Title: Elemental analysis of Gunshot Residue (GSR)-related evidence

A. Purpose: To analyze samples for the possible presence of elements relating to gunshot residue (GSR)

B. Responsibility: Analysts authorized to conduct analyses within the GSR category of testing

C. Procedure:

1. Preparation

   Upon starting analysis, document all label information on an appropriate worksheet.

2. Scanning Electron Microscopy Analysis

   A scanning electron microscope (SEM) with a backscatter electron (BSE) detector, secondary electron imaging (SEI) detector, and an energy dispersive X-ray spectroscopy (EDS) detector will be used for this analysis. A copper quality control (QC) reference standard will be analyzed prior to a batch of evidence sample discs to ensure instrument operability. Each QC analysis will be saved, documented, and stored appropriately.. A quality control disc containing known GSR particles (i.e., containing barium, antimony, and lead) will be analyzed monthly and documented accordingly.

   a) Sample Loading – Mount sample and evacuate the specimen chamber.

      1) Manipulate the stem of the stub with tweezers to avoid contamination, never touch the surfaces of the discs with a bare hand. A control blank stub which is used to monitor ambient conditions also is run with each set of samples. Once all stubs are in the coordinated sample holes, close the chamber and then press EVAC on the vacuum control panel.

      2) Wait for EVAC light getting to show steady green. Apply operating voltage, working distance, magnification and spot size on the SEM control screen to appropriate levels. Focus the microscope and adjust Brightness and Contrast knots on the control module to obtain a good image.

   b) Analysis using BSE and EDS

      1) Start the Genesis GSR Analysis program

      2) Enter the sample labeling information. Move and locate the sample stub.

      3) Select BSE (backscatter electron) mode on the SEM control screen and adjust appropriate brightness contrast level.

      4) Start GSR analysis by selecting MULTI STUB.

      5) All stubs will be scanned and analyzed automatically and systematically based on morphology, based on intensity measurements, and based on preliminary elemental determination.

6) Initial data from particles will be automatically classified as possibly containing one or more of the elements: Pb, Sb, Ba by the software. Particles will usually be re-examined manually after the analyses of all of the stubs have been completed.

c) Confirming Elemental Identity using EDS

1) When the analyses from the software are completed, a result report is created and should be printed.
2) Click on the particle number to recall the stored spectrum and designated particle image.
3) If a spectrum reveals the possible presence of lead, antimony, and/or barium, move the stage to the designated particle field and re-collect a new spectrum (e.g., 50 seconds lapse time or until the spectrum reveals enough data to determine if lead, antimony, and/or barium are present).
4) As needed, collect the secondary electron image (SEI) with adequate resolution and clarity.
5) Print out a result page with the particle image and spectrum.
6) During confirmations, when possible, a minimum of three (3) particles per disc, or per kit, will be analyzed, data collected, and data printed when elemental information from those particles will be used in a conclusion or in a report. If fewer than three particles are detected and elemental data will be used in a conclusion or a report, then as many spectra as possible will be obtained, printed, and retained in the case file.
Note: If analysts choose to minimally confirm only three (3) particles per kit, then conclusions within reports must be limited to addressing the entire kit (i.e., 4 stubs, inclusive) and not imply that he conclusions refer to individual stubs. However, if conclusions within reports actually do refer to individual stubs/discs, then three (3) particles per disc must be confirmed and appropriate data retainind within case files.
7) The maximum number of re-examined particles will be at the discretion of the analyst depending on the number of elements detected. However, when at least three (3) particles on a stub are found to contain all three elements (Ba, Sb, Pb) and the morphology of all three particles are consistent with GSR particles, then no further particles on that stub need to be confirmed.
8) Document the result (e.g., using a worksheet) and record all appropriate case and sample information.
9) Instrumental parameters must be recorded within the examination documents (e.g., beam voltage, collection time, magnification, working distance).
10) When a standard four-stub GSR kit has been submitted for analysis (i.e., left-hand back, left-hand front, right-hand back, right-hand front) and one of the stubs has at least three (3) confirmed particles which satisfy step 7 (above), then the remaining stubs should not be analyzed. If

submitting agencies specifically request that all four stubs be fully analyzed, then all four will be analyzed.

11) Remove samples stubs from chamber and put each one back in its original container.

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

13) Appropriate negative and positive controls can be analyzed contemporaneously, if necessary.

3. Reports
   a) Reports will be written based on the data that was obtained from the confirmatory/elemental identification of the particles.
   b) The verbiage of the reports will indicate what elements were confirmed/identified within each stub. When not all of the stubs in a GSR kit were fully analyzed, the report will state this occurrence.

D. 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 1588-94, "Standard Guide for Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive 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.