

# Handheld XRF (pXRF) User Guide

V374 | March 2018

## Manual Information

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Origination date:	2017 372
Original version:	V371T   July 2017
Revised:	V374 March 2018
Domain:	X-Ray
System:	XRF
Keywords:	Elemental analysis

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## Introduction

When X-ray radiation from the handheld XRF instruments' X-ray tube (source) excites the atoms in the sample, the atoms release fluorescent X-rays. The energy level of each fluorescent X-ray is characteristic of the element excited. As a result, one can tell what elements are present based on the energies of the X-ray emitted. The **Olympus DELTA Premium** handheld XRF (**pXRF**) detects and determines the fluorescent X-ray energies, produced. These energy patterns are characteristic to specific elements and allows for elemental measurements on section halves and discrete samples.

## Theory of Operation

X-ray fluorescence is the production of X-rays in the electron orbits. The handheld XRF functions by bombarding the atoms of the target sample with x-rays. Some of the photons collide with K (and L) shell electrons of the sample, dislodging them from their orbits. This leaves a vacant space in the K (L) shell, which is immediately filled by an electron from the L, M, or N (M or N) shell. This is accompanied by a decrease in the atom's energy, and an X-ray photon is emitted with energy equal to this decrease.

Since the energy change is uniquely defined for atoms of a given element, it is possible to predict definite frequencies for the emitted X-rays. This means that when electrons are dislodged from atoms, the emitted X-rays are always identical. These X-rays are analyzed with an X-ray detector and the quantity of K shell and/or L shell X-rays detected will be proportional to the number of atoms of the particular element or elements present in the sample.

## Apparatus, Reagents, & Materials

### Laboratory Apparatus

#### Discrete Samples

- XRF Sample Cups (32 mm Double Open Ended)
- SPEX Ultralene thin film for XRF (0.16 mil; 4 µm thick)
- Whatman circles (Grade 540; 24 mm)
- Foam plugs
- Powdering equipment (dependent on sample hardness):
  - Agate mortar & pestle
  - SPEX Mixer/Mill®
  - SPEX Shatterbox® (**take note:** only tungsten carbide disks are available on board)

#### Section Halves

- Leaded section half shielding
- Shielded box & gun stand

### Instrument Hardware

- Olympus DELTA Premium handheld analyzer
  - Processor
  - Colour touch screen
  - Membrane navigation keys
  - Detectors and tube anode material
- DELTA docking station

- Prolene® Windows
- Four Li-ion Batteries
- Rugged waterproof transport case
- Calibration check (CalCk) reference coupon/coin
- USB data cables
- Battery charger
- AC power adaptor
- Portable Flex Stand (test stand)
- Power cords
- Shielded mount for discrete samples

### Computer & Software

- XRF Laptop & power bar
- Section half & discrete barcode scanner
- Innov-x Delta advanced PC Software

## Apparatus Preparation & Set up

### General Precaution: Isolate the Instrument

The pXRF station needs to be mobile, easily accessible, and maintain a small footprint. The station should be in a location where section halves can be brought in and out easily without disrupting other work stations or without danger of dropping or mishandling core material. As a safety precaution, the pXRF should be set up in a location with minimal people working nearby. Potential locations are the Down Hole lab, Paleo Prep lab, or the correlators' station (only if no stratigraphic correlator is sailing). Check with scientist and co-chiefs for preferred location. After finding an appropriate location for the pXRF, begin setting up section half and discrete sample stations.

### Prepare Safety Talk

Work with the Co-Chiefs, Staff Scientist, and LO to identify the pXRF Users. Each user is required to wear a Dosimeter Ring while operating the pXRF. After identifying the scientists collect the dosimeter rings from the LO. Each ring has a unique ID and is assigned to a specific person. Check out the rings and record which ring is assigned to which person. Make sure to check out a ring for yourself as you will be demonstrating how to use the Gun and will perform safety checks on the instrument.

Prepare a safety talk and demonstration for the scientists who will be using the pXRF. For safety resources please to the IODP manual 'XRF Safety Guide' and the vendor provided DELTA Family safety chapter to familiarize yourself with the safety precautions. Make these documents available to the scientists.

### Apparatus Preparation

#### pXRF Analyzer

Inside the pXRF case are: the pXRF, docking station, micro-USB cable, docking station power supply, pXRF AC power supply, and pXRF batteries.

The pXRF can be operated through the User Interface on the analyzer or through a dedicated pXRF laptop. Be consistent with the chosen method.

First take the docking station and place in in your dedicated area (Figure 1). The pXRF will rest in the docking station when not in use. Connect the docking station to power through the labeled power supply.



Figure 1. Docking Station

Then take out the pXRF and rest it in the docking station (Figure 2).



Figure 2. pXRF in Docking Station

The pXRF has two options for receiving power: an AC power supply inserted into the gun and connected to a wall socket or batteries inserted into the gun. For measurements taking place over a small amount of time the batteries are fine, however for longer periods the AC Power Supply is preferred. If using the batteries understand that if the pXRF powers off before data has been 'exported' from the software, all measurements will be deleted. The Olympus DELTA Premium pXRF has a small internal battery which allows the lithium battery packs to be "hot-swapped" without losing power to the pXRF. The internal battery will keep the pXRF running for approximately thirty seconds, so work quickly to slot the new battery into place.

Pull the rubber latch at the bottom of the handle open (Figure 3). Insert the preferred method of power supply into the handle (Figure 4). The battery and AC Power Supply are keyed and only fit into the analyzer one way



Figure 3. pXRF handle open



Figure 4. AC battery source inserted into pXRF



You can now turn on the gun. Press the power button on the top of the analyzer for a couple of seconds (Figure 5). The button will illuminate green when on, as shown below in Figure 6.



Figure 5. pXRF power button outlined in red. pXRF is off.



Figure 6. pXRF turned on

*The following step is only applicable at this stage if controlling the pXRF through the computer. If choosing to use the pXRF alone move on to 'Discrete Samples'. The pXRF interface users will use the laptop during the 'Export Files' Section later on.*

Open the dedicated pXRF laptop and login to the computer with the following:

user: XRFHost

Password: B113@iodp.

To operate the gun and have it correctly write files administrator privileges are needed. If you login with something different, such as 'daq' 'daq', there are no administrator privileges and the gun will not work properly.

Connect the pXRF to the computer via the micro USB cord (Figure 7). Micro USB ports are very fragile so take care when plugging in or removing.

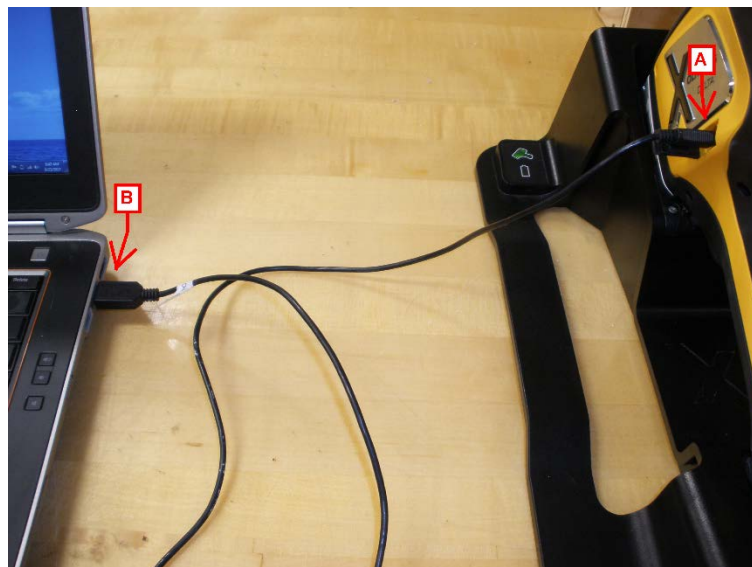


Figure 7. pXRF connected to laptop via micro USB cable. A. micro USB port. B. Laptop usb port

### Discrete Samples

1. To set up the discrete sample station collect the FlexStand Base and shielded sample chamber (Figure 8).

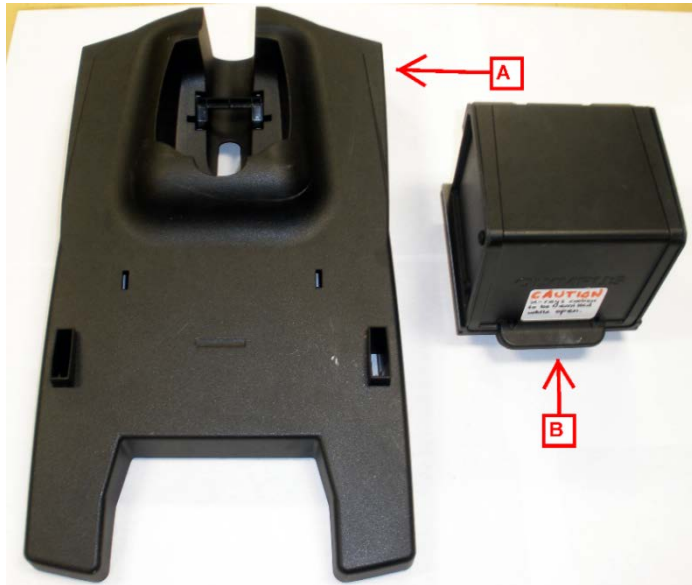


Figure 8: FlexStand components. A. FlexStand Base B. Sample Chamber

2. Place the back of the analyzer into the indented portion of the FlexStand Base. The analyzer nose will point into the air as in Figure 9. Take note of the two indents in the analyzer nose.

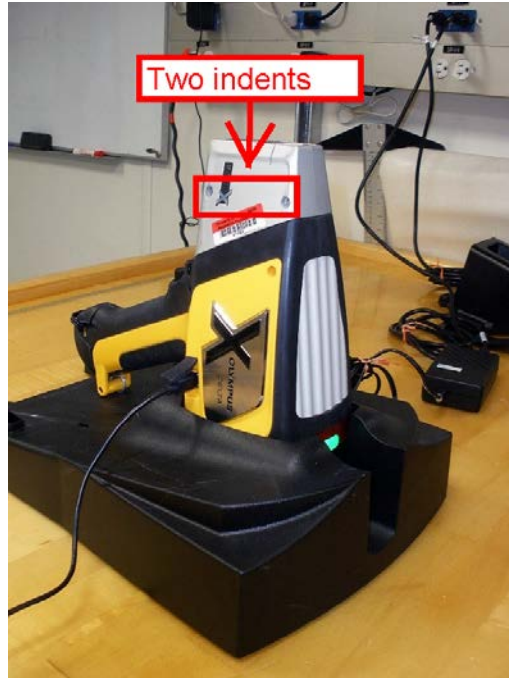


Figure 9. pXRF in FlexStand base. Red arrow indicating two indents on analyzer nose

3. On the Sample chamber there are two pegs and a release handle (Figure 10). The release handle controls the pegs.



Figure 10. Bottom of Sample Chamber. A. Two pegs B. Release handle



4. The two pegs will fit into the two indents on the analyzer nozzle. Orient the sample chamber so the pegs will fit into the holes and lower the chamber over the nose (Figure 11). The pegs will snap in when inserted.



Figure 11.  
Sample Chamber resting over pXRF nose.

**Note:** The **Olympus DELTA Flex Stand** can be set up in a second orientation, with the instrument standing upright. This is not recommended on board, because the samples are more likely to rock with ship's motion and give inconsistent results.

5. When it is time to remove the sample chamber pull the release handle and while holding the handle, lift off the sample chamber (Figure 12)



Figure 12. Release handle being pulled

## Section Halves

In preparing for section half analysis, consider the safety requirements of the instrument. Prepare the area carefully, ensure everyone working with or around the instrument understands X-ray safety, and use the lead drape to protect from scattered X-rays. When the pXRF is in contact with a section half, most of the X-ray energy is absorbed by the sediment/rock. The scattered radiation is at a low (but measurable) level, so maintain reasonable distance between the pXRF and the user. Do not place your hands close to the nose of the instrument; keep them on the grip/trigger area or butt of the instrument.

Remember **ALARA**: keep your exposure As Low As is Reasonably Achievable.

1. Set up an aluminum core tray to hold the **unwrapped** section half (you will wrap it with Ultralene later, but GLAD® Plastic Wrap is thick enough to interfere with measurements. Place the core tray far enough back from the edge of the bench that the pXRF will be several feet away from the operator.
2. Put a long ruler next to the aluminum tray to measure the measurement offset.
3. Place the docking station to the side of the core tray.
4. Collect the rubber lead impregnated shield. When it is time to take a measurement the pXRF will lock in to the mat as shown in Figure 13.



Figure 13. pXRF being lowered into section half shielding. pXRF oriented with indents facing set screws.

Again, please note that it will be necessary to cover the core surface with Ultralene film and GLAD® Plastic Wrap in order to protect the nose of the instrument and the underside of the shield from contamination. See

**Preparing Section Halves**, below.

**Important! Do not measure the core section half without covering it with film!**

## Sample Preparation

Sample preparation is split into two main categories: discrete samples and section halves.

### Preparing Discrete Samples

Discrete samples are dried, ground, and loaded into a XRF sample cup. Discrete samples allow the analysis of a specific interval or feature on a section half. This process is more involved, time-consuming, and invasive (destructive) compared to the section half measurement. However, discrete samples can be run along with powdered standard reference materials and the data can therefore be reduced for quantitative elemental data. Powdering a sample also creates a homogenous sample. If the

powder is fully homogenous and the sample cup is loaded to sufficient thickness and uniform powder density, quantitative error should be low.

### Drying Samples

It is important to dry discrete samples prior to grinding them. Freeze-dry samples for 12 hours. For details on how to operate the Freeze Dryer refer to the **XRD User Guide**.

### Grinding Samples

Once dry, samples are ground into a talc-like powder. Grinding is accomplished by three methods: agate mortar and pestle, SPEX Mixer/Mill®, or SPEX Shatterbox®. The specific grinding method used is dependent on sample hardness. For more details on grinding methods refer to the **XRD User Guide** and **ICP Hard Rock Prep User Guide**.

### Loading Samples into an XRF Sample Cup

To create a discrete XRF sample, assemble the following components as shown in Figure 13, below:

- Powdered & dried sample
- Scoopula
- XRF sample cups (32 mm Double Open Ended)
- SPEX Ultralene thin film (0.16 mil; 4 μm thick)
- Whatman Filter Paper Circles (32mm) or Parafilm M® film
- Foam plug

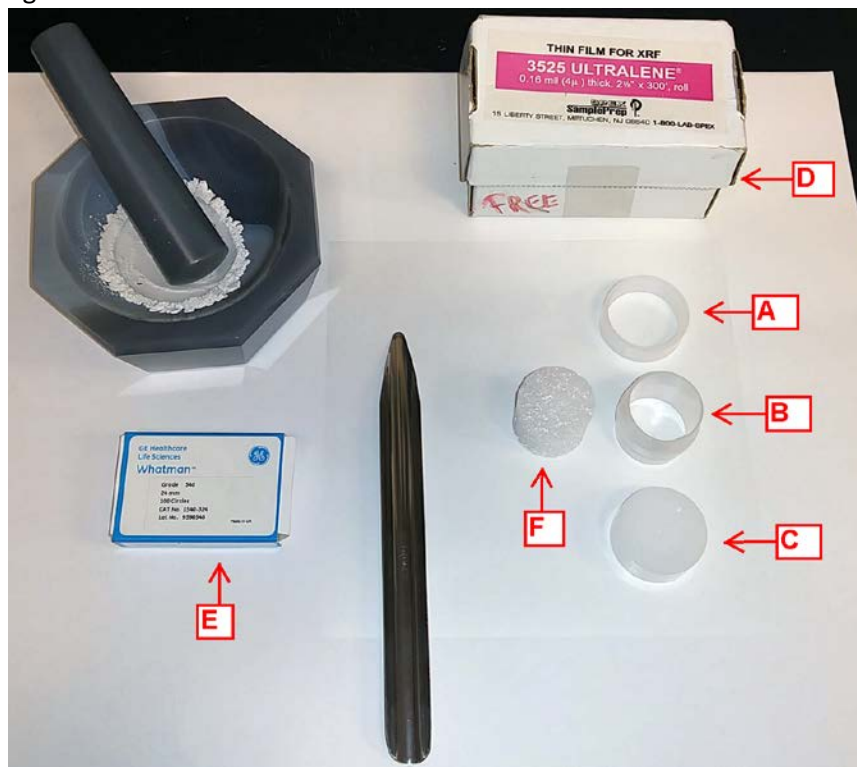


Figure 14. Mortar and pestle along with scoopula. XRF Sample Cup components denoted with red arrows A. Open-end Ring B. Cup C. Cap D. Ultralene® film box E. Whatman Filter Paper F. Foam plug inserts

1. A XRF Sample cup has three components: cup, ring, and cap. Take a piece of Ultralene® film and stretch it over one end of the cup. Place the open ring over the film and cup and snap it firmly onto the cup. This will create a tight window on one end of the sample cup (Figure 15). Ultralene® is very thin and prone to ripping. Check for rips before and after sample powder is added.



Figure 15.  
Ultralene® covering  
of one side of cup

2. Flip this unit upside down and begin to load the sample into the cup. Remember that the discrete sample will be back loaded. A couple centimeters of sample is sufficient. The sample layer cannot be too thin, or X-ray penetration (and the results) will be inconsistent. Make sure the powder is evenly dispersed, maintaining a relatively flat, even surface.
3. Next place a barrier layer on top of the powder, either Whatman Filter Paper or Parafilm. This creates a seal that prevents contamination and keeps powder secured in place. Take a piece of Whatman filter paper and put it on the powder or cut a square of parafilm, wrap it over the end of the foam plug and put inside the cup. Press down evenly until it is firmly on the powder.
4. Insert the foam plug. Take care not to insert it too quickly and cause air displacement that might disturb the powder. Depending on how much sample has been loaded into the sample cup; a couple centimeters of the foam may need to be chopped off.
5. Once you have filled the unit properly, place another piece of Ultralene® film across the back of the sample cup and snap the closed cap on the cup. If you have over-filled the sample cup, this is the point where the film will break, so examine both sides of the cup after you snap the lid on.
6. It is important to create some outward “bowing” of the film so that the powder stays well-packed and stationary. Cracks, gaps, or inconsistent filling of the powder will create inconsistent results. Be careful, however, not to pack too tightly or the Ultralene® film will break and you will have to start over.

Your finished product should look like the cup in Figure 16.



Figure 16. Properly filled XRF cup; note the slight bulge of the powder.

Put a sample label onto the cup. Remove excess Ultralene to help the label stick. The discrete sample is now ready for analysis.

### Preparing Section Halves

**Note:** Ultralene® film is quite expensive (about \$1 US/meter) and it is not practical to cover the entire length of each core section.

1. Cut a piece of Ultralene® approximately 10 cm long and cover the core section at the locations you intend to measure. Cover the rest of the section in GLAD® Plastic Wrap so the instrument and rubber, lead-impregnated shield does not get dirty. Make sure the top of the plastic is clean, dry, and mud-free, especially the Ultralene®-covered locations.

**Important!** Even a small amount of sediment on top of the thin film will be transferred to the pXRF, and will affect the measurement of the next core section half.

## Starting the Software

**Note:** Independent of sample type (discrete or section half) all samples follow the same procedures for taking and storing XRF data.

The computer interface and the pXRF interface are different. Follow the appropriate instructions.

### Computer Interface

#### Getting Started

Double-click the **Innov-X Delta Advanced PC Software** icon on the desktop (Figure 17) to launch the software.



Figure 17. Innov-X software icon.

The initial start-up window will appear (Figure 18, below); click the **Import Data Only** button. **Import Data Only** moves only the analytical data from the pXRF to the PC. If the pXRF was just turned on then wait a moment and select 'Close Device App' then continue with 'Import Data Only'. The PC Software will not let you continue on if the Device Application is active.

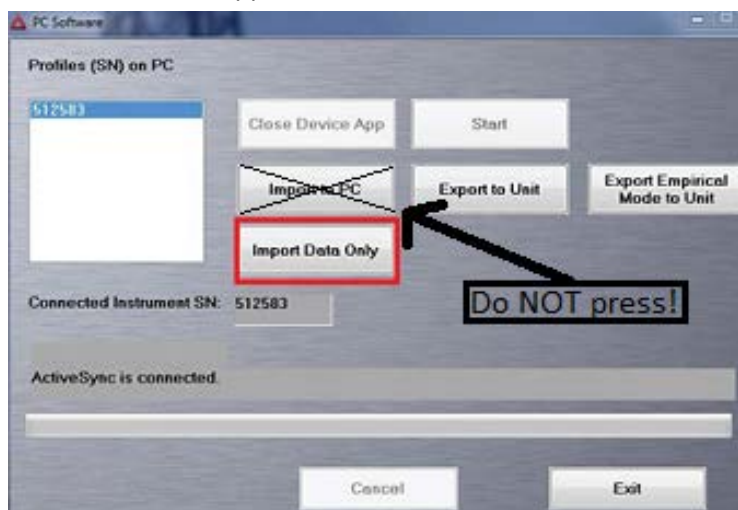


Figure 18. Innov-X Start-up Screen



**Important! Do not click the Import to PC button!**

The **Import to PC** button overwrites the saved method on the PC with the method stored on the pXRF unit. This is likely to be factory settings and not the methods needed for the analysis, so it is a very undesirable action.

Once the **Import Data Only** button has been clicked, the **Start** button will become active after a brief pause to sync the instrument with the software. Click **Start**. The login screen appears (Figure 19). Enter the following information:

- User Name: innovx
- Password: 1776



Figure 19. Login screen. Login button outlined in red

The main measurement window then pops up.

### Performing a Cal Check

Before any measurements are taken, a **Cal Check** needs to be performed. A Cal Check is an internal calibration check in which the analyzer measures a particular stainless steel alloy to ensure the unit is measuring properly. A calibration check is required when the gun is turned on and every 10 hours thereafter. The information bar indicates when a Cal Check is needed by displaying “Perform a Cal Check” as shown in Figure 22, arrow B, below. It is not possible to proceed until the check is done. Place the gun in its docking station.

**Note:** When using the **Flex Stand**, it is possible to perform the **Cal Check** by simply placing a stainless steel disk on the instrument nose and closing the lid of the **Flex Stand**. An extra stainless steel disk can be found in the pXRF supplies.



Figure 20. Performing a Calibration Check. A. CalCheck Button B. Indicator Bar C. Disabled 'Start' button

Click the **Cal Check** button located on the bottom left as shown in Figure 20, arrow A. The instrument will measure the stainless steel disk and check the peaks and their ratios to ensure that it's getting the expected results. The **Start** [analytical measurement] button will remain grayed-out and disabled until the pXRF passes the CalCheck. If the analyzer fails a Cal Check please refer to the '**CalCheck Fails**' Section in Troubleshooting.

### Setting Parameters

Parameters that require setting or editing include: mode and measuring time.

#### Selecting the Mode

Choose either the Geochemical or the Soil Mode; each one has specific scanning parameters, and preferentially scans certain elements. Discuss with scientists which mode is preferred.

**Note:** Once a mode is selected, it does not have to be re-chosen before each measurement or when the gun is turned on/off.

Click on **Mode**, located top right in Figure 21 (arrow A). On the left hand panel, you can choose between Geochemistry and Soil modes (Fig. 21, B). Click on each to see the elements scanned and the beam conditions used in each mode.

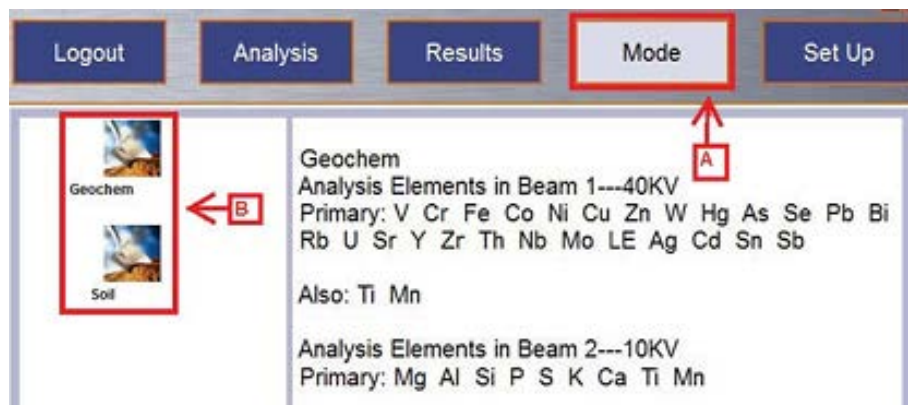


Figure 21. Mode window. A. 'Mode' tab B. Measuring Mode options

## Selecting Measuring Time

Establish the measuring time for each beam. To access this, click the **Set Up** button at the top of the screen as shown in Figure 22, arrow A, below. On the left hand panel, click **Test Condition**. This will show the minimum (**Beam # Min**) and maximum (**Beam # Max**) time currently set for each beam. Adjust the settings as desired in seconds and click **Save**.

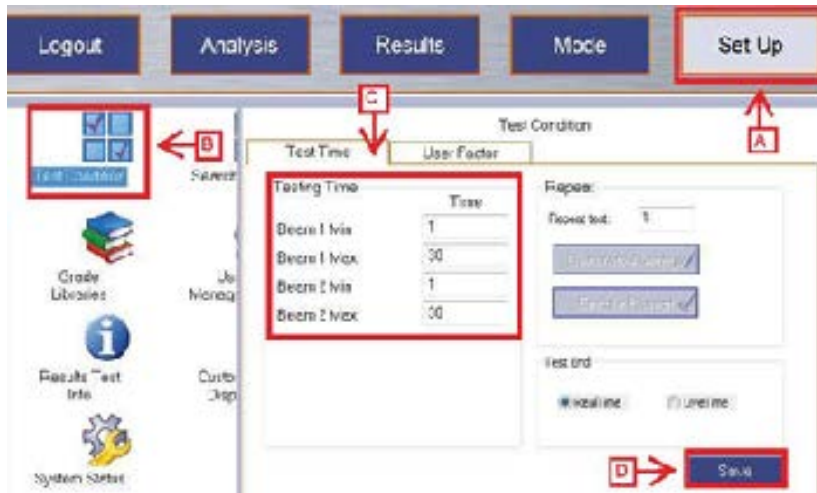


Figure 22. Innov-X Set Up Screen. A. Set Up tab B. Test Condition button C. Test Time area D. Save button

The behavior of the Test Time settings is as follows. If the minimum time is set to 1 s and the maximum time is set to 30 s (as shown in Fig. 22), the pXRF will scan until it determines that it has a good profile (via manufacturer-defined quality conditions) and stop measuring before it gets to 30 s. However, even if the pXRF has not reached a “good profile,” it will stop at the maximum time.

If the user wants to ensure the pXRF measures for the same time on each measurement, set the minimum and maximum time to the same number (e.g., Beam 1 Min = 30, Beam 1 Max = 30). We do not recommend measurement times below 30 seconds, and longer times may be necessary to get good resolution of weak lines.

## Naming the Sample for Measurement

In the Analysis Window, click the blue button with the three dots located in the upper left corner as shown by the B arrow in Figure 23.

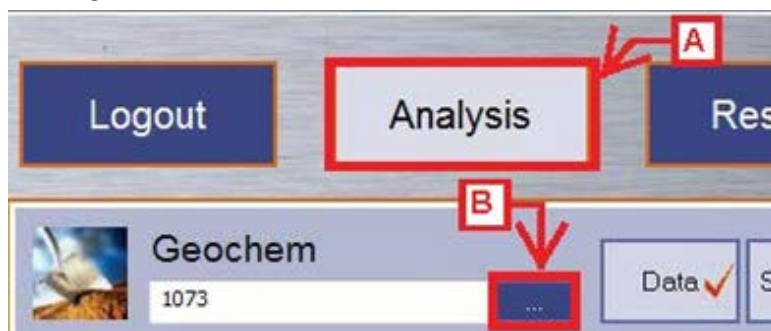


Figure 23. Analysis screen closeup. Clicking on the blue “...”

The measurement naming window will appear (Figure 24, below).

This window contains fields to identify the sample.

Test Info for Geochem			
Run Number	1073	Label 9:	Label not enabled
Sample Name	366-U1498B-23R-3-W-2E	Label 10:	Label not enabled
Text_ID	PWDR8638211	Label 11:	Label not enabled
Offset for SHL		Label 12:	Label not enabled
Comment	oxidized powder	Label 13:	Label not enabled
Label 6:	Label not enabled	Label 14:	Label not enabled
Label 7:	Label not enabled	Label 15:	Label not enabled
Label 8:	Label not enabled	Label 16:	Label not enabled

Figure 24. Test Information window. IODP-defined fields are circled in red (arrow A). B. Save button

Arrow A in Figure 24 highlights the IODP-defined editable fields, defined as follows:

- **Run Number:** A sequential number increased by +1 with every measurement, ex: 1, 2, 3... This number should start at 1 for the first measurement of the expedition. This should be unique throughout the expedition, so the use of a logsheet is highly recommended
- **Sample Name:** Sample name according to the sample or section half label. Ex: 366-U1234A-20F1-W.
- **Text ID:** Unique database identifier for each sample or section half located under the barcode on the label. Ex: SHLF8638181
- **Offset for SHLF:** The point where a measurement was taken, measured as distance from the top of the section. Leave this field blank for discrete samples.
- **Comment:** A brief description of the measurement. An optional field.

First enter the run number. Then put the cursor in the 'Sample Name' field scan the label with a barcode scanner. Use the barcode scanner labeled **SECTION HALF** for section halves and the scanner labeled **DSICRETE** for discrete samples. This will populate the 'Sample Name' and 'Text ID' fields. If measuring a section half enter the 'Offset for SHLF'; leave this field blank for discrete measurements.

**Important!** Click **Save** in the lower right portion of the screen.

**If you do not click Save, the sample information will revert back to values entered for the previous run and the new measurement will have the same name as the previous one.** The measurements are parsed by time so measurements will not overwrite one another; however, there will be multiple measurements with the same information, and the data will have to be sorted out later.

These barcode scanners have been programmed to properly parse the different kind of label for section halves and other types of samples. The fields should fill with the label ID (e.g., 366-U1498B-23R-3-W...) and text ID (e.g., PWDR8638211). If they are not working correctly reprogram the scanners by going to IODP\_SHARE > XRF > Barcode Rules and scan the rule set.

If the user needs additional fields to describe the sample or the measurements being done, the technician can modify the method to enable up to 11 additional user-defined fields. To see how to add fields refer to the section 'How to Add Fields to Sample Information Window'

## pXRF User Interface

### Getting Started

The DELTA's user interface starts with the start-up radiation safety and initialization screens (Figure 25). Read the radiation safety notice screen, and then confirm that you are a certified user



Figure 25. Start-up Radiation safety and initialization screens.

System initialization begins immediately after confirmation (Fig. 26). The analyzer launches a test screen using the previously selected mode.

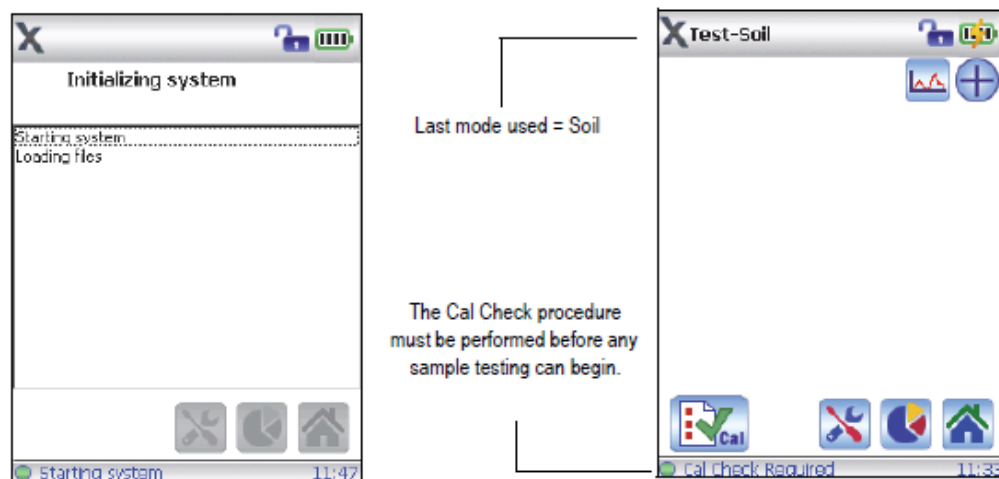


Figure 26. Initialization and test screens.



## Performing a Cal Check

Before sample testing can begin, a Cal Check needs to be performed. This will be shown by a “Cal Check Required” message located on the bottom left of the screen. A Cal Check measurement tells the gun to measure the stainless steel disk located in the stand. This is an internal calibration which ensures the gun is measuring properly.

*Note: Cal Checks must be performed when the analyzer requests the procedure (once when the program is started, and once after every ten hours of testing). The Start Test button and trigger are disabled until a successful Cal Check is achieved. The Cal Check can be run any time during software operation, except during a test. When the Cal Check is in progress, the X-ray indicator light assembly blinks. This indicates that the X-ray tube is energized.*


To perform a manual Cal Check: Place the analyzer in the DELTA docking station. Tap the Cal Check button.




*Note: The deadman trigger are overridden when the analyzer is mounted in the docking station. If necessary, unlock the software trigger lock by tapping the Lock button at the top of the screen.*

If the “Cal Check – Passed” message appears on the Results screen, the analyzer is ready for sample testing. If the Cal Check refer to the ‘CalCheck fails’ section in ‘Troubleshooting’.

## Setting Parameters

Tap the Home button . You are now in the main measurement window. First set the Mode in which you’d like to measure in. The gun has a Geochemical and Soil Mode. Each one has different scanning parameters which preferentially pick up some elements better than others. The gun will remember the chosen mode and does not have to be chosen each time a new measurement is taken or the Gun is turned off/on.

To access the Modes click on ‘Mode’ button . This directs you to the ‘Mode’ Screen where you can choose between the Geochemistry and Soil modes (Figure 27). Click on each to see it’s corresponding features and select the one best for your analysis.

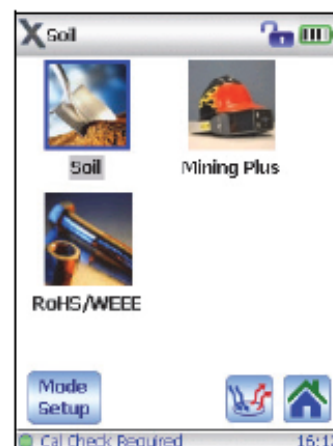


Figure 27. Mode Screen.

Now that the Mode is set, establish the measuring time for each beam. Each Mode has preset measuring times but these are changeable. To access the Measurement Time, in the Mode Setup screen click on 'Test Condition.'



In the Test Conditions screen, tap the Test/Tools button. This brings you to the Test Setup screen (Fig. 28). Enter the Minimum and Maximum time currently set for each beam (Fig. 8). Adjust the settings to your liking and click 'Save'.



Figure 28. Test Setup screen.

*Note: If the minimum time is set to 1 and the maximum to 30, if the gun determines it has a good profile in less than 30 seconds it will stop actively measuring. If you want to ensure it measures for the full time, set the minimum and maximum time to the same number. Ex: Minimum 30s and Maximum 30s*

## Naming a Measurement

Staying in the Test Setup screen, tap the Label Defaults button located in the bottom left corner.



This is where you can manually input sample information. Editable fields are as follows: Run Number, Sample Name, Text ID, SHLF Offset, and Comment (optional field).

**Run Number:** A sequential number increased by +1 with every measurement, ex: 1, 2, 3... This number will start at 1 for the measurement of the expedition.

**Sample Name:** Sample Name according to the sample or section half label. Ex: 366-U1234A-20F1-W

**Text ID:** Unique 11 digit ID for each sample or section half located under the barcode on the label.

**Offset for SHLF:** The point where a measurement is made on a section half, measured as distance from the top of the section. This field will only have a value for section half measurements.

**Comment:** A brief description of the measurement. An optional field.

Click 'Save' in the lower right portion of the screen. If you do not click save the information will revert back to the prior run and there will be two measurements with the same name. The measurements are

parsed by time so measurements will not overwrite one another there will just be multiple measurements with the same information.

## Preparing the Sample for Measurement

### Discrete Measurement

1. Open the mount door. The measurement window should lay flush with the bottom interior of the sample chamber.
2. Insert the discrete sample oriented face down towards the X-ray beam. ALWAYS make sure only smooth, flat surfaces go on the X-ray window. The pXRF has a very thin Prolene window that can be easily torn and is the only barrier between the beryllium window and the sample. If the window becomes torn stop measuring immediately and replace the window. The Beryllium window is very fragile and brittle, any particles coming into contact with it can ruin the window and make the pXRF unable to measure accurately.
3. Close the mount door, and make sure the shielded mount is firmly and safely attached to the analyzer. The discrete sample is ready to be analyzed.

**Warning! NEVER activate the X-ray source when the Flex Stand door is open! The Sample Chamber door does not have a safety trigger and the pXRF will continue to emit X-Rays if door is open.**

### Section Half Measurement

1. Locate the area on the section half to be measured. Make sure that portion has a layer of Ultralene on it. Put a layer of glad wrap on the rest of the core that the lead shield might touch. We do not want to contaminate the core or pXRF.
2. Place the shield over the core so the point of measurement is centered. Use the plexiglass view-finder to help find this spot.
3. Take note of the measurement position on the core using the ruler. Lower the pXRF into the shielding oriented so the two indents on the analyzer are on the same side as the tightening screws.
4. Tighten the screws until it feels firm. This locks the pXRF into place and helps stabilize it. Keep a hand on the handle while measuring to ensure it does not fall.

## Starting a Measurement

If the minimum time has been set to the same number as the maximum time for each beam setting (see **Setting Parameters**, above), the run time will be exactly what was set. If the minimum time is smaller than the maximum time, the pXRF may stop the measurement early, if the quality of the measurement satisfies its internal quality criteria. (These criteria cannot be adjusted by the end user.)

**Important!** Record each measurement taken on the paper log sheets provided by the technician. The log sheet will be a life-saver later in the expedition.

### PC Interface


Press the green **Start** button located on the bottom left of the screen as shown in Figure 29, below. The measurement can be started in either the **Test Information** or **Analysis** window.



Figure 29. Innov-X Start Button

The spectral graph and elemental concentrations will populate after each beam has completed measuring.

### pXRF Interface

Navigate to the Test screen (Figure 30). Start the test by pulling the trigger or tapping the Start Test button. .

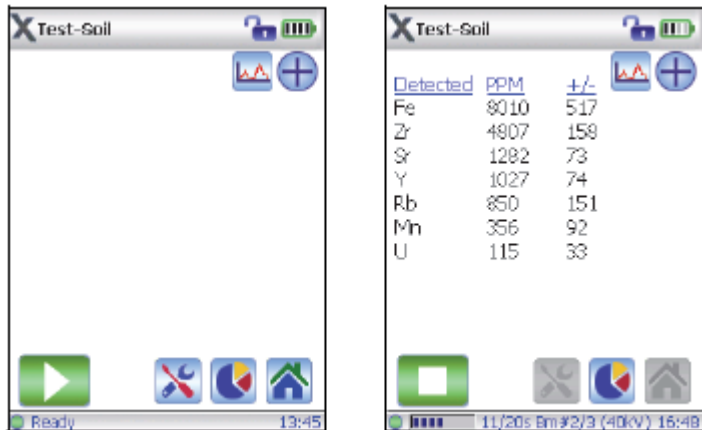


Figure 30. Start and mid-way through an active test.

The results are displayed immediately upon test completion. Tap the Spectral Plot button to view the spectrum results (Figure 31).

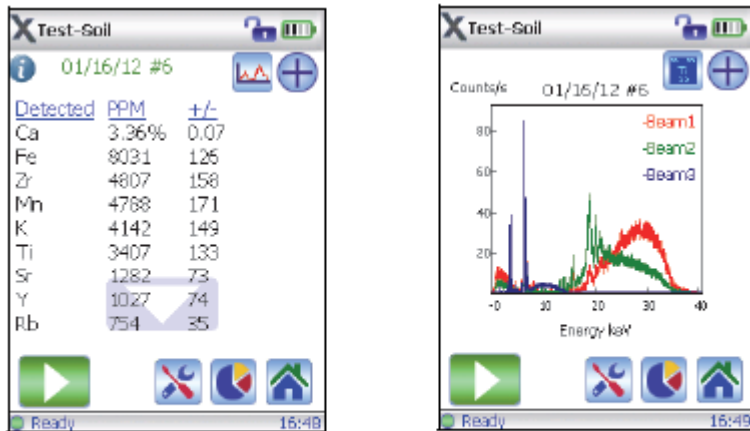


Figure 31. Completed test and results. Results shown as both a list and as a spectrum.

Complete test results with a list of the elements.

The Spectrum screen for these results.

## Processing Sample Results

Results are exported to a Data > IN Folder and then uploaded to the LORE database via Mega Uploader Tron, MUT. pXRF interface Users follow instructions on connecting the gun to the laptop. PC Users jump ahead to 'Exporting Results'.

### pXRF Interface

If using the pXRF interface at this point the laptop needs to be turned on and logged in as pXRF host. Then connect the gun to the computer using the micro-USB cable. Open the software icon. Click on the

'Import Data Only' Button. This allows data storage on the computer. **Do NOT** click 'Import to PC' as this removes all configurations from the gun. Then login with:

Username: innovx

Password: 1776

### Exporting Results – All Users

The gun writes one file per day that contains all the measurements taken in that 24-hour period. Until that file is exported the measurements are not permanently attached to the file.

**Important!** If files have not been exported **DO NOT** disconnect the gun, turn off the software, or turn off the gun; otherwise those un-exported measurements will be lost. For this reason, be sure to keep an eye on remaining battery life (or use the AC power adapter to provide continuous power to the pXRF).

To export a file, click **Set Up** on the top right corner of the screen. Click **Data Management** located on the left hand panel as shown in Figure 32, arrow B.

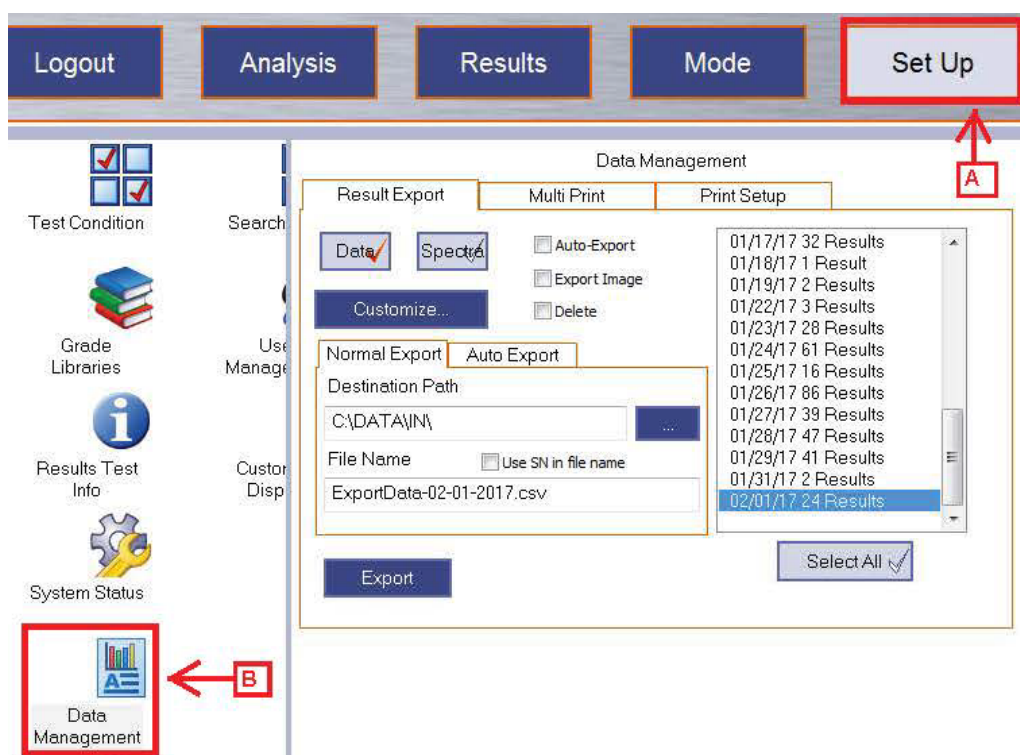


Figure 32. Accessing the Data Management Screen. A. Set Up tab B. Data Management Button

Once the **Data Management** screen appears, files can then be exported. The results are organized by date in the window on the right hand-panel as shown in Figure 26. Select the date-referenced file you wish to upload. Make sure the **Data** or **Spectra** button is selected (designated by the presence of a check mark; in Figure 30, above, the **Data** button is selected).

- **Data:** reduced data will be exported; extension is .csv.
- **Spectra:** raw data and metadata will be exported; extension is .spm.



Export a data file and a spectra file for each file. It is important that the filename be the same for both files. Follow this naming convention: **ExportData-MM-DD-YYYY.extension**, examples shown here:

- **Data File:** ExportData-07-23-2017.csv
- **Spectra File:** ExportData-07-23-2017.spm

Other file types are possible, but are not our normal procedure; talk to the technician if you need a different file type.

Once you have selected the type and set the file name, make sure the values are correct as shown in Figure 33.

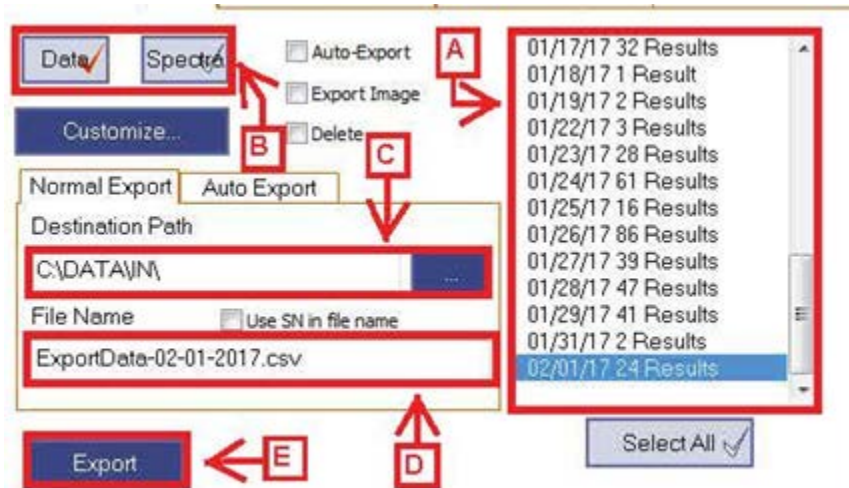


Figure 33. Exporting a file. A. List of all stored files. B. Data or Spectra selection C. Pathway where exported data goes D. Filename E. Export button

In Figure 33, A indicates the date-referenced data files. These can be exported one by one, or multi-selected. (Or, click Select All to highlight and export all of the files, but this will be difficult to sort out.)

Arrow C indicates the destination path. This should always be set to C:\DATA\IN\. Arrow D is the filename; make sure it matches the file format defined above.

Once the data type (we suggest always starting with **Data** to help prevent mistakes) is selected, the date-referenced results are selected, the path set, and the filename assigned, click **Export**. Repeat the process for the **Spectra** file, again making sure the filename is the same as the **Data** file. The active filetype has the red checkmark.

After the files have been exported the measurements are attached to the file. Multiple exports can be done in the same day; however, **the measurements will still be written to the same date file.**

**Example:** 10 measurements are exported at 11:00 on 03-15-2017. Then 5 new measurements are taken and exported at 15:00 on 03-15-2017. The 03-15-2017 file now has 15 measurements that are exported in spite of 10 measurements being exported earlier in the day. This may cause duplicate records to be uploaded to the LIMS database; see a technician if this occurs.

## Uploading Files to LIMS

This step is done by the technician.

Open MegaUploadatron (MUT). If the file(s) are not yet in the DATA > IN; transfer them now. MUT requires two file types per uploaded file: '.csv' and '.spm'. The filenames must be identical for MUT to recognize the two as linked and MUT will not allow an upload unless both files are present.

Check the boxes for the files to be uploaded. The **Misc** column identifies files that have not been uploaded as shown in Figure 34.

	Status	Analysis	ErrorMessage	Filename	Misc	Sample
<input type="checkbox"/>	✓	Handheld X-Ray ...		ExportData-01-29-2017.bt	No new items	
<input type="checkbox"/>	✓	Handheld X-Ray ...		ExportData-01-29-2017.spm	No new items	
<input checked="" type="checkbox"/>	✓	Handheld X-Ray ...		ExportData-02-01-2017.spm	24 new items	PWDR8614351 ...
<input checked="" type="checkbox"/>	✓	Handheld X-Ray ...		ExportData-02-01-2017.bt	24 new items	PWDR8614351 ...

Figure 34. MUT XRF Uploader.

Click **Upload** in the bottom right of the window. This brings up the **pXRF Pre-upload Form** as shown below in Figure 35. This form lists all measurements and allows files to be edited. The fields in white are editable: Run Number, Sample Name, TextID, Offset for SHLF, and Comment. Measurements can also be

This form allows you to review and edit the XRF data that's about to be uploaded to LIMS.

C:\DATA\IN\ExportData-02-01-2017.bt

Date	Time	Reading	Mode	Unit	Run Number	Sample Name	TextID	Offset for SHLF	Comment	Mg	Al	Si	P	S
2017-02-01	16:29:02	#1	Cal Check	%										
2017-02-01	16:32:58	#2	Geochem	%	1051	BHVO-2			Standard	44347.97	74042.47	224765.25	905.11	ND
2017-02-01	16:36:07	#3	Geochem	%	1052	366-U1497A-6F-1-W-53-XRF	PWDR8614351			231263.11	1560.49	190855.62	ND	213.1
2017-02-01	16:38:14	#4	Geochem	%	1053	366-U1498B-23R-3-W-26-XRF	PWDR8638211		Ignited	258895.27	3004.14	197615.55	ND	ND
2017-02-01	16:39:48	#5	Geochem	%	1054	366-U1498B-21R-1-W-54-XRF	PWDR8638191		Ignited	55103.39	62650.57	232871.60	441.01	ND
2017-02-01	16:41:25	#6	Geochem	%	1055	366-U1498B-23R-1-W-67-XRF	PWDR8638201		Ignited	69587.66	45239.32	223858.70	1792.79	ND
2017-02-01	16:43:06	#7	Geochem	%	1056	366-U1498B-20R-1-W-24-XRF	PWDR8638181		Ignited	269458.93	2887.86	195773.74	ND	ND
2017-02-01	17:01:23	#8	Geochem	%	1057	366-U1498B-19R-CC-W-17-XRF	PWDR8638171		Ignited	244669.68	2889.78	193542.95	ND	ND
2017-02-01	17:03:06	#9	Geochem	%	1058	366-U1498B-18R-1-W-14-XRF	PWDR8638161		Ignited	250153.97	3186.05	192643.59	ND	ND
2017-02-01	17:04:59	#10	Geochem	%	1059	366-U1498B-13R-3-W-106-XRF	PWDR8638151		Ignited	259859.34	1528.37	192264.29	ND	ND
2017-02-01	17:06:56	#11	Geochem	%	1060	366-U1498B-13R-3-W-16-XRF	PWDR8638141		Ignited	250084.26	2813.28	185780.17	ND	ND
2017-02-01	17:09:07	#12	Geochem	%	1061	366-U1498B-18R-2-W-25-XRF	PWDR8638131		Ignited	288534.07	ND	180312.08	ND	629.7
2017-02-01	17:11:01	#13	Geochem	%	1062	366-U1498B-12R-2-W-128-XRF	PWDR8638121		Ignited	256371.30	3441.54	188756.93	ND	ND
2017-02-01	17:13:06	#14	Geochem	%	1063	366-U1498B-4R-2-W-77-XRF	PWDR8636381		Ignited	249637.77	2293.50	185871.82	ND	236.1
2017-02-01	17:16:10	#15	Geochem	%	1064	366-U1498B-4R-2-W-36-XRF	PWDR8636371		Ignited	283559.57	1412.69	183911.14	ND	ND
2017-02-01	17:19:00	#16	Geochem	%	1065	366-U1498B-3R-3-W-56-XRF	PWDR8636361		Ignited	268049.51	1582.52	195048.00	ND	106.0
2017-02-01	17:21:04	#17	Geochem	%	1066	366-U1498B-3R-3-W-22-XRF	PWDR8636351		Ignited	264785.24	2899.34	193263.16	ND	1360.
2017-02-01	17:22:37	#18	Geochem	%	1067	366-U1498B-7R-4-W-123-XRF	PWDR8636331		Ignited	262760.26	2996.00	202671.53	ND	ND
2017-02-01	17:24:09	#19	Geochem	%	1068	366-U1498B-7R-4-W-13-XRF	PWDR8636321		Ignited	240387.37	3757.55	189748.04	ND	ND
2017-02-01	17:25:58	#20	Geochem	%	1069	366-U1498B-6R-1-W-123-XRF	PWDR8636311		Ignited	277379.58	2616.99	193351.06	ND	286.1
2017-02-01	17:28:09	#21	Geochem	%	1070	366-U1498B-6R-1-W-93-XRF	PWDR8636301		Ignited	247701.91	1935.28	186009.07	ND	ND
2017-02-01	17:30:06	#22	Geochem	%	1071	366-U1498B-5R-1-W-9-XRF	PWDR8636291		Ignited	244810.62	2576.21	185161.77	ND	531.5
2017-02-01	17:31:55	#23	Geochem	%	1072	366-U1498B-3R-1-W-70-XRF	PWDR8636341		Ignited	272277.76	2741.14	196761.14	ND	2202.
2017-02-01	17:33:56	#24	Geochem	%	1073	BHVO-2			Standard	45810.68	69442.84	219085.02	945.47	ND

Check all
  Uncheck all
  Show only new items

Figure 35. MUT pXRF Pre-Upload Form. A indicates the selection boxes. B. Editable fields

checked or unchecked to upload or not upload to LIMS. Cross check measurements with the log sheets and make edits as necessary.

Keep a close eye out for duplicate lines. It is easy to inadvertently create multiple measurements with the same name, if the **Save** button isn't pressed when identifying the sample (see **Naming the Sample for Measurement**, above).

If any changes need to be made to the editable fields or if scientists require a different file type, from the MUT main screen, click **Options > Set active analyses...** to open the **Configure Active Analyses** window as shown in Figure 36. Follow the prompts in the window and select **Handheld X-Ray Fluorescence Analysis** to see the configurable options. The option wheel icon will only appear when the analysis type is on the Active uploaders side and is highlighted.

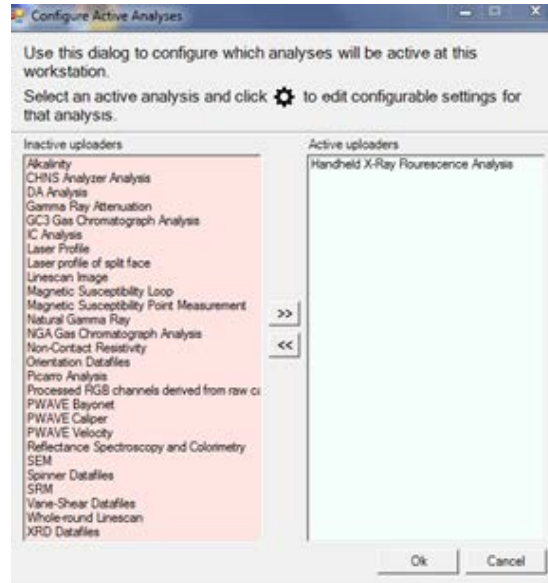


Figure 36. Configure Active Analyses screen (pXRF shown active).

Once the wheel is clicked, the window in Figure 37 appears.

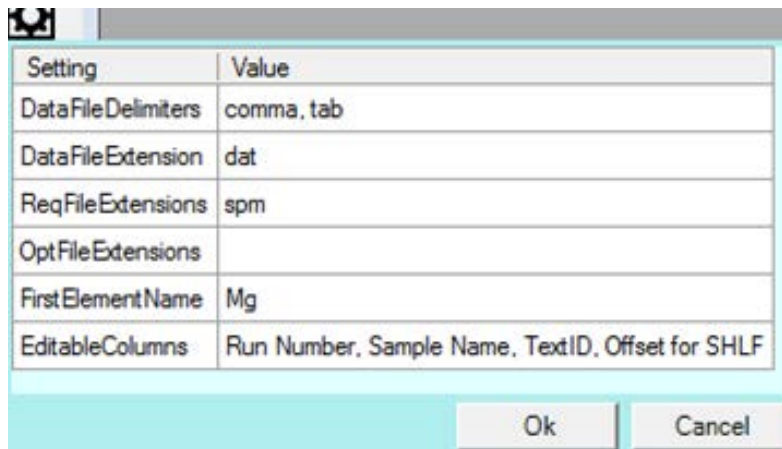


Figure 37. MUT Settings for pXRF Analysis.

These settings determine what MUT is searching for to validate the data before upload. These values are editable, but it is **strongly** recommended to have a developer assist you with this. If other labels (see **Naming the Sample for Measurement**, above) are enabled in the Olympus Innov-X software, those columns can be added here. Add the column name to the **EditableColumns** value exactly as it appears in the Innov-X menu.

## Quality Assurance/Quality Control

This measurement is highly affected by surface conditions, the heterogeneity of the sample, the size of the features in a heterogeneous sample, and the quality of the instrument-sample connection. Even a slight tilt of the instrument, for example, may have a significant effect on data quality.

It is highly recommended for the scientist to develop a quality control plan, to measure known standards for discrete samples. This is not possible for section half measurements; the user cannot judge accuracy of the elemental chemistry on section halves—only precision by measuring some of the positions more than once. (Be sure to label deliberate duplicates carefully!)

When measuring discrete samples for quantitative elemental chemistry, it is important to follow good laboratory practices: bracket the elements of interest (wherever possible) by the standards, ensure that samples are uniformly made (so that packing density is not an issue), etc.

When measuring section halves, unfortunately, the instrument **cannot** make quantitative measurements without significant post-pXRF work. In general terms, this means taking a representative number of discrete samples for each lithology, measuring them by another method like ICP-AES, and then calibrating the pXRF data **for that lithology**. Differences in moisture content, porosity, composition, surface smoothness (for good contact for the pXRF) will all have an impact—often a strong impact—on the quality of the results.

That being said, the power of pXRF for qualitative measurements of sediment and hard rock is very great (presuming heterogeneity is accounted for). A technique of evaluating elemental peak ratios can help to normalize results (but **not** quantify) for variances in physical properties, for example, and downhole trending can be displayed (similar to XRF Core Scanner results).

### Measuring Accuracy

It is not straightforward to determine accuracy for section halves, so this section applies to discrete samples. Calibration curves can be created by measuring powdered rock and sediment reference materials whose elemental responses bracket the samples.

Properly calibrated energy-dispersive XRF results, on homogenized and consistently-packed samples (or pressed pellets or flux fusion beads), can be expected to produce results of similar data quality to that of ICP-AES. (The detection limits are quite different, of course.)

It is recommended to measure some of the standard reference materials as unknowns during the analytical process in order to characterize the accuracy of the method. If all of the variables are properly controlled, accuracy of  $\pm 5\%$  or better should be achievable.

### Measuring Precision

Precision measurements are quite straightforward for pXRF for both discrete samples, but for section halves there are complications that must be discussed.

#### Discrete Samples

Discrete sample precision is expected to be quite high, with percent differences  $< 1\%$  for replicate measurements of either samples or standards. Differences higher than that may represent non-homogenous packing of sample cups.

#### Section Halves

If the pXRF is not moved between measurements, precision will be very high (similar to that for discrete samples). However, if the pXRF is moved and is not placed in the exact same spot as the previous

measurement, the precision is likely to be significantly lower, depending on the characteristics of the core section in question.

It may be quite useful, therefore, for the user to make such measurements, attempting to place the pXRF back in the same place, in order to define some of the limits of the precision of the measurement.

## Maintenance and Troubleshooting

### Maintenance

It is important to keep the pXRF clean and if the Prolene® window becomes dirty (or breaks), it should be replaced immediately. For instructions on how to replace the Prolene® Window please refer to the Olympus 'DELTA\_Family--User—International' User Manual.

Keep the batteries away from extreme temperatures, and talk to the ET's about the best way to promote long battery life.

One of the things I worry about the most is the mini-USB connector on the side of the instrument to connect to the PC. The cables are somewhat easy to damage, but the biggest concern is the port itself. Mini-USB ports are **very** easy to damage, so take care to protect it against force from the side (i.e., pulling the cable sideways).

### Delete Files from Software

In the Set Up > Data Management area select the box next to 'Delete'. Then select the files to be deleted. A blue 'Delete' button will appear at the bottom of the screen. Click the 'delete' button. Restart the software to see be removed.

### Replace Ultralene Window

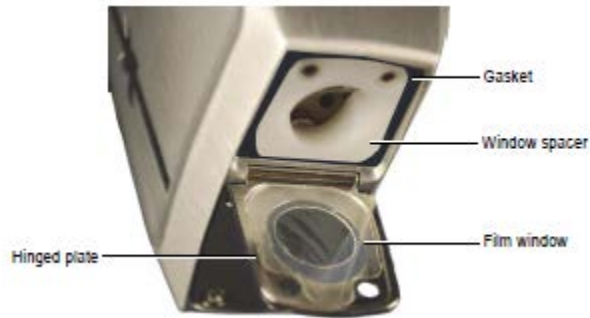
It is critical during the process to keep the surrounding area and analyzer clean. Do not touch any internal components or allow anything inside the analyzer.

1. Turn off the analyzer and make sure the surrounding area is clean.
2. Collect a Prolene® Measurement window from the bin labeled 'XRF Supplies', a small Phillips head screwdriver, and a box of KimWipes
3. Orient the analyzer so the nose is pointing sideways. This will help minimize the likelihood of anything falling into the analyzer.
4. Remove the two screws on the plate using the Philipps Head screwdriver (Figure)





5. Swing the face plate outward. The film window is now exposed.



6. Remove the damaged window.
7. Take the new window, grabbing it by the edges, and place it on the plate. Orient the window so the backside (side fully covered with film) facing upward.
8. Double check the white window spacer and black rubber gasket are seated properly, and then rotate the hinged plate upwards.
9. Insert the screws and tighten until snug.

*Directions and images for this section were provided by Olympus Delta Family User Manual. For more details on this procedure please refer to the 'DELTA\_Family—User—International Guide'.*

#### Add User Defined Fields

1. Go to 'Setup' tab at the top and then 'Result Test Info' in the left panel.
2. Select 'Customize..'
3. Labels with the Red Check are enabled. Select the 'enable' box next to the next Test label number.
4. On the right side select the appropriate test label number from the dropdown.
5. Define the 'style' from the next dropdown as 'text', 'fixed list', 'auto incrementing order'.
6. Give the test label a name.

The label is now created and it has to be setup for being exported.

1. In the left panel go to 'Data Management'
2. Select 'Customize' underneath the Data and Spectra boxes.
3. Under the 'Fields' panel select the test label number you just made from the 'Not Exported' panel. Then click the left pointing arrow to move it into the exported field.
4. Click the 'Up' button to move the label into position. The vertical order transposes left to right in the .csv and lore report.
5. Click 'Save' at the bottom of the screen.

#### User Factor

The pXRF has a feature to apply linear regressions for elements, quantifying data as it is measured. It is NOT recommended for use. Data Quantification should be done outside of the software by the scientists. Please note that this feature can provide erratic results and not detect elements that are present.

## Troubleshooting

### Indicator Bar shows a 'CommError'

This indicates a Communication Error between the pXRF and the computer. If this error shows up at the initial startup window, close and restart the software. The gun must be connected via the micro-USB cable before turning on the software. If this error shows up while the software is already open, close and restart the software. In this instance the connection between the analyzer and computer was broken, either through the cable being unplugged or the Gun turned off. If that happens.

### Files are missing from the Data Management Panel

This could be a range of issues:

1. (Most Likely) Connection between the analyzer and computer was broken before files were exported. If a file is not exported before the communication is broken, either intentionally or unintentionally, the measurements are not saved.
2. The memory card on the XRF Gun was too full and the gun started deleting files
3. The software versions on the PC and the analyzer do not match.

### Looking through the results tab and it shows no or a partial list of samples

A 'filter' has likely been applied to the results display. Select 'Filter' in the lower right hand corner and adjust as needed to include the results.

### Cal Check Fails

Go through these steps:

Remove the gun and clean the stainless steel disk and Prolene<sup>®</sup> window. Try to measure again. If it still fails, restart the software and gun then try again (make to export any data first). If it still fails, replace the stainless steel disk and measure again. If none of this resolves the issue, then contact Olympus and David Houpt.

## Health, Safety, and Environment

### Handheld XRF Analyzer Safety Design

To control X-ray emissions, and thereby minimize the possibility of accidental exposure, the **Olympus DELTA Premium** pXRF analyzer has a standard **safety interlock structure** consisting of two features listed below. Be sure to understand the **software trigger lock** setting to understand whether the trigger is "armed" or not.

#### Software proximity sensor

Within two seconds of starting a test, the analyzer attempts to detect backscatter radiation from the sample. If it fails to detect this backscatter radiation (i.e., no sample is detected), the test aborts to prevent accidental exposure. Upon an abort action, the filter wheel returns to the 0 position, the X-rays shut off (the tube current decreases to 0.0  $\mu\text{A}$ ), and the red light stops blinking. If the probe is pulled away from the sample while a test is in progress, testing will stop and X-rays will shut off.

This safety works slightly differently inside the sample chamber. If the pXRF starts measuring inside of the sample chamber without a sample inside the X-rays will continue emitting until it switches to Beam 2. At the start of the second beam the analyzer will abort the measurement. Beam 1 is a higher voltage and the analyzer detects the x-rays bouncing off the walls in there. If a run was started but a sample was not put in the chamber it is **NOT SAFE** to open the sample chamber door.

### Software trigger lock

After a five-minute lapse between tests (default time), the trigger automatically locks. The pXRF analyzer also has a dead man trigger protocol, in which the user is required to pull and hold the trigger for the duration of the test. When the dead man trigger is enabled, releasing the trigger prematurely will abort the test. However, it is not recommended to use the trigger—instead, trigger the measurements from the PC, and thus further away from the instrument.

## Indicators and Alarms

### X-ray Indicator

An X-ray indicator alerts the operator when the tube is receiving power and when X-rays are emitted from the analyzer through the measurement window. The X-ray indicator is located on the upper rear portion of the analyzer. This indicator consists of a six-element red LED array, and has three states:

#### *X-ray Indicator Lights OFF*

This signifies that the X-ray tube is disabled, and that there is no possibility of radiation exposure to you or bystanders. The instrument can be carried or set down safely in this condition.

#### *X-ray Indicator Lights Continuously ON*

**Solid red LED array.** This signifies that the X-ray tube is enabled, and that there is no radiation exposure to you or bystanders. The instrument can be carried or set down safely in this condition.

#### *X-ray Indicator Lights Flashing ON and OFF*

**Blinking red LED array.** This signifies that the X-ray tube is powered on and that the analyzer is emitting X-ray radiation through the measurement window. In this condition, the analyzer must be pointed toward a test sample and **never** at a human being.

### Audible Alarm Protocol

When enabled, the **audible signal** emits three tones as a warning when the X-ray tube is about to emit X-rays. During the subsequent testing period, the audible alarm maintains a “chirping” signal throughout the duration of the test. If the pXRF is to be set up in a potentially high-traffic area (which is not recommended in the first place, but could be necessary), it is suggested to use this feature to help minimize exposure of bystanders.

### Infra-Red Sensor

The pXRF has an **infrared sensor** that detects the presence of an object in front of the pXRF source. If no object is detected, the pXRF will not activate the source.

## Radiation Safety Tips for Using the pXRF

All operators should follow minimum safety requirements discussed below. When handled properly, the amount of radiation exposure received from the pXRF will be negligible. However, the following safety tips are provided to help ensure safe and responsible use:

1. **WARNING:** No one but the operator should be allowed to be closer than 3 feet from the pXRF, particularly the beam port. Ignoring this warning could result in unnecessary exposure.
2. **WARNING:** The operator should never defeat the IR sensor in order to bypass this part of the safety circuit. Defeating this safety feature could result in over-exposure of the operator.
3. Do not allow anyone other than trained personnel to operate the pXRF.
4. Be aware of the direction that the X-rays travel when the red light is blinking and avoid placing any part of your body (e.g., eyes, hands) near the X-ray port to stabilize the instrument during operation.
5. Never hold a sample up to the X-ray port for analysis by hand; hold the instrument to the sample.
6. Establish a no-access zone at a sufficient distance from the instruments measurement window, which will allow air and distance to attenuate the beam.
7. Enclose the beam working area with protective panels (e.g., >3.0 mm stainless steel) if practical.
8. Wear an appropriate dosimeter (see the Laboratory Officer for more information on when a dosimeter is called for).
9. The operator is responsible for the security of the handheld XRF. When in use, the device should be in the operator's possession at all times (i.e., either in direct sight or a secure area).
10. Always store the instrument in a secure location when not in use.
11. During transport to and from the set up location, store the instrument in a cool, dry location.
12. **WARNING: Pregnant women should not use the pXRF or work in proximity to it. See Additional Note, Pregnancy, above, for more information. Radiation exposure can be harmful to an embryo or developing fetus!**