

XRS-FP2 Combined Standards Calibration Tutorial

Note: This document describes how to do a multiple-standard least-squares (MLSQ) calibration as well as how to implement this calibration with the Scattered-Intensity Ratio method (SIR-FP) in association with an example data set.



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DOCUMENT CHANGE LOG

Date	Person	Pages	Description
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1 MLSQ OVERVIEW

In XRS-FP2 there is a software option where multiple standards can be used for the FP calibration of each element. This secondary least-squares calibration of the FP coefficients (MLSQ) can be done with various models, in order to allow for any variations in the theoretical FP calibration coming from either errors in the physical models or the FP database itself. There is incomplete and inaccurate knowledge of some of the inter-element effects, especially in the low-energy range, or where the standards may not be completely homogeneous.

The MLSQ method will be described below in more detail and can be used with either multi-element type standards or pure-element standards. In fact, there are few limits on the types of standards that can be used, and they can be mixed together in any way. For example, a standard list might include single elements that are in the bulk (infinitely thick) state, or as thin films, and they can also be multi-element standards with all or some of the total range of elements to be calibrated.

Note that MLSQ method is not the same as LSQ method, which uses a simple empirical calibration curve of intensities vs. concentrations, using a least-squares fitting technique without FP.

In a multi-standard calibration **ALL** entered **standards are employed**, along with several models for how to allow for variations of the calibration coefficients with different concentrations and mass thicknesses of each element. This method will be described in more detail below. But typically for this method, you will first calibrate each standard in turn. You can perform these single standard calibrations either with live spectra or with saved spectrum files. Using these calibrations, information from each of these single-standard calibrations is then merged into the defined MLSQ multi-standard model for a final secondary calibration that is the basis of the MLSQ method. The single-standard FP Theoretical Calibration Coefficients (TCCs) from several standards are “blended” together, choosing from several different models to perform a secondary calibration for each element. A least-squares fitting procedure allows the matrix of TCC values for the different standards to be expressed in terms of 1 to 3 different secondary coefficients for each element, which allow the generation of “variable” TCC values over the range of the calibration matrix. See the following sections in this document for more detail.



2 QUICK GUIDE: MLSQ CALIBRATION

The following procedure briefly explains the steps required to do a Multiple-Standard Least-Squares (MLSQ) calibration, where calibration coefficients from all standards are used together. First we are going to calibrate each standard individually, and then merge them together with MLSQ.

- a. In XRS-FP2 “Expert Mode” go to the Application workflow by clicking on the **Application** button from the Home page.
- b. Then click on the **Methods** button and under the “Quant” tab in the R-hand panel select the radio button for **“Single Standard”** in the Calibration Modes section.
- c. Click on the **Standards** button which will take you to the Standards workflow.
- d. Select the **Create** button and enter the sample information for the first standard either by entering components in the Component Table (R-hand panel) or by using the Periodic Table/Compounds tabs. Enter the concentration (“Conc.”) and units for the components in the standard. Note that the “Type” can be left as “Calc” for standards input.
- e. Then click on either the **Import** or **Acquisition** button to either load or acquire a spectrum (file), respectively.
- f. Next select the **Process** button followed by the **Execute** button. This step calculates the elemental intensities and generates the theoretical calibration coefficients (TCC).
- g. Now select the **Save** button, type a filename for the standard and then click the **Execute** button.
- h. Repeat steps (d-g) for **ALL** the individual standards to be used for the multi standard calibration, MLSQ.
- i. Then click on the **Finish** button in the Standards workflow, which will bring you back to the **Methods** section of the Application workflow.
- j. Under the “Quant” tab in the R-hand panel select the radio button for **“Multiple Standards”** in the Calibration Modes section.
- k. Now click on the **Calibration** button, which will take you to the MLSQ multi-standard workflow.



- I. Enter all the individual standard (*.ana) files in the MLSQ Standards Setup Table by double clicking in the “Standard Filename” column and selecting the standard ANA file. Use the down arrow or the plus button to add additional rows for the individual standards.
- m. In the R-hand panel go to the “**Model Setup**” tab and select the type of model to be used. Click on the “Ordinate (Y)” drop-down listbox and select one of the options. The “Intensity” option is commonly selected and can be used with both multilayer thin-film standards and single-layer standards, including bulk samples. Then click on the “Polynomial” drop-down listbox and select either the Linear or Quadratic option. The linear model is particularly suitable for removing some constant bias from a series of measurements. This model performs a linear fit of the measured intensity of the element on the Y-axis against a theoretically calculated intensity based upon the TCC value on the X-axis. Note that the Model number is displayed under the model chart in the R-hand panel. With the linear and intensity options selected the Model number is 9. See the MLSQ Standards Calibration section of the “XRS-FP2 Software User Manual” for more detail.
- n. Click the **Execute** button to perform the merge of all the single-standard TCC values.
- o. You can then click the **Plot** button to display the least squares plot for each element.
- p. To finish, select the **Save Calibration** button to save the complete MLSQ calibration file. This will save 2 files for the MLSQ calibration - a *.cal file for the merged MLSQ calibration and a *.ana file that contains all the final coefficients for future analysis.



3 MLSQ TUTORIAL

The following tutorial along with associated files (*.ana and *.cal) guides the user through an MLSQ FP calibration. This document describes a multipoint calibration using bulk materials with XRS-FP2, but the same principles apply if you are creating a multilayer (thin-film), multipoint calibration (assuming the MTFFF option has been purchased).



MLSQ Calibration:

1. Launch XRS-FP2
2. Go to the **File Manager** button in the top ribbon and select the “RockStd1.ana” file in the R-hand panel (from the MLSQ Analysis Tutorial Folder). Then select Execute to load the analysis file for the first standard. See Figure 1 below.

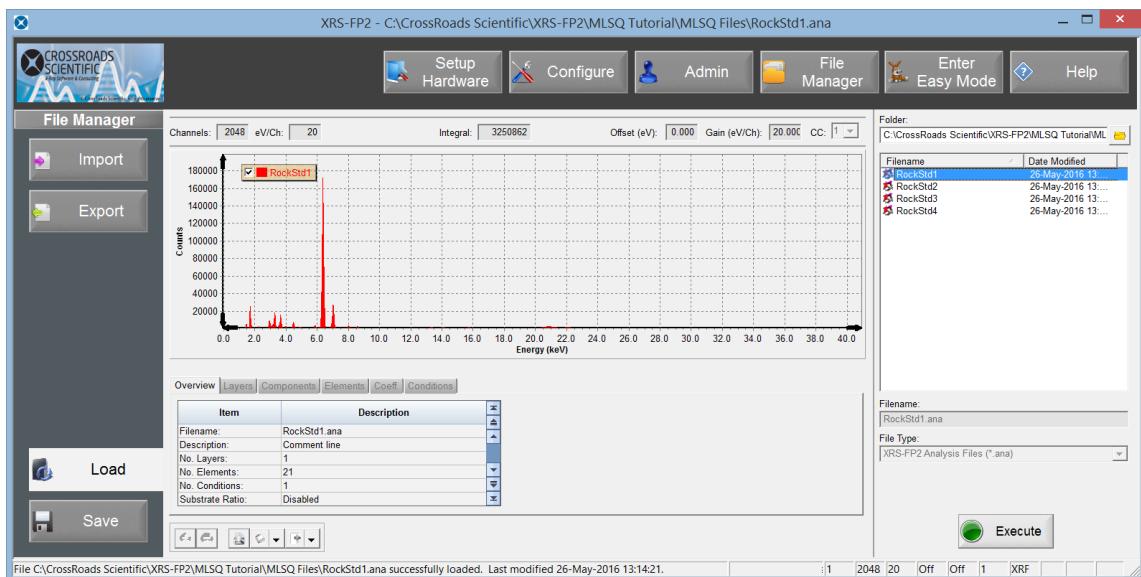


Figure 1. File Manager - Load. Open the first MLSQ standard ANA file, e.g. “RockStd1.ana”.

3. Then select the Home button and then select the **Application** button. Notice that the Component Table and Layer Tables are defined for this standard, including the component names, concentrations, units, calculation method, etc. The component concentrations for the standard add up to 100%, as shown in the Layer Table (i.e. Normalized to 100%).



4. Now select the **Methods** button. In the R-hand panel choose “Single Standard” as the Calibration Mode.
5. Then select the **Standards** button which brings us into the standards workflow.
6. With the **Create** button selected the standard component values (concentrations) can be entered – in the case of this tutorial these are already predefined. Note that the component values are entered in the “Conc” column of the standard Component Table (or they can be selected using the period table), along with the units and calculation method.
7. At this point you have the option to either import or acquire a spectrum for the standard. In the case of this tutorial, the spectrum for each standard has previously been saved to the ANA file for each individual standard. Select the **Import** button - notice that the spectrum for “RockStd1” is displayed in the spectrum view so there is no need to do anything more in this case. However, notice that the R-hand panel allows the user to select a spectrum file to open (i.e. *.MCA file).
8. Now select the **Process** button. Then click on the **Execute** button in the R-hand panel. This will process the spectrum and generate FP Theoretical Calibration Coefficients (TCCs) in one step. Notice that the TCC values are displayed under the “Coeff.” Tab. See Figure 2 below.

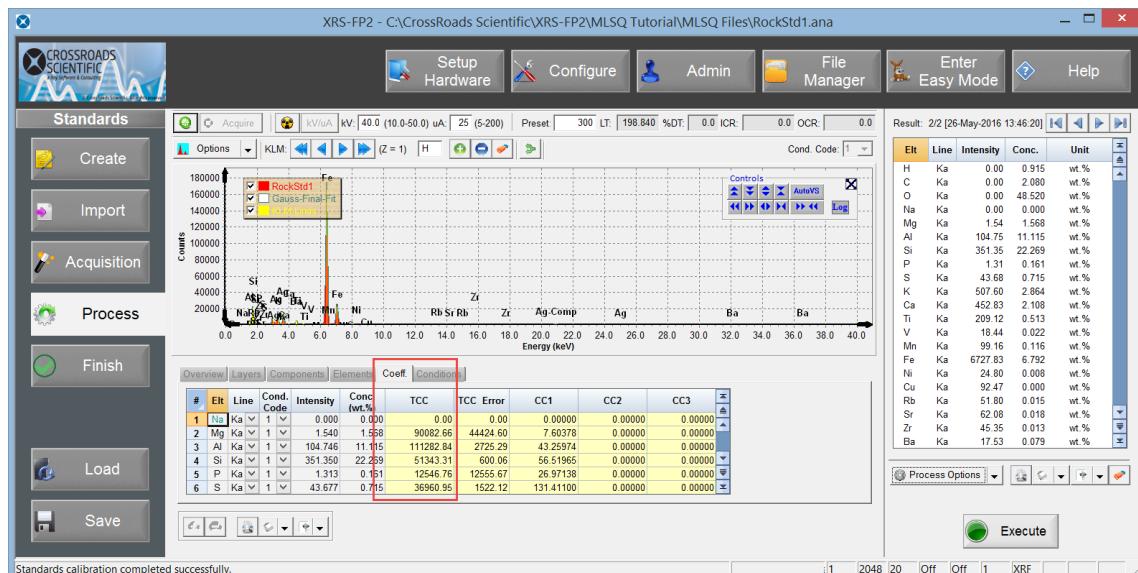


Figure 2. Standards Workflow - Process. The first MLSQ standard spectrum (“RockStd1.mca”) has been processed and TCC values obtained.

9. Save the standard ANA file by selecting the **Save** button. This can be re-saved as “RockStd1.ana” or as an alternate filename of choice.
10. Now select the **Load** button and load the next standard, i.e. select the “RockStd2.ana” file.



11. REPEAT steps 6-10 above for ALL the individual MLSQ standards (single standards) within the “MLSQ Analysis Tutorial” folder. Remember to save each of the four MLSQ standards (i.e. “RockStd1”, “RockStd2”, “RockStd3”, “RockStd4”).

12. Having obtained the 4 ANA files above for the individual MLSQ standards, select the **Finish** button.

13. This will bring up the **Methods** button in the Application workflow shown below in Figure 3. Change the application quant method (calibration mode), in the R-hand panel under the “Quant” tab, to “Multiple Standards” (Fig. 3 below).

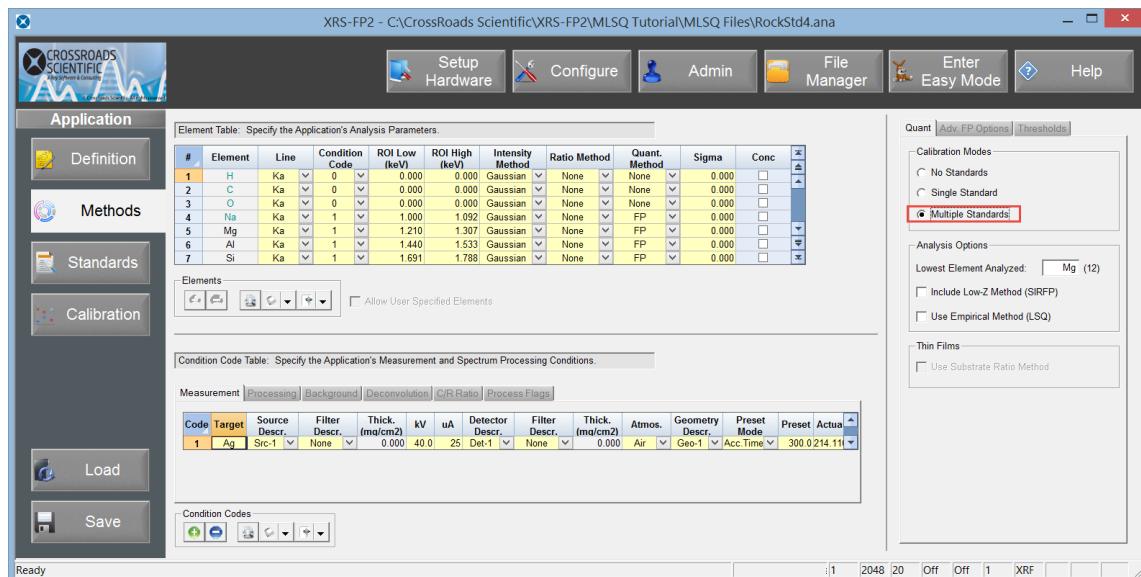


Figure 3. Application Workflow – Multiple Standards Calibration Mode. Select “Multiple Standards” for the “Calibration Mode”.

14. Go to the Calibration workflow by selecting the **Calibration** button (Fig. 4 below).

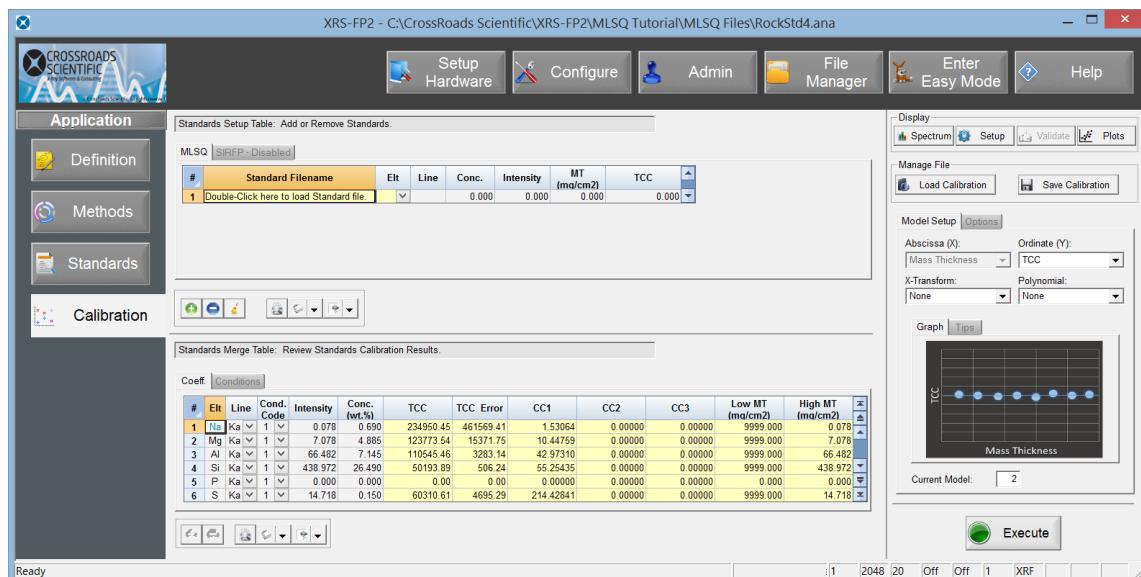


Figure 4. Application Workflow – Calibration. Select the “Calibration” button to move to the multipoint calibration (MLSQ) setup page.



15. Double click in the MLSQ “Standard Filename” column to load the ANA filename to be used for the MLSQ calibration (eg. “RockStd1.ana”). To add additional ANA files (standards) simply use the down-arrow key or select the plus button to add a row. Then load “RockStd2.ana” and subsequently use the same method for the other two standards, “RockStd3.ana” and “RockStd4.ana”. See Figure 5 below.

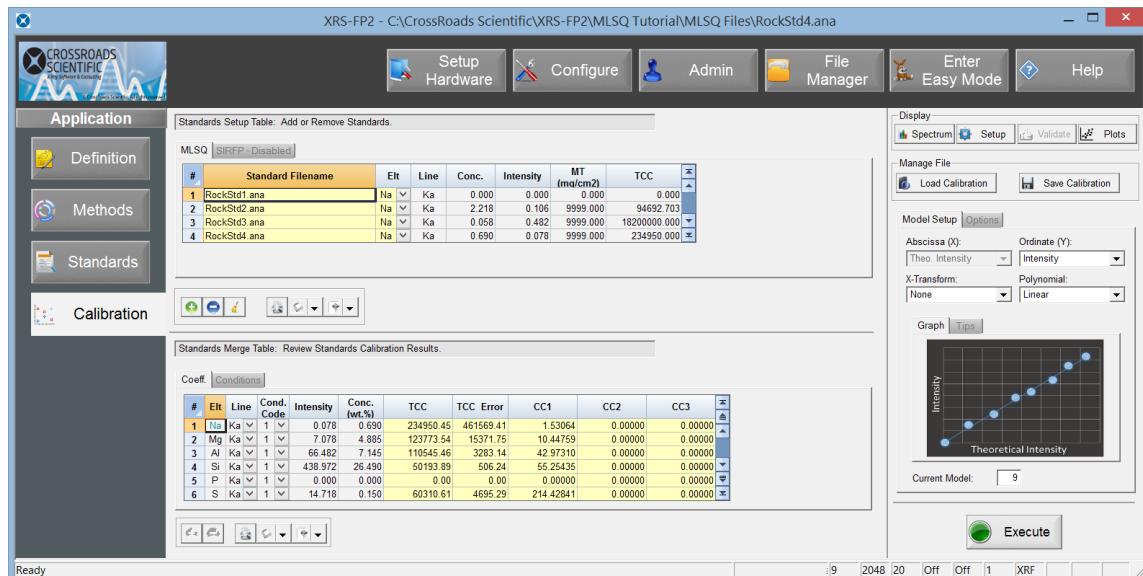


Figure 5. Application Workflow – Calibration. Add all the single standard ANA files to be used for the multipoint calibration (MLSQ).

16. Now go to the “Model Setup” tab in the R-hand panel (Fig. 6 below). Select the type of model to be used by clicking on the “Ordinate (Y)” drop-down menu button. Select the “Intensity” model in the “Ordinate (Y)” column for this method. The least-squares fitting column “Polynomial” can either be “Linear” or quadratic, which has an additional quadratic term. Use “Linear” for this tutorial (Model #9).



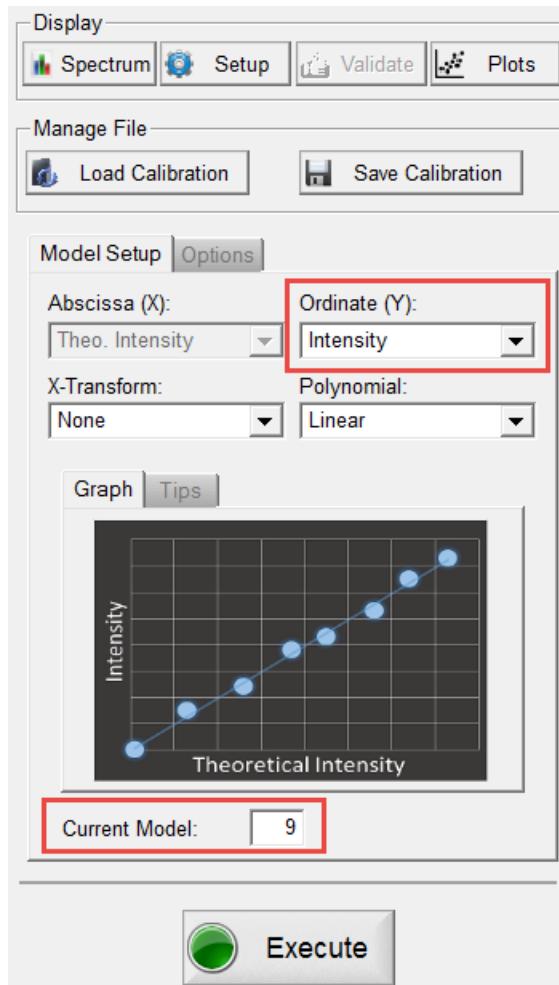


Figure 6. Model Setup for MLSQ Fitting. Under the “Ordinate (Y)” column select the drop-down menu button to select the type of model to be used with this method – “Intensity” (Model 9) in this case.

17. Now click the **Execute** button in the R-hand panel. This will perform a merge of the calibration coefficients (TCCs) for each element in the multipoint calibration.
18. After the MLSQ calibration has been completed, the **Plot** button can be selected to display the least squares plot for each element (see Fig. 7 below). Use the “Previous” and “Next” buttons to display plots for each of the elements. Scroll through all the plots and observe the quality of the fit for each element. After scrolling through all plots, the panel will close or you can use the **Exit** button at any time.



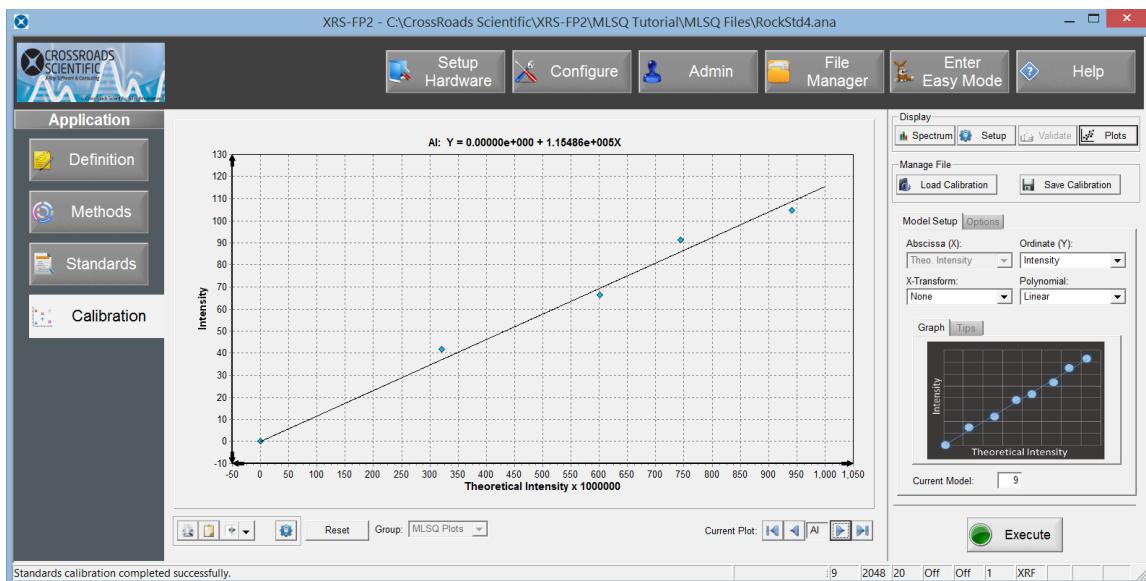


Figure 7. MLSQ Least Squares Plot for AI. Least squares plot for AI.

- Now click the **Save Calibration** button (see Fig. 8 below) to save the full MLSQ calibration. This will save 2 files. The *.cal file contains a list of all the ANA files used in the calibration along with the chosen model. The *.ana file contains all the secondary coefficients necessary to generate TCC values over the range of the calibration standards. For example, the two files saved in this tutorial are the default names: “MultiStdsTutorial.cal” and “MultiStdsTutorial.ana”. Save these files in this location: C:\CrossRoads Scientific\XRS-FP2\MLSQ Analysis Tutorial. Click “OK” to continue and to acknowledge that the two files, “MultiStdsTutorial.cal” and “MultiStdsTutorial.ana” have been saved.

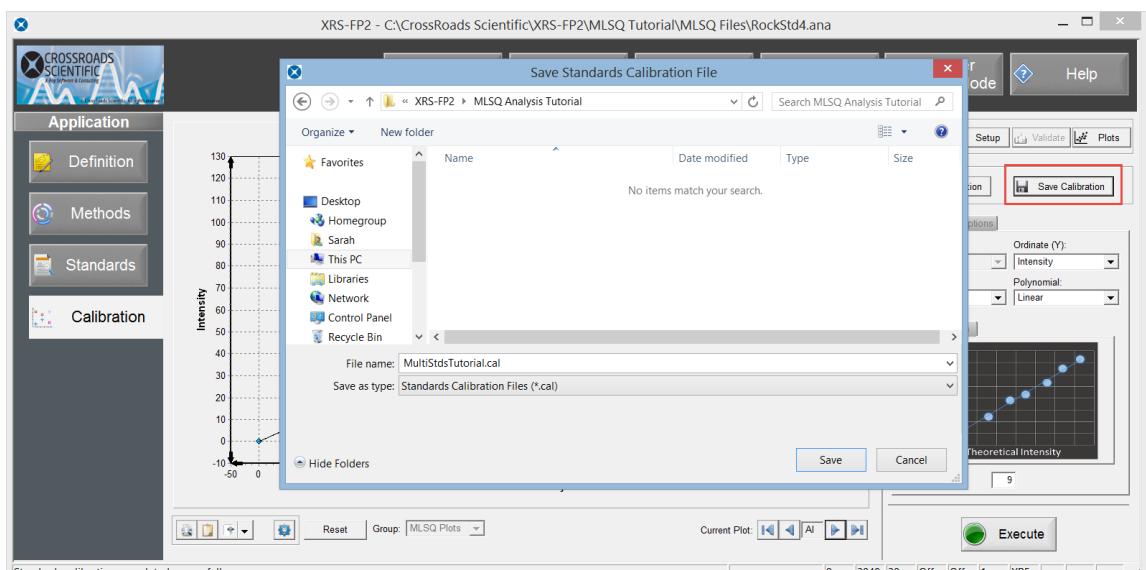


Figure 8. Save MLSQ Calibration. Save the full MLSQ calibration (a *.cal file and an *.ana file) using the “Save Calibration” button.



- 20.** We now have a complete multipoint (MLSQ) calibration. At this point, this method can be used for analysis of an unknown sample. You only need to open the ANA saved from the MLSQ calibration (step 14 above) in order to start analyzing unknown samples. All the information is contained within this single ANA file. For example, you can select the **Load Calibration** button and then open the "MultiStdsTutorial.ana" file. This file not only contains the necessary coefficients, but also the complete sample definition. It is worth noting that any further ANA files saved, with results from the unknown samples, will also contain all the secondary coefficients and may also be used as a "master" file for calibration, just like the file saved above.
- 21.** You may change the sample definition, by adding or deleting components. Deleting merely results in the loss of the coefficients for the elements in that component. Adding components will result in elements that do not have defined TCC coefficients (just like the situation with single-standard calibrations). In this case, TCC values will compute at runtime by interpolation from neighboring elements in the periodic table.
- 22.** Now you can go to the Analyze workflow and load or acquire an "unknown" spectrum for analysis. Then select the **Quantify** button followed by the **Execute** button (R-hand panel). This will perform a full analysis of the "unknown" using the MLSQ method, thus yielding concentrations for the elements in the unknown sample.



4 SIR-FP APPLICATION

The following tutorial along with associated application (*.ana) files guides an XRS-FP2 software user through the SIR-FP calibration and subsequent analysis. This includes:

1. SIR calibration using low-Z standards
2. Integrating the SIR calibration with a MLSQ FP calibration
3. Analysis of an unknown using the complete SIR-FP method

Note: You must choose to integrate the SIR-FP calibration with EITHER the single standard FP calibration OR the MLSQ FP calibration.

The same SIR-FP method is also available for multilayer thin-films (if the MTFFP option has been purchased).

Please note that spectra collected for the SIR and the FP calibrations must be obtained under the same conditions. The same is true for the analysis of unknowns using the SIR-FP calibrations.

This tutorial was created using the analysis of rocks as an example. Secondary standards with varying atomic number (from low Z, such as carbon, to medium Z, such as Ti) will form the basis of the SIR calibration. This calibration will construct a least-squares fit of C/R ratios vs. Z for all the standards. It is recommended that at least 6 standards are used for this method. Following the SIR calibration, an FP calibration will be described for all measured elements. Combining the SIR and FP calibrations will then allow analysis of “unknown” samples.

*It is recommended that you keep two full copies of the “SIR-FP Tutorial” folder on your computer. One will serve as the “working” folder and the other will be the “backup” folder, as some files will be overwritten during the tutorial.



5 SIR-FP TUTORIAL

The following tutorial along with associated files (*.ana and *.cal) guides the user through the Scattered-Intensity Ratio method (SIR-FP).



SIR Calibration with MLSQ Calibration:

1. Launch XRS-FP2.
2. Go to the **File Manager** button in the top ribbon and select the “C:\CrossRoads Scientific\SIR-FP2 Tutorial\SIR Files” folder (or equivalent location), then select the “Initial.ana” file in the R-hand panel (from the SIR Files Folder). Select **Execute** to load this analysis file, which acts as a template for this application. See Figure 1 below.

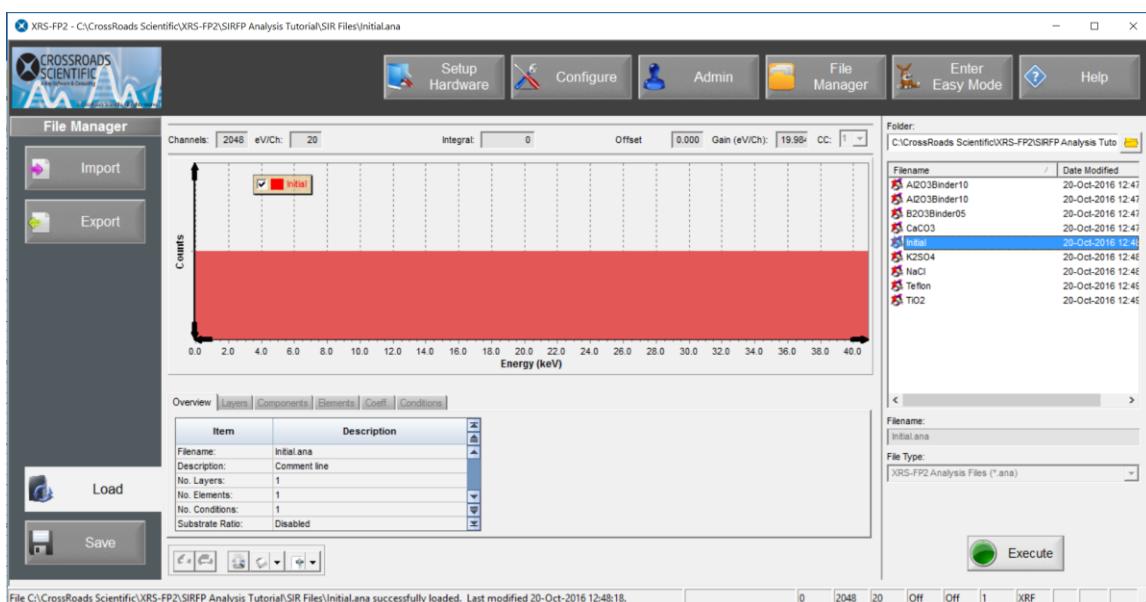


Figure 1. File Manager - Load. Open the SIR template file, e.g. “Initial.ana”.

Using this template file, click on the **Home** button, then the **Application** button, then go to the **Methods** button. Notice that the Calibration Method under the “Quant” tab is set to “Multiple Standards” and the Analysis Options is set to “Include Low-Z Method (SIRFP)”. Although the Component and Elements Tables have no data at this point, the Measurement & Processing information (spectrometer configuration) is already pre-defined and loaded for this application. See Figure 2 below. Note: if there is data in the “Condition Code Table” for the Compton and Rayleigh columns under the “C/R Ratio” tab, **un-check and then re-check the “C/R Ratio” box**. This will



clear the Compton and Rayleigh data in this table and create a “clean sheet” to begin the application (see Fig. 2).

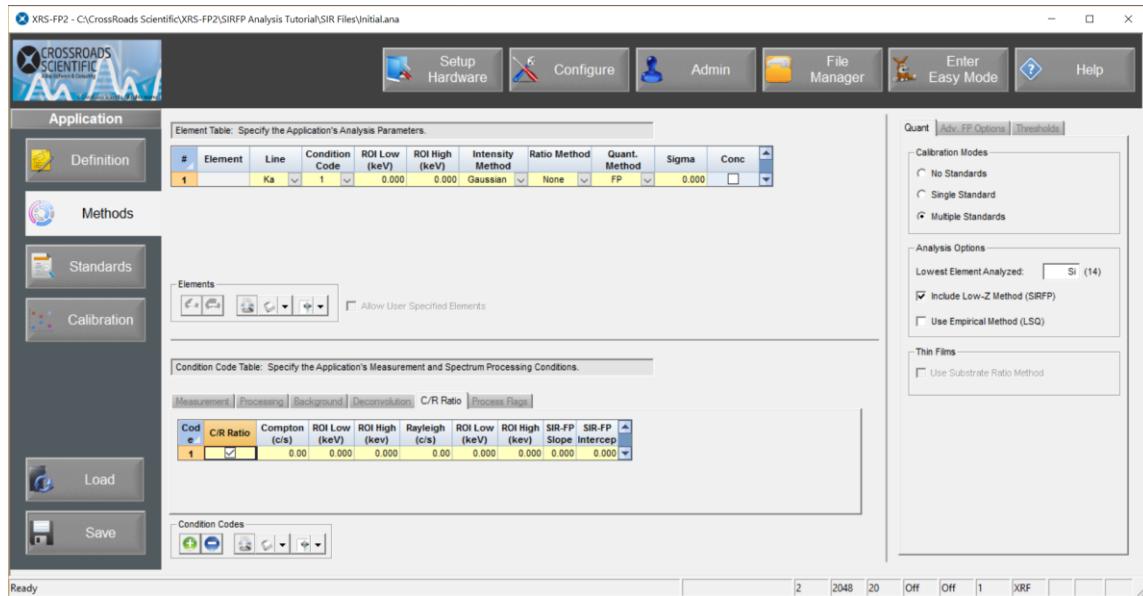


Figure 2. Application - Methods. The application is set for “Multiple Standards” to “Include Low-Z Method (SIRFP)”.

Note that since the Compton and Rayleigh regions of interest (ROIs) are zero, the Gaussian deconvolution for the spectrum processing will automatically assign the regions for the first sample analyzed. For more information on setting and using ROIs see the section “Process Spectrum – Compton Peak” in the “XRS-FP2 User Manual”.

- Now select the **Load** button. In the R-hand panel select the “Al2O3Binder10.ana” file. This is the ANA analysis file for our 1st low-Z standard. See Figure 3 below. Note that the spectra for this tutorial are already adjusted and in calibration.

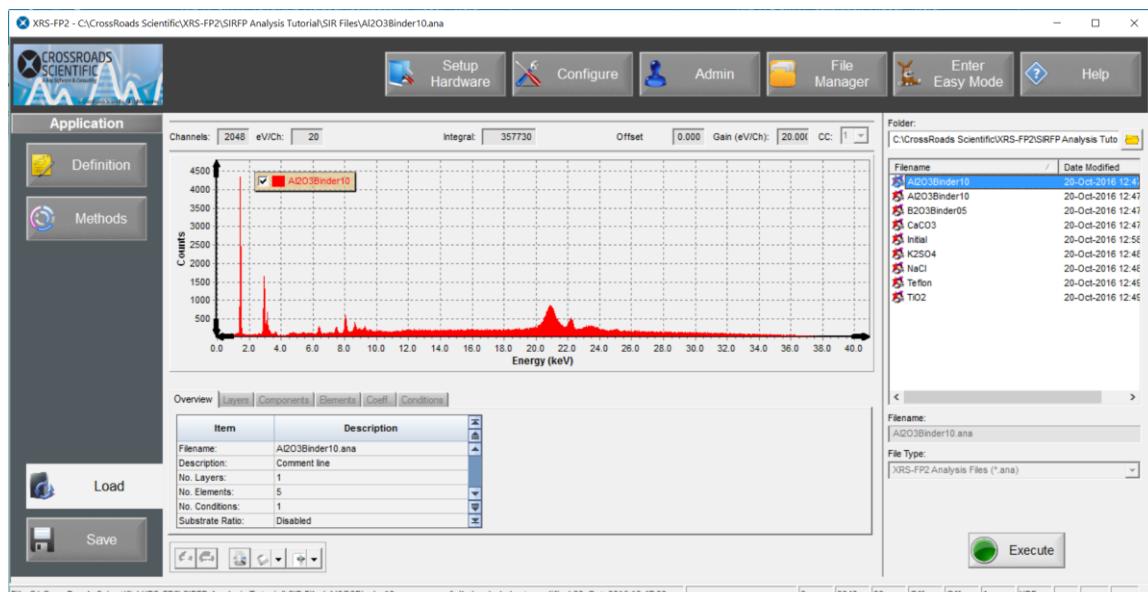


Figure 3. Application - Load. Load the first low-Z standard *.ana file (e.g. Al2O3Binder10.mca).

4. Go to the **Definition** button. Enter the component information in the “Component Table” (see Fig. 4). For this first low-Z standard (“Al2O3Binder10”) enter the following components: “Al2O3” then click “enter” (which will fill the individual elements in the “Element Table”), then “H74C37ON” and “enter”. Set the “Concentration” to 90 and 10 wt.%, respectively.

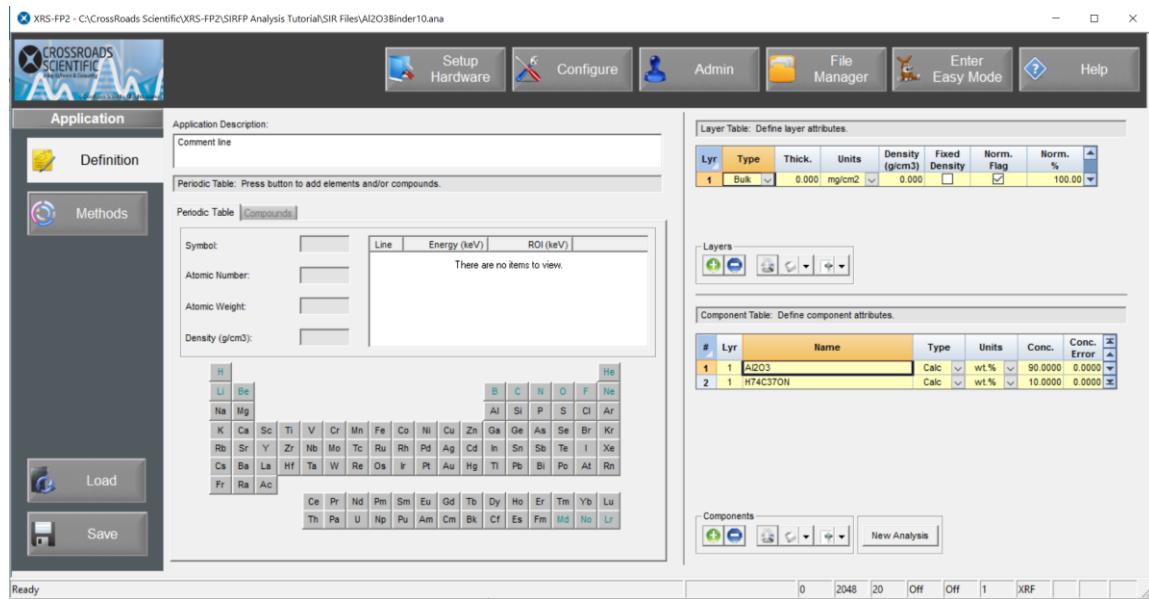


Figure 4. Application - Definition. Note the component information of the low-Z standard is entered in the “Component Table” and the thickness is set to “Bulk” and “Normalized” to 100% in the “Layer Table”.

Note that the Layer “Type” is set to “Bulk” in the “Thickness Information” table (which is mandatory for use with an SIR-FP calibration) and it is “Normalized” to a “Total” of 100% (see Fig. 4 above).

5. Now select the **Methods** button and navigate to the “C/R Ratio” tab in the “Condition Code Table”. (see Fig. 5 below). Note that the **“C/R Ratio” checkbox is selected**. This must be selected for use with the SIR-FP calibration.



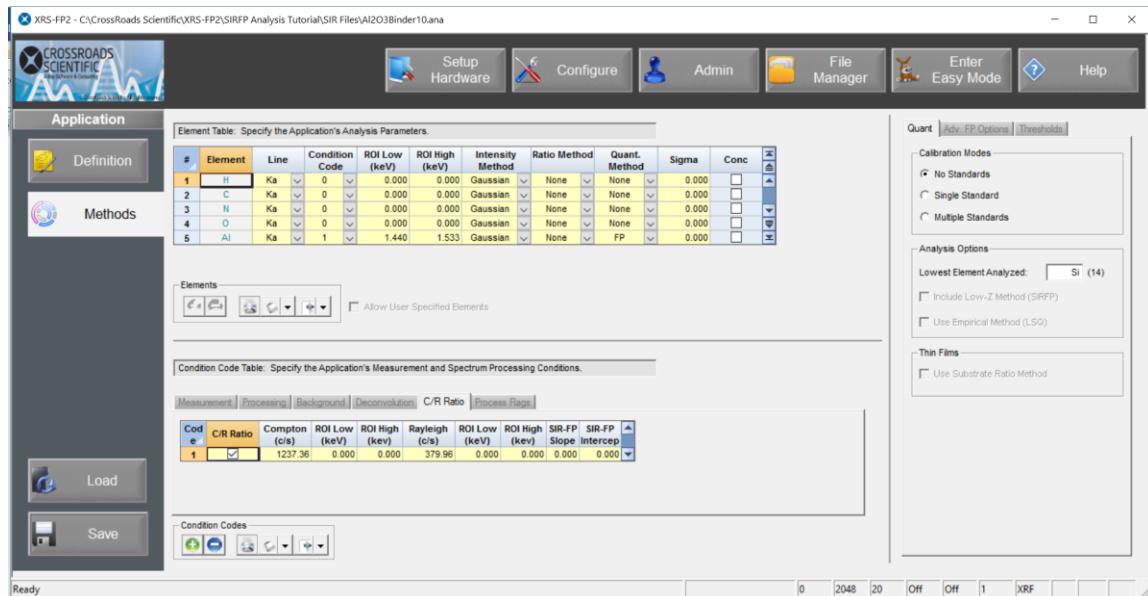


Figure 5. Application - Methods. Note that the “C/R Ratio” box is selected under the “C/R Ratio” tab.

Note: All processing conditions must be the **SAME** for all files used in the SIR-FP calibration. That is, they must all have the same ROI high and low values.

6. Select the **Home** button and then the **Analyze** button. Then click on the **Quantify** button. From the Quantify Options button in the R-hand panel select the **Process All** option (which performs the full spectrum processing in one step). Notice that there is now data for the Compton (c/s), Compton ROI (keV), Rayleigh (c/s) and Rayleigh ROI (keV) columns in the “C/R Ratio” tab under the “Conditions” tab (see Fig. 6). Note that the Compton (c/s) and Rayleigh (c/s) values may vary due to the background removal during processing.

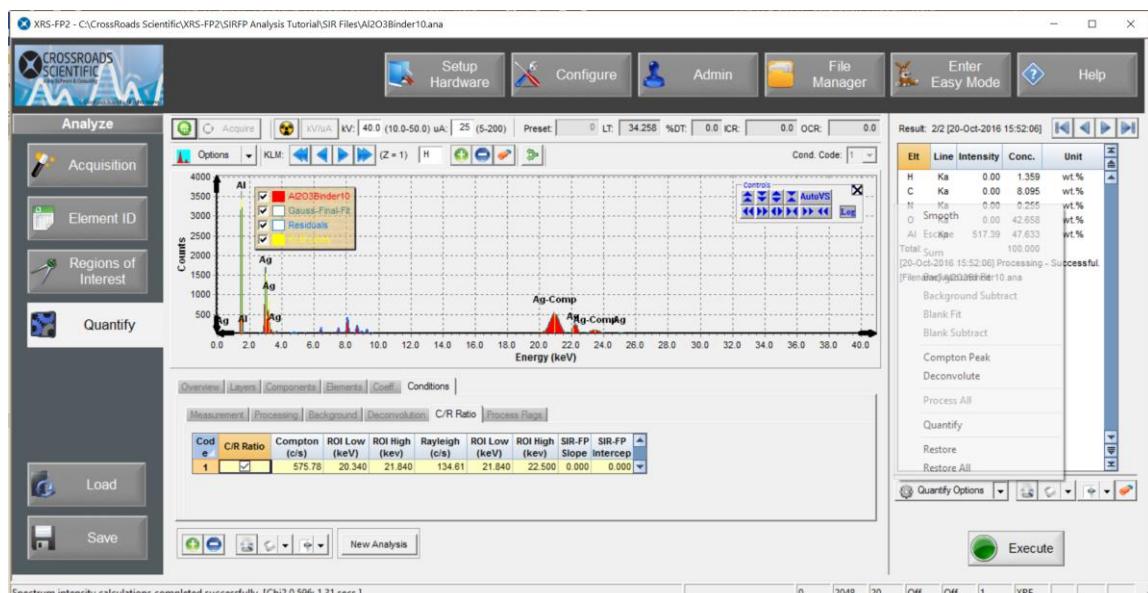


Figure 6. Analyze - Quantify. Data is shown under the Compton and Rayleigh columns after spectrum processing.



7. Now save the ANA file by selecting the Save button followed by the Execute button. This can be saved as “Al2O3Binder10.ana” or as an alternate filename of choice.
8. **REPEAT steps 3 - 7 above for ALL the low-Z standards** within the SIR folder. Check that the layer information is normalized to 100% and the C/R Ratio in the Conditions table is checked. Remember to enter the C/R information in the “C/R Ratio” tab under the “Conditions” tab (see Fig. 6 above). Make sure to use the SAME Compton and Rayleigh ROIs for each standard. If necessary, enter the high and low ROI (keV) values for consistency. After processing the spectrum, remember to save the ANA file before moving on to the next standard! The following figures (Fig. 7-10) show screen shots of the XRS-FP2 Methods view for each of the remaining SIR low-Z standards (data shown is after completion of step 8 above for each standard).

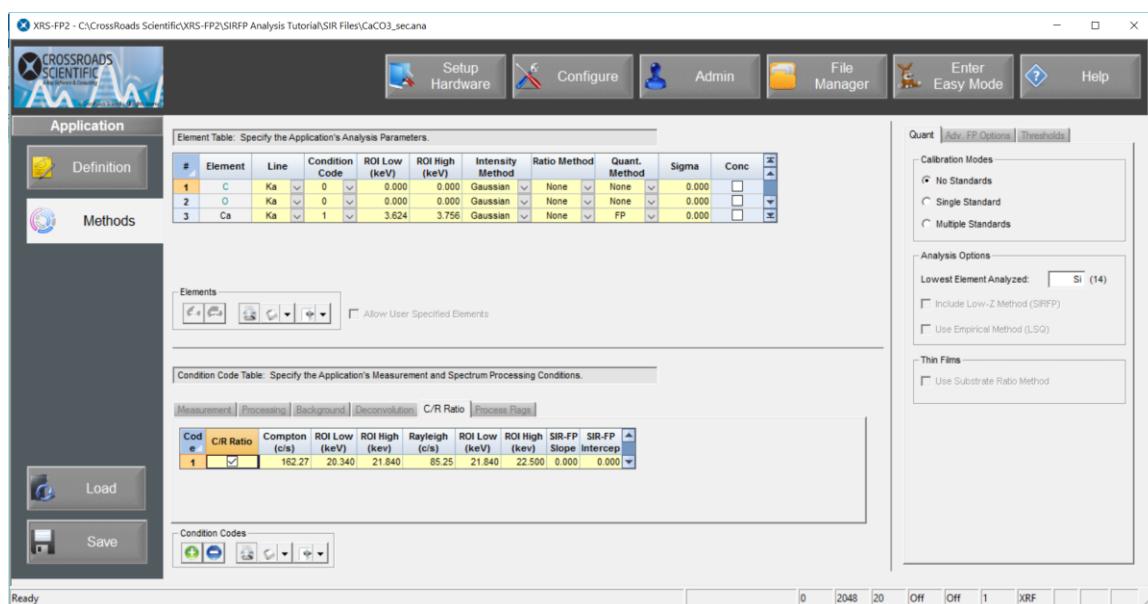


Figure 7. Application - Methods. Data after processing CaCO3.ana. Note that for XRS-FP2 to process the carbon and oxygen elements in this low-Z standard, the compound MUST be entered as “CaC O3” in the “Component Table”, i.e. with a space between the C and the O. Also be sure to enter “O” for oxygen, as the number “0” will not be recognized as the element oxygen.



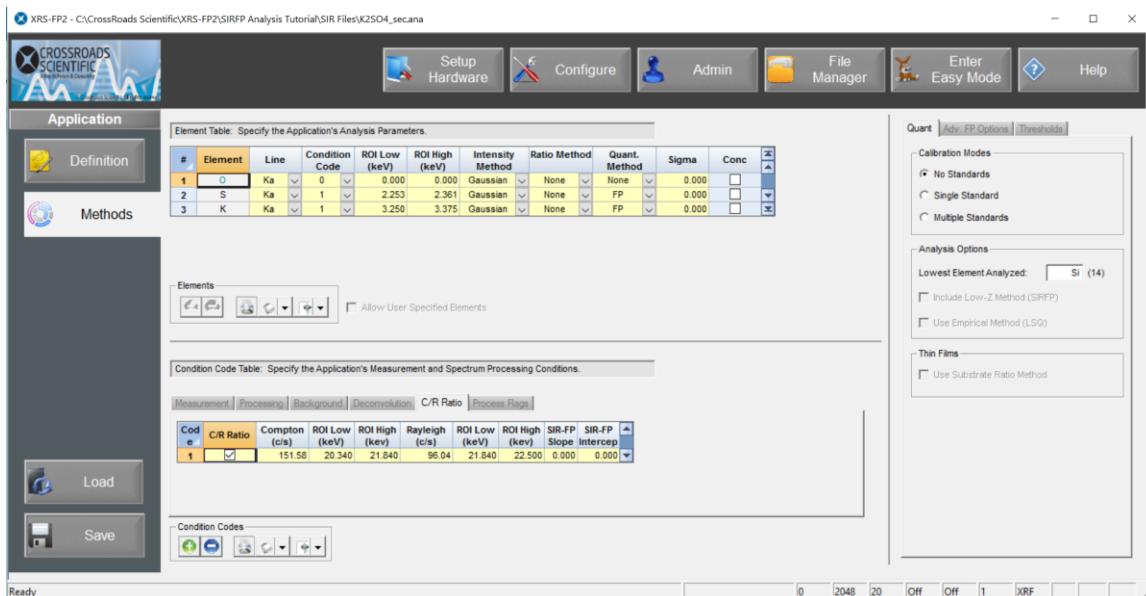


Figure 8. Application - Methods. Data after processing K2SO4.ana.

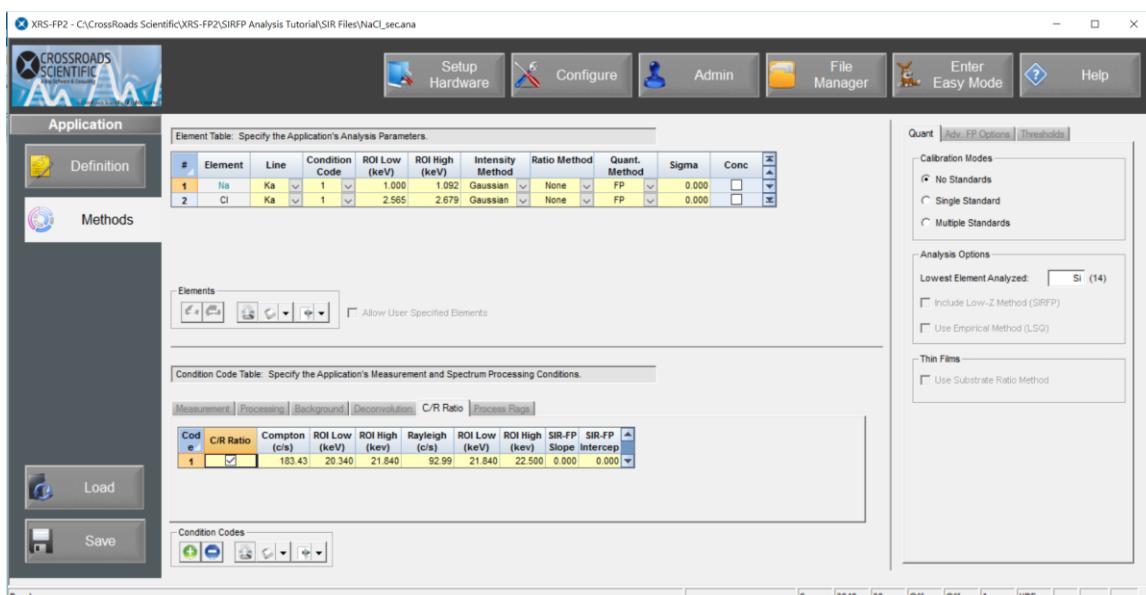


Figure 9. Application - Methods. Data after processing NaCl.ana.



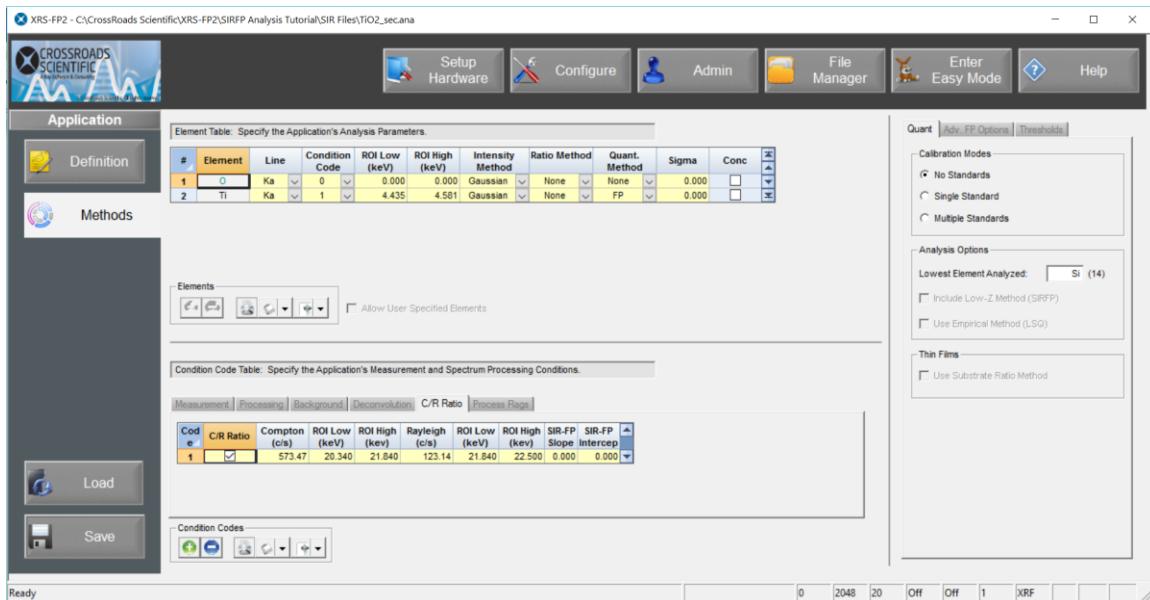


Figure 10. Application - Methods. Data after processing TiO2.mca.

9. Having completed the calibration of the low-Z standards, we will now make the multipoint (MLSQ) calibration as the next step. Select the **Load** button then select the “RockStd1.ana” file in the R-hand panel under the “C:\CrossRoads Scientific\SIR-FP2 Tutorial\MLSQ Files” folder (or equivalent location).
10. Now select the **Methods** button. Set the Calibration Mode under the “Quant” tab to “Multiple Standards” and the Analysis Options to “Include Low-Z Method (SIRFP)” if it is not already set. See Figure 11 below.

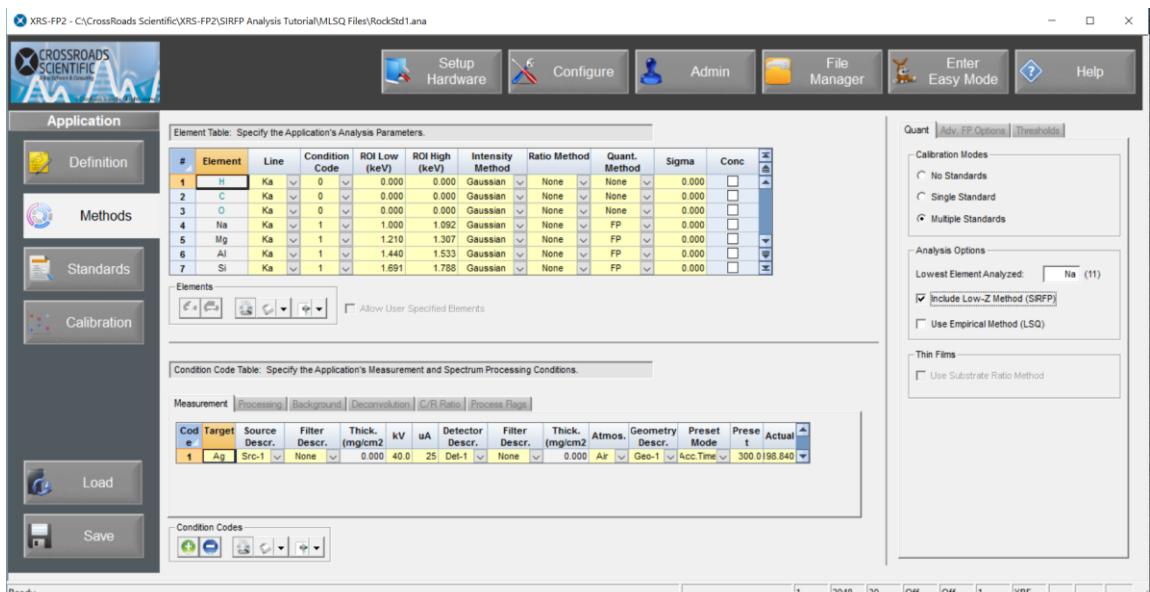


Figure 11. Application - Methods. In the R-hand panel set the Calibration Mode (under the “Quant” tab) to “Multiple Standards” and the Analysis Options to “Include Low-Z Method (SIRFP)”.



11. Then select the **Standards** button which brings us into the standards workflow.
12. With the **Create** button selected the standard component values (concentrations) can be entered – in the case of this tutorial these are already predefined. Note that the component values are entered in the “Conc” column of the standard Component Table (or they can be selected using the period table), along with the units and calculation method.
13. At this point you have the option to either import or acquire a spectrum for the standard. In the case of this tutorial, the spectrum for each standard has previously been saved to the ANA file for each individual standard, so we will simply load the ANA file for each standard. So select the **Load** button and load one of the MLSQ standards, such as “**RockStd1.ana**” file.

Note that if the **Import** button is selected a spectrum is displayed in the spectrum view. The R-hand panel allows the user to select a spectrum file to open (i.e. *.MCA file).

14. Now select the **Process** button. Then click on the **Execute** button in the R-hand panel. This will process the spectrum and generate FP Theoretical Calibration Coefficients (TCCs) in one step. Notice that the TCC values are displayed under the “Coeff.” Tab. See Figure 12 below.

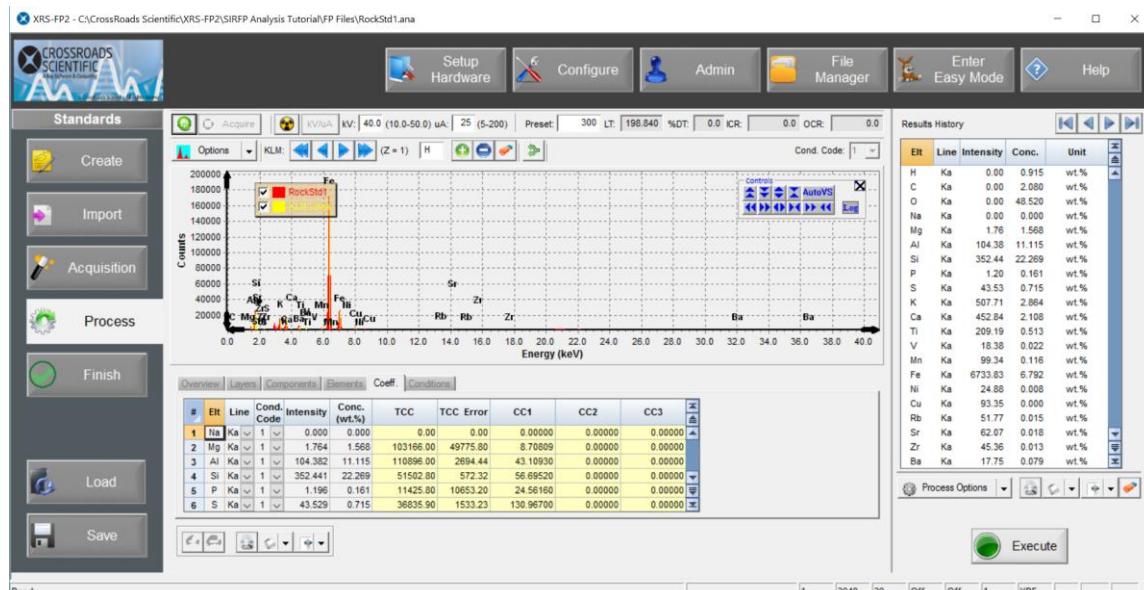


Figure 12. Standards Workflow - Process. The standard spectrum (“RockStd1.mca”) has been processed and TCC values obtained.

15. Save the standard ANA file by selecting the **Save** button. This can be re-saved as “RockStd1.ana” or as an alternate filename of choice.
16. Now select the **Load** button and load the next standard, i.e. select the “**RockStd2.ana**” file.



17. REPEAT steps 13-15 above for ALL the individual MLSQ standards (single standards) within the “MLSQ Analysis Tutorial” folder. Remember to save each of the four MLSQ standards (i.e. “RockStd1”, “RockStd2”, “RockStd3”, “RockStd4”).

18. Having obtained the 4 ANA files above for the individual MLSQ standards, select the **Finish** button.

19. Double check that the Calibration Mode under the “Quant” tab to “Multiple Standards” and the Analysis Options to “Include Low-Z Method (SIRFP)” if it is not already set. Also change the Quant Method to “SIRFP” for ALL elements. See Figure 13 below. Note: select the **Save** button and save the ANA file if any changes are made.

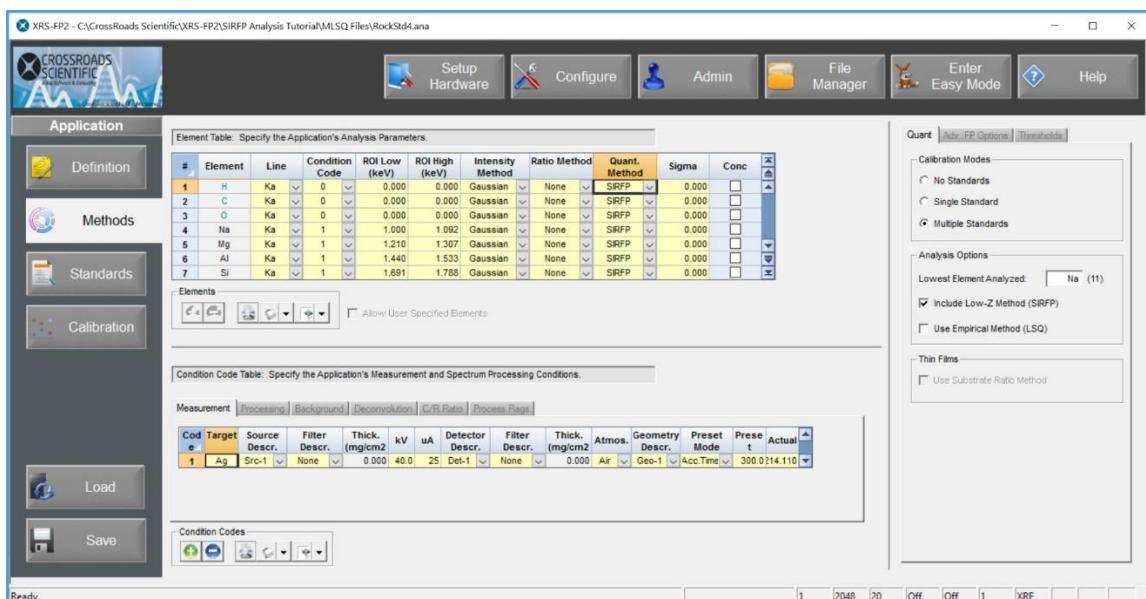


Figure 13. Quant Method. Set the Quant Method to “SIRFP” for all elements.

20. Then select the **Calibration** button in the Application workflow shown below in Figure 14. This is where we will merge the SIR-FP calibration from above with the multiple standard (MLSQ) calibration.



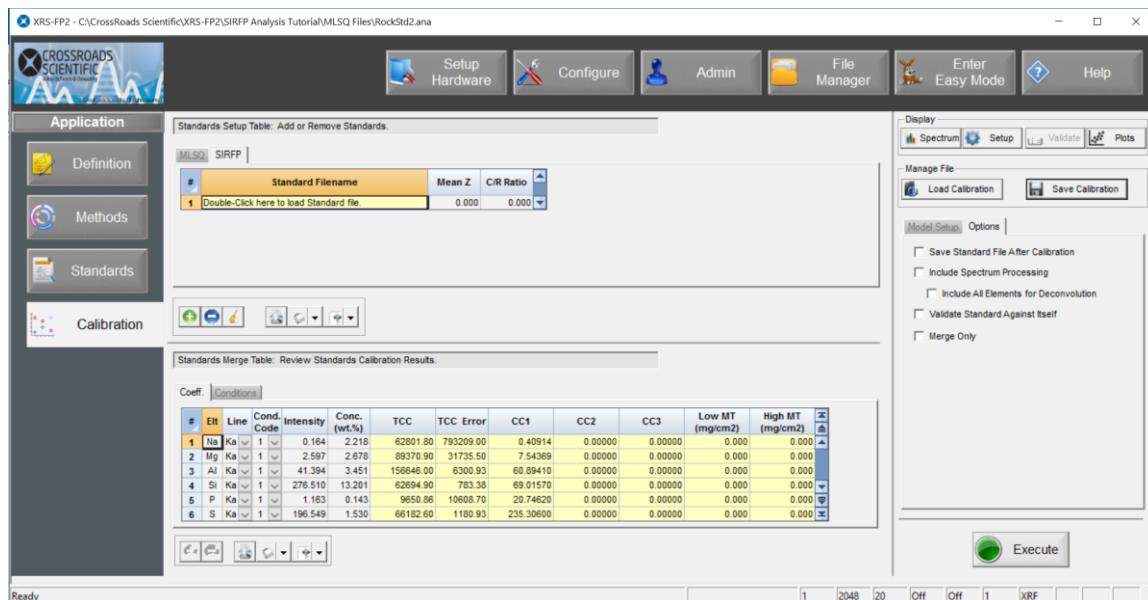


Figure 14. Application Workflow – Calibration. Select the “Calibration” button.

21. Under the “SIRFP” tab, double click in the SIRFP “Standard Filename” column to load the ANA filename to be used for the SIRFP calibration (e.g. “Al203Binder10.ana”). To add additional ANA files (standards) simply use the down-arrow key or select the plus button to add a row. Then load “CaCO3.ana” and subsequently use the same method for the rest of the SIRFP standards (Fig. 15 below).

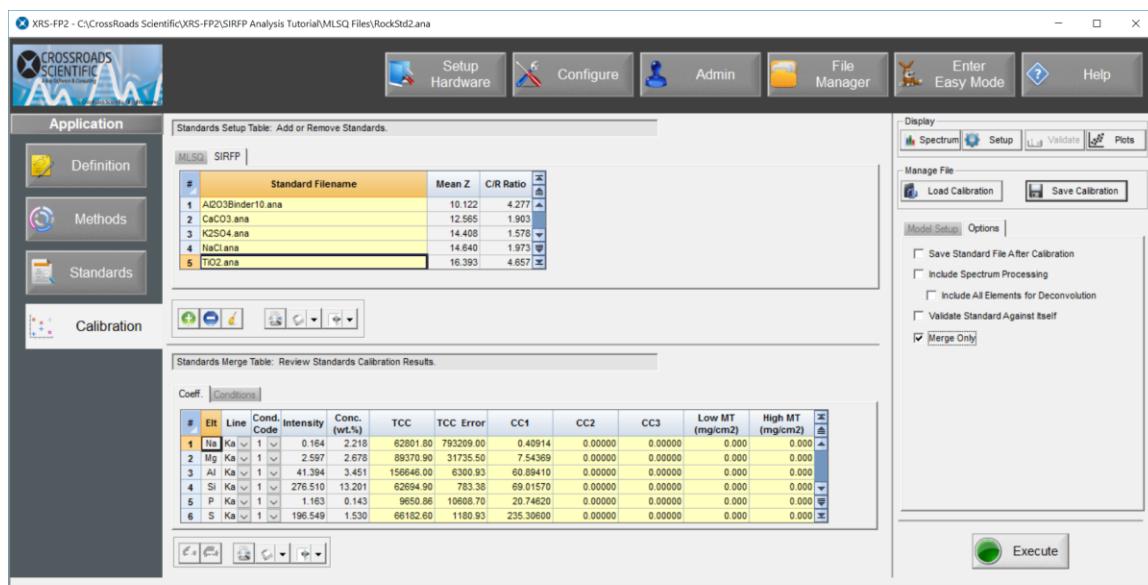


Figure 15. Application Workflow – Calibration. Add all the SIRFP standard ANA files to be used for the low-Z calibration.

22. Then go to the “MLSQ” tab and double click in the MLSQ “Standard Filename” column to load the ANA filenames to be used for the MLSQ calibration (e.g. “RockStd1.ana,” “RockStd2.ana,” etc.). See Figure 16 below.



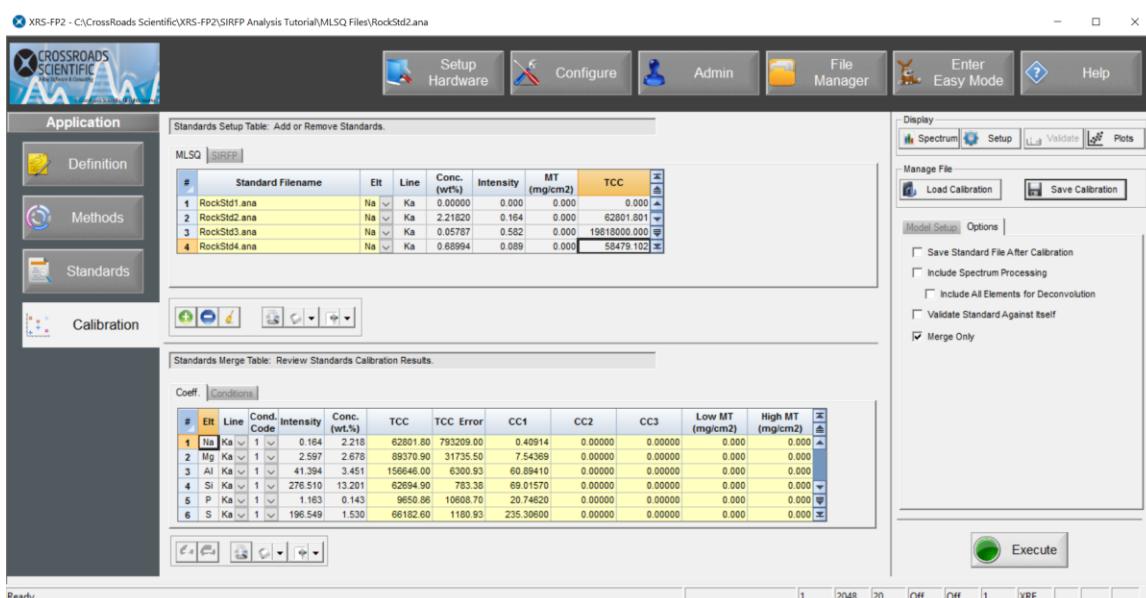


Figure 16. Application Workflow – Calibration. Add all the MLSQ standard ANA files to be used for the multipoint calibration.

23. In the R-hand panel select the “Options” tab and then the box for “Merge Only”. There are several options here; however, since we have already processed and collected the calibration information for each individual standard file, there is no need to re-process the individual standards. Although the user has the option to do this if they desire.

24. Now go to the “Model Setup” tab in the R-hand panel (Fig. 17 below). Select the type of model to be used by clicking on the “Ordinate (Y)” drop-down menu button. Select the “Intensity” model in the “Ordinate (Y)” column for this method. The least-squares fitting column “Polynomial” can either be “Linear” or quadratic, which has an additional quadratic term. Use “Linear” for this tutorial (Model #9).



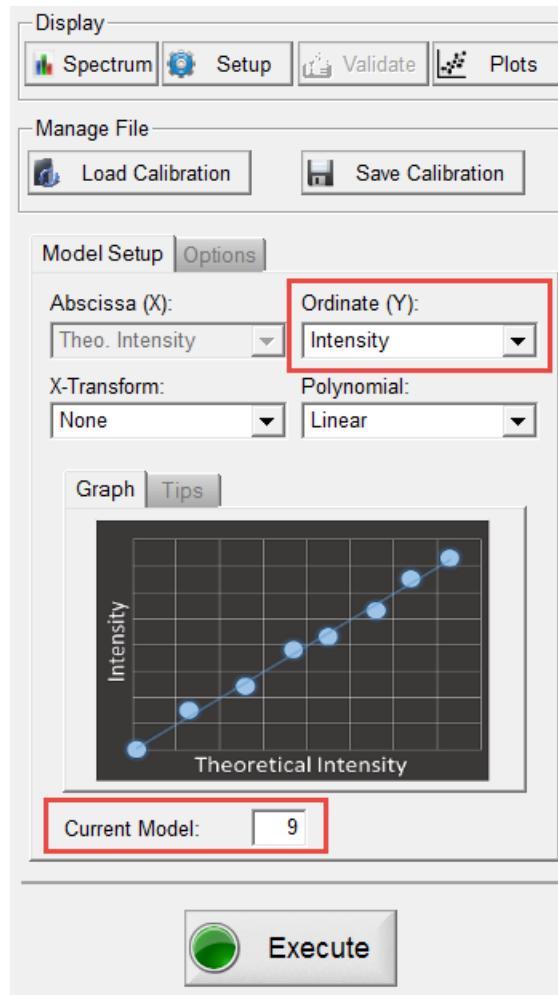


Figure 17. Model Setup for MLSQ Fitting. Under the “Ordinate (Y)” column select the drop-down menu button to select the type of model to be used with this method – “Intensity” (Model 9) in this case.

25. Now click the **Execute** button in the R-hand panel. This will perform a merge of the calibration coefficients (TCCs) for each element in the multipoint calibration along with a merge of the SIRFP information (Fig. 18 below).



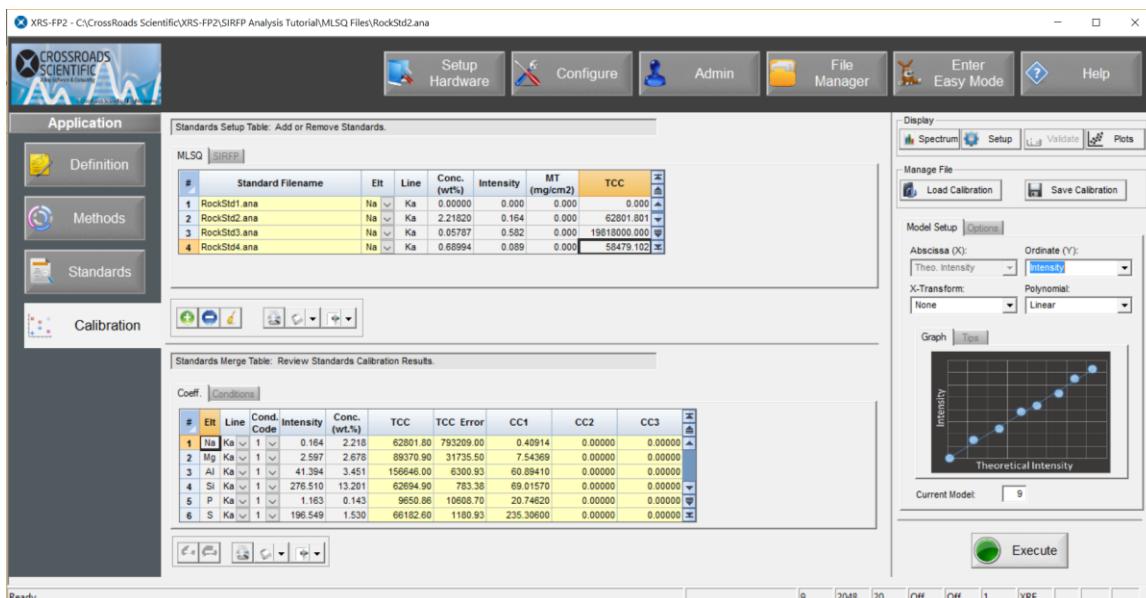


Figure 18. Application Workflow – Calibration. Add all the MLSQ standard ANA files to be used for the multipoint calibration. Then select the model (i.e. Model #9 here). Then select the Execute button to perform a merge of the MLSQ and SIRFP standards.

26. After the SIRFP - MLSQ calibration has been completed, the **Plot** button can be selected to display the SIRFP plot as well as the least squares plot for each element (see Fig. 19 below). Use the “Previous” and “Next” buttons to display plots for each of the elements. Scroll through all the plots and observe the quality of the fit for each element. After scrolling through all plots, the panel will close or you can use the **Exit** button at any time.

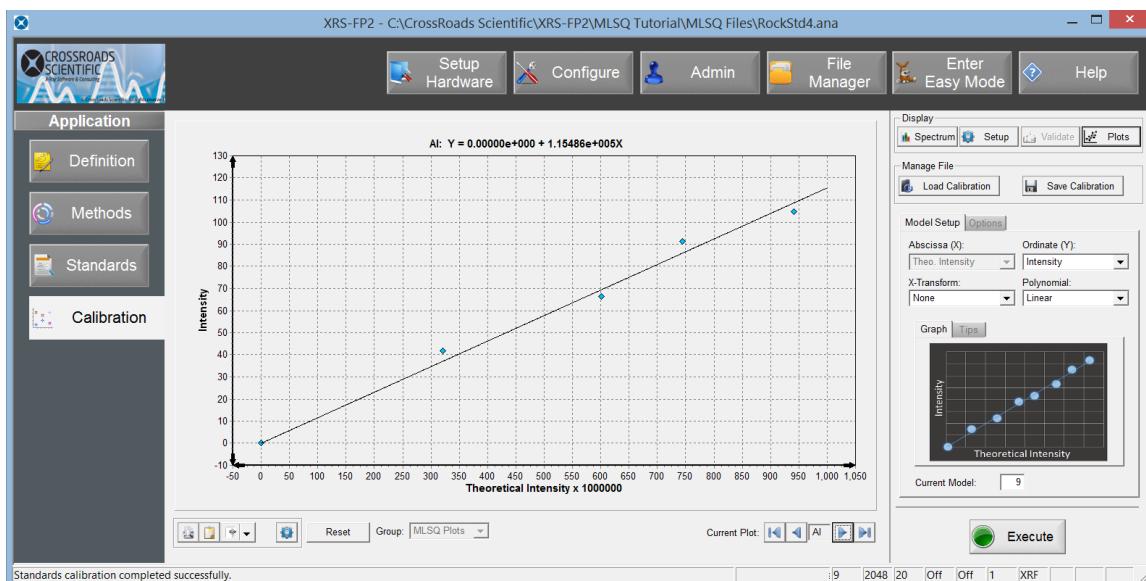


Figure 19. MLSQ Least Squares Plot for Al. Least squares plot for Al.



- 27.** Now click the **Save Calibration** button (see Fig. 20 below) to save the full SIRFP - MLSQ calibration. This will save 2 files. The *.cal file contains a list of all the ANA files used in the calibration along with the chosen model. The *.ana file contains all the secondary coefficients necessary to generate TCC values over the range of the calibration standards along with the slope and intercept information. For example, the two files saved in this tutorial are the default names: "SIRFP-MLSQ_Tutorial.cal" and "SIRFP-MLSQ_Tutorial.ana". Save these files in this location: C:\CrossRoads Scientific\XRS-FP2\SIRFP Analysis Tutorial. Click "OK" to continue and to acknowledge that the two files, "SIRFP-MLSQ_Tutorial.cal" and "SIRFP-MLSQ_Tutorial.ana" have been saved.

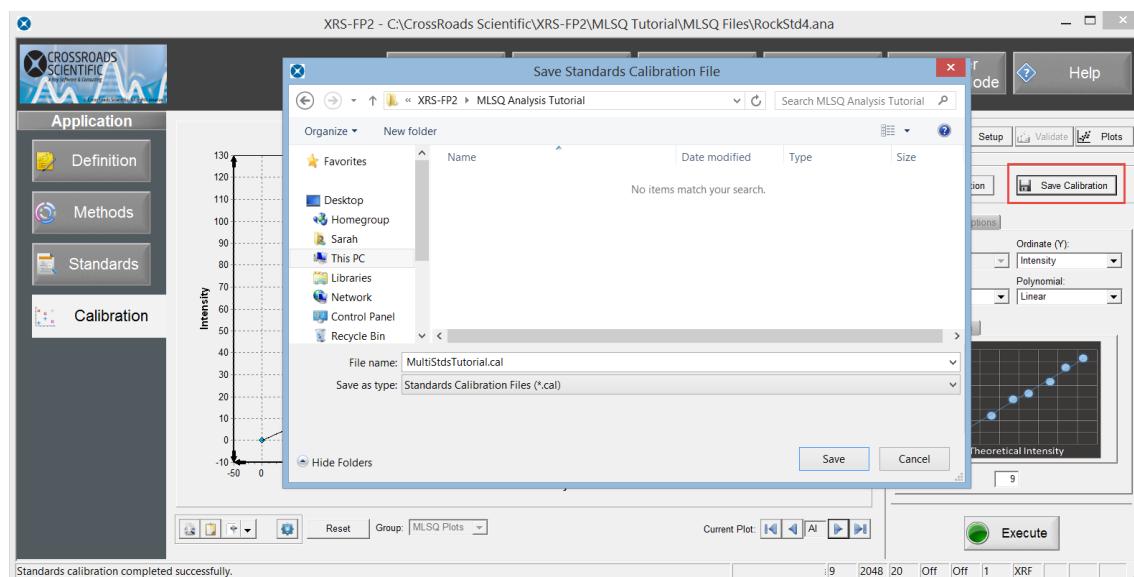


Figure 20. Save MLSQ Calibration. Save the full MLSQ calibration (a *.cal file and an *.ana file) using the "Save Calibration" button.

- 28.** We now have a complete, combined SIRFP and multipoint (MLSQ) calibration. At this point, this method can be used for analysis of an unknown sample. You only need to open the ANA saved from the MLSQ calibration (step 27 above) in order to start analyzing unknown samples. All the information is contained within this single ANA file. For example, you can select the **Load Calibration** button and then open the "SIRFP-MLSQ_Tutorial.ana" file. This file not only contains the necessary coefficients, but also the complete sample definition. It is worth noting that any further ANA files saved, with results from the unknown samples, will also contain all the secondary coefficients and may also be used as a "master" file for calibration, just like the file saved above.
- 29.** You may change the sample definition, by adding or deleting components. Deleting merely results in the loss of the coefficients for the elements in that component. Adding components will result in elements that do not have defined TCC coefficients (just like the situation with single-standard calibrations). In this case, TCC values will compute at runtime by interpolation from neighboring elements in the periodic table.



- 30.** Now you can go to the Analyze workflow and load or acquire an “unknown” spectrum for analysis. Then select the **Quantify** button followed by the **Execute** button (R-hand panel). This will perform a full analysis of the “unknown” using the SIRFP-MLSQ method, thus yielding concentrations for the elements in the unknown sample.

