# Amptek Experimenter's XRF Kit

Quick Start and Demonstration

Amptek's Experimenter's XRF Kit contains the key components necessary to begin carrying out X-ray analyses. This kit was designed so users can rapidly set up a prototype XRF system and customize it for experiments or for specific measurements. It is not a turn-key system because it has not been optimized or calibrated for specific applications; the user is responsible for optimization and calibration. This requires you to understand both (a) the principles of XRF and (b) how to use Amptek's products. You will use the "Expert Mode" of the software, which presumes expertise in both topics. This guide walks the user through the set up and configuration of the system to do one analysis (standardless analysis of a stainless steel sample) as a system demonstration.
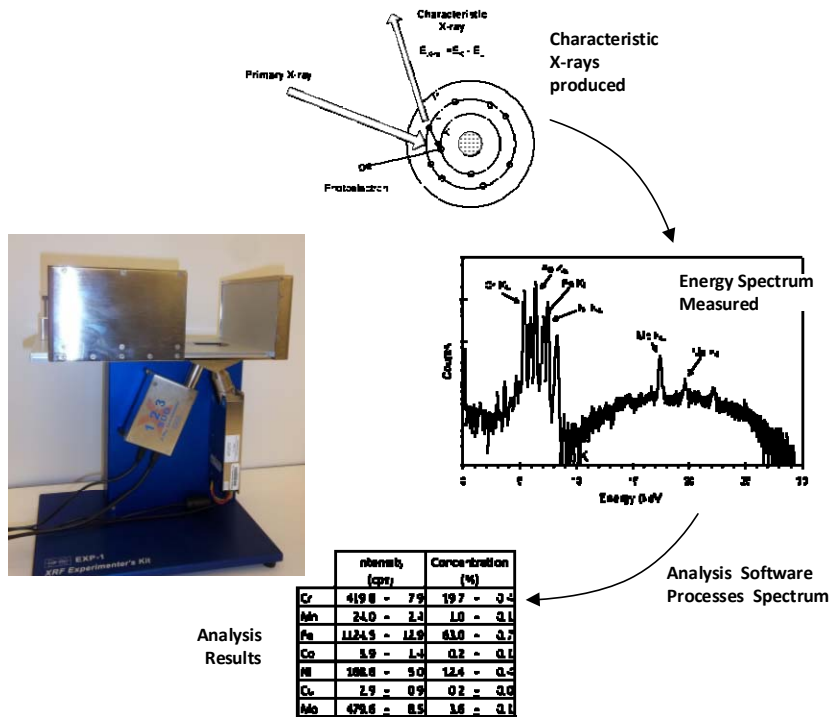
Warnings and Precautions

THE MINI-X GENERATES X-RAY RADIATION DURING NORMAL OPERATION AND PRESENTS A SAFETY HAZARD. The EXP-1 includes radiation shielding around the sample chamber, safety interlocks, and both a visual and audio alarm. THE EXP-1 MUST BE ASSEMBLED CORRECTLY. DO NOT DISABLE OR TAMPER WITH THE SAFETY FEATURES OF THE EXP-1.

High voltages are present in the Mini-X and the X-123. The Mini-X voltage (up to 50 kV) presents a potential personnel safety hazard.

THE DETECTOR AND X-RAY TUBE BOTH CONTAIN THIN, FRAGILE BERYLLIUM WINDOWS. If the windows are damaged, the units will be destroyed and cannot be repaired. Do not touch the windows! Do not drop anything on the windows! Be very careful when working around the detector!

THE USER MUST VERIFY THAT THE EXP-1 MEETS THE RADIATION REQUIREMENTS OF HIS/HER INSTITUTION. Warnings and precautions are discussed in more detail in the *Experimenter's Kit Application Notes.*

Release Note: This document, rev D0 (Aug 2014), describes procedures for X-123 units containing the EXP-1 fixture, a DP5 with FW6, and for XRS-FP version 5.2.1 or later
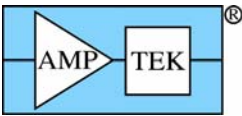
1. Equipment List

Amptek Supplied

- ☐ X-123 spectrometer. Includes X-ray detector, preamp, digital pulse processor & power supplies. It is mounted on a spacer.

  Several detector options are available from Amptek. You need to know the options for your detector (detector type, area, and window thickness).

- ☐ Mini-X X-ray tube and controller, with mounting bracket (attached).
- ☐ Mini-X collimator & filter kit for EXP-1
- ☐ EXP-1 mechanical fixture. Includes radiation shielding and a sample plate.
- ☐ Six 4-40 screws

- ☐ SS316 test sample
- ☐ 2 Mini-USB cables
- ☐ X-123 +5 VDC adapter
- ☐ Mini-X +12VDC adapter
- ☐ Amptek software installation CD
- ☐ XRS-FP USB HASP plug

Customer Supplied

- ☐ Computer with Windows XP or Win7 and 3 USB ports
- ☐ Phillips screwdriver
- ☐ Optional: Safety secondary window (discussed below)

Documentation

The Experimenter's Kit combines three separate, standard Amptek products and a unique mechanical fixture. The components are (1) the X-123 spectrometer (which includes a detector and the DP5 digital signal processor), (2) the Mini-X X-ray tube (which includes a collimator with a place to mount filters), (3) XRS-FP X-ray analysis software, and (4) the EXP-1 mechanical fixture which provides radiation shielding, safety interlocks, and a sample chamber.

The X-123, Mini-X, and XRS-FP are provided with extensive, separate documentation (quick start guides, user manuals, software installation guides, and application notes). These can all be found on the Amptek Installation CD and Amptek's website. The software has a help file with much information. Please refer to these documents to understand the individual components in the kit. The "Experimenter's Kit" documents focus primarily on assembling and initial operations of the kit rather than offering details on the various components.
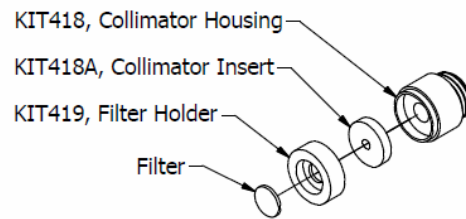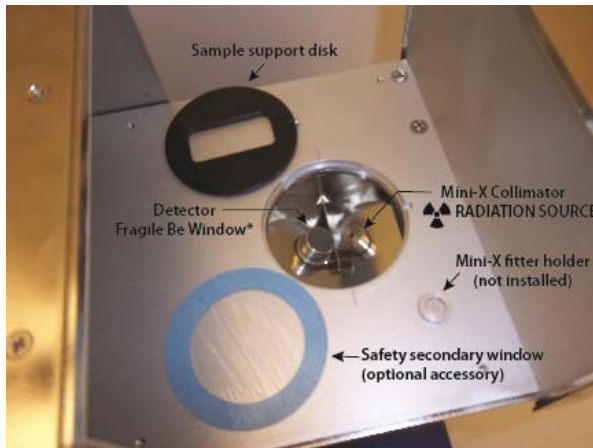
2. Mechanical Assembly

The drawing to the right shows an assembled EXP-1 fixture. To assemble:

1. Place the EXP-1 fixture on its back for easy access. Make sure there is nothing inside the sample chamber (e.g. the sample support disk).

2. Install the cables onto the Mini-X. There are three cables: power (to the +12V AC/DC adapter), USB, and a four pin interlock cable.

3. The Mini-X has been shipped with its mounting bracket attached. Align with the holes in the fixture and attach with two ¼" long 4x40 screws. Plug the interlock cable into the fixture.

4. Install the cables onto the X-123. There are two cables: power (to the +5V AC/DC adapter) and USB.

5. The X-123 has been shipped with its baseplate attached. It also has a black protective cap on the Be window. Very carefully slide the X-123 extender through the hole in the shielding (the black cap can be left in place). Attach the X-123 with four ¾" long 4x40 screws.
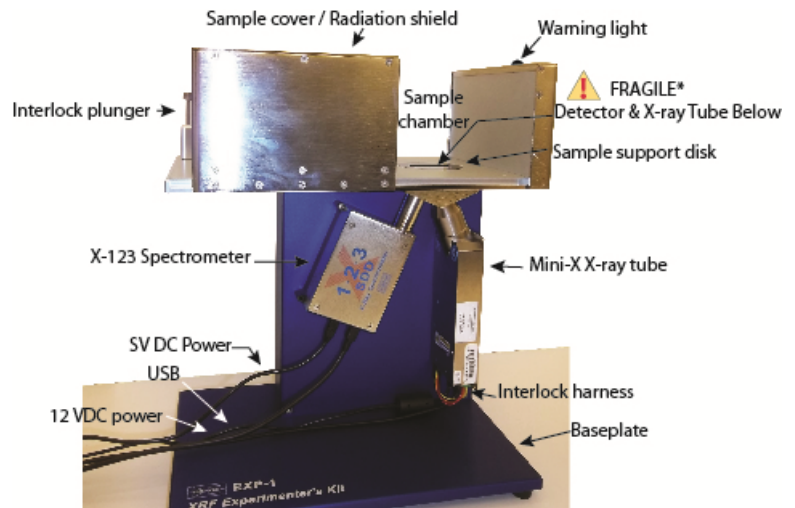
6. Turn the fixture upright and install the Mini-X collimators and filters. BE VERY CAREFUL NOT TO TOUCH OR DROP ANYTHING ONTO THE DETECTOR BE WINDOW. The unit is shipped from Amptek with the collimator housing (drawn below) installed on the Mini-X. Place the tungsten collimator insert into the housing. In the Mini-X filter holder, place a 1 mil tungsten filter and then on top of this place a 40 mil aluminum filter. These filters can be found in the Mini-X collimator kit. VERY CAREFULLY, slip the holder with filters over the collimator housing. You can do this with the black cover on the detector. Once the filters are in place, remove the cover.

7. Place the sample mounting plate (the black plastic disk with a rectangular cutout) into the opening in the sample chamber. You may wish to cover this with a safety secondary window. This is a thin polymer window, to avoid accidental damage to or contamination of the detector Be window. Commercial sources and part numbers are in the *Experimenter's Kit Application Notes.*

8. Place the stainless steel 316 test sample over the cutout in the sample holder.

9. Close the cover of the sample chamber (the cover is vital for radiation shielding). Close the interlock plunger behind the cover. The chamber and the plunger are a tight fit; it is necessary that the plunger be closed for the Mini-X X-ray tube to operate and necessary to fully close the chamber for the plunger to seat properly.
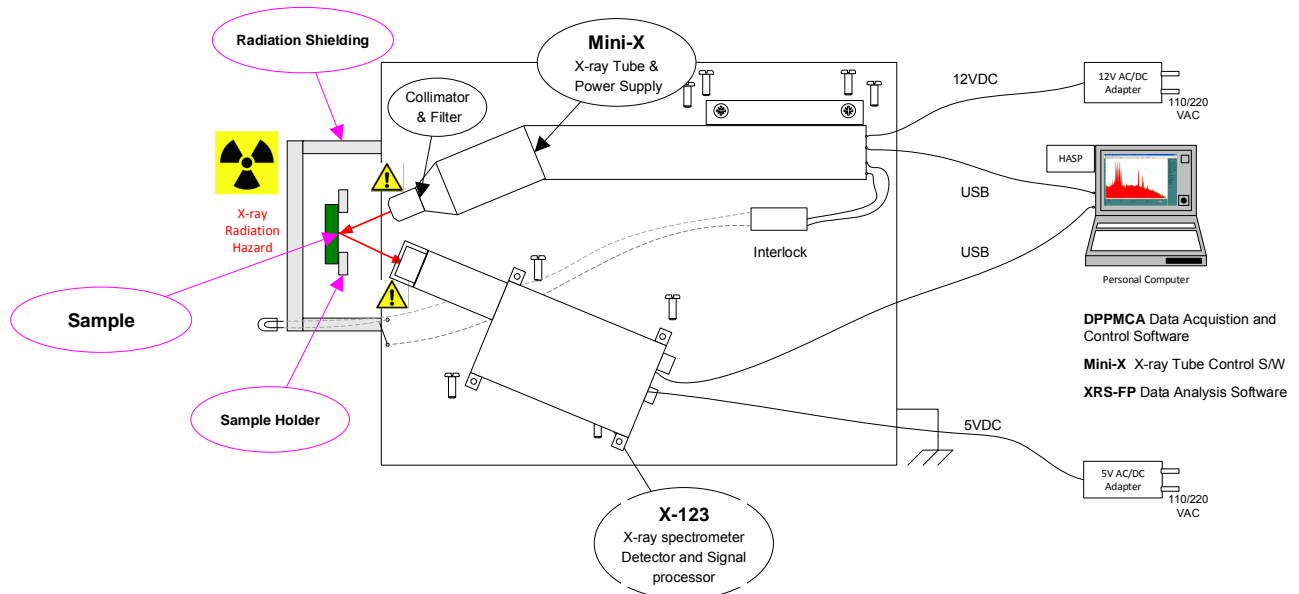
Schematic drawing of the Experimenter's Kit.
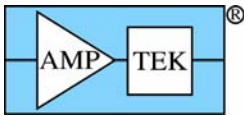
3. Connect Modules & Install Software

Three software packages are needed. "DPPMCA.EXE" is used to configure the X-123 spectrometer. "MINIX.EXE" controls the Mini-X X-ray tube. "XRS-FP.EXE" controls the X-123 after initial configuration, acquires the data, processes the spectra, and analyzes the results. Each of these software packages requires a USB driver to be loaded. The user should follow the software installation instructions carefully to avoid problems.

Connect X-123 and install DPPMCA Software and Drivers

1. Turn on the PC. Log on to Windows as an administrator. Insert the Amptek Installation CD (or download software from *www.amptek.com*).

2. Connect the X-123 to the PC via a Mini USB cable. Plug the X-123 5VDC supply into a 110/220 VAC outlet and into the X-123 back panel.

3. A "Found New Hardware" or "Installing Device Driver" box opens. Follow the instructions for "Automatic" installation over Windows Update. The steps are slightly different for XP and Win7 machines (see the instructions in "Amptek DPP Software Installation.PDF").

4. Create *C:\Program Files (x86)\Amptek\DPPMCA.* From the CD, copy the contents of *DP5 PX5…\DPPMCA* into the new folder. Create a shortcut to DPPMCA.EXE and place this on the desktop.

Install XRS-FP Analysis Software

5. Plug the HASP into the PC USB port.

6. A "Found New Hardware" or "Installing Device Driver" box opens. Follow the instructions for "Automatic" installation over Windows Update.

7. Locate the *XRF-FP with Spectra-X* folder on the Amptek Installation CD. Run (as administrator under Win7) the "XRSFP_Setup.exe" file and follow the instructions.

Install Mini-X Software and Drivers

8. Locate the Mini-X folder on the Amptek Installation CD. Run (as administrator under Win7) the "Mini-X Setup.exe" file and follow the instructions.

9. Connect the Mini-X via the mini-USB cable to the PC. Plug the Mini-X 12VDC supply into a 110/220 VAC outlet and into the Mini-X.

10. When the unit connects, Windows will prompt for driver installation. Select "Install from a list or specific location: the "Mini-X\MiniX Drivers" folder on the Amptek Installation CD. There is a warning because the drivers are not WinQual certified but install anyway. Repeat for Controller B (there are two controllers).

Quickstart Files

11. On the Amptek Installation CD is the *Experimenter's XRF Kit* folder, with subfolders for the two main detector configurations, the SDD and the SiPIN. Find the folder for your detector. Copy MASTER.TFR and XRS-FP.INI from the appropriate folder into *C:\Program Files (x86)\CrossRoads Scientific\XRS-FP\.*

4. Check Hardware

Check X-123 spectrometer

1. Double-click "DPPMCA" icon. A "Select DPP Communications Port" dialog opens. The device should be listed in the USB section. Click "Connect".

    A green USB symbol should appear at the bottom and the DP5 serial number at top right.

    Note: The X-123 boots up using the configuration stored internally when it was powered down. When you receive it, the unit was last powered down using a configuration stored at the factory where it was tested with a ^{55}Fe source.
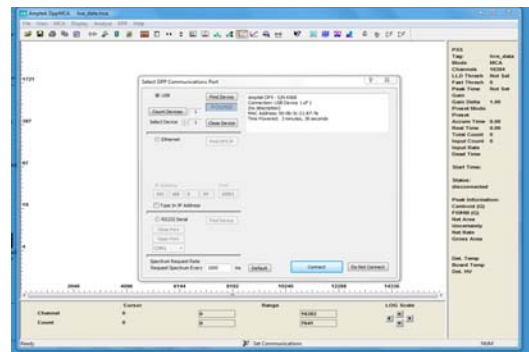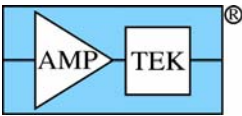
2. It is normal to see noise counts towards the left as the system stabilizes. Click "Delta" mode on the upper right (this updates the data every second rather than accumulating a spectrum and is useful during setup). The noise counts should disappear in a couple minutes.
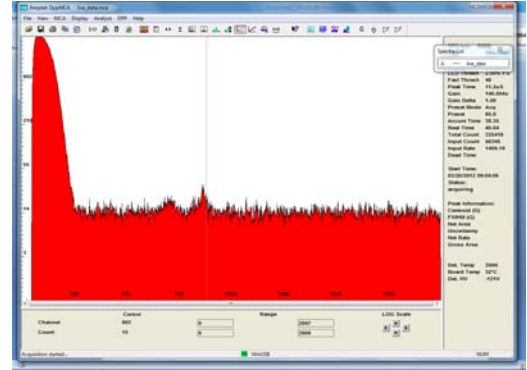
3. Double click the "Mini-X" icon on the desktop. Click "Start Mini-X".   The serial number should appear in the top middle and the "HV ON" and "OFF" boxes will become active.

Check X-ray tube and shielding

1. Verify that the shielding and stainless steel sample are installed.
2. Verify that Mini-X software is set to 25 kV and 20 μA (starting at a low level for safety).
3. Ensure that the Mini-X safety interlock is closed. In the Mini-X software, Click "HV On", then select "Yes" on the box that appears. The Mini-X should begin beeping, a red light on the unit will flash, and a symbol will appear on screen.
4. Use the survey meter to verify that the shielding is effective, looking particularly for scattered X-rays.

Check that X-rays are detected

5. Counts should be recorded in the spectrum. Click "Delta" to accumulate data, and the display should look similar to the screen on the right. The "Info Pane" (right) should show counts, fast and slow. The peaks visible here are the Cr, Fe, and Ni peaks in stainless steel.
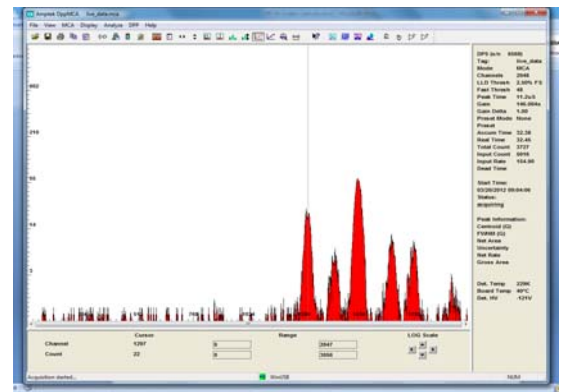
Radiation safety check:

6. Increase Mini-X HV setting to 50 kV (maximum) and the current to 80 μA (maximum).   Use the survey meter to verify that the higher energy radiation is stopped by the shielding.
7. Set HV to 30 kV and the current to 100 μA. Click "HV Off" to turn off the tube.

5. Configure X-123 Spectrometer

1. The configuration of the X-123 will need to be changed from the factory test configuration to one appropriate for this XRF application. In particular, the gain should be adjusted to give approximately 40 keV full scale. A gain of 30 - 35 is typical.
2. In DPPMCA, select the "Acquisition Setup" button, then the "Gain" tab. Set gain to 30, then click "Accept".
3. On the MCA tab, set the preset time to "30 seconds", set MCA channels to 2048, then click "OK".
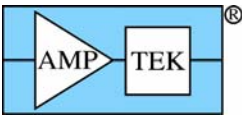
4. Verify that the HV is 30 kVp and the current 100 µA. Turn the HV on, then turn "Delta mode" off. When data acquisition stops, click "HV OFF" button to turn off the X-ray tube. The spectrum should look like the one displayed below.

Calibrate the energy scale

To calibrate the energy scale, you need (at least) two peaks of known energy. For best results, use peaks that are widely separated in energy, with good statistics and no overlapping peaks. In this example, we use the Fe and Mo K_α peaks in stainless steel 316. What you will do is mark regions of interest (ROIs) around the two peaks, open the calibration dialog, then define the energies of the centroids. The software performs a linear regression. The calibration obtained in DPPMCA will be transferred to the XRS-FP program.
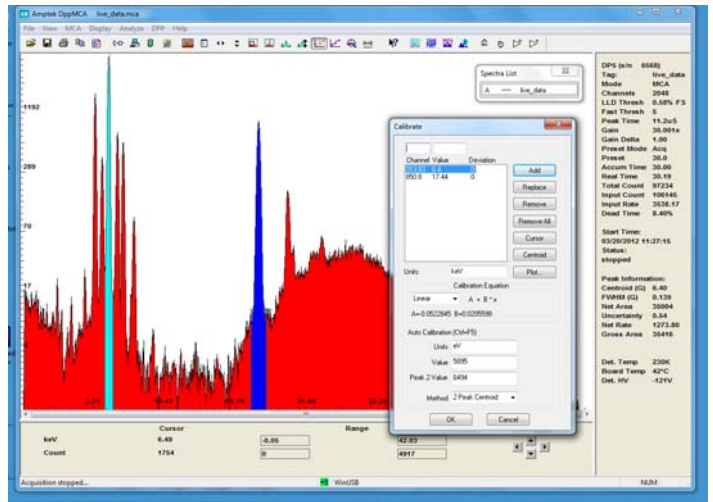
1. To mark the ROIs, click the "Edit ROI" button, then click, drag, and release for each peak. You may want to zoom in on the peaks, using the arrows near the bottom of the display.

2. Click the "Calibrate" button. The dialog shown on the right appears.

3. Select the Fe K_α peak in the spectrum display, then click the "Centroid" button in the "calibrate dialog", then enter the energy (6.40 keV) in the box beside the channel. Click "Add".

4. Repeat for the Mo K_α peak at 17.44 keV. Click "OK", then "Enable Cal" on the toolbar.

5. Verify the calibration by checking the centroids of other peaks, e.g., the second largest peak (with SS316) is Cr K_α at 5.4 keV.

6. Check the energy resolution. The FWHM of the Fe K_α peak should be slightly larger than the FWHM listed on the data sheet supplied with the detector (e.g., an SDD should give about 140 eV).

7. Check if the upper channel (right hand edge) is about 40 keV. If not, then adjust the gain. You may use the ">" and "<" buttons on the toolbar, and may find it convenient to turn on the tube and use "Delta Mode". The gain can also be changed using the "Gain" tab under "Acquisition Setup".

8. Under the "MCA" tab, set the "Preset time" to 30 sec. Advanced users should adjust other parameters at this time.

9. After all parameters are set, turn off the tube, clear the data, and start an acquisition with no counts. Click the "Tune Slow/Fast" button to set thresholds. Turn the X-ray tube back on and do a final energy calibration for the new settings.

10. Save the spectrum as a .MCA file. Open the "Acquisition Setup" dialog, click "Save Configuration File", and save the configuration as DP5.TXT in *C:\Program Files (x86)\CrossRoads Scientific\XRS-FP*.

11. Close DPPMCA.EXE and MINI-X.EXE.
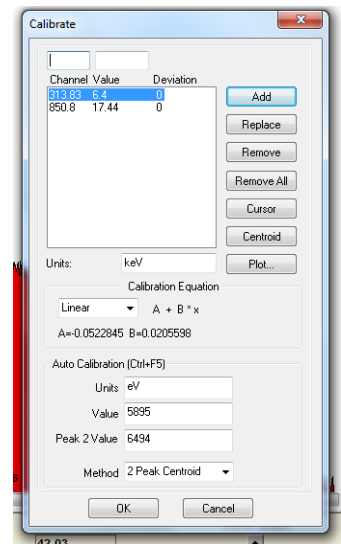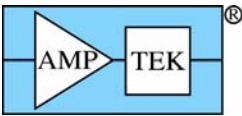
6. Configure XRS-FP Software

Properly configuring and calibrating the XRS-FP software is vital for obtaining accurate results. For the SS316 demonstration, you can start with existing configuration files appropriate for stainless steel and your detector. For other samples and measurements, care must be taken to properly configure and calibrate this software.

1. Open the XRS-FP software. Click "Expert Mode" in the gray box.

2. Two additional boxes appear: a light blue "Spectrum Display" box and the "XRS-FP" main screen. The main screen has several key components.

- The "Setup" menu item provides access to the many parameters defining the basic setup, the hardware and processing options.

- The "Calibrate" menu items are used to calibrate the energy scale and to calibrate the parameters used in quantitative analysis from reference materials.

- The "Thickness Information" table is used to define the sample's thickness, density, and layers.

- The "Specimen Component Table" is used to enter the elements to be analyzed.

- The "Element Table" is used to define analytical methods for each element.

- "The Measurement and Processing Conditions" table is used to define certain parameters which can be adjusted for each particular measurement.
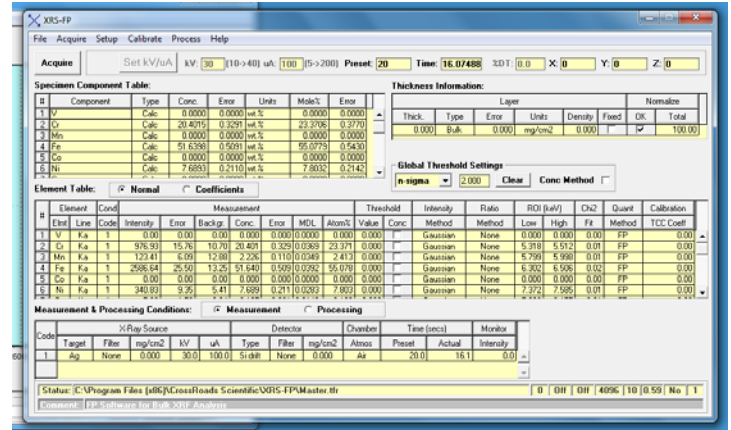
Select parameters defining the hardware

3. Select "Setup" from the menu, then "DPP/MCA". Click "Connect".

4. A large box appears listing all the X-123 parameters. Review these, then click "OK". Check that the correct device type and serial number appear in the lower left corner of this box. Click "Exit".

5. Select "Setup", "Detector" and verify the detector properties, particularly the area of the detector, the collimated area, and the thickness of the Be window. Amptek supplies many different options for these. The MASTER.TFR files you copied does not necessarily reflect the details of your detector.

6. If you are using anything other than an Ag anode tube with 1 mil W and 40 mil Al, use "Setup", ""Tube/Source" to enter the values. For multilayer filters, use the "Multiple layer window" pull-down.

7. If you have made any modifications to the fixture, use "Setup", "Geometry" to correct values. It is very important that hardware parameters be set accurately.
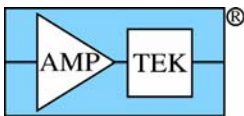
Select spectrum processing parameters

Enter the correct energy resolution. This is important for fitting the spectrum and depends on the detector and on hardware settings. Use the value measured in DPPMCA using the Fe K_α peak. The corresponding Mn K_α value must be entered. Select "Setup", "Processing", then the small box with 3 dots to the right of "Peak Width (FWHM)". Another box appears.

8. For "Energy", type 6.40. For "FWHM", the bottom box, type the value you measured (e.g., 140). Click "Get Mn FWHM", which returns 134.4 for this example. Close the dialog and enter this value (e.g., 134.4) for "Peak Width (FWHM) at Mn Ka".

9. For "Low Pass Filter Width", enter a value roughly 1.5 times the Mn FWHM (200 in this example).

Select quantitative analysis parameters

10. Select "Setup", "Quant", and verify that the "Fundamental Parameters" and "Standardless" buttons are selected.

Enter sample parameters

11. To perform an analysis, one must select which elements to analyze and how the photopeak intensity is modeled. The default TFR file already contains the elements found in stainless steel 316 and uses a Gaussian peak fit. To remove an element from analysis, select it in the "Element" table and click "Delete". To add one, select a line, then click "Insert" and type the symbol.

12. The default TFR file presumes that the sample is thick and normalizes the elemental concentrations to 100%. The "thickness information" table permits one to analyze other types of samples.

13. When all configurations options are set, save the MASTER.TFR file (over-write existing file). This is loaded at start up. We recommend also saving it with a different file name for reference (e.g., SS316 baseline.TFR).
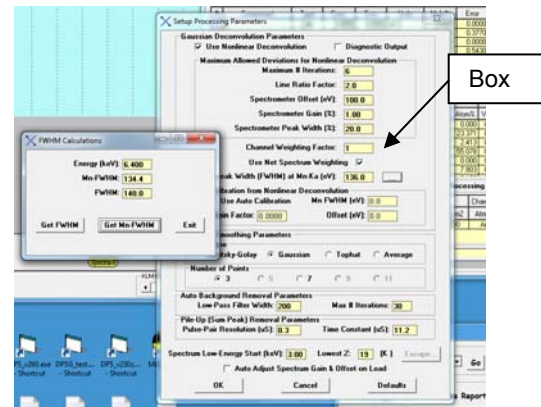
7. Calibrate the energy scale

Measure the spectrum calibration

1. In "Setup", "DPP", set the "Auto Adjust on acquire" option to OFF.

2. Turn on the X-ray tube HV by clicking "HV ON" in the Mini-X window. The click "Set HV and current" to set these to 30 kV and 100 μA.

3. In XRS-FP, select "Acquire". The spectrum should appear in the blue box and the time will increment in the main screen.

4. When the 30 seconds has elapsed and the acquisition is finished, turn off the X-ray tube.

Calibrate

5. Open "Calibrate", "Spectrum". Press "reset gain and offset" to start with defaults. Enter 6.400 into the "low energy peak" box and 17.443 into the "high energy peak" box. See figure on next page.

6. Go to the blue, Spectra-X window and use the cursor to find the centroid of the Fe and Mo peaks. Enter these values as the "Low energy" and "High energy" peak values in the "Manual Calibration section

7. Click "Calculate Gain & Offset" (you will see some change in offset and gain values).

8. Now use the automatic calibration method by click the "Calibrate Spectrum" button to get refined values of the gain and offset. Press "Adjust spectrum" to adjust the spectrum to match the new values.

9. In "Setup", "DPP", set the "Auto Adjust on acquire" option to ON.
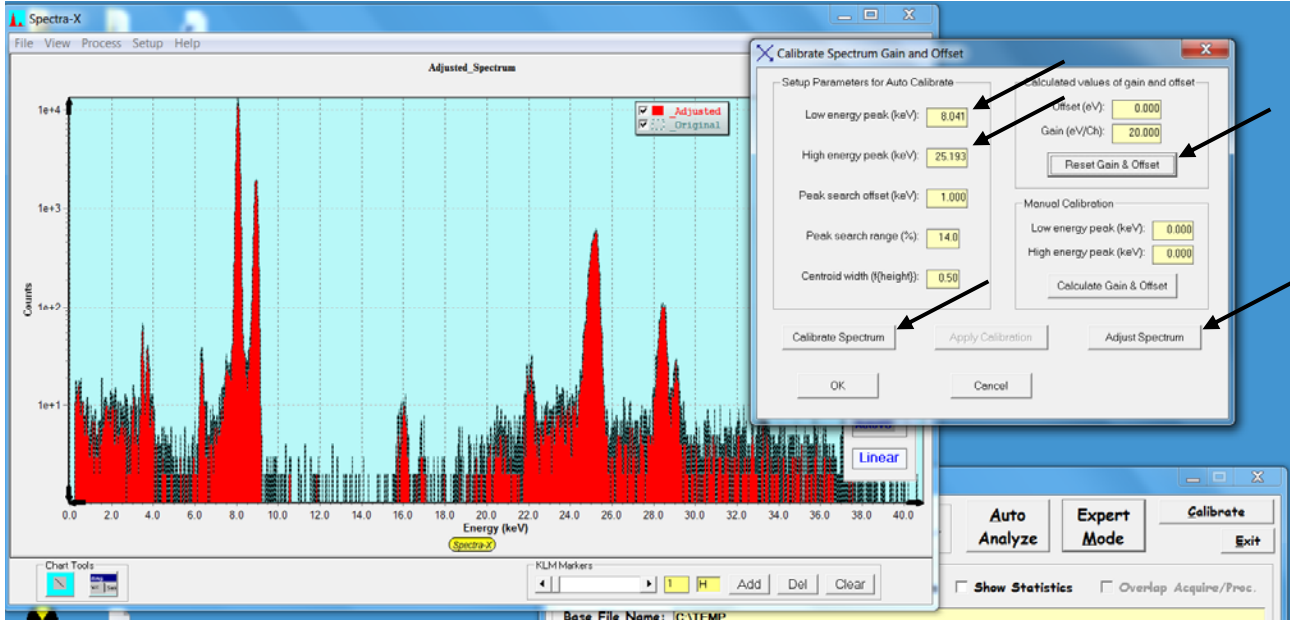
8. Acquire Sample Spectrum

Measure the spectrum to be analyzed

1. Turn on the HV of the X-ray tube by clicking "HV ON" in the Mini-X window. The click "Set HV and current" to set these to 30 kV and 100 A.

2. In XRS-FP, select "Acquire". The spectrum should appear in the blue box and the time will increment in the main screen.

3. When the 30 seconds has elapsed and the acquisition is finished, turn off the X-ray tube. In the main screen, save the file as a .MCA type.
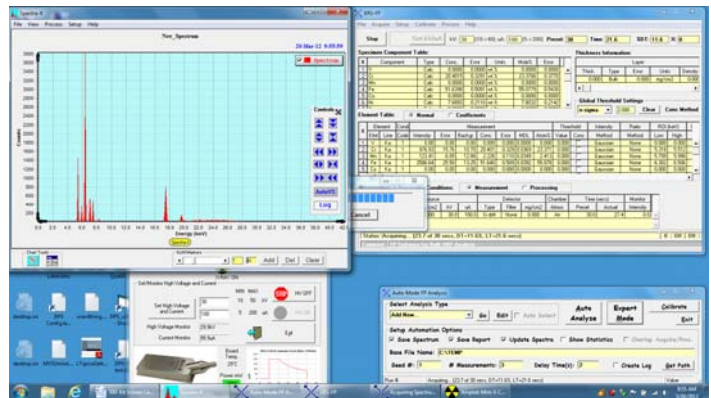
Quick check of spectrum

4. Expand the blue window with the spectrum to full screen. In the lower right, one can select elements and the software will draw lines at the X-ray energies. Move the slider to the elements which are known to be present in SS316: Cr, Fe, Ni, Co, and Mo. Verify the energy calibration by making sure the lines should are near the centroids of the photopeaks. It may be helpful to use the log scale or zoom in.
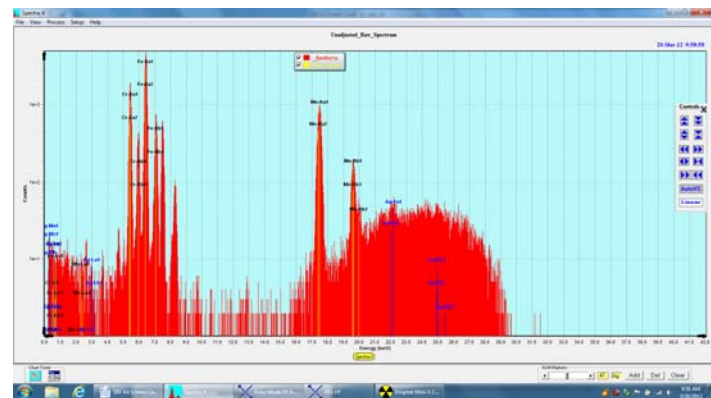
9. Process Spectrum

1. Click "Process," "Spectrum," "Smooth." Observe the result of smoothing in the spectrum window.
2. Click "Process," "Spectrum," "Escape Peaks." Observe the result in the spectrum window. You can see both the corrected spectrum and the escape peak correction.
3. Click "Process," "Spectrum," "PileUp Removal." Observe the result in the spectrum window.   You can see both the corrected spectrum and the sum peak correction.
4. Click "Process," "Spectrum," "Background Removal."   Observe the result in the spectrum window. You can see both the photopeaks and the background that was removed.
5. Click "Process", "Compton Peak". Nothing is visible in the window but values were calculated.
6. Click "Process" "Spectrum" "Deconvolute." It may take many seconds but the result should be as right.
7. The "Element table" will show intensities: the rate of X-rays observed in each photopeak.   This is the output of the processing step.

Deconvolved Spectrum
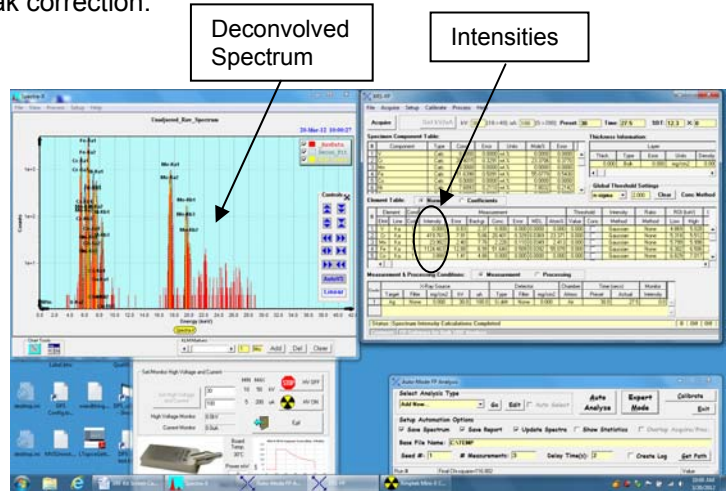
Intensities

Note: The "Process," "All" button will do all of these steps automatically, in sequence, as defined in the "Measurement and Processing Conditions" table. We have done them step by step to show what occurs during processing.

10. Analyze Intensities

1. Click "Process," "Analyze." FP now uses the intensities and applies corrections for matrix effects and attenuations to determine concentrations.
2. The result is shown as "Concentration" in the "Specimen Component Table". By default, these are in units of weight %.
3. Print the report, then save the file. Enter a name for the sample, e.g., "Steel.TFR." The .TFR is a CSV file (in ASCII) which can be read by Excel or Wordpad.

Specimen Component Table:

#	Component	Type	Conc.	Error	Units
2	Cr	Calc	24.814	0.188	wt.%
3	Cu	Calc	1.168	0.039	wt.%
4	Fe	Calc	35.920	0.248	wt.%
5	Mn	Calc	1.689	0.043	wt.%
6	Mo	Calc	1.155	0.012	wt.%
7	Ni	Calc	35.144	0.258	wt.%
8	Pb	Calc	0.028	0.005	wt.%

Element Table: ⊙ Normal   ○ Coefficients

Congratulations!   You have now set up the system and completed a standardless analysis.

11. Improving the analysis

Optimize the configuration

It is very important that the configuration of the system be optimized for each particular application. This includes

- Optimizing the excitation source: the HV bias, the filtering, and the current are all important.
- Optimizing the X-123: the gain, the peaking time, number of channels, and thresholds are very important. There are many other parameters but these are the most vital.
- Optimizing the deconvolution parameters: the "setup processing" tab and the "element table" contain many parameters which affect the accuracy and precision of the analysis results.

Calibrate the analysis parameters

This summary describes "standardless" analysis: the software uses simple models and nominal values to estimate the coefficients which relate the X-ray intensity to the concentrations. Much greater accuracy results
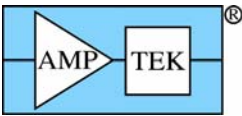
are found by calibrating the analysis coefficients. These are calibrated from spectra measured using standards, reference materials of known compositions.

This is described in more detail in the "Startup Guide" and in the XRS-FP documentation. The first step is to obtain reference materials of known composition with the elements one wishes to analyze. Pure element standards are very helpful. One measures the spectrum (for a relatively long time) for a standard, processes it, then saves the processed .TFR file. Once all the standards are measured, the "Calibrate MLSQ" menu is used to load all of these TFR files and produce a set of calibration coefficients. "Multiple Standards" must be selected under the "FP Calibration Mode" in the "Setup Quant" menu. After all the files are saved, processed, and the calibration completed, the result should be stored as a new TFR file.

12. Learning More

The Installation CD contains a *Documentation* folder which includes user manuals, application notes, software installation guides, and a great deal of information describing the components in the Experimenter's Kit.

  o The *DPPMCA* software's "Help" file includes detailed information on setting the parameters.
  o The *XRS-FP Software Guide* provides detailed information on how to configure the analytical software.

For XRF novices, Amptek has a few application notes which may be helpful. These are all found on the "Links and PDFs" section of Amptek's web site.

  o An X-ray emission line chart, showing the characteristic X-ray energies for each element
  o A "Description of X-ray Fluorescence" and a note "Understanding characteristic X-rays"
  o Three application notes showing the use of Amptek systems in EDXRF (one on RoHS/WEEE materials, one on light elements in water, one on used automotive catalytic converveters)
  o A note describing the processing software, "XRF spectra and analysis software".

Other references include

  o *Principles and Practice of X-ray Spectrometric Analysis*, 2nd Ed, E.P. Bertin, Plenum Press, (1975).
  o *Principles of Quantitative X-Ray Fluorescence Analysis*, R. Tertian, F. Claisse, Heyden & Son Ltd., (1982).
  o *Handbook of X-Ray Spectrometry: Methods and Techniques*, eds. R. van Grieken, A. Markowicz, Marcel Dekker, (1993).
  o *Quantitative X-ray Spectrometry,* 2nd Edition, R. Jenkis, R. Gould, D. Gedcke, Marcel Dekker (1995)
  o *Handbook of Practical X-ray Fluorescence Analysis,* eds. B. Beckhoff, B. Kanngiesser, et al, Springer (2006)
 o http://xdb.lbl.gov/

13. Frequently Asked Questions

Can I just use the default calibrations and settings?

No! There has been no calibration at Amptek. You should expect no meaningful results if you use the configuration in the X-123 when it shipped or the calibration values in the default files.

Is this XRF Experimenter's Kit comparable to a turn-key XRF system?

No. This kit contains all of the critical hardware and software required to do energy dispersive X-ray fluorescence (EDXRF) but it is NOT a turn-key system. To get accurate results requires fabrication of radiation shielding and sample mount hardware, determining the optimum configuration of the hardware and software for your samples, and calibration. Obtaining quality results takes considerable effort.

A turn-key system is generally designed to handle a wide range of measurement applications. If a user has a specific application, particularly one that is challenging for turn-key systems, the Experiment's Kit lets the user optimize the entire system for that one application. It is a very powerful tool for specific and challenging measurement applications. But to obtain its advantages, the user must invest the time to fully optimize the hardware, the software, the calibration procedures, and so on.
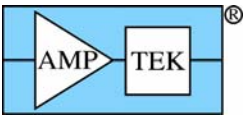
A turn-key system is designed for an operator with minimal training. The operator turns on the system, takes a measurement, and the system gives the answer. The designers of the turn-key system have already optimized the configuration, calibrated the system, and evaluated its measurement uncertainty. The Experimenter's Kit requires a user to carry out these steps and to have the knowledge to carry them out.

Where do I turn if I am having a problem?

There is a "Troubleshooting Guide" in the documentation section of the Installation CD. You can refer to this or to the User Manuals for the various components.