XRS-FP2 Basic Setup & Analysis

Note: This document describes the basic setup and configuration of XRS-FP2 and implementation of a standardless analysis.

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DOCUMENT CHANGE LOG

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1 QUICK GUIDE: XRS-FP2 SETUP & ANALYSIS FLOWCHART

A flowchart illustrating the steps involved in the basic setup and configuration of XRS-FP2 for standardless analysis is shown below in Figure 1:
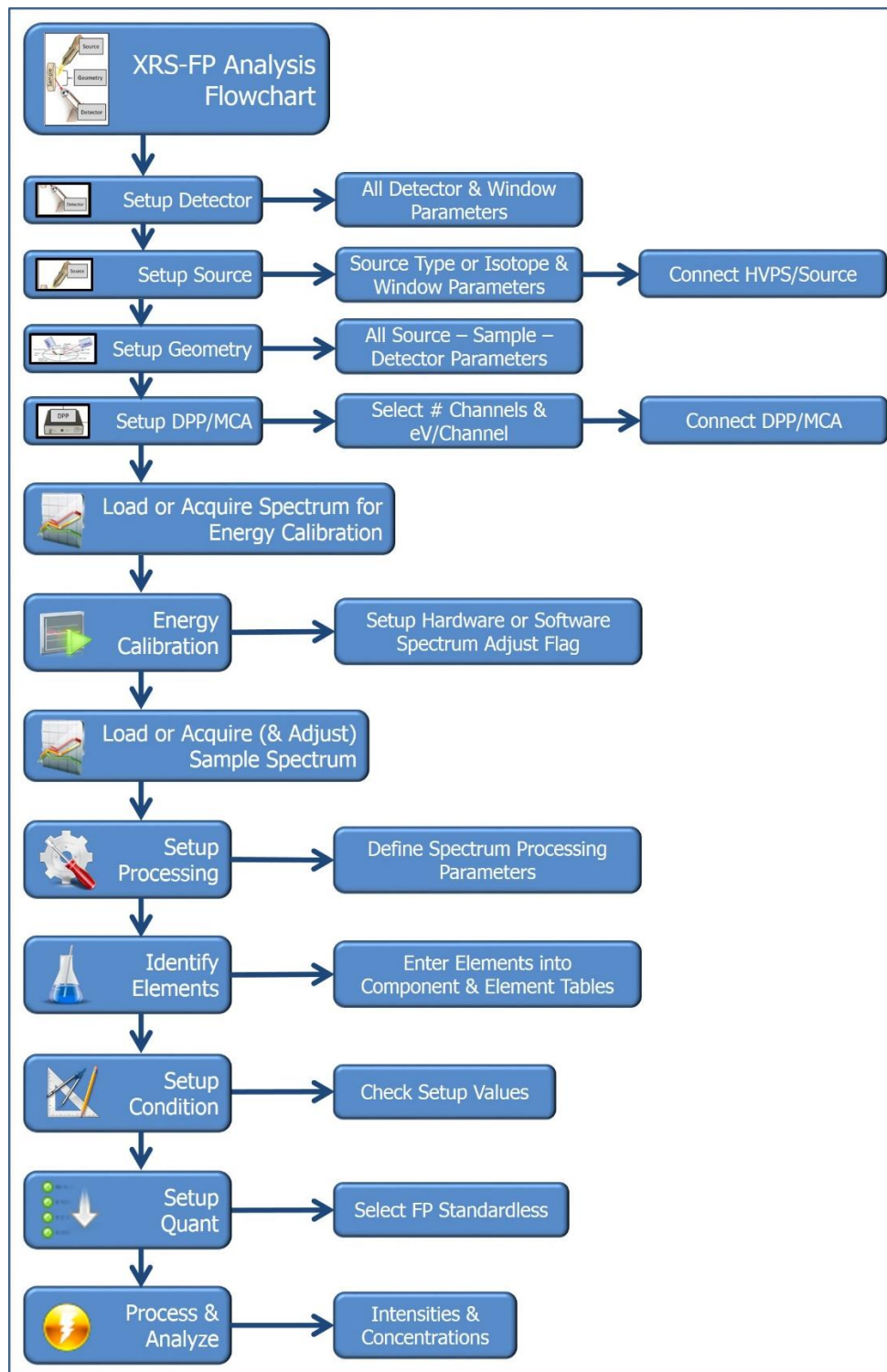

Figure 1. XRS-FP2 Flowchart. This flowchart describes the steps needed for basic setup and configuration of XRS-FP2.

2 XRS-FP2 SETUP & STANDARDLESS ANALYSIS TUTORIAL

The following tutorial describes the basic setup of a ***general*** XRF system for analysis, the configuration of XRS-FP2, energy calibration using Amptek hardware (although briefly, as this is discussed in detail in the "XRS-FP2 How to Calibrate with Amptek HW" document) and standardless analysis of stainless steel 316 (SS 316). This tutorial along with the associated application (*.ana) files guides an XRS-FP2 software user through the basic setup of their XRF system and subsequent standardless analysis of SS 316. This may be achieved in one of two ways:

a) Using a live XRF setup for hardware (HW) control and data acquisition (note: that this is a generic guide and the particulars of a given XRF system will vary from instrument to instrument).

b) Using the provided application (*.ana) ***without*** HW to emulate the setup and analysis.

> Note: It is not necessary to connect the XRS-FP2 program to any hardware unless you wish to acquire spectra directly with XRS-FP2. For **BOTH** methods listed above, the provided application (*.ana) file will be used. If using method (a) various input parameters may need to be changed depending on the type of HW, geometry, etc., used for a specific XRF system.

1. If using a live XRF system ensure that the hardware (HW) components are setup in the desired geometry and connected appropriately (power cables, signal cables, etc.). Please see appropriate guides proved by the HW manufacturer(s) for correct setup and **safety** measures.
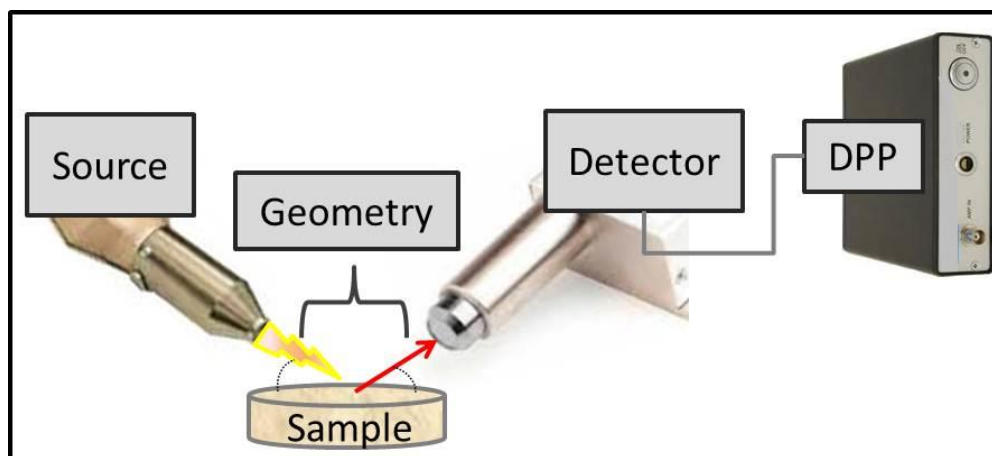

Figure 2. XRF Generic Setup. Above is a simple schematic of an XRF setup, including the source, detector and DPP. Note that for live acquisition both the source (x-ray tube) and DPP need to be connected to a computer for data acquisition and hardware (HW) control, and for running the XRS-FP2 analysis software; however, if working offline on previously acquired data or if using the files provided with this tutorial without HW there is obviously no need for the HW to be connected.

2. Launch XRS-FP2 – either run the software from the Windows Start-Programs menu, or double-click on a shortcut (if one has been created on the desktop). First, a splash screen will load and then XRS-FP2 will be displayed. See figure 3 below. The splash screen will briefly show the values and options for the HASP security plug, and the software libraries that have been loaded. This can also be displayed by selecting the **Help** button (located in the top ribbon).
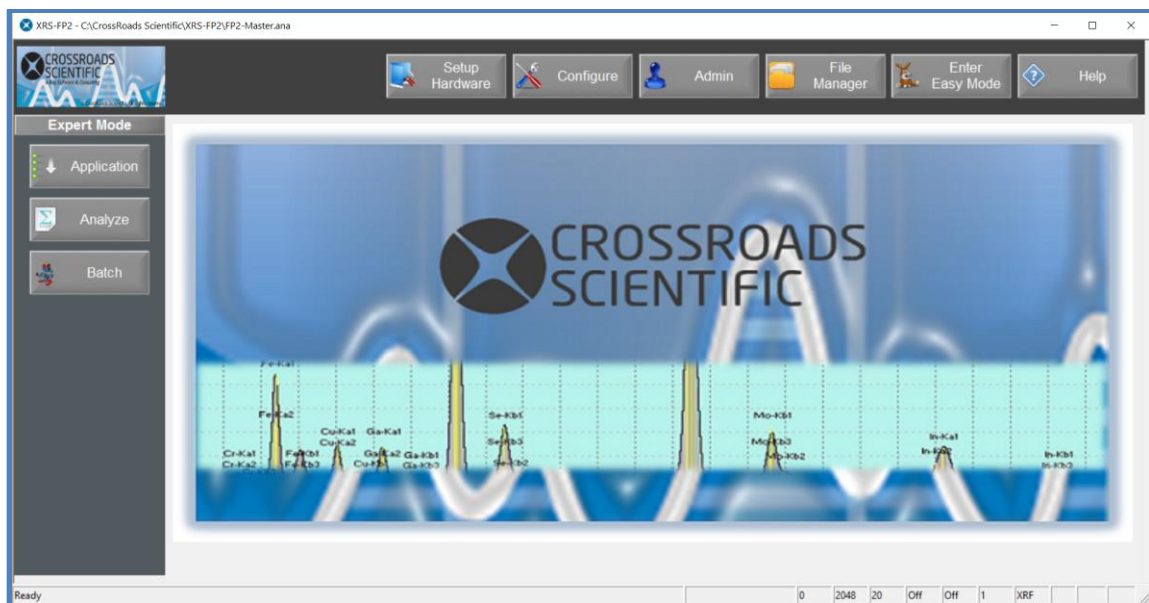

Figure 3. XRS-FP2. XRS-FP2 home page.

3. Select the **File Manager** button from the top ribbon. Then select the **Load** button and locate the "SS316 - Initial.ana" file in the **"C:\CrossRoads Scientific\XRS-FP2\Basic Setup Tutorial"** directory (or equivalent location). Select the "SS316 - Initial.ana" file in the right-hand panel of XRS-FP2 and either double click or use the **Execute** button to load the file. See figure 4 below.
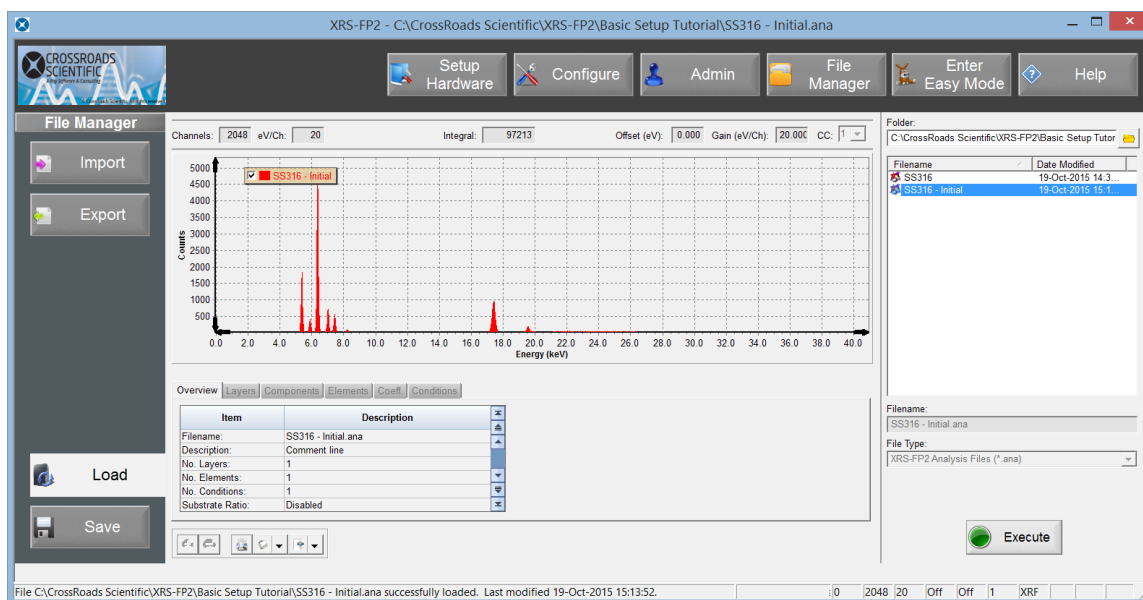

Figure 4. File Manager. Open the stainless steel SS316 Initial ANA file, e.g. "SS316 - Initial.ana".

This ANA ("analysis") file, acts as a template for this tutorial. An ANA file contains the elements and calibration coefficients (if employed) that are required for routine XRF analysis, along with a description of the various analysis and system setup. Although the components tab, elements tab and layer tab have no results at this point, the conditions tab (spectrometer configuration) is already pre-defined and loaded for this application. See Figure 5 below.

| Overview | Layers | Components | Elements | Coeff. | **Conditions** | | | | | | | | | | |

| **Measurement** | Processing | Background | Deconvolution | C/R Ratio | Process Flags | | | | | | | | | | |

Code	Target	Source ID	Filter	Thick. (mg/cm2)	kV	uA	Detector ID	Filter	Thick. (mg/cm2)	Atmos.	Geometry ID	Preset Mode	Preset	Actual
1	Ag	Src-1	Al	539.780	45.0	10.0	Det-1	None	0.000	Air	Geo-1	Off	0.0	27.5

Figure 5. XRS-FP2 Conditions Tab. The "SS316 - Initial.ana" file acts as a template for this application. Note that this file contains the proper measurement & processing information for this application (i.e. spectrometer configuration information).

> Note: If you are connected to HW, various input parameters may need to be changed in the TFR file depending on the type of HW, geometry, etc. used for a specific XRF system. The following steps will outline which parameters may need to be changed. Regardless of the method used for this tutorial (i.e. with or without HW), the steps outlined below will guide you through the process of setting up and configuring XRS-FP2 for use with an XRF system.

4. From the top ribbon of XRS-FP2 select the **Configure** button. This is where all the detector, geometry, source and filter properties are set. With the **Detector** button selected go to the "Edit" tab. If using HW with this tutorial, change the parameters so that they correspond to the XRF system in use. Otherwise, make sure all parameters are identical to those shown below in Figure 6. Note that the most important parameters involved in the detector setup are the detector material, thickness, resolution (Mn-Ka) and window type. If you are using your own spectra, you may need to change the Mn-Ka FWHM to a different value.
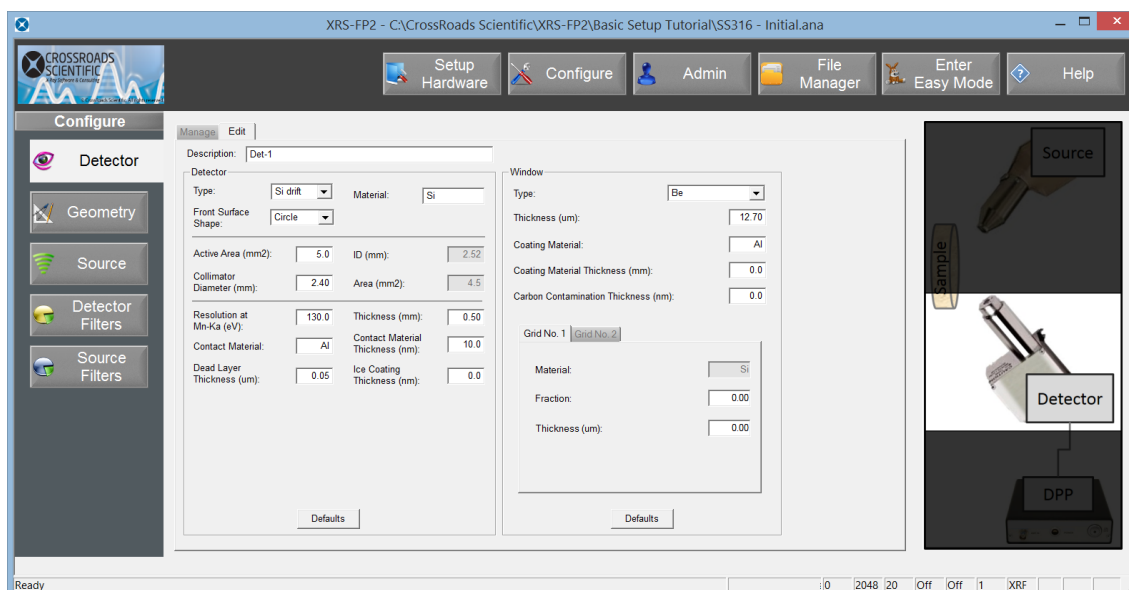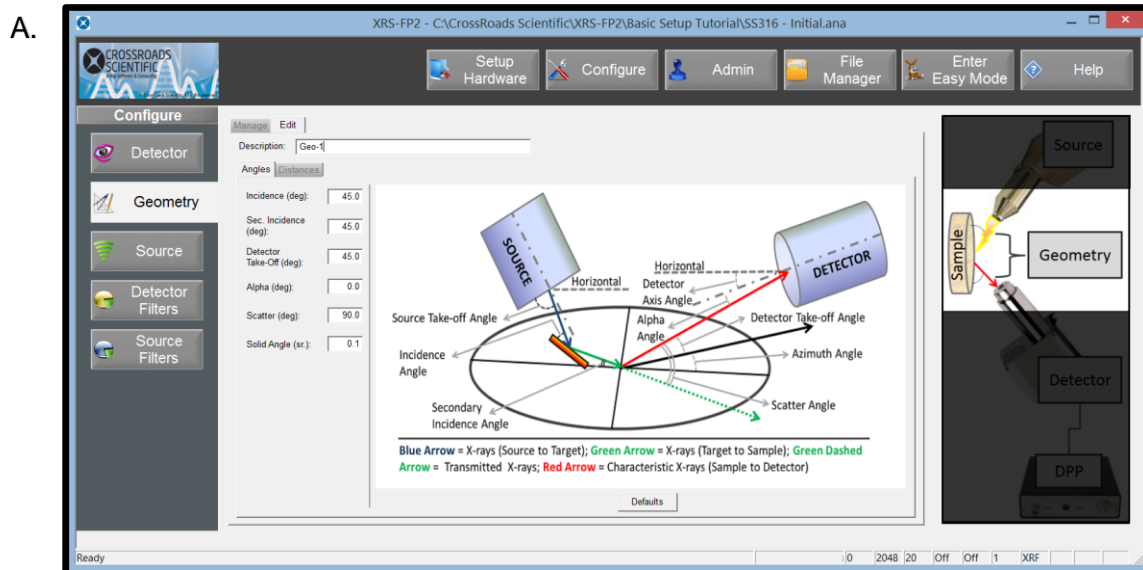
Figure 6. Configure Detector & Window Parameters. Check that the Detector "Type," Window "Type," and all corresponding parameters are as shown here, unless HW is being used with this tutorial.

5. Now, still in the configure workflow, select the **Geometry** button and go to the "Edit" tab. This is where all the angle (Fig. 7A) and distance (Fig. 7B) parameters are set. If using HW with this tutorial, change the parameters so that they correspond to the particular geometry of the XRF system in use. Otherwise, make sure all parameters are identical to those shown below in Figure 7. Note that the most important parameters involved in the geometry setup are the incidence angle, take-off angle, the tube-to-sample distance and the sample-to-detector distance.
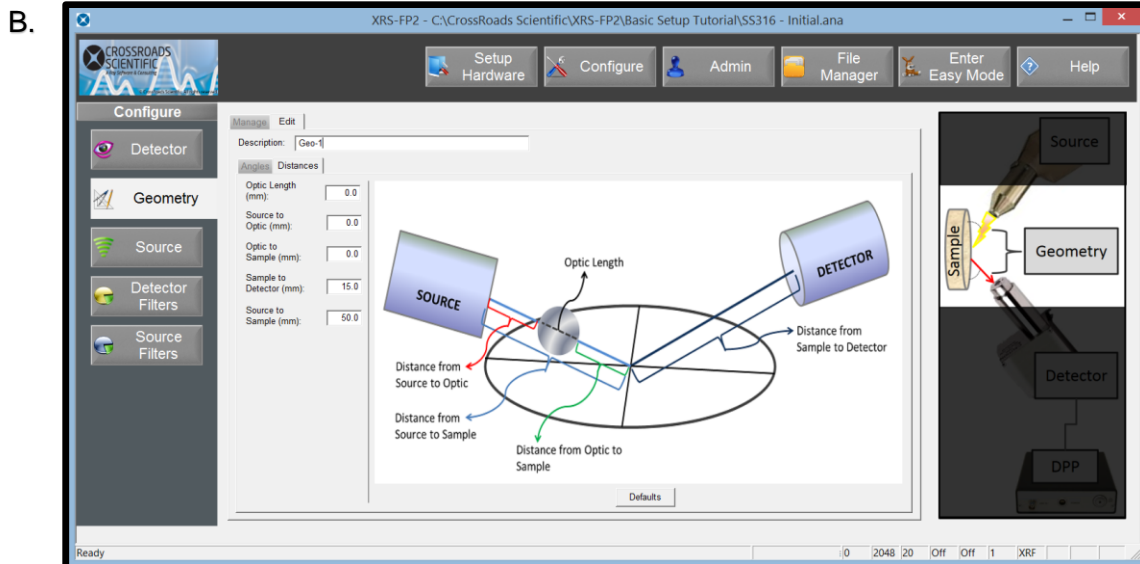
A.

B.

Figure 7. Setup Geometry Dialog. Check that the angle (A) and distance (B) parameters are as shown here, unless HW is being used with this tutorial.

6. Now, select the **Source** button and go to the "Edit" tab. This is where all the source properties are set. If using HW with this tutorial, change the parameters so that they correspond to the particular source of the XRF system in use. Otherwise, make sure all parameters are identical to those shown below in Figure 8. Note that the most important parameters involved in the source setup are the tube type, model, tube anode material (element) and the window type.
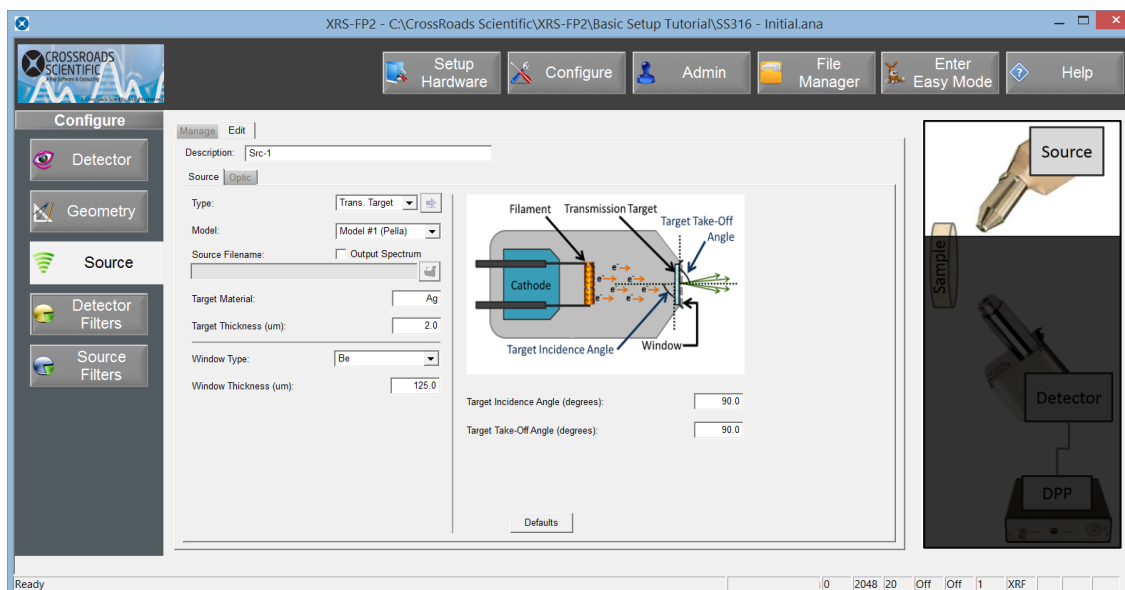

Figure 8. Configure Source Parameters. Check that the Source "Type," "Target Material," "Window Type" and all corresponding parameters are as shown above, unless HW is being used with this tutorial.

7. From the XRS-FP2 ribbon select the **Setup Hardware** button and go to the **DPP-MCA** button. If working offline (without HW) go to the MCA tab on the right hand panel and check or enter the number of channels, with the eV per channel or range (keV). See figure 9 below. ***If working with HW please see the following document for stepwise details on performing an energy calibration in XRS-FP (with Amptek HW): "XRS-FP2 How to Calibrate with Amptek HW.doc".*** Note that the two most important parameters involved in the DPP/MCA setup are the number of channels and range. Figure 9 shows the Amptek DPP/MCA Acquisition Setup parameters.
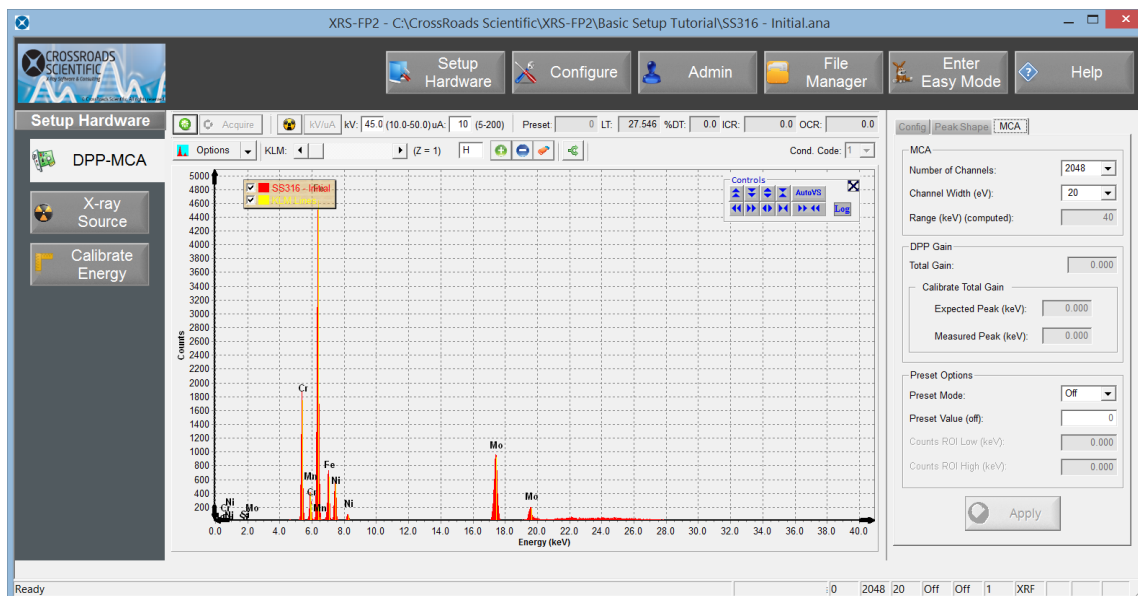

Figure 9. Setup DPP-MCA. Amptek DPP/MCA Acquisition Setup showing the No. Channels, eV/Ch and Range.

8. Now that the system has been configured and hardware setup, we move to the ***application workflow***, where the application and methods are defined. First click on the **Home** button (Fig. 10, yellow circle), then select the **Application** button (Fig. 10, red circle).
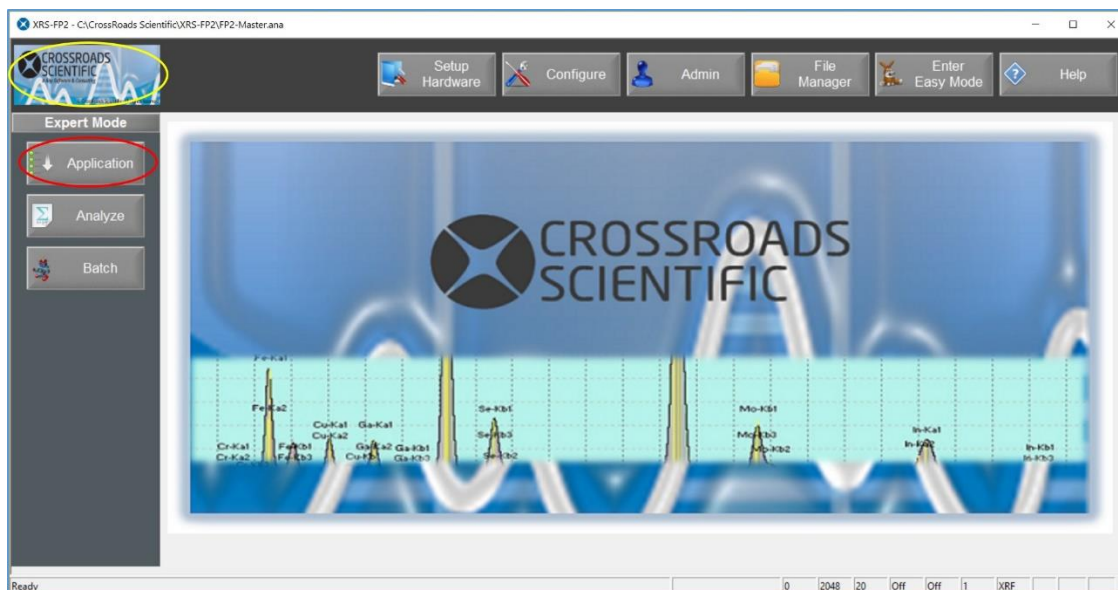
Figure 10. XRS-FP2 Application Setup. From the XRS-FP2 home screen select the application button to setup the application.

a. **Definition Button:** This is where the sample elements and compounds can be entered either by clicking on the periodic table or by entering them in the Component Table (in the right-hand panel).
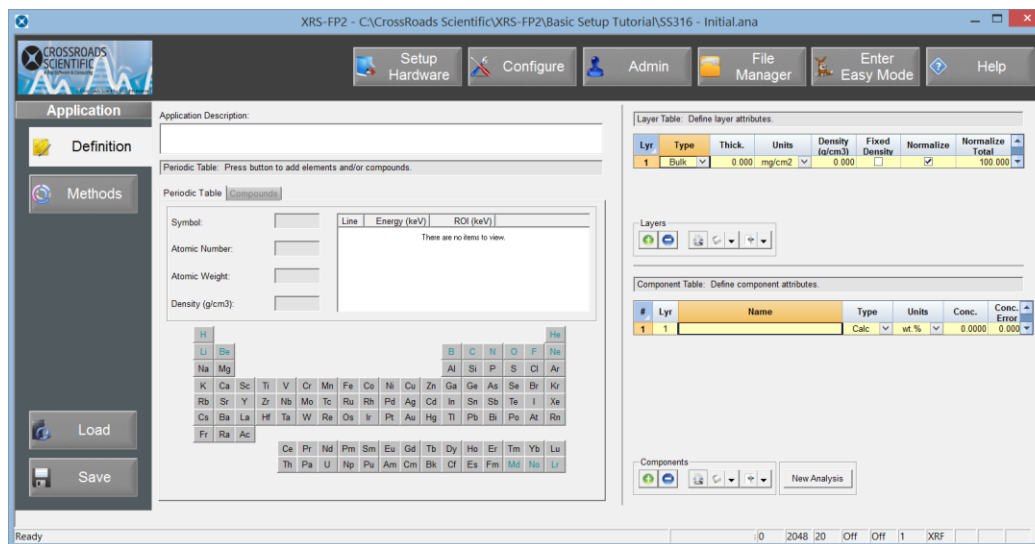

Figure 11. XRS-FP2 Application Workflow. The application definition and methods are input here.

For this tutorial, select or enter **Cr** as the first component. Having entered a component in the table, use the down arrow key of your computer keyboard or the "plus" and "minus" buttons to enter additional components. Now enter **Mn** as the second component, **Fe** as the third component, **Ni** as the fourth component, **Mo** as the fifth component and **Si** as the sixth component. Notice that the "Type" is automatically set to "Calc" so the SW will

calculate the concentration of each component; however, notice that the "Type" for **Si** is set to "Fixed" and the Concentration is set to 1.0000 in figure 12 below. The reason for this is there are several minor elements such as Si, S, P and C which cannot be analyzed. To account for this, we use 1% Si as a rough estimate. In the Specimen Component Table *enter* **"Fixed"** as the component "Type" for Si and set the "Conc" to **1.000**. See entered components below in Figure 12.

Figure 12. XRS- FP2 Definition Panel. The components, Cr, Mn, Fe, Ni, Mo and Si, are now filled in for this application.

Note: to create a "clean sheet" for future analysis click the **New Analysis** button below the component table (Fig. 12). This will clear all of the application information, i.e., Component Table, Elements Table and Thickness information; however, the Measurement information (spectrometer configuration) will be retained.

In addition to defining the sample components, the layer attributes of a sample are also defined here. Accurate XRF analysis requires definition of the thickness of the sample material. In the case of this tutorial, the stainless steel is infinitely thick, which means that it has greater thickness than the escape depth of the highest energy line. This is defined as "Type" "Bulk" in the "Layer Table", which is the default type setting. The default thickness

for bulk analysis is zero (0). The "Normalize" field of the thickness table refers to component concentrations. For standardless analysis the results **MUST** be normalized (see Fig. 12 above). Normalizing to 100% is not required for all bulk analyses, but is often employed.

b. **Methods Button:** As components are defined (see above), the "Element Table" is filled in and the line is set to the highest energy line that gets excited by the source kV. Note that the line must have an associated edge energy that is below the specified kV in the Condition Table, otherwise the intensity will be reported as zero. For example, the Cr Kα line is selected when Cr was entered for this application (see Fig. 13 below).
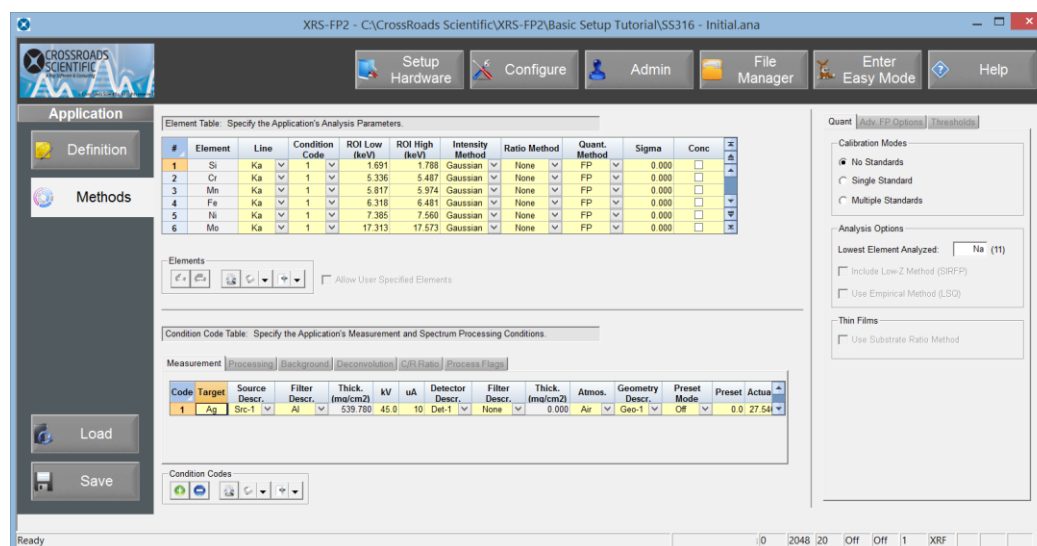

Figure 13. XRS-FP2 Methods Panel. As components are entered in the "Component Table" (Definition button), the "Element Table" is filled in and the line is set to the highest energy line excited by the source kV (e.g. the Cr Kα line was selected when Cr was entered).

Note: In general, the appropriate line for analysis (Ka, Kb, La, Lb, Lg or Ma), MUST be selected. Although this line is filled in by the software, it is important to check that it is the best one for the application at hand.

In addition to defining the application analysis parameters and measurement and processing conditions, the application quant method (calibration mode) is also defined here. Notice that the "Calibration Mode" for this application is set to "No Standards" (i.e. standardless) in the right-hand panel under the "Quant" tab (Fig. 13).

9. At this point our application, stainless steel 316, has been defined, XRS-FP2 has been configured for the setup of the XRF system used for acquisition (either the live XRF system in use or for the system previously used to collect the tutorial files) and it is calibrated (energy calibration). If using the supplied tutorial files *without* hardware proceed to step 10 below. If **using hardware** to do a

live acquisition, ensure that the DPP is connected and *now* turn the source x-rays on (Analyze button -> HV on). Details for a specific hardware setup are beyond the scope of this tutorial; however, please ensure that all safety measures are followed according to the hardware manufacturer guidelines! Once the system is running and stable, acquire a spectrum of the stainless steel 316 standard.

10. Now that the application has been defined, we move to the ***Analyze workflow***. The next step is to extract intensities from the stainless steel 316 spectrum (associated with this *ana file). To do this, go to the **Home** button and then select the **Analyze** button (see figure 14 below).
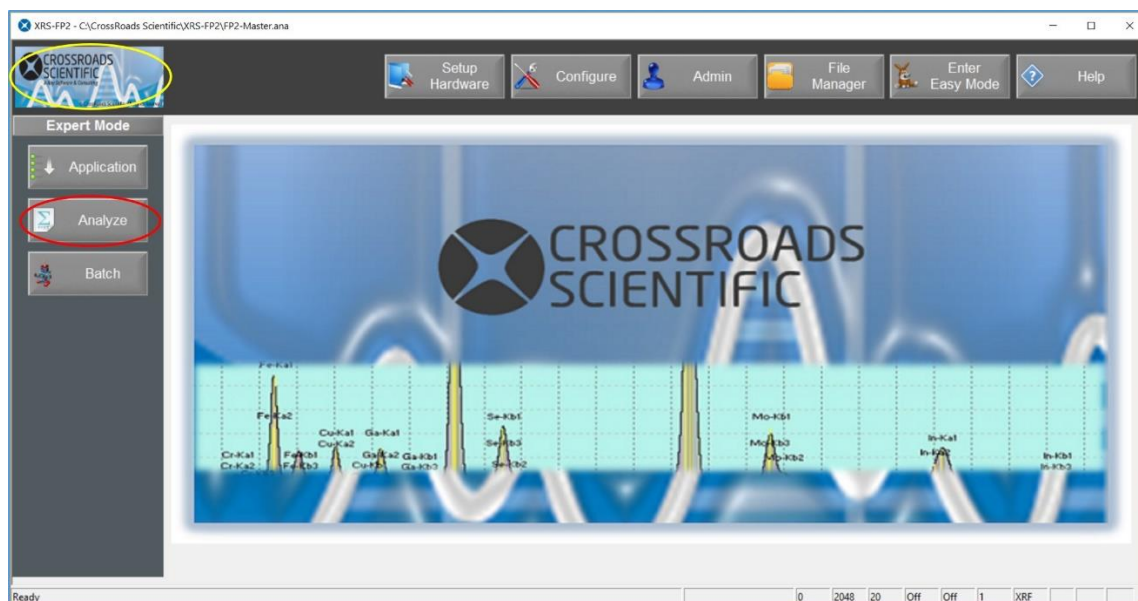

Figure 14. XRS-FP2 Analyze Workflow. From the XRS-FP2 home screen select the analyze button to collect, process and quantify spectra.

Then select the **Acquisition** button (Fig. 15 below). Note that spectra supplied for this tutorial have been energy adjusted (see the "Spectrum Adjust" section of the XRS-FP2 Software Guide). To avoid doing a double adjust in this case, verify that the "Auto-Adjust Spectrum" box is ***unchecked*** in the right-hand panel. See Figure 15 below.
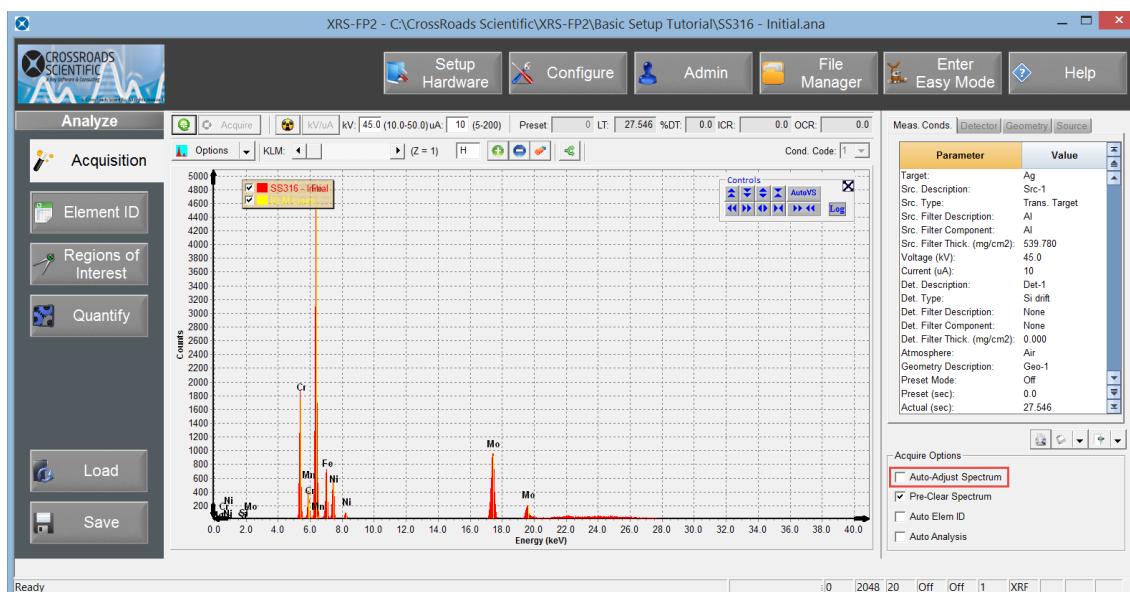
Figure 15. XRS-FP2 Analyze Workflow, Acquisition Button. From the XRS-FP2 home screen select the acquisition button, then check that the "Auto-Adjust Spectrum" box is ***unchecked*** given that spectra supplied for this tutorial have already been energy adjusted.

Now select the **Quantify** button to bring you to the following screen (Fig. 16 below):
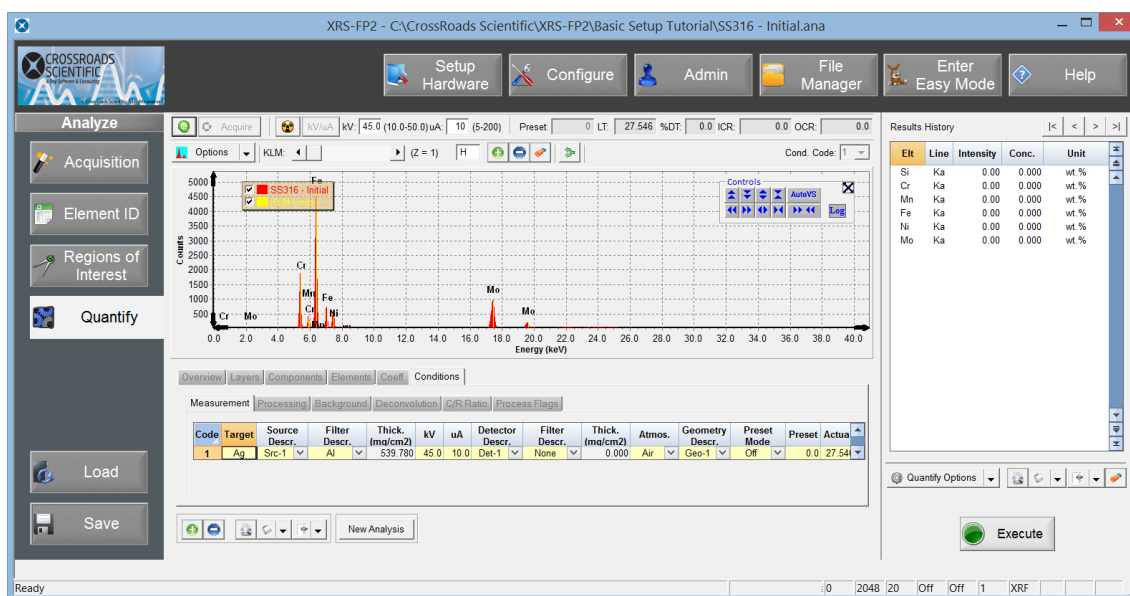

Figure 16. XRS-FP2 Analyze Workflow, Quantify Button. Select the Quantify button to process and quantify spectra.

Now select the **Execute** button (Fig. 16 above) to extract intensities from the stainless steel 316 spectrum. Note that the Execute button does a complete "process all" and quantification of the spectrum. You may also select the Quantify Options dropdown button to individually process and quantify the spectrum in a stepwise fashion.

Notice that there are now intensities and concentrations displayed for each element in the results listed in the right-hand panel (see Fig. 17 below). These results are also listed in the table below the spectrum display under the "Components" and "Elements" tabs.
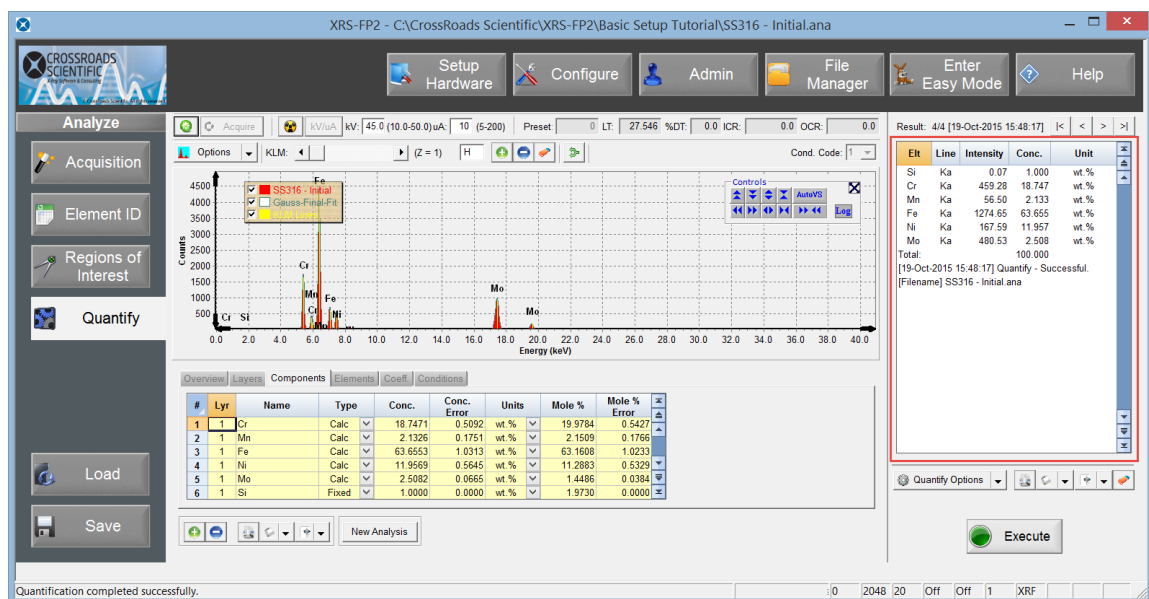

Figure 17. XRS- FP2 Quantify Panel. Note that intensities and concentrations are now displayed for each element.

11. You can now save the complete ANA file by selecting the **File Manager** button and then selecting the **Save** button followed by the **Execute** button. This can be saved as **"SS316.ana"** or as an alternate filename of choice. The analysis results should be similar to those shown below (Fig. 18).

Figure 18. SS316 Composition. A) Concentrations for the five calculated components in XRS-FP2 are shown here; B) the average composition for SS316 is shown here.

3 APPENDIX

XRS-FP2 is analysis software for use with X-Ray Fluorescence (XRF) Spectrometry, a widely used analytical technique. XRF analysis is based on the fact that incident x-rays excite atoms in a sample through ionization (the loss of an electron), which then emit characteristic radiation (x-rays of a particular energy) when they relax (an inner shell is filled by an electron from an outer-level shell), see Figure 19 below (for more detail see the XRS-FP2 Software Guide, "About X-ray Fluorescence").
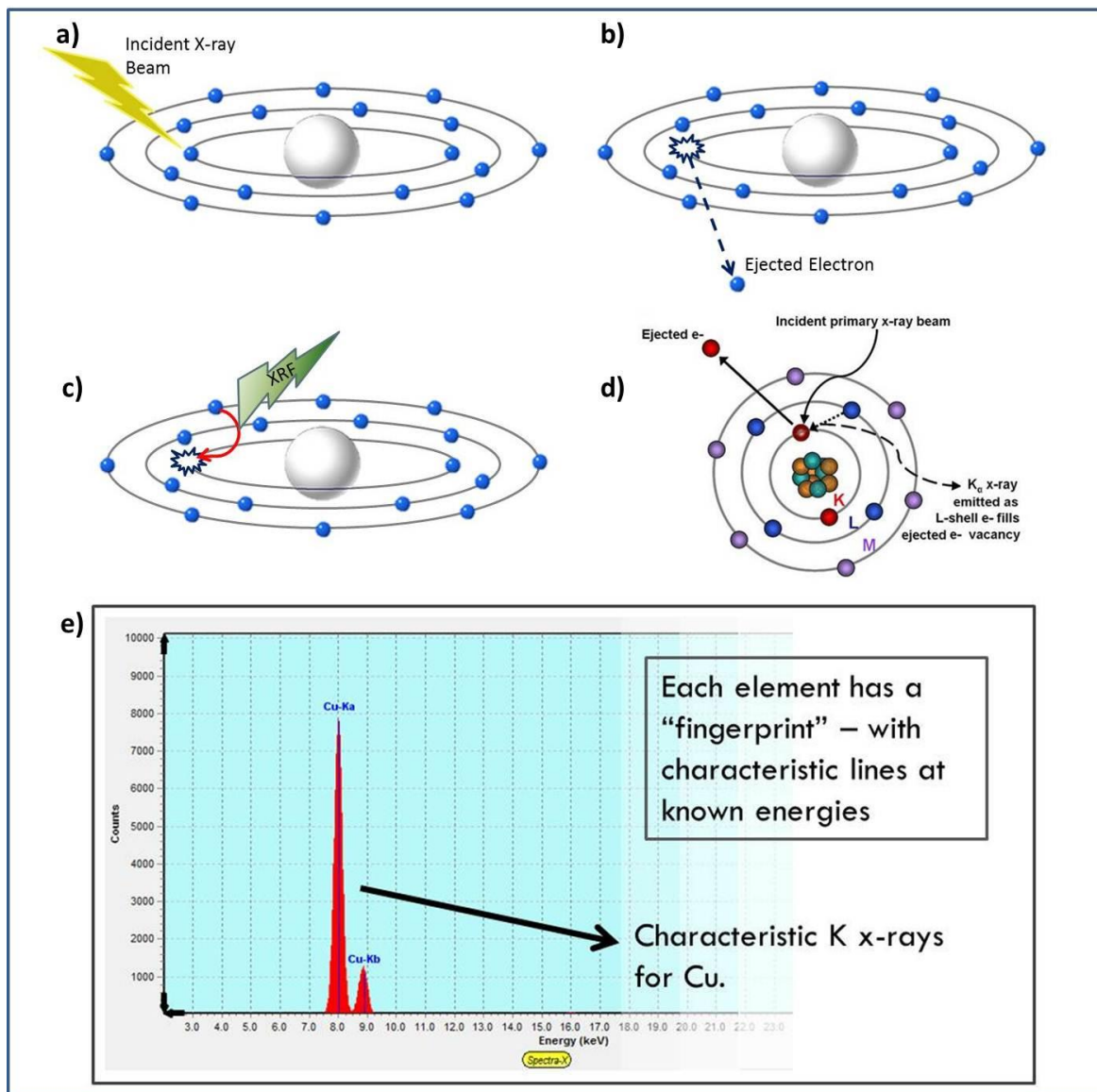

Figure 19. XRF Basics. a) Interaction of an x-ray photon with an atom (of sufficient energy), b) ionization of the atom by ejection of a core shell electron, c) vacancy filled by higher shell electron resulting in emission of an x-ray (photon). d) Schematic illustrating steps (a) through (c) including the emission of a characteristic K_α x-ray. d) Chemical elements emit characteristic radiations unique to each element when subjected to appropriate excitation.

A detector and signal processing electronics measure each emitted x-ray energy and stores them in "bins" as a histogram, or "spectrum." The X-ray analysis software, XRS-FP2, processes these "raw" spectra to obtain elemental peak areas that when divided by the acquisition time are known as intensities (counts per second, c/s). These values can then be used to determine the elemental concentration and/or sample thickness, using the so-called Fundamental Parameters (FP) method. XRF is capable of analyzing most elements in the periodic table non-destructively, and is therefore one of the most versatile analytical techniques currently available (Willis, Turner, & Pritchard, 2011).

The XRS-FP2 software consists of a main program that provides the user interface, and various hardware input and output functions, together with at least two libraries (DLL's) that provide the spectrum processing and FP calculations. It runs on standard PC's, and operating systems (Windows XP, Windows 7, Windows 8/8.1, and Windows 10), with 2 GB RAM.

Complete XRF analysis is possible, with or without standards, using an internal database of fundamental parameters (FP) such as absorption coefficients, fluorescence yields, transition probabilities, etc., and complete modeling of detectors and sources. Spectrum processing libraries are available for removing spectrum artifacts (escape and sum peaks), background and for various least-squares peak-fitting models using synthetic Gaussian or experimental reference profiles.

The typical XRF "system" (Fig. 20 below) includes an x-ray source (either an x-ray tube or radioisotope) and detector/electronics, together with a sample within a chamber, which can be evacuated or operated in air. In addition, filters may be used with either the x-ray source and/or the detector. It is extremely
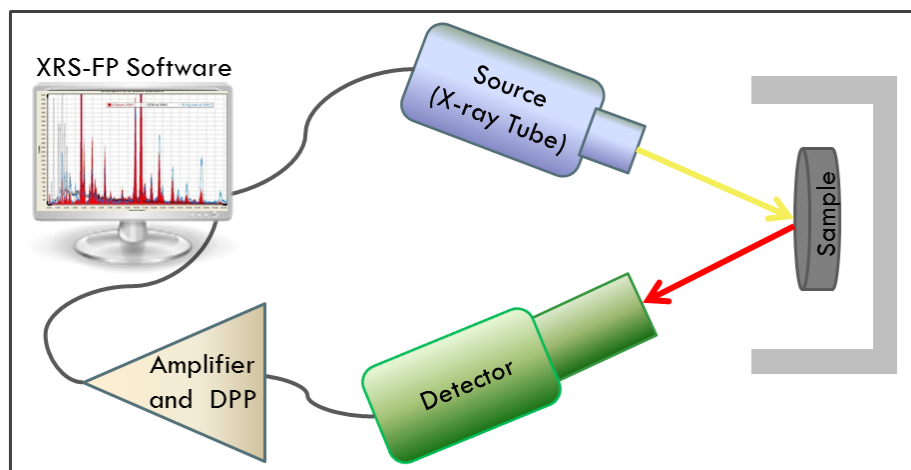

Figure 20. XRF "system". Key components of an XRF instrument are shown above. This includes the source (x-ray tube or radioisotope), the sample and sample holder, the detector, an

amplifier and signal processor and a computer for data acquisition, system control and for sample analysis using XRS-FP software.

important to know the geometry of your system and details about the components before setting up the XRS-FP2 software and doing any calibration or analysis work.

As briefly described above, the purpose of XRF analysis with FP is to convert elemental peak intensities (see previous section) to elemental concentrations and/or film thicknesses. This is achieved typically though a calibration step, where the XRF response function (related to parameters that are independent of the sample matrix) for each element is measured using a known standard of some kind. In some circumstances the analysis may be purely based upon theoretical equations, and the fundamental-parameter database, without any need for a calibration step. This is possible for simple bulk materials or single-layer films where the thickness is fixed, assuming the results can be normalized to 100%.

There are typically two steps in XRF analysis, whether or not the FP method is used. The first step is to calibrate each element from one or several standards (called the FP "Calibration" step). The second step is to produce the sample analysis of a given material, using the previously stored calibration coefficients, and the FP-based algorithms given a definition of the sample (i.e., elements, layers, and the distribution of the elements in the layers).