Technical Procedure for the Examination of Gunshot Residue Evidence

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- **1.0 Purpose** This technical procedure shall be followed for the examination of gunshot residue evidence.
- **Scope** This procedure applies to gunshot residue (GSR) collection kits, clothing and adhesive lifts collected from surfaces other than hands.
- **3.0 Definitions** N/A

4.0 Equipment, Materials, and Reagents

4.1 Equipment

- Oven
- Inductively Coupled Plasma/Mass Spectrometer (ICP/MS)
- Scanning Electron Microscope/Energy Dispersive X-Ray Detector (SEM/EDX)
- Fourier Transform Infrared Spectrometer (FT-IR)
- NIST Traceable Thermometer

4.2 Materials

- Cotton swabs
- Polypropylene 13x100 mm test tubes
- Polypropylene 15 mL conical tubes
- Polypropylene 50 mL centrifuge tubes
- Eppendorf 1000 μL pipette
- Eppendorf 10 mL pipette
- Adhesive lifts
- Spot Plate
- 100 mL volumetric flask
- 1000 mL volumetric flask
- Graduated cylinder
- Scissors
- Test tube rack(s)

4.3 Reagents

- HPLC grade water
- Nitric acid Optima grade
- 10 % nitric acid
- 5 % nitric acid
- 1 % nitric acid
- 1000 µg/mL barium (Ba) standard
- 1000 μg/mL antimony (Sb) standard
- 1000 µg/mL lead (Pb) standard
- 1000 µg/mL indium (In) standard
- 1000 µg/mL lutetium (Lu) standard
- Internal standard (0.2 µg/mL In/Lu in 10 % nitric acid)

- GSR standards 1, 2, 3, 4, 5, 6
- GSR control standards (Blank, QC1, QC2)
- Diphenylamine solution
- **4.3.1** See **Appendix 1** for reagent preparation instructions.

5.0 Procedure

5.1 Antimony (Sb), barium (Ba), and lead (Pb) are the three most common elements associated with gunshot residue.

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- **5.2** Each GSR collection kit shall contain the following cotton swabs and adhesive lifts that were used to collect evidence from the hands of an individual.
 - **5.2.1 Five pairs of cotton swabs,** in vials labeled:
 - Control
 - Right Back
 - Left Back
 - Right Palm
 - Left Palm
 - **5.2.1.1** Each vial of cotton swabs shall contain two swabs.
 - **5.2.2 Four adhesive lifts,** labeled:
 - Right Back
 - Left Back
 - Right Palm
 - Left Palm
- 5.3 Analytical Approach for GSR hand kit examination
 - **5.3.1** Review the request for analysis.
 - **5.3.2** Assign a unique sequential number to each GSR collection kit upon receipt from the evidence technician.
 - 5.3.3 Open kit. Label each piece of evidence within the kit with the unique sequential number, the date, and the Forensic Scientist's initials. This information, plus the Laboratory case number, shall be placed on the cotton swab vial labeled Control.
 - 5.3.4 If a GSR Analysis Information Form is present in the kit, label the top of the form with the Laboratory case number, unique sequential number, Laboratory item number, Forensic Scientist's initials, the date, and the subject's last name.
 - 5.3.5 Scan the GSR Analysis Information Form into Forensic Advantage (FA). If the form is not present in the kit, note this in the FA GSR case record worksheet.

- **5.3.6** Determine if the GSR collection kit is suitable for examination.
 - **5.3.6.1** If the GSR collection kit contains adhesive lifts only (no cotton swabs), analyze the adhesive lifts utilizing the Trace Evidence Section <u>Technical Procedure for SEM/EDX for GSR Casework</u>.
 - **5.3.6.2** GSR collection kits that meet one or more of the following criteria shall not be examined and a report shall be generated.
 - **5.3.6.2.1** Wooden handled cotton swabs were used for the collection of the cotton swab handwipings.

- **5.3.6.2.2** The GSR Analysis Information Form revealed that the subject had washed his/her hands prior to GSR hand kit collection.
- **5.3.6.2.3** The GSR Analysis Information Form revealed that a time greater than 4 hours had elapsed between discharge of weapon and collection of GSR hand kit.
 - **5.3.6.2.3.1** This does not apply to GSR hand kits collected from incapacitated or deceased subjects.
- **5.3.6.2.4** The cotton swab handwipings were not properly collected.
- **5.3.7** If the GSR collection kit is suitable for examination, label five polypropylene test tubes with:
 - C & unique sequential number
 - RB & unique sequential number
 - LB & unique sequential number
 - RP & unique sequential number
 - LP & unique sequential number
- **5.3.8** Visually inspect the cotton portion of the swabs for the presence of burned or unburned particulates.
- **5.3.9** If particulates are observed, remove and perform the following methods for conformation and record the results:
 - 5.3.9.1 Diphenylamine Spot Test
 - **5.3.9.1.1 Purpose** A color test to indicate the presence of oxidizing ions such as nitrates, nitrites, chlorates, and ferric ions.
 - **5.3.9.1.2** Procedure
 - **5.3.9.1.2.1** Place the diphenylamine color test solution in a white spot plate. The solution should be clear and colorless.

- **5.3.9.1.2.2** Add particulate to the solution.
- **5.3.9.1.2.3** Note any color formation.
- **5.3.9.1.2.4** The formation of a blue color is a positive indication for the presence of an oxidizing ion.

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5.3.9.2 FT-IR

- 5.3.9.2.1 Analyze the particulate by following the Trace Evidence Section <u>Technical Procedure for Infrared Spectroscopy.</u>
- **5.3.10** Remove the cotton portion of the swabs and place in the corresponding polypropylene test tubes labeled in **5.3.7**.

5.4 Preparation of Analytical Standards

- **5.4.1** Label seven polypropylene test tubes:
 - Blank
 - Standard 1
 - Standard 2
 - Standard 3
 - Standard 4
 - Standard 5
 - Standard 6
- **5.4.2** Label five polypropylene test tubes QC2.
- **5.4.3** Cut two new cotton swabs into each polypropylene test tube from **5.4.1** and **5.4.2**.
- **5.4.4** Spike each labeled polypropylene test tube with the corresponding amounts:
 - Blank 100 µL of 5 % nitric acid
 - Standard 1 100 µL of 0.5/2.5/2.5 ppm Sb/Ba/Pb
 - Standard 2 100 μL of 1/5/5 ppm Sb/Ba/Pb
 - Standard 3 100 μL of 2/10/10 ppm Sb/Ba/Pb
 - Standard 4 100 μL of 4/20/20 ppm Sb/Ba/Pb
 - Standard 5 100 μL of 8/40/40 ppm Sb/Ba/Pb
 - Standard 6 100 μL of 16/80/80 ppm Sb/Ba/Pb
 - QC2 (five polypropylene test tubes) 100 μL of 2/10/10 ppm Sb/Ba/Pb

5.5 Digestion of Cotton Swabs from GSR Hand Kits

5.5.1 The temperature readings for ovens used for gunshot residue examination shall be monitored once a month. The temperature reading shall also be monitored once every 12 months with a NIST traceable thermometer. The acceptable temperature range for an oven shall be 75 °C to 85 °C. If the temperature is outside of the range, the oven

temperature shall be adjusted and allowed to equilibrate until a temperature within this range is acquired.

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- **5.5.2** Place labeled polypropylene test tubes containing the removed cotton from **5.3.10** in an oven set at approximately 80 °C for a minimum of 8 hours.
- **5.5.3** Place labeled polypropylene test tubes containing analytical standards from **5.4.4** in an oven set at approximately 80 °C to dry for a minimum of 8 hours.
- **5.5.4** Remove polypropylene test tubes from the oven.
- 5.5.5 Add 2 mL of internal standard to polypropylene test tubes to bring the concentration of the standards to the following:
 - Blank 0/0/0 ppm Sb/Ba/Pb
 - Standard 1 0.025/0.125/0.125 ppm Sb/Ba/Pb
 - Standard 2 0.05/0.25/0.25 ppm Sb/Ba/Pb
 - Standard 3 0.1/0.50/0.50 ppm Sb/Ba/Pb
 - Standard 4 0.2/1/1 ppm Sb/Ba/Pb
 - Standard 5 0.4/2/2 ppm Sb/Ba/Pb
 - Standard 6 0.8/4/4 ppm Sb/Ba/Pb
 - QC2 0.1/0.5/0.5 ppm Sb/Ba/Pb
- **5.5.6** Place polypropylene test tubes in an oven set at approximately 80 °C for a minimum of two hours.
- **5.5.7** Remove the polypropylene test tubes from the oven and allow them to cool to room temperature. Vortex the test tubes until the cotton swabs break away from applicator stick.
- **5.5.8** Centrifuge the polypropylene test tubes for approximately two minutes.
- **5.5.9** While the polypropylene test tubes are centrifuging, label a new 15 mL conical tube for each of the polypropylene test tubes, except for the five QC2 polypropylene test tubes.
- **5.5.10** Remove the polypropylene test tubes from the centrifuge and prepare a 1:10 dilution for each 15 mL conical tube using HPLC grade water. A minimum of 5 mL is needed.
- **5.5.11** Combine the five QC2 polypropylene test tubes into a new 50 mL centrifuge tube and prepare a 1:10 dilution using HPLC grade water. Prepare a 50 mL total solution.
- **5.5.12** The 1:10 dilution will bring the final concentration of the standards to the following:
 - Blank 0/0/0 ppm Sb/Ba/Pb
 - Standard 1 0.0025/0.0125/0.0125 ppm Sb/Ba/Pb
 - Standard 2 0.005/0.025/0.025 ppm Sb/Ba/Pb
 - Standard 3 0.01/0.050/0.050 ppm Sb/Ba/Pb
 - Standard 4 0.02/0.1/0.1 ppm Sb/Ba/Pb
 - Standard 5 0.04/0.2/0.2 ppm Sb/Ba/Pb

- Standard 6 0.08/0.4/0.4 ppm Sb/Ba/Pb
- QC2 0.01/0.05/0.05 ppm Sb/Ba/Pb
- **5.5.13** Recap all tubes and shake vigorously.
- **5.5.14** Remove caps and analyze tubes on an ICP/MS using the Trace Evidence Section Technical Procedure for Inductively Coupled Plasma Mass Spectrometry.
- 5.5.15 If significant levels of Sb/Ba/Pb are present after ICP/MS analysis the corresponding adhesive lifts from the GSR Hand Kit shall be analyzed using the Trace Evidence Section Technical Procedure for SEM/EDX for GSR Casework.
 - **5.5.15.1** If the GSR kit is from the hands of a reported suicide victim, report findings.

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- **5.5.15.2** Significant levels, based on the Forensic Scientist's training and experience, of Sb/Ba/Pb correspond to the following concentration level guidelines:
 - Sb \geq 0.03 ppm
 - Ba \geq 0.2 ppm
 - Pb \geq 0.3 ppm
- **5.5.16** SEM/EDX is used to determine if particles characteristic to gunshot residue are present on the corresponding adhesive lifts.
 - **5.5.16.1** Characteristic GSR particle(s) are spheroidal particle(s) that contain Sb/Ba/Pb.
 - **5.5.16.2** Characteristic GSR particles may also contain the following elements: Si, Ca, Al, Cu, Fe, S, P, Zn, Ni, K, Cl, and Sn.
- **5.5.17** Report findings.

5.6 Analytical Approach for the Collection on GSR on Clothing

- **5.6.1** See **5.11** for a list of sampling limitations regarding the collection and analysis of GSR on clothing.
- **5.6.2** Assign a unique sequential number to each item of clothing upon receipt from the evidence technician.
- **5.6.3** Clean examination surface with bleach solution.
- **5.6.4** Place a new clean piece of paper over the examination surface.
- 5.6.5 Open packaging containing clothing. Label each piece of clothing with the Laboratory case number, unique sequential number, Laboratory item number, Forensic Scientist's initials and the date.
- **5.6.6** List and describe the item of evidence.

5.6.7 Examine the item of evidence for smokeless powder visually or with the aid of an illuminated magnifier, UV light or stereomicroscope.

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- **5.6.8** Sample the item of clothing using blank adhesive lift(s).
- The adhesive lift(s) shall be analyzed using the Trace Evidence Section <u>Technical</u> Procedure for SEM/EDX for GSR Casework.
- **5.6.10** Report findings.

5.7 Analytical approach for adhesive lifts collected from surfaces other than hands.

- **5.7.1** Assign a unique sequential number to each adhesive lift kit upon receipt from the evidence technician.
- 5.7.2 Open kit. Label each piece of evidence within the kit with the unique sequential number, the date, and the Forensic Scientist's initials.
- 5.7.3 If a GSR Analysis Information Form is present in the kit, label the top of the form with the Laboratory case number, unique sequential number, Laboratory item number, Forensic Scientist's initials and the date.
- **5.7.4** Scan the GSR Analysis Information Form into Forensic Advantage (FA). If the form is not present in the kit, document this in the FA GSR case record worksheet.
- 5.7.5 The adhesive lift(s) shall be analyzed until characteristic particles of gunshot residue are found or completion, using the Trace Evidence Section <u>Technical Procedure for SEM/EDX for GSR Casework</u>.
- **5.7.6** Report findings.

5.8 Guidelines for Gunshot Residue Results Statements

- **5.8.1** All results shall be based on the Forensic Scientist's knowledge and experience and the case being examined. The Forensic Scientist has the responsibility to conduct analyses deemed necessary and appropriate and report the results based on his/her findings and experience.
- **5.8.2** The following is a list of guidelines for reporting results of gunshot residue analysis:

5.8.3 Report #1

- **5.8.3.1** Barium, antimony and lead, indicative of gunshot residue, were not present in significant concentrations on the handwipings submitted. However, this does not eliminate the possibility that the subject could have fired a gun.
 - **5.8.3.1.1** This statement should be used when the concentrations of barium, antimony, and lead do not meet the gunshot residue threshold levels as set out in **5.5.14.2**.

5.8.4 Report #2

- **5.8.4.1** Barium, antimony and lead, indicative of gunshot residue, were not present in proper concentrations or distribution on the handwipings submitted to allow the Forensic Scientist to give an opinion with regard to whether the subject could have fired a gun.
 - **5.8.4.1.1** This statement may be used when one or more elements of barium, antimony, and lead do not meet the gunshot residue threshold levels or are not in the proper distribution.

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5.8.5 Report #2A

- **5.8.5.1** Barium, antimony, and lead, elements associated with gunshot residue were present in elevated levels on the handwipings. However, due to the distribution of these elements on the handwipings, the results are inconclusive as to whether the individual could have fired a gun.
 - **5.8.5.1.1** This statement may be used when the concentrations of barium, antimony, and lead meet the threshold levels but one or more of the elements are not in the proper distribution.

5.8.6 Report #3A

- **5.8.6.1** Barium, antimony and lead, indicative of gunshot residue, were present in significant concentrations and distribution on the handwipings submitted. These results show that the subject could have fired a gun or been in close proximity to a firearm when it was discharged.
 - **5.8.6.1.1** This statement may be used when the concentrations of the elements barium, antimony, and lead meet the gunshot residue levels, and all three are in the proper distribution in the handwipings of a subject who is the victim of a gunshot wound and/or is suspected of discharging a firearm.

5.8.7 Report #4

- **5.8.7.1** Examination of the gunshot residue kit failed to identify particles characteristic of gunshot residue. These results are inconclusive as to whether the subject could have fired a gun.
 - **5.8.7.1.1** This statement shall be used when the examination of adhesive lifts collected from the hand(s) fails to identify particles characteristic of gunshot residue.

5.8.8 Report #4 Item(s)

5.8.8.1 Examination of the adhesive lifts collected from Item(s) # _____ failed to identify particles characteristic of gunshot residue.

5.8.8.1.1 This statement shall be used when the examination of adhesive lifts collected from a surface (other than hands) fails to identify particles characteristic of gunshot residue.

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5.8.9 Report #5

- **5.8.9.1** Examination of the gunshot residue kit revealed the presence of a particle characteristic to gunshot residue. A characteristic particle could have originated from the discharge of a firearm, the handling of a discharged firearm, being in close proximity to a firearm when it was discharged or from some other source which produces similar particles.
 - **5.8.9.1.1** This statement shall be used when the examination of adhesive lifts collected from the hand(s) identifies **ONE** particle characteristic of gunshot residue.

5.8.10 Report #5A

- **5.8.10.1** Examination of the gunshot residue kit revealed the presence of particles characteristic to gunshot residue. Characteristic particles could have originated from the discharge of a firearm, the handling of a discharged firearm, being in close proximity to a firearm when it was discharged or from some other source which produces similar particles.
 - **5.8.10.1.1** This statement shall be used when the examination of adhesive lifts collected from the hand(s) identifies **particles** characteristic of gunshot residue.

5.8.11 Report #5 Item(s)

- **5.8.11.1** Examination of the adhesive lift(s) collected from item(s) # _____ revealed the presence of a particle characteristic to gunshot residue. A characteristic particle on an item could have originated from being in close proximity to a firearm when it was discharged, being in contact with another item with gunshot residue on it or from some other source which produces similar particles.
 - **5.8.11.1.1** This statement shall be used when the examination of adhesive lifts collected from a surface (other than hands) identifies **ONE** particle characteristic of gunshot residue.

5.8.12 Report #5A Item(s)

5.8.12.1 Examination of the adhesive lifts collected from item(s) # _____ revealed the presence of particles characteristic to gunshot residue. Characteristic particles on an item could have originated from being in close proximity to a firearm when it was discharged, being in contact with another item with gunshot residue on it or from some other source which produces similar particles.

5.8.12.1.1 This statement shall be used when the examination of adhesive lifts collected from a surface (other than hands) identifies **particles** characteristic of gunshot residue.

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5.8.13 Report #7

- **5.8.13.1** Examination of the evidence revealed that wooden handled swabs had been used to collect the handwipings. Since these swabs contain barium, which is one of the elements used to determine the presence of gunshot residue on the subject's hands, it is not possible for this Laboratory to give an opinion with regard to whether a subject has fired a gun. Therefore, the evidence (handwipings) was not examined.
 - **5.8.13.1.1** This statement shall be used when it is noted that the handwipings procedure has been conducted using wooden swabs.

5.8.14 Report #8

- **5.8.14.1** Examination of the evidence data sheet revealed that the subject had washed his/her hands prior to the gunshot residue kit being administered. Washing one's hands successfully removes gunshot residue. Therefore, the evidence (handwipings) was not examined.
 - **5.8.14.1.1** This statement shall be used when it is noted that the subject had washed his/her hands after the discharge of the firearm and prior to the collection of the gunshot residue kit.

5.8.15 Report #9

- 5.8.15.1 Examination of the evidence data sheet revealed that a time greater than (4) hours had elapsed from the time the weapon was discharged to the time the handwipings were collected. The concentration of gunshot residue significantly declines after approximately (4) hours. Therefore, the evidence (handwipings) was not examined.
 - 5.8.15.1.1 This statement shall be used when it is noted that a time greater than four hours had elapsed between discharge of the firearm and collection of the gunshot residue kit. *This applies only to live subjects.*

5.8.16 Report #10

5.8.16.1 Examination of the evidence revealed that the instruction sheet was not followed for the proper collection of handwipings. In such cases it is not possible for this laboratory to give an opinion with regard to whether a subject has fired a gun. Therefore, the evidence (handwipings) was not examined.

5.8.16.1.1 This statement shall be used when it is noted that the proper procedures were not used in the collection of the gunshot residue kit.

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5.8.17 Report #10A

- **5.8.17.1** This item was not examined due to an improper collection technique. Proper collection for characteristic gunshot residue particles from inanimate objects requires the use of adhesive lifts.
 - **5.8.17.1.1** This statement shall be used when improper media are used in the collection of potential gunshot residue particles. Adhesive lifts (i.e., Carbon backed adhesive SEM lifts) are the only acceptable media for analysis in this Laboratory.

5.8.18 Report #11

- **5.8.18.1** Examination of item(s) # _____ revealed the presence of characteristic particles of gunshot residue; therefore, this item was not analyzed.
 - **5.8.18.1.1** This statement shall be used when characteristic particles of gunshot residue are identified on an item collected from the same subject.

5.8.19 Report #12

- **5.8.19.1** Examination of the evidence revealed that the control swabs were contaminated. In such cases it is not possible for this laboratory to give an opinion with regard to whether a subject has fired a gun or has been in close proximity to a firearm when it was discharged.
 - **5.8.19.1.1** This statement shall be used when the control swabs are contaminated visibly with a foreign object not considered part of the original kit.

5.8.20 Report #12A

- **5.8.20.1** Examination of the evidence revealed that the control swabs contain (barium/antimony/lead). Since these swabs contain (barium/antimony/lead), an element used to determine the presence of gunshot residue on a subject's hands, it is not possible for this laboratory to give an opinion with regard to whether a subject has fired a gun or has been in close proximity to a firearm when it was discharged.
 - **5.8.20.1.1** This statement shall be used when the control swabs in a suicide victim kit contain one or more of the elements barium, antimony, and lead. This statement shall also be used for kits that do not contain adhesive lifts.

5.8.21 Report #13

- **5.8.21.1** This item is being returned unworked. If you have any questions, please contact the Forensic Scientist who issued this report.
 - **5.8.21.1.1** This statement shall be used when an item of evidence is being returned unworked.

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5.8.22 Report #13A

- **5.8.22.1** This item is being returned unworked. Clothing from a subject that is a victim of a gunshot wound is not examined in the Trace Evidence Section for characteristic particles of gunshot residue.
 - **5.8.22.1.1** This statement shall be used when an item of evidence is being returned unworked specifically because it is the victim's clothing.
- **5.9 Calibrations** No additional calibrations or performance checks are required. See associated technical procedures for instrumental performance checks.
- **5.10 Maintenance** No additional maintenance is required. See associated technical procedures for instrumental maintenance.

5.11 Sampling and Sample Selection

- **5.11.1** No sampling is performed. When sample selection occurs, it shall be based on the Forensic Scientist's training and experience.
- **5.11.2** Items submitted for examination shall be analyzed based on the following criteria:
 - **5.11.2.1** When an agency requests gunshot residue examination for both a gunshot residue collection kit and clothing from the same subject, the gunshot residue collection kit shall be examined first.
 - 5.11.2.1.1 If the gunshot residue collection kit reveals particles characteristic of gunshot residue, the clothing shall not be examined.
 - 5.11.2.1.2 If the gunshot residue collection kit fails to reveal the presence of particles characteristic of gunshot residue, the clothing shall be examined, unless the subject is the victim of a gunshot wound.
 - **5.11.2.2** Gunshot residue kits and clothing from a subject that is the victim of a gunshot wound shall not be examined in the Trace Evidence Section for characteristic particles of gunshot residue.
 - **5.11.2.3** Inanimate objects other than clothing shall only be analyzed based on the forensic scientist's training and experience. Examples of items that will not be analyzed for gunshot residue include, but are not limited to, firearms and inanimate objects with known bullet holes.

5.12 Calculations – N/A

5.13 Uncertainty of Measurement – N/A

6.0 Limitations

6.1 The presence of characteristic particles of gunshot residue on hands or clothing does not conclude that the subject fired a gun. Characteristic particles could have originated from the discharge of a firearm, the handling of a discharged firearm, being in close proximity to a firearm when it was discharged or from some other source which produces similar particles.

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- 6.2 The absence of characteristic particles of gunshot residue on hands, clothing, or inanimate object does not conclude that a subject **did not** fire a gun. These results are inconclusive.
- 6.3 Items of clothing packaged together shall be treated as one item. GSR particles can be transferred from item to item if packaged together.

7.0 Safety

- **7.1** Concentrated acids and known standards must be used in a well ventilated area with proper precautions being exercised to minimize skin contact.
- 7.2 Items of clothing may have blood or other body fluids present. Use proper protective equipment when dealing with items that may contain biohazard material.
- 7.3 X-Rays from the EDX detector of the scanning electron microscopes are considered to be a minimal health risk. Badges are located near the systems to monitor radiation levels.

8.0 References

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9.0 Records

Oven Log

10.0 Attachments

- Appendix I: Reagent Preparation Instructions
- Appendix II: General Flow Diagram for Gunshot Residue Collection Kit Analysis

Revision History		
Effective Date	Version Number	Reason
09/17/2012	1	Original ISO Document
03/08/2013	2	5.8.11.1 and 5.8.12.1: Added opinion statement to result 5.8.17: Added Report #10A which resulted in format renumbering of remaining document 5.8.19.1: Added close proximity to Report #12 5.8.21: Added Report #13A 5.11.2: Revised examination criteria 8.0: Added Wright and SWGGSR references
09/30/2013	3	5.5.1: Added use of NIST traceable thermometers to 4.1 and traceable thermometer to Appendix 1: Corrected element listed in Internal Solution within QC1 Standard Preparation from "Bi" to "Lu" 5.10: Maintenance Section was not formatted in version 2. Added proper Formatting number (5.10) which shifted all numbers after 5.10 5.8.19.1 Reworded Result #12 5.8.20.1 Added Result #12A

APPENDIX I: REAGENT PREPARATION INSTRUCTIONS

Notes:

- 1) All standards must be prepared in plastic ware, preferably PFA, PMP, or a Teflon plastic.
- 2) All prepared standard listed below will expire one year from preparation date.

10% Nitric Acid

Add 100 mL concentrated nitric acid to 900 mL HPLC Grade water into a 1000 mL plastic graduated cylinder.

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5% Nitric Acid

Add 50 mL concentrated nitric acid to 950 mL HPLC Grade water into a 1000 mL plastic graduated cylinder.

1% Nitric Acid

Add 10 mL concentrated nitric acid to 990 mL HPLC Grade water into a 1000 mL plastic graduated cylinder.

Internal Standard (0.2 ug/mL In/Lu in 10% Nitric Acid)

Combine 0.2 mL of 1000 ppm Indium, 0.2 mL of 1000 ppm Lutetium, and 100 mL nitric acid in a 1 L plastic volumetric flask. Adjust volume to 1 L with HPLC Grade water.

GSR Standard 6 (16/80/80 ppm Sb / Ba / Pb in 5% Nitric Acid)

Combine 1.6 mL of 1000 ppm antimony (Sb), 8 mL of 1000 ppm barium (Ba), 8 mL of 1000 ppm lead (Pb), and 5 mL nitric acid in a 100 mL plastic volumetric flask. Adjust volume to 100 mL with HPLC Grade water.

GSR Standard 5 (8/40/40 ppm Sb / Ba / Pb in 5% Nitric Acid)

In a 100 mL plastic volumetric flask, aliquot 50 mL of GSR Standard 6 and fill to volume with 50 mL 5% Nitric Acid.

GSR Standard 4 (4/20/20 ppm Sb / Ba / Pb in 5% Nitric Acid)

In a 100 mL plastic volumetric flask, aliquot 50 mL of GSR Standard 5 and fill to volume with 50 mL 5% Nitric Acid.

GSR Standard 3 (2/10/10 ppm Sb / Ba / Pb in 5% Nitric Acid)

In a 100 mL plastic volumetric flask, aliquot 50 mL of GSR Standard 4 and fill to volume with 50 mL 5% Nitric Acid.

GSR Standard 2 (1/5/5 ppm Sb / Ba / Pb in 5% Nitric Acid)

In a 100 mL plastic volumetric flask, aliquot 50 mL of GSR Standard 3 and fill to volume with 50 mL 5% Nitric Acid.

GSR Standard 1 (0.5/2.5/2.5 ppm Sb / Ba / Pb in 5% Nitric Acid)

In a 100 mL plastic volumetric flask, aliquot 50 mL of GSR Standard 2 and fill to volume with 50 mL 5% Nitric Acid.

QC 1 (0.02 / 0.02 ppm In / Lu in 1% Nitric Acid)

Prepare a 1:10 dilution of the Internal Standard (0.2 ug/mL In/Lu in 10% Nitric Acid) by taking 100 mL of Internal Standard into a 1 L plastic volumetric flask. Adjust volume to 1 L with HPLC Grade water.

QC 2 Stock Solution (20 / 100 / 100 ppm Sb / Ba / Pb in 5% Nitric Acid)

Add 1 mL of 1000ppm Sb, 5 mL of 1000ppm Ba, and 5 mL of 1000ppm Pb to a 50 mL falcon tube. Add 39 mL 5% Nitric Acid.

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QC 2 Working Solution (2/10/10 ppm Sb/Ba/Pb in 5% Nitric Acid)

Prepare a 1:10 dilution of the QC 2 Stock Solution by taking 5 mL of QC2 Stock Solution and adding 45 mL 5% Nitric Acid in a 50 mL falcon tube.

Diphenylamine Solution

Add 1 g diphenylamine, 40 mL water, and 200 mL concentrated sulfuric acid in a 500 mL flask. The solution is then QC checked with a known nitrate (i.e., KNO₃). If the solution turns blue in the presence of the known nitrate, this is considered a positive QC check.

APPENDIX II: GENERAL FLOW DIAGRAM FOR GUNSHOT RESIDUE COLLECTION KIT ANALYSIS

Version 3

