Technical Procedure for Scanning Electron Microscope/ Energy Dispersive X-Ray System (SEM/EDX) for GSR Casework

1.0 Purpose – This technical procedure shall be followed for the operation of the Scanning Electron Microscope/Energy Dispersive X-Ray System (SEM/EDX). This procedure shall be used for GSR casework.

2.0 Scope – This procedure applies to the EVO MA 15/Oxford and the LEO/Oxford Systems. These instruments are used for high resolution and magnification imaging with enhanced depth of field for trace evidence and non-destructive elemental analysis of gunshot residue particles, paint, metals, powders, and other trace particulate material.

3.0 Definitions – N/A

4.0 Equipment, Materials, and Reagents

4.1 Equipment

- EVO MA 15 Scanning Electron Microscope
- LEO 1450 Variable Pressure Scanning Electron Microscope
- Oxford Energy Dispersive X-ray System (SDD Detector)

4.2 Materials

- Mounting tweezers for SEM lifts
- Kimwipes
- Nitrile gloves
- Adhesive lifts with carbon-backed tape for blank standard
- Stainless Steel 316 Standard
- PLANO GSR Standard
- Manganese (Mn) / Rhodium (Rh) Standard
- Cobalt (Co) / Rhodium (Rh) Standard
- Manganese Standard
- Nitrogen gas, compressed (Purity Grade 5.0)

4.3 Reagents – N/A

5.0 Procedure

5.1 EVO/Oxford or LEO/Oxford SEM/EDX

5.1.1 SEM Start-Up Procedure & Loading of Samples

5.1.1.1 Press green “ON” button on microscope and turn on computer.

5.1.1.2 Load the SmartSEM software.

5.1.1.3 Open RemCon32 software.

5.1.1.4 Verify stage is initialized. If not initialized, remove sample holder and select Stage Initialize.

5.1.1.5 Using mounting tweezers, place samples in the holder and note position of each sample. Tighten the screws on the holder for each mounting position. Place the holder back on the stage, ensuring that the flat edge of the mount is against the flat area of the stage.

5.1.1.6 Close the SEM sample chamber and select "pump." Allow system to reach vacuum.

5.1.1.7 Turn on the Extra High Tension (EHT) and Filament.

5.1.1.8 Adjust saturation of the filament slightly below or at the second crossover.

5.1.1.9 Adjust the working parameters of the instrument as necessary by accessing Tools through the User Toolbar and/or use of control panel with rotary controls.

5.1.2 Setting up a GSR Analysis in Oxford

5.1.2.1 Load INCA Software. Select GSR (LEO) or Feature (EVO) Tab.

5.1.2.2 Identify the Project name and sample information for each adhesive lift in the holder.

5.1.2.3 Go to the Recipe tab. Create a database and select "NC Crime Lab GSR" recipe. ***NOTE* Do not "Lock" or "Embed database within the project file."**

5.1.2.4 Go to the Microscope tab. Set the stage point to the Mn or Co standard. Increase the Mag to 766 and deadtime to approximately 40%.

5.1.2.5 Go to the Quant Optimization tab. Choose Mn or Co standard and acquire a spectrum.

5.1.2.6 Go to the Area Layout, select areas tab, and highlight the adhesive lift associated with each sample.

5.1.2.7 Go to the GSR or Feature Detection tab. Select Calibration and Mn/Rh standard or Co/Rh standard. Acquire a spectrum and optimize the working parameters to between 145 and 255.

5.1.2.8 Run automated analysis.

5.1.3 Particle Relocation & Identification

5.1.3.1 Review results of automated analysis. Assess instrumentally identified particles and determine whether there are Characteristic Gunshot Residue Particles. Use this information to formulate conclusion on sample. Characteristic Gunshot Residue Particle(s) are spheroidal particle(s) that contain antimony, barium, and lead. Characteristic Gunshot Residue Particles may also contain the following elements: silicon, calcium, aluminum, copper, iron, sulfur, phosphorus, zinc, nickel, potassium, chlorine, and tin. Note additional particles present in the population, including particles that contain antimony, barium, and/or lead.

5.1.3.2 Relocate and create documentation of image and spectra of Characteristic Gunshot Residue Particle(s).

5.1.3.3 When analysis and relocation are complete, remove the samples.

5.1.4 Performance Check (Resolution vs. Process Time) – Performed monthly if in use.

5.1.4.1 Open INCA software and select Analyzer from drop down.

5.1.4.2 Move stage to Manganese standard. Select "Quant Optimization" and acquire a Spectrum of Manganese.

5.1.4.3 Select "Quant." Under the Spectrum details tab note the detector resolution.

5.1.4.4 Pass Criteria: The extrapolated strobe resolution at process time 6 shall be no greater than the detector resolution of 127 eV. If resolution requirement is not met, maintenance shall be performed or a service engineer called. Once maintenance is performed and this criterion is met, the instrument may be returned to service.

5.1.4.5 When test is complete, use the Snipping Tool to save resolution result.

5.1.4.6 Load and analyze the Stainless steel 316 Standard. Save report. Pass Criteria - The following peaks should be present: Fe, Ni, Si, Mo, Cr, and Mn. If these peaks are not present, maintenance shall be performed or a service engineer called. Once maintenance is performed and this criterion is met, the instrument may be returned to service.

5.1.4.7 Fill out SEM-EDX Performance Check Log and save reports in the Monthly Check folder on the D-drive.

5.1.5 Shut Down Procedure

5.1.5.1 Turn off the filament & and select high vacuum mode.

5.1.5.2 Close the SEM user software. Close the EMServer. Close the RemCon32 software. Shut down the SEM computer.

5.1.5.3 Press the yellow "STANDBY" button on the microscope.

5.1.5.4 Close all windows in INCA software and shut down Oxford computer.

5.1.6 Performance Verification for New Instrument Set-Up

5.1.6.1 A new SEM with EDX detector shall be installed by a certified engineer according to the manufacturer's guidelines.

5.1.6.2 Spectra shall be obtained from a Manganese/Rhodium Standard and/or Cobalt/Rhodium Standard, and a Stainless Steel 316 Standard.

5.1.6.3 An analysis shall be performed on a PLANO GSR Standard and then compared to the known amount of GSR particle's on that standard.

5.1.7 Standards & Controls - This instrument requires the use of a Manganese/Rhodium standard or a Cobalt/Rhodium standard with a Manganese standard for performance checks. In addition a Stainless Steel 316 Standard shall be used for performance checks and verifications. The PLANO GSR standard is used for performance verifications. These standards have no special storage requirements.

5.1.8 Maintenance - Routine maintenance shall be performed such as changing pump oil, checking liquid chiller status (LEO), and replacing a filament. Any maintenance performed shall be documented in the maintenance log for that particular instrument.

5.2 Sampling and Sample Selection – No sampling is performed. When sample selection occurs, it shall be based on the Forensic Scientist's training and experience

5.3 Calculations - N/A

5.4 Uncertainty of measurement - N/A

6.0 Limitations – N/A

7.0 Safety

7.1 The greatest safety concern is radiation from the X-ray tube. The x-ray system is monitored for leaks on a regular basis.

7.2 There is a high voltage/current safety concern which can cause electrocution. Avoid contact with any live circuitry components. Potentially lethal voltages exist with the high voltage x-ray supply.

8.0 References

ASTM Standard E 1588 – 95 (2001), "Standard Guide for Gunshot Residue Analysis by Scanning Electron Microscopy/ Energy—Dispersive Spectroscopy." ASTM International, West Conshohocken, PA, 2001, www.astm.org.

Andrasko, J. "Detection of Gunshot Residue on Hands by Scanning Electron Microscopy." *Journal of Forensic Sciences* 22.2 (1977): 279-287.

DeGaetano, Douglas, et al. "A Comparison of Three Techniques Developed for Sampling and Analysis of Gunshot Residue by Scanning Electron Microscopy/Energy Dispersive X-Ray Analysis (SEM/EDX)." *Journal of Forensic Sciences* 37.1 (1992): 281-300.

Nesbitt, R.S., et al. "Detection of Gunshot Residue by Use of the Scanning Electron Microscope." *Journal of Forensic Sciences* 21.3 (1976): 595-610.

Wolten, G.M., et al. "Final Report on Particle Analysis for Gunshot Residue Detection." The Aerospace Corporation, ATR-77 (7915)-3, 1977.

9.0 Records

- ASPEX SEM/EDX Performance Check Log
- Maintenance Log
- Results For Instrumental Analysis Of Evidence For Gunshot Residue

10.0 Attachments

- APPENDIX 1: Guidelines for GSR Acquisition parameters.

Revision History		
Effective Date	Version Number	Reason
09/17/2012	1	Original ISO Document
02/01/2013	2	Request for Instrumental Analysis of Evidence for Gunshot Residue was added as a record. Revised title for Attachment 1 and measure dwell time.
09/30/2013	3	5.1.4 - Removed from performance check that it shall be performed by first person to use the instrument
10/18/2013	4	Added issuing authority to header
09/05/2014	5	Updated header to Physical Evidence Section – Trace Unit, issuing authority to Physical Evidence Section Forensic Scientist Manager. 5.1.1.2, 5.1.1.3, 5.1.2.3, 5.1.2.5, 5.1.2.7, 5.1.2.8, 5.1.2.10, 5.1.3, 5.1.3.1, 5.1.3.2, and 5.1.3.5 were updated to clarify the instructions for use. Added Appendix 1. Removed: 5.1.1.1, 5.1.1.4, 5.1.1.5, 5.1.1.6, 5.1.3.2, 5.1.3.4. Moved instructions to SEM Training.
07/01/2016	6	Added Instrumentation for GSR Analysis (Section 5.2). GSR samples can now be examined on the LEO/Oxford SEM/EDX.
07/27/2018	7	Updated 2.0, 4.1, 4.2, 5.1, to reflect new equipment and materials. The following were updated to clarify instructions for use on current instrumentation and software: 5.1.1.1, 5.1.1.3, 5.1.1.4, 5.1.1.6, 5.1.1.9, 5.1.2.1, 5.1.2.4, 5.1.2.5, 5.1.2.7, 5.1.4.1, 5.1.4.2, 5.1.4.3, 5.1.4.4, 5.1.4.5, 5.1.4.6, 5.1.4.7, 5.1.5.1, 5.1.5.2, 5.1.5.3, 5.1.5.4, 5.1.6.2, 5.1.6.3, 5.1.7, 5.1.8. Edited Appendix 1 to show parameters as they appear in current software. Removed information regarding ASPEX instrument that is no longer in service: 2.0, 4.1, 4.2, and 5.1 ASPEX, 5.1.6.2, 10.0. Deleted extra spacing: 5.1.2.6, 5.1.3.3, and between 5.1.6 and 5.1.7. Misspelling 5.1.1.8

APPENDIX 1: Guidelines for GSR Acquisition parameters

The default parameters for GSR are saved as recipe "NC Crime Lab GSR" file in INCA software.