XRS-FP Basic Setup & Analysis

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



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DOCUMENT CHANGE LOG

Date	Person	Pages	Description
10-Mar-14 Sarah Cross		All	Created document
25-Mar-14	Sarah Cross	Most	Updated document
7-Apr-14	Sarah Cross	Many	Updated document
15-May-14	Brian Cross	15	Changed path to find files after installation of XRS-FP. Document v7.



1 QUICK GUIDE: XRS-FP SETUP & ANALYSIS FLOWCHART

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



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

2 XRS-FP SETUP & STANDARDLESS ANALYSIS TUTORIAL

The following tutorial describes the basic setup of a *general* XRF system for analysis, the configuration of XRS-FP, energy calibration using Amptek hardware (although briefly, as this is discussed in detail in the "XRS-FP How to Calibrate with Amptek HW" document) and standardless analysis of stainless steel 316 (SS 316). This tutorial along with associated spectra (*.mca) and application (*.tfr) files guides an XRS-FP 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 spectra (*.mca) and application (*.tfr) files *without* HW to emulate the setup and analysis.

Note: It is not necessary to connect the XRS-FP program to any hardware unless you wish to acquire spectra directly with XRS-FP. For **BOTH** methods listed above, the provided spectra (*.mca) and application (*.tfr) files 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.

 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 <u>safety</u> 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-FP 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-FP 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, the splash screen will load and then the Auto-Mode FP Analysis window and a separate Spectra-X window will be displayed. 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 -> About menu option.
- **3.** From the Auto-Mode FP Analysis window, click **Expert Mode** (Fig. 3 below). This will show the contents of the "Master.tfr" file (see section 4.1 of the "XRS-FP Software Guide" for additional information on loading the software). Ensure the "Update Spectra" option is checked (on) (Fig. 3).

X Auto-Mode FP Analysis		X
Select Analysis Type	Auto Expert	<u>C</u> alibrate
Add New Go Edit Auto Select	Analyze Mode	<u>E</u> xit
Setup Automation Options		
Save Spectrum / Save Report / Update Spectra	Show Statistics Overlap	Acquire/Proc.
Base File Name: C:\TEMP		
Seed #: 1 # Measurements: 3 Delay Time	≥(s): 2	<u>G</u> et Path
Run # Preset Livetime = 20		Value 💋

Figure 3. Auto-Mode FP Analysis Panel. Select the "Expert Mode" button. Ensure the "Update Spectra" option is checked (on).

From the XRS-FP Expert Panel: Select File -> Open -> "C:\CrossRoads Scientific\XRS-FP Basic Setup SS316 Tutorial Files\SS316 - Initial.tfr" (or equivalent location). See Fig. 4 below.

X Open				23
CrossRoads Sc	ientific XRS-FP Basic Set-up SS316 Tutorial Files	▼ 4j	Search XRS-FP Basic S	iet-up SS 🔎
Organize 🔻 New folder				
☆ Favorites	Name	Date modified	Туре	Size
🧮 Desktop	SS316 - Initial.tfr	8/29/2013 1:44 PM	TFR File	4 KB
🐌 Downloads	SS316.tfr	8/29/2013 1:44 PM	TFR File	4 KB
S Dropbox				
Google Drive	=			
Recent Places				
🔁 Libraries				
Documents				
J Music				
Pictures				
Videos				
😽 Homegroup	+ (
F 1			VDE D	
File name:	55310 - Initial.ttr	•	ARE Reports (".ttr)	
			Open 🔻	Cancel

Figure 4. File Open Menu. Open the stainless steel SS316 TFR file, e.g. "SS316.tfr".

This TFR ("Thin Film Report") file, or configuration file, acts as a template for this tutorial. A TFR 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 Component Table, Elements Table and Thickness information have no results at this point, the Measurement & Processing information (spectrometer configuration) is already pre-defined and loaded for this application. See Figure 5 below.



Figure 5. XRS- FP Expert Panel. The "SS316 - Initial.tfr" 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). Also note that in the "Thickness Information" table the "Type" is set to "Bulk" and it is "Normalized" to 100%.

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-FP for use with an XRF system.

a. Specimen Component Table: This is where the sample components are added to the TFR (also see section 5.2, "XRF Elemental FP Calibration or Standardization," of the XRS-FP Software Guide). Components can be elements or compounds. For this tutorial, *enter* Cr as the first component. Having entered a component in the table, use the down arrow



key of your computer keyboard 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. 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 6.

XRS-FP	10.00.0	1000	and the second s							
File Acquire Setup Calibrate Process Help										
Acquire Set kV/uA kV: 45 (10->50) uA: 10 (5->200) Preset: 50 Time: 27.546 \$DT: 0.0 0CR: 0.0										
Specimen Component Table: Thickness Information:										
# Component Type Conc. Error Units Mole% Error Layer Normalize										
Cr Calc 0.0000 0.000	00 wt.% 0.0000 0.0000	 Thick. Type 	Error Units Density Fixe	OK Total						
2 Mn Calc 0.0000 0.000	00 wt.% 0.0000 0.0000	0.000 Bulk	0.000 mg/cm2 0.000	100.00						
4 Ni Calc 0.0000 0.000										
5 Mo Calc 0.0000 0.000	00 wt.% 0.0000 0.0000									
Si Fixed 1.0000 0.000	0.0000 0.0000	Global Threshold S	ettings							
Element Table: C. Namel C. Castilia		n-sigma 💌 2.00	Clear Conc Method							
Element rable. (• Normal () Coerricit	ents									
# Element Conc Measure	ment Thre	eshold Intensity Ratio	ROI(keV) Chi2 Quant	Calibration						
Elmi Line Code Intensity Error Backgr. Co	nc. Error MDL Atom% Value	Conc Method Method	Low High Fit Method	TCC Coeff						
1 Si Ka 1 0.00 0.00 0.00 1	.000 0.000 0.0000 0.000 0.000	Gaussian None	0.000 0.000 0.00 FP	0.00						
2 Cr Ka 1 0.00 0.00 0.00 0		Gaussian None	0.000 0.000 0.00 FP	0.00						
4 Fe Ka 1 0.00 0.00 0.00 0		Gaussian None		0.00						
5 Ni Ka 1 0.00 0.00 0.00 0	.000 0.000 0.0000 0.000 0.000	Gaussian None	0.000 0.000 0.00 FP	0.00						
6 Mo Ka 1 0.00 0.00 0.00 0	.000 0.000 0.0000 0.000 0.000	Gaussian None	0.000 0.000 0.00 FP	0.00						
Measurement & Processing Conditions:	leasurement C Processin	g								
X-Ray Source	Detector Cha	amber Time (secs)	Monitor							
Target Filter mg/cm2 kV uA	Type Filter mg/cm2 Ati	mos Preset Actual	Intensity							
1 Ag Al 539.780 45.0 10.0	Sidrift None 0.000 /	Air 50.0 27.546	0.0							
			-							
5 Status: Updated Element able Loncentration	\$		j U Uff Un 2	048 20 0.39 No 1						
Comment: FP Software for Bulk XRF Analysis										

Figure 6. XRS- FP Expert Panel. The components, Cr, Mn, Fe, Ni, Mo and Si, are now filled in for this application. Notice that the elements are automatically entered as the components are entered and that the highest energy analyte line is also automatically selected as the components are entered (e.g. the Cr Kα line was selected when Cr was entered).

Note: to create a "clean sheet" for future analysis (select **File** -> **New** from the XRS-FP Expert Panel. 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.

b. Element Table: 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. 6 above).

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.

- c. Thickness Table: 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 "Thickness Table" and 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. 6 above). Normalizing to 100% is not required for all bulk analyses, but is often employed.
- 5. From the XRS-FP Expert Panel: Select Setup -> Detector. This is where all the detector properties are set. 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 7. Select "OK" to exit the dialog and save the parameters. Note that the most important parameters involved in the detector setup are the detector material, thickness, resolution and window type.

Detector Properties	Window Properties
Detector Type: 5: Si drift Element: Si Front-Surface Shape © Circle © Square Detector Area (mm2): 5.0 ID (mm): 2.52 Collimator Diameter (mm): 2.40 Area(mm2): 4.5 Detector Thickness (mm): 0.50 Front Contact Material: Al Contact Thickness (nm): 10.0 Dead Layer Thickness (micron): 0.05 Ice Layer (nm): 0.0 Resolution at Mn-Ka (eV): 130.0	Window Type: Be Window Thickness (micron): 12.70 Coating Material: Al Window Coating (nm): 0.0 Carbon Contamination (nm): 0.0 Grid1 Element: Si Grid1 Fraction: 0.00 Grid1 Thickness (micron): 0.0 Grid2 Element: Grid2 Fraction: 0.00 Grid2 Thickness (micron): 0.00
Set Defaults OK	Set Defaults Cancel

Figure 7. Setup Detector & Window Parameters Dialog. Check that the "Detector Type," "Window Type," and all corresponding parameters are as shown here, unless HW is being used with this tutorial.

6. Now, from the XRS-FP Expert Panel: Select Setup -> Tube/Source. 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. Select "OK" to exit the dialog and save the parameters.



Figure 8. Setup Tube Parameters Dialog. Check that the "Source Type," "Target Element," "Window Type" and all corresponding parameters are as shown above, unless HW is being used with this tutorial. The four different source types are illustrated here, a) side window tube, b) end window tube, c) transmission target tube and d) radioisotope.

Figure 8 displays schematic diagrams of the four source types - side window tube, end window tube, transmission target tube and radioisotope source - that can be selected from the XRS-FP **Setup** -> **Tube/Source** dialog. The data acquired for this tutorial used a transmission target tube. The source angle parameters and all other associated geometry of the XRF system are very important and will be discussed further in step 7 in more detail.

7. Again from the XRS-FP Expert Panel: Select Setup -> Geometry. This is where all the angle and distance 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 9. 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. Select "OK" to exit the dialog and save the parameters.

X Setup Geometry
Tube-Sample Incidence Angle: 45.0 °
Secondary Incidence Angle: 45.0 °
Take-Off Angle: 45.0 °
Alpha Angle: 0.0 °
Scatter Angle: 90.0 °
Tube-to-Sample (mm): 50.0
Tube-to-Optic (mm): 0.0
Optic Length (mm): 0.0
Optic-to-Sample (mm): 0.0
Sample-to-Detector (mm): 15.0
OK Cancel

Figure 9. Setup Geometry Dialog. Check that the angle and distance parameters are as shown here, unless HW is being used with this tutorial. See Figures 10 and 11 below for explanation of the various angle and distance parameters.

Figures 10 and 11 below show schematic diagrams of the various angle and distance parameters involved in the geometry setup of an XRF system.



Blue Arrow = X-rays (Source to Target); Green Arrow = X-rays (Target to Sample); Green Dashed Arrow = Transmitted X-rays; Red Arrow = Characteristic X-rays (Sample to Detector)



Figure 10. Geometry - Angles Schematic. All angle parameters listed for the geometry setup are described here.



Figure 11. Geometry - Distances Schematic. All distance parameters listed for the geometry setup are described here.

8. From the XRS-FP Expert Panel: Select Setup -> MCA/DPP. If working offline (without HW) just enter the number of channels, with the eV per channel or range (keV). If working with HW please see the following document for stepwise details on performing an energy calibration in XRS-FP (with Amptek HW): "XRS-FP 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 12 shows the Amptek DPP/MCA Acquisition Setup parameters.



Amptek DPP/MCA Acquisition Setup	
Connected: No	COM Port (1 or 2): 1 ect USB Port: Ethernet Select: Addr: 0
Acquire Options Preset Mode: LiveTime(: v Auto Adjust Spectrum during Acquire	Preset Value: 50 e 🔽 Preclear on Acquire: 🔽
Spectrum ROI for Preset Counts Moo ROI Low (keV): 1.000 Preset Integral (Cour	le ROI High (keV): <mark>10.000</mark> nts): <u>0</u>
Slow Threshold: 0.59 Fast Threshold: 5.25 Peaking Time (uS): 0.8 Flat-Top Time (uS): 0.20	No. Channels: 2048 eV/Ch: 20 Range (keV): 40 Gain:Calibrate
Clock 80	jection 🗖 Rise-Time Discriminator Config File Read Config from DPP
Device Serial # DPP: Firmware FPGA TEC: DPP Config File HV	Configure DPP Exit

Figure 12. Setup MCA/DPP Dialog. Amptek DPP/MCA Acquisition Setup showing the No. Channels, eV/Ch and Range.

9. Now select: Setup -> Processing from the XRS-FP Expert Panel. This will display the "Setup Processing Parameters" Dialog shown below in Figure 13 (see section 14.3, "Process Spectrum," of the XRS-FP Software Guide for more detail). Note that spectra supplied for this tutorial have been energy adjusted (see section 12.11, "Setup Spectrum Adjust" of the XRS-FP Software Guide). To avoid doing a double adjust in this case, verify that the "Auto Adjust Spectrum Gain & Offset on Load" is unchecked in the "Setup Processing Parameters" dialog. See Figure 13 below. Note: if you are using your own spectra, you may need to change the Mn-Ka FWHM to a higher value.

Setup Processing Parameters
Gaussian Deconvolution Parameters
✓ Use Nonlinear Deconvolution
Maximum Allowed Deviations for Nonlinear Deconvolution
Maximum # Iterations: 5
Line Ratio Factor: 2.0
Spectrometer Offset (eV): 100.0
Spectrometer Gain (%): 1.00
Spectrometer Peak Width (%): 20.0
Channel Weighting Factor: 1
Use Net Spectrum Weighting 🔽
Peak Width (FWHM) at Mn-Ka (eV): 130.0
Auto Calibration from Nonlinear Deconvolution
Use Auto Calibration Mn FWHM (eV): 0.0
Gain Factor: 0.0000 Offset (eV): 0.0
Spectrum Smoothing Parameters
Filter Type
Savitsky-Golay (* Gaussian (* Topnat (* Average
Number of Points © 3 0 5 0 7 0 9 0 11
Auto Background Removal Parameters Low-Pass Filter Width: 250 Max # Iterations: 30
Pile-Up (Sum Peak) Removal Parameters
Pulse-Pair Resolution (uS): 0.40 Time Constant (uS): 0.80
Spectrum Low-Energy Start (keV): 0.50 Lowest Z: 12 (Mg) Escape
Auto Adjust Spectrum Gain & Offset on Load
UK Lancel Defaults

Figure 13. Setup Processing Parameters Dialog. Observe that the "Auto Adjust Spectrum Gain & Offset on Load" button is unchecked if using the files supplied with the SS316 tutorial, as the spectra have already been energy adjusted.

10. Then select: Setup -> Quant from the XRS-FP Expert Panel. Check that the radio button for the "Quant Analysis Method" is set to "Fundamental Parameters" (FP) and the "FP Calibration Mode" is set to "Standardless" (see Fig. 14 below).

📉 Setup F	P Calibration Method	X
	Quant Analysis Method Fundamental Parameters FP with Scatter Ratios Simple Least-Square Fitting	FP Calibration Mode Standardless One Standard Multiple Standards
SIR-FP	File Name:OK	Cancel

Figure 14. Setup Quant Dialog. Select the "Fundamental Parameters" method and the "Standardless" mode.

- 11. At this point our application, stainless steel 316, has been defined, XRS-FP 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 12 below. If using hardware to do a live acquisition, ensure that the DPP is connected and *now* turn the source x-rays on (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. Select File > Save As from the XRS-FP Expert Panel and save the spectrum as, for example, "SS316.mca" (or equivalent).
- 12. The next step is to extract intensities from the stainless steel 316 spectrum (*mca file). Select File -> Open ->"C:\CrossRoads Scientific\XRS-FP\Basic Setup Tutorial\SS316.mca" (or equivalent location). See Figures 15 and 16 below. Note that the spectra provided for this tutorial are already adjusted and in calibration.

X Open				23
CrossRoads Sci	entific XRS-FP Basic Set-up SS316 Tutorial Files	▼ 4⁄7	Search XRS-FP Basic S	et-up SS 🔎
Organize 🔻 New folder				
Stropbox	^ Name	Date modified	Туре	Size
Google Drive Recent Places	SS316.mca	7/31/2013 6:36 AM	MCA File	16 KB
 ➢ Libraries ➢ Documents J Music ➢ Pictures ☑ Videos ở Homegroup ➢ Computer 	E			
DVD RW Drive (E:) Audio C	• •			•
File name:	SS316.mca	•	Amptek Spectra (*.mca Open 🛛 🗸) Cancel

Figure15. File Open Menu. Open the stainless steel spectrum, *.mca file (e.g. SS316.mca).



Figure16. Spectra-X Window. Loaded stainless steel mca file (SS316.mca).

13. To extract intensities from the stainless steel 316 spectrum, select Process -> Spectrum -> All, from the XRS-FP Expert Panel. Notice that there are now intensities displayed for each element in the "Element Table" (see Fig. 17 below).

	S-FP	1.444	-								10.1					120		X	
File	Acquire	Setup	Calibrate	e Proc	cess Hel	р													٦
Acquire Set kV/uA kV: 45 (10->50) uA: 10 (5->200) Preset: 0 Time: 27.546 &DT: 0.0 ICR: 0.0 OCR: 0.0																			
Specimen Component Table: Thickness Information:																			
#	Compo	nent	Туре	Con	c. Erro	ır Unit	s Mole	% Err	or				Layer	r			Norm	alize	
1 Cr			Calc	0.0	000 0.0	000 wt.%	0.0	000 0.0	000	-	Thick.	Туре	Error	Unit	s [Density Fix	ed OK	Total	
2 M	n		Calc	0.0	000 0.0	100 wt.% 100 wt.%	0.0	000 0.0	000		0.00	0 Bulk	0.000	mg/ci	m2	0.000		100.00	-
4 N	; i		Calc	0.0	000 0.0	000 wt.%	0.0	000 0.0	1000										- 1
5 M	0		Calc	0.0	000 0.0	000 wt.%	0.0	000 0.0	1000		<u></u>								
6 Si			Fixed	1.0	000 0.0	000 wt.%	0.0	000 0.0	1000	-	Global	I hreshold :	ettings	1	<u> </u>		_		
Elem	ent Table		Normal		Coeffic	ients					<mark>n-sigma</mark>	2.0		ear	Lonc	Method			
	Element	Conc 🥖			Measu	rement			Thresh	old	Intensity	Ratio	ROI (keV)	Chi2	Quant	Calibration		
EI EI	ml Line	Cod Int	ensity	Error	Backgr. 0	Conc. Err	or MDL	Atom%	Value C	onc	Method	Method	Low	High	Fit	Method	TCC Coeff	1	
1 9	ii Ka	1	0.068	0.73	1.16	1.000 0.0	0000.0000	0.000	0.000		Gaussian	None	1.691	1.788	0.01	FP	0.00		-
20	∫r Ka	4	58.899	12.43	4.47	0.000 0.0	0000.0000	0.000	0.000		Gaussian	None	5.330	5.490	0.00	FP	0.00	김 -	
3 M	n Ka	12	56.421	4.74	6.17			0.000			Gaussian	None	5.809	5.977 6.495	0.04	FP FP	0.00	4	
5 N	e Ka li Ka	1 1	67.653	7.89	2.61	0.000 0.0	000 0.0000	0.000	0.000		Gaussian	None	7.374	7.566	0.00	FP	0.00	ή l	
6 M	lo Ka	1 4	80.103	12.90	4.39	0.000 0.0	0000.0000	0.000	0.000		Gaussian	None	17.245	17.609	0.01	FP	0.00	j .	-
Meas	urement	E Pro	Co	ndition	s: 🔎	Measurer	nent	C Proce	essing										
		X	Ray Sourc	се			Detector		Chamb	per	Time (s	secs)	Monitor						
_ode	Target	Filter	mg/cm	2 kV	/ uA	Туре	Filter	mg/cm2	Atmo	s	Preset	Actual	Intensity						
1	Ag	Al	539.78	0 45	5.0 10.0) Si drift	None	0.000	Air		0.0	27.546	0.0	_					
														-					
Sta	itus: Spe	trum In	tensity C	alculat	ions Com	pleted				_				() Of	F On 2	048 20 0.	59 No 1	1
Con	nment: F	P Softwa	are for B	ulk XRI	F Analysis	3								,					
										_									

Figure 17. XRS- FP Expert Panel. Note that intensities are now displayed for each element in the "Element Table."

14. Then select: **Process** -> **Analyze** from the XRS-FP Expert Panel. Notice that there are now concentrations displayed for each component in the "Component Table" (see Fig. 18 below).

XRS-FP		1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1					
File Acquire Setup Calibrate Process Help	1						
Acquire Set kV/uA kV: 45 (10->50) uA: 10 (5->200) Preset: 0 Time: 27.546 %DT: 0.0 ICR: 0.0 OCR: 0.0							
Specimen Component Table: Thickness Information:							
# Component Type Conc. Error	Units Mole% Error	Laye	r Normalize				
1 Cr Calc 18.7455 0.50	79 wt.% 19.9768 0.5413	Thick. Type Error	Units Density Fixed OK Total				
2 Mn Calc 2.1311 0.17	90 wt.% 2.1494 0.1806	0.000 Bulk 0.000	mg/cm2 0.000 🔽 🔽 100.00				
4 Ni Calc 11.9686 0.56	33 wt.% 11.2994 0.5318						
5 Mo Calc 2.5078 0.06	74 wt.% 1.4484 0.0389						
6 Si Fixed 1.0007 0.0000 wt.% 1.9730 0.0000 U							
Element Table: Coefficients							
Element Conc Measur	ement Threshold	Intensity Ratio ROI	(keV) Chi2 Quant Calibration				
# ElmI Line Code Intensity Error Backgr. C	onc. Error MDL Atom% Value Conc	Method Method Low	High Fit Method TCC Coeff				
1 Si Ka 1 0.068 0.73 1.16	.000 0.000 6.5224 1.973 0.000	Gaussian None 1.691	1.788 0.01 None 0.00				
2 Cr Ka 1 458.899 12.43 4.47 18		Gaussian None 5.330	5.490 0.00 FP 0.00				
3 Mn Ka 1 56.421 4.74 6.17 2		Gaussian None 5.809	5.977 0.04 FP 0.00				
5 Ni Ka 1 167,653 7,89 2,61 1	969 0.563 0.0476 11 299 0.000	Gaussian None 7.374	7,566 0.01 FP 0.00				
6 Mo Ka 1 480.103 12.90 4.39	2.508 0.067 0.0045 1.448 0.000	Gaussian None 17.245	17.609 0.01 FP 0.00				
Measurement & Processing Conditions: Measurement C Processing							
X-Ray Source	Detector Chamber	Time (secs) Monitor	T				
Target Filter mg/cm2 kV uA	Type Filter mg/cm2 Atmos	Preset Actual Intensity	1				
1 Ag Al 539.780 45.0 10.0	Sidrift None 0.000 Air	0.0 27.546 0.0					
Status: Completed FP Quantitative Analysis 0 Off 0 n 2048 20 0.59 No 1							
Comment: FP Software for Bulk XRF Analysis							

Figure 18. XRS- FP Expert Panel. Observe that the concentration for each component is now displayed in the "Specimen Component Table."

15. Now save the TFR file by selecting File -> Save As from the XRS-FP Expert Panel. This can be saved as "C:\CrossRoads Scientific\XRS-FP Basic Setup SS316 Tutorial Files\SS316-StandardlessAnalysis.mca" or as an alternate filename of choice. The analysis results should be similar to those shown below in Figure 19.

Layer # Thi 1 0.0	r Tabi ick)0	le Type Bulk	Ern 0.(or Un)0 mg	its D /cm2	ensity 0.00	Norm. To On 10	otal 00.00				
Sampl	le Tal	ble —										
Layer	r Cor	nponer	nt	Type	Concn	. Error	Units	Mole%	Error			
1	Cr			Calc	18.74	5 0.508	wt.%	19.97	7 0.541			
1	Mn			Calc	2.13	1 0.179	wt.%	2.14	9 0.181			
1	Fe			Calc	63.64	7 1.035	wt.%	63.15	3 1.027			
1	Ni			Calc	11.96	9 0.563	wt.%	11.29	9 0.532			
1	Mo			Calc	2.50	8 0.067	wt.%	1.44	8 0.039			
1	Si			Fixed	1.00	0 0.000	wt.%	1.97	3 0.000			
Eleme	ent Ta	able -										
Elmt	Line	Cond	Ratio	Thte	nsitv	Error	Intensit	V Conc	Conc	Calibr	ation	
	Code	Code	Metho	d (c	/s)	(c/s)	Method	1 001101	Method	Coeffi	cient	
Si	Ka	1	None	0.06	8	0.7328	Gaussian	1.000	None	0.000		
Cr	Ka	1	None	458.	899	12.4342	Gaussian	18.745	FP	0.000		
Mn	Ka	1	None	56.4	21	4.7401	Gaussian	1 2.131	FP	0.000		
Fe	Ka	1	None	1273	.657	20.7086	Gaussian	63.647	FP	0.000)	
Ni	Ka	1	None	167.	653	7.8904	Gaussian	11.969	FP	0.000		
Mo	Ka	1	None	480.	104	12.8983	Gaussiar	1 2.508	FP	0.000)	
Analy	ysis (Condit	ions									
# Tar	rg I	filtei		Thick.	κV	uA	Detecto	or 1	hick. A	tmos	Preset	Actual
				mg/cm2	45.0	10.0.0	ype Fi	liter m	ig/cm2		Time(s)	Time(s)
1 Ag		AL		539.78	45.0	10.0 51	drift No	one u	0.0 A	ır	0.0	27.5
Processing Conditions # No. Escape Sum Back C/R BlankBlank Smths Peaks Peaks Type Ratio RemFile 1 2 Yes Yes Auto No No												

Figure 19. SS316 Analysis Results. Concentrations for the five calculated components are shown here.

16. For comparison, Figure 20 below shows average compositions for SS316.

Stainless Steels Chromium-Nickel-Molybdenum

Types 316 (S31600), 316L (S31603), 317 (S31700), 317L (S31703)

316/316L Stainless Steel Chemical Composition

Represented by ASTM A240 and ASME SA-240

	Percent by Weight					
Element	Maximum Unless Range is Specified					
	316	316L				
Carbon	0.08	0.030				
Manganese	2.00	2.00				
Silicon	0.75	0.75				
Character	16.00	16.00				
Chromium	18.00	18.00				
Nickal	10.00	10.00				
Inckei	14.00	14.00				
Molybdanum	2.00	2.00				
holybuenum	3.00	3.00				
Phosphorus	0.045	0.045				
Sulfur	0.030	0.030				
Nitrogen	0.10	0.10				
Iron	Bal.	Bal.				

Figure 20. SS316 Average Composition. Average composition for SS316 is shown here.



3 APPENDIX

XRS-FP 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 21 below (for more detail see the XRS-FP Software Guide v51X, section 2 "About X-ray



Figure 21. 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.

Fluorescence"). 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-FP, 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-FP 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 8, 7, Vista, XP, 98, etc.), with 512 MB RAM of memory.

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. 22 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



Figure 22. 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.

detector. It is extremely important to know the geometry of your system and details about the components before setting up the XRS-FP 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).

