Title: Analysis of primer-Gunshot Residue (pGSR) evidence

1. Introduction:

This 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 a firearm cartridge burns and generates gases that force projectiles (i.e., bullets) out of a firearm. Expended residues from the chemicals within the primer are potentially deposited on the hands of shooters and anything else in close proximity of the firearm's discharge. The detection of residues from primer discharge are based on the "Sinoxid" primer formulation which contains lead styphnate (possibly with other lead compounds), antimony sulfide, and barium nitrate. The discharge of firearms produce a range of chemicals, including residues 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 firearm discharge related to primer chemicals, including metal oxides. The term "primer discharge residue" (or pGSR) is sometimes used to make this distinction more clear. The chemicals from gunshot residue can also be broken down into inorganic and organic GSR. The particles of interest within this procedure will only be inorganic, particularly those containing Ba, Sb, and/or Pb.

Gunshot primer particles can be transferred to clothing or other nearby individuals. Residues can also be transferred indirectly based on touch-to-touch interactions, a process commonly referred to as secondary transfer. Because of the possibility of secondary transfer and environmental interferences, it can be challenging to interpret analytical results, be they positive or negative. The detection of pGSR particles occurs in two stages within this procedure. The first stage is a screening portion wherein analytical software, based on certain parameters, determine possible pGSR particle(s) on a GSR stub's surface, and summarize particle information with location. The second stage involves examiner confirmation. The number of pGSR particles reported by the software during the preliminary screening portion of the exam will be limited and may be greater than the number of particles actually confirmed to have characteristics indicative of pGSR.

2. Scope

This procedure is limited to the qualitative screening of specimens. Only inorganic particles will be analyzed. Comparison of inorganic particles to specific types of primer chemicals is possible, but prior approval is required. The number of characteristics (i.e., specific elements [barium (Ba), antimony (Sb), and lead (Pb)], shape, size) present within a sample will determine how strongly associated detected particles will be when compared to primer-gunshot residue particles. 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 analysis of AA swabs (consult with the lead Examiner or higher prior to any AA swab analysis) . The transfer of AA swabs (that have not been exposed to any acid/liquid solutions) to GSR stubs is possible, however it must be understood that not all particles may transfer from the swabs to the stub surfaces. 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. Stubs with carbon adhesive (GSR stubs)

b. Tweezers

c. Scanning electron microscope (SEM) with secondary electron imaging (SEI), backscatter electron (BSE) and energy-dispersive X-ray Spectroscopic (EDS) detectors

d. Copper reference standard (Sigma or equivalent)

e. PLANO reference standard (or equivalent)

f. Isopropanol (reagent grade or better) (or equivalent)

g. Deionized water (DI) (or equivalent)

7. Standards and Controls

Positive and Negative Controls: May be purchased (e.g., PLANO) or generated in-house (e.g., GSR stub from known firearm discharge). Store either within the SEM chamber or within individual plastic containers. If generated in-house the controls must be verified appropriately and the documentation will be retained. 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 notes/files.

8. Calibration

This procedure is qualitative only.

9. Sampling

Not applicable.

10. Procedure:

a. Preparation

i. Upon starting the analysis record appropriate sample information.

ii. Examiners will wear gloves during sample handling and will change gloves between new samples. Tools (e.g., tweezers) will be pre-cleaned with water and isopropanol (or equivalent solvent) when used to handle evidence. GSR stubs will only be handled by their stems to avoid contamination. The touching of GSR disc adhesive surfaces with other objects will be avoided.

iii. Item number information should be recorded on each GSR stub base, as well as on their respective containers, if possible.

iv. Appropriate negative and positive controls will be analyzed. A negative control GSR stub is used to monitor ambient conditions. Both a positive and negative control will be analyzed with each batch of samples.

v. Individually place stubs in proper locations using the appropriate SEM sample/stage holder.

vi. All stubs will be itemized (or sub-itemized) within the laboratory information management system (LIMS). If stubs are generated in-house (i.e. collection from clothing or other items) the sub-item creation will occur on the day the [GSR stub] sub-items are generated.

vii. If AA swabs or other items of evidence (e.g., clothes) 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 pGSR particles from the items to the GSR collection stubs.

1. Since this involves residue material, examination areas will be cleaned with DI water and isopropanol and then lined with examination paper in-between each item of evidence. Proper negative controls will be collected from the examination paper.

Note: If AA swabs never make contact with the examination paper, then a negative control for sampling will not be required.

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

b. Scanning Electron Microscopy [with multiple detectors] Analysis

A scanning electron microscope (SEM) with a backscatter electron (BSE) detector, secondary electron imaging (SEI) detector, and an energy dispersive X-ray spectroscopic (EDS) detector will be used for this analysis. A combination of software and manual examination is used to render conclusions.

i. Quality Assurance/Quality Control (QA/QC):

1. Appropriate quality control (QC) reference standards will be analyzed according to the QA/QC procedure for that instrument.
2. When the instrument is ready set operating voltage, working distance, magnification, and spot size on the SEM control screen to appropriate levels.
3. Focus the microscope and adjust brightness and contrast as needed.

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

1. Select BSE (backscatter electron) mode on the SEM control screen and adjust appropriate brightness/contrast level and other settings.
2. Set up the parameters within the GSR analysis program.
3. Move and locate the sample stubs.
4. Enter the sample labeling information (e.g., case #, item #).
5. Appropriate collection information (e.g., job summary, particle lists, particle images, etc.) will be printed and stored in the casefile.
   Any necessary information not readily printed will be accurately described in Examiner case notes (e.g., brightness and contrast settings).
   The information within the case file <u>must be</u> sufficient so that another authorized Examiner would be able to reproduce the findings within the report.
6. All stubs will be set to 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., 1/2/3-component or none).
7. The maximum number of automatically classified 3-component particles per stub can be set at a user-defined number (e.g., 30) within the collection parameters.
8. The entire surface of a stub does not have to be completely analyzed. However, at least 70% of a stub's surface area should be mapped with fields within the software. The process of how the software is set up to search each stub (i.e., the starting/ending field and analysis path) can be user-defined.

Note: If (3) 3-component particles cannot be manually confirmed (on a stub or within a group of stubs from the same source) when the maximum number of automatically classified 3-component particles was reached, then the lead examiner or higher will be consulted. In this scenario it is likely that the instrument will need to be readjusted and the batch of affected stubs reanalyzed completely.

9. Start the GSR analysis.
10. Particles will be re-examined manually after the automated analyses of the batch of stubs has been completed.

iii. Manual confirmation using EDS and BSE

1. When the analyses from the software are completed a result summary is created.
2. Review the particle data for each stub (i.e., classifications, spectra, particle images, etc.).
3. Collect the backscatter electron image (BSE) of any particle used to render a conclusion within the report. The BSE images will contain adequate magnification, resolution, and clarity to document pGSR particles. Such images will be saved electronically with filenames unique to the case.
4. Re-collect a new EDS spectrum using at least a 100 second live time acquisition (to reveal enough data to adequately determine if lead, antimony, and/or barium are present). Spectra used in rendering conclusions will be saved electronically with a filename unique to the case.
5. Print any confirmed particle images and spectra.
6. During the analysis of a stub(s), if (3) 3-component particles are manually confirmed, all with a Pb/Sb/Ba composition and acceptable morphology, then no further particle confirmation from the same stub(s), kit of stubs, or stubs from the same source, is required.
7. There must be a minimum of (3) confirmed particles of the same class (i.e. component number) in order to stop confirming additional particles. For example, if only (2) 3-component particles were confirmed on a single stub, then the Examiner would only stop confirming additional particles once (3) such particles were confirmed across multiple stubs from the same kit or source. If less than (3) 3-component particles are confirmed, then the above steps will be followed for 2-component particles. Correspondingly, if less than (3) 2-component particles are confirmed, then the above steps will be followed for 1-component particles.
8. Record results and appropriate case and sample information within case notes.
9. Instrumental parameters will be recorded (e.g., beam voltage, collection time, magnification, working distance).
10. All data (e.g., images and spectra) from particles used to render conclusions within final laboratory reports must be maintained within case files.

c. Remove GSR stubs from the SEM chamber and return to their original containers.

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

11. Decision Criteria:

The following criteria are used as guidelines in determining the acceptability of the data produced in this procedure. Results from the automated GSR software analysis of stubs provides a list of possibilities which have select characteristics of pGSR 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 pGSR particles based on size, image intensity measurements, and elemental composition (i.e., 1-component, 2-component, or 3-component).

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 any particles with elemental compositions related to pGSR, then the associated positive control stub must be acceptable (at least (3) 3-component particles confirmed). If not, then the lead examiner or higher will be consulted, the instrument re-optimized, and all stubs within the affected batch re-analyzed.

b. Morphology and Size

i. Particles identified as characteristic of or consistent with gunshot residue primer 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 pGSR formation. However, such particles have occasionally been observed in known primer-GSR residues. Since morphology can vary greatly, it will never be considered as the only criterion for identification of pGSR.

c. Elemental Characteristics

i. The elemental composition is the most diagnostic property to determine if a particle could be a pGSR particle. Within this procedure the three elements: barium (Ba), antimony (Sb), and lead (Pb) will be used to determine the possibility of a pGSR particle. However, it is understood that some primer components may not have compounds that contain such elements.

When appropriate, the elemental composition of the recovered residues can be compared with case-specific known source primer formulations.

ii. Particles will be classified as being <u>characteristic of</u> pGSR when they contain the following elemental composition:

1. Barium, Antimony, and Lead.

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

iii. Particles will be classified as being consistent with pGSR when they contain any one of the following elemental compositions:

1. Lead and Antimony
2. Lead and Barium
3. Antimony and Barium

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

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

1. Barium
2. Antimony
3. Lead

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

v. The following classifications will be used within laboratory reports to indicate whether a residue particle possibly resulted from gunshot primer (from highest to lowest): 'characteristic of' > 'consistent with' > 'commonly associated with'. Individual elements will not be listed within reports.

vi. If the particle confirmations for a group of stubs (from the same kit or source) meets the minimum requirements set forth by the "procedure" section of this document, then additional manual particle confirmation on any remaining stubs is not required (even if particles were detected and classified during the automated analysis on said stubs). These stubs would be reported as "unconfirmed."

d. Positive/Negative Control

i. Results from the data analysis of controls must correspond to the appropriate control. Positive controls will have at least three (3) pGSR particles confirmed (along with supporting data and BSE images).Negative controls will have no particles with elemental compositions also confirmed on associated evidentiary stubs. If the negative control stub has one or more particles related to pGSR that are confirmed, then the Lead Examiner (or higher) will be consulted to determine the source of particles and how to proceed with reporting.

12. Limitations

The analysis of evidence for the presence of GSR (pGSR) can provide information to an investigation. However, the presence or absence of GSR (pGSR) cannot absolutely determine whether a person discharged a firearm or was in the presence of a firearm being discharged.

While the classifications used to indicate whether a residue particle possibly resulted from gunshot primer are ranked (from highest to lowest): 'characteristic of' > 'consistent with' > 'commonly associated with,' the determination that particles found on a GSR stub are, in fact, definitively from a discharged firearm cannot be made using this procedure.

The absence of particles either 'characteristic of,' 'consistent with,' or 'commonly associated with' pGSR does not mean that the sampled evidence was not in the close proximity of a discharged firearm. Studies have shown that evidence sampled in close proximity to a discharged firearm may not contain pGSR particles.

Some ammunition contain lead-free primers – thus causing evidentiary stubs from such firearm discharge areas to only have the possible result, 'consistent with pGSR' or 'commonly associated with pGSR' to be in final laboratory reports. Other ammunition may have other limited elements within their primers, causing similar limitations to occur within final laboratory reports.

13. References

Hitachi S-3700N Scanning Electron Microscope Operators Manual on the help menu of the program.

EDAX Genesis GSR Analysis software user's manual and movie (Appendix B).

Law Enforcement Development Group of the Aerospace Corporation. "Final Report on Particle Analysis for Gunshot Residue Detection". Prepared for the National Institute of Law Enforcement and Criminal Justice Law Enforcement Assistance Administration, U.S. Department.

Meng, H.H., Caddy, B., "Gunshot Residue Analysis-A Review", Journal of Forensic Sciences, 1977; Vol.42, No.4, pp.553-570

Wolten, et.al, "Particle Analysis for the Detection of Gunshot Residue. I: Scanning Electron Microscopy/Energy Dispersive X-ray Characterization of Hand Deposits from Firing", JFS, Vol. 24, No. 2, April 1979, pp 409-422.

Wolten, et.al, "Particle Analysis for the Detection of Gunshot Residue. II: Occupational and Environmental Particles", JFS, August 1978.

GSR Summary, Dennis Ward, FBI Academy.

ASTM Designation E 1588-17, "Standard Practice for Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive X-Ray Spectroscopy.