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Forensic Glass Comparison: Background Information Used in Data Interpretation

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Introduction

Glass can be found in most localities. It is produced in a wide variety of forms and compositions, and these affect the properties of this material. It can occur as evidence when it is broken during the commission of a crime. Broken glass fragments ranging in size from large pieces to tiny shards may be transferred to and retained by nearby persons or objects. The mere presence of fragments of glass on the clothing of an alleged burglar in a case involving entry through a broken window may be significant evidence if fragments are found. The significance of such evidence will be enhanced if the fragments are determined to be indistinguishable in all measured properties from the broken window. On the other hand, if the recovered fragments differ in their measured properties from the glass from the broken window, then that window can be eliminated as a possible source of the glass on the subject's clothing (Koons et al. 2002).

Glass is technically defined as "The inorganic product of fusion which has cooled to a rigid condition without crystallizing" (ASTM C162-05). Glass is most accurately defined by its atomic structure. In contrast to crystalline solids, which have an ordered internal arrangement of atoms, the internal structure of glass consists of a network of atoms lacking long-range symmetry. This condition is referred to as the *vitreous*, or *glassy*, state (Varshneya 1994).

The vast majority of the raw materials used for making glass are derived geologically. North American glass manufacturers annually use more than 20 million tons of raw materials (Carr 1994). In addition to the geologically derived raw materials, recycled broken glass, or *cullet*, is added to the batch to act as a flux, and some manufacturers use synthetic soda ash (Na^2CO^3) (Guttman 1996). All of these materials contain impurities that can produce measurable variations in the final glass product.

The raw materials for glass manufacturing are first mixed together to form a batch and then melted in a furnace to produce liquid glass. Most modern, commercially produced glass is manufactured in a nonstop process wherein raw materials are fed continuously into one end of a melting tank and liquid glass is drawn from the other end. A typical melting tank can contain up to 2000 tons of liquid glass with a throughput of several hundred tons per day. The composition of the glass changes gradually as more raw materials are added (Arbab 2005).

The time required to completely flush a particular glass from the tank during continuous production can be days to weeks, and the glass that is produced in the transition will have an intermediate composition (Arbab 2005). Some specialty glasses such as optical glass, novelty glass, or glass that is difficult to melt are produced in pot furnaces or day tanks. These furnaces are capable of producing a limited amount of glass, ranging from one to five tons of glass per day (Tooley 1974).

After the glass is formed, it is cooled in a controlled manner through the glass transition range in an annealing oven, or lehr, to avoid cracking at lower temperatures. Various fabrication methods can be employed after the glass is formed, for example, tempering or lamination.

In tempering, the surface of the glass is placed in compression by either chilling it while the glass object is cooling through the glass transition range or through ion exchange on the surface of the glass. Tempered glass is typically four to five times more resistant to breakage than nontempered glass (Varshneya 1994). Heat strengthening is a form of tempering wherein the glass is chilled to a lesser degree. Heat-strengthened glass is usually about twice as strong as nontempered glass.

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Laminated glass is produced by heat-sealing thin layers of plastic between two or more panes of heat-strengthened glass. In the United States, laminated glass must be installed in the windshields of vehicles, and tempered glass must be installed in the side and rear windows (U.S. Department of Transportation, Code of Federal Regulations [CFR], 49 CFR 571.205).

Although modern glass manufacturing is a highly automated process that produces glass with large-scale uniformity, minor variations in the properties of the resulting glass remain. Each of the raw materials used to produce glass contains impurities that are uncontrolled by the manufacturers and consequently vary in amount and composition over time. The mixing of raw materials during batching is incomplete, and the batch will unmix during transport and delivery to the furnace. Some mixing occurs as the molten glass flows through the furnace, but it is not sufficient to make an absolutely uniform product. The refractory materials lining the glass furnace are gradually eroded into the glass melt over the lifetime of the furnace. These factors result in glass products with small but measurable variations in their chemical, optical, and physical properties both within and between production runs (Koons et al. 2002).

Transfer and Persistence

When a glass object breaks, fragments can be ejected from the object in all directions (Pounds and Smalldon 1978), including backward toward the direction of the breaking force (Nelson and Revell 1967). During experimental studies, glass fragments have been recovered from up to four meters away from a breaking glass object (Francis 1993; Locke and Unikowski 1991). Glass fragments can be transferred onto anything within this distance. The number of glass fragments that can be transferred is controlled by a number of factors:

- The closer something is to the breaking glass, the more likely it is to have glass fragments transferred to it (Allen and Scranage 1998). The number of fragments transferred decreases with distance from the break (Pounds and Smalldon 1978).
- The person breaking a window will have more glass on him or her than a bystander, and the more blows required to break out the glass, the more glass that will be transferred (Allen et al. 1998b).
- The number of glass fragments generated by a break is independent of the size and thickness of the window but increases with greater damage to the glass (Locke and Unikowski 1992).

Whether the glass that is deposited on clothing persists to be recovered by a forensic examiner depends upon additional factors:

- Less glass is retained on slick clothing, such as nylon jackets, than on rough clothing, such as wool sweaters. Wet clothing retains more glass than dry clothing (Allen et al. 1998b).
- Glass fragments fall off clothing over time, and larger pieces fall off before smaller pieces (Cox et al. 1996a, 1996b, 1996c; Hicks et al. 1996; Hoefler et al. 1994).
- Glass falls off faster if the person wearing the clothing is active (Batten 1989; Cox et al. 1996c; Hicks et al. 1996).

It should be noted, however, that the transfer and persistence of glass is highly variable, and the total number of fragments transferred in a single type of experiment can vary by an order of magnitude or more (Curran et al. 2000).

The above studies chiefly involve primary transfer—a transfer from the broken glass object to something else. Primary transfer also can occur when a person or object comes into contact with previously broken glass (Allen et al. 1998a). Additionally, there can be secondary transfer of glass between people and objects, such as when glass is transferred from a person to a vehicle seat (Allen et al. 1998c). During a glass examination, it cannot be positively determined whether the glass fragments found on an object were acquired through primary transfer, secondary transfer, or through contact with previously broken glass (Koons et al. 2002).

Recent studies have been conducted to determine the background level of glass on clothing from randomly selected individuals. In 1997, Lau et al. examined 213 garments and reported that 1% of upper garments and 3% of lower garments had fragments on their surface. Of the garments with glass, 5 had only one fragment, and 1 garment had two fragments. Petterd et al. (1999) found one glass fragment each on only 6 of 2008 upper garments examined. Roux et al. (2001) examined 776 pairs of footwear and found that 5.9% had glass embedded in the sole, 1.9% had glass on the uppers, and only 0.3% had glass on both the soles and uppers.

These studies and many others (Davis and DeHann 1977; Harrison 1978; Harrison et al. 1985; Lambert et al. 1995; McQuillan and Edgar 1992; McQuillan and McCrossan 1987; Pearson et al. 1971; Zoro and Fereday 1982) demonstrate that it is unlikely that glass fragments will be found on people who have not been present when a glass object is broken or who have not come into contact with broken glass. Nevertheless, it is still a reasonable possibility, so characterization of the glass is useful.

Analysis

A forensic glass analysis is typically a comparison of two or more glass fragments in an attempt to determine if they originated from different sources. Less frequently, it is a question of determination of end use, or glass provenance. For example, a provenance question might be: Is this fragment of broken glass from a light bulb? These analyses require the determination of class characteristics that may associate objects with a

group of similar objects such as containers, but never to a single object. It is important to note, however, that although there may be several objects with identical properties, glass fragments can originate only from broken and not intact objects. Only physically matching two or more broken glass fragments allows for their association with each other to the exclusion of all other sources (Scientific Working Group for Materials Analysis [SWGMA] 2005c).

Every analytical test available is not always performed on each specimen. There are several reasons for this occurrence. The aim of a comparative glass analysis is to exclude possible sources. When a difference is detected, no further comparison is necessary. Additionally, sampling is typically beyond the control of the forensic scientist and is subject to what happens to be preserved on suspects, on victims, and at crime scenes. The glass must then be recovered.

It is not always possible to assess every potential point of comparison in each glass specimen because not all fragments transferred, recovered, and submitted for forensic analysis will express every feature. If a fragment does not possess a particular feature, it is not necessary to assess that feature in comparison specimens. For example, not all glass fragments will possess original surfaces. Without original surfaces, it is impossible to determine the thickness of the glass. It would therefore be pointless to measure thickness in one glass fragment if the fragment it was being compared with does not possess original surfaces.

Finally, a glass fragment may be too small to be analyzed with reproducible results even when a feature is preserved. Consequently, the actual tests performed on a set of specimens depend on the size and shape of the glass fragment, as well as analytical considerations.

For a typical examination, after determining that the specimens in question are actually glass, their physical properties are assessed. The optical properties of the specimens are measured next. In some laboratories, glass density is measured either in addition to or instead of measuring optical properties. Chemical composition of the glass is typically measured last. If at any point a difference is noted between comparison specimens, it is no longer necessary to continue the analysis. The two specimens have been shown to have different sources.

Initial Examinations

Because there are many materials that can be mistaken for glass at a casual glance, it is first necessary to determine if the specimen is actually glass. Many disciplines address this question, most notably the fields of igneous petrology and gemology. Methods typically employed by igneous petrologists and gemologists to identify glass include the observance of conchoidal fracture, determination of hardness, reaction to a hotpoint, microscopy, and spectroscopy (Gemological Institute of America [GIA] 1996; GIA 1998; Hatch et al. 1972; Hurlbut and Klein 1977; Kerr 1959; Williams et al. 1982). Although a glass mimic may have some of the same characteristics as glass, it is not possible to duplicate all of the characteristics of glass (GIA 1996, 1998). Forensic scientists use many of these same techniques in their examinations (SWGMA 2005e).

The physical properties used for comparison include glass color, fluorescence, thickness, surface features, and curvature. These can be assessed using various methods. These tests are rapid and nondestructive.

Color

Materials can be added to the batch to produce glass in practically any color (Tooley 1974). Alternately, impurities present in the raw materials used to produce glass can impart unintentional color (Doyle 1994). Differences in color represent a change in glass chemistry and can be used to differentiate specimens.

The assessment of color in glass can be difficult. It is typically not possible to reliably perform colorimetry on glass fragments in forensic casework because the fragments usually are too small and their color density is too low. Color assessment is performed visually against a white background in natural light with the particle on edge. Side-by-side comparison should be used with similarly sized particles (Koons et al. 2002; SWGMA 2005e).

Fluorescence

Many glass specimens will fluoresce when exposed to short-wave (~254 nm) and/or long-wave (~350 nm) ultraviolet light. This fluorescence can be used as a basis to differentiate glass specimens (Lloyd 1981). Fluorescence throughout the body of a piece of glass can be caused by the presence of certain elements such as uranium in chrome-green glass (Tooley 1974).

During the production of modern glass manufactured using the float process, liquid glass is poured onto a bath of molten tin (Doyle 1994). The glass surface that was in contact with the tin bath will fluoresce when exposed to short-wave ultraviolet light. This fluorescence is caused by the uptake of tin on one side of the glass during manufacturing. It has been reported that some coatings applied to glass will also fluoresce (Stoecklein 1996).

As with the assessment of color, visual comparisons of fluorescence should be performed side by side. Fluorescence examinations can also be performed using fluorescence spectroscopy on specimens as small as 0.05 mm² (Lloyd 1981). Fluorescence on a glass surface will be detected only if the surface that will fluoresce is preserved, collected, and analyzed.

Thickness

ASTM International (ASTM), formerly known as the American Society for Testing Materials, Standard C1036-06 lists the tolerances for standard thicknesses of flat glass. The tolerances for flat glass in the thickness ranges typically encountered in forensic casework are on the order of ±0.25 mm (ASTM C1036-06), but the range observed in casework for a pane of flat glass is usually much lower (Koons et al. 2002).

Thickness is usually tightly controlled by the manufacturer, on the order of thousandths of an inch in a single sheet. Variations in thickness can produce unsightly ripples in the glass sheet, lessening the market value of the glass. Thickness is a function of the viscosity of the glass. Viscosity can be changed only by changing the glass composition or the furnace temperature (Tooley 1974; Varshneya 1994). Changing the glass composition can produce other, even less desirable, changes in the end product, and increasing the furnace temperature is expensive because of increased fuel costs (Greenman 2008; Tooley 1974).

Manufacturers can measure thickness continuously along a ribbon of flat glass using radioisotope thickness gauges (Doyle 1994). Thickness in the forensic laboratory can be measured using a micrometer or caliper but requires that fragments possess both original surfaces. Because thickness produces a quantitative measure, the precision and accuracy of the micrometer must be considered when evaluating the results of thickness measurements. When the thickness of a piece of flat glass is measurably different from the range expressed in the known glass standard, those glasses can be determined to have come from different sources.

Surface Features

Certain surface features are imparted during glass manufacturing and fabricating processes or during use. These features can include mold and polish marks, mirrored backings, scratches, and decorative finishes such as texturing, etching or frosting, and coatings. The majority of these features can be compared visually using a stereomicroscope, but coatings are not usually apparent to the naked eye and may require sophisticated instrumentation for detection and comparison. Transmission electron microscopy (Bravman and Sinclair 1984), X-ray scattering (Misture 1999), atomic force microscopy (Arribart and Abriou 1999), and Fourier transform infrared spectroscopy (DeRosa and Condrate 1999) have all been used for the analysis of glass coatings. These features also can be used to discriminate between glass objects.

Curvature

Whether or not a fragment of glass is flat or curved often can be determined visually with the aid of low-power magnification. For small particles, interferometry can be used (Locke 1984). The curvature of glass can be used as a point of comparison and as a method of determining a broad product type.

Optical Properties

Refractive index (n) is a unitless measure of the speed of light in a transparent medium and is defined by Snell's law as the ratio of the velocity of light in a vacuum to the velocity of the wave in the transparent medium (Stoiber and Morse 1981). Refractive index is a function of chemical composition and atomic arrangement (Stoiber and Morse 1981). In glass, these are controlled by the composition of the batch (chemical composition) and the cooling history of the glass (atomic arrangement) (Varshneya 1994). Tempering changes the cooling rate of the surface of the glass relative to the interior and imparts a continuous change in the refractive index from the surface to the center of the glass (Varshneya 1994).

Refractive index is the most commonly measured property in the forensic examination of glass fragments (Koons et al. 2002), because:

- Precise refractive indices can be measured rapidly on the small fragments typically found in casework.
- It can aid in the characterization of glass.
- It provides good discrimination potential. (Koons et al. 2002)

Refractive index varies with wavelength of light and temperature (Bloss 1961). Dispersion is the change in refractive index with a change in wavelength of illumination. For glass, relative dispersion, or dispersive power, is used to quantify dispersion (Koons et al. 2002). Relative dispersion (V) is defined as the difference between the refractive index at different wavelengths of light, typically n_C (486 nm), n_D (589 nm), and n_F (656 nm), which is mathematically expressed as

$$V = (n_D - 1)/(n_F - n_C) \quad \text{(Bloss 1961).}$$

Refractive index and dispersion can be measured by many methods. The precision refractometer measures indices only at the surface of the glass (Skoog and West 1980). A V-block refractometer can measure only the average refractive index through a block of glass. Although both methods when applied appropriately are accurate to six decimal places, they require the use of large polished glass samples. Samples adequately large for these methods are rarely encountered in forensic casework (Koons et al. 2002).

Immersion Methods

Immersion methods are used to measure refractive index in some laboratories. These methods take advantage of the fact that when using monochromatic light, a particle immersed in a liquid of identical refractive index will become invisible (Bloss 1961). The particle is viewed through a microscope. A classic technique used is called the Becke line method (Bloss 1961; Kerr 1959; Nesse 1986; Stoiber and Morse 1981). As summarized in the SWGMAT Glass Refractive Index Determination guideline (2005d):

In the Becke line method, a bright halo (Becke line) is observed around the particle. Movement of the Becke line with respect to the particle on changing the microscope focus indicates refractive index of the particle relative to the immersion oil. The amount of contrast between the particle and the immersion liquid indicates the magnitude of the difference in refractive index. The fragment is then removed from the liquid, washed, and placed in another liquid with a refractive index closer to the match point. This process is repeated until

the refractive index of the match point has either been reached or bracketed by two oils. When the match point is approached, the results can be plotted on Hartmann dispersion nets, which allows for the extrapolation of the results between liquids. (SWGMA 2005d)

Dispersion staining is very similar to the Becke line method. In dispersion staining, a stop is placed in the back focal plane of the objective. Slight differences in refractive index between the particle and the liquid are seen as colored halos. The color of the halo is characteristic of the difference in wavelength to the refractive index match point. As with the Becke line method, particles can be recovered, cleaned, and placed in another liquid closer to the match point until the match point is identified or bracketed. When the match point is approached, the results can be plotted on Hartmann dispersion nets, which allows for the extrapolation of the results between liquids (McCrone et al. 1997).

Both the Becke line method and dispersion staining method produce rapid results using equipment that is readily available in most laboratories: a microscope and calibrated liquids. The results of these methods can be reported with certainty only to ± 0.001 under the best conditions, but are typically less reliable in actual use (SWGMA 2005d). Variability in refractive index across a 12-foot-wide ribbon of flat glass is approximately 0.0001 (Almirall 1996) to 0.0002 (Underhill 1980). Variability from the interior to the glass surface is 0.003 (Davies et al. 1980). The expected variation within a single float source is in the range of ± 0.00004 for annealed glass and ± 0.0016 for tempered glass (Locke et al. 1985). Becke line and dispersion staining methods cannot, therefore, measure the true variability of glass and may not be able to differentiate between glasses with similar but genuinely different refractive indices. Because of this, they are not typically used in forensic glass comparisons, although they are excellent screening techniques to rapidly discriminate between glasses with very different refractive indices.

Emmons Double Variation

R. C. Emmons first described the double-variation method in 1928 (Emmons 1928). He suggested the use of a monochromator and hot stage to allow for the variation of temperature and wavelength simultaneously. This method was formally described in the *Official Methods of Analysis of the Association of Official Analytical Chemists (AOAC)*, “Method 973.65, Characterization and Matching of Glass Fragments: Dispersion Microscopy (Double Variation Method)” (Association of Official Analytical Chemists 1990). In this method, a phase-contrast microscope converts the difference in index between a particle and the immersion liquid into a difference in brightness contrast. This brightness contrast enhances the Becke line (Abramowitz 1987). Although the AOAC method describes varying temperature while holding wavelength constant for multiple wavelengths, in practice, because of problems with thermal lag in hot stages, most practitioners hold temperature constant while varying wavelength for multiple temperatures.

For the Emmons double-variation method, fragments of glass are mounted in the previously calibrated, appropriate immersion liquid on a glass microscope slide and covered with a glass cover slip. The slide is inserted into a hot stage mounted on a phase-contrast microscope. The hot stage is set to a temperature within the stable range of the liquid, and the wavelength of the monochromator is adjusted until the match point is reached. The match point and temperature are noted. This process is repeated for at least two additional temperatures.

By comparing these measured points with the previous calibration data for the liquid, the refractive index of the particles at a particular wavelength—normally n_C , n_D , and n_F —can be calculated or determined graphically. Results are typically reported to the nearest 0.0001 (SWGMA 2005d). The precision of the method is approximately 0.00004 to 0.00006 (Cassista and Sandercock 1994).

Automated Method

An automated method for refractive index determination of glass fragments using a phase-contrast microscope, hot stage, and monochromatic light source has been published by ASTM [ASTM E1967-98(2003)]. In this method, a video camera captures the image of the particle edge, and a computer calculates the point of minimum contrast—the match point—across the particle edge while automatically varying the temperature. This method parallels the AOAC method in that the wavelength is fixed and the temperature varies.

As with the Emmons double-variation method, fragments of glass are mounted in the previously calibrated, appropriate immersion liquid on a glass microscope slide and covered with a glass cover slip. The slide is inserted into a hot stage mounted on a phase-contrast microscope. The temperature is adjusted so that the refractive index of the liquid is higher than that of the glass sample. The instrument lowers the temperature of the preparation through the match point for the glass. The contrast between the fragment and the liquid is monitored, and the match point is noted automatically. This process is repeated as the temperature is then raised through the match point.

These values are recorded as the *match temperature on cooling* and the *match temperature on heating*, which are averaged to give the *match point temperature* for the sample. The refractive index of the sample is calculated automatically from the calibration data (SWGMA 2005d). The precision of the automated method of glass refractive index measurements determined from repeat measurements of n_D of an optical glass is 0.00002 (Locke 1985), which is typically better than the measurable variation of a glass object.

Density

Density is mass per unit volume. Like refractive index, density is a function of chemical composition and atomic arrangement, which are controlled by the composition of the batch and the cooling history of the glass, respectively (Varshneya 1994). The typical density variation measured in a bottle is 0.002 g/cm^3 , and the typical density variation measured across a sheet of glass is 0.001 g/cm^3 (Koons 2002). Density in glass

can be assessed either quantitatively by direct measurement or qualitatively by simultaneous comparison of two or more specimens.

Density measurements are performed less frequently than refractive index determinations because:

- The glass fragment must be scrupulously clean and free of inclusions.
- Accurate density measurements require a sample that is two to three millimeters in diameter (SWGMA 2005b), much larger than particles typically encountered in forensic casework. Additionally, particles of this size are suitable for chemical analysis, which is a more discriminating technique.
- Density and refractive index are correlated in the majority of glass samples (Smalldon and Brown 1973). Refractive index determinations are more rapid and can be performed with smaller samples, so most laboratories choose to perform refractive index determinations first.
- Until recently, density measurements required the use of hazardous liquids, such as bromoform (Koons 2002).

Most quantitative density measurements are performed in forensic laboratories using a density meter. The density of a solution can be changed by adding small amounts of miscible liquids with different densities. When a particle in this solution is suspended, the density of the solution will match the density of the particle. The density of the liquid is then measured using a density meter. The precision of digital density meters is 0.0001 g/cm³ (Beveridge and Semen 1979), which is better than the measurable variation of a glass object.

Relative density determinations can be made by using density gradients (McCrone and Hudson 1969). Density gradients are seldom used, however, because density gradients are difficult to make and cannot be reused (Koons 2002). A description of the method is provided in the SWGMA Glass Density Determinations guideline (2005b):

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.

. . . . 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. . . . 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.

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. (SWGMA 2005b)

Another relative method for measuring density is the sink-float comparator method. In this method, glass fragments are placed in a heavy liquid mixture in a test tube. The tube is placed in a water bath that is heated automatically at a uniform rate. As the particles settle and become suspended, the temperature is noted. The precision of this method is 0.0001 gm/cm³ (ASTM C729-05; Knight 1945), which is better than the measurable variation of a glass object.

Elemental Analysis

Manufacturers control the concentrations of many chemical elements to impart specific properties to their glass product. The concentrations of trace elements, however, are generally uncontrolled unless they alter the glass in some undesirable way (Koons 2002; SWGMA 2005a). Trace elements can be introduced to glass as impurities in the raw materials (Varshneya 1994) and/or from deterioration of the glass furnace (Doyle 1994). Glass composition analysis, therefore, can be used to differentiate between glasses made by different manufacturers, glasses from different production lines of the same manufacturer, and glasses made over a period of time in a single production line (Koons 2002).

Glass composition analysis is performed infrequently in forensic laboratories for several reasons:

- Most methods of glass composition analysis are destructive.
- Most methods require glass samples larger than those routinely encountered in forensic casework.
- Most of the instrumentation used to measure glass composition is expensive to purchase and maintain, and much of the instrumentation has few other applications.

- Because of the complexity of the calculations, Bayesian statistical analysis including compositional data is extremely difficult to apply.

Despite these drawbacks, chemical analysis remains the best means for differentiating glass specimens (Koons and Buscaglia 1999; Koons and Buscaglia 2002). Koons and Buscaglia (1999) succinctly stated the case for the use of chemical analysis despite its drawbacks:

The forensic scientist should use the most discriminating technique available in the examination of glass or other form of trace evidence because it is the most effective means of both avoiding false associations and excluding two similar, but separate, sources. It is in the best interest of the court for the scientist to use the most discriminating analytical technique even if this means that exact probability figures for a conclusion cannot be calculated. (Koons and Buscaglia 1999)

Many methods have been used for compositional analysis of glass. These methods include semiquantitative techniques such as scanning electron microscopy-energy dispersive spectrometry (Ryland 1986; Terry et al. 1982) and X-ray fluorescence (Andrasko and Maehly 1978; Reeve et al. 1976) and quantitative techniques such as neutron activation analysis (Coleman and Goode 1973), flameless atomic absorption spectrometry (Hughes et al. 1976), spark-source mass spectrometry (Dabbs et al. 1973), inductively coupled plasma-optical emission spectrometry (Hickman 1987; Koons et al. 1988), inductively coupled plasma-mass spectrometry (Zurharr and Mullings 1990; Parouchais et al. 1996), and laser ablation-inductively coupled plasma-mass spectrometry (Moenke-Blankenburg et al. 1992).

Scanning Electron Microscopy and X-ray Fluorescence

Both of these semiquantitative methods, scanning electron microscopy (SEM) and X-ray fluorescence (XRF), are rapid and essentially nondestructive and use XRF to determine chemical composition. Although it is possible to analyze small, irregularly shaped particles, flat surfaces provide more precise and accurate quantitation. To achieve a flat surface, an irregularly shaped particle is embedded in resin and polished. This preparation technique is destructive (SWGMA 2005a).

Most forensic laboratories have access to scanning electron microscopy-energy dispersive spectrometry (SEM-EDS). In SEM-EDS, a focused beam of electrons systematically scans across a specimen and produces many signals, including X-rays with energies characteristic of specific elements (Postek and Howard 1980). Ratios of the intensities of some of the major and minor elements in glass can be used to discriminate between sources of glass, with 38 of 40 specimens being distinguishable in one study (Andrasko and Maehly 1978). This method also has been used successfully to classify glass fragments into sheet or container categories (Ryland 1986; Terry et al. 1982).

Wavelength dispersive detectors also are available for scanning electron microscopy-wavelength dispersive spectrometry (SEM-WDS) and can be used to produce quantitative data. Most forensic laboratories do not use SEM-WDS because of its higher cost and greater complexity (SWGMA 2005a). Although SEM-WDS is available in the FBI Laboratory, it is not used for glass analysis because of the availability of more discriminating chemical analysis techniques. Therefore, it will not be discussed in this paper.

X-ray fluorescence uses a primary beam of X-rays from a source, either an X-ray tube or a radioactive source, to excite secondary X-rays from the specimen. As with SEM-EDS and SEM-WDS, the X-rays emitted by the glass specimen have characteristic energies and wavelengths, which can be used to identify the elements present, and the intensities of the peaks can be used to quantify the results (Skoog and West 1980), or ratios of the intensities can be used for semiquantitative results. Semiquantitative XRF has been used successfully to distinguish 79 of 80 glass sources (Reeve et al. 1976). X-ray fluorescence requires the use of specimens that are larger than specimens analyzed using SEM-EDS (SWGMA 2005a). Quantitative results are difficult to achieve because they require the use of matrix-matched standards for comparison (Skoog and West 1980).

Neutron Activation Analysis

The basis for neutron activation analysis (NAA) is the measurement of the radioactivity induced as a result of irradiation by nuclear particles. A gamma-ray spectrometer is used to measure radiation of different energies. By comparing these energies with those of a standard, the type and quantity of atoms can be determined. Neutron activation analysis has the advantage of being a nondestructive and extremely sensitive form of chemical analysis. Unfortunately, the specimens are rendered radioactive (Skoog and West 1980). Neutron activation analysis also requires the use of a nuclear reactor, which is not readily available to most forensic laboratories. Nevertheless, NAA has been used successfully in the analysis of glass fragments (Coleman and Goode 1973; Schmitt and Smith 1970).

Spark-Source Mass Spectrometry

In spark-source mass spectrometry, the specimen is vaporized into a gaseous ionic plasma by a radio-frequency spark source, and the resultant ions are swept into a mass spectrometer. Spark-source mass spectrometry has been used successfully to discriminate specimens that were indistinguishable by refractive index and density determination (Dabbs et al. 1973). This technique is not readily available to forensic science laboratories, and applications to glass analysis have not been widely studied.

Flameless Atomic Absorption Spectrometry

In flameless atomic absorption spectrometry (FAAS), the specimen is first dissolved, then introduced to a graphite furnace and vaporized. A beam of monochromatic light is directed through the vapor, and the absorption is measured. The wavelength of the light is matched to the characteristic absorption of the element of interest. Absorption is proportional to the number of atoms in the light path (Skoog and West

1980). Flameless atomic absorption spectrometry provides accurate, precise measurements and is a well-developed and widely accepted technique (Koons 2002). It has been used successfully for discriminating and classifying sub-milligram-sized glass fragments (Howden et al. 1977; Hughes et al. 1976). However, FAAS is a relatively slow analysis because each element is analyzed sequentially. Consequently, use of FAAS has been supplanted largely by various inductively coupled plasma (ICP) methods because ICP can analyze multiple elements simultaneously.

Inductively Coupled Plasma Methods

All of the ICP methods rely on the use of a plasma torch to produce extensive atomization, ionization, and excitation of the atoms of the specimen. In inductively coupled plasma-optical emission spectrometry (ICP-OES), the detector is a spectrometer that detects the characteristic wavelengths of light emitted by the excited atoms. The intensity of the light is proportional to the concentration of the atoms (Skoog and West 1980). In inductively coupled plasma-mass spectrometry (ICP-MS), the excited ions are swept into a mass spectrometer, similar to spark- source mass spectrometry, but using the plasma to ionize the specimen (Skoog and West 1980). In ICP-OES and ICP-MS, a glass specimen is first digested in hydrofluoric acid, brought to dryness, and then placed into solution (SWGMA 2005a). The dissolved glass is introduced into the plasma as a solution by a nebulizer (Skoog and West 1980). In laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS), instead of digesting the glass in hydrofluoric acid, a laser is used to ionize glass (Latkoczy et al. 2005), which is then swept into the ICP torch and analyzed as any other ICP-MS specimen (SWGMA 2005a).

Detection limits for most elements by ICP-OES are on the order of 0.01 µg/g; and for ICP-MS, 0.001 µg/g. The better detection limit for ICP-MS is acquired at a slight loss in precision and accuracy (SWGMA 2005a). Inductively coupled plasma techniques are in routine use in laboratories with the resources to buy the instrument. Numerous studies have been published on this subject. A summary of some of the literature is found in the SWGMA Elemental Analysis of Glass guideline (2005a):

... An inductively coupled plasma-optical emission spectrophotometry analytical method was developed to determine the concentrations of Mn, Fe, Mg, Al, and Ba in glass fragments (Catterick and Hickman 1981). Over the next several years, the concentrations of additional elements in glass by inductively coupled plasma-optical emission spectrophotometry were determined, and 6 to 10 element classification schemes based on comparison with a glass database divided into nine product categories were developed (Hickman 1981; Hickman et al. 1983). Currently, the protocol most widely used for casework was developed for determining the concentrations of 10 elements (Al, Ba, Ca, Fe, Mg, Mn, Na, Ti, Sr, and Zr) with excellent analytical precision in milligram-sized glass fragments (Koons et al. 1988). A combination of five of these elements was shown to provide good classification into the two categories of sheet and container glass. Inductively coupled plasma-optical emission spectrophotometry has also been used to associate food containers to the manufacturing plants in which they were made and to identify sources of contaminant glass in cases involving product tampering (Wolnik et al. 1989).

In further studies, the distributions of up to 22 elements, most measured by inductively coupled plasma-optical emission spectrophotometry, in various glasses were shown to provide excellent discrimination capability among sources within a product class (Hickman 1983; Hickman et al. 1983). In a study measuring the concentrations of 10 elements in automobile side-window glasses, the probability that two glasses from different vehicles would be indistinguishable was reported to be one in 1,080, compared with one in five for refractive index alone and one in ten for energy dispersive X-ray fluorescence spectrometry analysis alone (Koons et al. 1991). Studies have shown that using inductively coupled plasma-optical emission spectrophotometry, sheets of glass produced within minutes of each other in a single float-glass production line can be differentiated. In a recent study using statistical analysis of samples collected in casework, it was reported that inductively coupled plasma-optical emission spectrophotometry measurements provide very high discrimination capability. The probability that two glass fragments from different sources will have indistinguishable concentrations of ten elements is extremely small (Koons and Buscaglia 1999). (SWGMA 2005a)

Interpretations/Conclusions

In order to differentiate between two or more glass sources, the observed and measured properties between sources must be different. The variations of the properties of glass within a single source are usually small (Bottrell et al. 2007), typically below the resolving power of the techniques used to measure them. The variation among glass objects from different sources can be observed and measured (Hickman 1983; Koons et al. 1988; Koons and Buscaglia 1999; Koons and Buscaglia 2002) and are usually much greater than the variation within a single object. The variation in glass properties between types of glasses is pronounced (Boyd et al. 1994; Corning, Incorporated 1998; Doyle 1994; Varshneya 1994). The most important of these considerations in interpreting the results of glass analyses is that the variations within an object are typically smaller than the variations among objects.

Databases of refractive indices and/or chemical compositions of glass received in casework have been established by a number of crime laboratories (Koons et al. 1991). Although these glass databases are undeniably valuable, it should be noted that they may not be representative of the actual population of glass, and the distribution of glass properties may not be normal. Although these are not direct indicators of the rarity in any specific case, they can be used to show that the probability of a coincidental match is rare.

Koons and Buscaglia (1999) used the data from a chemical composition database and refractive index database to calculate the probability of a coincidental match. They estimated that the probability of a random occurrence of glass_D with indistinguishable chemical composition and n_D to be between 10⁻⁵ and 10⁻¹³. In other words, the chance of finding a coincidental match in forensic glass casework using refractive index and chemical composition alone is 1 in 100,000 to 1 in 10 trillion, which strongly supports the supposition that glass fragments recovered from an item of evidence and a broken object with indistinguishable n_D and chemical composition are unlikely to be from another source and can be used reliably to assist in reconstructing the events of a crime.

When no differences within the limits of the analytical techniques of the assessed properties can be identified, the possibility that the glass fragments originated from the same source cannot be eliminated. Currently, the FBI Laboratory uses range overlap as the comparison criteria. In range overlap, multiple measurements are made on each specimen, and the range is defined by the lowest value of the control specimen measured minus the measurement uncertainty to the highest value of the control specimen measured plus the measurement uncertainty. The number of fragments selected for analysis is based on the statistical sampling scheme presented in the paper by Sandercock (2000), “Sample Size Considerations for Control Glass in Casework.” The measured values of each recovered fragment are compared with the range for the control. If the measured values of the recovered sample overlap the control range, the specimens are considered indistinguishable.

The “Trace Evidence Unit: Casework Assignment and Review Procedures” section of the *Trace Evidence Unit Quality Assurance Manual* (2006) states:

A glass association is defined as two or more glass samples that can be fracture fitted together, or that exhibit indistinguishable observable properties and/or range overlap in all measured properties. Observed properties can include but are not limited to: glass type, color, and manufacturing methods. Measured properties can include but are not limited to: thickness, refractive index, dispersion and chemical composition. Glass associations are confirmed by a second qualified examiner. (Federal Bureau of Investigation [FBI] 2006)

Other methods for evaluating glass analytical data have been suggested, such as Bayesian statistical analysis (Curran et al. 2000) or various t-tests (Curran et al. 1997). Each technique has its advantages and disadvantages. One of the important considerations in choosing an evaluation technique is where to set the threshold for differentiation. A conservative threshold will differentiate all samples from different sources but may also indicate that a difference exists in specimens that are actually from the same source. A high threshold for differentiation may not be able to differentiate all specimens from sources that are genuinely different but will not differentiate specimens that are actually from the same source.

Range overlap on glass analytical data that include chemical composition data is considered a conservative standard. In one study, on a data set consisting of three replicate measurements each for 209 specimens, the range-overlap test discriminated all specimens, and all other statistical analysis-based tests performed worse (Koons and Buscaglia 2002).

Range-overlap tests, however, may achieve their high discrimination by indicating that two specimens from the same source are differentiable. Another study showed that when using a range-overlap test, the number of specimens differentiated that were actually from the same source may have been as high as seven percent (Bottrell et al. 2007).

The range-overlap approach, however, seems prudent given that other tests with higher thresholds for differentiation, such as t-tests with Welch modification (Curran et al. 2000) or Bayesian analysis (Walsh 1996), lower the number of specimens differentiated that were actually from the same source by worsening the ability to differentiate specimens that are genuinely different, a result that is unacceptable.

Reporting Conclusions

In most cases, the conclusions reached by the forensic glass examiner are officially communicated to the submitting agency in writing after a review process. At the FBI Laboratory, it is a three-stage process in which the results of the examiner are:

- 1. Verified by a second qualified examiner.
- 2. Technically reviewed by a second qualified examiner.
- 3. Administratively reviewed.

The technical reviewer ensures that the proper examinations have been conducted, confirms that a documented verification has been performed, and ensures that the conclusions are supported by the examination documentation and are within the limitations of the discipline. The administrative reviewer ensures that the report is concise, accurate, and complete; that the report and examination conform to FBI Laboratory practices; and that the prior two stages of the review have been conducted (FBI 2007).

Accredited laboratories follow this same general process, although the details on how it is carried out may differ (International Organization for Standardization/International Electrotechnical Commission [ISO/IEC] 2005). Conclusions may also be presented in sworn statements, either through deposition or testimony.

Guidance on the content of reports is provided through various accrediting bodies (American Society for Crime Laboratory Directors/Laboratory Accreditation Board [ASCLD/LAB] 2006; International Laboratory Accreditation Cooperation [ILAC] 2002; ISO/IEC 2005). Additionally, SWGMAT’s Expert Reporting Guideline (SWGMAT 2009) offers additional advice on what should be included in a report. The guideline mainly addresses written reports.

In general, the SWGMAT guideline recommends that reports contain enough administrative detail for a

reader to determine the identities of all parties to the report and the nature of the request. The report also should contain the results and conclusions of the examiner. All supporting data must either be included in the report, included as an addendum, or maintained in the laboratory's files and provided on request from the client (SWGMAF 2009). Greater transparency in reporting can be achieved if these guidelines are followed.

Conclusions

Glass analysis is used by both the glass industry and the forensic community. Abundant data have been published in peer-reviewed journals in either industry or forensic literature that validate the analytical techniques used in glass analysis and the theory behind comparisons. Additional work in these areas is ongoing as advances are made and instrumentation improves. Glass analysis has been demonstrated to provide excellent discrimination potential, making it an outstanding tool for forensic analysis.

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