Standard Operating Procedure

Rigaku ZSX Primus II XRF

Yale West Campus
Materials Characterization Core
ywcmatsci.yale.edu

ESC II, Room A119C
810 West Campus Drive
West Haven, CT 06516

Version 2.0, February 2024

- Please **FOLLOW the SOP strictly** to keep the facility in good condition. Any **explorations are strongly prohibited** unless permitted by lab manager.
- **Only** use the Core USB drive on the XRF computer.
- **NEVER** surf the web on the computer to minimize the risk of the computer being hacked
- Users should acknowledge MCC in their publications. The general acknowledgement for XRD should read:
    - "The XRF data was taken using the Rigaku ZSX Primus II at Yale West Campus Materials Characterization Core."
- The core reserves the right to use the data for core promotion.

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Rigaku ZSX Primus XRF Standard Operating Procedure

1 Introduction

1) Instrument features:
   - > Wavelength Dispersive X-ray Fluorescence (WDXRF) systems with high resolution (typically 5 – 20 eV) and minimal spectral overlaps
   - > Analysis of elements from Be to U
   - > Tube above optics minimizes contamination issues
   - > Micro analysis to analyze samples as small as 500 µm
   - > Mapping feature for elemental topography/distribution
   - > Helium seal means the optics are always under vacuum
2) Location

   Materials Characterization Core
   Room A119
   810 West Campus Drive
   West Haven, CT 06516

3) Primary Staff Contact

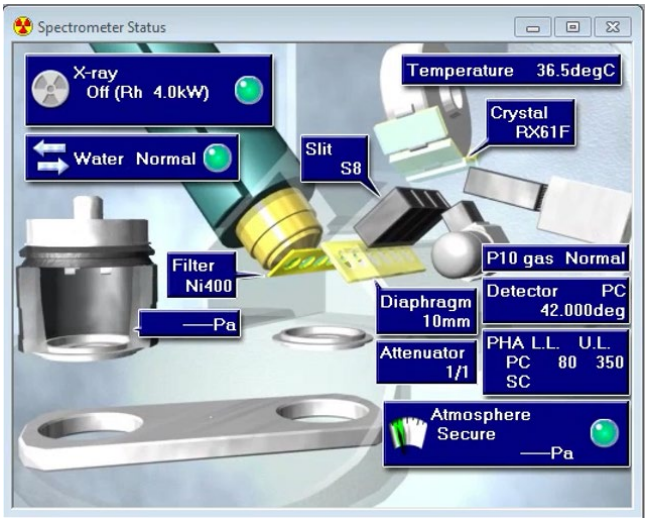
   Dr. Min Li
   Tel: 203-737-8270
   Email: min.li@yale.edu
   Office: ESC II, Room A119D

The Yale West Campus MCC Facilities are operated for the benefit of all researchers. If you encounter any problems with this facility, please contact the staff member listed above immediately. There is never a penalty for asking questions. If the equipment is not behaving exactly the way it should, contact a staff member.

Notice: Please **follow** strictly the **SOP** to keep the facility under good condition. Please **DO NOT** explore the operation program unless approved by core manager.

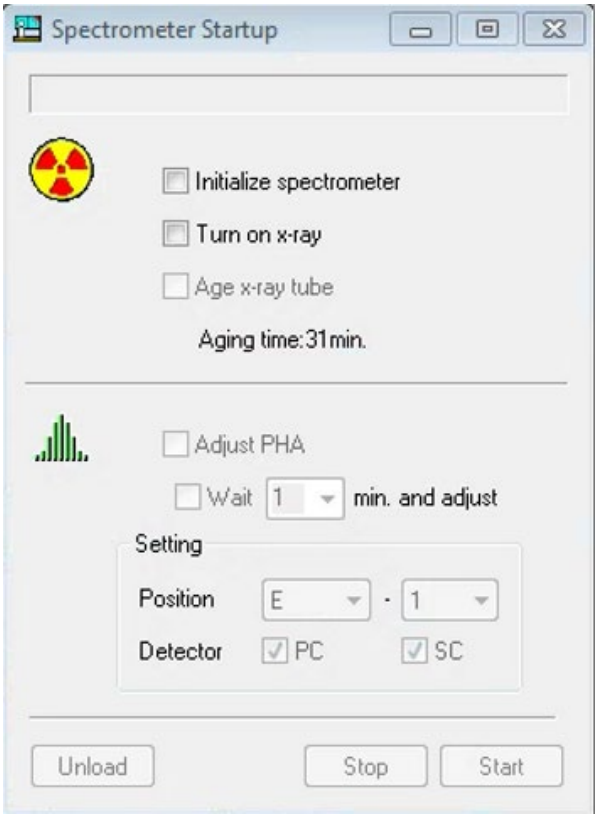
2 System status check

1) Sign in the logbook on the bench.
2) Check the **Spectrometer Status** below and make sure no warning messages appear. If not, contact the manager immediately.

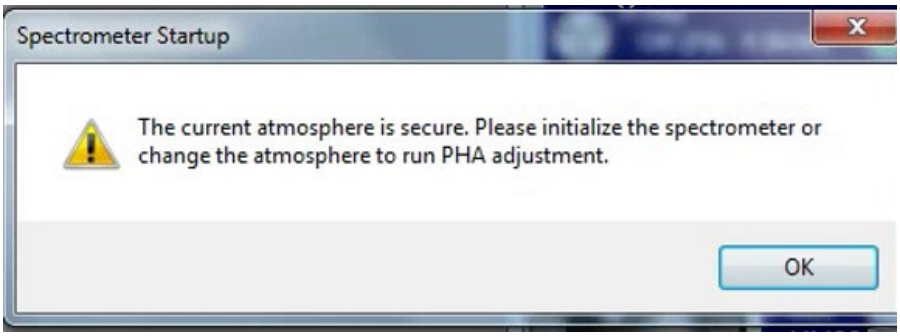


3 System Startup

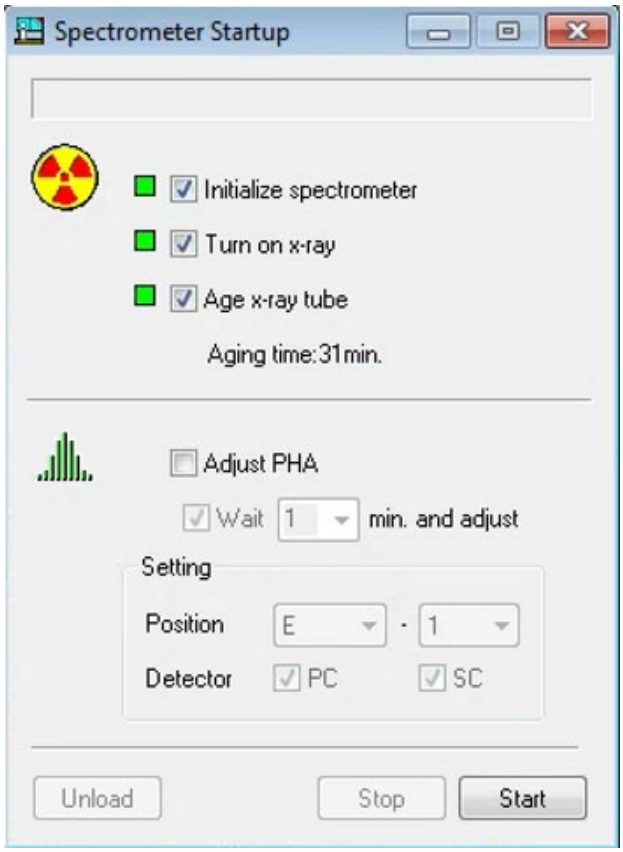
1) On the top menu, click on Startup/ Shutdown > Startup> to open the Spectrometer Startup window below:



2) If a popup window below shows up



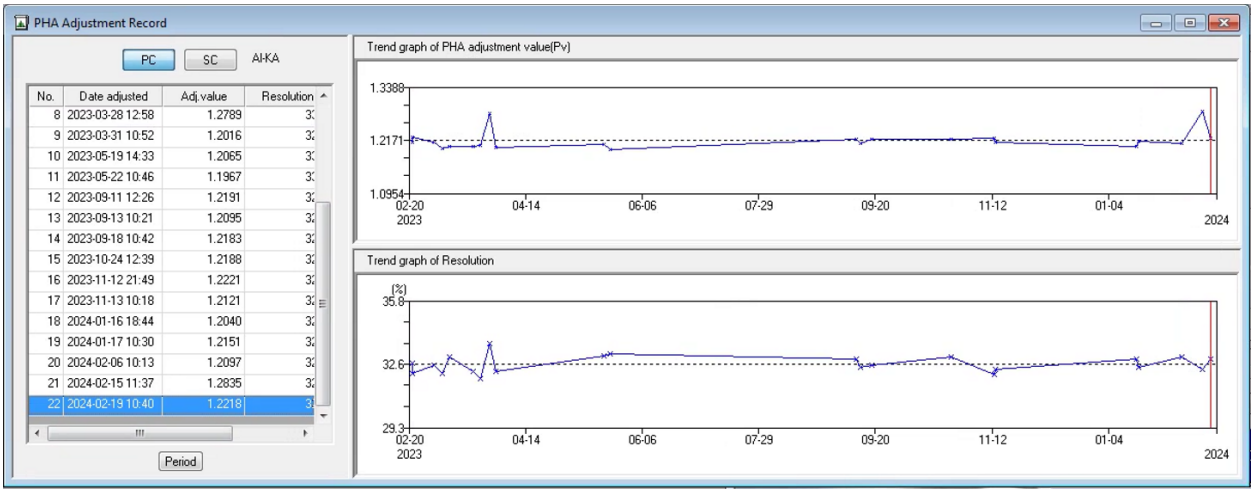
3) Click OK button above and check the small boxes in front of **Initialize spectrometer**, **Turn on x-ray** and **Age x-ray** below to finish the system startup/warmup. It could take from 31mins to 1 hour, depending on if the time since last use.



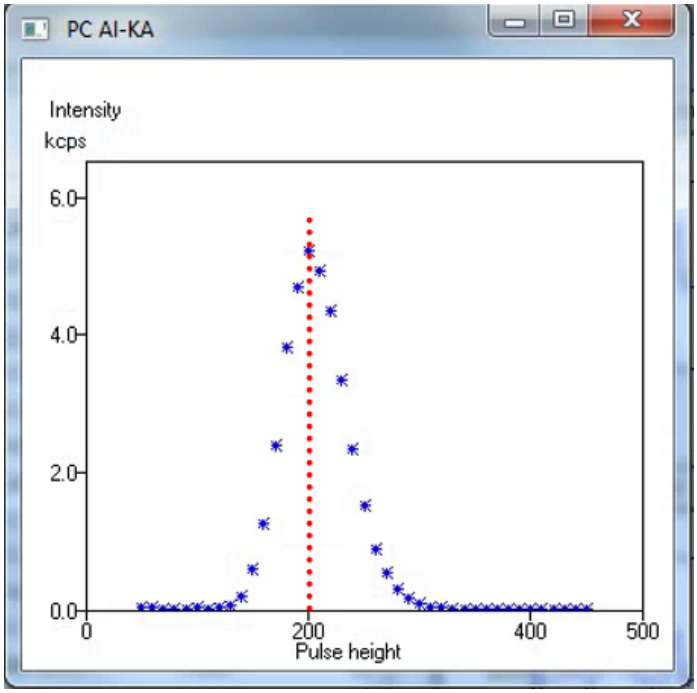
4 Check PHA Analysis result

a) Once the measurement finishes, click the **Maintenance > Maintenance Records > PHA Adjustment** on the top menu.

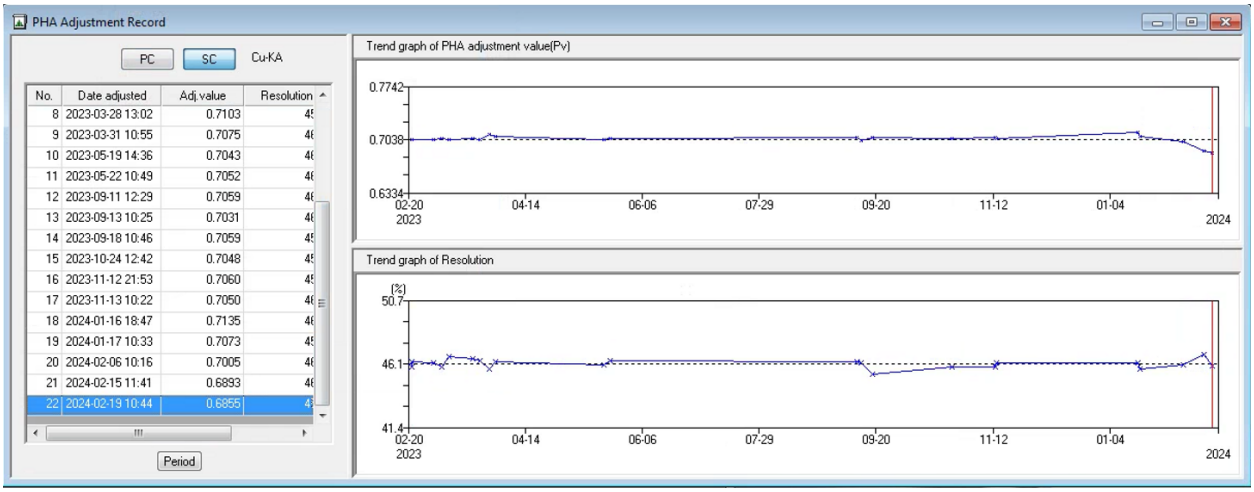
b) In the **PHA adjustment Record** window below with **PC** (proportional counter) highlighted on the top, scroll down to the bottom and click the most recent time record. A red line will show up at the end of the **Trend graph of Resolution** curve as show below:



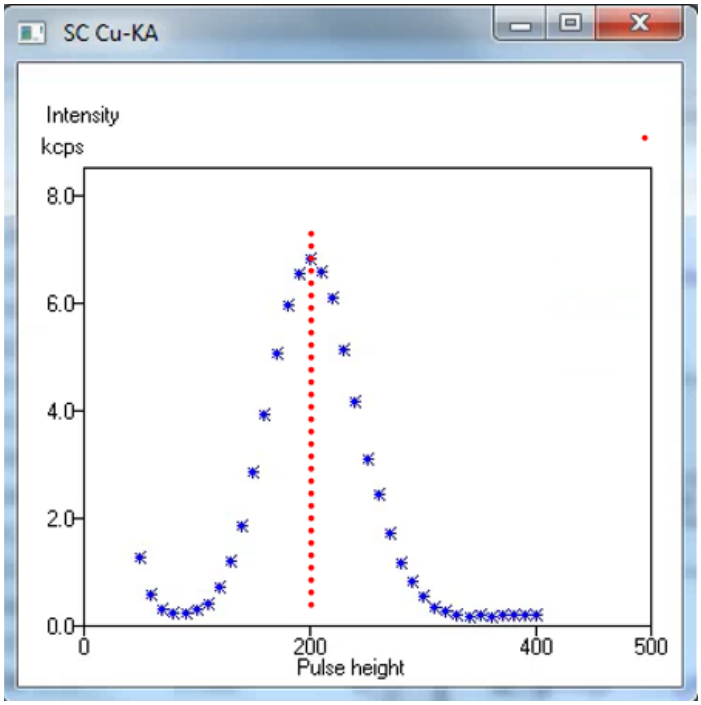
c) Move the mouse to the red line, right click and select **PHA curve** on the popup menu top open the **PC Al-KA** window below. Make sure the Gaussian shaped peak is centered at 200 and intensity between 4.0 and 6.0 kcps as highlighted. Contact the manager if not.



d) Close the window above and click **SC** (scintillator counter) button on the top of the **PHA Adjustment Record** window as shown below. Scroll down to the bottom and click the most recent time record. A red line will show up at the end of the **Trend graph of Resolution** curve as show below:



e) Move the mouse to the red line, right click and select **PHA curve** on the popup menu top open the **SC Al-KA** window below. Make sure the Gaussian shaped peak is centered at 200 as highlighted and intensity between 6.0 and 8.0 kcps. Contact the manager if not.

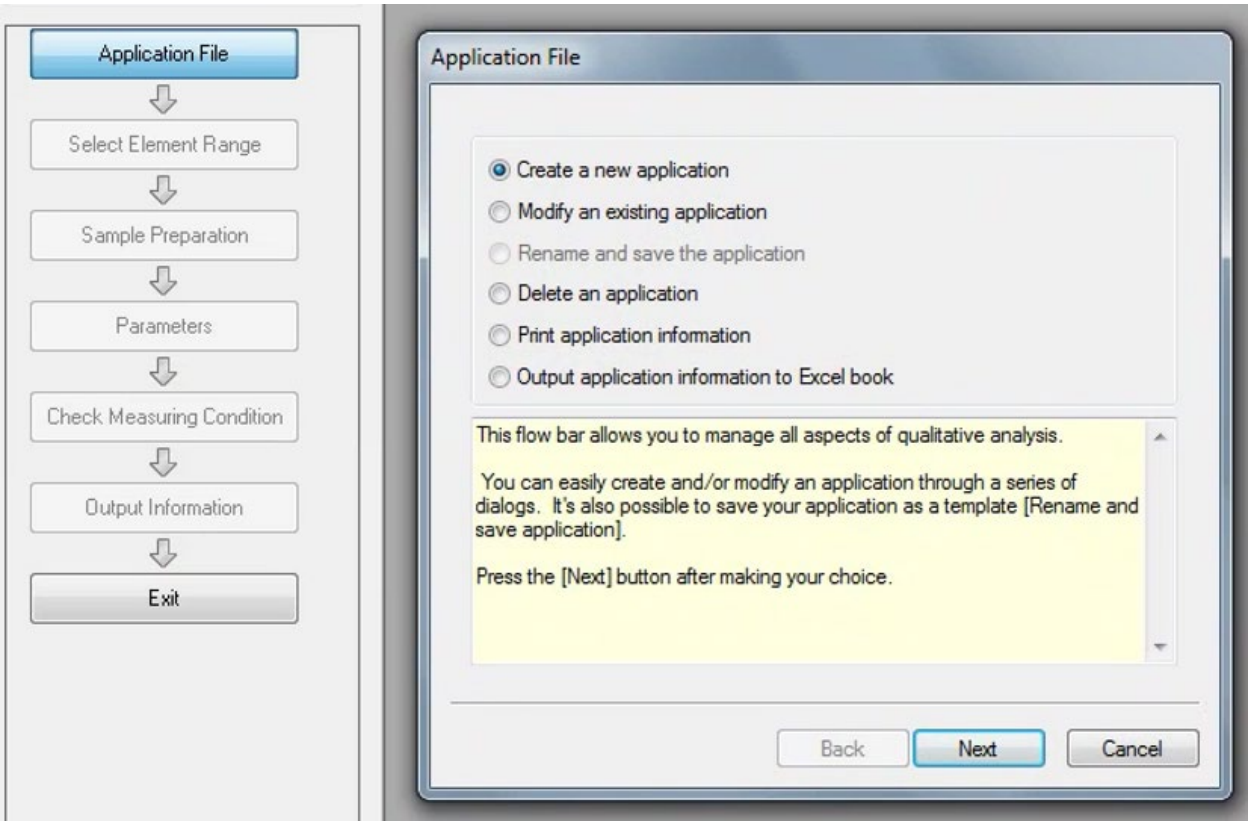


5 Sample preparation (wear gloves)

1) For solid samples, make sure the surface is flat and the sample is larger than chosen metal masks of the sample holder. Blow the surface with nitrogen inside the fume hood.
2) For powder samples, sandwich with prolene membranes provided by core. Maximize the surface area to increase the signal counts.
3) No liquid sample allowed.

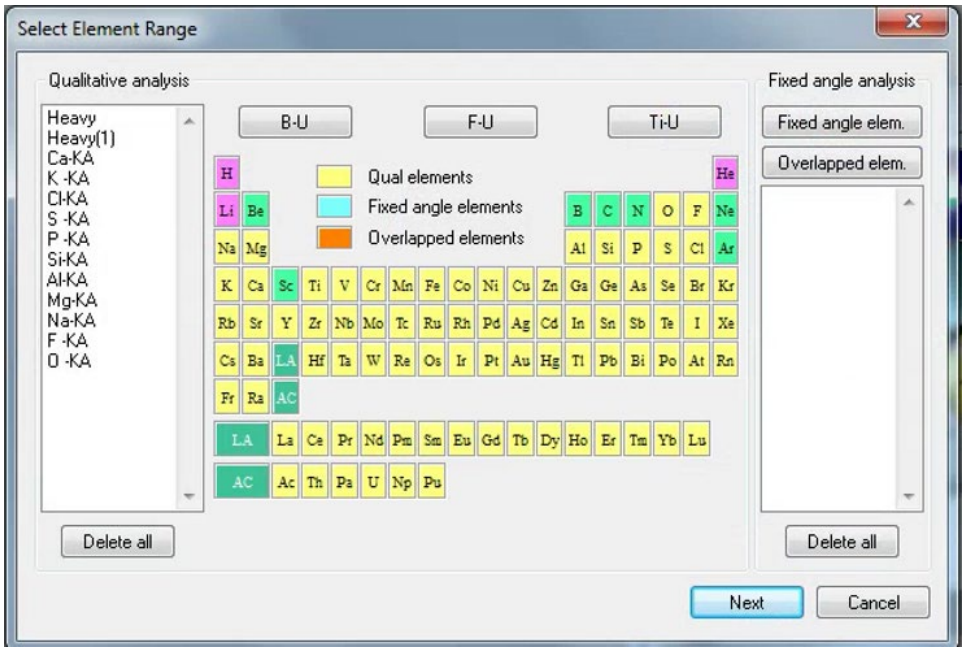
6 Setting up Application File

1) Click **Qual Application** on the top menu to open the Application File setup window below:

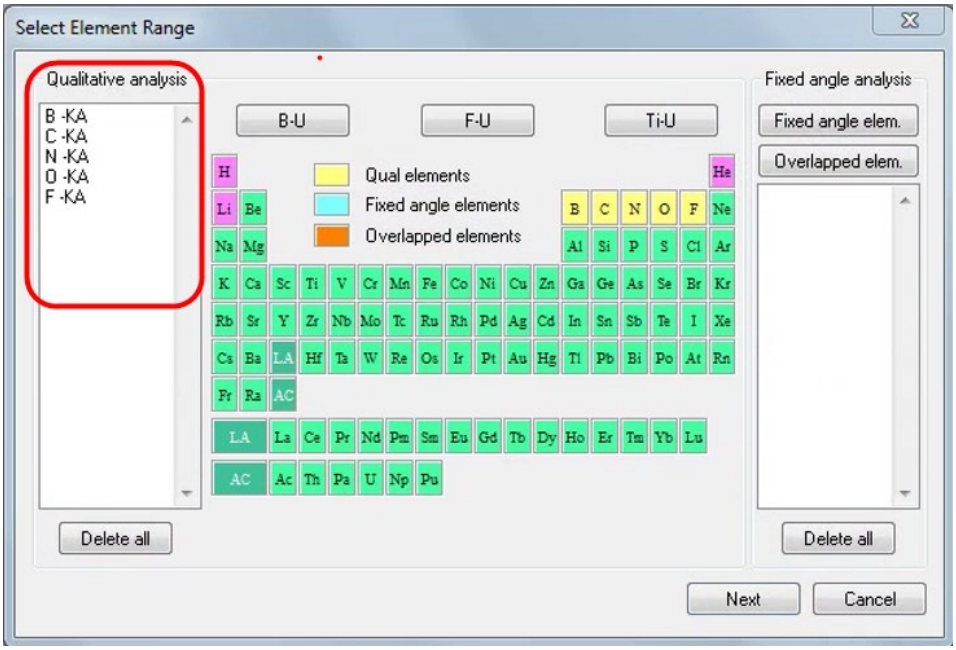


2) Select **Create new application** or **Modify an existing application** and click **Next** button.
   a) Inside the **Select an Application Template** window, select **Metal & Alloy.**
   b) Create the **Name of application** and choose/create a folder to save the application file. Click **Finished** button.

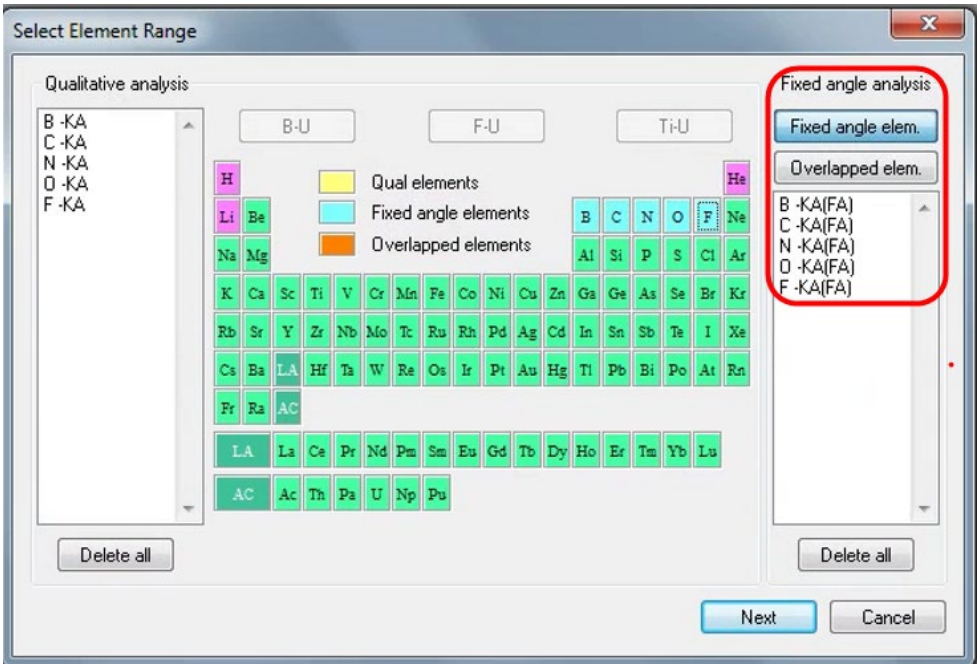
3) Ins the Select Element Range window below:



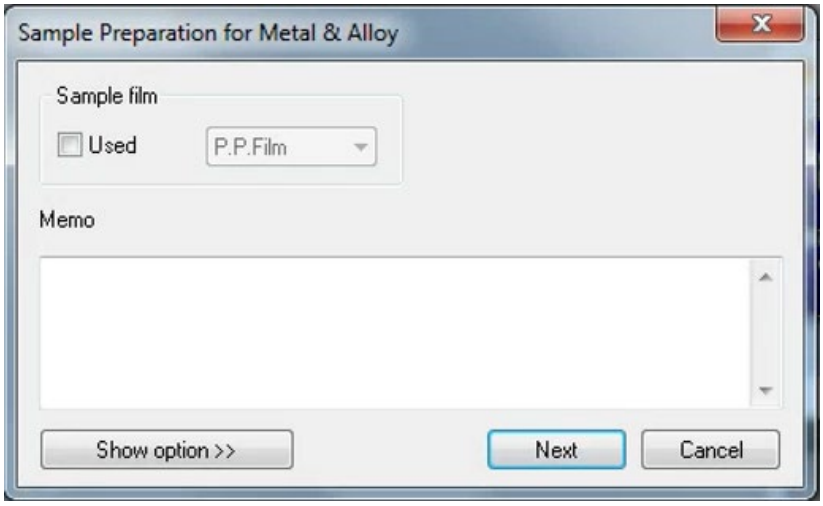
a) Click **Delete all** button on the bottom left corner to remove the elements already listed in the left space for Qualitative analysis.
b) Select interested elements inside your sample by clicking on the periodic table which will appear yellow in the table and appear in the left side space as shown below:



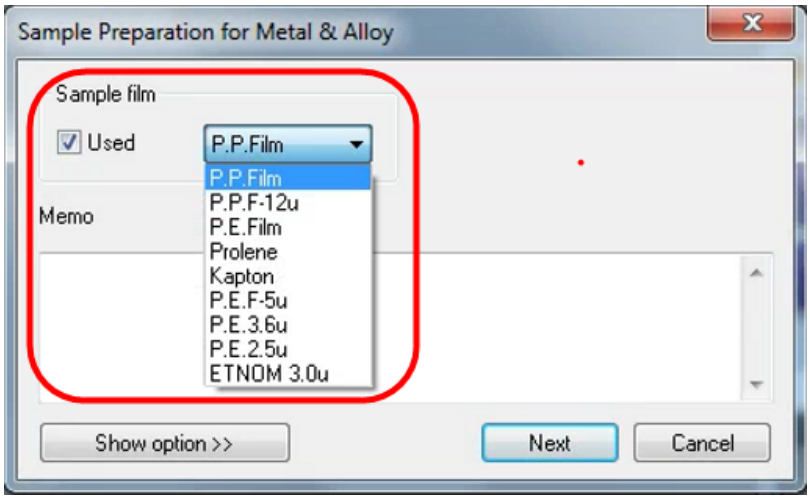
c) Click the **Fixed angle elem.** button on the top right corner of the window above and click previously chosen elements which will appear cyan and listed in the right space for the **Fixed angle analysis** as show below:



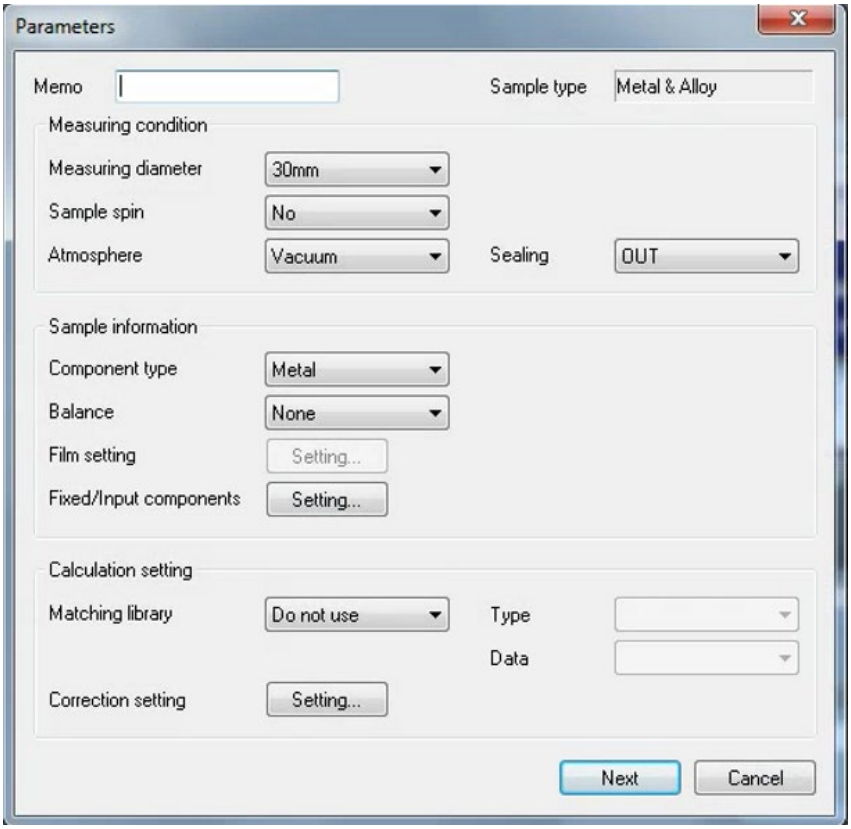
a) Click Next button above top open the Sample Preparation for Metal & Alloy window below. **For powder sample** that has been sandwiched between prolene



films, check **Used** box and select **Prolene** in the dropdown menu. **For solid samples**, leave the **Used** box unchecked.



4) Click the Next button above top open the Parameters window below:



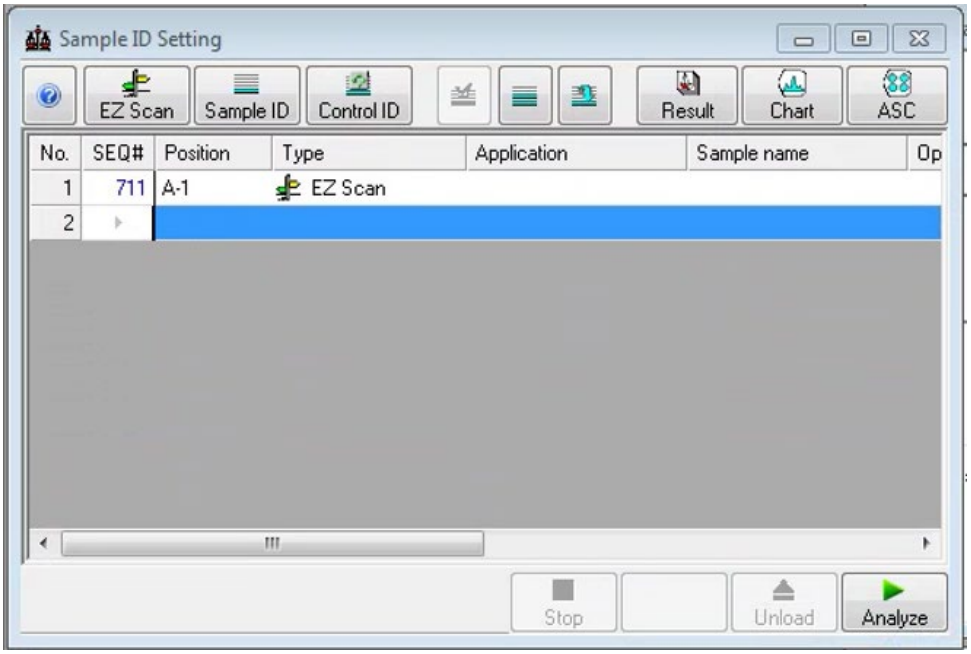
a) **Memo:** leave it blank.
b) **Measuring diameter**: **20 mm** if using 30mm metal mask on the sample holder; **10 mm** if using 20mm mask to avoid signals from the metal cover. Make sure your samples are larger than the metal mask openings.
c) **Atmosphere:** Vacuum.
d) **Sealing**: OUT (for solid samples).
e) **Component type**: Metal.

f) **Balance**: None. Unless there are possible impurities to choose from.
g) **Machine library:** Do not use. Unless created before from standards.

5) Click **Next** button.
6) In the Check **Measuring Condition window**:
   a) Double click each row top open the **Scan Condition** for specific element.
   b) If an element has a small concentration/mass %, choose the **Step** of 0.010 degrees and change the **Speed** to 1-10 degrees/minute.
   c) Click blue FA labeled row to open the Fixed Angle Condition for specific element. For small concentration elements, choose larger numbers for **Time Peak** and **Background**.
7) Click **Next** button.
8) In the **Output Information** window:
   a) **Chart output**: Yes.
   b) **Peak list**: Yes.
   c) **SQX result**: Yes.
   d) **Smoothing**: keep default values unless further smoothing of less smoothing is required on collected spectral peaks.
   e) Check the box in front of the **Output detection limit for undetected element**.
   f) Click **Next** and **Exit** buttons to finish the Application File Setup.

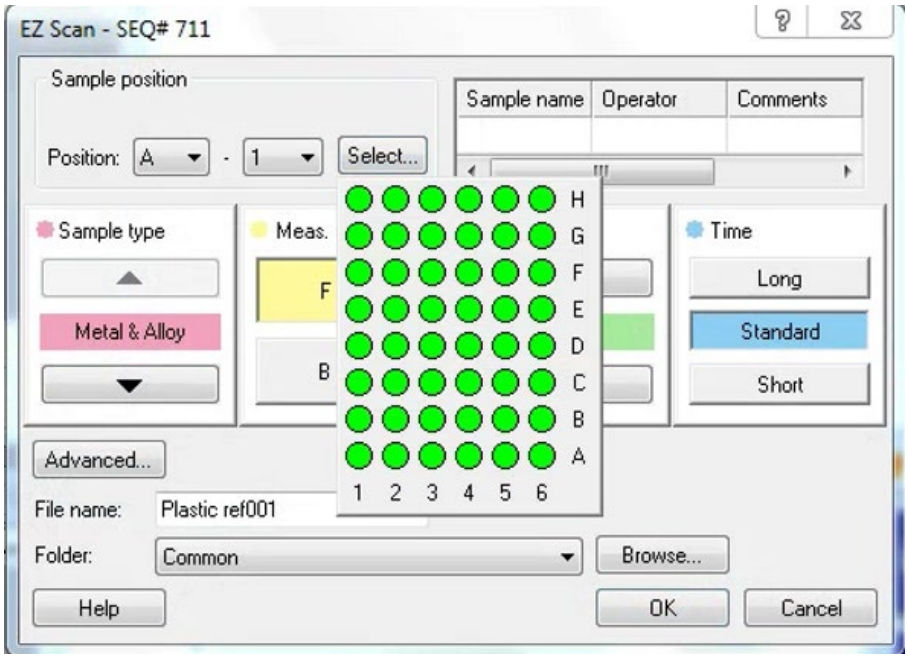
7 Survey Scan

1) Click **EZ Analysis** in the top menu and click **EZ Scan** button in the **Sample ID Setting** window below:



2) Double click the first row inside the table to open the **EZ Scan - SEQ#** window below:

a. **Position**: click the Select button and choose the right sample holder position from 2D array panel below:



b. **Sample type**: leave **Metal & Alloy** unchanged.
c. **Meas. Range**: choose **F to U** or **B to U** based on sample requirements.
d. **Diameter**: choose **20mm** for 30 mm metal mask and **10mm** for 20 mm mask.
e. **Time**: choose **Standard** (~15 mins) or **Long** (~30 mins) if survey scan is more interested than elemental scan for known matrix elements.

f. **File name**: type in the filename specific to each sample.

g. **Folder**: click **Browse** button and choose/create folder for data storage.

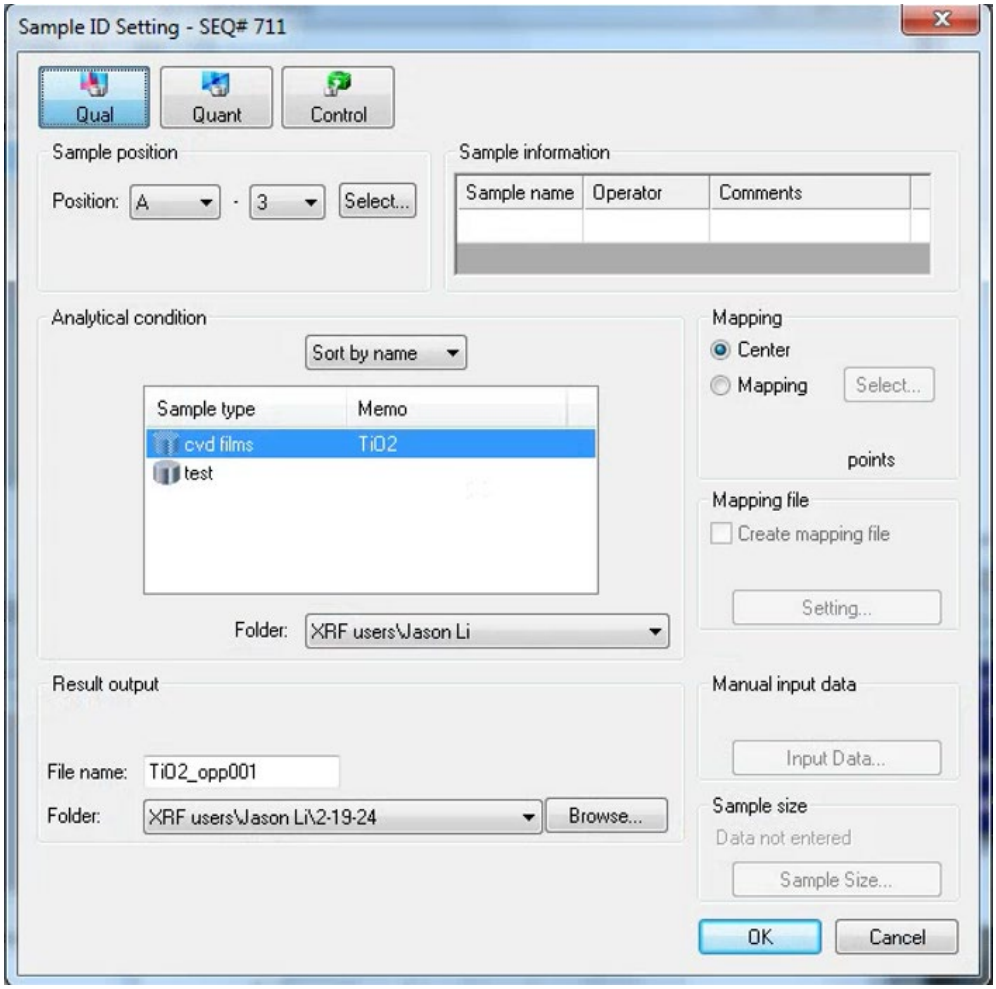
h. Click **OK** to finish EZ Scan setup.

3) Repeat the steps above to finish rest of samples.

4) Click **Analyze** button on the bottom corner of the **Sample ID Setting** window to start scan.

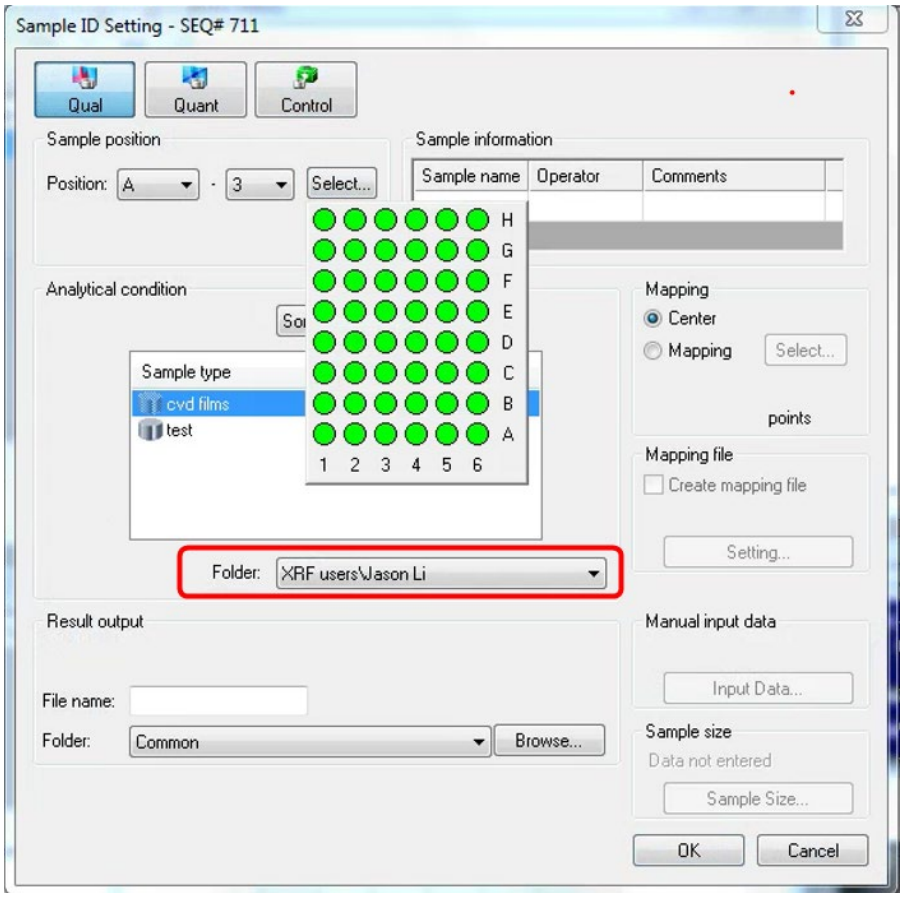
8 Elemental Scan

1) Click sample ID button in the **Sample ID Setting** window below:



a) Click **Qual** button on top of the window.

b) **Position**: click the Select button and choose the right sample holder position from 2D array panel below:

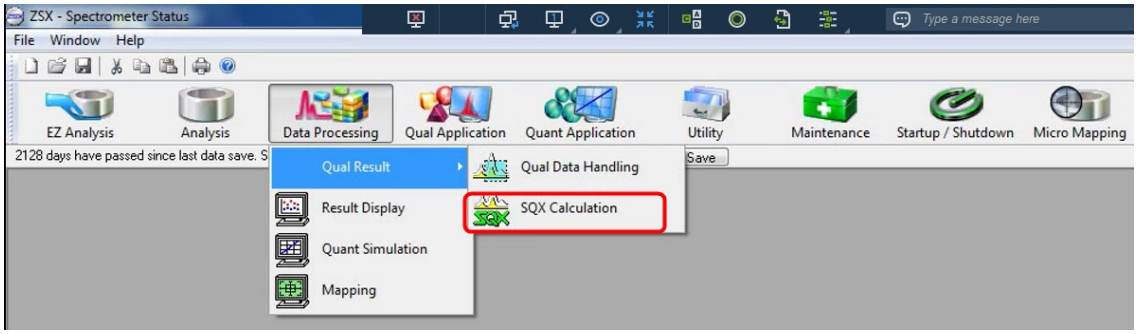


c) **Analytical condition**: click on the **Folder** space highlight above and select the folder where the application file was created; choose the right file to use if more than one was listed.
d) **Mapping**: choose **Center** to skip mapping.
e) **File name**: type in the filename specific to each sample.
f) Click **OK** to finish Elemental Scan setup.

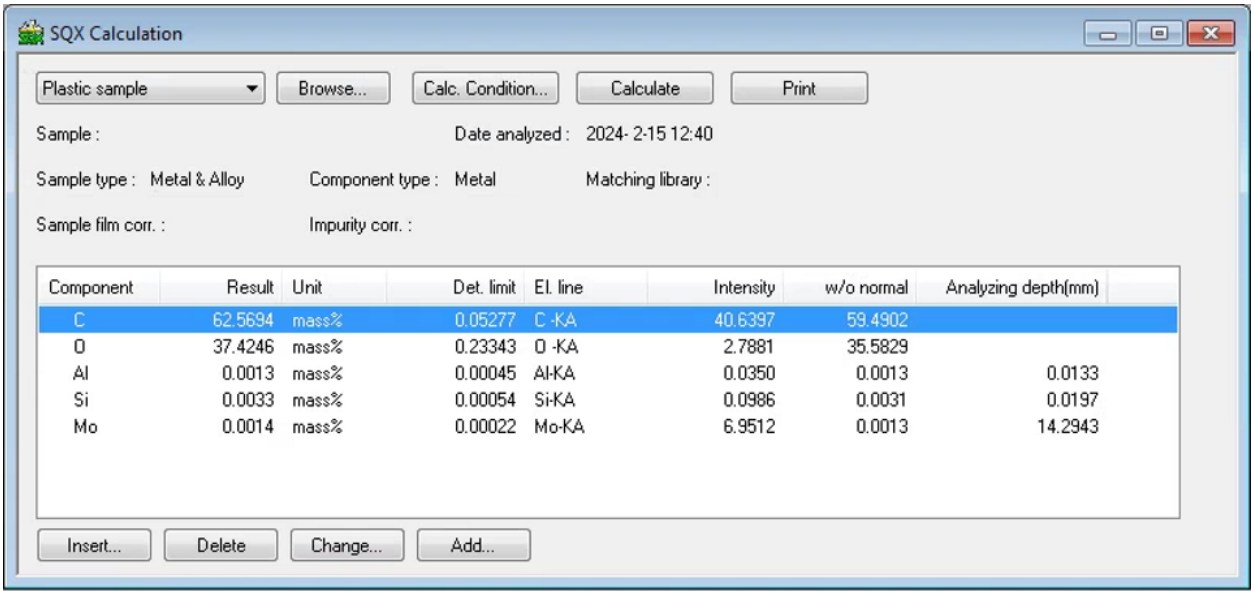
2) Repeat the steps above to finish rest of samples.
3) Click **Analyze** button on the bottom corner of the **Sample ID Setting** window to start scan.

9 Check Mass% Results

1) Close the **Sample ID Setting** window.
2) Click **Data Processing** on the top menu and choose **Qual Result** > **SQX Calculation** as shown below:



3) In the SQX Calculation window below:



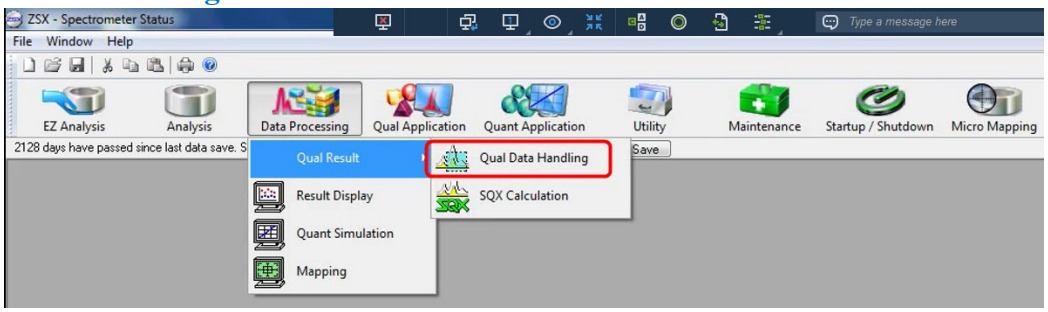
a. Click **Browse** button above to locate the data folder and choose the **File name** in the popup **Data File Selection** window.
b. Click **Calc. Condition** button to change the **Sample type**, **Comp. type** and other settings if needed in the **Calculation Condition** window. Click **OK** to quit the window.
c. Click **Calculate** button to refresh the mass% result after modifying settings in the step above.
d. Click **Insert**, **Delete**, **Change** or **Add** button to modify matrix elements for mass% calculation if needed, and click **Calculate** button to refresh the result.

4) Mass% result table export:
   a. Click **File** > **Data Export…** on the top menu.
   b. In the **Data Export** window, **Browse** to find the right folder. Change the **File name** if needed and click **OK** to finish.

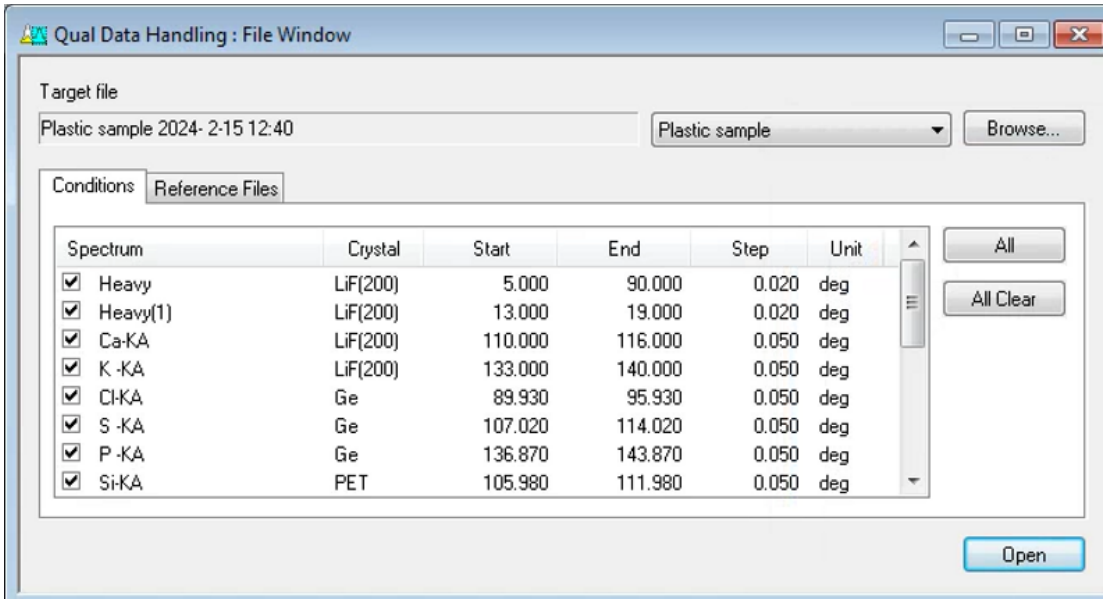
5) Close the **SQX Calculation** window after finish. Click **Yes** on the popup **Confirmation** window.

10 Check spectra

1) Click **Data Processing** on the top menu and choose **Qual Result** > **Qual Data Handling** as shown below:



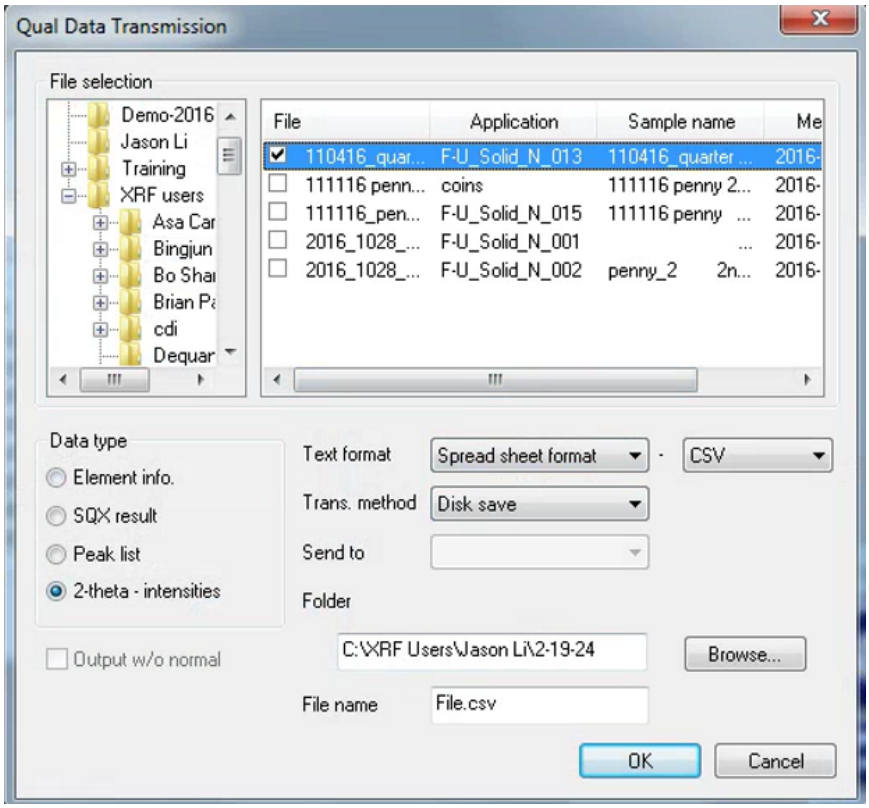
2) Choose the collected data file from the **File Load** window and click **OK** to open the **Qual Data Handling: File Window** as shown below:



3) Select interested elements and click Open button to check spectra.

4) Spectra data export:

   a. Click **File** > **Transfer Qual Data…** on the top menu to open the **Qual Data Transmission** window below:

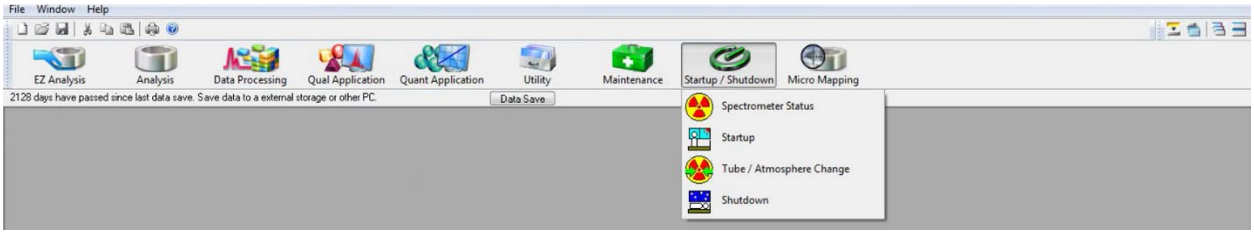


b. Choose the interested data file from the window above.
c. **Data type**: **2-theta-intensities**.
d. **Text format**: **Spread sheet format** and **CSV**.
e. **Trans. Method**: Disk save.
f. **Folder**: choose/create the output folder.
g. **File name**: create output data filename.
h. Click **OK** to finish.

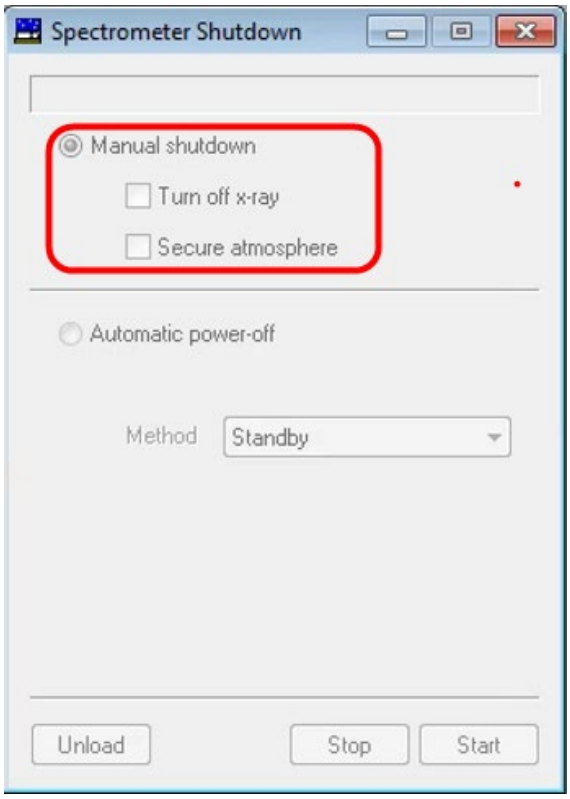
5) Close the **Qual Data Handling: File Window** after finish.

11 Closing Steps

1) Click Startup / Shutdown on the top menu and click Shutdown.



2) In the **Spectrometer Shutdown** window below, select **Manual shutdown,** choose **Turn off x-ray** and click **Start**. If not planning to use the machine within a week, also choose **Secure atmosphere** (to stop vacuum pumping) and click **Start**.



3) Wait for the x-ray power is turned off and the pump has stopped pumping.
4) Keep the ZSX software open.
5) Upload your data to the cloud drive such as Box or One Drive. DO NOT use personal USB drive.
6) Sign off the logbook.
7) Clean the bench sample preparation area, the sample holders and glass slides with clean wipes and isopropanol.
8) Put the sample holder and other tools back into the toolbox.