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 adhesive carbon-coated stubs are analyzed for the presence of particles that contain general morphology and size, along with select elemental compositions, which can be found within 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 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 within cardboard boxes (GSR kits). While 4-stub GSR kits from hand samplings have historically been accepted for analysis, 2-GSR stub 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. Transfer of AA swabs 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.

While the GSR kit may have a submission number, each GSR stub will have its own item number and description.

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. Carbon coater

7. Standards and Controls

Positive and Negative Controls: Can be purchased (e.g., PLANO) or generated in-house (e.g., GSR stub from known firearm discharge). Store either in SEM chamber or within plastic container – can be carbon coated. If generated in-house, the controls must be verified appropriately and documentation retained. Controls may be sent to outside lab for further confirmation – any documentation must be retained for

QA/QC purposes. All controls will have unique identifiers 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 document information from the sample labels on appropriate worksheet(s).

ii. Examiners will wear gloves during sample handling. Pre-cleaned tools (e.g., tweezers) will be used when handling the stems of GSR stubs to avoid contamination. The touching of GSR disc adhesive surfaces with other objects will be avoided.

iii. Ensure item number information is recorded on each GSR stub base, as well as their respective containers. Label control stubs accordingly. Add lab number information to stubs, 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 and record the positions within case notes or worksheets.

vi. It is recommended to mark the side of the GSR stubs and align with the markings of the sample holder. This assists with reproducibility if GSR stubs have to be re-mounted and analyzed in the future.

vii. All stubs will be itemized (or sub-itemized) within the Laboratory Information Management System's (LIMS) JusticeTrax LIMS-Plus software.

viii. If AA swabs or other items of evidence (e.g., clothes) have been submitted, each item will be sampled using individual GSR stubs as best as possible 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 and lined with brown examination paper in-between each item of evidence and proper negative controls will be collected.

Note: If swabs never touch the brown paper/examination area surface, then negative controls for AA swabs will be handled differently (i.e., not be required) than negative controls for items actually resting on examination surfaces (e.g., clothing).

2. Transfer of actual fibers from swab tips or cloth-like items should be avoided.
3. GSR stubs from such samples may be carbon coated in order to reduce 'charging effects,' but this is not required.
4. If carbon coating is performed, such information and description of the carbon coating process (including instrument model information) will be recorded within the Examiner's case notes.
5. Sub-items will be created within JusticeTrax for GSR stubs used to sample other items. Appropriate sub-item numbers and lab numbers will be recorded on the newly created GSR stubs. This creation will occur on the day the sub-items are generated.

ix. Other samples (e.g., dirty stubs) may be carbon coated in order to reduce the amount of 'charging' interference when the GSR software evaluates samples. Carbon coater information and the process for coating will be recorded within Examiner case notes.

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 to obtain good image(s).

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 Genesis GSR analysis program.
3. Move and locate the sample stubs.
4. Enter the sample labeling information (e.g., case #, item #).
5. All appropriate collection information (e.g., software printouts, screen capture images, case notes) will be printed and stored in the casefile.
   Any information not readily printed will be accurately described in Examiner case notes (e.g.,

how brightness and contrast were adjusted and specifically set for a given analysis).
The information within the case file must be enough so that another authorized Examiner will 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 morphology, intensity measurements, and preliminary elemental determination.
   Initial data from particles will be classified by the software.
   The maximum number of automatically classified 3-component particles (i.e., 3-COMP) within a GSR kit for a single person (or a set item) can be set at a user-defined number (e.g., 30) within the collection parameters for each stub.
7. The entire surface of stubs do not have to be completely analyzed. However, at least 70% of stub areas should be mapped with fields in the software. The process of how the software is set up to search each stub (i.e., the start/end path) can be user-defined. For example, if thirty (30) 3-COMP particles are detected by the time only 10% of the stub has been analyzed and the analysis automatically stops, then that is acceptable if at least three (3) of the particles can be verified as containing GSR-related characteristics.

Note: If at least three (3) 3-component particles cannot be confirmed when using a limited-number of automatically classified 3-COMP particles, then more of the stub's surface will be analyzed.

8. Start the GSR analysis.
9. Select particles will be re-examined manually after preliminary analyses of all of the stubs by the software have been completed.

iii. Manual confirmation using EDS and SEI (GSR kits of the same individual, item, or area of interest)

1. When the analyses from the software are completed a result summary is created.
2. Click on a particle number to recall the stored spectrum and designated particle image.
3. If a preliminary EDS spectrum reveals the possible presence of lead, antimony, and/or barium, move the stage to the designated particle field position.
4. Collect the secondary electron image (SEI) of any particle used to render a conclusion within the report. The SEI images will contain adequate magnification, resolution, and clarity to document pGSR particles. Such images will be saved electronically with filenames unique to the case, printed, and printouts kept within the case file.
5. Re-collect a new EDS spectrum using a 100 second live time acquisition (thus revealing 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, printed, and the printouts kept within the case file.
6. Print out a result page with particle images and spectra.

7. During the analysis of a stub within a GSR kit, if three (3) pGSR-related particles (i.e., contain Ba, Sb, and Pb, and have shapes and sizes of pGSR particles) are found and confirmed, then no further confirmations of other particles within that stub will be performed.
8. If one stub within a GSR kit (2-stub or 4-stub kit) has at least three (3) pGSR-related particles (i.e., all contain Ba, Sb, and Pb, and all have shapes and sizes of pGSR particles) then confirmation of particles from other stubs within the same kit is not required.
9. If one stub has at least three (3) pGSR-related particles that all contain Ba, Sb, and Pb, and all have shapes and sizes of pGSR particles, then confirmation of more pGSR-related particles from other stubs within the same kit/sampling/grouping is not required.
10. The maximum number of particles [of the same component number] to be confirmed within a GSR kit (or within stubs of the same person/object) will be six (6).
    For example: If only one or two 3-COMP particles are identified, then no more than six (6) 2-COMP or 1-COMP particles are to be confirmed.

Note: There must be a minimum of three (3) such particles per stub in order to stop confirming additional particles. For example, if only two (2) 3-element pGSR particles were found on one stub, then the Examiner would only stop when three (3) such particles were identified on multiple stubs. No more than six (6) 3-element particles will be confirmed within a GSR kit (or similar items of evidence). If no '3-COMP' particles exist then the above steps will be followed for 2-COMP particles. Correspondingly, if no '2-COMP' particles exist then the above steps will be followed for '1-COMP' particles.

11. If submitting agencies specifically request that all stubs within a GSR kit (or within all submitted clothing) be fully analyzed, then all will be analyzed. This specific request must be documented in writing by the submitting agency and will be kept within LIMS-Plus.
12. Document results (e.g., using a worksheet) and record all appropriate case and sample information within case notes. All data printouts will contain the instrument name (i.e., SEM-01, SEM-02), date(s) of acquisition, and the Analyst(s) identifiers (i.e., initials or name) who acquired and is responsible for the data.
13. Instrumental parameters must be recorded within the examination documents (e.g., beam voltage, collection time, magnification, working distance). This should preferably be done electronically, but handwritten notes are acceptable.
14. 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 put each back into 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 allow Examiners to confirm pGSR particle qualities.

a. Software results

i. The Genesis GSR software will produce a list of particles which are possibilities for GSR particles based on size, image moiety, and elemental composition (i.e., 1-component, 2-components, or 3-components).

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 '3COMP' particles (i.e., presumptive positive '*characteristic* pGSR particle'), then the number of '3COMP' particles found from the software's analysis of the positive control stub (used for the batch analysis) must be at least 70% of the accepted number of '3COMP' particles from said positive control based on QA/QC acceptability set for that control. If not, then the instrument will be re-optimized and only the negative and non-3-component particle-evidentiary stubs will be re-analyzed.

b. Morphology and Size

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 + in size, or both. In general, it is not consistent with the mechanisms of pGSR formation to find particles by SEM displaying crystalline morphology. 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

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. Occasionally, pGSR particles with apparent unusual elemental compositions can be encountered and elemental compositions of evidence can be compared to case-specific sources, such as cartridges or ammunition/weapon test fire deposits, if specifically requested and approved by the Deputy Director.

i. Particles classified as being characteristic of pGSR must contain the following elemental composition within the particle's EDS spectra: Barium, Antimony, and Lead.

These particles have compositions which are infrequently found in particles from any other source other than pGSR-related particles.

ii. Particles will be classified as being consistent with pGSR when they contain one (1) of the following elemental compositions:

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

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

iii. Particles classified as commonly associated with pGSR must contain only one (1) of the following elements: Barium, Antimony, or Lead
These particles have compositions which are also found in particles from environmental sources. When such particles are found on stubs from a GSR kit or from clothing that contain 'characteristic' and/or 'consistent with' particles, then they have more significance towards pGSR-related particles. However, when no 'characteristic' and/or 'consistent with' particles are found with 'commonly associated' particles, then such particles have little significance to pGSR presence.

iv. 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.

d. Positive/Negative Control

Results from the data analysis of controls must correspond to the appropriate control.

i. Positive controls will have at least three (3) pGSR particles confirmed (along with supporting data and SEI images).

ii. Negative controls will have no particles that are related to pGSR which need to be confirmed.

1. If the negative control stub has one or more 3-component particles that are confirmed, then the Lead Examiner (or higher) will be notified. All stubs analyzed in the same batch will be reported as inconclusive with an explanation in the report.

2. If the negative control stub has one or more 1-component or 2-component particles that are confirmed, then the Lead Examiner (or higher) will be notified and evidentiary stubs in the same batch that have 1-COMP or 2-COMP data will be rejected and, unless at least three (3) 3-COMP particles have been confirmed, the analyses will be reported as inconclusive with an explanation in the report.

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.

Revision #	Revision History
2	Revised title. Updated Responsibility section. Updated procedure. Removed references to manuals, other SOPs, and to appendices. Added minimum number of confirmatory spectra to be analyzed and documented per sample. Added step to allow that not all stubs in a GSR kit needed to be fully analyzed if certain parameters were met. Added requirement to include instrumental parameters within case file. Added positive and negative controls. Added a 'Revision History' section to the document.
3	Removed the requirement of company-specific instruments (e.g., Hitachi, EDAX) within section C. Changed and updated section C.2.c.6. so that the number of particles to be confirmed, printed, and kept within case files could be cumulative (i.e., from an entire GSR kit) and not necessarily restricted to three (3) particles per disc/stub.
4	Edited title, removed "Purpose" section. Added "Introduction", "Scope", "Principle", and "Specimens" sections. Edited "Responsibility" section. Added "Equipment/Materials/Reagents", "Standards and Controls", "Calibration", and "Sampling" sections. Edited "Procedure" section to include detailed guidelines for handling of various types of GSR casework, utilization of the SEM and associated detectors, analyzing and confirming particles, the minimum and maximum number of particle types to be confirmed, and the necessary documentation for GSR casework. Added "Decision Criteria" and "Limitations" sections.

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