Glass Density Determination

Scientific Working Group for Materials Analysis (SWGMAT)

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1. Scope

This document describes the guidelines for density determination as it relates to forensic glass analysis. The guideline outlines density measurement using density gradient columns, analytical balance and plummet, density meter, and sink/float (comparative) methods. The guideline does not purport to address all of the safety problems, if any, associated with density determinations. It is the responsibility of the user of this guideline to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to analysis.

2. Reference Documents

2.1. Scientific Working Group for Materials Analysis Documents

Trace evidence recovery guidelines
Quality assurance guidelines

2.2. American Society for Testing and Materials Standard

C 729 Standard Test Method for Density of Glass by the Sink-Float Comparator
E1492 Practice for Receiving Documenting, Storing, and Retrieving Evidence in a Forensic Laboratory

3. Terminology

Density is mass per unit volume (g/cm^3).

Density gradient is a column containing a liquid mixture that undergoes a gradual decrease in density from the bottom level to the top level.

Density meter is an electronic device for measuring density.

Plummet is a sealed, immersible chamber of known volume and density.

Temperature controlled column is an item specifically made to contain density liquid and remain at a constant temperature.

4. Summary of Guideline

The density of an unknown sample of glass is determined by placing a glass sample into suspension in a liquid solution. The density of that liquid is then either measured directly or used comparatively with another sample of glass.

5. Significance and Use

5.1. Density, a fundamental property of glass, varies with changes in composition and thermal history. Density determined either by direct measurement or comparison is suggested if elemental analysis is not available. It is impractical to measure density on glass fragments smaller than two to three millimeters. If two samples of glass can be

differentiated by density, they could not have originated from the same source. Further limited discrimination may be possible by doing a density comparison in conjunction with refractive index comparison.

5.2. Density may be used as a screening technique where large numbers of larger fragments are encountered. This may be particularly useful in identifying multiple sources present in the known and/or questioned samples.

6. Sample Handling

6.1. Proper sample preparation is a prerequisite for obtaining reliable results. Density measurements are not appropriate for fragments of glass that contain inclusions or cracks or when comparing a surface fragment to a bulk fragment.

6.2. The variability of the known sample should be determined when possible. An effort to sample the known glass may include breaking a fragment of tempered glass and including fragments from the bulk and surface to compare to the unknown.

6.3. Density determinations and comparisons between known and questioned samples should be made using fragments of approximately equal size.

6.4. The samples must be clean and dry before proceeding. Cleaning may be accomplished in a number of ways, such as rinsing with laboratory-grade detergent solution followed by several rinses of deionized water in order to remove dust and dirt. The samples must be dried following cleaning. (See Section 6.5 in the *Scientific Working Group for the Materials Analysis Collection, Handling, and Identification of Glass.*)

7. Analysis

7.1. Density Gradient Column Method (Kirk 1951)

7.1.1. Scope

The method involves placing, in a vertical glass tube, a liquid containing a gradient of density. The gradient is such that the density at any level is less than that at any level lower in the tube and greater than that of any level higher in the tube. When glass fragments are introduced to the column, each will become suspended in the liquid at the level that is the same density as that glass fragment. Fragments of different density will settle to different levels in the column.

7.1.2. Materials and setup

Gradient tubes are usually 25cm to 45cm in length and 6mm to 18mm in diameter. A heavy liquid, such as 1,4 dibromobenzene or bromoform, is mixed with a lighter liquid, such as bromobenzene or ethanol, in varying proportions to form a density gradient. For most purposes, about five layers of liquids are used. The bottom layer of the density gradient tube consists of heavy liquid only. The second layer consists usually of three parts of the heavy liquid to one of the light. The third consists of equal mixtures of heavy and light liquids. The fourth layer is made of three parts light liquid and one part heavy liquid. The top layer consists of light liquid only. Each layer is added to the prior very slowly using a pipette so as to not allow mixture at the interface. The bottom layer is typically about a quarter of the total height of the column. The second, third, and fourth layers should each be about half the height of the first layer. The top layer should be the same height as the bottom layer. The gradient tube should stand overnight before being used so

that the liquids will diffuse into each other to form a gradient.

7.1.3. Use

The fragments should be properly documented prior to their addition into the density gradient column so as to facilitate identification when they are recovered from the gradient. The fragments to be compared are gently placed in the density gradient and allowed to settle completely. The position of the glass in the column may be better viewed using back-illumination. Care should be taken to avoid changes in the temperature of the column.

7.2 Sink/Float Method

7.2.1. Comparative Method

7.2.1.1. Scope

The method involves suspending glass fragments in a density solution within a constant temperature bath. If two or more fragments are suspended, their densities are the same. The numeric density value is not determined in this method but can be determined using the absolute density determination methods described in Section 7.2.2. of this guideline.

7.2.1.2. Materials and setup

A temperature-controlled density column containing a mixture of bromobenzene and bromoform is typically used.

7.2.1.3. Use

Place the glass fragments to be compared in the density column after temperature equilibrium has been achieved. Add small amounts of the density liquids until at least one fragment becomes suspended.

7.2.2. Absolute density determination methods

After glass fragments are suspended in a density solution using the sink/float method, a numeric determination can be made using the following methods.

7.2.2.1. Analytical balance and plummet method (Koons et al. 2002)

7.2.2.1.1. Scope

The method involves measuring the suspending density solution using an analytical balance and weighed plummet.

7.2.2.1.2. Materials and setup

An analytical balance capable of accurately determining weights within 0.0001g with a tall weighing chamber and a plummet are required. The plummet must sink in a liquid whose density is above 2.6g/cm^3 (Beverage and Semen 1979). Analyze a reference glass sample of known density prior to each use to ensure that the system is operating within acceptable parameters.

7.2.2.1.3. Use

Determine the volume of the plummet by weighing it in air and then in water at a known temperature. Place the glass fragment in the density

column and suspend it using the density liquids. Weigh the plummet immersed in the density column. Calculate the density of the liquid and the glass (Koons et al. 2002).

7.2.2.2. Density meter method (Beveridge and Semen 1979)

7.2.2.2.1. Scope

The method involves measuring the density of a suspending liquid mixture using a density meter.

7.2.2.2.2. Materials and setup

A density meter with its sample chamber at the same constant temperature as the density column is required (to +/- 0.1 degrees Celsius). Analyze a reference glass sample of known density prior to each use to ensure that the instrument is operating within acceptable parameters.

7.2.2.2.3. Use

A portion of the suspending density liquid mixture is transferred into a calibrated density meter and the density is recorded.

8. Considerations

8.1. Glass samples exhibit a range of density values. Density is a function of the chemical composition of the glass and its thermal history. The composition of a glass sample can be measured by a variety of techniques. Thermal history is typically assessed through a measurement of either refractive index or density.

8.2. The variations in either refractive index or density at fixed values of the other indicate that even with precise measurement of one property, the other is capable of providing some degree of additional discrimination. If density and refractive index are used in conjunction when one parameter is measured, the second gives an improvement in discrimination capability of approximately twofold (Koons et al. 2002; Stoney and Thornton 1985).

8.3. Because there is a high correlation between density and refractive index, a limited amount of additional significance can be placed on an association based on the combination of these techniques.

8.4. Samples that are distinguished by density did not originate from the same source.

9. References

Beveridge, A. D. and Semen, C. Glass density measurement using a calculating digital density meter, *Canadian Society of Forensic Science Journal* (1979) 12(3):113-116.

Kirk, P. L. *Density and Refractive Index:* Their *Application in Criminal Identification*. American Lecture Series. Publication 112. American Lectures in Public Protection. Thomas, Springfield, Illinois, 1951.

Koons, R. D., Buscaglia, J., Bottrell, M., and Miller, E. T. Forensic glass comparisons. In: *Forensic Science Handbook*. 2nd ed. R. Saferstein, ed. Prentice-Hall, Upper Saddle River, New Jersey, 2002, Volume 1, pp.161-213.

Stoney, D. A. and Thornton, J. I. The forensic significance of the correlation of density and refractive index in glass evidence, *Forensic Science International* (1985) 29:147-157.