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Density, Refractive Index, and Dispersion in the Examination of Glass: Their Relative Worth as Proof

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ABSTRACT: By statistical analysis of probabilities, the value of density, refractive index, and dispersion for identifying the source of glass samples is evaluated. Empirical tests validated the statistical conclusions. It was determined that density has by far the greatest differentiating power of the three parameters tested; assuming equal thresholds of discrimination, density has a differentiating power some six times greater than that of refractive index. Dispersion offers little, if any, improvement over refractive index. Density and refractive index together are somewhat greater in value than density alone.

KEYWORDS: criminalistics, glass, physical properties

In an earlier report [*1*] an estimate was given of the probability that two glasses from different sources would have the same density. A value of approximately 1 in 50 was determined from a sample of 52 glasses. Later, Reeves et al [*2*] reported the results of additional studies on the same 52 glasses to determine the further differentiating value of refractive index and elemental composition as determined by energy dispersive X-ray fluorescence analysis. That report gave extensive data to show the added worth of elemental analysis but did not give quantitative information as to the gain, if any, in "proof value" offered by determining the refractive index.

On many occasions the particle size of the glass is restrictively small for the determination of its density. Finding the refractive index of such glass to be indistinguishable from glass from a reference source would lead some workers to assume that the refractive index has the same proof value as comparisons based on density. Such a judgment would be based on the high coefficient of correlation reported for these two properties [*3*] and the assumption that both have equal discriminating power. That there is a high correlation cannot be disputed. The assumption of equal discriminating power is troubling, however, because if refractive index is exclusively relied upon, the criminalist risks a possibly harmful error if discriminating power is in truth less than that of density. This is a matter of some concern, especially for personnel in laboratories not equipped for elemental analysis.

The work reported here was principally intended to test the validity of assuming equal proof value for density and refractive index. However, the method used for determining refractive index also gave a value for dispersion. Thus the worth of each of the three properties was evaluated singly and in combination.

Glass sent as reference samples by law enforcement agencies in Santa Clara County, CA,

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from 1974 to 1980 formed the study population. All samples were window glass, principally from businesses; they were clear except for some with a smoky tint. Some safety glasses were included. A total of 50 glasses known to be from different sources was used.

Density Determinations

The density of the glasses was characterized with a sink-float multiple tube comparator similar to that described in the ASTM Test for Density of Glass by the Sink-Float Comparator (C 729-75). Figure 1 shows details of the apparatus, which was constructed from parts available in the laboratory. The liquid used to determine density was a mixture of dibromomethane (density ρ = 2.4970 at 20°C) and 1,1,2,2,tetrabromoethane (ρ = 2.9656 at 20°C). These were chosen to prepare a solution whose densities at temperatures from 20 to 50°C would cover the range of glass densities commonly encountered in practice (2.470 to 2.525). The fluid mixture used in the study had a density of 2.5245 at 20°C. The tubes were filled with this liquid to a level below the level of water in the 2-L breaker. In two of the tubes a series of six calibrated Cargille density beads were sequentially placed according to their

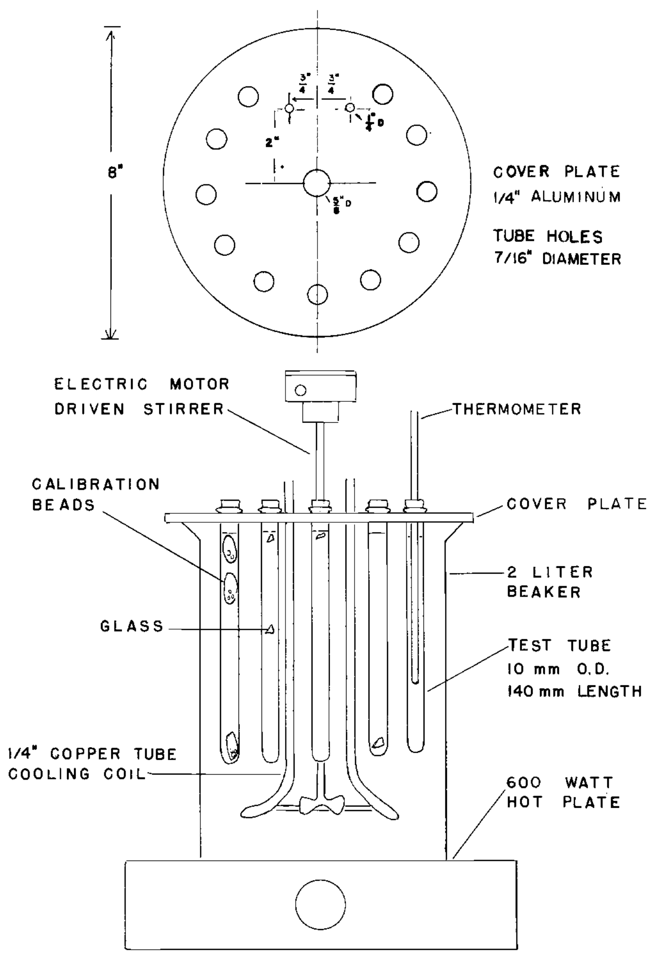

FIG. 1—*Multiple tube density comparator.*

densities, three in each tube. Density determinations were made in a vented hood and were greatly aided by a strong source of illumination.

The hot plate (wattage rating—600) was set for an approximately 3°C/min rise in temperature. With the stirrer on, the temperature at which each of the beads began to descend was read from the thermometer and recorded to 0.1°C. When all the beads had descended, power to the hot plate was turned off; water from a tap was moved through the cooling coil and the temperature at which each of the beads began to rise was recorded. The descent and ascent temperatures for each of the calibration beads were easily determined to a precision of 0.1°C.

The values for temperature match and nominal bead density were entered into the linear regression routine of an electronic hand calculator (Texas Instruments TI-55). From this routine it was possible to rapidly calculate a linear estimate of density for any temperature entry. For six calibration beads the correlation coefficient was determined to be −0.99. Also calculated was the density change for each 1°C change in temperature, $d\rho/dT$. For the fluid mixture used in the study, $d\rho/dT = 0.0025$.

The multiple tubes of the apparatus enabled several glasses from different sources to be simultaneously analyzed, with each tube including many particles from the same source. Other tubes were used for calibration beads; one was used to contain a thermometer certified by the National Bureau of Standards (covering 0 to 50°C in 0.1°C gradations). All were filled with the same density fluid mixture.

Values for ρ were determined for each of the 50 glasses studied. The glass particles were selected to be approximately 3.2 mm (⅛ in.) in the largest dimension. The density values calculated were based on the ascent temperature, thus avoiding surface tension effects, which can cause error when the temperature of descent is used. All glasses were tested within a period of one week, the progress of change in the liquid density being monitored through the calibration beads during each series of determinations. No change was detected.

Determination of Refractive Index and Dispersion

Characterization of refractive index and dispersion for the glass samples was effected through observation of dispersion staining effects. The instruments used were a microscope equipped with a 10× dispersion staining objective and a Mettler FP-52 hot stage together with a Mettler FP-5 controller. Dispersion staining effects and their production by introduction of annular or central stops at the back focal plane of a microscope objective are discussed by McCrone et al [4]. Application of these effects to characterization of a wide variety of particulate matter, including glass, has also been described [5].

Glass that had been reduced to uniform particle size by grinding in a carbide mortar and pestle was placed on a microscope slide and immersed in a high-dispersion liquid selected to be higher in refractive index at 25°C and 589 nm n_D^{25} than that expected for the glass. A cover glass was placed over the preparation and care was taken to avoid excessive tilt. The preparation was inserted into the Mettler hot stage at about 28°C and the glass particles were observed for color with the central stop of the dispersion staining objective in place. The presence of green or bluish-green colors indicated that the glass n_D was less than the n_D^T of the liquid. The controller was set to cause a temperature rise of 3°C/min. Observations of colors and the corresponding wavelengths were as follows: first red—620 nm; blue-violet and red-orange—560 nm; violet and orange—520 nm; yellow and violet—485 nm. The match temperature for the appearance of each of these colors was recorded. Based on the observations made, an analytical dispersion staining curve was plotted. The refractive index for each match point was calculated from the change in refractive index for each 1°C temperature change dn/dT. Figure 2 shows a typical analytical dispersion curve plotted on a Hartman net dispersion staining chart for a Kofler glass standard with $n_D = 1.5204$.

Applying the correction of $dn/dT = 0.00047$ for the liquid ($n_D^{25} = 1.525$) at each of the match temperatures gave values for n_D^T of 1.5221 at 620 nm, 1.5183 at 560 nm, 1.5152 at 520 nm, and 1.5109 at 486 nm. The calculations were based on the equation:

$$n_D^T = n_D^{25} - 0.00047\,(T - 25)$$

where n_D^T is the refractive index n_D^{25} of the Cargille liquid at the matching temperature.

The initial Becke-line color assignment, specified as the first red, was selected because of the abruptness of its appearance. That it was indeed the first red was verified through movement of the preparation to study other fields. If such study revealed particles that were more vividly red, the most intense was studied by lowering the temperature until the red color disappeared. The controller was set to heat slowly (3°C/min) until the red edge effect reappeared. At this point, the temperature was recorded as the match point for 620 nm. Continued elevation of temperature resulted in rapid appearance of additional particles with red edge effects, confirming that the first red recorded was valid. If additional reds were not seen

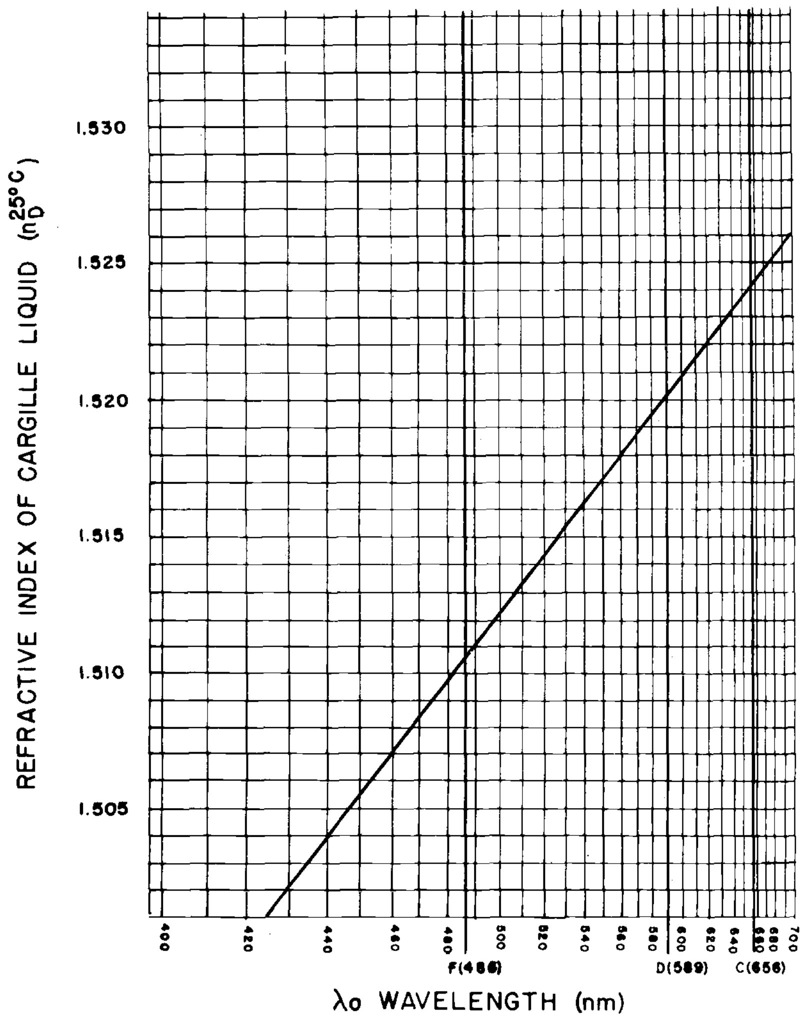

FIG. 2—*Analytical dispersion staining curve for Kofler certified glass powder with* n_D = *1.5204.*

in immediate succession the first red was considered suspect. Such a condition was occasionally noted in some glasses, especially when the particles were believed to include the surface, that is, tramlines as described by Underhill [6]. All other colors designated to be observed through continued heating were recorded at the temperature of their first and definite appearance.

TABLE 1—*Density, refractive index, and dispersion values.*

Glass	Density	Refractive Index			Dispersion,
		n_C	n_D	n_F	$n_C - n_F$
1	2.4763	1.5183	1.5140	1.5037	0.0146
2	2.4782	1.5190	1.5150	1.5058	0.0132
3	2.4788	1.5192	1.5145	1.5038	0.0154
4	2.4791	1.5199	1.5159	1.5058	0.0140
5	2.4797	1.5188	1.5140	1.5052	0.0136
6	2.4820	1.5190	1.5155	1.5062	0.0128
7	2.4851	1.5213	1.5161	1.5038	0.0175
8	2.4851	1.5207	1.5163	1.5055	0.0152
9	2.4859	1.5212	1.5170	1.5055	0.0157
10	2.4862	1.5215	1.5173	1.5073	0.0143
11	2.4864	1.5213	1.5167	1.5056	0.0157
12	2.4869	1.5198	1.5169	1.5076	0.0122
13	2.4875	1.5215	1.5175	1.5077	0.0138
14	2.4877	1.5215	1.5175	1.5077	0.0138
15	2.4882	1.5219	1.5177	1.5074	0.0145
16	2.4885	1.5222	1.5178	1.5079	0.0143
17	2.4886	1.5223	1.5181	1.5081	0.0142
18	2.4895	1.5223	1.5181	1.5081	0.0142
19	2.4903	1.5224	1.5183	1.5079	0.0145
20	2.4903	1.5215	1.5174	1.5076	0.0139
21	2.4916	1.5217	1.5177	1.5082	0.0135
22	2.4916	1.5216	1.5174	1.5075	0.0141
23	2.4918	1.5220	1.5181	1.5098	0.0122
24	2.4921	1.5228	1.5187	1.5087	0.0141
25	2.4921	1.5230	1.5187	1.5088	0.0142
26	2.4922	1.5234	1.5193	1.5091	0.0143
27	2.4929	1.5223	1.5180	1.5079	0.0144
28	2.4930	1.5230	1.5185	1.5074	0.0156
29	2.4931	1.5231	1.5187	1.5085	0.0146
30	2.4931	1.5230	1.5187	1.5085	0.0145
31	2.4949	1.5239	1.5197	1.5087	0.0142
32	2.4953	1.5237	1.5195	1.5086	0.0151
33	2.4974	1.5246	1.5203	1.5098	0.0148
34	2.4974	1.5240	1.5197	1.5092	0.0148
35	2.4996	1.5255	1.5216	1.5119	0.0136
36	2.5042	1.5267	1.5219	1.5112	0.0155
37	2.5055	1.5265	1.5224	1.5123	0.0142
38	2.5064	1.5270	1.5228	1.5130	0.0140
39	2.5086	1.5291	1.5249	1.5149	0.0142
40	2.5107	1.5280	1.5239	1.5140	0.0140
41	2.5110	1.5273	1.5233	1.5133	0.0140
42	2.5112	1.5275	1.5235	1.5139	0.0136
43	2.5117	1.5283	1.5238	1.5133	0.0150
44	2.5120	1.5279	1.5236	1.5136	0.0143
45	2.5133	1.5286	1.5245	1.5140	0.0146
46	2.5135	1.5280	1.5240	1.5142	0.0138
47	2.5153	1.5290	1.5249	1.5147	0.0143
48	2.5156	1.5284	1.5242	1.5140	0.0144
49	2.5164	1.5294	1.5250	1.5158	0.0136
50	2.5221	1.5307	1.5266	1.5168	0.0139

The color judgments were confirmed by inserting filters in the pathway of the light source and making observations for the Becke line with the objective free of focal screens. Filters were used for the temperature match points of 656 nm (n_C), 589 nm (n_D), and 486 nm (n_F), respectively.

Table 1 gives values of density and refractive index for each of the 50 glasses studied. From the data given, together with the discrimination threshold for each of the properties, the probability that two glasses would be indistinguishable in density and in refractive index, considered singly or in combination, was calculated. Also, the data permitted the additional discriminating worth of dispersion to be estimated.

Thresholds for Discrimination

The discrimination thresholds used are given in Table 2. The values for ρ were arrived at through separate determinations on 21 glass particles taken from scattered locations throughout a sheet of 457- by 610-mm (18- by 24-in.) plate glass. The refractive index and dispersion threshold values were arrived at through 21 separate determinations on Kofler certified glass powder with $n_D = 1.5204$.

The threshold values given are based on the standard deviations of the 21 determinations for each of the properties. For example, the standard deviation for ρ was ± 0.0001 and the threshold was 0.0002, 0.0004, and 0.0006 for one, two, and three standard deviations, respectively.

TABLE 2—*Discrimination threshold values for density, refractive index, and dispersion of glass.*[a]

Confidence Level, %	Standard Deviation	Discrimination Threshold ($\times 10^{-4}$)		
		ρ	n_D	Dispersion
67	± 1	2	3	7.5
95	± 2	4	6	15
99.7	± 3	6	9	23

[a]Values given resulted from 21 determinations on the same glass sample.

TABLE 3—*Relative probabilities of distinguishing two samples of glass from a population of 50.*

Properties Examined[a]	Discrimination Threshold ($\times 10^{-4}$)			Probability of Indistinguishability, %
	ρ	n_D	ν[a]	
ρ	4	...	...	2.2
$\rho + n_D$	4	6	...	1.8
ρ	6	...	...	3.1
$\rho + n_D$	6	9	...	2.5
$\rho + n_D + \nu$	6	9	15	2.1
$\rho + n_D + \nu$	6	9	25	2.3
n_D	...	3	...	4.8
$n_D + \nu$	...	3	7	3.3
n_D	...	6	...	12.6
$n_D + \nu$	...	6	15	12.2
n_D	...	9	...	17.9

[a]ρ = density, n_D = refractive index, and ν = dispersion.

Determination of Worth

Combining the data given in Table 1 and Table 2 permits the determination of the desired evidentiary worth values. Table 3 gives a summary of these values when the data are considered individually and in combination.

The manner in which the probability values in Table 3 were arrived at may be conveniently described by considering Glasses 39 to 44, inclusive. Under the criterion of a threshold value for ρ of 0.0006, Glass 39, for which $\rho = 2.5086$, is easily distinguished from Glass 40, for which $\rho = 2.5107$. Glass 39 then falls into a group of one. Glass 40 and Glass 41, for which $\rho = 2.5110$, cannot be discriminated, since their densities are within 0.0006 of one another, so they compose a group of two. Under the same threshold criterion Glass 41 and Glass 42 compose another group of two and Glasses 43 and 44 still another. The same thought processes were applied to all 50 glasses.

The calculation of the probability that two glasses chosen at random from the 50 glasses studied would be indistinguishable in density within the threshold limits of $\rho = 0.0006$ was made as follows:

	10		1		10	
	15		2		30	
There is/are	3	group(s) of	3	having	9	members.
	1		4		4	
	1		6		6	

The total number of group members exceeds 50 because of overlap. If the first sample is taken at random there is

a. A probability of 10:50 that it will belong to a group of one, in which case the probability that the next sample belongs to the same group is 0.

b. A probability of 30:50 that it belongs to a group of two, in which case the probability that the next sample is the other member is 1/49.

c. A probability of 9:50 that it belongs to a group of three, in which case the probability that the next sample belongs to the same group is 2:49.

d. A probability of 4:50 that it belongs to the group of four, in which case the probability that the next sample belongs to the same group is 3:49.

e. A probability of 6:50 that it belongs to the group of six, in which case the probability that the next sample belongs to the same group is 5:49.

Summation of the probability values resulting gives:

$$(30/50 \times 1/49) + (9/50 \times 2/49) + (4/50 \times 3/49) + (6/50 \times 5/49) = 0.0366$$

To account for the overlapping group members the foregoing process is repeated, but with the values of 59 and 58 substituted for the denominator values of 50 and 49:

$$(30/59 \times 1/58) + (9/59 \times 2/58) + (4/59 \times 3/58) + (6/59 \times 5/58) = 0.0263$$

The probability P that the two samples are indistinguishable is:

$$P = (0.0366 + 0.0263)/2 = 0.0314 \text{ or } 3.1\%$$

Combinations of two or more properties are dealt with in the same way.

Proof of Calculations

The method of calculation was subjected to an empirical test to determine its validity. Data on n_D from Table 1, rearranged in order of increasing n_D, were examined to find a series of glasses that presented a serious problem of overlapping groups under the threshold for differentiation of 0.0006. Sixteen glasses ranging in n_D from 1.5169 to 1.5185 were selected and used for the test.

The last two decimal places of the n_D values for the 16 glasses were 69, 70, 73, 74, 75, 75, 77, 77, 78, 79, 80, 81, 81, 81, 83, and 85. Under the criterion of a threshold of 6 ($\times 10^{-4}$), these 16 glasses would be found to compose:

1		6		6	
1		7		7	
2	group(s) of	8	having	16	members
1		9		9	
1		10		10	

Calculations performed as previously described gave a value of 0.79, that is, a 79% chance that two glasses, selected from the 16 glasses considered, would fall within the threshold specified.

The numbers 69, 70, 73, and so on were recorded onto small corks. The 16 corks were placed in a large container and shaken thoroughly, and two corks were withdrawn. The numbers on the corks were recorded and the corks replaced in the container. This procedure was repeated until 200 pairs of draws had been completed. The result was that 148 pairs were found to be within the threshold limits, for a calculated observed value of 0.74, which agrees well with the expected value of 0.79.

A χ^2 test of the hypothesis that the method of calculation used was valid, assuming no bias in the drawing procedure, was applied as follows: the observed frequencies of indistinguishability and distinguishability are, respectively, $o_1 = 148$ and $o_2 = 52$. The expected frequency of indistinguishability and distinguishability based upon calculations are, respectively, $e_1 = 158$ and $e_2 = 42$.

$$\chi^2 = (o_1 - e_1)^2/e_1 + (o_2 - e_2)^2/e_2 = (148 - 158)^2/158 + (52 - 42)^2/42 = 3.01$$

The number of categories $k = 2$. The number of degrees of freedom $\nu = k - 1 = 2 - 1$. The critical value $\chi^2_{0.95}$ for one degree of freedom = 3.84. Since the calculated value does not exceed 3.84 the hypothesis is valid with 95% confidence.

Summary and Conclusions

The conclusions that can be drawn from the results given in Table 3 may be summarized as follows:

1. The power for differentiation is far greater by density than by refractive index. For equivalent thresholds for discrimination, that is, $\rho = 0.0006$ and $n_D = 0.0009$ (three standard deviations in both cases), ρ has a power for differentiation that is approximately six times greater than that of n_D.
2. Dispersion offers little, if any, added value over refractive index.
3. From the foregoing, it follows that if the particle size of glass permits the determination only of refractive index, the results cannot be assumed to have the same proof value as determination of density.
4. If density is determinable, then some value is gained from determining refractive index as well.

Confidence Levels

The estimates of relative proof worth rest principally on discrimination thresholds derived from successive determinations of samples from the same glass source. The results were statistically calculated ranges of experimental error, or discrimination thresholds. For this reason, a high level of confidence can be assigned to the calculated relative estimates. Also involved are the relative ranges found for the properties, for example, 0.040 for density and 0.010 for refractive index. A larger number of study samples would not alter these relative ranges since there is a very high degree of correlation between the two properties.

Calculations at the 95% confidence level for the estimated frequencies of chance indistinguishability based on the 50 samples in the study gave values of 0.031 $\pm$ 0.05 for density and 0.18 $\pm$ 0.11 for refractive index at the respective discrimination thresholds of 0.0006 and 0.0009. To reduce these wide ranges to more acceptable values, say density to 0.031 $\pm$ 0.015 and refractive index to 0.18 $\pm$ 0.035, the statistician would require determinations of glasses from 500 sample sources. Such a large number was impractical for this study. Nevertheless, while the number of samples was lower than desired, the results are believed to give useful rough approximations, which is an improvement over no approximations at all.

Discussion

Cognizance is taken of results reported in the literature [*3,8*] for the high differentiating power of refractive index in the forensic science examination of glass. The results reported in this study show proof worth for refractive index to be substantially different from values expressed by other workers.

The differences can possibly be reconciled through consideration of the methods used for determination, namely, dispersion staining as described in this study versus the Becke line method using a Mettler FP 2 hot stage [*3*], and phase contrast microscopy using a Mettler hot stage [*8*]. The standard deviation of 0.00002 reported for the first method is approximately equal to the differentiating value of the second method, which is 0.00004. This is astonishing when it is considered that the latter has the added advantage of phase contrast microscopy.

Lack of suitable phase contrast capability at this time prevents inquiry directed towards confirming the reported high differentiating power for n_D. It is anticipated that this situation will be remedied in the future. Rationally, the power claimed must be achieved before it can be accepted that refractive index has discriminatory worth equaling that of density as determined in this study. This conclusion is based on the narrow range of glass refractive indices encountered in practice as reflected in the data given—approximately 0.010—as compared to the wider range of glass density values—approximately 0.040. Under the considerations of these relative ranges and the discrimination value of 0.0006 for density determined in this study, the discrimination threshold for refractive index must be on the order of 0.00015 (with a standard deviation of $\pm$0.000025) before refractive index can be said to have a proof value equal to that of density.

Work reported in the forensic science literature alludes to the added value of dispersion. Miller [*7*] cites a selected case to illustrate its worth. Grabar and Principe [*5*] cite literature values for dispersion of optical glass as ranging from 0.007 to 0.023, giving a range of possible values of 0.016. They estimate their range of experimental error as being 0.0016 for $n_F - n_C$ and calculate a discrimination index of 10 based on the ratio 0.016:0.0016, suggesting an increase in value by a factor of 10 by using dispersion.

Both of the works cited lack the quantitative explicitness that would be desired to support a claim of substantial improvement in proof worth through use of dispersion. Surely, such worth is not offered by a selected case in which there is no discrimination threshold specified. What is equally sure is that optical glass is not representative of glass encountered in practice. Also, the assumption of uniform frequency distribution of dispersion cannot be justified.

Concluding Observations

Quantitative studies aimed at showing the proof worth of commonly occurring particulate evidence are recognized as highly desired. Glass, as one kind of evidence, is exceptional in that it lends itself readily to studies of well-known, immutable properties and samples in a practically defined population source are readily obtained.

The results from such studies can be extremely rewarding, since they provide the worker with an objective basis on which he can assess the risks involved in a particular decision. For example, from the results in this study, if the risk the worker is willing to assume is 5% for density (threshold for discrimination: 0.0004), then he at least knows the risk. If he chooses to be more guarded and sets his density discrimination threshold at 0.0006, equivalent to a 0.3% risk, he knows that he can be approximately 15 times more certain of his conclusion.

Other objective rewards accrue. Consider that the distribution of density over the population range is such that 50% of the samples in this study fall within the class interval from 2.485 to 2.495, while only 12% fall within the class interval from 2.475 to 2.485. Occurrence of indistinguishability in the latter class can be reasonably estimated to have a value four times greater than in the former. Additional relative class values can be easily estimated. Consider also that the data permitted the calculation of a coefficient of correlation between density and refractive index. This was 0.98, higher than what would be expected from the literature [*3*]. Such a high correlation would lead one to believe that determining one physical property effectively determines the other. The data show that this is substantially true if density is the value determined, but not the reverse.

A positive result of this work would be if other workers were encouraged to perform studies of their own based on their methods and the properties of glass in their geographical areas. Whether or not their results would substantiate this work, all or in part, cannot be predicted. What is predicted is that we will all gain insight into the forces at play in the matter of proof as developed by the forensic scientist.

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