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A Study of the Refractive Index Variations Within and Between Sealed Beam Headlights Using a Precise Method

One of the primary goals of the forensic scientist is to be able to individualize physical evidence. Associated with this goal is the underlying assumption that no two things are exactly alike. Individualization has been obtained if it can be unequivocally stated that this fingerprint is that of John Doe, or that this piece of glass is from this sealed beam head-lamp and no other. Unfortunately, due to practical limitations, individualization is not always possible. In such a case the forensic scientist must examine all information available and then make an "estimate of the situation" [1] often based upon his past experience. The purpose of this paper is to aid the forensic scientist so that he may make a better estimate of the forensic value of automobile headlamp glass when confronted with this type of evidence.

Historically, physical properties have long been exploited in the examination of physical evidence. This is especially true of density and refractive index (RI) with reference to glass samples. One reason for the past success of these approaches stems from the fact that small variations in chemical composition could be detected more readily by measurements of these properties than by chemical analysis due to limitations in quantitative accuracy of available chemical techniques. Even present day techniques are limited in this regard. Typical ranges in quantitative uncertainty for elemental techniques such as emission spectrography and neutron activation analysis are relatively large (± 10 percent).

Recently there has been concern in forensic laboratories about the value of such physical properties as density and refractive index due to the high uniformity of these physical properties as a result of recent improvements in the techniques of mass production and the large variety of glass products currently manufactured.

Kingston [2] conducted a survey among criminalists on a nationwide basis to ascertain which evidential materials were of most concern with respect to the need for improved methodology. The respondees stated that standard methods and procedures were most needed for glass, hair, paint, soil, fibers, and blood (in that order). The method and results presented below should aid the forensic scientist in evaluating the significance of RI measurements in cases involving automobile headlamp glass.

The method combines the use of the Mettler Hot Stage (Mettler Instrument Corp., Princeton, N.J.) and the phase contrast microscope and features accurate match point

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410 JOURNAL OF FORENSIC SCIENCES

determinations coupled with accurate and convenient control of the RI of the immersion fluid by utilizing the temperature variation method [3]. This method was used to examine the variations of RI in sealed beam headlamps manufactured in the United States. Pointto-point variations of RI within the lens and reflector portions of headlamp glass were examined and compared with the overall variations of RI within the product line of the manufacturer. Variations in refractive indices between daily production line samples obtained from a manufacturer of automobile headlamp glass are presented along with the variations of RI in sealed beam headlamps obtained from an automobile wrecking yard.

Theory and Methods

Obtaining a precise RI match between the immersion fluid and the glass particle has long been a problem. This problem may be separated into two parts: (1) adjusting the refractive index of the immersion medium to match the RI of the glass particle and (2) sensitively detecting the RI match point.

One of the commonly used methods for adjusting the RI of the immersion medium requires the mixing of two liquids which bracket the RI of the glass specimen. The proportions of these liquids are varied until a match is obtained between the fluid and the glass particle and at this point an aliquot of the mixture is measured with a refractometer. Another means of adjusting the RI of the immersion fluid is the temperature variation method. The data presented here, were obtained by this method. The Mettler FP-2 Hot Stage is used to accurately control the temperature of the immersion medium [4,5].

The most well known method for observing a RI match between a transparent particle and the immersion fluid that surrounds it was first described by Becke [6]. One drawback of this method is that it becomes increasingly difficult to see the Becke line as one gets closer to the match point of the immersion fluid and the glass fragment. Faust [7] notes that every disappearance of the Becke line does not necessarily correspond to equality of RI. He states that the line arises from an asymmetrically diffracted wave originating at the specimen edge. He also points out that the contrast of the diffraction pattern (Becke line) is influenced by the shape and dimensions of the specimen and by the degree of defocusing of the microscope. These factors in turn, affect the accuracy of the match.

Phase contract microscopy is used to accentuate refraction images and for this reason presents a very sensitive means of accurately locating match points. The use of phase contrast microscopy in conjunction with the Mettler Hot Stage appears to offer a nearly ideal means of measuring the refractive indices of small particles of glass. We have presented a detailed evaluation of this technique in an earlier paper [8]. Because of the excellent accuracy and precision of this method it was selected for the critical examination of automobile headlamp glass.

Apparatus and Materials

Phase contrast microscopes manufactured by American Optical Co. and Nikon were utilized. Both instruments gave equivalent results. All work was done with $10 \times$ phase objectives. A Nikon microscope (model SKE) was used for most of the work. The phase contrast objective was a Nikon BM 10 (0.30 NA).

It was found necessary to use a long working distance phase condenser (Nikon #77049, 12–15 mm working distance) because of the thickness of the hot stage. In fact, the central aperture in an inexpensive plain flat stage (Nikon "P" #77775) was enlarged so that the condenser could protrude through it to raise the focal point to the sample level within the hot stage. This alteration could have been avoided if the Nikon #77035 extra long working distance condenser had been used (40 mm).

The Mettler Hot Stage consists of the hot stage portion, which is mounted on the microscope stage, and a control unit. The instrument may be temperature programmed for linear heating or cooling rates of 10, 3, 2, 1 or 0.2 C per minute. The advantage of using this apparatus is that the temperature can be conveniently controlled to 0.1 C. In addition, the temperature can be noted with a digital display/recording device supplied as an integral part of the instrument. In this manner an uninterrupted view of the preparation is possible during the course of the experiment, while the temperature data are simultaneously recorded. Dabbs and Pearson [4] evaluated the hot stage and found the temperature lag to be less than 0.3 C at a heating rate of 1 deg/min. The lag was less than this at the slower heating rate and was found to be reproducible.

Monochromatic illumination at the D-line of sodium was obtained by the use of an arc lamp (G. W. Gates and Co., Long Island City, N.Y.) or alternatively an interference filter (Baird Atomic, Cambridge, Mass.).

Internal glass standards were used which eliminated the need for a refractometer. The glass standards were optical glasses on which the N_D^{20} for each was known to ± 0.00003 (Schott Optical Glass Inc., Duryea, Pa.). One important factor which must be taken into account when using internal standards is the temperature coefficient of RI for each of the glass standards used. For example, one standard labeled FK50 ($N_D = 1.48616$) had a dn/dT of $-6.7 \times 10^{-6}/\text{deg C}$ in the 20 to 40 C temperature range. Its match point with dibutylphthalate was 36.7 C. Therefore, the correction factor was (36.7 – 20.0 C) \times $(-6.7 \times 10^{-6}/\text{deg C})$ or -0.00011 RI units, where 20 C is the temperature at which the glass standard was calibrated. This correction factor which would be negligible by most immersion methods is significant with the method utilized here, and becomes even more significant at higher temperatures. It is not necessary to have a calibration curve if two pieces of glass are to be compared side-by-side. However, it might be prudent to know the approximate dn/dT of the immersion liquid so that the absolute RI difference between the two samples may be determined. Similarly, if the standards and the unknowns have similar dn/dT (which is generally the case), then the RI correction at various temperatures would be almost completely self-compensating.

Immersion fluids such as silicone oils or dibutylphthalate will maintain relatively stable refractive indices if they are kept free from contamination. We have found that contact with the rubber bulb portion of a glass dropper bottle can seriously alter the RI of dibutyl-phthalate. Precautions should be taken to keep the immersion liquid pure, and in addition, regular periodic calibrations are advisable.

Procedure and Results

Samples of headlamp glass were collected from Corning Glass Works, Greenville, Ohio; Anchor Hocking, Lancaster, Ohio, and General Electric, Richmond Heights, Ohio.

At the Greenville plant there are two continuous-flow glass tanks (designated A and B). The raw materials enter the back end of the tank in premeasured proportions and flow toward the front. About 15 percent of the raw materials that enter the tank consist of scrap glass (cullet). As the molten glass reaches the front (about $11/_2$ days) it is skimmed (tap stream) from both top and bottom. The glass from the tap streams is subsequently used as cullet. The glass flows to forming machines (6 per tank) where the glass is pressed into headlamp components. The output for the two tanks is in hundreds of thousands of pounds per day. Operation is constant, 24 hours a day, 7 days a week, except for two weeks during the summer.

Figure 1 demonstrates the variation in RI of glass manufactured between March 22nd and 23rd 1970 for both Corning tanks. During this 24-h period a total of 48 glass specimens

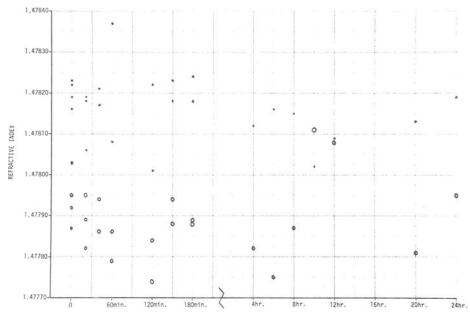


FIG. 1—Variation in refractive index of automobile headlamp glass. More than one RI value in any given time period indicates the RI values for consecutive production line samples. The two different symbols represent the two sample tanks, A and B.

were collected. At the beginning of the collection period consecutive production line samples were taken from the conveyor belt, and are noted on the figure by more than one point at a given time. The fragments of glass taken for measurement were removed from convenient locations on the specimen. Removal of a fragment without shattering the specimen (lens or reflector) was the main criterion for convenience in this instance. Two of these consecutive production line samples (reflectors, tank A) were each sampled at 9 separate locations. The RI variation within the two samples is graphically displayed in Fig. 2. The cross (+) represents the approximate location from which the sample was taken. The letters in the pictorial portion of the diagram refer to the RI at that point as listed on the left-hand side of the figure. The number in parentheses represents the difference in RI at that point from that at the center times 10⁵. Corning also provided daily samples from each tank (65 samples each) during the period of January 17 to March 22, 1970. The RI variation of the samples taken during this time period (tank A) are presented in Fig. 3. Variations in tank B are shown in Fig. 4.

Anchor Hocking manufactures lens and reflector components from one continuous-flow glass tank. Figure 5 shows the RI variations of 43 separate components manufactured during the period of January 16 to March 24, 1970. Consecutive production line samples of headlamp glass (reflectors) were each sampled at 9 separate locations. The RI variation within these samples is graphically displayed in Fig. 6.

General Electric, the third United States manufacturer of automobile headlamp glass uses one continuous-flow glass tank. General Electric provided 56 separate glass samples which had not been formed and 19 samples which had been formed into lenses. The formed samples were all produced during a single 24-h period. Two consecutive production line samples were each sampled at 9 separate locations. The RI variation within these samples is presented in Fig. 7. The RI of the unformed samples (manufactured during the period of February 6 to May 5, 1970) is presented in Fig. 8.

Twenty-eight complete sealed beam headlights were obtained from an automobile wrecking yard located in Queens, New York. Samples of glass from the front (lens) and rear (reflector) portions were examined with regard to RI. Figure 9 presents the results of RI measurements on these 28 headlights (56 samples). In most cases the front and rear portions are close in RI (within 0.0005 of each other). Four of these sealed beam head-lights were cut into fourths with a diamond saw, and sampled at 9 separate locations along the lens portion, and at 9 locations along the reflector portion. Point-to-point variation in RI for the front and rear portions of these headlights is presented in Figs. 10 through 13.

During this study a grand total of 492 specimens (excluding calibrations) were examined. The total is composed of the following: 194 samples of Corning Glass, 82 samples of Anchor Hocking glass, 96 samples of General Electric glass, and 120 samples of glass from an automobile wrecking yard (24 headlights + 4 headlights sampled at 18 separate locations—48 + 72 = 120). Figure 14 shows the distribution of RI of the samples from the

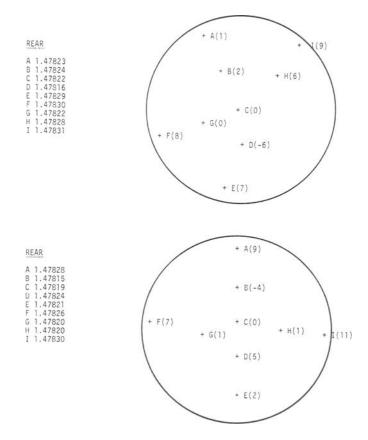


FIG. 2—Variations of refractive index within two consecutive production line samples of automobile headlamp glass (reflectors). Reflector is viewed from concave side.

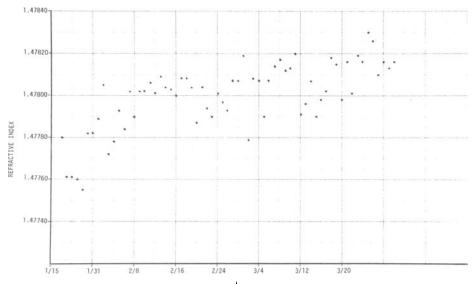
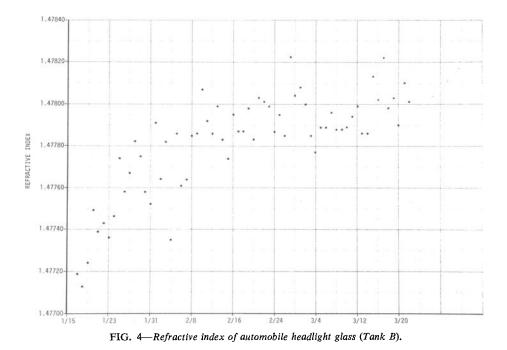


FIG. 3—Refractive index of automobile headlight glass (Tank A).



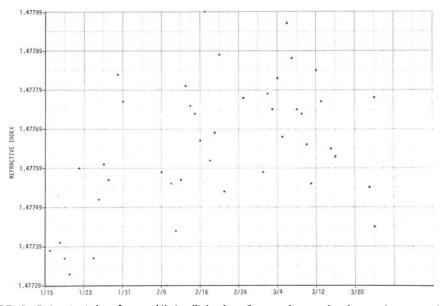


FIG. 5—Refractive index of automobile headlight glass. One sample was taken from each specimen for measurement.

three manufacturers of automobile headlamp glass and the distribution of RI of the samples obtained from an automobile wrecking yard. The distribution of refractive index from these four sources was broken down into eight groups labeled A through H. It must be remembered that the samples from the manufacturers represent a time period of approximately three months while the wrecking yard samples (although few in number) represent variation of RI over an indeterminate period of time, probably a number of years. The RI range of all headlight glass examined was 1.47657–1.48020 or 0.00363 RI units. As can be seen from Fig. 15, the headlamp glass manufactured by Corning during the three month period of time previously mentioned had a RI range from 1.47713 to 1.47837 or 0.00124 RI units, while the glass from Anchor Hocking had a RI range from 1.47729 to 1.47799 or 0.00070 RI units, and those headlamps from General Electric had a RI range from 1.47775 to 1.47881 or 0.00106 RI units. The automobile headlamp glass from these three manufacturers had an overall range of 0.00221 RI units.

Figure 15 also illustrates the range of variation of RI within a selected single lens or reflector from each general source along with the range of variation in the next consecutive production line sample. Variations within four headlights (front and rear portions) obtained from an automobile wrecking yard are also listed. As will be discussed below, it can be seen that in most cases the variation within the reflector portions of a headlight was greater than the variation within the lens portion of the same headlight. The range of variation within the reflector portion of one of the sealed beam headlamps obtained from an automobile wrecking yard was as large as 0.00112 RI units. This is equivalent to 31 percent of the total refractive index range found in the wrecking yard samples. Tables 1 and 2 summarize the information from Fig. 15.

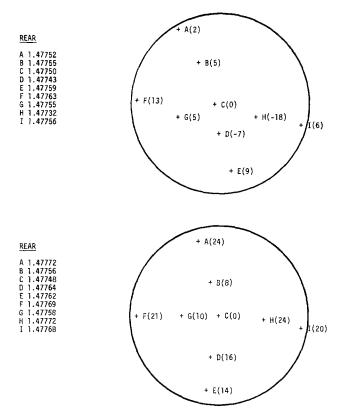


FIG. 6—Variation of refractive index within two consecutive production line samples of automobile headlight glass (reflectors). Reflector is viewed from front (concave side).

 TABLE 1—A comparison of the total refractive index range in automobile headlamp glass produced by various manufacturers over a three month time period with the range of point-to-point RI variations in consecutive production line samples.

Company	Total RI Range	Number of Separate Samples Examined	Range: Prod. Line Samples	Range: Consecutive Prod. Line Sample	Percentage of Total Range
Corning	0.00124	178		•••	100
			0.00015		12
				0.00015	12
Anchor Hocking	0.00070	66			100
			0.00031		44
				0.00024	34
General	0.00041	20			100
Electric			0.00013		32
(formed)				0.00027	66
General Electric (unformed)	0.00106	56			•••

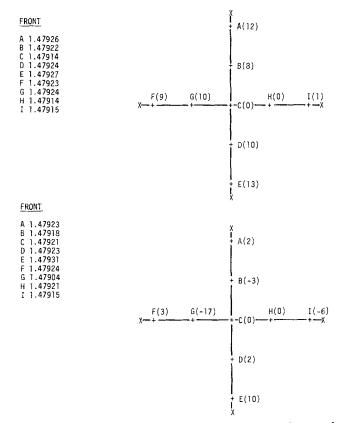


FIG. 7—Variation of refractive index within two consecutive production line samples of automobile headlight glass (lenses). Lens is viewed from front (convex side).

TABLE 2—A comparison of the refractive index range found within four individual headlamps with the overall RI range (0,00363) found between samples of 28 headlamps (56 samples) obtained from an automobile wrecking yard. The four selected headlamps were examined at eighteen locations.

Brand of Headlamp	Refractive Index Range: Lens	Refractive Index Range: Reflector	Percentage of Total Range
Tung-Sol	0,00026		7
		0.00076	21
General Electric	0,00009		2
		0.00033	9
Westinghouse	0.00018		5
	•••	0.00054	15
Guide Lamp	0.00048		13
(General Motors)		0.00112	31

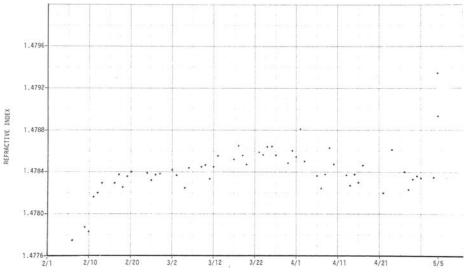


FIG. 8-Refractive index of unpressed samples of automobile headlight glass.

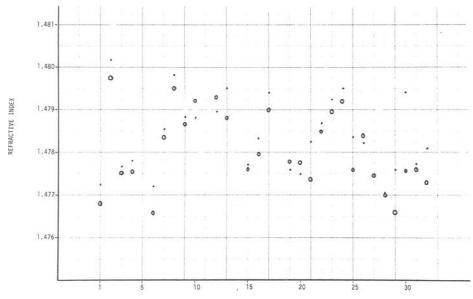


FIG. 9—Refractive index in the front (*) lens and rear (°) reflector portions of complete automobile headlights. Numbers along abscissa are for identification purposes.

Discussion and Conclusions

It should be pointed out that the actual dn/dT of the headlamp glass was not known, although an estimate of $-6.5 \times 10^{-6}/\text{deg C}$ for borosilicate glasses has been supplied [9]. The results presented here do not reflect a correction for the dn/dT of the headlamp glass samples. If it is assumed that the dn/dT for all the headlamp glass samples examined are

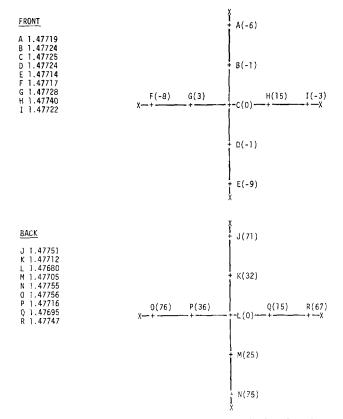


FIG. 10—Variation of refractive index within the front (lens) and back (reflector) portions of Tung-Sol sealed beam headlamp. Both portions are viewed from the front.

similar, the relative RI differences between the values presented would still remain essentially the same. The refractive indices given here are the indices of the glass samples at their match point temperatures. Correction factors (ψ) utilizing the -6.5×10^{-6} /deg C figure for the dn/dT of these glass samples can be derived and added to the results given here to yield the $N_{\rm D}^{20}$ for each of the samples. The formula is

$$\psi = \frac{N-n}{S} \left(-6.5 \times 10^{-6} / \text{deg C}\right)$$
(1)

where N is the refractive index of the immersion liquid at 20 C, n is the refractive index of the glass sample at the match point temperature, and S is the slope of the calibration curve for the immersion liquid (that is, the dn/dT of the liquid). Using dibutylphthalate as the immersion liquid this simplifies to:

$$\psi = (1.49270 - n) \ 1.64 \times 10^{-2} \tag{2}$$

Similar correction factors can be derived for other samples of dibutylphthalate or other immersion liquids.

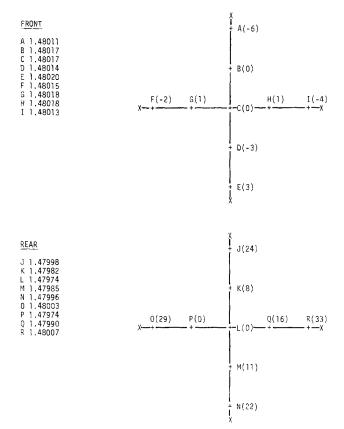


FIG. 11—Variation of refractive index within the front (lens) and rear (reflector) portions of a G.E. sealed beam. Both portions are viewed from the front.

The production line variations in refractive index between different headlamps produced over a period of time are in large measure masked by the internal variations in each article. These relatively large internal variations suggest that the melt in the large tanks is not completely homogeneous or that some inhomogeneities in RI are introduced in the molding process or both. The latter, if they exist, are evidently not completely relieved in the annealing step. Some support for the view that inhomogeneities arise as a result of the molding and fabricating operations is given by the observation that in general the RI variations within the reflector portions of the lamps were greater than those within the lens portions although the same melt is often used in producing both components.

It has been shown that when two samples of glass of "identical composition" are annealed at two different temperatures, they will exhibit different densities and refractive indices. Tool et al [10] investigating phenomena associated with the annealing of glass, found the average magnitude of the equilibrium temperature coefficients for density and for refractive index (at the sodium D-line) to be about 28×10^{-5} and 37×10^{-6} /deg C, respectively. These measurements were made at ambient temperatures. Thus, the differences observed in RI in an individual specimen may be due to compositional differences or to inhomogeneities arising in the fabricating or annealing process or both.

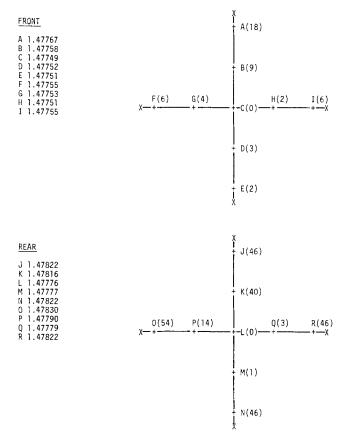


FIG. 12—Variation of refractive index within the front (lens) and rear (reflector) portions of a Westinghouse sealed beam headlamp. Both portions are viewed from the front.

As can be seen from an examination of Table 1 the ranges of refractive index between the production line samples of the three United States manufacturers are relatively small. Approximately 60 percent of the Anchor-Hocking samples fell within a range of only 0.0003 RI units. An absolute determination by the simple Becke line method (accuracy ± 0.001) would yield the same quantitative result for all of these if not for the great majority of the samples produced by the three manufacturers during the three month period (overall range $\simeq 0.002$). Side-by-side comparison of two samples, although tedious, would make somewhat higher degrees of discrimination possible with the simple Becke line method. The significance of the differences observed, however, would be difficult to interpret under these conditions. The utility of the conventional method (Becke line without temperature variation and control) in an actual case involving modern headlamp glass would be severely limited, and thus, the property of refractive index would have no practical significance with respect to headlamp glass evidence.

The method utilized in this study provides an improvement of nearly two orders of magnitude over the simple Becke method. The error for an absolute RI determination with this method is dependent upon the standards used to calibrate the dibutylphthalate and hot stage. The standards used for this study were calibrated to 3×10^{-5} RI units.

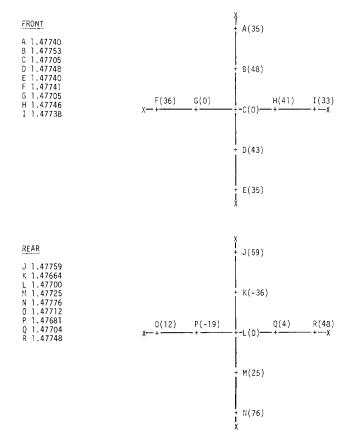
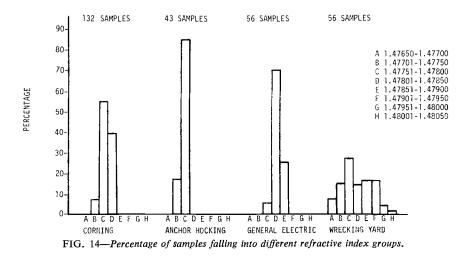


FIG. 13—Variation of refractive index within the front (lens) and rear (reflector) portions of a Guide Lamp (G, M) headlamp. Both portions are viewed from the front.



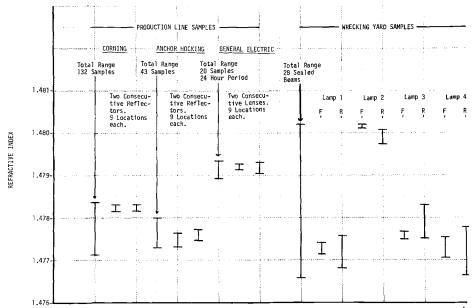


FIG. 15—Variation of refractive index in automobile headlight glass produced by three individual manufacturers over a three-month period.

This resulted in an average standard deviation of 3.4×10^{-5} RI units for absolute RI determinations. More accurately calibrated standards would improve the accuracy of the method. For example, if the calibration standards had been accurate to 1×10^{-6} , the average standard deviation of absolute determinations would have been 1.7×10^{-5} RI units. If relative differences in RI are needed, such as in the case of comparative RI measurements, average standard deviations of 8.8×10^{-6} RI units or better may be obtained. Even higher degrees of discrimination are possible with side-by-side comparisons. This increase in sensitivity and accuracy over the conventional method makes the study of the small variations in refractive index of the headlight glass feasible.

The range of variation in headlight glass, although quite small, is relatively large by comparison to the range of uncertainty in the method described here. The twenty-eight sealed beam headlamps obtained from the automobile wrecking yard (56 samples) had the largest RI range (0.00363). It can be seen that this is about 100 times larger than the sensitivity of the present method. Unfortunately this favorable situation is complicated by the rather large internal variations within the lamp components. As was mentioned earlier one of the reflector portions of the four lamps that were critically examined had a refractive index range of nearly one third of the total range of all the wrecking yard samples. These point-to-point variations within headlamp glass seriously affect the significance of refractive index measurements for the purposes of individualization. It would be possible to attach more significance to the results in actual practice if it can be ascertained that the known and questioned samples are from the same region of the article. Such a situation might occur where an object remains largely intact after fracture. If this is not possible, it would be necessary to compare the ranges of the indices in each sample.

424 JOURNAL OF FORENSIC SCIENCES

Other matters for further investigation suggest themselves. Determinations of the dispersion of headlamp glass samples could be accomplished by making refractive index measurements at three different wavelengths utilizing two additional interference filters (such as, the F and C lines of hydrogen). These data could then be used to calculate the "nu value" or some other measure of dispersion for each sample. It is quite possible that the variations in dispersion between modern headlamp glass samples may be too small to be of use in individualization as has been indicated in earlier studies [11], but this point warrants further investigation due to the fact that the earlier work was accomplished with less sensitive techniques.

Studies using the present method could be undertaken in other glass sample populations such as safety glass, window glass, bottle glass. Such investigations would serve to make statistical data available to the practicing forensic scientist.

The technique employed here could be improved still further. With the availability of more accurately calibrated standards, more accurate control and readout of the hot stage temperature would be desirable. The sensitivity of determining the match point could be improved by modifications in the phase contrast system along the lines previously suggested [8]. Commercially available phase contrast systems are generally optimized for use with biological samples and are not necessarily the best for refractive index work.

Summary

The combination of phase contrast microscopy and the Mettler Hot Stage makes very accurate determinations of refractive index (3.4×10^{-5}) with small fragments of glass possible. Consecutive measurements may be carried out with a precision of 8.8×10^{-6} RI units. This high sensitivity allows small variations within and between modern sealed beam headlamps to be examined. Consecutive production line headlamp glass samples along with samples manufactured over a period of three months were obtained from three companies. The overall variation in RI between the samples of headlamp glass produced by the three manufacturers was found to be $\simeq 0.002$. Thus, the bulk of these variations could not have been measured with the simple Becke line technique (accuracy ± 0.001). It was also found that the internal RI variation in some headlights was of the same order of magnitude (although smaller) as the variation between headlamps. The internal variations were generally somewhat larger in the reflector portions of the lamps than in the lenses. This is particularly true of the components of lamps that were recovered from an automobile wrecking yard.

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