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), including ASPEX and the LEO/Oxford systems. This procedure shall be used for non-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 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
- 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)

4.3 Reagents – N/A

5.0 Procedures

5.1 EVO/Oxford or LEO/Oxford SEM/EDX System

5.1.1 Microscope Startup

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 mount 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 Oxford EDX Start-Up

5.1.2.1 Load INCA software.

5.1.2.2 Select Detector Control. In Thermal Tab, the "Detector State" shall say "operate" and "COLD" and in the Slide tab the Slide Position shall say "in."

5.1.2.3 Select Microscope Control. Check the box for "HV."

5.1.3 Performance Check

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

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

5.1.3.1.2 Obtain an optimal image by adjusting the microscope settings.

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

5.1.3.1.4 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.

5.1.3.2 **Resolution vs. Process Time Check** – Performed monthly if in use.

5.1.3.2.1 Open INCA software and select Analyzer from drop down.

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

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

5.1.3.2.4 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.

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

5.1.3.2.6 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.

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

5.1.4 Performance Verification for New Instrument Set-Up

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

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

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

5.1.5.1 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.

5.1.5.2 Perform sizing of particles if needed.

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

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

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

5.1.6.1 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.

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

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

5.1.6.4 During and after collection you may expand different regions of the spectra and identify spectral peaks either automatically or manually.

5.1.6.5 Print spectra in .pdf format and save in FA Case Record.

5.1.7 Shut-Down Procedures

5.1.7.1 Turn off the filament and select high vacuum mode.

5.1.7.2 Close the SEM user software. Close the EMServer. Close the RemCon32 software.

5.1.7.3 Shut down the computer.

5.1.7.4 Once computer is shutdown, press the yellow "STANDBY" button on the front panel of the instrument to keep the electronics on, but in a standby mode. Pressing the red "OFF" button will remove all the voltage to the instrument and will lose stage settings. Therefore, never press the red "OFF" button except for major resetting of the instrument or for servicing of the system.

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

5.1.8 Standards and Controls – This instrument requires the use of Manganese/Rhodium standard or 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.

5.1.9 Instrument 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 logbook 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 Concerns

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 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

ASPEX, LLC. Personal SEM *Console User Manual: Perception Suite User Documentation.* Version 1.37, 2002.

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

- LEO-Oxford SEM/EDX Performance Check Log
- Maintenance Log
- ASPEX SEM/EDX Performance Check Log
- Request for Instrumental Examination of Evidence

10.0 Attachments – N/A

Revision History		
Effective Date	Version Number	Reason
09/17/2012	1	Original ISO Document
02/01/2013	2	Request for Instrumental Examination of Evidence was added as a record.
09/30/2013	3	5.1.3.2 - 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, 5.1.1.9, 5.1.1.11, 5.1.2.1, 5.1.2.4, 5.1.2.5, 5.1.3.1.1, 5.1.3.2.2, 6.1.3.2.4, 5.1.3.2.8, 5.1.6.3, 5.1.7.1, 5.2.1.1, and 5.2.1.6 were updated to clarify the instructions for use. Removed: 5.1.1.1, 5.1.1.4, 5.1.1.5, 5.1.1.7, 5.1.1.8, 5.1.2.2, 5.1.2.3, 5.1.3.2.6, 5.1.5.4, 5.1.6.5, 5.2.1.2, 5.2.1.3, 5.2.1.5, 5.2.2.2, 5.2.4.4, 5.2.4.7. Moved instructions to SEM Training.
07/27/2018	6	Updated 2.0, 4.1, 4.2, 5.1, and delete 5.2 to reflect current equipment and materials. Delete extra periods: 5.1.1.2, 5.1.1.7. Clarify instructions: 5.1.1.1, 5.1.1.4, 5.1.1.6, 5.1.1.9, 5.1.4.1, 5.1.3.1.1, 5.1.3.2.1, 5.1.3.2.2, 5.1.3.2.3, 5.1.3.2.4, 5.1.3.2.5, 5.1.3.2.6, 5.1.3.2.7, 5.1.3.2.8, 5.1.3.2.9, 5.1.4.2, 5.1.7.1, 5.1.8, 5.1.9.