## Title: SEM/EDS analysis of samples for gunshot residue (GSR)

A. **Purpose:** To analyze samples for the presence of gunshot residue (GSR)

**B. Responsibility:** GSR analyst or designee

**C. Procedure:**

1. Preparation
   a. Upon starting analysis, document all label information on an appropriate worksheet(see attached).

2. Carbon Coater Procedure (when necessary)
   a. If bare aluminum discs were used for collection, they need to be carbon-coated before SEM/EDS analysis. See separate SOP for carbon coating (IN-03) for details.

3. SEM/EDX Analysis

   An automated Hitachi scope and EDAX detector will be used for this analysis. A copper QC check will be run prior to a batch of evidence sample discs. Each QC run will be documented and saved(QC SOPIN05). A quality control disc will be run monthly(see QC SOPIN05).

   I. Sample Loading – follow instructions in the operation manual referring to mounting a sample and evacuating the specimen chamber(Appendix A)

   Notes: 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.

II. Starting Genesis GSR Analysis program – follow instructions in EDAX operator's manual (Appendix B)

Notes: 1) Enter the sample labeling information. Move and locate the sample stub.

2) Select BSE(backscatter) mode on the SEM control screen, adjust appropriate brightness contrast level by turning these knots on the control module.

3) Start GSR analysis by selecting MULTI STUB.

4) All stubs will be scanned and analyzed automatically and systematically.

Initial data from particles classified with one or more of three elements(Pb, Sb and Ba) will be re-examined manually after the analysis of all the stubs has been completed.

III. Confirming Elemental Identity

1) When the analysis is completed, a result report is created.
2) Click on the particle number to recall the stored spectrum and designated particle image.
3) If a spectrum reveals the possible presence of the elements lead, antimony and/or barium, move the stage to the designated particle field, re-collect a new spectrum with about 50 seconds lapse time or until this spectrum reveals the strong presence of one or more of these elements.
4) Collect this image and print out a result table with this particle image and spectrum.
5) Document the result on the appropriate worksheet.- See attached
6) Remove samples stubs from chamber and put each one back in its original container.
7) Store all stubs in the secured evidence storage area.

IV. Reports are written according to criteria in the quality manual(GL-1) andSOPFLIN11



**D. References**

1) Hitachi S-3700N Scanning Electron Microscope Operators Manual on the help menu of the program Software (Appendix A).
2) Edax Genesis GSR Analysis software user's manual and movie (Appendix B).
3) 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.

4) Meng, H.H., Caddy, B., "Gunshot Residue Analysis-A Review", Journal of Forensic Sciences, 1977; Vol.42, No.4, pp.553-570
5) 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.
6) Wolten, et.al, "Particle Analysis for the Detection of Gunshot Residue. II: Occupational and Environmental Particles", JFS, August 1978.
7) GSR Summary, Dennis Ward, FBI Academy.
8) ASTM Designation E 1588-94, "Standard Guide for Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive Spectroscopy.