

## **Technical Procedure for X-Ray Fluorescence Spectrometer (XRF)**

**1.0 Purpose** – This technical procedure shall be followed for the operation of the X-ray Fluorescence spectrometer.

**2.0 Scope** – This procedure applies to the Thermo NORAN Quan-X EC Energy Dispersive X-ray Fluorescence Analyzer located in the Trace Evidence Section. This can be used as a non-destructive elemental analysis of trace evidence such as glass, larger metal fragments, powders, other bulk evidence, and unknown liquids.

**3.0 Definitions** – N/A

### **4.0 Equipment, Materials, and Reagents**

#### **4.1 Equipment**

- Thermo NORAN Quan-X EC Energy Dispersive X-ray Fluorescence Analyzer
- Edwards High Vacuum Pump Model RV8
- WinTrace Software

#### **4.2 Materials**

- Polyethylene X-ray cell
- Kapton Pre-cut Circles
- Mylar Pre-cut Circles
- Ultralene Pre-Cut Circles
- KeveX Spectrace OFHC Copper Standard

#### **4.3 Reagents**

- High purity helium gas

### **5.0 Procedure**

#### **5.1 Verification of New Instrument**

**5.1.1** Run certified standards of red phosphorus, iodine, and soda-lime container glass and verify that the elemental composition of each standard is correctly identified.

#### **5.2 Start-up and Performance Check**

**5.2.1** Instrument may remain on at all times. If for some reason the power has been turned off, follow these steps to power the instrument.

**5.2.1.1** Open the samples chamber lid.

**5.2.1.2** Turn on the QuanX Power switch. The instrument will initialize and a final click will denote the completion of initialization. The filter wheel and the

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sample wheel will rotate. Open the top left cover and confirm the Quest DPP light match the parameters listed in the QuanX EC Technical Manual p. 5-8.

- 5.2.1.3** If instrument does not initialize, power off and power up again. If problem persists, contact the service representative.
- 5.2.2** An energy check of the XRF System shall be performed prior to the use of the instrument. A printed copy of each “Energy Adjustment Report” shall be placed in the Log Book.
  - 5.2.2.1** Click once on the Start menu in Windows. Select ACQUISITION MANAGER.
  - 5.2.2.2** Click Once on Analyze from the toolbar menu. Select Energy Adjustment. This will open the “Start Energy Adjustment” window.
  - 5.2.2.3** Place the Cu calibration disk in position 20 of the sample tray. In general, it is recommended to leave the Cu standard in position 20.
  - 5.2.2.4** Select “OK” when ready to begin the acquisition. The energy check will continue automatically until all criteria are met (see Thermo QuanX technician manual for details). If it is necessary to stop calibration while running, select “ACQUIRE” from the main menu and select “STOP.”
  - 5.2.2.5** When the energy adjustment is complete, a window will appear displaying the message “Energy Adjustment has completed.” Click “OK.” The Energy Adjustment Report will be displayed on the screen.
  - 5.2.2.6** Click on the printer icon to print Energy Adjustment report and retain copy with instrument.
  - 5.2.2.7** The Error (eV) must be less than +/-1.5. Repeat **5.2.2** if the error criterion is not met. If a problem persists, contact the service representative. Once maintenance has been performed, the error check must pass in order to continue use for casework.

### **5.3 Setting up WinTrace Software for Analysis**

- 5.3.1** Click on method tray list shortcut on desktop for analysis needed. These are denoted by the .mtl file extension.
  - 5.3.1.1** For clandestine laboratory analysis samples click on “Clanlab.mtl.”
  - 5.3.1.2** For glass analysis samples click on “Glass Intensities Only.mtl.”
  - 5.3.1.3** For unknown liquid analysis click on “General Scan LIQUID.mtl.”
  - 5.3.1.4** For unknown solid analysis click on “General Scan SOLID.mtl.”
- 5.3.2** Open the Quantitative Tray List.

- 5.3.3 Enter standard and sample names in sample column, beginning samples with the row that states "Enter sample name here."
- 5.3.4 Verify that the method file listed is the correct method for analysis by clicking on the document icon.
- 5.3.5 For each sample, enter proper position tray number for each sample.
- 5.3.6 Click on "Edit" in the main menu and select "Spectrum Location." Create a new folder for each batch run. Save all spectra in a folder such as C:\My Documents\. Click on "Make a New Folder" and enter folder name (i.e., 03-03-03-BOB). Click "OK."
- 5.3.7 Place samples into tray according to the qualitative tray list.
- 5.3.8 When finished entering sample information, click once on the first empty row after all samples. The tray list will not run the last sample if the cursor is positioned on a row with a sample.
- 5.3.9 Turn on the helium if running a liquid sample and/or clandestine laboratory samples. Set the pressure at approximately 20 psi.
- 5.3.10 To start analysis, click on "Analyze" from the main menu and select "Start."

#### 5.4 Post-Run Data Analysis

- 5.4.1 Click on Method Explorer at the bottom toolbar to maximize the window.
- 5.4.2 The window contains two columns. The left column lists all Method Contents including Sample Lists, Spectra, and Report Analysis. Clicking on an object in the left column will display more details in the right column.
- 5.4.3 Double click on Spectra in the left column. The spectra bar will expand and to display all data files associated with the previous run.
- 5.4.4 To view spectra of interest, click once on the spectrum data file. The spectrum will open in the right column.
- 5.4.5 To identify an element's KLM lines, select the KLM icon in the toolbar. Hold down the control key while pressing the left or right arrows to shift lines to proper elemental peaks.
- 5.4.6 To label the peaks, click on the Thumbprint icon on the toolbar. To remove peak label, double click on the labeled element.
- 5.4.7 Before printing the first spectrum, click on the File menu and select Printer Setup. Select the Landscape option, and click OK.
- 5.4.8 To print spectrum, click on the printer icon or select Print from the File menu.

#### 5.5 Shutdown Procedure

**5.5.1** Exit the Acquisition Manager. The Method Manager should close on its own.

**5.5.2** The instrument can remain on. The x-ray is not on while the instrument is idle.

## **5.6 Standards**

**5.6.1** KeveX Spectrace OFHC Copper Standard

**5.6.2** Copper standard shall be stored in position 20 on the auto sampler tray.

**5.7 Maintenance** – Record any maintenance performed in the Maintenance Log.

**5.8 Sampling and Sample Selection** – N/A

## **5.9 Calculations**

**5.9.1** For glass comparisons, calculate Ca/Fe and Zr/Sr peak ratios.

- Ca/Fe ratio = Measured response for Calcium (cps/mA) / Measured response for Iron (cps/mA)
- Zr/Sr ratio = Measured response for Zirconium (cps/mA) / Measured response for Strontium (cps/mA)

## **5.10 Uncertainty of Measurement**

**5.10.1**  $\leq 175\text{eV}$  resolution for 5.9keV X-rays at 1,000 cps.

**5.11 Calibration** – N/A

## **6.0 Limitations**

**6.1** This instrument is unable to identify the following elements: hydrogen, helium, lithium, beryllium, boron, carbon, nitrogen, oxygen, fluorine, and neon.

## **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. 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 stick 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 and wash area thoroughly. Check skin for beryllium particles.

## **8.0 References**

### **8.1 Mtl Method Appendix**

- Clandestine laboratory analysis. Clanlab.mtl
- Glass analysis. Glass Intensities Only.mtl
- Unknown liquid analysis. General Scan LIQUID.mtl
- Unknown solid analysis. General Scan SOLID.mtl

### **8.2 Thermo Electron Corporation, Technical Manual QuanX EC Energy Dispersive X-ray Fluorescence Analyzer**

### **8.3 Thermo Electron Corporation, QuanX EC Operator's Manual WinTrace Software**

## **9.0 Records**

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

## **10.0 Attachments – N/A**

| <b>Revision History</b> |                |  |
|-------------------------|----------------|--|
| 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              | Removed Performance Check Log from Records (9.0), Report now maintained with the instrument.<br>Removed mandatory preventative maintenance from Maintenance (5.7)<br>Edited wording in 5.4.5 and removed 5.4.9 |
| 10/18/2013              | 4              | Added issuing authority to header  |
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