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

2.0 Scope – This procedure applies to the EVO MA 15/Oxford and the Vega4/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 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
- VEGA-4 Scanning Electron Microscope
- Oxford Energy Dispersive X-ray System (SDD Detector)

4.2 Materials

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

4.3 Reagents

- Methanol

5.0 Procedure

5.1 SEM Start-Up Procedure & Loading of Samples

5.1.1 Turn on microscope and then turn on SEM computer.

5.1.2 Load the SEM software.

5.1.3 For the EVO MA 15:

5.1.3.1 Open RemCon32 Software.

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

5.1.4 Vent chamber. Using mounting tweezers, place samples in the holder and note position of each sample. Tighten the screws on the holder for each mount position. Ensure sample tray is securely mounted.

5.1.5 Close the SEM sample chamber and allow system to reach vacuum by starting the pump in the software.

5.1.6 Turn on the electron beam.

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

5.1.8 Adjust the working parameters of the instrument as necessary.

5.2 Collection and Storage of Data (Non GSR) on SEM

5.2.1 Set the parameters of the SEM instrument (e.g., different accelerating voltage, current setting, signal collector, or display variable), based on training and experience, to enhance the image on the display monitor.

5.2.2 When comparing known and questioned samples, begin with the questioned items unless case constraints dictate otherwise.

5.2.3 Perform sizing of particles if needed.

5.2.4 Label pertinent information on the monitor using Annotation drop down menu.

5.2.5 Print image and import into Forensic Advantage (FA) Case Record.

5.3 Collection and Storage of Data (Non GSR) on OXFORD EDS

5.3.1 Use Acquisition Rate 3 for most analysis. If performing quantification, a higher process time of 4/5/6 may be needed, depending on sample type.

5.3.2 Analysis time will vary, with 100 seconds being an average time.

5.3.3 Click on "Point and ID" icon. To set up analysis, follow the sequential steps, starting with "PROJECT." Save the project with a unique name before starting analysis.

5.3.4 When comparing known and questioned samples, begin with the questioned items unless case constraints dictate otherwise.

5.3.5 During and after collection different regions of the spectra may be expanded and spectral peaks may be identified either automatically or manually.

5.3.6 Create documentation of sample spectra to include in the FA case record.

5.3.7 When analysis is complete, vent the chamber and remove the sample(s) from the sample holder by loosening the screws on the holder and using mounting tweezers.

5.4 Performance Check (Quantitative) – Performed when needed

5.4.1 A quantitative performance check is required whenever quantitative results are needed.

5.4.2 Using either a Cobalt or Manganese Standard, in INCA, select the Quant Optimization icon.

5.4.3 Obtain an optimal image by adjusting the microscope settings.

5.4.4 Use a minimum Acquisition Rate of 4 for optimum resolution.

5.4.5 Select the element of choice. Once the element selection is made, this program runs automatically. The system will state the quantification was successful when finished.

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

5.5.1 Open INCA software and select Analyzer from drop down.

5.5.2 Move stage to Manganese standard. Select "Quant Optimization" and acquire a spectrum of Manganese.

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

5.5.4 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.

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

5.5.6 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.

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

5.6 Shut-Down Procedures

5.6.1 Turn off the filament and select high vacuum mode.

5.6.2 Close the SEM user software.

5.6.3 Shut down the SEM computer.

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

5.7 Performance Verification for New Instrument Set-Up

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

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

5.8 Standards and Controls – This instrument requires the use of Manganese/Rhodium standard or a Cobalt/Rhodium standard with a Manganese standard for performance checks. In addition, the Stainless Steel 316 Standard shall be used for performance verifications. These standards have no special storage requirements.

5.9 Instrument Maintenance – Routine maintenance shall be performed such as changing pump oil and replacing a filament. Any additional maintenance performed shall be documented in the maintenance logbook for that particular instrument.

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

5.11 Calculations - N/A

5.12 Uncertainty of measurement - N/A

6.0 Limitations – N/A

7.0 Safety Concerns

7.1 The greatest safety concern is stray x-ray radiation. The x-ray system is monitored for leaks on a regular basis.

7.2 There is high voltage/current 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

Carl Zeiss SMT Nano Technology Division. *SmartSEM Operating Software for Felid Emission Scanning Electron Microscopes Manual*. V05.04, Printed 2009.

Hearle, J.W., J.T. Sparrow and P.M. Cross. *The Use of the Scanning Electron Microscope.* Oxford: Pergamon Press Ltd, 1972.

Oxford Instruments Analytical. *INCA Energy Operator Manual Issue 1*. Printed 2004. (Located on Desktop of X-ray computer).

Oxford Instruments Analytical. *INCA Feature Instruction Manual.* (Located on Desktop of X-ray

computer), 2007.

9.0 Records

- Performance Check Log
- Maintenance Log
- Request for Instrumental Examination of Evidence

10.0 Attachments – N/A

Revision History		
Effective Date	Version Number	Reason
09/08/2023	7	Edited section 1.0 and 2.0 Removed LEO SEM from document Added Vega-4 SEM to document Removed reference from section 8.0 Edited section 9.0