MATERIALS CHARACTERIZATION FACILITY
UC SAN DIEGO MATERIALS RESEARCH SCIENCE AND ENGINEERING CENTER
University of California San Diego • 9500 Gilman Drive • La Jolla, California, 92093
mrsec.ucsd.edu

Epsilon 1 XRF S.O.P

Instrument:

Epsilon 1 XRF from Malvern Panalytical

Software:

Epsilon Dashboard

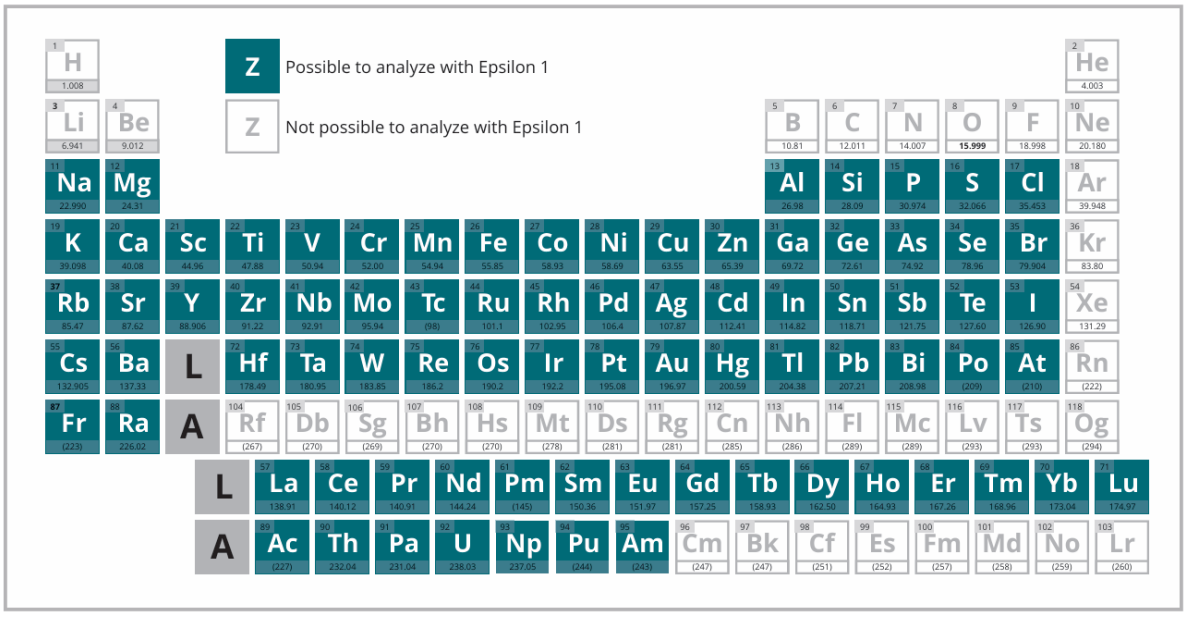
Purpose:

For Quantitative Elemental Analysis, in the range of Sodium to Americium in the Periodic Table, of liquids or solids through X-Ray Flouorescence.

To Begin:

- **Know the specifications of the instrument.**

Specifications	Epsilon 1
Source	Silver Anode X-Ray tube (10W)
Detector	Silicon Drift Detector
Calibrations	Omnian Factory Calibrated*
Sample Cells	Large or small volume options
Sample Dimension Max	15 x 12 x 10 cm (W x D x H)
Range	7-50 kV
Elemental Range	Na-Am



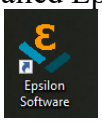
Periodic Table Image: From Malvern Panalytical BR-Epsilon-1-PN11841

- **Steps to Start Up the Instrument**
  - **NOTE:** The X-Ray Source is powered on however will only ignite when initiated by the system. X-Ray indicating lights will shine when source X-Rays are active
  - **NOTE:** Use of the instrument requires prior completion of Research Radiography from UC Learning Center. Documentation required for access, failure to provide documentation will prevent access

1. The instrument should be on, if not there is a power switch in the top left of the system
2. Log in to the computer.
   - There is no computer password.

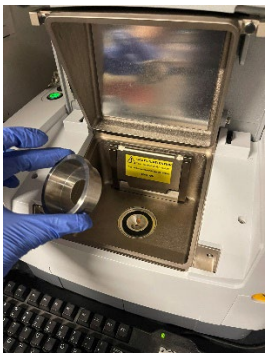
I. Open Software

1. The software for the XRF is called Epsilon Software



II. Take a Gain Measurement

1. The software will ask you to measure the gain at the start of a new experimental session.
2. Put on a new pair of Nitrile Gloves
   - The reference sample contains beryllium and is extremely toxic.
3. Open the sample chamber and remove the sample cup.



4. Place the reference sample face down over the detector and then shut the sample chamber.

5. Press OK. The gain will take about 5-7 minutes to measure. Now is a good time to prep your sample.
6. Once the gain is taken, return the reference sample to its standby position and place the sample cup back into its place.

III. Sample Prep

❖ There are several sample holders that you can use based on your needs. Setup between the different holders is similar to the procedure below.

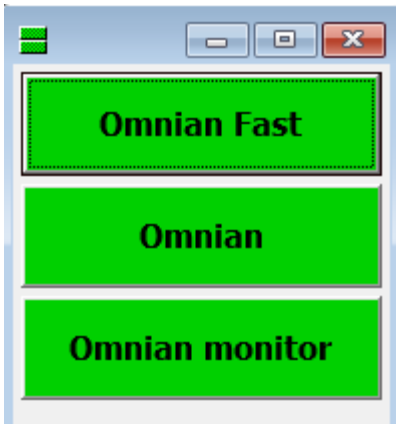
1. The plastic standard sample holders consist of an inner ring, an outer ring, and a 3.6 µm mylar film. The liquid sample holders also have a cap.
2. Place the outer ring with the inner curved edge face up.
3. Place the mylar film over the top of the outer ring.
4. Press the inner ring into the outer ring so that its curved outer edge is face down and will stretch the mylar film taut on the bottom of the sample holder.

5. Add your sample to the holder, and then place it in the sample chamber.

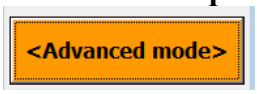
6. Close the sample chamber and prepare to take a measurement.

IV. Running a Measurement

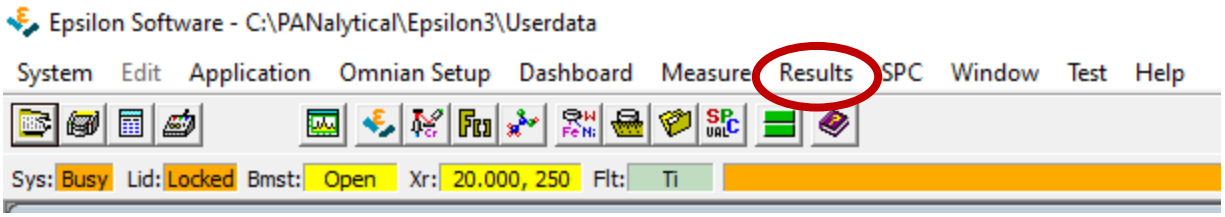
1. Once your sample is loaded, select the type of measurement that you would like to run
   - Omnian fast: Fast XRF scan of your sample. ~10 min wait time.
   - Omnian: Full XRF scan of your sample. ~30 min wait time.
   - Omnian monitor: for calibration only, staff use only.

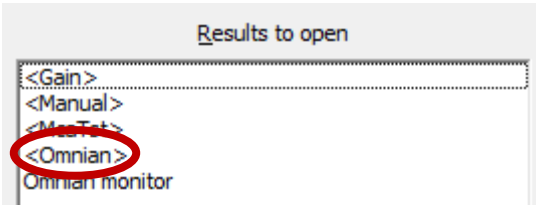


2. The program will ask you to name your sample. Once you hit "OK" the chamber interlocks will activate and your scan will proceed.
3. Once your scan has completed, it will display the results in the "Last Results" Window.
4. In order to modify the analysis and quantification parameters, activate "Advanced Mode". The password for Advanced Mode is **Epsilon**.

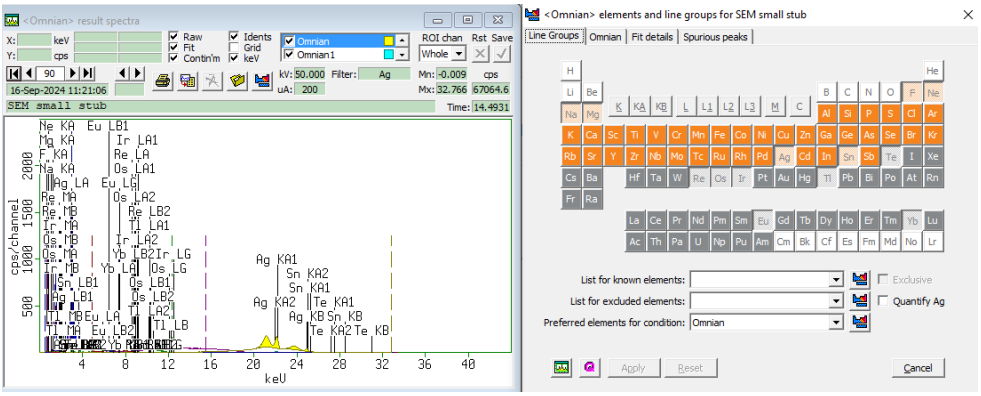


5. Open the results tab (at the top of the window) and select <Omnian> results.

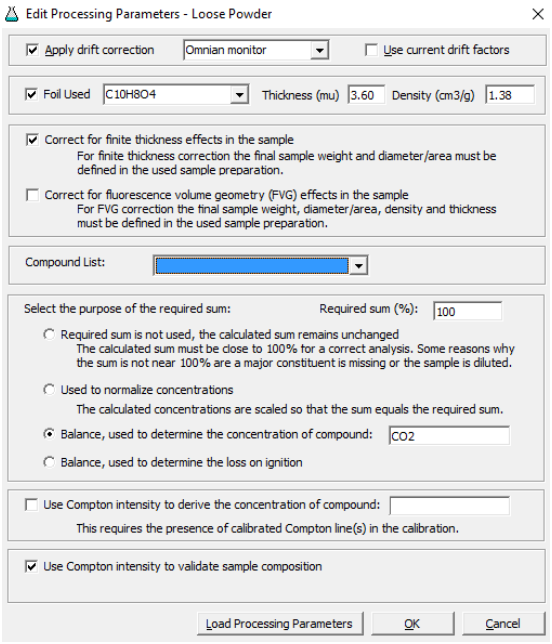




6. In the new results window, select the periodic table icon 
   - This will open up 2 new windows, depicted below.



7. In the window with the periodic table, double click elements that you want to detect in your sample. Any element icons that are not "pushed-in" will be ignored in the final analysis.
   - You can also select premade lists for known or excluded elements.
   - If your sample contains Ag, check the box next to Quantify Ag.
   - When done selecting your elements, select "Apply" and you will see the spectra window update with the elements the program looks for.
8. In the spectra window, look through all the spectra individually to double check element spectral identification. Some peaks can be misidentified by the program and need to be manually corrected using the periodic table window.
   - If you want to export the spectra, select the "Export Spectrum" button  and export your selected spectra as a .mps file.
9. Back on the periodic table window, select the Omnian Tab, and then select "Edit Processing Parameters"
   - If you have premade processing parameters, select "Load Processing Parameters" and select it from the list.
   - If you don't have premade parameters, select the correct processing parameters for your analysis. This includes:
     1. Applying a drift correction
     2. Selecting Mylar 3.6 µm film as the foil used
     3. Apply for relevant corrections
     4. Selecting a compound list (select the empty option if not interested in a specific compound list)
     5. Determine the purpose of the required sum
        a. "Balance, used to determine the concentration of compound" is better for extracting concentrations, can change this to your solvent for liquid samples
        b. "Used to normalize concentrations" is used if only interested in relative percentages.

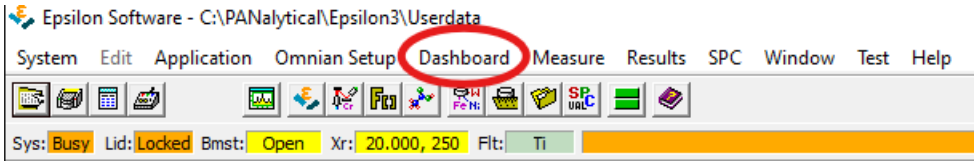


- Select "OK" when done the correct parameters are selected.

10. Once your correct parameters and elements of interest are selected, select "Quantify" on the omnian tab. This will update your results tab with new elemental analysis.
    - This table can be exported using the print to pdf option.

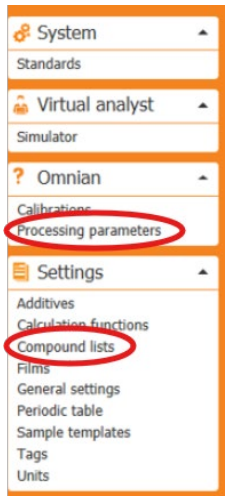
V. Custom Processing Parameters and Compound Lists

1. You can make your own processing parameters and compound lists using by opening Epsilon Dashboard using the Dashboard tab.



2. The password to access this is also "Epsilon"
3. This will launch a separate application. The tabs most relevant to data processing are highlighted in the image below.
    - WARNING: Do not delete or overwrite a setting or list that already exists. This will disrupt other users of the XRF and could affect the instrument's performance.
    - Instead, copy existing lists that are close to what you need and modify that instead. Name your lists and settings something unique so that you can quickly find them in the future.

- If you need to make custom settings for other functions, please consult MCF staff before doing so.



4. You can close Epsilon Dashboard once you have saved your custom settings.

VI. Ending your Session and Clean Up

1. Remove your sample from the sample chamber. The sample cups are disposable and can go into the properly labeled waste container nearby.
2. Close Epsilon and leave the XRF turned ON.
3. Collect your data using a thumbdrive.