

Procedures For Gunshot Residue (Gsr) Analysis

The SBI laboratory uses the methods of flameless atomic absorption spectrophotometry (F.A.A.S.) And scanning electron microscopy coupled with energy dispersive x-ray analysis (SEM/EDS) for the analysis of gunshot residue kits. Barium, antimony, and lead are the elements currently being used for gunshot residue analysis. The F.A.A.S. instrumentation currently being used is sold by Perkin Elmer Corp. and consists of the following:

Simaa 6000 Simultaneous Multi element Graphite Furnace Atomic Absorption Spectrophotometer
Transverse Zeeman Graphite Furnace
As-72 Auto-Sampler
Dell Optiplex GX-260 Computer
Oki Microline 320 Printer

The SEM/EDX instrumentation currently being used consists of the following:

- 1) Cambridge (Leica) Steroscan 200 Scanning Electron Microscope
Tracor (Noran) 5500 Energy Dispersive X-Ray Analyzer
Tracor (Noran) 5600 PAC Controller for stage automation control
- 2) LEO 1450 Variable Pressure Scanning Electron Microscope
Oxford Energy Dispersive X-ray Analyzer

Once the evidence has been received from the evidence control technician the following procedure should be followed:

A. Receiving the Evidence

1. Check all laboratory numbers and item numbers to ensure that you are receiving all of the evidence assigned to you.
2. Sign all of the evidence sheets and place your identifying marks on all pieces of evidence in your custody.
3. Store all evidence in a secure area until you are ready to work on the case.

B. Preparation & Analysis of Swabs by F.A.A.S.

Several handwiping kits may be analyzed during the same run. Therefore, extreme care must be taken to identify all samples with the proper numbers. This is best accomplished by opening only one handwiping kit at a time, identifying its contents and preparing each sample for analysis before another handwiping kit is opened. The procedure should be as follows:

1. Remove the selected cases from your storage area, and check all laboratory numbers.
2. Assign each handwiping kit a sequence number and place this number on each piece of evidence ie. #1RB, #1LB, #1RP, #1LP, etc.
3. After the handwiping kit and their contents have been identified, label clean unused test tubes to correspond to each piece of evidence ie. #1RB, #1LB, #1RP, #1LP, etc.
4. Check the number on each item to ensure that they correspond to the correct test tube, then visually and microscopically, if needed, individually inspect each swab for the presence of gunpowder. If gunpowder is observe on the swab, remove it using a tungsten probe and dissect it into three pieces on a microscope slide. Conduct a flame test on one of the pieces by placing it with foceps in an open flame and record the results. Conduct dermal nitrate test on the second piece by placing it in a spot well and dropping one drop of dermal nitrate on the piece record the color change. Prepare the final piece for analysis by **Micro FTIR (Follow analytical and instrumental procedures for analysis of particles by Micro FTIR)**. Then remove the cotton portion of the swab and place it in it's respective tube. Repeat this for all the swabs contained in the kit. Once all swabs are in their respective tubes, place tubes in oven, for drying, at approximately 80 degrees c. for (24) hours.
5. After drying add one (1) ml of 5% nitric acid to each tube, and place the samples in an oven at approximately 80degrees c. For approximately one (1) hour.

NOTE: Most commercial handwiping kits contain two (2) swabs per area of the hand. In this case place both swabs in one test tube and add two (2) ml. of nitric acid instead of 1 ml.

6. At the end of the one hour of heating, remove the samples from the oven and allow them to cool to room temperature. Then using a vortex mixer, agitate the samples until the cotton swab breaks up.
7. Centrifuge each sample for approximately two (2) minutes before decanting the liquid into analyzer cup. The cups are placed onto the auto-sampler.
8. The analysis of barium and lead require that the samples be diluted. Take 50ul. aliquot of sample to bring it up to 1,000ul. Total volume. The analysis of antimony requires no dilution of the samples.
9. The instrument is set up according to the procedure outline in the instrumentation operational manual.

Note: Operating parameters are listed at the end of this procedure outline.

10. Once all samples are analyzed for barium, antimony and lead, the data is printed out and then transferred to the case files.

11. All envelopes, plastic bags, vials, etc. that were used to package the evidence will be returned to the submitting agency along with a copy of the SBI laboratory report.
12. After all work has been double checked, and everything is correct, then the liquid samples may be discarded.

C. Analysis of Tapelifts by SEM/EDS

Each kit should contain four (4) different tapelifts labeled:

- Right Back (RB)
- Left Back (LB)
- Right Palm (RP)
- Left Palm (LP)

Results of F.A.A.S. analysis should be utilized to determine which of the tapelifts from each kit should be examined. Significant levels of Barium, Antimony and Lead are determined by the analyst on a case by case basis. A reference point for significant levels for this laboratory is Barium 0.20ppm., Antimony 0.03ppm., and Lead 0.30ppm. Designated tapelifts should be placed in the SEM in a predetermined ordered and examined both visually and elementally (see SEM/EDS procedures and methods).

Recommended settings are as follows:

- Accelerating voltage: 20kv
- Electron Collector: Backscattered
- Working Distance: 15-20mm
- Specimen Current: 500 PA or less
- X-Ray Dead time: 30% or less (45% for zeppelin program)
- Spectrum Energy Range: 0-20KV
- Manual Search Magnification: 400-500x
- Zeppelin Search Magnification: 100x

Tapelifts are examined for discrete particles of molten metal which are in the micron size range (usually below 20microns in size; most frequently 5 microns and below). These particles often appear spheroid in shape but may have an irregular appearance at times. After noting the size and shape of a particle, and elemental analysis of the particle is performed. Major elements are barium, lead and antimony.

Noted particles can be classified as characteristic of gunshot residue (GSR). Attached is a profiling of possible composition for each type as recognized by a published Aerospace report. Interpretation of the results is covered in the training manual for GSR.

Supervisor Approval _____

Date _____