Using Spectro Xepos EDXRF Software

Assuming the instrument is running and the software has been loaded, this document provides instructions for performing some routine procedures using the software that runs this instrument. The manual is of course a more comprehensive resource, it is in a white 3-ring binder near the instrument.

Before measuring a sample you should be aware of some preliminaries and precautions in using the instrument.

1. Never pull vacuum on a liquid or powder sample! Never use a method that will involve a measurement under vacuum if you have a liquid or powder in the sample compartment. This can ruin the instrument. At the end of an analysis, prior to pulling a vacuum, be sure you have removed all of your samples from the sample compartment.

2. If using a method that involves a helium purge, do pull a vacuum after removing samples from the sample compartment.

3. Analyze liquids immediately after preparation. Always test liquid samples for leaks by placing upside down onto a paper towel prior to analysis.

4. Never analyze liquids or powders in sample position number 1.

The MCA calibration standard should always be run if the instrument has not been on for a while. Once on, the standard should be run twice a week. This calibrates the multichannel analyzer. Peform the following procedure:

1. Place the MCA calibration standard in an aluminum cup and place in sample position number 1 in the sample compartment.

2. Go to File --> Method

3. Select the method “mca-2347”

4. Copy the 1st (top) sample to the clipboard

5. Go to File --> Routine --> Routine. This should bring up the “sample plate”.

6. Select the Routine for MCA Calibration

7. Confirm all messages and start the measurement. Make sure both measure and evaluate are checkmarked.

The calibration runs automatically. If no message appears no error has occurred and the calibration ran fine.

Measuring a sample

1. The spectrometer is equipped with a 12-position autosampler, so up to 12 samples can be programmed in to be analyzed at any one time. You need to keep track of which samples are placed in which positions so you can tell the software to keep track of the samples and appropriate measurement conditions for each sample. Each sample should be loaded in a plastic cup covered with prolene or mylar film, unless it has been appropriately pressed.

NOTE: The sample compartment is interlocked so you cannot open it while the x-rays are on. To open the sample compartment see if the X-ray LED is lit on the front of the instrument. If it is, click on the standby button on the top right of the main screen. The spectrometer should always be on standby (x-rays on, sample compartment evacuated) if the instrument is on and not in use.

2. Go to File --> Routine --> Routine. This should bring up the “sample plate” or table of samples which can be used to create new samples to be analyzed.

3. Select the number of positions on the sample plate – 12 in our case. This should be sample plate number 1.

4. Select the position of your sample on the sample plate with a mouse click --> New. This opens a new window “Enter/Edit Sample Data”

5. Each sample needs at least 3 things: a name, a method, and a job. So name the sample, then select the method. This is important as it will set the measurement conditions to the default settings of the method. **If you are measuring a liquid or powder sample, do not use a method which involves evacuation of the sample!** Various methods may be developed and exist on the hard drive. The turboquant methods (TQ) preinstalled on the instrument are most often used for an overview type of analysis to determine what types of elements are present and their approximate concentrations. There are two types, one for pressed pellets which uses a vacuum, the other for powders which uses a helium purge. Choose the appropriate method for your sample.

Also select the appropriate job. The job number can be thought of as a folder or subdirectory in which your results are stored. There are usually jobs created for various projects (see below). Job 0 is the default “collective” job for miscellaneous samples. Job 1 is for instrumental analysis, etc.

6. Click OK to close the window and you will return to the sample plate.

7. Create additional samples if desired on different positions of the sample plate.

8. Select the actions you wish to perform such as Measure, Evaluate, or both, and Standby.

9. Click Start to run the samples. If the method you are using requires a vacuum or helium purge a warning message will appear to inform you of these measurement conditions. Take heed, if you are sure about the measurement conditions confirm with yes and the measurement will begin. Remember if the measurement requires a helium flush, make sure to remove your samples and evacuate after the analysis is complete.

Creating a New Job

You may want to create your own job or folder to store your samples if you are undertaking a project. Each job can store up to 1000 samples.

1. Go to File --> Job --> New

A new window pops up to edit the name and other job information. As defaults for the new job the settings of the old job are copied. Change the Job Number and Job Name. The Job Number must be unique, the Job Name is just an additional description. Both are required fields. Type in any additional information and select OK.

Creating a New Method

This is a task which should not be taken lightly. You will need the software manual and perhaps the phone number of the applications chemist at Spectro, currently Dr. Meredith Daniel. This is just to get you started.

1. Go to File --> Method --> Select. Select a method to use as a template

2. Click New (Top one) and give it a name.

3. --> Edit Measurement Conditions. Generally leave the measurement parameters alone, the measurement times can be safely changed. Put in the appropriate information. The sample mass, Dilution mass, and diameter are only important for Turboquant methods.

4. --> Edit Deconvolution --> Select Elements. In deconvolution parameters make sure elements you are interested in are concentration elements.

5. --> Edit Calibration --> Calibration. The “Lucas-Tooth Price is a “straight line” correlation with correction factors.

6. --> Edit Output