Title: PANalytical Epsilon 5 XRF Spectral Acquisition, Processing and Reporting

Copy No: ##

Effective Date: July 3, 2013

QSM Approval:	
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Location: ###

PANalytical Epsilon 5 XRF spectral acquisition, processing and reporting

1. Introduction and Scope

SOP No.: 6.19/1.2/S

- 1.1. This Standard Operating Procedure provides instruction for the operation, acquisition and processing of spectra followed by preparation of final reports for elements in ambient aerosols using the PANalytical Epsilon 5 spectrometer determined by method 6.9/*.*/M, "Multi Element Analysis of Ambient Aerosols using Energy Dispersive X-ray Fluorescence Spectrometry".
- 1.2. Spectra are acquired using the Epsilon 5 software for Windows®.
- 1.3. Spectra are processed using the Epsilon 5 software for Windows®.
- 1.4. Results are reported using Microsoft Excel®.

2. XRF Operation and Spectral Acquisition

2.1. Instrument Start Up

Turn CPU and monitor on and enter passwords to start windows.

Press the Power ON button located on the front of the instrument control panel to the on position, the button will light up to indicate that the power is on. This switches the Epsilon 5 into the power on state.

Turn the HT key switch, on the control panel of the Epsilon 5, a quarter turn clockwise. This enables the High Tension.

When the X-Ray lights on top of the instrument turns on then Double click the Epsilon 5 Software icon to start the analytical software via windows. The software will start the Epsilon 5 multi document interface (MDI) window.

The instrument is now ready for operations.

The spectrometer and computer are always ready for use. (There is no warm up needed for this instrument due to the Epsilon 5 X-ray tube always running at 80KV and 4.0mA).

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2.2. **Loading of Samples**

Open the sample chamber cover and remove the stainless steel sample trays containing the filters from the instrument (Tray for 2x4 sample cups).

Remove and invert the stainless steel sample cup holders. Then, remove them from their liners, exposing the filters. Remove the existing filters and return them to their appropriate petri dishes.

Remove the new filters to be measured from their petri dishes and place them, facing up, onto the liners. Replace the sample cup holders over the liners and replace them re-inverted onto the sample tray.

Open the sample chamber cover of the spectrometer and place the sample trays back into their appropriately labeled positions.

Close the sample chamber cover of the spectrometer.

2.3. **Preparing Spectrometer for Measurement of Filters**

Open the Sample Changer Measurement window by clicking on the "Open sample changer window" icon located at the top of the "PANalytical Epsilon 5 Software' window.

Once open, drag and drop an empty tray from the 'Tray, cup & insert' area on the labeled tray sections within the 'sample changer' area to start defining new trays for measurement.

In the right upper corner, the list with available 'application' area is shown, i.e. Air Filter. Drag and drop an application to a tray. (Note the standards that are defined in an application are show if the tab 'standard' is selected).

Enter the sample data and other pertinent information into the 'Sample detail' area located below the 'Tray, cup & insert' area for each sample that must be measured.

Acquiring Spectra

Press the measure button to transfer the samples into the 'Measure queue' area. If only one tray or an individual sample is to be measured then, drag the tray or sample to the "Measure queue' area.

Press the start button to begin the measurement of the current samples from the measuring queue. This button will toggle to STOP once pressed.

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The Stop button finishes the current measurement, and pauses the queue. This button will toggle to START once pressed.

The Abort button aborts the current measurement and will pause the queue (not delete the queue items)

The spectra that are being generated can be viewed by clicking the "Show the spectrum preview" icon.

When the queue measurement is finished, the results can be viewed in the last results window by clicking the "Display the last results window" icon.

2.5. Shutdown of the Epsilon 5

If it is necessary to shut down the system, perform the following steps:

- 1. Close the analytical software.
- 2. Confirm with Yes on the question 'Do you want to switch off the detector and generators high tension.

Note: Confirm with a No on the question "Do you want to switch off the detector and generators high tension if performing a simple computer reboot.

- Turn the Key switch, on the panel, a quarter turn anti-clockwise, 3. this disables the HT.
- Press the power 'On' switch on the control panel of the Epsilon 5 4. to off.
- 5. Switch off the computer, printer, and monitor.

Note, to maintain the Epsilon 5 system performance according to the manufactures specification, it is important that the instrument is left switched on (Epsilon 5 system, computer and software).

Spectral Processing

The spectra are generated and processed automatically during a spectral acquisition and/or during the queue measurement. Also, the spectra being generated can be viewed by clicking the "Show the spectrum preview" icon.

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Report Preparation

- 4.1. When the samples in the queue measurement are finished, the results can be viewed and processed by clicking the "open window" icon followed by pressing the results button within this window. The results that are wanted can be found under the appropriate batch name. This will begin the process to obtain the data from the spectra that where produced earlier
- Under the sample identification heading within this window you can select 4.2. the desired samples to report or view. To view spectra details click on "Show in spectrum window" icon and to view the report click on the "Show report" icon.
- Once the data is shown in the 'Report' area located at the bottom of the 4.3. screen, this information can be transferred from the PANalytical Epsilon 5 Software to an Excel® document by clicking the 'Print...' icon under the file tab. Make sure the 'Clipboard', 'Delimited', and 'Tab' are select before pressing 'OK'. Then name and save the result file to a desired location on the computer
- Open E5_DYXX_Report Template.xls, located in the directory XRF 4.4. Lab\Documents\Work Forms. Click "Enable" to activate the macros within the temple and enter the XRF lab password. The file can now be renamed and saved in XLS format. Click "File" then "Save As", then in the "Save as type:" space, choose "Microsoft office Excel® Work (*.xls) and the directory where you would like to save this information. The macros within this worksheet will create the XRF report document for the client by changing the data layout, perform a zero adjustment on (-) numbers and inserting the correct IDLs.
- 4.5. Open the previously saved result file and from this worksheet run the conversion macro embedded within the E5_DYXX_Report Template.xls file. Once the macro is finished running this file can be closed without saving.
- Enter all pertinent and current information into the newly named file and format changes. Then run the finalize macro embedded within the file to remove all link by performing a copy paste special on each worksheet within the workbook.
- 4.7 This document also includes the Duplicates and Blank Evaluation and Supervisor Approval Letter that must be completed.

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5. Applicable Methods and SOP's

6.09/*.*/M, "Multi Element Analysis of Ambient Aerosols Using Energy 5.1. Dispersive X-ray Fluorescence Spectrometry

Revisions

Sep. 2008:

• Author: John Coltess: New document

May. 2011:

- Reviewer: John Coltess
- Section 1.2: Edited software name
- Section 1.3: Edited software name
- Section 4.1: Changed the word "application" to "batch" to reflect current reporting processes. Removed phrase "i.e. Air Filter".
- Section 4.5: Changed the acronym "DL" to "MDL". Edited directory path to reflect the current location of the macro file.
- Section 4.6: Changed the acronym "DL" to "MDL". Edited directory path to reflect the current location of the macro file.

June. 2013:

- Reviewer: John Coltess
- Section 2.1: updated process on instrument start up
- Section 4.3: Edited information to better reflect the process of "Report Preparation"
- Section 4.4: Renumbered to 4.5. Edited information to better reflect the process of "Report Preparation". Changed the acronym "MDL" to "IDL". Edited directory path to reflect the current location of the macro file
- Section 4.5: Renumbered to 4.4. Edited information to better reflect the process of "Report Preparation.
- Section 4.6: Deleted

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- Section 4.7: Deleted. Information combined with Section 4.4 to better reflect the process of "Report Preparation.
- Section 4.8: Renumbered to 4.6. Edited information to better reflect the process of "Report Preparation".
- Section 4.9: Renumbered to 4.7.

7. References

- "PANalytical Epsilon 5 System User's Guide 4th ed. 2005" 7.1.
- "PANalytical XRF Epsilon 5 Training Manual. 2005 7.2.

Lead Reviewer John Coltess

Title: Technologist, Particulate Characterization Unit

Approved By: David Mathieu

Title: Supervisor, Particulate Characterization Unit

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