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## Technical Procedure for Micro X-Ray Fluorescence ( $\mu$ -XRF) Spectrometry

- 1.0 Purpose** – This technical procedure shall be followed for the operation of the Micro X-ray Fluorescence spectrometer.
- 2.0 Scope** – This procedure applies to the Orbis PC Elemental Analyzer System with SDD (Silicon Drift Detector) located in the Trace Evidence Section. This can be used for non-destructive elemental analysis of trace evidence such as glass, paint, metals, and other materials.
- 3.0 Definitions** – N/A
- 4.0 Equipment, Materials, and Reagents**
- 4.1 Equipment**
- EDAX Orbis PC Elemental Analyzer System with SDD (Serial Number 1200)
  - Orbis Vision Software
- 4.2 Materials**
- Polyethylene X-ray cell (XRF sample cup)
  - Ultralene film
  - Glue stick
  - Double Coated Carbon Conductive Tape
- 4.3 Reagents**
- Acetone
  - Methanol
- 5.0 Procedure**
- 5.1 Sample Preparation**
- 5.1.1** The method of sample preparation may vary based on the size and type of sample being analyzed.
- 5.1.2** It is recommended that small samples, such as glass fragments, be secured in glue on Ultralene film or on carbon tape. The glue or tape may be adhered to an XRF sample cup to elevate the sample from the stage and prevent it from moving during analysis. Solvents such as acetone and methanol can be used to dissolve the glue for removal of the sample after analysis.
- 5.1.3** For glass samples, a dot from a black Sharpie marker may be added to the surface requiring analysis to aid in focusing the sample.

**5.1.4** Secure loose powders that require analysis under vacuum. The powder may be placed between two films of Ultralene in an XRF sample cup or firmly adhered to glue or tape.

**5.1.5** Analysis of liquids and/or corrosive materials is not recommended by the manufacturer.

## **5.2 Instrument Notes**

**5.2.1** Instrument and computer shall be turned off when not in use.

**5.2.2** When switching the chamber to vacuum, wait for the “Vacuum Level Warning” to disappear and the Chamber dropdown menu to change to green before making any changes to the software.

**5.2.3** Use caution when choosing ReLoad to place samples back into the chamber. This will automatically reset the stage to the previous X, Y and Z parameters. Should the stage crash safety feature fail, the samples could crash into the x-ray beam or cameras.

**5.2.4** The chamber door may be opened while the X-ray button above the door is illuminated so long as the Shutter button above the door is NOT illuminated.

## **5.3 Verification of New Instrument**

**5.3.1** Run glass certified reference material (ex. NIST SRM 1831) and a known elemental standard (ex. copper) and verify that the elemental composition of each is correctly identified.

## **5.4 Instrument Start-up**

**5.4.1** Press “POWER” button above chamber.

**5.4.2** Turn on the computer. Orbis Notifier will automatically start up after logging into Windows. This will check the status of the following: Communication, SafetyInterLock, HV Fuse, Power Fuse, Tube Temp, Door Switches, WatchDog, Xray, Shutter, and XrayHiVis. If the Notifier encounters a problem, it will display a message on the screen. Follow the directions displayed to resolve the problem such as rebooting the system. If this does not resolve the issue, contact the service representative. The Orbis Notifier must be running before starting Orbis Vision software.

**5.4.3** Open the sample chamber to remove any samples that are on the sample tray by moving handles to the vertical position and then swing chamber door to the left. Close the sample chamber.

**5.4.4** Press x-ray “on” button. This will take approximately 5 seconds to activate.

**5.4.5** Open Orbis Vision Software to initialize the instrument. Ensure that no samples are in the chamber and then click “OK” to recalibrate the stage. If instrument does not initialize, power off and power up again. If problem persists, contact the service representative.

## **5.5 Performance (Calibration and Resolution) Check**

- 5.5.1** A calibration and resolution check of the Micro XRF System using the Aluminum 2024 calibration disk shall be performed prior to the use of the instrument. A printed copy of each “Energy Adjustment Report” shall be saved in an electronic format.
- 5.5.1.1** Open the sample chamber and place the Aluminum 2024 standard on the stage. Close the sample chamber.
- 5.5.1.2** Select “vacuum” from the Chamber dropdown menu.
- 5.5.1.3** Adjust the stage to focus on the Aluminum 2024 standard. The Z position of the Aluminum 2024 standard is approximately 96.9.
- 5.5.1.4** Set the accelerating voltage to 50kV and adjust beam current to optimize detector dead time to between 30% and 50% (~100 $\mu$ A).
- 5.5.1.5** Under the Setup menu, select “Edam & Calibration” and choose “Auto All TC”. A “Congratulations” or “Fail” message notifies user if the calibration was successful. Select “PrintAll TC” to save the Energy Adjustment Report.
- 5.5.1.6** Inspect the Energy Adjustment Report.
- 5.5.1.6.1** A detector resolution no greater than 180eV must be achieved for all time constants.
- 5.5.1.6.2** Check X-ray source performance by noting the maximum counts per second. This shall be within +/- 20% of the reading on the previous Energy Adjustment Report.
- 5.5.1.7** Repeat the calibration and resolution check if the criterion are not met. If a problem persists, contact the service representative. Once maintenance has been performed, the calibration and resolution check must pass in order to continue use for casework.

## **5.6 Setting up Orbis Vision Software for Analysis**

- 5.6.1** Select “Unload” in Instrument Console and allow chamber to achieve atmospheric pressure. Open sample chamber by moving handles to the vertical position and then swing chamber door to the left.
- 5.6.2** Load samples on tray. Sample tray can be removed from chamber if needed to position samples.
- 5.6.3** Close chamber door and select chamber “vacuum”.
- 5.6.4** Adjust the stage to focus on the sample. The Z position for an XRF sample cup is approximately 75.

- 5.6.5** Select the appropriate run parameters for the type of analysis needed. The following parameters are recommended:
- 5.6.5.1** For all samples: Adjust probe current to optimize detector dead time. A dead time between 30% and 50% is recommended.
  - 5.6.5.2** For analysis of glass samples: Accelerating voltage= 50kV, amp time= 12.8 $\mu$ s, and an acquisition time of at least 1500 live seconds. In general, a probe current of approximately 150 $\mu$ A will optimize dead time. Compare samples of similar sizes. Multiple measurements should be made on glass fragments that do not have both original surfaces.
  - 5.6.5.3** For analysis of metal samples: Accelerating voltage= 50kV, amp time= 3.2 $\mu$ s, and probe current= 75 $\mu$ A.
  - 5.6.5.4** For analysis of paint samples: Accelerating voltage= 20kV, amp time= 12.8 $\mu$ s, and an acquisition time of at least 100 live seconds. If paint samples are prepared on an adhesive lift a blank should be run to show the elemental contributions the preparation material has on the sample spectra.
- 5.6.6** Adjust live time under Setup menu, Preset & Memory, "Preset Time and Memory Selection".
- 5.6.7** For manual sample runs, adjust the z-position to focus on the sample and select the "collect spectrum" icon on the toolbar to acquire a spectra.
- 5.6.8** For automated sample runs, select data file location, live time, and "Minimize Power at Finish" using the "Automation Setup" located in the Auto menu under Setup Output.
- 5.6.8.1** Set up the points for analysis by completing the Table under the Stage menu. Focus on the sample to be analyzed, save the point, and enter the sample information. Repeat this for as many points as needed.
  - 5.6.8.2** Select "Start Auto Run" from the Auto menu to begin the sample run.
  - 5.6.8.3** Once the automated run is complete the x-ray tube will go into standby mode, which reduces the accelerating voltage and probe current, while the instrument is idle.
- 5.6.9** Turn off instrument upon completion of analysis following the steps under **Instrument Shutdown**.

## **5.7 Post-Run Data Analysis**

- 5.7.1** Spectral data can be analyzed using either ORBIS Vision software or EDAX Spectral Processing Utility.

### **5.7.2 ORBIS Vision**

- 5.7.2.1** Open the .spc file and select “All Disk” to view the sample spectrum.
- 5.7.2.2** The “SPC Preview” option from the File menu will display a window with all .spc files in the folder.
- 5.7.2.3** To identify an element’s KLM lines, select the Peak ID icon in the top right toolbar or use the ID menu. Right click on the peak of interest on the spectrum to get a list of possible elemental sources. Highlight the matching elemental KLM line and select Add to properly label the elemental peaks.
- 5.7.2.4** Before printing, confirm that the printer orientation is landscape under “Printer Setup” and also when choosing “Print” from the File menu.

### **5.7.3 EDAX Spectral Processing Utility**

- 5.7.3.1** Open the folder containing spectral data and select file(s) to be viewed. All .spc files in that folder will appear in the left column.
- 5.7.3.2** To print, highlight spectra to be printed and choose SPC>>Printer from the toolbar.

## **5.8 Instrument Shutdown**

- 5.8.1** Select Unload and remove samples from the stage.
- 5.8.2** Close the Orbis Vision Software.
- 5.8.3** Power down the computer.
- 5.8.4** Shut off the Orbis by pressing the green power button located above the chamber door.

## **5.9 Standards**

- 5.9.1** Aluminum 2024 Standard (Aluminum-Copper alloy)
- 5.9.2** Known elemental standard (ex. Copper)
- 5.9.3** Glass certified reference material (ex. NIST SRM 1831)

## **5.10 Maintenance – Record any maintenance performed in the Maintenance Log.**

## **5.11 Sampling and Sample Selection – N/A**

## **5.12 Calculations**

- 5.12.1** When the area of a characteristic energy peak for an element has a signal-to-noise ratio of

ten or more, that element may be used for peak intensity ratio comparisons.

**5.12.2** The following are some peak ratios that may be used for glass comparisons: Ca/Mg, Ca/Ti, Ca/Fe, Sr/Zr, Fe/Zr, and Ca/K peak ratios may be used. Additional ratios may be used depending on the elements present in the sample.

### **5.13 Uncertainty of Measurement**

**5.13.1** EDS detector resolution <180eV.

### **5.14 Calibration – N/A**

## **6.0 Limitations**

**6.1** This instrument is capable of identifying all elements from sodium to berkelium.

## **7.0 Safety**

**7.1** The greatest safety concern is radiation from the X-ray tube. NEVER operate the X-ray tube without the x-ray shield in place or with any of the x-ray tubes removed. The sample chamber is protected by an interlock. The X-ray will not be on if the interlocks are working properly. OrbisNotifier checks both the Safety Inter-Lock and the Door Switches and will notify the operator if they are working properly prior to use. The x-ray system shall be 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 within the AC distribution box, DC power supplies, and the high voltage x-ray supply.

**7.3** The entrance and exit windows of the x-ray tube are made of a thin beryllium window. Beryllium is a highly toxic metal. Do not touch the foil. Do not expose the window to corrosive substances. If the beryllium window does break, remove the pieces with tweezers or the sticky side of tape and place in a properly labeled, sealed, unbreakable container. Contact the section safety officer for proper disposal. If beryllium comes in contact with the skin, remove beryllium and wash area thoroughly. Check skin for beryllium particles.

**7.4** Methanol is a High Risk Chemical. Refer to Appendix 1 for Chemical Hygiene and Safety Precautions.

## **8.0 References**

**8.1** E2926-17 Standard Test Method For Forensic Comparison of Glass Using Micro X-ray Fluorescence ( $\mu$ -XRF) Spectrometry

**8.2** EDAX Operator's Manual for Orbis PC Elemental Analyzer System

**8.3** EDAX Operator's Manual for Orbis Vision Software

#### 8.4 Laboratory Safety Manual- Chemical Hygiene Plan and Hazardous Communication Program

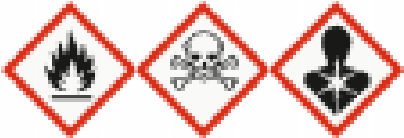
### 9.0 Records

- Maintenance Log
- Daily Check Form
- Energy Adjustment Reports
- Request for Instrumental Examination of Evidence

### 10.0 Attachments – N/A

Revision History		
Effective Date	Version Number	Reason
09/08/2020	1	Original Document

## Appendix 1: Chemical Hygiene and Safety Precautions

Methanol	
<b>DANGER: HIGH RISK SUBSTANCE *</b>	
	<b>HEALTH</b> 2
	<b>FLAMMABILITY</b> 3
	<b>REACTIVITY</b> 0
<b>Detection of Release</b>	Colorless liquid with a sweet, pungent odor.
<b>Signs/Symptoms of Exposure</b>	Headache, Nausea, Dizziness, Eye damage. May cause intoxication that includes central nervous system depression, headache, dizziness, nausea, lack of coordination, and confusion.
<b>PEL</b>	OSHA (TWA) 200 ppm
<b>Associated Hazards</b>	Flammable. Acute oral, dermal, and inhalation toxin. Toxic if swallowed, comes in contact with skin, or inhaled. Specific target organ toxicity of eyes.
<b>Controls</b>	Use under fume hood. Avoid contact with skin, eyes and clothing. Wash hands before breaks and immediately after handling the product. Use eye protection. Handle with gloves. Wear lab coat. Gloves: nitrile (break through time less than 1 minute), butyl-rubber (break through time greater than 8 hours)
<b>Safe handling, storage, disposal</b>	Avoid contact with skin and eyes. Avoid inhalation of vapor or mist. Use explosion-proof equipment. Keep away from sources of ignition. Take measures to prevent the build-up of electrostatic charge. Dispose in Hazardous Chemical Waste. Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.
<b>Emergency Procedures</b>	<p><b>Eye Contact:</b> Flush eyes with water as a precaution.</p> <p><b>Inhalation Exposure:</b> If inhaled, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.</p> <p><b>Ingestion:</b> After swallowing: fresh air. Make victim drink ethanol (e.g. 1 drinking glass of a 40% alcoholic beverage). Call a doctor immediately (mention methanol ingestion). Only in exceptional cases, if no medical care is available within one hour, induce vomiting (only in fully conscious persons) and make victim drink ethanol again (approx. 0.3 ml of a 40% alcoholic beverage/kg body weight/hour).</p> <p><b>Skin Contact:</b> Wash off with soap and plenty of water. Take victim immediately to hospital. Consult a physician.</p> <p><b>Spills:</b> Avoid breathing vapors, mist, or gas. Ensure adequate ventilation. Remove all sources of ignition. Evacuate personnel to safe areas. Beware of vapors accumulating to form explosive concentrations. Vapors can accumulate in low areas. Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Small spills: Contain spillage, and then collect with non-combustible absorbent material, (e.g. sand, earth, diatomaceous earth, vermiculite) and place in container for disposal. Large spills: Turn off sources of heat if possible; evacuate area and call 911 (Haz Mat).</p>