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Estimation of the Absolute Density of Glass Following the Sink/Float Technique

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ABSTRACT: Following the sink/float technique for the determination of comparative density, the refractive index of a bromoform/bromobenzene mixture may be determined by means of an Abbe refractometer. Reference to a calibration curve of the density and refractive index of bromoform/bromobenzene mixtures will then permit an estimation to be made of the density of the liquid, and by extension, the glass. This represents an alternative to the pycnometer method for the determination of the absolute value of the density. An estimation of the density will permit an opinion to be reached concerning how common or unusual the sample is by reference to a scattergram of an extended set of glass density and refractive index data.

KEYWORDS: forensic sciences, glass, density (mass/volume), refractive index

The comparison of evidence and exemplar glass using density and the refractive index may be accomplished in either an absolute or a comparison mode. In the absolute mode, a value is obtained for each of the two properties. In the comparison mode, values are not developed, but instead a determination is made as to whether the samples share the same, but unspecified, value.

A common comparison mode method for the determination of density is the sink/float method, and a common comparison mode technique for the refractive index is the hot stage immersion liquid method. In this latter technique, if the two samples agree in the temperature at which the Becke line disappears, they are construed to have the same refractive index (within the limits of experimental precision). The absolute value of the refractive index may then be taken from a calibration chart, but some workers elect not to convert temperature into a value for the refractive index.

In the sink/float method, organic liquids, typically bromoform and bromobenzene, (or bromoform and ethylene bromide), are mixed to yield a liquid in which the samples neither sink nor float, but are suspended in the liquid. When both samples are thus suspended, they are construed to have the same density (again, within the limits of experimental precision). The density of the liquid, and by extension the glass samples, can be determined by means of the pycnometer method [*1*], the density plummet and balance method [*2*], or the Mettler (Parr) digital density meter method [*3*]. Many workers, however, use density only in a *comparative* sense, and elect not to determine the *absolute value* for density.

The present authors view the practice of comparing samples without the development of

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absolute values for density and the refractive index as inappropriate to many case situations. Strict comparison is perhaps adequate if the question asked is simply whether the samples share the same value for the two properties. But if the question is refined somewhat to ask what it *means* with respect to the universe of glass evidence if two samples share the same values, then absolute values must be determined and projected against past experience. The authors believe that good professional practice requires that this latter question be posed, rather than the former.

The most extensive glass evidence data set available for forensic science purposes is that compiled by the FBI Laboratory. This data set has been given widespread informal distribution in the form of a computer printout,[2] and various graphical representations of certain of these data have been published [*4,5*]. Figure 1 illustrates a scattergram of 1054 samples of glass from an edited [*6*] set of data from the 1979 edition of the FBI data. Reference to a scattergram of this sort will permit a statement to be made concerning the degree of "commoness" or "unusualness" of a sample of glass. For example, it will be seen that a sample of glass of refractive index 1.5185 and density 2.495 would represent a prosaic, commonly encountered glass, while a sample of refractive index 1.5210 and density 2.5000 would represent a very unusual glass, and therefore of greater probative value as an evidentiary material.

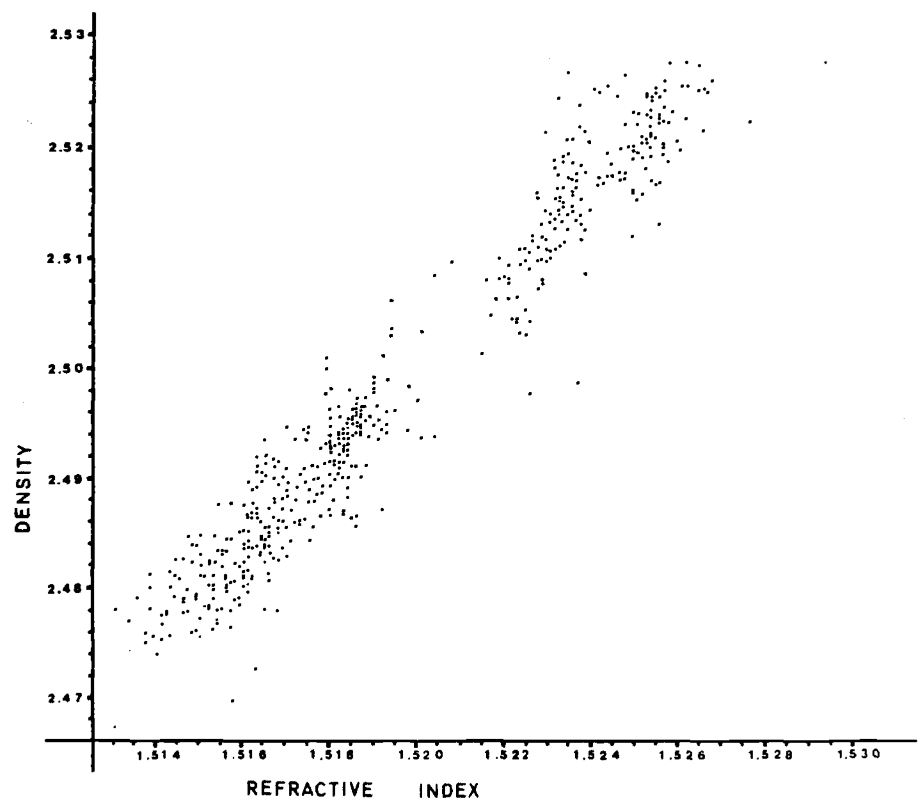

FIG. 1—*Scattergram of 1054 samples of glass from an edited set of data* [4] *from the 1979 edition of the FBI data set. (Not all 1054 data points are apparent because redundant data points are hidden in the plot).*

[2]E. T. Miller, personal communication, FBI Laboratory, 1979.

To make a judgement such as this, however, the absolute values of both refractive index and density must be derived.

In the case of the hot stage immersion method, this is easily accomplished; all that is required is that the worker refer to a calibration curve, that is, a regression line of temperature versus refractive index. These data are easily accessible in the literature [4], and in the case of the Cargille liquids, the value for the change in refractive index with a change in temperature, that is, dR/dT, is placed on the label on the bottle.

In the case of density, it is not so easy. Certainly the absolute density of the liquid (and by extension the glass) can be determined by the pycnometer method or the Mettler density meter method following the sink/float technique. But the Mettler equipment is not available in many operational laboratories and the pycnometer method is tedious, frequently frustrating, and therefore as a practical consequence often omitted. However, it would be possible to refer to the scattergram seen in Fig. 1 and formulate an opinion concerning how common or uncommon the glass is if the analyst could arrive at an *approximation* of the density. The present writers suggest that this can be accomplished in one or two minutes by determining the *refractive index* of the liquid following the sink/float technique by means of a refractometer, and then referring to a regression line of density versus refractive index for bromoform/bromobenzene mixtures.

Experimental Procedure

Reagents

The reagents used were bromoform, Fisher Purified Grade, Lot 74428, (BP) 149.5°C, density (20°C) 2.8899, refractive index (20°C, 589 nm) 1.5976 and bromobenzene, Fisher Certified Grade, Lot 771104, BP 156°C, density (20°C) 1.4950, refractive index (20°C, 589 nm) 1.5597.

Equipment

The equipment used were a Mettler 600-g pan balance, accurate to 0.1 mg; an Abbe refractometer, accurate to 0.0001 units; and a Fisher 2-mL pycnometer, accurate to 0.01 mL.

Method

The pycnometer method used was basically that of Kirk [1]. Bromoform/bromobenzene mixtures were prepared in 15-mL screw capped vials by using 1- to 5-mL pipettes to create ratios from 10:1 to 1:10 bromoform/bromobenzene. The neat solutions of bromoform and bromobenzene were also included, making a total of 15 liquids to be tested.

Density determinations were accomplished by comparing the tare weight of a 2-mL pycnometer to the average of two weight determinations on successive fillings of the pycnometer, taken at each concentration ratio. Weight values were recorded to 0.1 mg. The large volume pycnometer (2 mL) was used to reduce the weighing error to an insignificant fraction of the total weight of the filled pycnometer. Single refractive index measurements were taken on samples at each concentration ratio by taking a small aliquot (two to three drops) from the pycnometer and reading the refractive index with the Abbe Refractometer after the weight determinations were completed.

Sample preparation, weight, and refractive index determinations were carried out at a temperature of 25°C ± 0.5°C. Since the purpose of this work is to provide a density calibration curve to be used at nominal room temperature, no attempt was made to standardize measurements at the unrealistic temperature of 20°C.

Results

Table 1 illustrates the respective density and refractive index of the 15 ratios of bromoform and bromobenzene. Figure 2 illustrates a curve of density plotted against refractive index for mixtures of bromoform and bromozene. It should be clearly understood that this curve is not a plot of *glass* density and the refractive index, although it may be used to *estimate* the density of a sample of glass given the refractive index of the bromoform/bromobenzene mixture which matches the density of the glass.

Discussion

Figure 2 may be used to estimate the density of a sample of glass for the purpose of approaching a scattergram of an extended data set such as depicted in Fig. 1. Following the sink/float method, the refractive index of the bromoform/bromobenzene mixture may be determined easily with an Abbe Refractometer. If, for example, the refractive index of the mixture was determined to be 1.5838, then the density of the liquid, and by extension the glass, would be estimated from this curve as 2.4937.

The slope of the line in Fig. 2 was determined by standard regression analysis of the data illustrated in Table 1. Using the slope so derived, Fig. 2 is restricted to only those values of density that are to be expected in any analysis of glass evidence. Stated differently, Fig. 2 represents only one third of the entire plot of the tested mixtures of bromoform and bromobenzene; since density values below 2.467 are not to be expected, however, Fig. 2 represents an expansion of the plot from density 2.46 to 2.53. The remaining data, although contributing to the accuracy of the slope of the curve, are not further relevant to a discussion of glass evidence.

The estimate of density may be alternatively derived by employing the regression equation which describes the plot in Fig. 2. The regression equation was calculated to be:

$$\text{Density} = 37.2982(\text{refractive index}) - 56.5784 \quad (1)$$

TABLE 1—*Density and refractive index values of ratios of bromoform/bromobenzene ratios. The density value represents the average of two successive weight determinations versus the tared weight of the pycnometer. The values are for 25°C ± 0.5°C, and the refractive index is for a wavelength of 589 nm.*

Bromoform, mL	Bromobenzene, mL	Ratio, Bf/Bz	Density, g/cm^3	Refractive Index
10	1	0.91	2.7762	1.5911
5	1	0.83	2.6514	1.5890
4	1	0.80	2.6050	1.5865
3	1	0.75	2.5352	1.5848
2	1	0.67	2.4158	1.5819
3	2	0.60	2.3302	1.5792
2	2	0.50	2.1881	1.5753
2	3	0.40	2.0538	1.5717
1	2	0.33	1.9549	1.5692
1	3	0.25	1.8536	1.5663
1	4	0.20	1.7732	1.5644
1	5	0.17	1.7330	1.5632
1	10	0.09	1.6144	1.5605
neat	0	1.00	2.8787	1.5941
0	neat	0.00	1.4934	1.5575

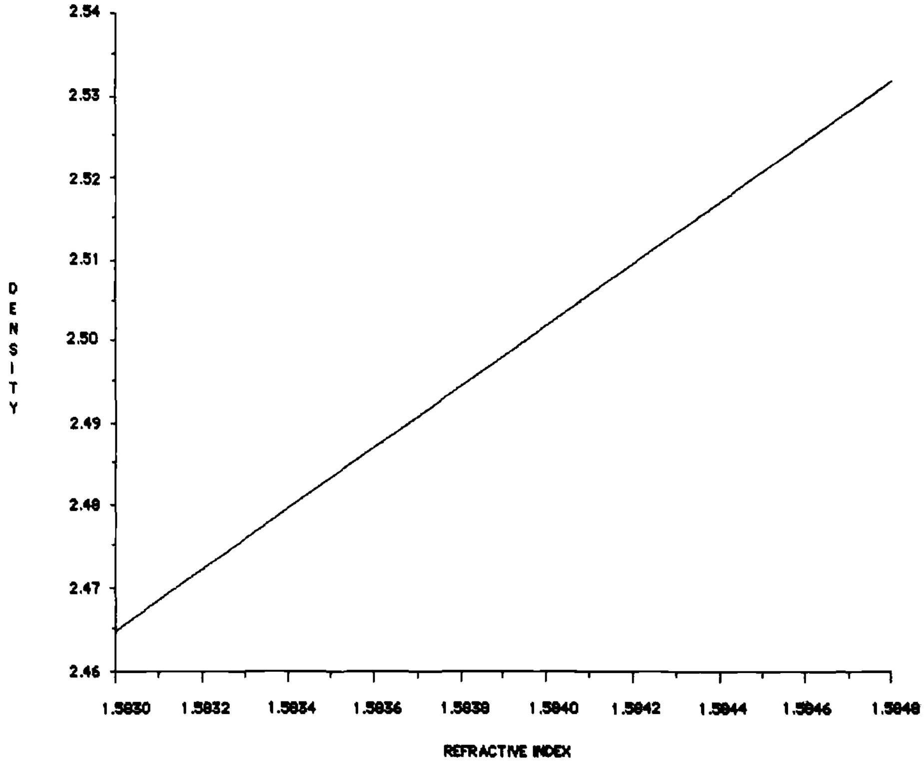

FIG. 2—*Regression line of the density and the refractive index of bromoform/bromobenzene mixtures in the region represented by glass evidence. Following the sink/float method. the refractive index of the mixture may be determined by means of a refractometer. Reference to this regression line, or, alternatively. to the regression equation, will provide an approximation of the density of the liquid and therefore of the glass.*

Upon determination of the refractive index of the bromoform/bromobenzene mixture, an estimated value for density may be calculated directly from this equation.

Conclusion

For those who, for whatever reason, are unable to apply the Mettler density meter technique or the density plummet technique, or who are reluctant to apply the pycnometer technique for the absolute determination of density following the sink/float method, the approach described here may provide a practical alternative. A determination of the refractive index of the bromoform/bromobenzene mixture by means of the Abbe Refractometer is a quick and easy procedure. From the calibration curve of density and the refractive index of bromoform/bromobenzene mixtures (Fig. 2), the density may be estimated. Reference then to a scattergram of an extended set of density and refractive index values (Fig. 1) will permit the analyst to formulate an opinion as to how common or usual the sample is.

The authors do not suggest that this approach is an acceptable substitute for the pycnometer, the plummet method, or the Mettler density meter method for the purpose of determining whether two samples could have originated from the same source. Stated differently, if any interpretation concerning provenance is to be made by comparing numbers, then those numbers should be determined as precisely as possible rather than simply estimated. If, however, the two samples are *already proven* to have the same comparative density (by means of the sink/float method), then this approach will provide the opportunity to project the samples under consideration against an existing data set.

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