Forensic Glass Examinations

1 Scope

1.1 Introduction

This document provides guidance for selecting/organizing an analytical scheme for identifying and comparing glass by Geologist-Forensic Examiners within the Trace Evidence Unit (TEU). The size and condition of the samples, as well as the information requested by the contributor will influence the selected analytical scheme.

The forensic examination of suspected glass is undertaken for investigative and intelligence purposes to:

- determine if an unknown particle is glass;
- determine the compositional class and product type;
- determine if two or more fragments originated from different sources;
- determine the significance of finding two or more glass fragments indistinguishable; and
- determine the cause and nature of breakage.

A comparison involves the recognition and evaluation of class characteristics that associate materials to a group but never to a single source to the exclusion of all other broken glass sources. Only when two or more broken glass fragments physically fit together can it be said that they were once part of the same object. When a difference is found between compared items, the examination may be immediately discontinued, and it can be reported that the compared fragments did not originate from the same broken glass source. Refer to *Introduction to Forensic Glass Examination, Initial Examinations of Glass*, and *Collection, Handling and Identification of Glass* by the Scientific Working Group for Materials Analysis for further discussion on these subjects.

1.2 Specimens

The various examination techniques described in this guideline apply to most types of glass, including the following:

1) flat glass used for items such as windows, doors, display cases, and mirrors
2) container glass
3) tableware glass
4) optical glass
5) decorative glass
6) specialty glass used for headlamps, cookware, and others

2 Equipment/Materials/Regents

- Cameras
- Light box equipped with daylight equivalent source
- Micrometer capable of measuring in inches to the fourth decimal place
- Personal protective equipment as needed
- Stereobinocular microscope with minimum magnification of 4 diameters
- Ultraviolet (UV) light source capable of producing short wavelength (nominally 254 nm) and long wavelength (nominally 350 nm) UV light
- Polariscope
- Polarized light microscope (PLM)
- Additional materials may be used at the discretion of the Geologist/Forensic Examiner.

3 Standards and Controls

Refer to the *Refractive Index of Glass by GRIM*, *Elemental Analysis of Glass by Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES)*, and *Elemental Analysis of Glass by Laser Ablation Inductively Coupled Plasma – Mass Spectrometry (LA-ICP-MS)* procedures for standards and controls used for each analysis technique.

4 Sampling

4.1 Refer to the *Refractive Index of Glass by GRIM*, *Elemental Analysis of Glass by Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES)*, and *Elemental Analysis of Glass by Laser Ablation Inductively Coupled Plasma – Mass Spectrometry (LA-ICP-MS)* procedures for sampling guidance for each analysis technique.

4.2 In cases where large quantities of glass are received in a sample, a portion of the entire sample may be selected for physical examination assessments based on a visual inspection of the entire sample.

5 Procedures

5.1 Glass may be received as suspected broken glass fragments or may be contained in debris recovered from objects. Thoroughly examine debris for glass fragments visually, using a stereobinocular microscope or other magnifiers as needed.

5.1.1 All suspected glass must first be identified as glass. Glass fragments can be identified using the following properties: it will not deform under pressure, it breaks with conchoidal fracture, it has a vitreous luster, it is non-crystalline, and it is isotropic.

5.2 Recovered glass (questioned glass) will be evaluated to identify characteristics suitable for comparison prior to comparison to exemplar glass samples. Characteristics not present or expressed in the recovered glass need not be assessed in the exemplar glass. Similarly, characteristics not present or expressed in the exemplar glass need not be assessed in the recovered glass.

5.3 If enough glass is present and the examination is requested or useful, perform fracture analysis. Refer to the Fractography of Brittle Materials Procedure for a description of glass fracture analysis. No further testing may be necessary after a fractography examination.

5.4 Assess the physical properties of the glass, including characteristics such as color, type of glass, etc. if they are expressed in the glass and will address the contributor's request. At the discretion of the Geologist/Forensic Examiner, the glass may be photographed.

5.4.1 Observe the broken fragments for indications of tempering. Tempered glass typically breaks into cube-shaped fragments or "dice." The fracture surfaces may exhibit mist hackle with a symmetrical pattern of secondary Wallner lines on either side (i.e., "frost lines"). Tempered glass exhibits internal stresses that may be resemble birefringence when observed in cross-polarized light on a polariscope or PLM.

5.4.2 Using natural light or a light box equipped with a daylight equivalent source, observe the color of the glass fragment on edge. No color may be apparent in small fragments, or even in larger fragments of lightly colored glass. Slight differences in color can be observed by comparing like-sized fragments side-by-side. For full-thickness fragments, observe color from edge to edge as well.

5.4.2.1 Glass fragments are considered to be distinguishable if the observed hue or saturation of color of like-sized fragments is different, or if the hue or saturation of color is different in dissimilarly sized fragments.

5.4.3 If an original surface is present, assess the glass for fluorescence under short wave and long wave UV light in a darkened room. The tin side of glass manufactured by the float process will fluoresce under short wave UV light. Glass samples may also exhibit an overall fluorescence.

5.4.4 For comparisons of flat glass specimens, measure the thickness of the fragment with a micrometer if the original surfaces of both the known and questioned glass fragments are present.

5.4.4.1 When measuring thickness of known glass, measure all fragments with two original surfaces or a minimum of 10 fragments in cases where there are more than 10 fragments with two original surfaces. To adequately assess the variability of the object, measure the thickness across the entire object when possible/available. If the glass measured falls into two or more distinct thicknesses populations, it may indicate the presence of multiple sources of glass in a

single sample. Additional thickness measurements may be useful in differentiating these populations.

5.4.4.2 When measuring the thickness of questioned glass, measure ten fragments with original surfaces that have not been previously disassociated by other properties. If ten fragments with original surfaces are not present, measure all glass fragments with original surfaces. If all of these fragments are distinguishable from the known glass by thickness measurement, additional questioned glass fragments, if present and suitable, will be measured. The total number of glass fragments measured is at the discretion of the Geologist/Forensic Examiner.

5.4.4.3 The items measured are considered indistinguishable in thickness if the questioned item(s) falls within the range of the known glass item. Each questioned glass fragment is compared individually. In cases where the variability of the known glass cannot be adequately assessed, the average of the measured values ± 0.001" will be used as the range for the known glass.

5.5 Optical properties will be measured if assessing the optical properties will address the contributor's request or be useful. Glass that has been heated in a fire may be thermally altered, and therefore the optical properties would be changed. For such samples, laboratory annealing before measuring refractive indices can remove strain introduced as a result of the fire. Refer to the *Laboratory Annealing of Glass* procedure.

5.5.1 Measure the refractive index of the glass at one or more wavelengths. Refer to the *Refractive Index of Glass by GRIM* Procedure for a description of measurement of refractive index of glass.

5.6 When questioned glass is not distinguishable in refractive index from the known glass, both may be annealed to determine whether or not they have similar thermal histories. If the glass may have been thermally altered, annealing before measuring refractive indices will remove strain introduced so that a meaningful comparison may be performed. Refer to the *Laboratory Annealing of Glass* Procedure.

5.7 Measure the elemental composition of the glass if the physical properties assessed are the same and the refractive indices are indistinguishable based on the association criteria, if there is sufficient glass present, and if measuring the chemical composition of the glass will address the contributor's request. The elemental composition of 18 to 20 elements in the glass can be measured by LA-ICP-MS. Refer to the *Elemental Analysis of Glass by Laser Ablation Inductively Coupled Plasma – Mass Spectrometry (LA-ICP-MS)* procedure. The LA-ICP-MS procedure is semi-destructive and does not require the use of hazardous chemicals. The elemental composition of ten elements in the glass can also be measured by ICP-OES. When the LA-ICP-MS system is not in use, the ICP-OES instrument may be used at the discretion of the Geologist-Forensic Examiner. Refer to the *Elemental Analysis of Glass by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)* procedure. Other methods of elemental analysis including scanning electron microscopy-energy dispersive X-ray spectrometry (SEM-

EDS), or X-ray fluorescence spectrometry (XRF) may also be used at the discretion of the examiner.

5.8 Record all pertinent observations, measurements and instrument data in case notes.

5.9 Verification

Glass associations will be verified by a second qualified Geologist-Forensic Examiner. A glass association is defined as follows:

1) When two or more pieces of broken glass physically fit together, the fragments were once part of the same broken object.

or

2) If elemental composition data has been acquired: When the physical properties assessed are the same, the average of multiple refractive index measurements of the questioned items falls within the range of refractive index values of the items from known sources, and the averages of the elemental concentrations of the elements measured falls within the modified 4σ interval, the glasses are said to be indistinguishable (see *Refractive Index of Glass by GRIM*, *Elemental Analysis of Glass by Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES)*, and *Elemental Analysis of Glass by Laser Ablation Inductively Coupled Plasma – Mass Spectrometry (LA-ICP-MS)* procedures).

3) If elemental composition data has not been acquired: When the physical properties assessed are the same and the average of multiple refractive index measurements of the questioned items falls within the range of refractive index values of the items from known sources, the glasses are said to be indistinguishable (see *Refractive Index of Glass by GRIM* procedure).

All other glass examination results may be verified by a second qualified Geologist-Forensic Examiner.

Verifications will be recorded in Forensic Advantage (FA). Any disagreements between the primary examiner and verifying examiner will be addressed under the FBI Laboratory Operations Manual, Practices for Resolution of Scientific or Technical Disagreement.

6 Calculations

Refer to the *Refractive Index of Glass by GRIM*, *Elemental Analysis of Glass by Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES)*, *Elemental Analysis of Glass by Laser Ablation Inductively Coupled Plasma – Mass Spectrometry (LA-ICP-MS)*, and *Laboratory Annealing of Glass* procedures for calculation requirements for each analysis technique.

An average thickness is calculated by summing the thickness measurements for a single item and dividing by the total number of thickness measurements for that item.

7 Measurement Uncertainty

7.1 Refer to the *Refractive Index of Glass by GRIM* procedure for measurement uncertainty for that technique.

7.2 The measurement uncertainty of thickness is on the order of ± 0.0002”, depending on the micrometer used. Refer to instrument manuals for uncertainty for a particular micrometer. The variation in thickness of flat glass ordinarily does not exceed 0.1”. Thickness variation in curved glass is typically greater than 0.1”.

8 Limitations

8.1 Refer to the *Refractive Index of Glass by GRIM*, *Elemental Analysis of Glass by Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES)*, *Elemental Analysis of Glass by Laser Ablation Inductively Coupled Plasma – Mass Spectrometry (LA-ICP-MS)*, and *Fractography of Brittle Materials and Laboratory Annealing of Glass* procedures for the limitations for each analysis technique.

8.2 The amount of sample and sample adulteration can limit the examination and resulting conclusions.

9 Safety

9.1 Broken glass will be handled while wearing appropriate personal protective equipment.

9.2 UV light in the range of 254 nm is classified as UVC. Prolonged exposure to UVC light can cause burning of skin, cornea, and conjunctiva, and can also cause nuclear cataracts. Care must be taken to minimize exposure to UVC light. A laboratory coat and opaque gloves will be worn to protect the skin and use of the UVC light should be severely limited to reduce the potential for damage to the eyes.

9.3 Eye protection and gloves will be used when breaking glass.

10 References

- Koons, Robert D., JoAnn Buscaglia, Maureen C. Bottrell, and Elmer Miller, Forensic Glass Comparisons, Forensic Science Handbook. R. Saferstein (ed), 2002. Prentice-Hall, 2002.

- Glass Refractive Index Determination, Scientific Working Group for Materials Analysis, *Forensic Science Communications*, Volume 7, No 1, January, 2005.

- Introduction to Forensic Glass Examination, Scientific Working Group for Materials Analysis, *Forensic Science Communications*, Volume 7, No 1, January, 2005.

- Garvin, Elizabeth J., and Koons, Robert D, Evaluation of Match Criteria Used for the Comparison of Refractive Index of Glass Fragments, *Journal of Forensic Sciences,* Volume 56, No 2, March 2011.

- Latkoczy, Christopher et al., Development and Evaluation of a Standard Method for the Quantitative Determination of Elements in Float Glass Samples by LA-ICP-MS, *Journal of Forensic Sciences*, 50(6): 1327-1341, 2005.

- Trejos, Tatiana, et al., Forensic Analysis of Glass by µ-XRF, SN-ICP-MS, LA-ICP-MS and LA-ICP-OES: Evaluation of the Performance of Different Criteria for Comparing Elemental Composition, *Journal of Analytical Atomic Spectrometry*, 28; 1270-1282, 2013.

- Introduction to Forensic Glass Examination, Scientific Working Group for Materials Analysis, *Forensic Science Communications*, Vol 7, No 1, Jan 2005.

- Initial Examination of Glass, Scientific Working Group for Materials Analysis, *Forensic Science Communications*, Vol 7, No 1, Jan 2005.

- Collection, Handling and Identification of Glass, Scientific Working Group for Materials Analysis, *Forensic Science Communications*, Vol 7, No 1, Jan 2005.

- Refractive Index of Glass by GRIM, Trace Evidence Procedures Manual (current version).

- Elemental Analysis of Glass by Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES), Trace Evidence Procedures Manual (current version).

- Elemental Analysis of Glass by Laser Ablation - Inductively Coupled Plasma – Mass Spectrometry (LA-ICP-MS), Trace Evidence Procedures Manual (current version).

- Laboratory Annealing of Glass, Trace Evidence Procedures Manual (current version).

- Chemistry Unit, Metallurgy, Operation of the Thermo QUANT'X X-Ray Fluorescence Spectrometer (current version).

- Chemistry Unit, Metallurgy, Compositional Analysis by Energy Dispersive X-Ray Fluorescence Spectrometry (current version).

- Chemistry Unit, Metallurgy, Operation of the Bruker M4 Tornado X-Ray Fluorescence Spectrometer (current version).

- FBI Laboratory Safety Manual (current version).

Rev. #	Issue Date	History
6	02/10/2020	Added section 5.2 and renumbered subsequent sections as necessary. Removed 'or Sample Selection' from Section 4 title.
7	09/01/2021	Reformatted Section 1.2 to bulleted list of examination techniques; removed automotive glass. Changed "Petrographic Microscope" to "Polarized Light Microscope" in Section 2. Added *Elemental Analysis of Glass by Laser Ablation Inductively Coupled Plasma – Mass Spectrometry (LA-ICP-MS)* procedure to Sections 3, 4.1, 5.7, 5.9, and 6. Updated Section 4.2 to a more general "physical assessments." Updated Sections 5.1, 5.4.1, 5.4.2 for clarity. Added reference to *Laboratory Annealing of Glass* procedure to Section 5.5. Changed Section 5.7 for instructions on the use of LA-ICP-MS versus ICP-OES. Add *Elemental Analysis of Glass by Laser Ablation Inductively Coupled Plasma – Mass Spectrometry (LA-ICP-MS)* procedure to References. Changed "Mineralogy" to "Geology" in Approval Section. Updated TL in Approval Section.

Redact - Signatures on File

Approval

Trace Evidence Unit Chief Date: 08/31/2021

Geology Technical Leader Date: 08/31/2021