Title: Examination of Primer Gunshot Residue (pGSR) Evidence

1. Introduction

The following procedure is used to analyze samples for the possible presence of primer gunshot residue (GSR or pGSR). Propellant within ammunition is ignited by chemicals after the primer is activated from a physical strike within a firearm. Propellant within firearm cartridges burn and generate gases that force projectiles (i.e., bullets) out of the firearm. Residue of the chemicals within the primer are potentially deposited on the hands of shooters and anything else in within range of the firearm's discharge. The detection of lead-bearing residue from primer discharge is based on the "Sinoxid" primer formulation which contains lead styphnate (possibly with other lead compounds), antimony sulfide, and barium nitrate. The discharge of firearms produces a range of chemicals, including residue from explosive material, oxidizers, reducing agents, sensitizers, fuels, binders, and primer (Romolo & Margot, 2001). Collectively, the chemicals being produced can be referred to as gunshot residue (GSR). In current forensic practices, however, the term GSR refers specifically to inorganic particulate residue that is formed after a firearm discharge and is related to the primer chemicals, including metal oxides. The term "primer gunshot residue" (pGSR) is sometimes used to make this distinction clearer. The chemicals from GSR can also be broken down into inorganic and organic GSR. The particles of interest within this procedure will only be inorganic, particularly those containing Pb, Sb, and/or Ba.

Gunshot primer particles can be transferred to clothing or other nearby individuals. Residue can also be transferred secondarily from one individual or surface to another through contact, a process typically referred to as secondary transfer. Because of the possibility of secondary transfer and environmental factors, it can be challenging to interpret analytical results, be they positive or negative. The detection of GSR particles occurs in two stages within this procedure. The first stage is an automated screening portion wherein analytical software, based on certain parameters, determines possible GSR particles on a GSR stub's surface, and summarizes particle information and location. The second stage involves manual confirmation. The number of possible GSR particles reported by the software from the screening portion of the examination may be greater than the number of particles actually confirmed to have elemental compositions related to GSR.

2. Scope

The following procedure is limited to the qualitative analysis of specimens. Only inorganic particles will be analyzed. Comparison of inorganic particles to specific types of primer chemical compositions is possible, but prior approval is required. Decision criteria will be in the form recommended by the relevant forensic scientific community.

3. Principle

Inorganic residues captured on carbon adhesive stubs are analyzed for the presence of particles with select elemental compositions and acceptable morphology and size comparable to those found amongst discharged primer ammunition particles. Using a scanning electron microscope with associated detectors, minute particles associated with gunshot residue primers can be detected. A combination of morphology, size, and elemental information is used.

4. Specimens

The analysis conducted within this procedure involves the examination of GSR stub collection devices which have been used on the surfaces of evidence. Collection devices typically are stubs with cylindrical surfaces that contain carbon with adhesive. These stubs should be stored within plastic containers and are usually submitted as GSR kits. While 4-stub GSR kits from hand samplings have historically been accepted for analysis, 2-stub GSR kits are preferred. Additionally, cotton-tipped applicator swabs have been used for atomic absorption (AA) related GSR analyses in the past, but these are not typically accepted and the analysis of any GSR stubs also submitted will take precedence over sampling from AA swabs (consult with the FSE 2 or higher prior to any AA swab sampling). Instrumentation is used to determine if particles on GSR stubs contain certain characteristics related to primer gunshot reside.

5. Responsibility

Analysts authorized to conduct examinations within the GSR category of testing.

6. Equipment/Materials/Reagents

a. Aluminum stubs (SEM/GSR stubs) (Electron Microscopy Sciences or equivalent)

b. Carbon adhesive tabs (double-sided, suitable for GSR collection) (Electron Microscopy Sciences or equivalent)

c. General laboratory equipment/materials (e.g., tweezers, Kimwipes, kraft paper, gloves, etc.)

d. Scanning electron microscope (SEM) system with backscattered electron (BSE) detector (Hitachi or equivalent)

e. Energy dispersive X-ray spectroscopy (EDS) system with detector and analyzer (Oxford or equivalent)

f. Copper (Cu) reference standard (99.9% purity or higher) (Sigma or equivalent)

g. Imaging standard (C, Co, Au, and Rh) (MAC or equivalent)

h. Gunshot residue (GSR) reference standard (PLANO standard) (Planotec GSR kit or equivalent)

i. Isopropanol (reagent grade or higher) (or equivalent)

j. Deionized water (DI) water (Millipore or equivalent)

7. Standards and Controls

Positive and negative controls may be purchased or generated in-house (e.g., a GSR stub collected after the discharge of a firearm as a positive control). Store controls either within the SEM chamber or within individual stub containers. If generated in-house, the controls must be verified and the documentation will be retained (physically and/or electronically). Controls may be sent to an outside laboratory for further confirmation. Any documentation must be retained for QA/QC purposes. All controls will have unique identifiers (i.e., lot numbers) which can be incorporated into case files.

a. Positive control in-house preparation (example):

  i. Immediately following the discharge of a firearm within the laboratory's firing range, GSR stubs are used to collect particles of interest from the shooter's gloved hands.

  ii. These stubs are then analyzed for GSR-related particles via SEM/EDS instrumentation.

  iii. Once particles characteristic of and consistent with GSR are confirmed, these stubs will be verified and acceptable for use as positive controls.

8. Procedure

a. Preparation:

  i. Upon starting the analysis, transfer and take custody of any items to be examined and record sample information in the laboratory information management system (LIMS) or on worksheets.

  ii. Analysts will wear lab coats and gloves during sample handling and will change gloves between items. Tools (e.g., tweezers) will be cleaned with DI water and isopropanol and wiped dry before handling evidence. GSR stubs will only be handled by their sides or stems to avoid contamination. Aside from intentional contact during sample collection from items, any other contact with the carbon adhesive surface of a GSR stub will be avoided.

  iii. All evidence stubs will be itemized within LIMS. Stubs generated in-house during evidence sampling will be itemized the day of sampling.

  iv. Negative and positive controls will be analyzed. A negative control GSR stub is used to monitor ambient conditions within and around the instrument. Both positive and negative control stubs will be analyzed with each batch of samples.

  v. Individually place stubs in the SEM stage sample holder and transfer custody to the appropriate instrument within LIMS.

  vi. If clothing or other items of evidence (e.g., AA swabs) have been submitted, each item (or group of items if packaged together) will be sampled using GSR stubs, as best as possible. The goal is to transfer any potential GSR particles from the items to the GSR collection stubs.

    1. Examination areas will be cleaned with DI water and isopropanol and then lined with a barrier layer of kraft paper between sampling different items of evidence. A negative control GSR stub

will be left open and exposed for the entire duration of sampling, within the same area and environment that the evidence sampling occurs.

2. Transfer of fibers from swab tips or cloth-like items should be minimized.

b. Scanning electron microscopy analysis:

An SEM with BSE and EDS detectors will be used for this analysis. A combination of automated and manual examination is used to render conclusions.

i. Quality assurance/quality control (QA/QC):

1. Necessary quality control standards will be analyzed according to the QA/QC procedure for that instrument and criteria acceptable prior to instrument examination of evidence.

ii. Automated analysis with GSR software using BSE and EDS detectors:

1. Load stubs for analysis.
2. Select the BSE mode (BSE-comp) in the SEM software and adjust instrument settings as needed.
3. Load an analysis template/profile and add the locations of the sample stubs.
4. Save the project, listing the case number, the date, and the analyst's initials in the project name.
5. All stubs will be scanned and analyzed automatically by the software based on particle size, intensity measurements, and preliminary elemental determination. Particles will be initially classified by the software (e.g., characteristic, consistent, commonly associated, or no classification).
6. The maximum number of automatically classified characteristic (Pb bearing) particles per stub will be set to 30 within the collection parameters.
7. A minimum of 250 fields at 200x magnification will be mapped for analysis on a standard circular GSR stub. If 250 fields cannot be mapped on a submitted stub, an FSE 2 or higher will be consulted.
8. Adjust the brightness and contrast thresholds as needed using the imaging standard and save the brightness and contrast calibration to the project.
9. Start the automated GSR analysis.
10. Collection information (e.g., individual stub reports, particle lists, etc.) will be generated and saved within the case file. List the information and parameters used during automated analysis (i.e., instrument, accelerating voltage, magnification, working distance, spot size, resolution, and process time) within the case file.

iii. Manual confirmation using EDS and BSE:

1. When the automated GSR analysis is complete, review the particle data for each stub (i.e., classifications, spectra, particle images, etc.).

2. Collect a BSE image of any particle used to render a conclusion within the report. Confirmed particle reports will list the parameters used during image capture (e.g., resolution, magnification, etc.)
3. Collect an EDS spectrum of said particles using a 30 second live time acquisition.
4. Save and include any confirmed particle images and spectra within the case file. These confirmed particle reports will list the information and parameters used during image and spectral capturing (i.e., instrument, accelerating voltage, magnification, working distance, spot size, resolution, live time, and process time).
5. Once 3 characteristic (Pb bearing) particles are manually confirmed, each with a Pb/Sb/Ba composition and acceptable morphology, then no further particle confirmations from the same stub(s), kit of stubs, or stubs from the same source, are required.
6. There must be a minimum of 3 confirmed particles of the same class in order to stop confirming additional particles of that class. For example, if only 2 characteristic (Pb bearing) particles are confirmed on a single stub, then the analyst would only stop confirming additional particles once 3 such particles are confirmed across additional stubs from the same kit or source. If less than 3 characteristic (Pb bearing) particles are confirmed, then the above steps will be followed for consistent (Pb bearing) particles. Only consistent (Pb bearing) particles with Pb/Sb, Pb/Ba, or Sb/Ba compositions will be confirmed. Commonly associated particles will not be routinely confirmed.
7. Record results and appropriate case and sample information in LIMS or on worksheets.
8. All data from particles used to render conclusions within final laboratory reports will be recorded in LIMS or on worksheets.

c. Remove GSR stubs from the SEM chamber. Label the underside of stubs with the case number, item number, and the analyst's initials, if possible. Return stubs to their respective containers and label those containers with initials and items numbers.

d. Properly seal and store all stubs in an approved and secure location.

9. Decision Criteria

The following criteria is used as a guideline in determining the acceptability of the data produced in this procedure. Results from the automated GSR software analysis provides a list of possible GSR particles. Manual acquisition of EDS spectra, along with particle imaging, allows for particle confirmation.

a. Software results:

i. The GSR software will produce a list of possible GSR particles based on size, image intensity measurements, and elemental composition.

ii. The software results will be used to select particles for confirmation purposes. Data from the software will not solely be used to render a conclusion within laboratory reports.

iii. If an evidentiary stub is negative for particles with elemental compositions related to GSR, then the associated positive control stub must be acceptable (at least 3 characteristic (Pb bearing) particles confirmed). If the positive control stub was not acceptable, then an FSE 2 or higher will be consulted, the instrument re-optimized, and all stubs within the affected batch re-analyzed.

iv. Unless noted otherwise, rejected particles are those which, upon manual particle review, do not match the originally screened particle classification (e.g., a particle screened as a characteristic (Pb bearing) particle (Ba/Sb/Pb) found to only have two, one, or none of those elements) and would not be selected for manual particle confirmation and/or would not be reported.

b. Morphology and Size:

i. Particles identified as characteristic (Pb bearing) or consistent (Pb bearing) using this method are often spheroid particles, typically between 0.5 µm and 5.0 µm in diameter. The remainder are irregular in shape or vary from 0.5 µm to 100 µm (or greater) in size, or both.

ii. In general, particles displaying crystalline morphology via SEM are not consistent with GSR formation. However, such particles have occasionally been observed in known GSR samples. Since morphology can vary greatly, it will never be considered as the only criterion for identification of GSR.

c. Elemental Characteristics:

i. The elemental composition is the most diagnostic property in determining if a particle could be GSR. Within this procedure the elements Pb, Sb, and Ba will be used to determine the possibility of a GSR particle. However, it is understood that some primer components may not have compounds that contain such elements.

ii. Particles will be classified as characteristic (Pb bearing) (i.e., characteristic of GSR) when they contain the following elemental composition:

1. Pb, Sb, and Ba

These particles have compositions which are infrequently found in particles from sources other than GSR.

iii. Particles will be classified as consistent (Pb bearing) (i.e., consistent with GSR) when they contain any one of the following elemental compositions:

1. Pb and Sb
2. Pb and Ba
3. Sb and Ba

These particles have compositions which are also found in particles from non-firearm sources.

iv. Particles will be classified as commonly associated with GSR when they contain any one of the following elemental compositions:

1. Pb
2. Sb
3. Ba

These particles have compositions which are also found in particles from environmental sources.

d. Positive/Negative Control:

   i. Positive controls will have at least 3 GSR particles confirmed. Negative controls will have no particles with elemental compositions also confirmed on particles from associated evidentiary stubs. If the negative control stub has one or more particles related to GSR confirmed, then the FSE 2 or higher will be consulted to determine how to proceed with analysis and reporting and that negative control stub will be discarded prior to the next analysis on that instrument.

e. Reporting:

   i. The particle classifications "characteristic of" and "consistent with" will be routinely reported. The particle classification "commonly associated with" will not be routinely reported. Consultation with the FSE 2 or higher will occur prior to the reporting of any "commonly associated with" particles.

   ii. Stubs without any confirmed characteristic (Pb bearing) and/or consistent (Pb bearing) particles will not routinely have any additional particle confirmations made. Said stubs will have reported results reflecting the lack of confirmed characteristic (Pb bearing) and consistent (Pb bearing) particles (e.g., "No particles characteristic of or consistent with pGSR were confirmed").

   iii. The number of confirmed particles associated with each classification will be reported, as will the elemental composition(s).

   iv. If the particle confirmations for a stub(s) (from the same kit or source) meets the minimum requirements set forth by this procedure, then additional manual particle confirmation on any remaining associated stubs is not required (even if candidate particles were detected and classified during the automated GSR analysis on said stubs). Said stubs will routinely be reported as not having been confirmed (e.g., "Particles were screened, but not confirmed").

10. Limitations

Ammunition containing lead-free primer compositions may only generate particles classified as "consistent with" or "commonly associated with" GSR during the discharge of a firearm. Ammunition primer compositions may also produce inorganic particles not confirmed or reported by this laboratory.

11. References

Hitachi SU3500/SU3800 SEM & software user manuals

Oxford Instruments Ultim Max 65 EDS & AZtec software user manuals

ASTM Standards E1588 and E3309