" for a range in Nd of approximately 0.001, it is obvious that for maximum accuracy in any absolute determination, all readings of liquid values should be made at a pre-selected position in the phase if consistent work is to be done. Although thick and irregular shapes may cause the Becke line to move in the "wrong" direction at times, observation shows that even a relatively thick and "chunky" particle can be made to disappear completely.

As to the color of halos during these experiments, as obtained with white light, it appears that color changes obtained in a given glass over a slight temperature range are not always sufficient to be recognizable with certainty—realizing that one has only a mental recollection of the color at the former temperature. At the same time, glass specimens known to be separated by only 0.001 to 0.003 in Nd have generally been clearly distinguishable by color, when examined in the same liquid.

In making color comparisons, it is important that observations be made on areas or edges of comparable thickness and shape—preferably medium thin edges. It has been noted also that of two fragments from a single source, where one fragment is quite thick and the other quite thin, color values and intensities may appear to vary noticeably throughout a range, yet at the exact end point will be effectively identical.

When utilizing only two basic liquids, with temperature variation, the dispersion of the liquid at 43 degrees will differ from the dispersion at 18 degrees. At the former temperature one would have a greater proportion of (higher dispersion) monobromobenzene to yield the same Nd as at the lower temperature wherein the volume of (lower dispersion) xylene would be relatively greater. For this reason, end-point
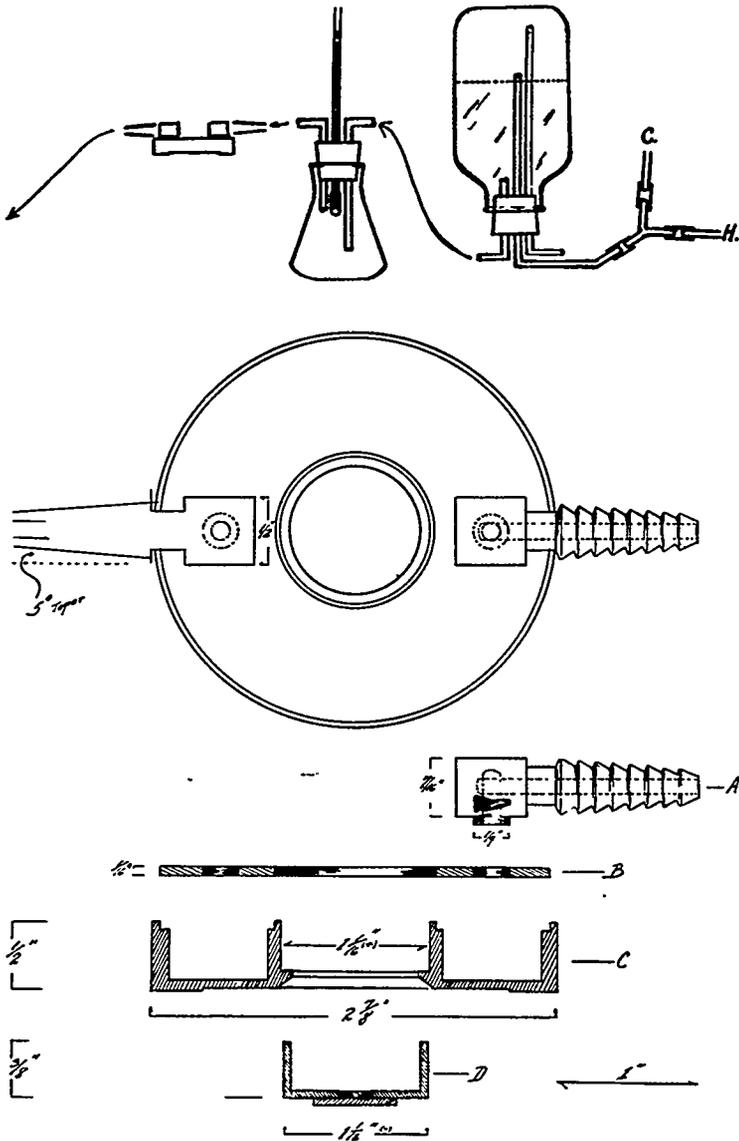
Figure 1

colors will vary somewhat depending upon temperature as it affects relative volumes and liquid-glass dispersions. At warmer temperatures an end point is likely to acquire a somewhat bluer halo than at colder settings.

Although the use of this temperature control cell has resulted in no greater speed or accuracy than that of the previous method, it has suggested a number of experimental projects, and facilitated work with glass fragments. Conclusions formed as a result of its application remain to be confirmed, in part at least, but are offered here for the benefit of those who may wish to do something along similar lines.

The Temperature-Control Device

The design of this unit necessitated a slight change in the construction of the glass immersion cell itself—both of which are illustrated in the accompanying sketch (Figure 1).

The cooling unit is essentially a hollow ring with water inlet and outlet at the top. Construction and dimensions are illustrated in the sketch. "A" represents a hose connection, "B" is the top plate, "C" is the base, a central portion of which is hollow clear through, and provided with a ledge upon which rests the glass cell. The device is made of brass, and soldered together to produce a leak-proof jacket.
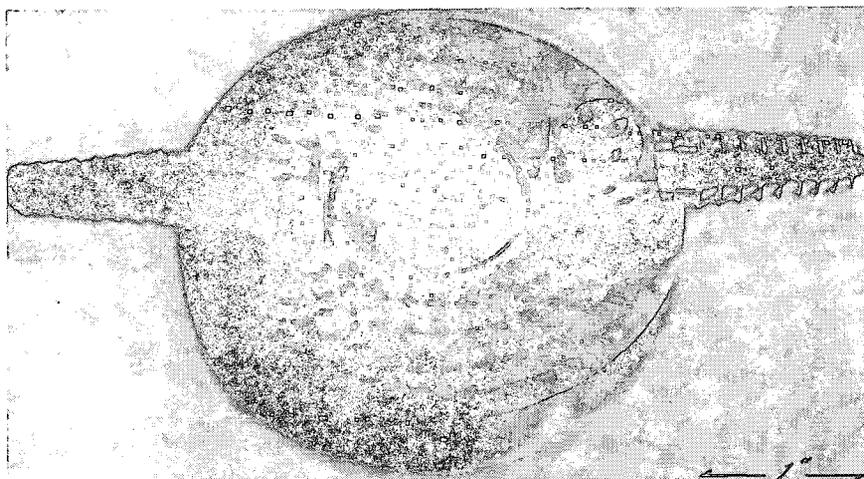

Figure 2

The glass cell (D) in this case is similar to the one described earlier, but is made from a shell vial of such size as will fit easily into the central opening of the cooling unit. A ¼-inch length of the bottom of a shell vial is cut off by means of diamond scoring and the use of a hot wire tubing cutter. A ¼-inch hole is drilled through the bottom of the vial, after which a second bottom plate is cemented in place as shown in the diagram.[2]

[2] Before drilling the hole the bottom of the vial should be cemented to a solid surface with pitch, dental compound or some similar material to prevent undue chipping of the edges of the hole at the "break through." The hole is best drilled with alumina abrasive in water or glycerine, using a ¼ inch brass tube on a drill press. The cell should be kept nearly full of water for cooling purposes. The "drill" should turn at about the rate used for drilling steel, and it should be lifted and lowered frequently in order to supply a fresh charge of grinding compound to the tube. Pressure should be applied rather lightly throughout—and particularly toward the end.

It may be necessary to grind the bottom (outside) of the cell to flatness lest a poor fit be obtained between the bottom plate and the vial itself. This may be done on a glass plate or an emery stone. The top of the cell should also be ground down to a smooth edge.

The bottom plate might be a cover-glass although it will be thinner than is to be desired. A better plate can be made by drilling a hole out of a microscope slide with a ½ inch brass tube and using the disc which is cut out.

As the glass cell will be smaller than the central hollow of the cooling unit, it is shimmed with a strip of aluminum foil which serves to hold it in place and provide better contact with the brass walls.

Although a thicker unit than this might permit water circulation underneath the glass cell, it would probably be so thick as to prevent use of the 48-mm. objective of the microscope where the body-tube of the microscope is already near the top of its excursion.

Figure 2 shows a completed unit of the dimensions shown on the sketch. (The large screw is merely a plug for a hole drilled overly deep in the hose connecting unit.) This unit will remain in position on the microscope stage without difficulty though it is provided with no special clips. Hose connections could be at an angle to one another but would tend to pull it out of position should the hoses be moved accidentally.

Lacking a constant temperature bath, this unit is supplied with water from a reservoir fed with both hot and cold tap water, as indicated in the small sketch at the top of the diagram. The reservoir is an inverted one-gallon bottle, supplied with an air tube (and overflow outlet combined). The other outlet tube extends just through the cork, from which it leads to a flask fitted with a thermometer—thence to the cooling unit. From here the water may be run into a refractometer, or directly to the sink. The thermometer will not give a reliable reading of the temperature of the cell fluids during periods of rapid temperature change, but is a suitable indicator otherwise.