Laboratory Methods

1 Purpose

The purpose of this manual is to provide the Forensic Scientist (GSR Examiner) with a set of standards and recognized methods for the examination of physical evidence. The methods and practices described apply to casework, proficiency tests and competency tests and may be applicable in other situations, as determined by laboratory management. Deviations from written methods and conventions are at times necessary and are permitted as circumstances dictate. Significant deviations from the methods provided in this manual must be approved by laboratory management and must be accurately reflected in the analyst's notes.

2 Documentation

In addition to the technical record requirements detailed in the Laboratory Quality Assurance manual, the following case documentation practices should be used whenever appropriate:

Photographs of evidence may be taken to document the physical condition of evidence. Annotation of photographs should include at minimum the case number, item number, date, examiner initials. Representative photographs will be included in the case record.

Data produced during an examination may be rejected. If data is rejected, the case record will include the reason the data was rejected, the date the data was rejected and the person rejecting the data. Rejected data will not be used for a report conclusion. Case notes should include a description of the evidence analyzed, the method of sample preparation, the analytical instrumentation used, and its operating parameters, whenever applicable.

Case notes will include a copy of all of the instrumental data.

2.1 Safety Considerations

Standard laboratory safety practices apply to all methods described in this manual (see the Laboratory Safety Manual).

3 Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy (SEM/EDS)

3.1 Introduction

The scanning electron microscope (SEM) is one of the most versatile instruments available for the examination of the microstructural characteristics of solid objects. It has imaging capabilities which provide high resolution, large depth of field and a three-dimensional image at both very high and very low magnifications. Further, these image characteristics are acquired non-destructively and often with little sample preparation.

A biproduct of the electron beam/sample interaction provided by the SEM is the generation of several complimentary useful signals, including x-rays. X-rays exhibit energies that are specific to the elements from which they originate. By combining the SEM with an energy dispersive x-ray spectrometer (EDS), the generated x-rays can be detected and characterized. The SEM/EDS combination provides structural, qualitative and, in some cases, quantitative inorganic compositional information about the sample in question.

3.2 Equipment/Instrumentation

- Scanning electron microscope equipped with an energy dispersive x-ray spectrometer
- Sputter coater
- Stereo microscope

3.3 Minimum Standards and Controls

The SEM/EDS will undergo the following routine maintenance and/or quality checks:

- A positive control (PLANO) and an EDS calibration will be run once a month.
- Preventative maintenance visits for the SEM only will occur yearly and be conducted by the instrument manufacturer.
- Filaments and nitrogen cylinders are changed on an as needed basis.
- Following scheduled preventative maintenance, routine software updates, repair or unusual equipment shutdown, a performance check using the PLANO standard will be run.
- Any modifications to the instrument recipe (other than run termination limits), changing to different analysis software or changing/upgrading the hardware may require re-validation of the system.
- All data and logs for these quality checks will be maintained in a network folder or in LIMS.

3.4 Sample Preparation Procedure

Coating - Some non-conductive samples require treatment to enhance surface conductivity, in order that x-ray analysis may be performed optimally. Insufficient conductivity may result in poor imaging of the sample and beam deflection from the intended analysis area.

3.5 Instrument Requirements and Operation

Most commercial-grade SEM/EDS systems should be adequate for GSR analysis given that the following criteria are met. Automated data collection of GSR involves some portion of the data collection being controlled by the instrument automation software. The extent to which the SEM and EDS systems communicate and are integrated varies according to the manufacturers and the capabilities of the hardware/software. The system shall have the ability to recall stage locations of particles for verification and software for particle recognition.

The following suggested instrument operating conditions are meant as general guidelines or starting conditions. Actual requirements may vary as the analyst determines specific analytical needs.

- Beam voltage is increased when higher energy line excitation is required.
- Beam voltage is decreased when greater spatial resolution is required.
- Pulse processor time constant is lengthened when greater spectral resolution is required.
- Pulse processor time constant is shortened when a greater count rate is required, (for construction of elemental distribution maps).
- Detector to sample distance can be reduced or increased to increase or decrease x-ray collection efficiency.
- Spectral energy display scale is expanded when sufficient detail is not evident.
- Beam current is increased when the X-ray count rate is too low. Decreasing the condenser lens current and/or increasing the final aperture size may increase beam current.
- Beam current is decreased when the X-ray count rate is too high. Increasing the condenser lens current and/or decreasing the final aperture size may decrease beam current.

3.5.1 Scanning Electron Microscope (SEM)

- The SEM, operating in backscattered electron imaging mode, shall be configured to detect particles down to at least 1.0µm in diameter.
    - This parameter was tested during in-house validation, performance checks and monthly with the PLANO standard.
- The SEM shall be capable of an accelerating voltage of at least 20 kV.
- SEM systems shall include a motorized stage with automated stage control.

3.5.2 Energy Dispersive X-Ray Spectrometry (EDS)

- The detector shall be configured to produce a resolution of better (less) than 150 eV during analysis, measured or extrapolated as the full width at half the maximum height of the Mn Kα peak.
    - Measurement acquired by the manufacturer during installation, as well as during in-house validation and performance checks.
- At minimum, the EDS spectrum shall be acquired at 20 eV per channel.
- Displaying the EDS output shall encompass the X-ray lines of analytical utility, with a minimum range of 0-15 keV.
    - The examiner shall set this during the preparation of the spectra for data output.
    - The range can be reduced when attempting to focus on finer peak detail. However, it must be accompanied by a spectrum in the 0-15keV range.
- Automated systems will also include software capable of acquiring X-ray spectra for a specified collection time or total X-ray counts.
    - Acquisition of spectrum is set to a minimum of 0.50 seconds. However, additional time can be set per the preference of the analyst.
- The instrument shall record spectrum from the analysis and store all of the particle location coordinates from the candidate particles.

3.5.3 Imaging Analysis

1. Utilizing the secondary electron (SE) signal detector, optimize instrument operating conditions as dictated by the sample to be examined.
2. Beginning at low magnification, focus and proceed to higher magnifications, as needed.
3. A backscattered electron image is useful for defining structures based on the average atomic number of the matrix. Structures containing elements with higher atomic numbers will generally appear brighter than those with lower atomic numbers. This is often useful for evaluating homogeneity and layer structure.
4. Photographically document or print visual image(s). SEM micrographs should include a measuring scale or magnification scale or both. The micrograph should also display which signal (backscattered electron or secondary electron) was used to produce the image.

3.5.4 Particle Analysis

- Evaluation of composition may be achieved by the analysis of specific particles. Generally, these particles appear bright in the backscattered electron image. An EDS spectrum shall be collected from the candidate particle by placing the electron beam in spot mode near the center of the particle or raster an area completely within the particle's volume. X-ray counts should be collected to permit robust manual identification of all elements of interest.

    - Because the beam interaction volume may be considerably larger than an individual particle, inclusion of other matrix components may be expected in the spectrum from an individual

particle. Lower beam voltages may be used to confine more of the interaction volume to the particle. It should be noted, however, that the use of lower beam voltages may result in the loss of characteristic lines that may be found at higher energies.

3.5.5 Qualitative analysis

- Once an X-ray spectrum is collected, a qualitative analysis is performed to determine the elements present. The process is straightforward for the peaks of elements present in major amounts and those not overlapping. Misidentification or omissions of minor components are possible unless a systematic approach to elemental identification is used which includes consideration of X-ray line families, spectral artifacts, escape peaks, sum peaks, and overlaps.
- Reference lines, or energies, may be obtained from several sources; including energy slide rules, published tables, and computer-generated KLM reference lines that may be superimposed on the spectrum. Additionally, manufacturers often provide an automatic element identification application. These aids often are used in complementary fashion.
- Identification begins with high-energy peaks and major peaks. High-energy peaks are generally less likely to overlap than lower energy peaks. If a major peak is present, generally a complete family of peaks can also be identified. Spectral artifacts, including sum peaks and escape peaks associated with major peaks, should be evaluated.
- As spectral interpretation alternates between the identification of major and minor peaks, the vertical (counts) scale should be adjusted to reveal required detail. In addition to the higher energy peaks, the presence of any lower energy families and their expected relative intensities should be noted. Individual asymmetric peaks and inconsistent peak ratios within a family may indicate a peak overlap. Superimposing and scaling KLM reference lines on the spectrum or referencing the actual spectrum of an elemental standard aids elemental identification. The analyst should be familiar with the characteristic pattern and relative intensities of peaks of various atomic numbers. The identification of major elements is usually straightforward.
- Following the identification of major elements, lower intensity peaks and overlapped peaks are identified. The limited number of characteristic peaks present for minor elements can limit their identification.
- The presence of an element can be considered unequivocal only when a distinctive, unique set of lines is produced or when a single peak occurs at an energy where it cannot be mistaken for another element or spectral artifact. Unequivocal identification may not be possible if an element is present in low concentration or if lines required for confirmation overlapped with the lines of other elements.
- Spectrum should be displayed on a scale that clearly demonstrates the peaks identified. To display peaks from elements with significant differences in concentration, the peaks from the elements in low concentration may be viewed by displaying the spectrum separately on different display scales.
- There may be an overlap of peaks in the energy dispersive X-ray spectroscopy spectrum of materials containing several elements. In order to resolve these overlaps, several methods may be employed.
    - The live time count can be increased.
    - The processing time of the pulse processor may be increased to improve spectral resolution.

- Mathematical spectral subtraction (deconvolution) methods supplied by the energy dispersive X-ray spectrometer manufacturer can be employed.
- An alternative method of elemental analysis or X-ray diffraction may be used.

3.6 References

1. Friel, J. J., “X-Ray and Image Analysis in Electron Microscopy”, Princeton Gamma Tech, Princeton, N. J., 1995.
2. Goldstein, J. I., et al., “Scanning Electron Microscopy and X-Ray Microanalysis”, second edition, Plenum Press, New York, New York, 1992.
3. Lifshin, E., “Advances in X-Ray Analysis”, Vol. 19, Kendall/Hunt, Dubuque, Iowa, 1975

4 Gunshot Primer Residue Analysis

4.1 Introduction

When a weapon is fired, several events occur immediately after the primer is struck. Initially the primer composition is crushed, causing ignition of the mixture. Ignited primer moves forward and ignites the propellant. Burning propellant produces a large volume of highly pressurized gases which force the projectile(s) out of the cartridge case and down the weapon barrel. As this happens, gases vent out of the barrel, cylinder gap, ejection port or other unsealed areas. These gases then rapidly cool and deposit on any nearby surface. The products of firearm discharge are collectively referred to as gunshot residue (GSR). Collection of GSR and subsequent analytical identification of primer components lead, barium and antimony can be used to associate an individual with a discharged firearm.

4.2 Minimum Standards and Controls

4.2.1 Analytical QA/QC

Known gunshot residue samples (positive controls) are to be analyzed under the following conventions:

4.2.1.1 A synthetic GSR positive control will be analyzed once per month. Documentation of the monthly positive control runs must be retained in a designated log.

- The monthly positive control shall be considered to have “passed” if the automated run finds and correctly identifies at least 90% of the 1 µ and larger Pb-Ba-Sb particles present on the analysis area of the sample.
- The positive control shall be considered to have “failed” if the appropriate minimum threshold of Pb-Ba-Sb particles cannot be identified. If the positive control fails, the instrument in question will not be used for GSR evidence analysis until the problem can be identified and corrected.

4.2.1.2 A laboratory produced GSR positive control will be analyzed for each case and will be run before and after the analysis of the sample stubs and prior to the analysis of the negative control.

- If additional case sample stubs are analyzed in a new instrument batch run, another positive control will be analyzed in the manner described above.
    - For the purposes of this method, a “batch” refers to all the sample stubs loaded onto the SEM instrument stage, intended to be run continuously or until all the samples on the stage have been analyzed. A “batch” of samples may include multiple items and/or cases.
- The positive control shall be considered to have “passed” upon the automatic identification and subsequent user confirmation of at least 3 Pb-Ba-Sb particles.
- A copy of the positive control sample analytical data must be retained as part of the case notes for each case.
- If both positive controls fail during a case run, the data shall be rejected and the case shall be fully re-run.
- The positive control sample should be stored to protect it from loss and degradation.

4.2.1.3 A negative control will be analyzed for each case and will be run after the analysis of the sample stubs and the second positive control. These samples will be of like design and subjected to the same preparation and analysis procedures as case samples.

- A negative control shall be considered to have "passed" if no Pb-Ba-Sb particles are identified. These stubs should be discarded after the analysis has been completed.
- A copy of the negative control sample analytical data must be retained as part of the case notes for each case.
- The negative control shall be considered to have "failed" upon the automatic identification and subsequent user confirmation of at least one Pb-Ba-Sb particle.
- If the negative control fails, any findings in that case shall be considered inconclusive.
- A new negative control will be used with each batch run.

4.2.2 Beam Current Stability

- The SEM beam current stability is set to be measured every thirty minutes using an appropriate reference sample (such as copper, cobalt, etc.).

4.2.3 Contamination control

- Sample preparation and analysis must be conducted in an environment that restricts potential gunshot residue contamination.
- All sample manipulation utensils must be cleaned with methanol (reagent grade or better) between samples.
- Cleaning of the sample preparation area and analysis equipment must be performed prior to each run.

4.3 Sample Preparation Procedure

4.3.1 Hand Samples

1. Clean countertop surface with cleaning wipes or methanol prior to exam.
2. Open evidence packaging and label packaging with case number, analyst initials, date and item number.
3. Mark vials with case number, analyst initials, date and item number.
4. If opening multiple cases, open evidence from one case and label before moving on to the next.
5. Scan datasheet if present.
6. Vent SEM and place stage in the load position.
7. Clean tweezers with methanol and dry on kimwipe. Use a new kimwipe per sample stub.
8. Remove the cap with the attached aluminum stub from the vial. While holding the cap, use the tweezers to remove the stub and place stub onto the stage.
9. Stage will be loaded from lower to higher number positions to prevent accidental contact with tweezers and/or other stubs.
10. Record the stage position in case notes.
11. Repeat steps 3-6 for each loaded sample.
12. Positive control sample will be located in position 1; negative control sample is located in position **4.**
13. Tighten stage screw for all samples loaded before **closing the SEM door.**
14. Pump the SEM to begin run setup.

15. When run is finished, vent SEM and place stage into load position to begin sample removal.
16. Unscrew all samples to be removed.
17. Clean tweezers with methanol and dry on kimwipe. Use a new kimwipe per sample stub.
18. Starting with the last sample loaded, remove the cap from the vial and place the finished stub into the cap and return to the vial. **Repeat until all samples have been removed.**
19. Wipe the stage with a cotton swab soaked in methanol to remove any debris.

4.3.2 Item Samples (clothing and surfaces)

1. Clean countertop surface prior to exam.
    - Cleaning wipes or methanol can be used. However, if the item has a DNA co-assignment, use bleach solution prepared by FB/DNA section.
2. Place item onto clean brown paper.
3. Take a photo of the item, with an included ruler, for documentation.
4. Label new sample vials with case number, analyst initials, date, item number and description of sample location.
5. Remove the cap with attached aluminum stub from vial.
6. While holding cap, repeatedly press collecting surface on sample area until desired area has been sampled, or until collection surface loses its adhesive quality.
7. Return the cap and stub to the vial.
8. Repeat steps 6 through 8 as necessary.
9. Fold item and roll brown exam paper around it, securing with tape.
10. Return item to original packaging and seal for return.
11. Sample collected stubs in LIMS and place into manila envelope.
12. Follow sample loading and unloading directions under the hand samples.
    - For item samples only, only one item's collected stubs can be run at a time. If more than one item is in a single item package, all the sample stubs can be run together.

4.3.3 Analytical progression

The intent of this test is to establish the presence or absence of particles characteristic of gunshot primer residue and to associate that finding with an individual. This is generally best accomplished by the identification of Pb-Ba-Sb particles on samples collected from the individual's hands. The analysis of clothing or other items generally does not satisfy the criteria given above and should be discouraged, however, they may be sampled and analyzed when the case facts warrant it.

4.3.4 Batching

- Multiple GSR kits may be run with each batch to efficiently utilize the SEM sample stage.
- Multiple cases may be analyzed together to accomplish this.

4.3.5 Hand samples

- Hand samples will be analyzed preferentially over non-hand samples unless special circumstances exist.

4.3.6 Item samples (clothing and surfaces)

- Clothing and/or surface samples associated with an individual or item may be analyzed if hand samples test negative or are unavailable.
- Sample progression will move from areas most likely to contain GSR towards areas less likely, based on case facts.
- Analysis of samples associated with one individual or item can be halted when one sample is found to be positive or when a sufficient representation of an area or an item has been analyzed.

4.4 Testing Parameters

The following recipe setup should be used for SEM/EDS GSR analysis:

GSR Recipe – Tescan Vega 4

SEM Microscope Settings	**SEM Settings**
Image Scan Size: 1024	Acc. Voltage (keV): 25
Dwell Time (µs): 2	Working Distance (mm): 15
Input Signal: BSE	Dead time on Cobalt (%): 45 to 55
Perform in-run threshold adjustment: Yes	
Deviation Limits:	
Above Starting Level (%): 30	
Below Starting Level (%): 20	
Interval (minutes): 30	
Sampled Area (pixels): 63 x 63	
Brightness/Contrast levels: 3500 (carbon)/26000 (cobalt)	
Feature Image Settings	**Detection Refinement Settings**
Image width (pixels): 1024	Gray Level Filters
Dwell Time: 2	Feature Image: None
Input Signal: BSE	Thresholds
Number of Frames: 1	Lower: 12000
Frame Time (s): 1.573	Upper: 32767
	Merge thresholds: No
	Invert thresholds: No
	Binary Image Filters: None
Feature Detection Settings	
Smallest Feature Size (pixels): 1	
Magnification: 800	
Allow Threshold Overlaps: No	
Disable large area field overlap rules: No	

Feature Analysis Settings	Analysis & Filter Options Settings
First Pass Dwell Time (µs): 2	Filter by Morphology: No
Second Pass Imaging: Yes	First EDS Acquisition Time (s): 0.50
Second Pass Dwell Time (µs): 40	Acquire Second Pass EDS: Yes
Leading pixels: 20	EDS Filter: Pb Ba Sb
Trailing pixels: 10	No Further EDS Analysis.
Morphology measurements only: No	Reject Features: No
Scan Mode: Scan Feature	Additional Time (s): 1.00
Energy Range (keV): 20	Acquire Third Pass EDS: No
Number of Channels: 2048	
Process Time: 3	
Acquisition Mode: Live Time	
Acquisition Time (s): 0.50	
Pulse Pile Up Correction: Yes	

Quant Setup	AutoID Settings
Processing Options: All Elements	Perform AutoID during acquisition: Yes
Normalize Results: Yes	Perform AutoID during requantification: No
Correct for window artefacts: No	
Deconvolution elements: Carbon, Oxygen	
Quant standardizations: Factory (Quant Standardizations (Extended Set)	
Threshold Quantitative Results	
Enable thresholding: No	
Sigma level: 3.0	
Element list: Current Spectrum	
Automatic line selection for all elements: Yes	
Classification list used: Ohio BCI	

Feature Automation Run Settings	Run Termination Settings
Restore column conditions for every area: Yes	Field
Turn beam off at the end of the run: Yes	Features: Stop analysis after 50 features
Auto brightness/contrast during run: No	Classification: None
Microscope auto focus during run: No	Time: None
Microscope auto brightness/contrast during run: No	Area
Align fields during acquisition: No	Features: None
Reconstruct feature across fields: No	Combined: None
Default Field Acquisition Order: Sequential	Time: None
	Options: None
	Specimen
	Features: 199000
	Classification: Stop analysis after 10 features detected in Rank 1 (for positive control, stop after 5 features)
	Time: None
	Options: None
Run Area Layout Settings	
Field coverage: inside	
Field acquisition: Sequential	
Magnification: 800	
Refine Field Layout	
Overlap: 0%	
Refine Acquisition	
Edge Boundary (mm): 0.00	

4.5 Automated Particle Classification List

Rank 1: Characteristic of GSR	Pb-Sb-Ba	
Rank 2: Consistent With GSR	Ba-Sb	
	Pb-Sb	
	Pb-Ba	
	Pb-Ba-Ca-Si	
	Ba-Ca-Si	
	Ba-Al	
Rank 3: Commonly Associated With GSR	Pb	
	Ba	
	Sb	
Rank 4: Commonly Associated With Lead-Free/Nontoxic Primers	Gd-Ti-Zn	
	Ga-Cu-Sn	
	Ti-Zn	
	Sr	
Rank 5: Commonly Associated With Airbags	Cu-Co	
	Zr	

Rank 6: Environmental/Other Elements	Cu-Ni	Fe
	Brass	Ti
	Ni	ZN
	Sn	Al
	Cu-Ni	Al-Ca
	Fe-Ni	Ca
	Bronze	Al-Fe
	Ni-Cr	Co
	W	Barium Sulfate
	Au	Zinc Sulfate
	Stainless Steel	Salt NaCl
	Lighter Flint Ce	Salt KCl
	Lighter Flint La	Bi
	Ag	

4.6 Morphology

GSR particles are often spheroid and between 1 to 10µ in size. They can, however, be irregular in shape and range from 1 to 100+µ in size. In general, they should not display a crystalline structure. Since morphology can vary greatly, it should never be considered as the only criterion for identification of GSR.

4.7 Elemental Composition

Particles classified as characteristic of GSR will have the following elemental composition:
Lead (Pb), Barium (Ba) and Antimony (**Sb)**

4.8 Data Collection

An automated SEM/EDS system collects elemental data from particles whose backscattered electron signal brightness exceeds the desired threshold setting. An electronic copy of the data including, at minimum, stage X and Y coordinates, total number of particles detected, and total number of particles classified as GSR will be included in the analyst notes.

Automated sample analysis may be terminated when any of the following criteria are met:

- Entire user described area has been analyzed.
- A total of ten (10) Pb-Ba-Sb particles have been identified on any combination of samples from an individual, single surface or item.
- A user specified time or total particle maximum (199,000) has been reached.
    - If the sample is positive when it reaches the total particle maximum, no further analysis is necessary. However, if the sample is negative when it reaches the total particle maximum, the sample should be turned 180 degrees clockwise from the analysis starting edge in order to maximize the coverage of the stub and run a second time.

Attended sample analysis may be terminated at any time, upon identification of characteristic gunshot residue particle(s).

Pb-Ba-Sb particles identified through automated analysis shall only be considered confirmed after manual relocation and re-acquisition of the X-ray spectrum (as described in section 3.5 of this manual). A detailed, quality image and spectrum of each confirmed GSR particle should be included in the case notes. At minimum, the relevant elements to be labeled shall be Lead, Barium and Antimony.

4.9 Interpretation Criteria

The following possible conclusions can be reached after evaluating GSR samples:

Positive:

A positive finding for the presence of particles characteristic of gunshot primer residue shall occur with the automatic identification and subsequent user confirmation of at least *one* Pb-Ba-Sb particle exhibiting characteristic GSR morphology.

Major amounts of sulfur, barium, magnesium, sodium, cobalt, manganese, zirconium, chromium, or titanium may be indications of non-firearm sources. Refer to the cited materials in this section for more information.

For example, individual characteristic Pb-Ba-Sb particles should NOT contain:

- major levels of iron (indicates particles consistent with brake dust)
- major levels of aluminum and magnesium (indicates particles consistent with fireworks)
- major levels of copper, cobalt and zirconium (indicates particles consistent with airbags)

Negative:

A negative finding for the presence of particles characteristic or consistent of gunshot primer residue shall occur when the above conditions for a “positive” finding cannot be met on any analyzed sample associated with an individual or item.

Inconclusive:

An inconclusive finding for the presence of particles characteristic of gunshot primer residue shall occur when the negative control run with that batch of samples is also deemed to be positive.

4.10 References

1. SWGGSR. “Guide for Primer Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive X-Ray Spectrometry.” November 29, 2011. Web. 03 Feb. 2012.<http://www.swggsr.org/documents.html>
2. Wolten, G. M., Nesbitt, R. S., Calloway, A. R., Loper, G. L., and Jones, P. F., “Final Report on Particle Analysis for Gunshot Residue Detection,” Report ATR-77 (7915)-3, The Aerospace Corporation, El Segundo, CA, September 1977.
3. Wolten, G. M., Nesbitt, R. S., Calloway, A. R., Loper, G. L., and Jones, P. F., “Particle Analysis for the Detection of Gunshot Residue I: Scanning Electron Microscopy/Energy Dispersive X-Ray Characterization of Hand Deposits from Firing,” Journal of Forensic Sciences, Vol. 24, No. 2, April 1979, pp. 409-422.
4. Wolten, G. M., Nesbitt, R. S., Calloway, A. R., Loper, G. L., and Jones, P. F., “Particle Analysis for the Detection of Gunshot Residue. II: Occupational and Environmental Particles,” Journal of Forensic Sciences, Vol. 24, No. 2, April 1979, pp. 423-430.
5. Wolten, G. M., Nesbitt, R. S., Calloway, A. R., Loper, G. L., and Jones, P. F., “Particle Analysis for the Detection of Gunshot Residue. III: Case Study,” Journal of Forensic Sciences, Vol. 24, No. 4, October 1979, pp. 864-869.
6. Mosher,P.V., et.al., “Gunshot Residue- Similar Particles Produced by Fireworks,” Can Soc. Forens.Sci. J., 31 (20) 1998.
7. Berk, R.E. Automated SEM/EDS Analysis of Airbag Residue. I. Particle Identification. Journal of Forensic Sciences, Vol. 54, No. 1, January 2009, pp. 60-68.
8. Berk, R.E. Automated SEM/EDS Analysis of Airbag Residue. II: Airbag Residue as a Source of Percussion Primer Residue Particles. Journal of Forensic Sciences, Vol. 54 No. 1, January 2009, pp. 69-76.
9. Torre, C.A Source of Non-GSR Particles containing Lead, Barium and Antimony, Journal of Forensic Sciences, 2002, (47), pp. 474-504.
10. Wallace, James Smyth. “Chemical Analysis of Firearms, Ammunition, and Gunshot Residue”, CRC Press, 2008.
11. Goldstein, J. I., et al., “Scanning Electron Microscopy and X-Ray Microanalysis”, second edition, Plenum Press, New York, New York, 1992.
12. Gunaratnam, L., and Himberg, K., “The Identification of Gunshot Residue particles from Lead-free Sintox Ammunition,” Journal of Forensic Sciences, Vol 39, 1994, pp. 532–536.
13. Romolo, F. S., and Margot, P. “Identification of Gunshot Residue: A Critical Review,” Forensic Science International, Vol 119, No. 2, 2001, p. 195.
14. Basu, S., “Formation of Gunshot Residues,” Journal of Forensic Sciences, Vol 27, 1982, p. 72.

15. Harris, A., “Analysis of Primer Residue from CCI Blazer* Lead-free Ammunition by Scanning Electron Microscopy/Energy Dispersive X-ray,” Journal of Forensic Sciences, Vol 40, 1995, pp. 27–30.
16. Garofano, L., et al., “Gunshot Residue—Further Studies on Particles of Environmental and Occupational Origin,” Forensic Science International, Vol 103, 1999, pp. 1–21.
17. Berk, R., “Automated SEM/EDS Analysis of Airbag Residue. 1: Particle Identification,” Journal of Forensic Sciences, Vol 54, No. 1, January 2009, pp. 60–68.
18. Zeichner, A., and Levin, N., “More on the Uniqueness of Gunshot Residue (GSR) Particles,” Journal of Forensic Sciences, Vol 42, No. 6, November 1997, pp. 1025–1026.
19. ASTM International. “Standard Practice for Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive X-Ray Spectrometry.” E1588-20.

5 Suggested Report Wording

Laboratory reports are generated in accordance with the accreditation requirements. Reports may include a Conclusions Table, a Remarks section, an Analytical Detail section or a narrative that ensures compliance with the Laboratory Quality Assurance manual policies.

5.1 Conclusion Table

Item – List all items received for analysis.

Description – Provide an item description. For example: GSR kit from John Doe, GSR collected from jacket.

Findings – Provide the examination findings.

Finding	Suggested Report Wording
Pb-Ba-Sb identified –one or more samples not analyzed	One or more particles characteristic of gunshot primer residue were identified on one of the samples. Analysis was not completed on the remaining sample.
Pb-Ba-Sb identified – all samples completed, all samples positive	One or more particles characteristic of gunshot primer residue were identified on the samples.
Pb-Ba-Sb identified – all samples completed, one sample positive	One or more particles characteristic of gunshot primer residue were identified on one of the samples.
No Pb-Ba-Sb found – all samples completed	One or more particles characteristic of gunshot primer residue were not identified on the samples.
Inconclusive – negative control failure	Due to a control failure, the results of the analysis were deemed inconclusive.
Not tested	--

Conclusions – Provide the conclusions of all testing performed on evidence. For example: Positive, negative, inconclusive, not tested.

5.2 Qualifying Statements

5.2.1 Hands

For GSR particles identified or not identified on hands:

Particles classified as characteristics of gunshot primer residue (those containing Pb-Ba-Sb) have compositions rarely found in particles from any other source.

A finding of “positive” for particles characteristic of gunshot residue on a person’s hands means that individual either discharged a firearm, was in the vicinity of a firearm when it was discharged or handled an item with gunshot residue primer on it. The number of confirmed particles cannot be used to determine which of these scenarios occurred.

A finding of "negative" for particles characteristic of gunshot primer residue does not preclude the possibility of any of the above stated events.

5.2.2 Items

For GSR particles identified or not identified on items:

Particles classified as characteristics of gunshot primer residue (those containing Pb-Ba-Sb) have compositions rarely found in particles from any other source.

A finding of “positive” for particles characteristic gunshot primer residue on an item means that the item, at some time in its history, was in the vicinity of a firearm when it was discharged or came into contact with another item with gunshot residue on it. The number of confirmed particles cannot be used to determine which of these scenarios occurred.

A finding of "negative" for particles characteristic of gunshot primer residue does not preclude the possibility of any of the above stated events.

5.2.3 Hands and Items

For GSR particles identified or not identified on hands and items:

Particles classified as characteristics of gunshot primer residue (those containing Pb-Ba-Sb) have compositions rarely found in particles from any other source.

A finding of “positive” for particles characteristic of gunshot residue on a person’s hands means that individual either discharged a firearm, was in the vicinity of a firearm when it was discharged or handled an item with gunshot residue primer on it. The number of confirmed particles cannot be used to determine which of these scenarios occurred.

A finding of “positive” for particles characteristic gunshot primer residue on an item means that the item, at some time in its history, was in the vicinity of a firearm when it was discharged or came into contact with another item with gunshot residue on it. The number of confirmed particles cannot be used to determine which of these scenarios occurred.

A finding of "negative" for particles characteristic of gunshot primer residue does not preclude the possibility of any of the above stated events.

5.2.3 Additional Statements

Circumstance	Suggested Report Wording
GSR evidence analyzed in Richfield/report written in Bowling Green	The GSR instrumental analysis of the above evidence was performed in the Richfield laboratory; the interpretation of the data was performed in the Bowling Green laboratory.
GSR evidence analyzed in Richfield/report written in London	The GSR instrumental analysis of the above evidence was performed in the Richfield laboratory; the interpretation of the data was performed in the London laboratory.
Report re-written	This report replaces the report dated (original report date), originally issued by Forensic Scientist, (analyst name), due to unavailability for testimony.
Routine Quality Assurance re-examination	The above listed item(s) was re-examined for quality assurance purposes and the findings concur with those listed on the report issued by (analyst name) dated (original report date).
Evidence re-examination	The above listed item(s) was re-examined. This report replaces the report dated (original report date), originally issued by Forensic Scientist (analyst name), due to unavailability for testimony.
Returned Evidence (initial analysis)	The evidence is being returned to your department for retention.
Returned Evidence (re-write)	The evidence was previously returned to your department for retention.

6 Report Content Examples

Example GSR

To:	Police Department	BCI Laboratory Number:	xx-xxxxx
	Det.		
	Road	Analysis Date: July 14, 2019	Issue Date: July 15, 2019
		Agency Case Number:	xxxxx
Offense:	Weapons under disability		
Subject(s):	John Doe		
Victim(s):	State of Ohio		

Submitted on May 24, 2019 by Steve:

1.
2.
3.
4.
4.1 :cted from hat

Item	Description	Finding	Conclusion
1	GSR Kit from John Doe	A particle characteristic of gunshot primer residue was identified on one of the samples.	Positive
2	GSR Kit from Jane Doe	Particles characteristic of gunshot primer residue were identified on the samples.	Positive
3	GSR Kit from Jim Smith	Particles characteristic of gunshot primer residue were not identified on the samples.	Negative
4.1	GSR samples collected from hat	Particles characteristic of gunshot primer residue were identified on one of the samples. Analysis was not completed on the remaining sample(s).	Positive

Remarks

Particles classified as characteristics of gunshot primer residue (those containing Pb-Ba-Sb) have compositions rarely found in particles from any other source.

A finding of "positive" for particles characteristic of gunshot primer residue on a person's hands means that individual either discharged a firearm, was in the vicinity of a firearm when it was discharged or handled an item with gunshot primer residue on it. The number of confirmed particles cannot be used to determine which of these scenarios occurred.

A finding of "positive" for particles characteristic of gunshot primer residue on an item means that the item, at some time in its history, was in the vicinity of a firearm when it was discharged or came in contact with another item with gunshot primer residue on it. The number of confirmed particles cannot be used to determine which of these scenarios occurred.

A finding of "negative" for particles characteristic of gunshot primer residue, does not preclude the possibility of any of the above stated events.

The GSR instrumental analysis of the above evidence was performed in the Richfield laboratory; the interpretation of the data was performed in the Bowling Green laboratory.

The evidence is being returned to your department for retention.

Analytical Detail
These findings were determined using scanning electron microscopy/energy dispersive x-ray spectroscopy analyses.

7 Appendix I: Note Abbreviations

Brn = Brown	~ = Approximately	S = Suspect
Blk=Black	+ = And	SO = Sheriff's Office
BPB = Brown Paper Bag	LB, LHB = Left Hand Back	SS = Submission Sheet
Cl = Clear	LH = Left Hand	V = Victim
Cont = Containing	LP, LHP = Left Hand Palm	Unk = Unknown
Contr = Container	NA, N/A = Not Applicable	w/ = With
Evid=Evidence	NC = Negative Control	w/o = Without
Env = Envelope	Neg., (-) = Negative	
HS = Heat Sealed	PC = Positive Control	
Man = Manila	Pos., (+) = Positive	
ME = Manila Envelope	RB, RHB = Right Hand Back	
MCE = Manila Coin Envelope	RH = Right Hand	
Mkd = Marked	RP, RHP = Right Hand Palm	
Pa = Paper		
PB = Paper Bag		
Pkg = Package		
Pkt = Packet		