030.0006.02.2

# User Guide

## PXRF

For use with Tracer III-V$^{+}$, Tracer III-SD, and Tracer IV-SD

# Table of Contents

# Overview

The PXRF software is used to record a spectrum, which can be saved to the PXRF library for comparison to other spectra. The settings on the instrument (tube, voltage, and filter) can be optimized to accommodate individual projects in order to better fluoresce the elements of interest and to consider the matrix density. This user guide describes procedures for changing those settings, as well as an overview of the software itself.

Before beginning, install the PXRF program on your PC.

# PXRF Basics

When the PXRF program is initiated, the program opens to a window similar to that in Figure 1.

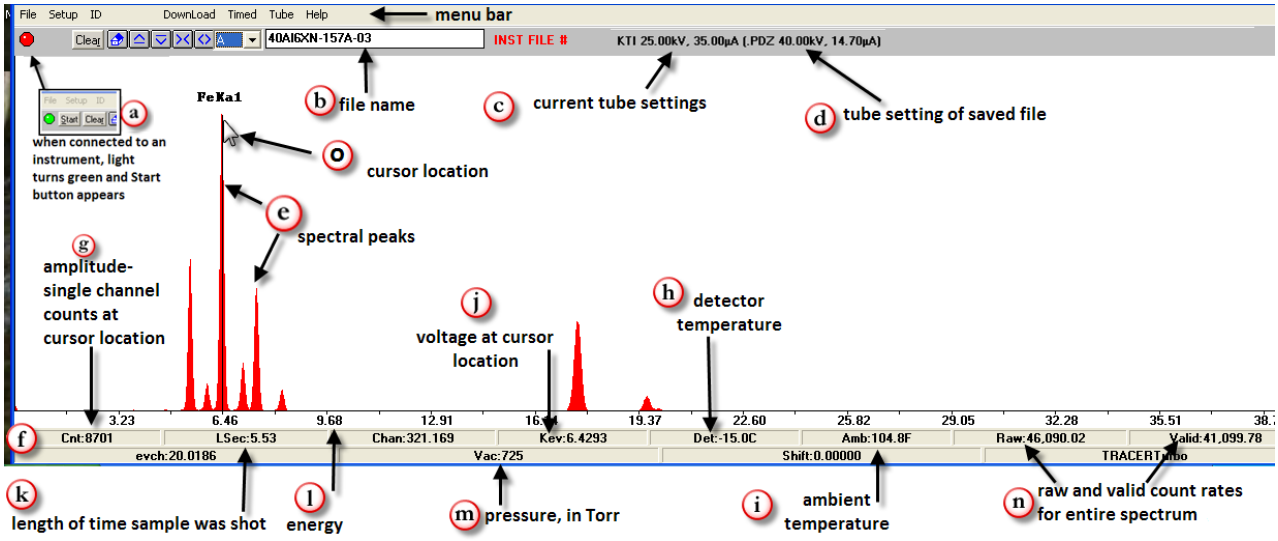



**Figure 1 PXRF**

a) **Start/Stop indicator**. Once the Tracer instrument is connected to the computer, click on the red button. If a connection is made, the light will turn green and the *Start* button will appear. If the light does not turn green, the software may not have been properly configured to the instrument. Refer to *Instrument Setup* on page 4.

b) The name of the **currently open file** appears in the file field. When first opened, the window will show the last saved or opened file. The series of buttons ( ) expands, shrinks, compresses, and broadens the spectra for easier viewing. The icon restores the spectrum to its original dimensions. The dropdown window removes or adds the background or foreground spectra.

c) When the Tracer instrument is on and connected to PXRF, the instrument's **current tube settings** will appear.

d) The **tube settings** used to record the current file (see b above) will appear in parenthesis.

e) When a file is open, or a sample is being analyzed, **spectra peaks** appear.

f) The **data bar** provides information about the spectrum. The readings are sometimes dependent upon where on the spectrum the cursor has been placed.
g) The **count rate** shows the amplitude at the point where the cursor has been placed.
h) The **detector temperature** (in Celsius) is displayed. This setting should not change. If it does change, the instrument may be damaged. **Turn off the Tracer and contact Bruker immediately**.
i) **Ambient temperature** (in Fahrenheit) shows the temperature internal to the Tracer. Although variable, the temperature generally should be in a range between 70 °F to 115 °F.
j) **Kev** is the **voltage** at cursor location. The reading will change depending upon the location of the cursor.
k) **LSec** is the **length of time** the sample was analyzed. If shooting a sample, the reading is the real time length of analysis. When collecting a timed assay (see page 7), the label will change to **RSec** and the timer will count down to zero.
l) The x-axis records **energy,** measured in Kev.
m) **Vac** shows the **pressure** in Torr within the nose of the instrument (between window and detector). When under vacuum, the value should be under 10.
n) **Raw** and **valid** count rates are recorded. Generally, the ratio should be less than 3:2 (raw:valid).
o) **Cursor location**. Placing the arrow in the spectral display and clicking the left mouse button will move the cursor to that location. In addition, the ←→keys on the keyboard, the location may be moved one channel at a time.

# File

The File tab is located on the Main Menu Bar. The File options are essentially the same as those in most Windows applications, with one exception: *PDZ Preview*. The PDZ Preview button opens a window of the stored PDZ (PXRF) files. By using the ↑↓ keys, you can preview each spectrum file. The function is especially helpful when comparing stored files to the current spectrum (see *Spectrum Overlay*, page 5).

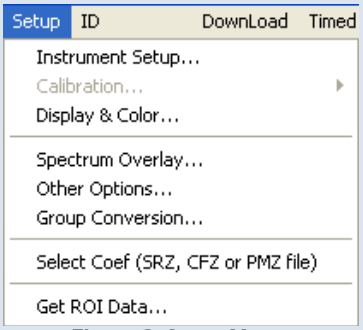


**Figure 2 Setup Menu**

# Setup

The Setup tab (Figure 2) provides several options to reconfigure the way information is displayed.

## Instrument Setup

*Instrument Setup* allows the user to reset basic instrument settings. Standard settings for the Tracer III-V$^+$, Tracer III-SD, and Tracer IV-SD are shown in Figure 3.

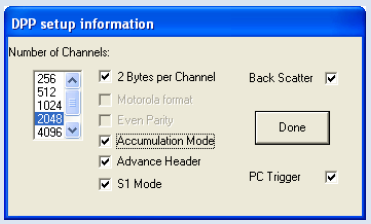


**Figure 3 Standard Settings**

## Display and Color

*Display and Color* (Figure 4) allows the user to change the appearance of the spectra. The radio buttons are used to select the item. When the *Color* button is selected, a color chart appears, providing color options. Selecting the *Element Name* allows the user to change both the color and the font. *Flood Fill* toggles the display for the primary (A) spectrum between a solid color and an outline.

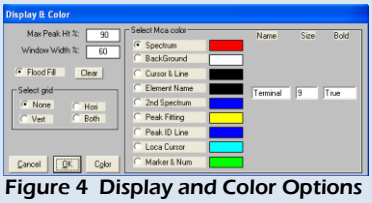



Figure 4 Display and Color Options

## Spectrum Overlay

*Spectrum Overlay* (Figure 5) enables the user to display two spectra at the same time.


Figure 5
Spectrum Overlay

Once an initial spectrum has been selected, select *Move A>>B* to place the spectrum as the background. Then select a second spectrum. If the two spectra are similar, use the + and – buttons to move lines of the overlying spectrum up or down for easier viewing.

*Normalize* adjusts the two spectra so that the ratios conform to each other (e.g., two spectra may have been shot over different time intervals and so have different count intensities; *Normalize* adjusts the spectra to match at the point of the cursor, so that the ratios can be compared). Generally, the user will click on the Compton peak and then click *Normalize*.

## Other Options

*Other Options* (Figure 6) includes a *Warning Beep* check box that enables/disables the audible alert, signifying that a timed assay is complete. Enter the *Start Kev for Compton* and *End Kev for Compton* to produce a Compton range that will be labeled on subsequent saved PDZ files. By default, L-alpha lines are used; if you wish to use L-beta labels, check the element box at the bottom of the window.

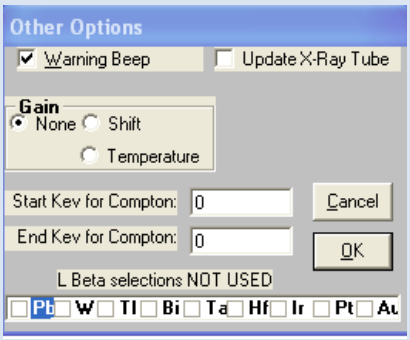



Figure 6 Other Options...

## Group Conversion

The *Group Conversion* option (Figure 7) allows the user to change the data format in an entire folder. Select a *PDZ Name* from the folder you wish to process, check the appropriate *Output* boxes, and then select which settings should be modified. Spectra may be compressed or expanded by selecting the appropriate radio button. Clicking on *Execute PDZ* will start the conversion of all PDZ files located in that directory. Selecting *Done* will exit without executing the command.

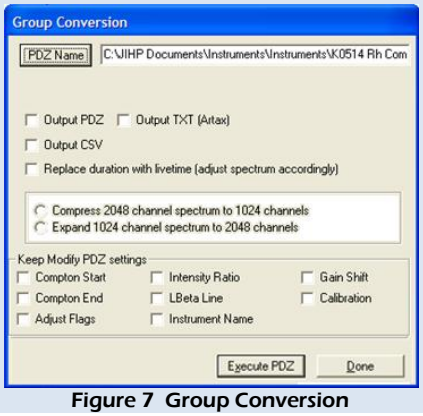



Figure 7 Group Conversion

PDZ files can also be converted to text (.txt) files. To convert files, select *Group Conversion* from the *Setup* tab. Click the *PDZ Name button* (Figure 7) and then select the PDZ folder you wish to convert. Check the *Output TXT (Artax)* and *Replace duration with livetime [adjust spectrum accordingly]* checkboxes. This box executes a command to correct all spectrum data to the instrument live time rather than real time, allowing direct

comparison of one spectrum to another in absolute terms. When this box is checked, the *FWHM* (Full Width Half Maximum) and *ELin* (Energy Line) fields will appear (Figure 8). Enter the correct information in the *FWHM* field (for PIN diode, the FWHM is typically 180 eV; for SDD systems, it is 150 eV). The ELin field may be left blank (the number is read from data stored with each spectrum file; however, it is typically 0.02 KeV for an SDD system, and 0.04 KeV for a PIN diode system). Click the Execute PDZ button to convert ALL spectra in the folder (the conversion will create files in the same folder with the identical name as the source file, but with a .txt extension). The .txt files can be analyzed using the Artax software, described in a separate user guide.

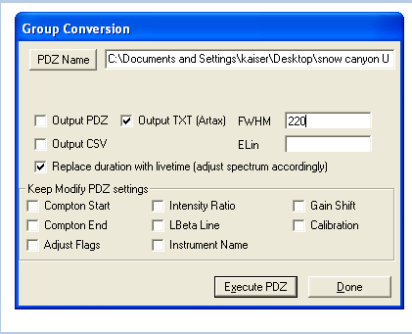



**Figure 8 FWHM and ELin fields**

*Caution! Once the file has been converted, it cannot be reconverted!*

**Select Coef (SRZ, CFZ, or PMZ file)**

See Quantification, page 12.

**Get ROI Data**

*Get ROI Data* displays the intensity of energy of the elemental channel range for the identified peak(s). Use of this feature is explained in the next section.

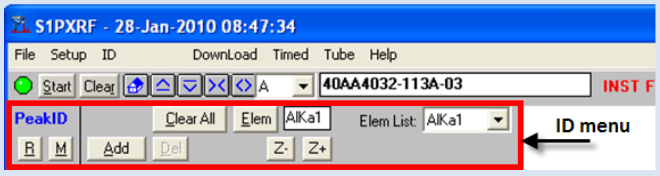
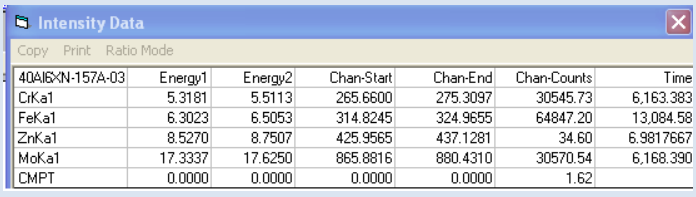



**Figure 9 ID Window**

## ID

Selecting the *ID* tab opens the *ID menu* (Figure 9). The ID Window is used to identify element lines and add labels. The R M buttons **R**emove the menu, or **M**oves the menu. To move, click on the *M* and then click again on the screen. The menu will move to that place. Select *Elem* to open the periodic table and choose an element of interest, as well as the K, L, or M line (see Appendix B). All selected elements will appear in the Elem List. *Z- and Z+* will scroll to the previous or subsequent element on the periodic table (at the K, L, or M line, as selected). As the elements are chosen, their spectral peaks will show on the spectrum. Select the element of interest and click *Add*. Right click on the element in the periodic table to open a window containing additional information, including energy and counts.




| 40AI6XN-157A-03 | Energy1 | Energy2 | Chan-Start | Chan-End | Chan-Counts | Time |
|---|---|---|---|---|---|---|
| CrKa1 | 5.3181 | 5.5113 | 265.6600 | 275.3097 | 30545.73 | 6,163.383 |
| FeKa1 | 6.3023 | 6.5053 | 314.8245 | 324.9655 | 64847.20 | 13,084.58 |
| ZnKa1 | 8.5270 | 8.7507 | 425.9565 | 437.1281 | 34.60 | 6.9817667 |
| MoKa1 | 17.3337 | 17.6250 | 865.8816 | 880.4310 | 30570.54 | 6,168.390 |
| CMPT | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 1.62 | |

**Figure 10 Intensity Data Window**

Once elements have been added to the spectrum, it is possible to receive quantitative information about the peaks. Select *Get ROI Data* from the *Setup* tab to open the *Intensity Data* window (Figure 10). For each element, the Energy and Channel

limits are displayed (*Energy 1, Energy 2, Chan-Start*, and *Chan-End*). *Chan-Counts* records the total counts in the region of interest (ROI) for the entire collection time. The *Time* column displays the ratios selected from the *Ratio Mode* tab. The Ratio Mode options include *Time* (counts divided by the acquisition time or counts per second); *Compton* (counts divided by the total counts in the Compton Peak); or *ROI* (counts divided by the total counts in the spectrum).

# Download

The download tab allows the user to set the Communication Port and Baud Rate. See Connect the Instrument (page 8), steps 7 and 8

# Timed

### Timed Assay

*Timed Assay* (Figure 11) is used to set up the instrument to take one or more assays of a sample for a predetermined length of time. Select the *Multiple Runs* box to collect a series of spectra with the same base file name (the name will be incremented for each spectrum). The data will be saved by *File Type*, determined by the checkboxes selected. Selecting the *Autosave* box will tell the instrument to save each assay of a multiple run; if *Autosave* is not selected, the files will be displayed but NOT be saved unless you save the spectrum manually. The file name is created manually for each run. The *FWHM* box is used with the Artax software to define the instrument resolution. When OK is clicked, the software will direct the user to choose a folder in which to save the file. When performing multiple runs, the file name will auto-increment. For more information, see Timed Assays, page 10.

### Auto

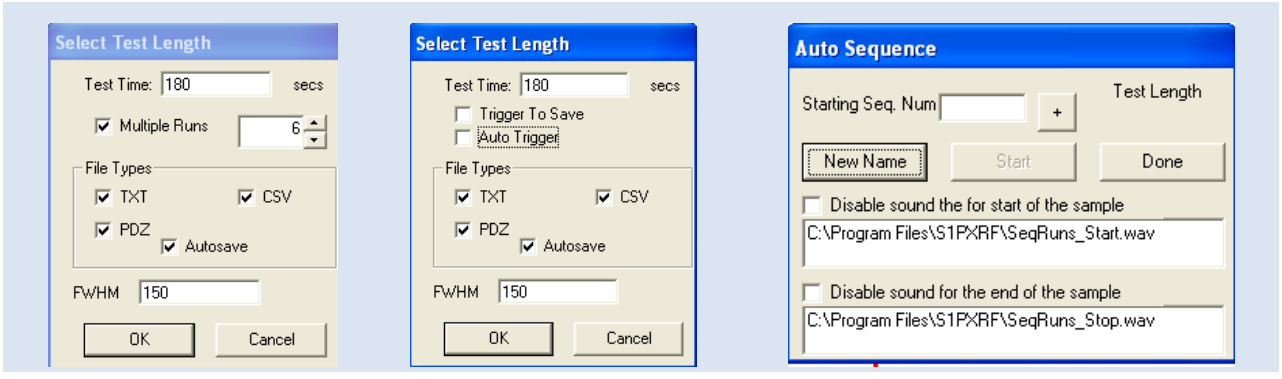


Figure 11 Time Assay Tab Figure 12 Auto Figure 13 Auto Sequence

*Auto* (Figure 12) allows the user to auto-increment the file names for each sample. To determine manually the length of the assay time, select *Trigger to Save.* To allow the software to run for the preset *Test Time*, select *Auto Trigger*. Files can be saved as TXT, CSV, or PDZ files. Once the folder has been selected, the *Auto Sequence* window (Figure 13) pops up. Enter the starting number for the sequence. After each assay is saved, using the + will increase or decrease the starting number for the next assay. *New Name* restores the defaults and allows the user to rename and configure the next assay. If *Trigger to Save* was selected, **Test Length** will appear in the upper left of the window (as in Figure 13). If *Auto Trigger* was selected, the predetermined **Test Time** will appear.

# Tube

See Appendix A.

To open the tube-setting window, turn the instrument on and connect to PXRF. Select the Tube tab from the Main menu, then select KTI Tube > Read.

# Recording Spectra

In order to characterize a sample, its spectrum must first be recorded. To record a new spectrum:

## Connect the Instrument

1. **ATTACH** the TRACER to your PC using the provided Serial/USB cable (4-foot gray cable).
2. **INSERT** the TRACER's battery OR **REMOVE** the battery and **CONNECT** the provided A/C power adaptor to the base of the instrument (Figure 14).
3. **PLACE** the TRACER in the instrument stand.
4. **TURN** the power key to the *ON* position.
5. **OPEN** the PXRF program from the *Start* menu of your PC.
6. **PLACE** the sample on the instrument.
   a. **ENSURE** that the sample completely covers the IR sensor. If the sample does not cover the IR sensor, **USE** the optional small sample table.
   b. **ENSURE** that the sample is flush on the face of the instrument with no gaps.
   c. **ENSURE** that the sample is centered on the aperture.

*Settings in steps 7 through 10 will become the default settings.*

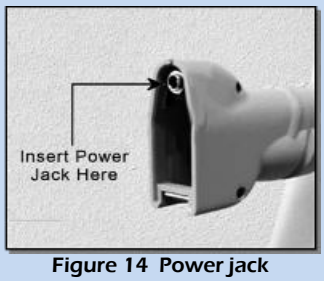


Figure 14 Power jack


Figure 15 Small-sample table

Figure 16 Download Menu

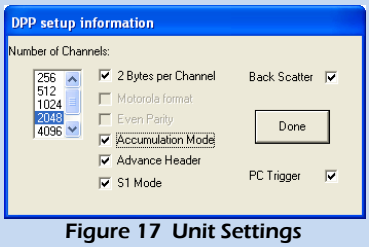


Figure 17 Unit Settings

7. **SELECT** the correct Com Port from the *Download* menu (Figure 16).
8. **SELECT** the correct baud rate from the *Download* menu (115200).
9. **OPEN** the *Setup* menu and **SELECT** Instrument Setup.
10. **ENSURE** the following settings (see Figure 17):
    a. Number of Channels (2048)
    b. 2 Bytes per Channel box is checked
    c. Accumulation Mode is checked
    d. Advance Header is checked
    e. S1 Mode is checked
    f. Back Scatter is checked (this is a safety feature which shuts off the X-ray if there is no sample in place)
    g. PC Trigger is checked (this allows the user to click the Start button in PXRF rather than having to manually pull the trigger to activate the X-rays). If communication to the instrument is broken, the PC Trigger box must be re-checked after communication is re-established.
11. **CLICK** the red button on the navigation bar to initiate communication between the PC and the device. If button is green, communication has been established.

## Collect a Spectrum

12. **SELECT** *Tube* from the menu bar and then *KTI Tube > Read*.
13. **ENSURE** that the Tube voltage, current, and filter settings are appropriate for the sample of interest (see X-Ray Tube Voltage/Current and Filter Control, page 13.)
14. From the PXRF navigation bar**, SELECT** *Start* (Figure 18)

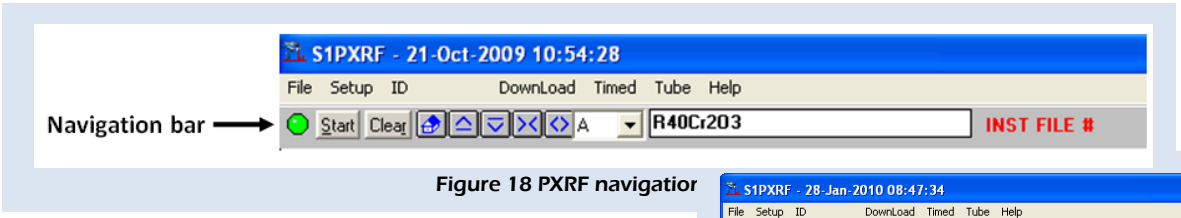



**Figure 18 PXRF navigation**

a. Within 2-3 seconds, a spectrum will begin to grow.

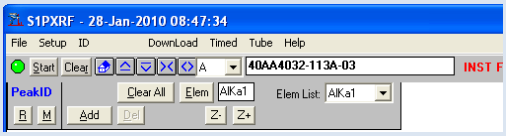



**Figure 19 ID Window**

*Note: Depending on the matrix and count rate, a longer assay time may be required. If the spectrum is to be used for calibration, a collection time 4 times the planned test time is recommended (180-second assay time is typical).*

15. After the desired measurement time has elapsed, **SELECT** *Stop* from the PXRF navigation bar.

16. **ID** and **LABEL** the element peaks:
    a. Select **ID** from the navigation bar.
    b. From popup window (Figure 19), **SELECT Elem** (see Appendix B for details on using the ID function).
    c. From popup Element Table (Figure 20), select K or L line.
    d. **CHOOSE** elements. A blue vertical line will appear identifying the chosen element. When the line corresponds with the spectral peaks, the element has been identified.

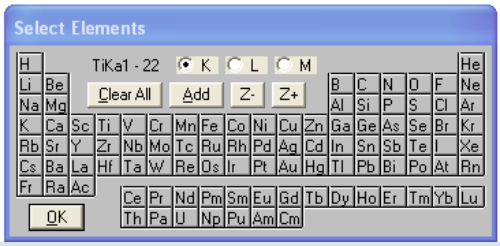


**Figure 20 Pop up window, Element Table**

17. **TAKE** a minimum of 3 assays from various locations on the sample to determine basic homogeneity of the sample (*if this is an alloy sample, skip this step and assume homogeneity).*
18. **SAVE** the spectrum to library folder.
    a. From menu bar, **SELECT** *File*.
    b. **SELECT** *Save As*.
    c. To save file to be read as a PXRF file, **SAVE** file as a .pdz file.
    d. To save file to be read outside of PXRF (e.g., Excel), **SAVE** file as a .csv file.

## Timed Assays

To take timed assays for each sample:

1. **PLACE** the sample on the instrument aperture.
2. From the PXRF Menu Bar, **SELECT** the *Timed* button.
3. From the drop down menu, **SELECT** *Timed Assays*.
4. **ENTER** desired Test Time (at least 60 seconds for calibration data) (see Figure 21).
5. **SELECT** *Autosave* as default mode (saves the spectra to selected file folder after the assay is complete).
6. **SELECT** PDZ box.
    a. If the file will be read by another program (e.g., Excel), **CHECK** both the PDZ and CSV boxes.
7. **CLICK** OK.
8. **ENTER** the desired file name to be saved.
    a. If Multiple Runs is selected, the program will automatically increment the file name.

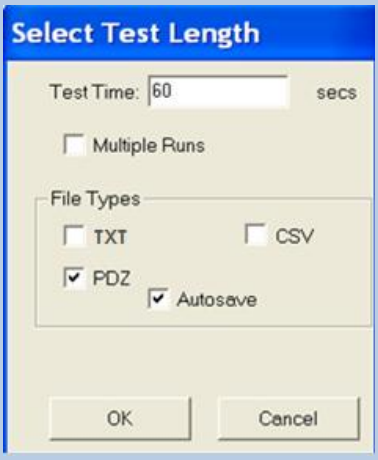


**Figure 21 Timed Assay Menu**

9. **CLICK** *Save*.
10. **CLICK** PXRF start button.
    a. If the PC Trigger has been selected the multiple runs will be performed automatically
    b. If PC Trigger is not selected, squeeze the trigger and release after the spectrum has been selected; repeat for each spectrum.
11. **RELEASE** the trigger between samples.
12. **REPEAT** for all the remaining samples.

# Reviewing Spectra

A new data folder should be created for each new sample matrix and/or new instrument parameters, (e.g., new voltage or current, new beam filter).

To view and compare a sample group:

1. **SELECT** *PDZ Preview* from the PXRF File menu (Figure 22).
2. **SELECT** *Path* and **BROWSE** to the desired data folder.
3. **OPEN** first spectra (.pdz file)
1. From PXRF Menu Bar, **SELECT** *Setup > Spectrum Overlay (*Figure 23*)*.
2. **SELECT** *Move A>>B*. This spectrum will appear in blue (spectrum B) and be moved to the background.

*The default setting makes the original spectrum a blue outline; however, the color scheme can be changed.* ***SELECT*** *Setup from the PXRF menu bar, and then* ***EDIT*** *the color scheme.*

3. From the preview window, **SELECT** and **OPEN** second spectrum. By default, this second spectrum will appear in red (spectrum A) and will appear in the foreground.
4. If matrixes or assay times for the two spectra are different, **CLICK** on the desired Compton peak and **SELECT** *Normalize* (e.g., for Rhodium target systems, place the black cursor at ~19.28 kV and select *Normalize)*.

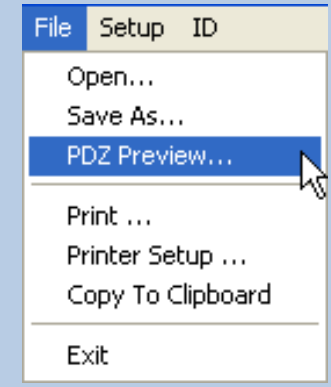



**Figure 22 PXRF File Menu**

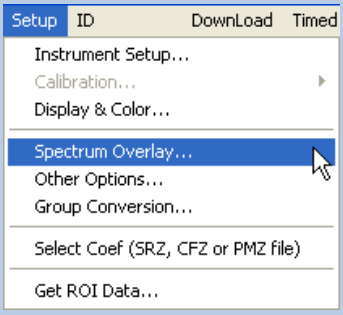



**Figure 23 Setup Menu**

# Quantification

1. **OPEN** PXRF application.
2. From Setup menu, **SELECT** *Select Coef*.
3. **SELECT** appropriate calibration file (.cfz) for the sample (Note: "Non-vac" vs. Al vac denotes Non-vacuum vs. vacuum calibrations and are stored in separate folders. Calibration files are named for their matrix (e.g., Fe2, Cu1).
4. **SELECT** a spectra file (.pdz) OR **USE** live data spectra. A *Conc* (Concentration) tab will appear on the menu bar (Figure 24).
5. From the PXRF menu bar, **SELECT** Conc (Concentration). This provides quantification of the elemental concentrations in a separate table.

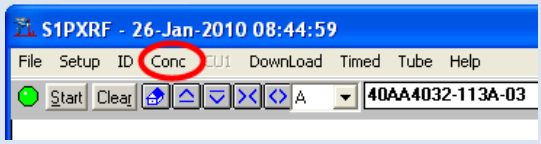



**Figure 24 Conc Tab**

# Appendix A X-Ray Tube Voltage/Current and Filter Control

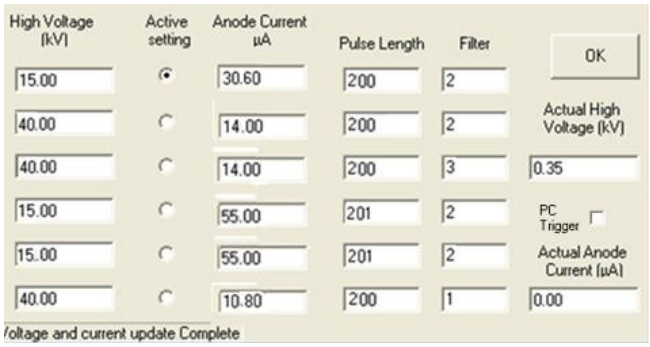



**Figure 25 Typical Voltage/Current and Filter Settings for TRACER III-SD**

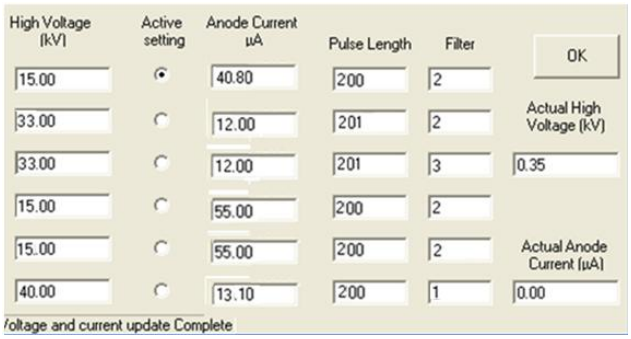



**Figure 26 Typical Voltage/Current and Filter Settings for TRACER IV-SD**

The current setting (µA) may vary slightly from instrument to instrument; however, the settings in Figure 25 are typical for the TRACER III-SD. TRACER IV-SD typically have settings similar to those in Figure 26.

Factory calibrations use the settings in the following Table:

| Calibration Group | Voltage | Current Range | Filter |
|---|---|---|---|
| **Non-Vacuum**<br>Iron, Cobalt, Nickel, Copper (Bronzes), Gold | 40kV | 10-20µA | 1<br>(Al-300um/Ti-25um) |
| **Vacuum Titanium** | 33 kV | 10-15 µA | 2<br>(Blank-No filter) |
| **Vacuum Aluminum** | 15 kV | 55 µA | 2<br>(Blank-No filter) |

When the energy of elements of interest change significantly, or the substrate density changes, you may need to change the voltage and/or current of the instrument. For example—if you would like to examine Si and Mg only, (especially in low concentrations), you may want to set the voltage to 15 kV (these are considered low energy or low "Z" elements). For low-density substrates, the current should be higher; for high-density substrates, the current should be lower. (The total raw/valid count ratio should ideally be less than 3:2, and definitely less than 2:1.)

*Warning! Clicking the PC Trigger from the Tube Setting window will activate the unit. Be sure that a sample is present and all safe precautions are engaged prior to checking the PC Trigger box*

*Note: The PC Trigger can be activated from this window by checking the PC Trigger box.*

# Appendix B Using the Element/ID Window

For first time users, it may be easiest to use the Duplex 2205 alloy sample that comes with each instrument so as to become familiar with the operation of the Element/ID window. When the Duplex 2205 alloy sample is used, the spectrum should look like the one below. The first peak is the Chromium (Cr-K-shell), the second large peak is the Iron (Fe-K-shell) and the peak at approximately 18 KeV is the Molybdenum (Mo-K-shell). To open the *Select Element ID* window, select *ID* from the main menu, and then click on the Elem button in popup window. Note that the Select Element ID window allows you to select K, L or M shell ID. You will want your Bruker Elemental periodic chart handy to help identify the K and L-shell energies, or you can right click on the Elements window for additional information. For a correct peak ID, the blue lines should line up perfectly in the center of each peak. There will be alpha and beta lines for each element.