

Chapter 1 Introduction

1.0 INSPECTING YOUR INNOV-X ANALYZER

Upon receipt:

1. Locate and remove the shipping papers and documentation from under the lid's foam padding.
2. Remove the Innov-X Analyzer and all of the components from the protective carrying case and identify each on the enclosed shipping list.
3. Connect the battery charger to an 110V-240V AC power source. Place one Li-ion battery on the charger and charge it for at least 2 hours. Charge the second battery.
4. Charge the HP iPAQ using the attached AC adaptor for at least ½ hour.
5. Read and review the "Quick Start" section of the User's Manual. Innov-X recommends that you read the entire manual.
6. Install the fully charged battery into the analyzer.
7. Press the ON/OFF button on the back of the analyzer and the power button on the iPAQ.
8. Select Innov-X from the start menu located in the upper left hand corner of iPAQ screen.
9. Select the desired analysis mode (i.e., Analytical, FastID, Pass/Fail or Soil). The instrument will undergo a one minute hardware initialization period.
10. Standardize the instrument with the 316 Stainless Steel mask. Standardize the instrument every 4 hours or as directed by the display.
11. Release the software trigger lock and analyze a sample of known composition, in order to verify the correct operation of the analyzer.
12. Analyze samples of unknown composition.

1.1 COMPONENTS INCLUDED WITH THE ANALYZER

Shown here are the various items which are included with the Innov-X portable XRF analyzer. Unless otherwise noted, all items are standard accessories.



Analyzer, with iPAQ attached.



Two, Li-ion batteries (one shown).



Battery charger and an AC adaptor. Battery shown mounted in charging system.



Standardization cap and weld mask (optional)

The standard standardization cap has no weld slit.



iPAQ cradle and AC adaptor. The cradle is used to connect the iPAQ to a PC for downloading data and reports.



Testing stand. This is the benchtop docking station for the analyzer. It is an optional accessory

1.2 QUICK START INSTRUCTIONS

The following section provides a quick overview to using the Innov-X portable XRF analyzer. This is intended to provide the basic startup and operational instruction needed to perform simple analyses. It is highly recommended that the user read the sections on Radiation Safety (Chapter 3) and the detailed description on operation (Chapter 4). The following Quick Start information is also provided as a separate, bound, laminated publication for quick reference.

1. Place a battery in the analyzer.
2. Power on the Analyzer (On/Off switch located on back of analyzer)
3. Power on the iPAQ (Button located in upper right hand corner of iPAQ)
4. Select Innov-X from the start menu located in the upper left hand corner of iPAQ screen.
5. Read the radiation safety notice and acknowledge that you are a certified user by pressing Start.
6. Select Desired Mode.
7. The analyzer will undergo a 60 second hardware initialization.
8. Place a standardization clip on the nose of the analyzer. Tap the button on the screen to standardize. (*Manual section 4.4 Standardization*)
9. When standardization is complete, remove the standardization clip.

10. Release the software trigger lock by tapping the locked icon on the iPAQ screen and tapping yes in response to the software prompt.
11. Test standard to verify instrument performance.
12. Results will display on screen. Subsequent tests may be started from either the Results or Analysis screens.

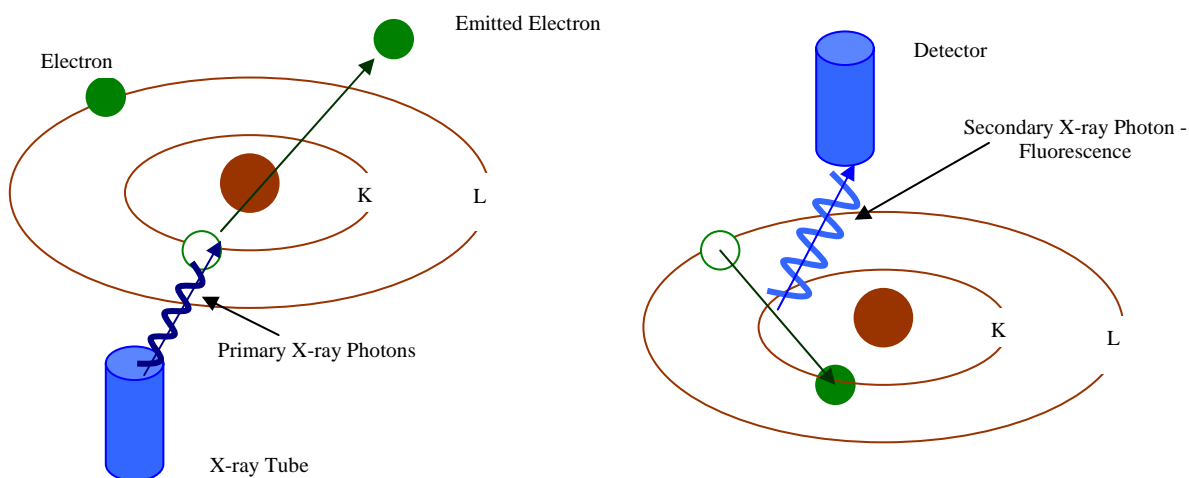
1.3 INTRODUCTION TO XRF: X-RAY FLUORESCENCE SPECTROMETRY OVERVIEW

Basic Theory

Although most commonly known for diagnostic use in the medical field, the use of x-rays forms the basis of many powerful analytical measurement techniques, including X-ray Fluorescence (XRF) Spectrometry.

XRF Spectrometry is used to identify elements in a substance and quantify the amount of those elements present. An element is identified by its characteristic X-ray emission wavelength (λ) or energy (E). The amount of an element present is quantified by measuring the intensity of its characteristic line. XRF Spectrometry ultimately determines the elemental composition of a material.

All atoms have a fixed number of electrons (negatively charged particles) arranged in orbitals around the nucleus. The number of electrons in a given atom is equal to the number of protons (positively charged particles) in the nucleus; and, the number of protons is indicated by the Atomic Number in the Periodic Table of Elements. Each Atomic Number is assigned an elemental name, such as Iron (Fe), with Atomic Number 26. Energy Dispersive (ED) XRF and Wavelength Dispersive (WD) XRF Spectrometry typically utilize activity in the first three electron orbitals, the K, L, and M lines, where K is closest to the nucleus. Each electron orbital corresponds to a specific and different energy level for a given element.



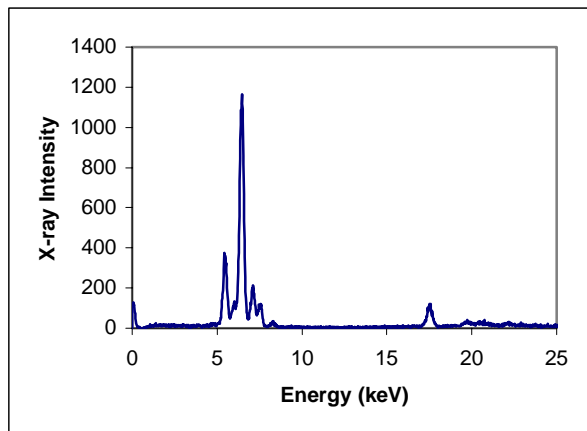
In XRF Spectrometry, high-energy primary X-ray photons are emitted from a source (X-ray tube) and strike the sample. The primary photons from the X-ray tube have enough energy to knock electrons out of the innermost, K or L, orbitals. When this occurs, the atoms become ions, which are unstable. Electrons seek stability; therefore, an electron from an outer orbital, L or M, will move into the newly vacant space at the inner orbital. As the electron from the outer orbital moves into the inner orbital space, it emits an energy known as a secondary X-ray photon. This phenomenon is called fluorescence. The secondary X-ray produced is characteristic of a specific element. The energy (E) of the emitted fluorescent X-ray photon is determined by the difference in energies between the initial and final orbitals of the individual transitions.

This is described by the formula

$$E=hc/\lambda$$

where h is Planck's constant; c is the velocity of light; and λ is the characteristic wavelength of the photon.

Wavelengths are inversely proportional to the energies; they are characteristic for each element. For example the $K\alpha$ energy for Iron (Fe) is about 6.4keV. The number of element-specific characteristic X-rays produced in a sample over a given period of time, or the intensity, can be measured to determine the quantity of a given element in a sample. Typical spectra for EDXRF Spectrometry appear as a plot of Energy (E) versus the Intensity (I).



History

Wilhelm Roentgen discovered X-rays in 1895. Methods for identifying and quantifying elements using XRF were first published by Henry Moseley in 1913. Much research and development of XRF continued after Moseley's pioneering work, especially during WWII when rapid developments in the aircraft, automotive, steel and other metals industries heightened the need to identify alloys quickly and reliably. However, the first commercial XRF Spectrometers weren't available until the early 1950's. Those systems were based on WDXRF technology and measured the characteristic wavelength of an element, one element at a time. Although the use of these systems was critical for elemental analyses, they were large, expensive, and required highly skilled operators to use and maintain them.

In the late 1960's, EDXRF technology, which measures the characteristic energy of an element, began to rival the use of WDXRF due to the development of Si (Li) solid state detectors, which offered better energy resolution of the signal. EDXRF systems offered the potential of collecting and displaying information on all of the elements in a sample at the same time, as opposed to one at a time with typical WDXRF systems. Many of the early EDXRF systems used radioisotopes for excitation instead of X-ray tubes, which could require changing sources to determine all the elements of interest. Some of those early EDXRF systems did not easily resolve multiple elements in a single analytical run.

As can be imagined, the equipment and applications of XRF Spectrometers have developed tremendously since the 1960's. Advancements in technology, electronics, computers, software and the use and modification of them for XRF Spectrometers by instrument manufacturers, research scientists & engineers, and industrial users alike have led to the current state of the art in XRF Spectrometers. Now a mature technology, XRF Spectrometry is routinely used for R&D, QC and analytical services in support of production.

Elemental Analysis

XRF Spectrometry is the choice of many analysts for elemental analysis when compared to the other techniques available. Wet chemistry instrument techniques for elemental analysis require destructive and time-consuming specimen preparation, often using concentrated acids or other hazardous materials. Not only is the sample destroyed, waste streams are generated during the analytical process that need to be disposed of, many of which are hazardous. These wet chemistry elemental analysis techniques often take twenty minutes to several hours for specimen preparation and analysis time. All of these factors lead to a relatively high cost per sample. However, if PPB and lower elemental concentrations are the primary measurement need, wet chemistry instrument elemental analysis techniques are necessary.

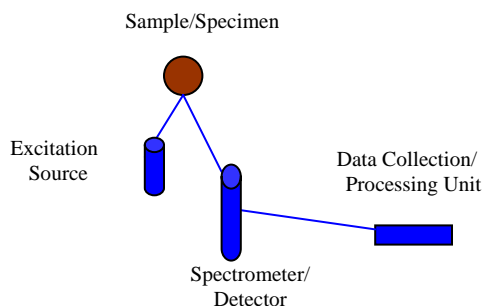
XRF Spectrometry easily and quickly identifies and quantifies elements over a wide dynamic concentration range, from PPM levels up to virtually 100% by weight. XRF Spectrometry does not destroy the sample and requires little, if any, specimen preparation. It has a very fast overall sample turnaround time. These factors lead to a significant reduction in the per sample analytical cost when compared to other elemental analysis techniques.

All elemental analysis techniques experience interferences, both chemical and physical in nature, and must be corrected or compensated for in order to achieve adequate analytical results. Most wet chemistry instrument techniques for elemental analysis suffer from interferences that are corrected for by both extensive and complex specimen preparation techniques, instrumentation advancements, and by mathematical corrections in the system's software. In XRF Spectrometry, the primary interference is from other specific elements in a substance that can influence (matrix effects) the analysis of the element(s) of interest. However, these interferences are well known and documented; and, instrumentation advancements and mathematical corrections in the system's software easily and quickly correct for them. In certain cases, the geometry of the sample can effect XRF analysis, but this is easily compensated for by grinding or polishing the sample, or by pressing a pellet or making glass beads.

Quantitative analysis for XRF Spectrometry is typically performed using Empirical Methods (calibration curves using standards similar in property to the unknown) or Fundamental Parameters (FP). FP is frequently preferred because it allows elemental analysis to be performed with no standards or calibration curves. This enables the analyst to use the system immediately, without having to spend additional time setting up individual calibration curves for the various elements and materials of interest. The capabilities of modern computers allow the use of this no-standard mathematical analysis, FP, accompanied by stored libraries of known materials, to determine not only the elemental composition of an unknown material quickly and easily, but even to identify the unknown material itself.

EDXRF Spectrometers

EDXRF Spectrometer systems are mechanically very simple; essentially there are no moving parts. An EDXRF system typically has three major components: an excitation source, a spectrometer/detector, and a data collection/processing unit. The ease of use, rapid analysis time, lower initial purchase price and substantially lower long-term maintenance costs of EDXRF Spectrometers have led to having more systems in use today worldwide than WDXRF Spectrometer systems.



EDXRF has been found most useful for scrap alloy sorting, forensic science, environmental analysis, archaeometry and a myriad of other elemental field-oriented analyses.

Handheld EDXRF Spectrometers for Field Analyses

It is clear that a future trend for elemental analysis is in rapid site investigation using techniques that are fast, inexpensive, reliable, and long-term cost effective. There is a need for immediate decisions to be made during the delivery of materials, industrial processing, and in the field for positive materials identification or environmental site assessment and remediation. It is also clear that EDXRF Spectrometry is the most suitable elemental analysis technique available for field analysis due to its simplicity, speed, precision, accuracy, reliability, and overall cost effectiveness.

Recent technological developments in cell phones, pocket PC's and other portable consumer electronics have led to the advancement of many high-performance, miniature components. X-ray equipment manufacturers began to take advantage of these developments in the late 1990's and developed Handheld EDXRF systems. An obvious advantage of Handheld EDXRF systems is that the analyzer is taken to the sample as opposed to bringing the sample to the analyzer and configuring it to fit in an analysis chamber. In addition to the per sample analytical cost savings, a key factor in using non-destructive EDXRF analysis, especially in the field, is the overall project cost savings due to improved and more timely decision making. The use of EDXRF for immediate positive materials identification or to guide an environmental site characterization will generally reduce the overall time required in the field due to the quick turnaround for the sample analysis; this invariably reduces the overall costs of analytical field work.

Of course, Handheld EDXRF technology has continued to evolve in concert with portable consumer electronic developments. Just like the early Benchtop EDXRF systems, early Handheld EDXRF systems used radioisotopes for excitation. There are several practical problems with the use of radioactive isotopes for handheld systems. The source decays and loses its testing speed over time. In addition to the loss in analytical capabilities, the sources have to be replaced incurring a cost. The use of radioactive isotopes also requires licensing (state-to-state in the US) and a radioactive materials control program; they are difficult to ship and transport, as they require hazardous materials declarations and/or permits. Consequently, the newest and most exciting development in Handheld EDXRF technology is the use of battery operated, miniature X-ray tubes, which was pioneered by the staff at Innov-X Systems.

Innov-X Systems Handheld EDXRF Spectrometers

Innov-X Systems specializes in Handheld EDXRF technology with the most advanced miniature components available for X-ray Tube sources, detectors, and PC 's. Innov-X Systems Handheld EDXRF Spectrometers are ideally suited for field analysis of alloys, lead-based paint, environmental soils, filters, dust wipes, forensics, archaeometry, and a variety of other elemental analyses in the field or around the plant. Innov-X Systems EDXRF Spectrometers are affordable, easy to use, reliable, and overall cost effective. The Innov-X Systems Handheld EDXRF units incorporate state-of the art components including a battery operated miniature X-ray tube, a high-resolution silicon pin detector, high speed data acquisition circuitry, and a Compaq IPAQ Pocket PC[®] handheld computer for calculations, results and operator interface.

Innov-X Systems EDXRF Spectrometers offer the following invaluable features:

- Portable
- Battery operated, rechargeable
- X-ray Tube-based (Ag or W anode, 10-40kV, 10-100uA)
- Si PiN diode detector.
- Integrated pocket PC
- Pistol-shaped design for difficult testing locations and welds
- Auto-compensation for irregular or small samples
- Fundamental Parameters for no-standard analyses
- Stored Grade Libraries for rapid Grade ID's
- Stored Fingerprint Libraries for rapid material ID's

- Docking station available for use as standard benchtop unit
- Results shown after a few seconds of testing time.

For more information on how to utilize your Innov-X Systems Handheld EDXRF Spectrometer optimally, please review this Instruction Manual or contact us directly.

Chapter 2. Usage and Assembly of Accessories

2.0 ACCESSORIES

This chapter describes the various accessories that are provided with an Innov-X XRF analysis system. Included are:

- Batteries
- Battery Charger
- iPAQ cradle and charger
- Testing Stand Assembly (not standard with all units)
- Standardization Clip or Standardization Clip/Welding mask.

2.1 ANALYZER BATTERY

The Innov-X Systems XRF Analyzer is powered by a replaceable, rechargeable Lithium ion battery. In addition, the iPAQ has its own internal battery.

Innov-X Systems Main Battery

The Innov-X Analyzer uses a rechargeable Lithium Ion Smart Battery. A picture of the battery is shown in Fig. 2.1. Two batteries are included with each analyzer. The batteries are charged an external battery charger. Batteries typically function for 4 to 8 hours, depending on usage patterns. Heavier duty cycles deplete the battery more quickly. Therefore, users who do longer and more frequent tests will need to replace their batteries more often than users who take shorter or fewer tests.



Figure 2.1. Li-ion Battery for analyzer

Replacement batteries can be purchased directly by calling Innov-X Systems at 781-938-5005. (P/N A003)

Battery power indicators:

There are two ways of determining the charge remaining on a battery: the LED indicator on the battery and the battery status icon on the analyzer screen. The battery icon, when tapped, will indicate the percent charge remaining on a battery inside the analyzer. Additionally, the battery icon will change from green to yellow when the battery gets low, indicating it has about 15 minutes left of charge.

To use the battery LED, push the button below the indicator. The lighting will indicate the % of charge. If possible, try to use batteries with at least 50% of their full charge, according to the indicator.

2.2 CHANGING A BATTERY

To change a battery, perform the following steps:

1. Hold the instrument by the handle, upside down, so the bottom of the instrument base is pointing upward. Please refer to Fig. 2.2.
2. Hold the instrument so that the nose is pointing away from the operator.
3. Open the battery door on the bottom of the handle. The batteries have a small tab attached for ease of removal.

4. Pull out the existing battery, and replace with a new battery.
5. Insert the charged battery into the analyzer such that the connectors on the top of the battery are facing to the right. Note that the battery slot is keyed so that the battery can only be inserted one way.



Figure 2.2a. Instrument handle. Pull the rubber latch and lift door. Reach into opening and remove battery.



Figure 2.2b Insert new battery into opening.

2.3 BATTERY CHARGER

The battery charger is shown in Fig. 2.3. It takes about 2 hours to completely charge a battery. The status of the charger is shown by two lights on the power adaptor. Table 2.1 lists the information conveyed by the lights.



Figure 2.3. Battery charger.

Left Light	Right light	Status
On	Off	Battery is charging
On	On	Battery is 80% charged
Off	On	Battery is completely charged
Blink	Blink	Error. Remove battery and replace on charger. If error persists, call Innov-X Systems Technical support.
Off	Off	No battery is on charger

Table 2.1 Battery charger status lights

2.4 HP IPAQ POCKET PC BATTERY

The iPAQ has an internal rechargeable battery, which can be recharged by using the power adaptor that is included with the unit. This adaptor can be connected either to the iPAQ itself, or to the cradle. If it is connected to the cradle, and plugged in, the iPAQ will recharge whenever it is placed in the cradle. In addition, the iPAQ Battery will recharge whenever the iPAQ is mounted in an Innov-X analyzer which is powered, but not actively taking a test. The amber light on the top of the iPAQ will blink whenever the battery is charging. It will remain solid when the battery is completely charged.

Since the iPAQ will be recharged whenever the Innov-X Systems Analyzer is in use, it may never be necessary to use the iPAQ power adaptor. However, care should be taken when the analyzer is not used for a period of several days, as the iPAQ uses some power even when it is powered off. It is therefore possible to completely discharge the battery simply by not using the iPAQ for several days, or by using it for several hours without recharging it.

If you do not use your Innov-X Analyzer on a daily basis, or if you will have a down period of more than several days, it is recommended that you remove the iPAQ from the Analyzer when it is not in use and plug in the iPAQ to a power outlet to recharge it. This will ensure that your iPAQ is always charged and ready for use. You should also always plug in the power cord whenever the iPAQ is removed from the analyzer for data transfer.

If you do allow the iPAQ battery to discharge significantly, either by allowing it to sit too long unused, or by using it for a period of time without it being connected to a power source, it may not be possible to operate your analyzer. If this happens, the Innov-X software will provide an error message indicating that the iPAQ battery is too low. Recharge the iPAQ for at least a half an hour before attempting another measurement.

If the iPAQ battery is completely discharged, it will not be possible to turn on the iPAQ until it is recharged. A complete power failure will erase anything that is stored in the Main Memory of the iPAQ. All Innov-X program and data files are stored on the storage card, rather than in Main Memory, so you will not lose any data or have to reinstall the Innov-X software.

1. If the battery on the iPAQ is completely discharged, charge it for at least one half hour.
2. You will be required to follow the prompts on the iPAQ screen before you can use the iPAQ. This procedure involves realigning the screen by tapping in several spots, and going through a quick tutorial.
3. The iPAQ will reinitialize the Innov-X Systems software. A message will appear indicating that this is going to happen. You must tap ok to initialize.
4. The software will open automatically; a message will appear indicating that several registries have been restored. Tap ok to dismiss this message.
5. Set the clock to the current time. **Note, this is very important**, as your data is indexed by date. If the date in the iPAQ is incorrect, you may not be able to locate your results. The instrument will not allow you to take a reading until the date has been changed.
 - a. From the Start Menu, tap Settings.
 - b. Select the System tab, and tap clock.
 - c. Set the proper date. Further details about this procedure can be found in the HP iPAQ user's manual.

2.5 REMOVING THE IPAQ FROM THE ANALYZER

It is very important to properly remove the iPAQ Pocket PC from the analyzer to avoid damaging the connector on the back of the iPAQ.

In order to remove the iPAQ, push the iPAQ retainer shown in Fig. 2.4 towards the front of the analyzer. Holding the retainer forward, grab the iPAQ from the sides, slide the iPAQ forward until it is clear of its

connector, then tilt the front end up enough so it clears the front holder allowing the iPAQ to be lifted out of the instrument.

Note: Never grab the iPAQ and twist it side-to-side to remove it from the analyzer. Always move the iPAQ retainer forward as instructed above, slide the iPAQ forward and remove from the analyzer.



Figure 2.4. Removing the iPAQ from the analyzer.

2.6 STANDARDIZATION CAP and/or WELD TESTING MASK

All analyzers are supplied with either a standardization cap or a combination standardization cap welding mask. The standardization mask is the standard accessory. Welding masks can be purchased as an additional accessory, or in lieu of the standardization mask.

Standardization Cap

The cap clips on the front end of the analyzer and is used to standardize the system as described in Chapter 4. To attach the cap, snap it onto the nose of the analyzer over the Kapton window.

Combination Standardization Cap/Welding Mask

The standardization/welding mask is shown in Fig. 2.5. The cap clips onto the front end of the analyzer and is used to standardize the system as described in Chapter 4. To attach the cap, snap it onto the nose of the analyzer over the Kapton window. Be sure that when attaching the cap, that the solid end (as opposed to the end with the 1/4" wide slit) is covering the window. To remove the mask, slide it off to either side.

The opposite end of the standardization cap serves as a welding mask. This mask is used to shield the base metal from analysis, when analyzing a weld. It is important to use this mask since failure to do so will produce an alloy chemistry that is a mixture of the base metal and the actual weld. For best results:

- a. Use the welding mask only for welds that are larger than the opening in the mask;
- b. Make solid contact between the surface of the mask and the material to test;
- c. Use the mask only in the Analytical Mode – not with the standard Fast ID library;
- d. Consider using longer test periods to compensate for the smaller testing area – especially with more difficult separations.

If it is desirable to use the welding mask in FastID mode, a user can create a special “Welding Mask Library.” Teach all relevant alloys with the welding mask in position. Make sure these fingerprints are

saved in library that contains ONLY fingerprints taught with a welding mask. When measuring a weld, make sure the “Weld” library is the only one selected. By creating a special finger print library using the welding mask, a user can get good results in the Fast ID Mode as well.



Figure 2.5 Standardization cap and welding mask. (Optional accessory)
The standard standardization cap does not have the welding slit.

2.7 TESTING STAND (optional accessory)

The testing stand is designed as a docking station for the handheld analyzer. It can be used as a bench-top system, or to test small samples. A list of components and an assembled stand is shown in Figure 2.6:



Figure 2.6. Assembled Testing Stand

Components of the testing stand:

1. Three (3) short legs
2. Three (3) long legs
3. Lower Stand
4. Upper Stand
5. Four (4) knobs for top plate
6. Test stand cradle
7. Clip for cradle.
8. Adaptor cable (connects serial connector on iPAQ cradle to auxiliary port on analyzer)

Assembly of Testing Stand

1. Insert the three Short Legs through the holes in the Lower Stand by inserting the threaded screw through the holes. This will balance the Lower Stand on the table top. (Fig. 2.7).



Figure 2.7. Mounting Lower Stand onto Short Legs.

2. Mount the three Long Legs onto the Lower Stand by inserting the threaded screws from the Short Legs into the holes on the Long Legs and turning until snug. Remove iPAQ from analyzer by following the instructions in Figure 2.4. Place the analyzer into the gap in the Lower Stand as shown. (Fig. 2.8).



Figure 2.8. Mounting Long Legs onto Lower Stand and inserting analyzer.

3. Mount the Upper Stand onto the Long Legs. The Upper Stand has holes for the screws at the end of each of the Long Legs. The Upper Stand will also fit snugly over the front end of the analyzer. Be sure that the Upper Stand is mounted so that all three screws are inserted through the holes, and the front end of the analyzer is flush with the top surface of the upper stand. (Fig. 2.9).



Figure 2.9. Mounting Upper Stand onto Testing Stand.

5. Put three knobs to secure testing stand onto analyzer. The iPAQ clip can be secured with any of the knobs. This clip grabs the base of the iPAQ cradle to hold the iPAQ securely in place.

6. Place the iPAQ in the cradle and connect it to the Auxiliary Port on the analyzer using the serial cable adaptor.



Figure 2.10. Connecting iPAQ to Auxiliary Port on analyzer.

Chapter 4 Operation

4.0 OPERATION - GENERAL

Power to the instrument is controlled by the ON/OFF button located at the rear of the analyzer. The green LED next to this button will illuminate when the analyzer power is on. The iPAQ operates on the Microsoft Windows CE ® operating system and is activated separately by the power button on the right top face, just over the display. The trigger is locked via the software.

4.1 WORKING WITH THE HP iPAQ Pocket PC®

The Microsoft Windows CE ® operating system and Innov-X software provided on the iPAQ handheld computer are operated by user input through the touch screen. For comprehensive details on the iPAQ's operation, please refer to the iPAQ reference materials included with your unit.

General tips

- The Start Menu is found in the upper left corner of the iPAQ screen. This is used to launch all applications, including the Innov-X Systems Analyzer software.
- The instrument is designed as a “point and shoot” system that requires little, if any, entry of information for most operations. In the event the user modifies the grade library, enters testing information data, or performs other functions, it will be necessary to enter data via the virtual keyboard, which can be accessed by tapping the keyboard icon in the lower right corner. The iPAQ also includes character recognition software. This can be selected from the drop-down menu to the right of the keyboard icon.
- The File toolbar which will be used to Change Functions, Screens and Options is located at the bottom of the screen.
- It is possible to cut, copy, rename and delete files from within Windows File Explorer by selecting the file to be modified and holding the stylus on the screen for 2 seconds.
- Pressing buttons on the bottom of the iPAQ will perform various functions that are described in the iPAQ documentation. The button on the right hand side of the analyzer is the iPAQ task manager. Pressing this button will show all programs that are currently open. Open files can be closed from this menu. Simply hold the stylus on the file for a few seconds. The option to close the file will appear.

4.2 OPERATION - MAIN SOFTWARE SCREENS

The Innov-X Software consists of three main screens:

- **Main Menu screen:** Used to select the analysis mode, open the results screen, and change the administrator password.
- **Analysis Screen:** Used to change settings, edit libraries, and perform tests.
- **Results Screen:** Displays results from current reading, allows scrolling back to previous test results. Allows recorded data to be exported to a comma delimited file which is directly compatible with Microsoft Excel.

4.2.1 Innov-X Main Menu

The main menu below appears upon startup. The Main Menu allows you to choose an analysis mode, as well as perform certain administrative functions such as changing your login password. The modes which

are available on the analyzer are shown in blue. For information on adding additional analysis modes to an analyzer, please contact the Innov-X Sales Department at 781-938-5005.

- **Use the Main Menu to select the desired analysis mode.**
The analysis mode can be selected by either tapping on the name of the method (shown in blue) or by selecting the appropriate mode from the Modes menu.
- The administrative password can be changed by selecting **Options → Change Password.**
- It is possible to go directly to the Results Screen by selecting **View→Results.** If the results screen is opened in this manner, it is possible to view results when the iPAQ is not connected to the analyzer.

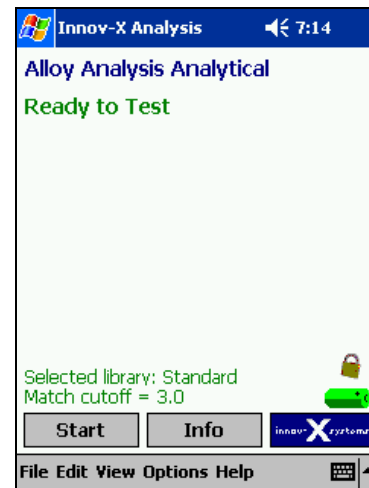


4.2.2 The Analysis Screen

Selecting a mode opens the analysis window for that mode. All data acquisition and analyzer control are done from this window. This window allows the user to start or stop an analysis, change testing parameters, and modify the fingerprint and grade libraries (Alloy Analysis only).

The analysis screen runs continually while during normal instrument operation. From the results menu, it is always possible to go back to the Analysis screen by selecting **File→Exit** or by tapping the X in the upper right hand corner of the screen.

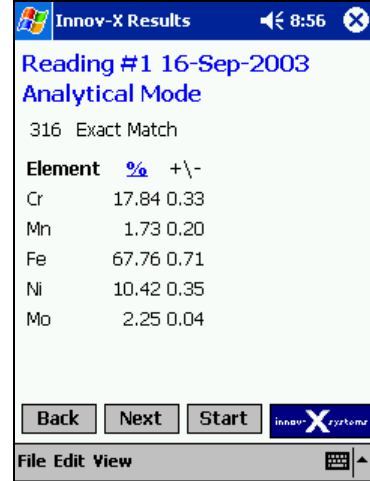
The analysis screen for Analytical mode is shown to the right. Screens from other modes are similar and will be described in later in this manual. The analysis screen shows the name of the mode that is currently active, a start/stop button (which is inactive in most cases), an info button that is used to enter descriptive information for any given test, a trigger lock and a battery indicator. In addition, a message appears directly below the name of the mode which will indicate the current state of the analyzer. Typically it reads “Ready to Test,” but also provides other information in certain circumstances. Any mode specific information will be displayed at the bottom of the screen above the menu choices.



4.2.3 The Results Screen

The Results screen displays the current reading and old data. All data handling functions such as exporting and deleting readings are carried out from this screen. Once the Results Screen is open, the user may start new tests without going back to the analysis screen by pulling and holding the trigger. Tapping the X in the upper right hand corner will return the user to the analysis screen without starting a test. If no analysis mode is running, an Exit button will appear which will close the Results screen.

The Results screen is automatically shown at the completion of any analysis. It can also be accessed from the analysis screen for any mode or the **Main Menu**, by selecting *View*→*Results*. Once the Results screen has been opened, the information which is displayed can be changed by selecting options from the View menu. The various viewing options will be described in detail in later chapters.



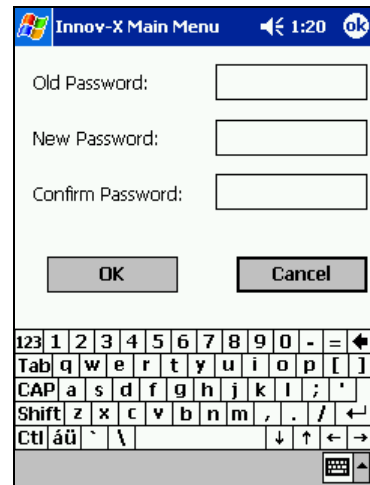
4.3 PASSWORDS - ABOUT PASSWORD PROTECTION

Certain functions such as adding and deleting fingerprints from the libraries, and Pass/Fail setup have been specified as Administrative Level Functions. These functions are described in detail in later sections of the manual. In order to use these functions, a password must be entered. The default password is set as the lowercase letter “z”. This password can be entered whenever the system prompts for a password.

Changing the Administrator Password.

The Administrator password may be changed at any time from the **Innov-X Main Menu** by choosing *Options*→*Change Password*. When the change password option is selected, this screen will appear.

If you are changing the password for the first time, enter the letter “z”; otherwise enter the current system password. Then, choose a password and enter it twice, once in the “New Password” box and again in the “Confirm Password” box. Passwords may be any combination of letters or numbers.



4.4 STANDARDIZATION

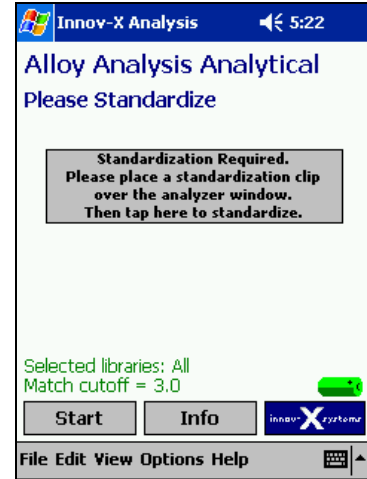
4.4.1 Standardization Procedure

Before performing tests, it is necessary to standardize the instrument. This automated procedure involves collecting a spectrum on a known standard (Alloy 316) and comparing a variety of parameters to values stored when the instrument was calibrated at the factory. If there are any problems with the instrument, they will be indicated by an error message.

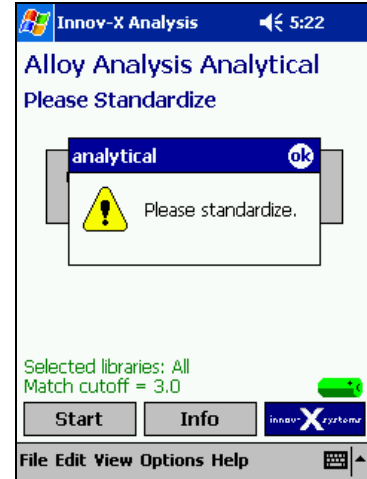
The standardization procedure takes about 1 minute. Standardization must be done any time the analyzer hardware is initiated or restarted and must be repeated if the instrument is operating for more than 4 hours.

It is possible to re-standardize the instrument at any point while the software is running. Standardization is always initiated from the Analysis Screen of any Mode.

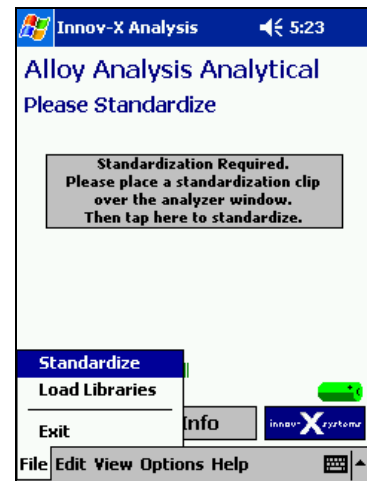
If the analyzer is restarted, you will be required to standardize the instrument before performing any measurements. This is indicated by the message “**Standardization Required. Please place a standardization clip over the analyzer window. Then tap here to standardize.**” on the analysis screen



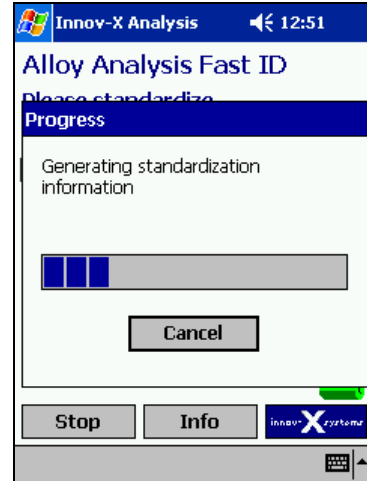
It is not possible to start a test before standardization. If the trigger is pulled before the standardization procedure is completed, a message box will appear. Press **ok** to acknowledge and clear the message.



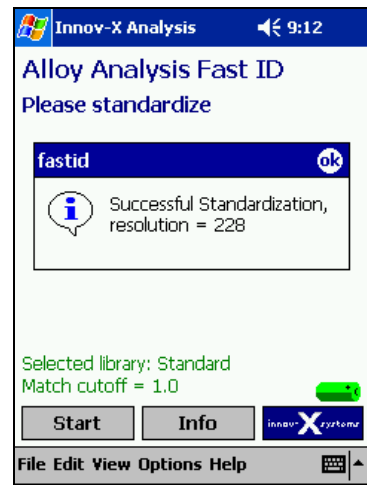
To initiate the standardization procedure, snap the standardization piece on the front of the instrument. Verify that it completely covers the analyzer window. When using a standardization mask with a weld collimator, be sure that the solid portion of the mask covers the analyzer window. Tap the grey box in the center of the screen or select **File**→**Standardize** to begin.



When standardization is in progress, the red light on the top of the instrument will blink, indicating that the X-ray tube is energized and the shutter is open. In addition, a status bar will appear, tracking the progress of the measurement.

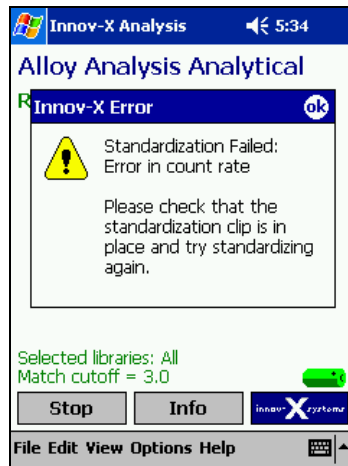
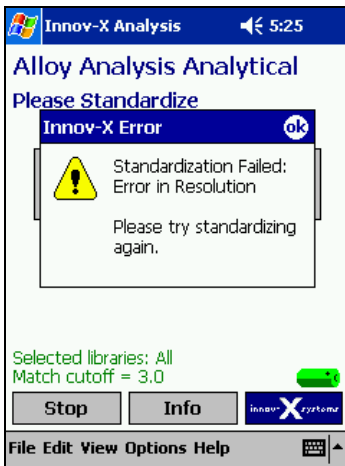


When standardization is complete, the message “Successful Standardization” will appear, along with the resolution of the instrument. Tap **ok** to acknowledge and clear the message. The instrument is ready for testing.

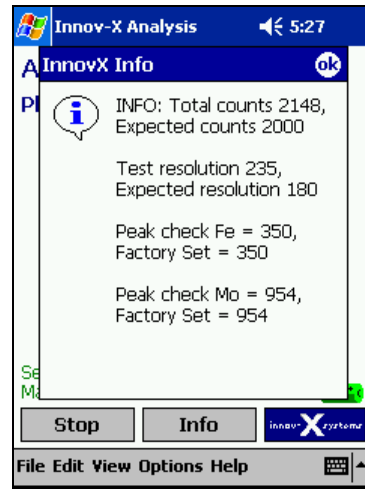
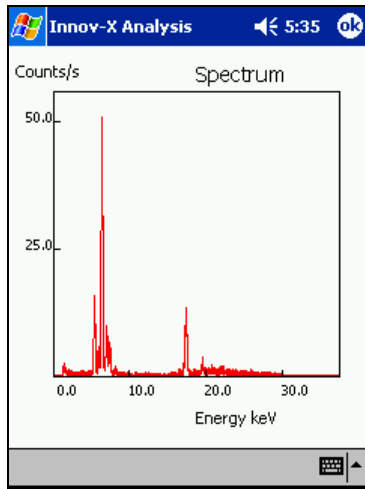


4.4.2 Standardization Errors

The analyzer performs several diagnostic checks during the standardization process. If the standardization fails, the instrument will prompt the user regarding the next step. Several errors could occur while standardizing: “Wrong Standardization Material,” “Error in Resolution” or “Error in Count Rate”

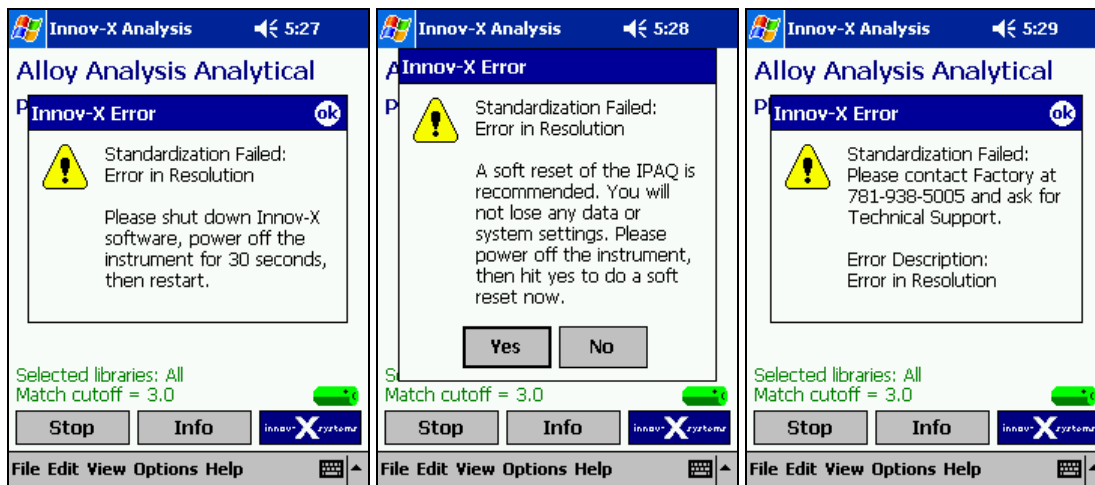


After closing the Standardization Failed message, two additional screens will appear. The first is a picture of the spectrum generated during the standardization. The second is a summary comparing factory set values for resolution, count rate, and peak positions to values calculated during the standardization.



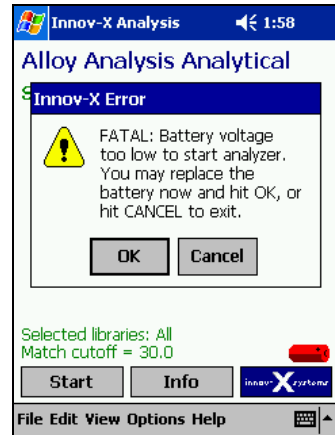
When standardization fails, verify that the standardization mask is in place, and attempt standardization again. To restandardize after a failure, tap the grey box in the center of the display, or choose **File**→**Standardize**. If you are using a weld collimator, make sure that the solid part of the mask is covering the window.

If standardization fails again, exit the analysis screen and power off the instrument. Restart and restandardize. If the standardization fails a 3rd time, you will be prompted to perform a soft reset of the iPAQ. Selecting Yes on this screen will automatically soft reset the IPAQ. You should also power cycle the instrument. Restart and restandardize. If the standardization fails again, replace the battery in the instrument and attempt another standardization. If this fails, please contact the Innov-X Systems service center at **781-938-5005**.



4.4.3 Battery Replacement and Initialization/Standardization

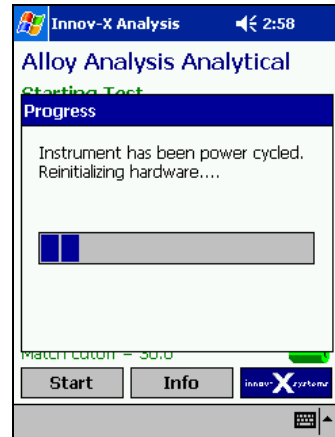
When the battery is too low to take a measurement, an error message will appear:



In order to continue testing, replace the battery immediately, and then tap “OK.” The analysis screen will remain open, and the instrument will reinitialize. This process will take 1 minute. It is not necessary to re-standardize, provided that less than 4 hours has elapsed since the last standardization and the battery swap is completed within 10 minutes.

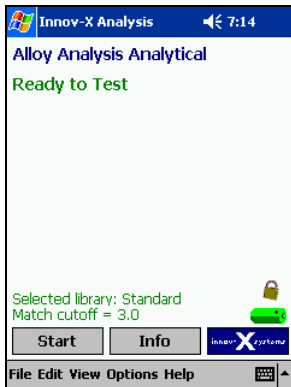
After re-initialization is completed, testing can continue.

If the battery is not replaced, and cancel is selected, the Analysis screen will close. When the software is restarted, the instrument will go through a complete 1 minute initialization and will require standardization.

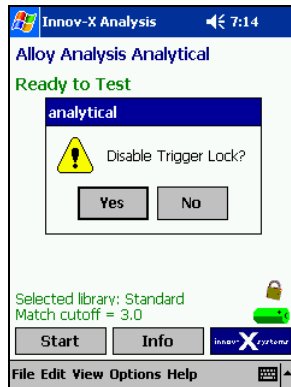


4.5 THE SOFTWARE TRIGGER LOCK

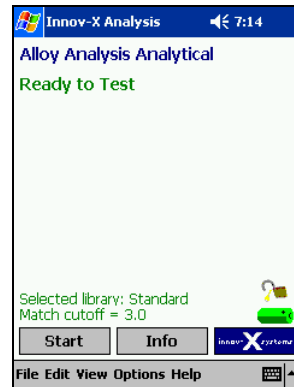
Innov-X analyzers are equipped with a software trigger lock which prevents the trigger from being actuated unintentionally. The lock is released by tapping an icon on the iPAQ screen. Once the lock is released, it will remain unlocked for subsequent tests, until more than five minutes has elapsed between tests. At that point, the trigger lock will be activated and will need to be disabled before additional testing can commence.



Tap the lock icon located directly above the battery indicator.



Select yes to disable the trigger lock



The open lock icon indicates when the trigger is disabled.

4.6 TEST INFORMATION - LABEL INPUT

Information such as sample name, and identifying characteristics can be stored with each measurement. This is done from the test information (Test Info) screen which can be accessed from the **Analysis Screen** of any mode by tapping the **Info** button, or selecting *Edit*→*Edit Test Information*.

The **Test Info** screen consists of eight fields. The name and format of each field can be changed by using the **Modify Test Info Template** feature described in section 4.6.1 **Modifying the Test Info Template**. The process of entering test information prior to each analysis is described in section 4.6.2 **Entering Test Information**. Finally, the process of entering or changing test information after the analysis has been completed is described in section 4.6.3 **Editing Test Info from the Results Screen**.

4.6.1 Modifying the Test Info Template

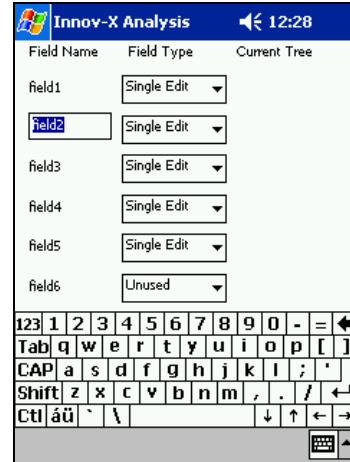
Test Info fields are modified via the Modify Test Info Template option found in the edit menu on the analysis screen in every software mode. Each field can be designated to be Direct Entry, Drop-down, or Tree. Direct entry fields allow users to enter characters directly from the virtual keyboard, or a bar code reader. Drop down menus provide a list of options to choose from. Trees are more complicated drop-downs; which allow users to subdivide large numbers of choices for ease in quickly locating the correct label. For example, a user may set up a tree with several parts for a main assembly. Subassemblies for the parts can be linked to their parent parts.

To make any changes to the Test Info format, select *Edit* → *Modify Test Info Template* from the analysis screen of any Mode. Modifications of Test Info screens are specific to each mode, and will need to be made to each mode if more than one is used.

Field Name	Field Type	Current Tree
Sample	Direct Edit	
field2	Direct Edit	
field3	Dropdown	
field4	Dropdown	
field5	Dropdown	
	Dropdown	
	Dropdown	
	Dropdown	

4.6.1a Changing Field Names

Field names can be edited by tapping on the current name. This will open an editable text box. A new name can be entered with the virtual keyboard. Selecting another cell or tapping **ok** will save this info.

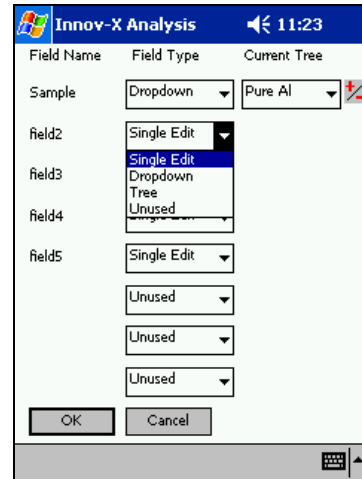


4.6.1b Selecting Field Type

From the Modify Test Info screen, the type of field can be selected from a drop-down menu. Simply tap the arrow in the Field Type box for the field being modified.

- Select **Direct Edit** for a text field which will accept data from the virtual keyboard, or a bar code scanner.
- Select **Drop-down** for a drop-down list
- Select **Tree** for a Drop-down menu with many choices, some of which may be grouped into categories and subcategories.

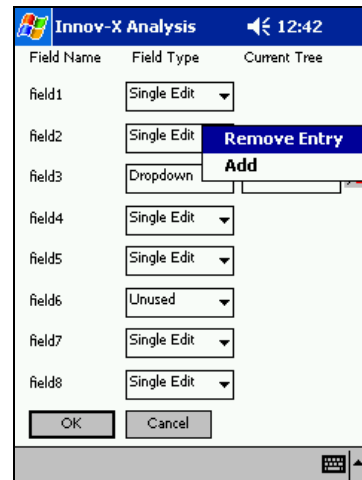
Select **Unused** to eliminate the field from the Test Info screen.



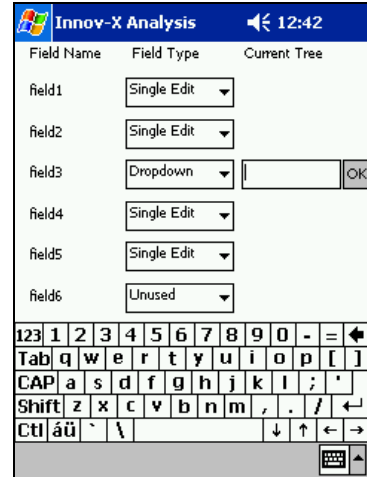
4.6.1c Changing Drop-down Menu Entries

Once a field has been designated a drop-down menu, entries can be added or deleted by clicking the +/- symbol to the right of the field. Two choices will appear; **Remove Entry** and **Add**.

To delete a drop-down entry, first select the label to be deleted, then press +/- and tap **Remove Entry**.

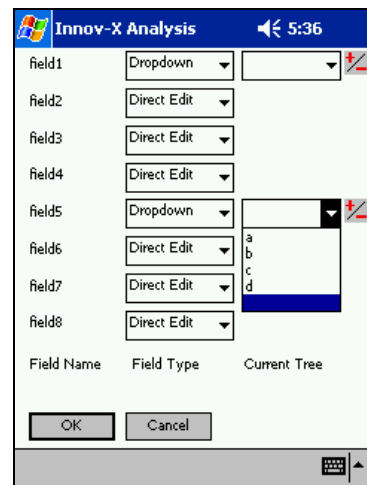


To add an entry from a drop-down list, tap the +/- symbol next to appropriate field, and select **Add**. Type the new info into the blank text box that appears. Select **OK** and the entry will be added to the drop-down menu.



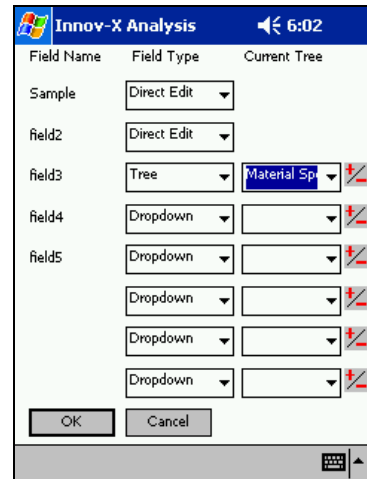
Repeat the process above to complete the complete drop-down list.

If it is anticipated that a drop-down field will not be used for all samples, enter an empty field as a choice so you can choose to leave the field blank.



4.6.1d Changing Tree lists.

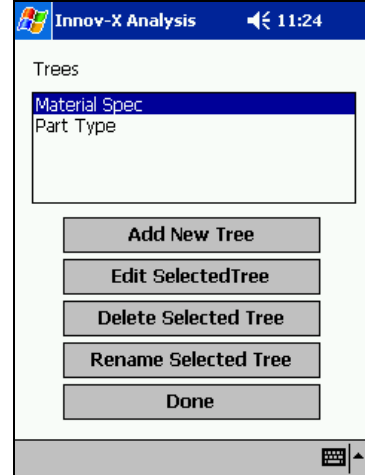
Once a field has been designated a tree, modifications to the contents of the tree can be made by tapping the +/- symbol to the right of the tree.



All modifications to trees are made from the menu shown on the right.

It is possible to add, edit, delete or rename trees. Select the appropriate choice from the menu to perform any of these functions.

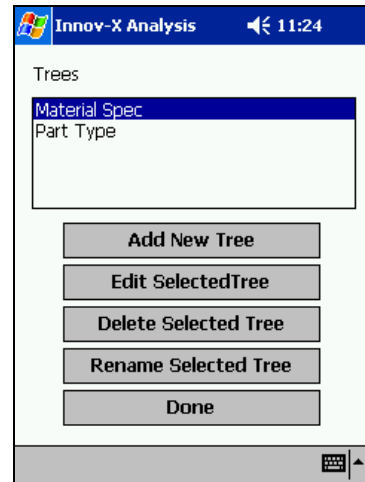
When you have finished creating/editing your tree, highlight it and select **Done**.



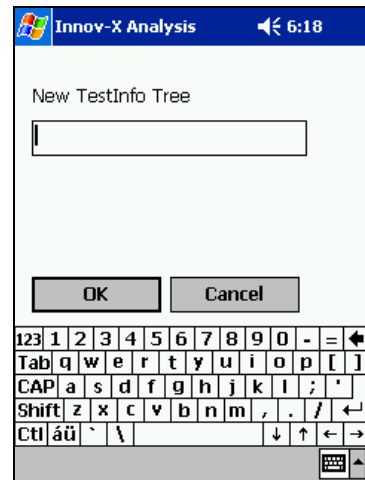
The following is an example of how a user might create a tree: A manufacturer of tubes and valves tests all parts to ensure that they're made of the proper material. The company's QC procedure involves labeling each test with the part number of the item. Rather than forcing operators to look through a long list of part numbers, a tree is created in order to subdivide the parts number into groups based on part type.

The procedure for creating the tree is as follows:

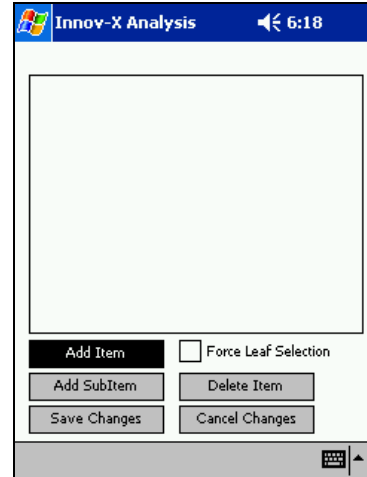
Select: Add New Tree:



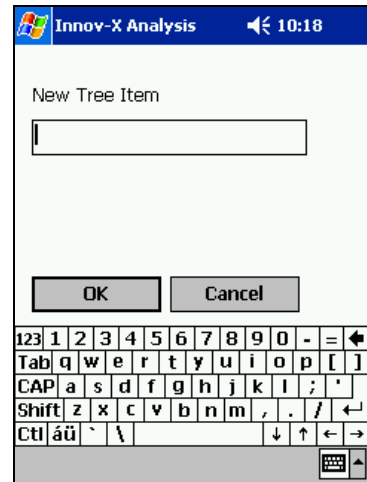
Enter the Name of the Tree in the text box and select OK.



Tap Add to add the first item

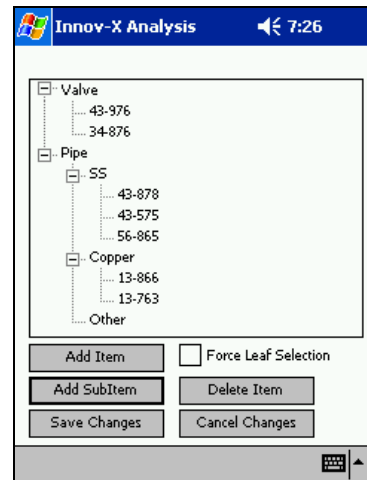


Enter the name of the item



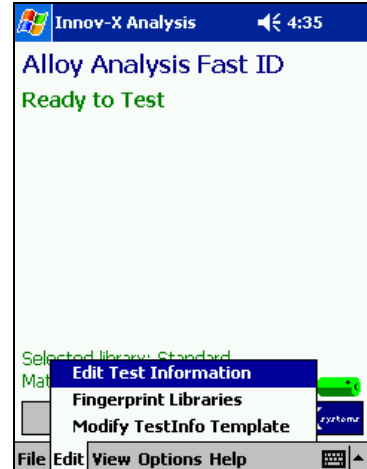
Once the tree is started, continue to Tap Add Item to add a top level menu item, or select an item and tap Add SubItem to link a subcategory to the item. Continue until all items have been added.

In this example, the part numbers for pipes and valves are separated into categories. The pipes are further subdivided by material type.

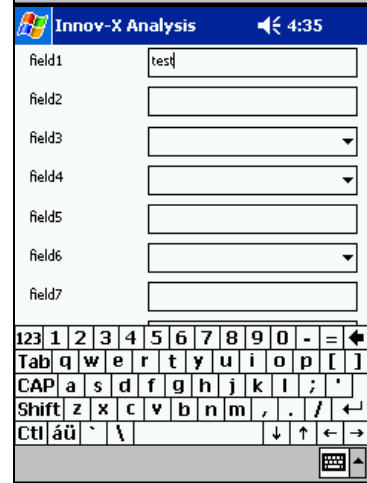


4.6.2 Entering Test Information

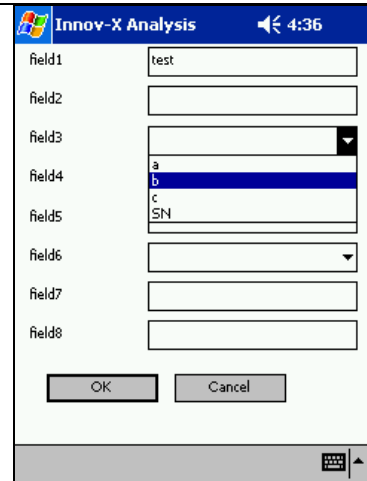
1. To enter the Test Info screen, you must be in the Analysis Screen. If the Results Screen is open, tap the ⊗ in the upper right hand corner to return to the Analysis Screen. From the Analysis Screen, select *Edit*→*Edit Test Information*, or tap the **Info** icon.



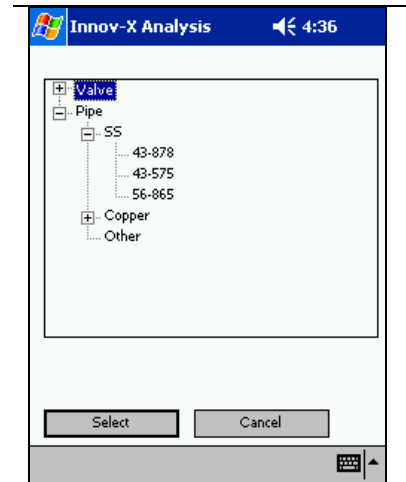
2. To enter a unique sample name or number, select a direct entry field by tapping anywhere within the field. Use the virtual keyboard to enter the information.



3. To select information from one of the drop-down menus, tap the arrow to the right of the box. Select the desired entry.



4. Some drop-down fields are formatted as trees. To select information from these fields, tap the arrow to the right of the box. A screen will appear showing options. The plus (+) symbol will appear before some choices indicating the presence of sub-items. Tap on the + symbol to expand the menu. Tap on any item or sub-item to select it, then press **Select**.



5. When all the necessary data have been entered, select **OK**
6. The information entered in the test info screen will be saved with each reading until the test info screen is modified again.

4.6.3 Editing Test Info from the results screen

Test information can be edited, or added to a test after its completion.

- From the results screen, scroll to the reading to be modified.
- Select **View** → **Test Info** to see in the information which is already stored.
- Select **Edit** → **Edit Test Info** to bring up the editing menu.



You will then be presented with the same test information screen described in **Section 4.5.2: Entering Test Information**.

4.7 EXPORTING AND ERASING DATA

Because the memory of the iPAQ is limited, you should periodically backup the data on your analyzer, and erase the memory. Depending on test volume, it is recommended that all data is erased on a weekly or monthly basis.

4.7.1 Installing ActiveSync

In order to copy files between the iPAQ and a desktop PC, Microsoft Active Sync Software must be installed on the desktop PC. Innov-X strongly recommends that you download the latest version of ActiveSync from the internet. ActiveSync v3.7 may be downloaded from <http://www.microsoft.com/windowsmobile/resources/downloads/pocketpc/activesync37.msp>

If it is not possible to download the latest version, an ActiveSync CD (v3.5) was shipped with your analyzer. Check behind the foam in the instrument case.

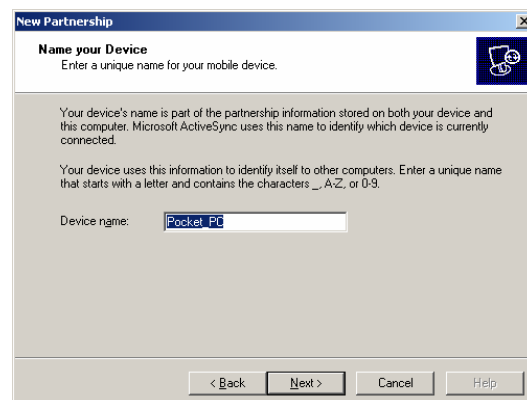
The iPAQ cradle should be hooked up to the USB port on the desktop computer before installing software.

The Procedure for installing and setting up ActiveSync is as follows:

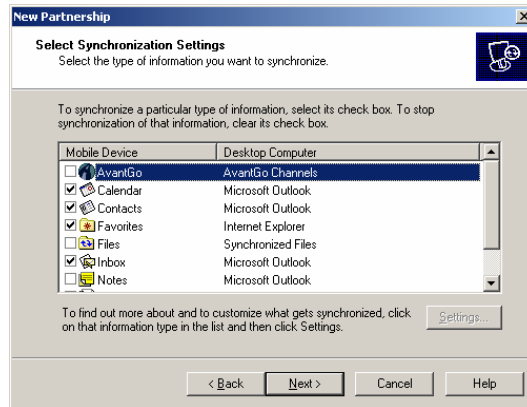
1. Insert the ActiveSync CD in your CD Drive. It will start automatically. The CD contains information about Getting Started with Your Pocket PC. This changes periodically, so it's difficult to describe exactly what the screens will look like. Step through the screens until you see the option "Install ActiveSync." Select this to start the installation process.
2. Follow the prompts on the screen. When given the choice, select "Run this program from its current location" and click OK.
3. Complete the install process. You will be required to restart your computer in order to complete the installation.
4. After restarting your computer, dock the iPAQ in the cradle. The iPAQ should automatically communicate with your computer. If it doesn't, check the connections and try removing the iPAQ and reseating it. If that doesn't work, try doing a soft reset on the iPAQ
5. When the computer communicates, you will be prompted to "Set Up a Partnership." Select "Yes, with this computer"



6. Enter a name for your iPAQ and click next.



7. You will be prompted to "Select Synchronization Settings." **Select "Files" only. It is important to make sure that Files is the only item checked. Otherwise, the files such as address books and emails will be copied from the desktop computer to the iPAQ.**



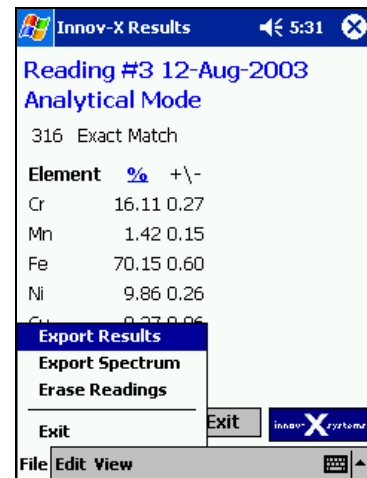
8. Step through the rest of the process.
9. A folder will automatically be created on the PC's desktop with the name of the device entered in step 8 above. Results files saved on the iPAQ will automatically be synched and will be stored in this folder. Opening this folder and clicking on the name of the file will open the file in Excel.
10. After ActiveSync is set up correctly, copying results to a desktop computer will consist of
 - a. Exporting results on the iPAQ. (described in section 4.6.2)
 - b. Synching the iPAQ to the computer
 - c. Opening the results in Excel for viewing, or printing.

4.7.2 Exporting Results

All data from your Innov-X Systems analyzer can be exported as a comma delimited text file (csv). This format allows the data to be easily exported to spreadsheet programs. It is possible to export all data from a single day, or to export all data saved in the iPAQ. Results and spectra are exported separately.

To export or erase data, you must be in the Results Screen. This is automatically opened when a reading is taken, or can be accessed by choosing *View*→*Results* from any analysis screen.

From the results screen, select *File*→*Export Results*



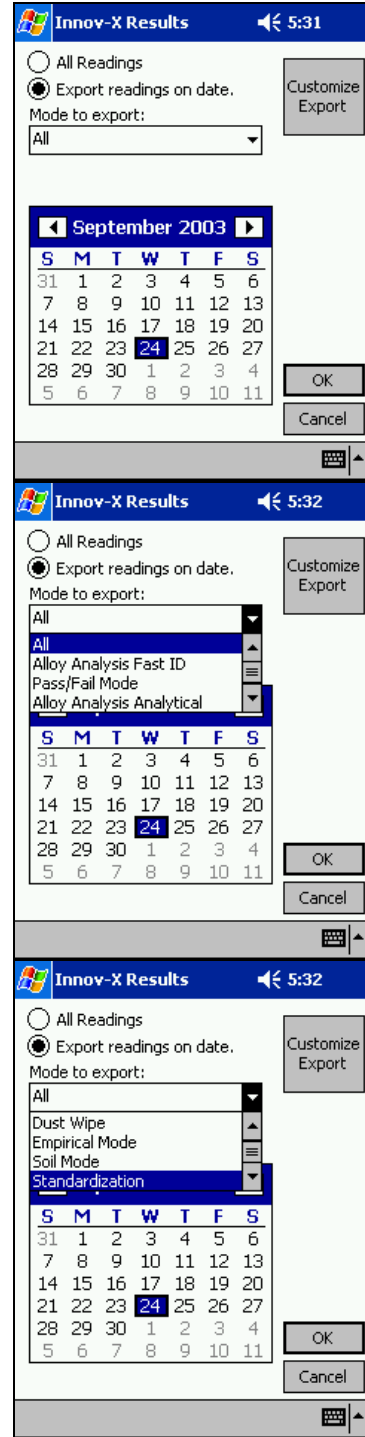
You can choose to export All Readings or just Readings on a specific date. Choosing **All Readings**: will export all readings saved in memory and is a good choice if you want to backup all data stored on the instrument before deleting. If a large number of readings stored, this option will take several minutes.

Choosing **Export Readings on date** requires that you pick a date from the calendar below. It is strongly recommended that you use this option and export data on a daily basis.

The customize export option allows users with administrative password privileges to customize the format in which data is exported. This is described in **Section 4.7.3: Customizing Results Export**.

After choosing which readings to export, you may choose to export all data, or just data from a specific mode. Selecting the arrow to the right of the mode to export will open a drop-down menu. Select the mode for which you want to export data.

All standardization data are stored as results files. These data are automatically included in exported results files when the selected “Mode to export” is **All**. Additionally, it is possible to export only the standardization data by selecting **Standardization** as the “Mode to export.”

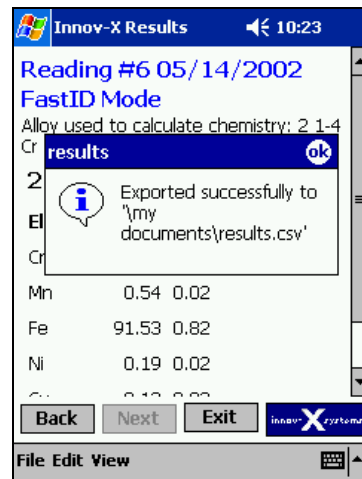


When the proper selections have been made, select **OK**. A **Save As** box will appear. Select the folder in which you want to save the data, and name the file. The file Type will always be **Comma Separated Values**. The recommended Location is Main memory and Folder is **None**. This will export files into the “My Documents” folder in the main Memory of the iPAQ.

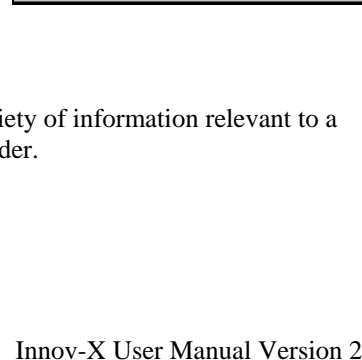
If you select a File Name which already exists, you will be asked if you want to replace the existing file. If you do, select **Yes**. Otherwise select **No** and choose another file name.



A status bar will indicate the progress of the export. It may take several minutes to export many readings. Daily downloading and weekly erasing of data simplifies and shortens this procedure.



When all readings are exported, a message will appear confirming the export. Tap **ok** to acknowledge and clear the reading.



4.7.3 Customizing Results Export

All units come with a standard results export format which reports a variety of information relevant to a test. Users can select which fields are exported as well as modify the order.

To modify exported results files, select **File** → **Export Readings** from the Results screen.

Tap the **Customize Export** box.

Enter the administrative level password when prompted.

Two columns appear on the screen; the column on the left lists fields which will NOT be exported, and the right-hand column lists fields which will be exported.

Fields can be moved from one column to another via the >> and << buttons located in the center of the screen

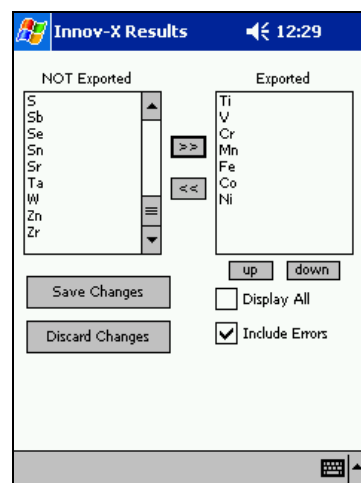
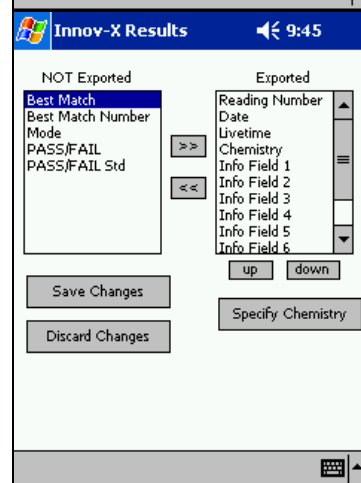
Exported field order can be changed by using the **Up/Down** buttons. Select a field and move it up or down as desired

Once all changes have been made, choose **Specify Chemistry** if changes need to be made to the list of exported elements.

In chemistry is not edited, select **Save Changes** to keep the modified settings, or **Discard Changes** to ignore any changes.

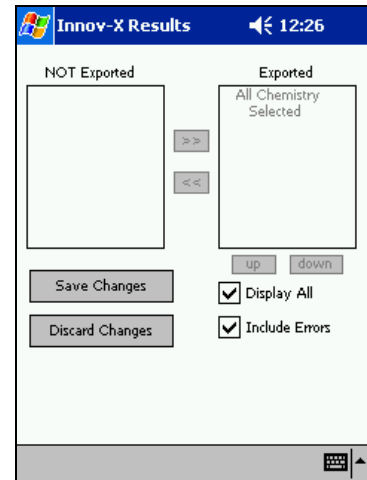
The Specify Chemistry screen resembles the previous screen. Move elements to the appropriate column, depending on whether or not an element should appear in exported files.

Select **Include Errors** to export the error associated with each measurement.



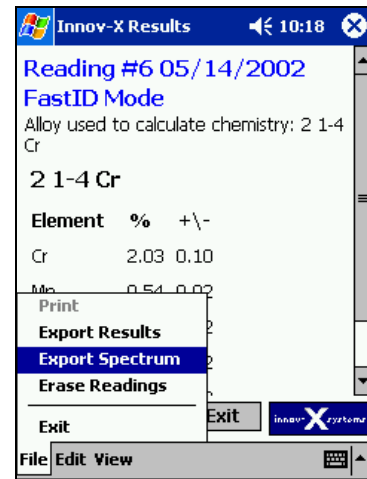
Select **Display All** to include all measured elements. *This setting is recommended, as it will ensure that all data measured with the instrument is exported.*

When all changes have been made, tap **Save Changes** or **Discard changes**, depending on whether the changes should be saved.

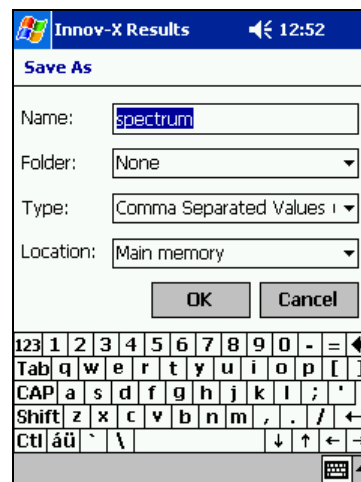


4.7.4 Exporting spectra

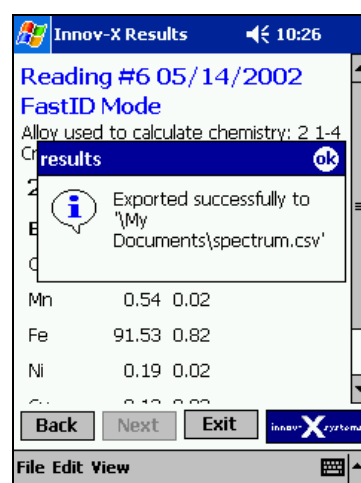
Only one spectrum may be exported at a time. In the results screen, scroll to the reading for which you wish to export the spectrum, and **Select File→Export Spectrum**.



Choose the File name, and make sure that **Comma Separated Values** and **Main Memory** are selected. This will save the spectrum to the My Documents folder in the Main Memory of the iPAQ.



A message will appear indicating a successful export. Tap **ok** to acknowledge and clear the window.



4.7.5 Erasing readings

It is possible to erase a single reading, a range of readings, all readings from a specific data, or all readings before a specific date.

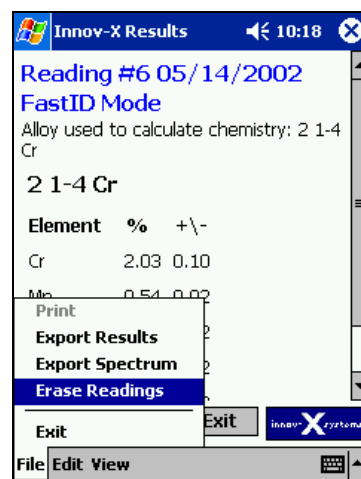
In order to erase a single reading, the reading to be erased must be displayed on the screen before selecting delete. If necessary scroll to the reading you wish to delete.

In order to select a range of readings, you must have a reading open from the date you wish to delete the readings. If a reading from the desired date is not open, you may select **View**→**Go to date**, and select the appropriate date.

The reading displayed in the results screen is not relevant if you want to delete all readings from a specific date, or all readings before a specific date .

From the results screen, select **File**→**Erase Readings**.

A message box will appear prompting you to enter your password. Enter your administrative level password and select **OK**.

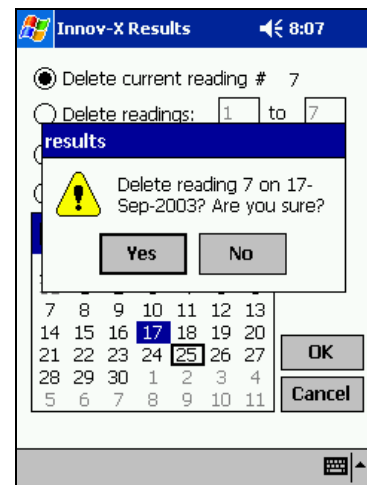


A dialogue box will appear allowing a choice of which results to delete. Select the appropriate choice:

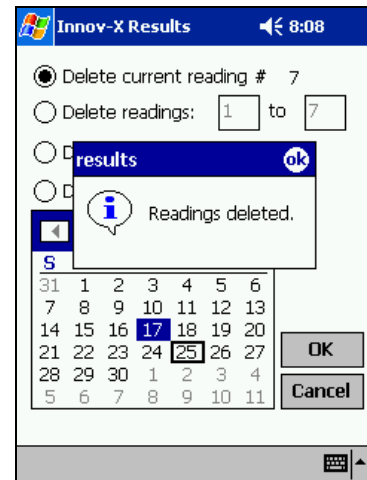
- Selecting **Delete current reading** will delete the reading that is currently open.
- Choosing **Delete readings XX to XX** will delete a range of readings from the date of the reading that is currently open.
- **Delete all readings on date** deletes all readings from a specific day.
- **Delete readings before date** deletes all readings taken prior to a specific day.

If you select **Delete all readings on date** or **Delete readings before date**, you must specify a date from the calendar. The default date is the current date.

When you've selected the readings to delete, Click **OK**. You will be asked if you're sure you want to proceed. If you want to proceed with the data erase, select **Yes**. Otherwise, click **No**.



A message will indicate the readings were successfully deleted. Tap **ok** to acknowledge and clear the message window.



Chapter 5 ALLOY ANALYSIS

5.0 ALLOY ANALYSIS INTRODUCTION

Three different modes exist for the analysis of alloys:

- ❑ **FastID Mode**
- ❑ **Pass/Fail Mode**
- ❑ **Analytical Mode**

Systems may be purchased with any combination of the three modes. Instruments can be upgraded for a fee at any point after purchase. General introductions to each of the modes, as well as basic operations are found in this chapter. Subsequent chapters describe each of the modes in greater detail.

FastID MODE

FastID mode is designed to quickly identify an alloy by matching the spectral signature of an unknown sample to the saved spectral signatures of reference standards in the FastID library. This mode can provide alloy chemistry if concentration data are entered for the standards. Chemistry results are a linear extrapolation from standard intensity data. FastID Mode is suited for determining accurate chemistry for alloys for which standards are available AND are loaded into the library. A standard library, as well as 3 user libraries can be used for matching. All libraries can be edited.

PASS/FAIL MODE

Pass/Fail mode is used to quickly test alloys to ensure that they meet quality control criteria. The operator chooses a stored spectral fingerprint which the system uses as a reference standard. Samples are compared to the reference, and a Pass or Fail result is displayed. Pass/Fail decision criteria can be spectral signature matching or concentration ranges for one or more elements. Pass/Fail mode uses the same fingerprint library as FastID mode.

ANALYTICAL MODE

Analytical Mode provides a full analysis of alloy chemistry using the method of fundamental parameters, as well as a grade match based on minimum and maximum grade specifications. This method uses a factory calibration, and requires no additional user supplied standards. In addition using the comprehensive grade library included with the analyzer, users may enter additional grade table specifications.

5.1 ALLOY ANALYSIS – STARTING THE INSTRUMENT AND TAKING A MEASUREMENT USING THE STANDARD LIBRARY

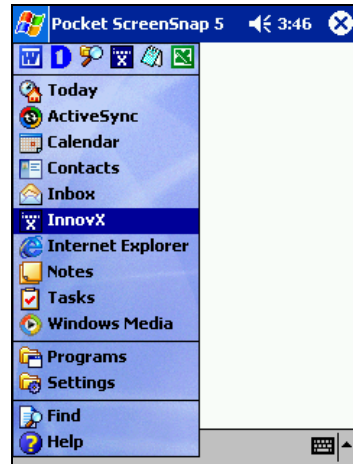
The basic startup and testing procedure is described below. Most screen shots were taken using **FastID Mode**; however, the basic procedure is the same for all three alloy modes.

BASIC OPERATION

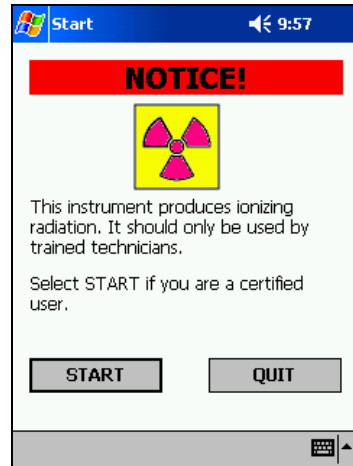
All Innov-X Systems Analyzers are shipped with a standard set of reference alloy standards that makes it possible to identify approximately 200 common alloys (35 in FastID). A list of the references in the library is provided in Appendix III. When you first receive your analyzer, it is recommended that you start by

analyzing the 316 standardization piece included with your analyzer to gain an understanding of how the analyzer works.

1. Install a freshly charged battery in the instrument.
2. Turn on the analyzer by pressing the power switch located at the back of the analyzer.
3. Verify that the iPAQ is correctly seated on the top of the unit. If the iPAQ is properly connected, the amber light on the upper right side of the iPAQ next to the power button will blink, indicating that the iPAQ is receiving charge from the analyzer.
4. If the iPAQ is not on, turn it on by pressing the power button on the upper right side of the iPAQ.
5. Start the Innov-X Systems Software by selecting the Start Menu from the upper left hand corner of the iPAQ screen. Select the Innov-X Systems Software from the drop down menu.



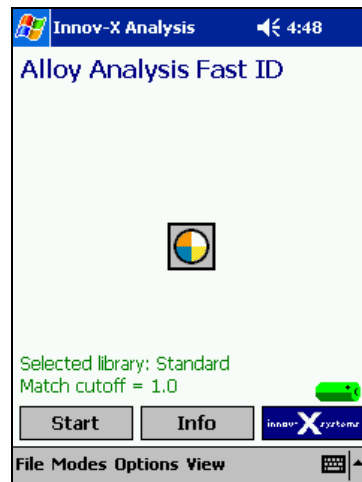
6. A notice will appear reminding the user that this instrument produces ionizing radiation and requires a trained user. . Select **START** to start the Innov-X Systems Software package. Selecting **QUIT** will exit the Innov-X Software.



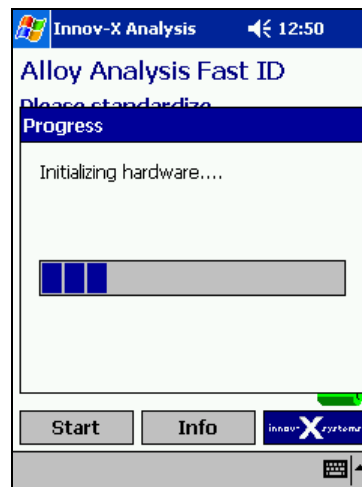
7. The Main Menu will open. Tap the name of the Mode you will be using to open it. First time users should Analytical Mode.



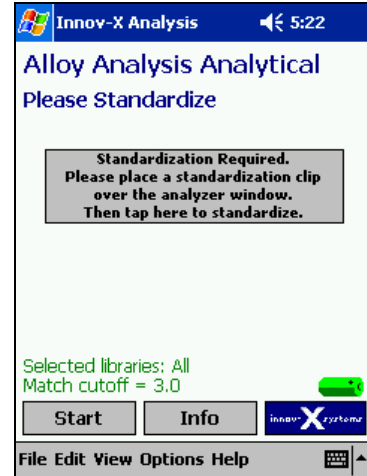
8. There may be a brief pause while the instrument loads the various parameters needed for operation. While this occurs, an icon will appear in the center of the iPAQ screen.



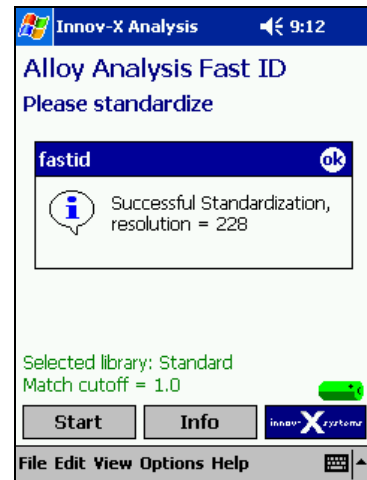
9. Once the analysis mode has been selected, the instrument will go through a 1 minute hardware initiation during which the electronics will stabilize and the detector cooling will be initialized.



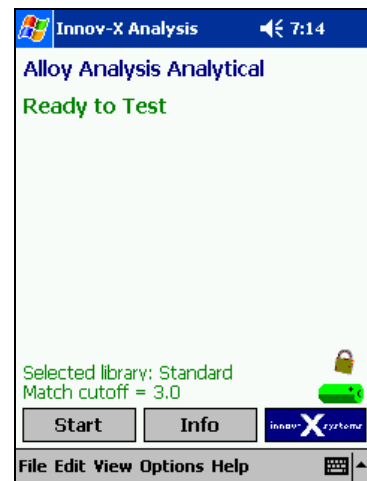
10. The message “**Standardization Required. Please place a standardization clip over the analyzer window. Then tap here to standardize.**” will appear. Standardization is required before testing can begin. Place the standardization clip in front of the analyzer window. Tap the message box. Standardization will take approximately 1 minute; a status bar will be displayed throughout the measurement. Standardization is described in more detail in **Section 4.4: Standardization.**



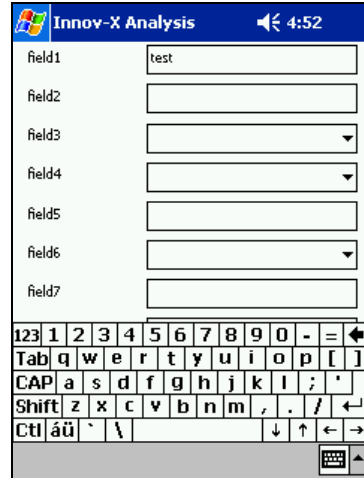
11. When standardization is complete, the resolution of the analyzer will be displayed. Tap **ok** to acknowledge and clear this screen.



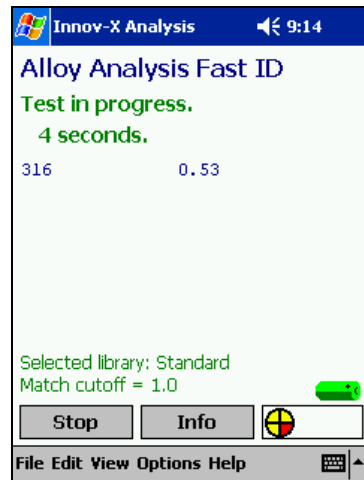
12. The analyzer is now ready to take a measurement. The Trigger lock must be unlocked before pulling the trigger will start a test.



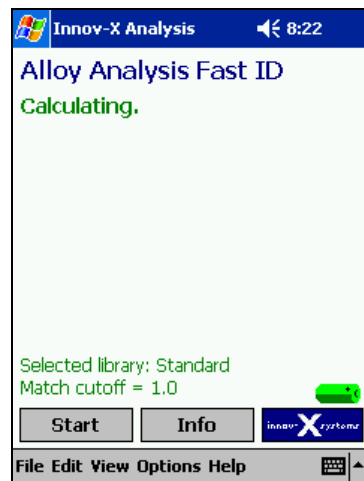
13. If you wish to enter a sample name or sample identifying characteristics, select **Edit**→**Test Info**. Enter information in text fields, or select items from drop down menus. Select **ok** to close the Test Info window. The format of this screen may vary depending on user settings. See **Section 4.6: Test Information** for more information.



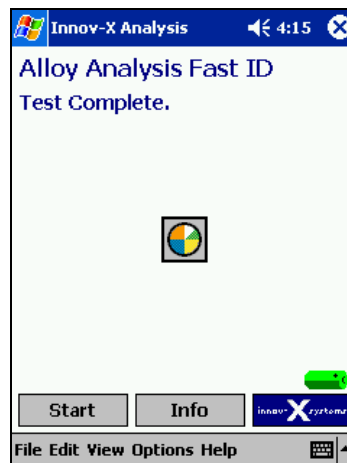
14. Hold the analyzer to the sample to be analyzed. Make sure the sample is as flush against the analyzing window as is possible. You may start an analysis by pulling and holding the trigger. Releasing the trigger will abort the test.
- After an analysis is started, the message “**Test in Progress.**” will appear, followed by the number of seconds elapsed during the measurement. For the duration of the test, the red light on top of the instrument will blink, and the “testing” icon will appear in the lower right corner of the IPAQ.



- When the measurement is complete, the analysis screen will display the word **Calculating**. There may be a slight delay while the instrument calculates the results. This will be indicated by the appearance of a “calculating” icon in the lower right hand corner of the IPAQ screen. Because the FastID calculation is very rapid, this icon is rarely seen, however there may be a few second calculation for Analytical Mode.

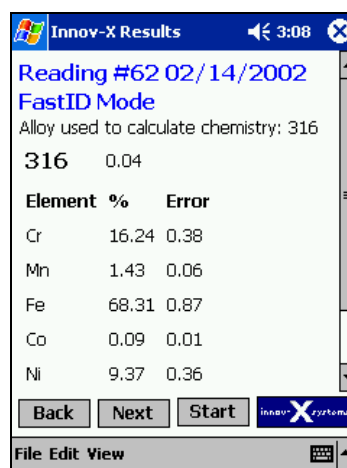


- c. When the calculations are complete, there will be a slight delay the first time the results screen is opened. An icon will appear in the center of the screen during this delay. This indicates that the results program is loading and re-indexing all saved results.



15. The Results screen will display the results. The information displayed on the screen may be changed by selecting one of the options under the View menu. This is described later in this chapter under the Results section. If you analyzed the standardization piece, the grade identification should be listed as 316.

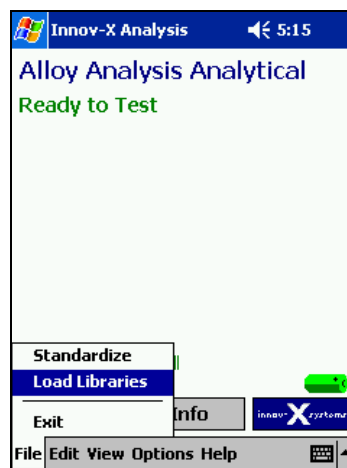
16. Once the results screen is open, subsequent readings may be started by depressing the trigger. If at any point, you wish to return to the analysis screen, select **File**→**Exit** or tap the X in the upper right hand corner of the screen.



5.2 SELECTING A LIBRARY

The Innov-X Software can search any one of four libraries when in **FastID** or **Analytical Modes**. **ALWAYS VERIFY THAT THE CORRECT LIBRARY IS BEING SEARCHED.** More detailed information on library functions can be found in Chapter 5.

To select which library to search, go to the **FastID** Analysis Screen or the **Analytical** Analysis Screen – whichever mode is in use – then select **File**→**Load Libraries**.



A menu appears. The first line will read **Use Grade Libraries** for Analytical Mode and **Use Fingerprints** for FastID Mode

Choose the Fingerprint or Grade Libraries you wish to search. For the most comprehensive search, select **All** libraries. This will search the entire Standard Library, as well as any fingerprints or grades that have been added by the user.

Users who are primarily concerned with sorting the most common specialty, stainless, nickel and high temperature alloys should always search the **Standard Library**. This will ensure that the factory-installed library will be searched. The Standard Library can be searched by itself, or in combination with any of the other libraries.

Users who are sorting a small group of alloys may prefer to create their own libraries using their own standards. In this case, only the appropriate user library should be selected.

When loading libraries from the **File→Load Libraries** Menu, the number of fingerprints or grades in the selected libraries is displayed at the bottom of the screen. For best results especially when measuring complete unknowns, a large number of fingerprints should be selected because the analyzer cannot identify an alloy that is not in a library.

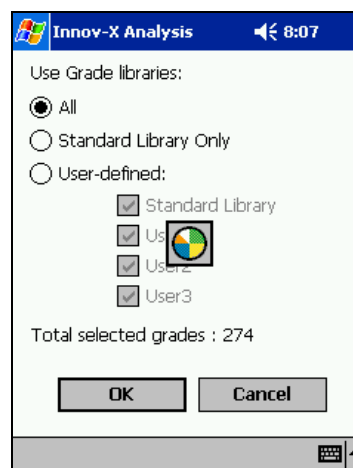
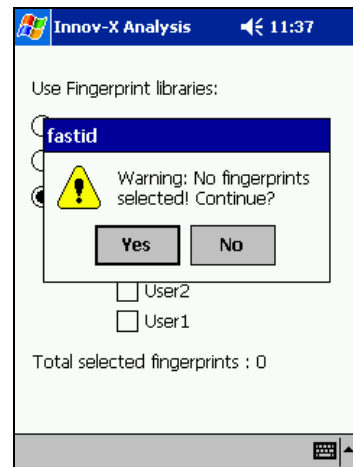
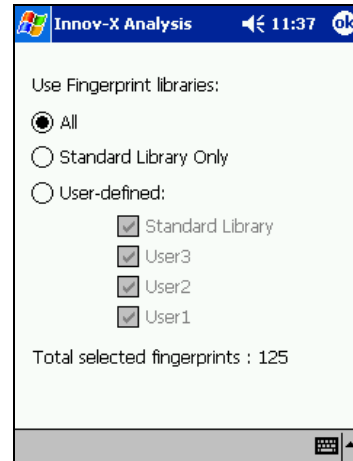
Since there is a good chance that searching a small number of libraries will result in No Matches, a warning message will appear if you select 10 or fewer grades or fingerprints.

Select **No** to return back to the Use Fingerprint Libraries screen to make another selection.

Select **Yes** to continue with the selected library. Keep in mind that with fewer fingerprints or grades being searched, you will likely get a larger number of No Matches.

If no fingerprints are selected, it will not be possible to get any valid results in FastID. If a user continues with no fingerprints selected, it will be necessary to teach fingerprints in the selected library before proceeding with the analysis. In Analytical Mode, chemistry will be calculated, but no grade matches will be displayed if no grades are loaded.

There will often be a pause of several seconds while the instrument loads the new libraries. A revolving icon will appear in the center of the screen indicating that the libraries are loading.



5.3 SETTING THE ANALYSIS TIME

The software allows the user to set up minimum and maximum testing times.

The minimum testing time must elapse in order for results to be calculated. If a test is stopped before the minimum testing time, it will be treated as an aborted test, and no results will be calculated. Additionally, if the Live Update feature described in section 5.4 is active, results will not be displayed on the screen until after the minimum time period.

The test will end automatically when the maximum testing time is reached. A test can be ended manually at any time by releasing the trigger.

To change Testing times:

From any analysis screen, select **Options**→**Set Testing Times**

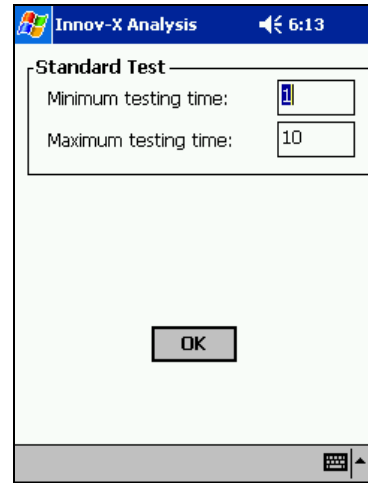
Typical settings for standard test times, in seconds are:

Minimum testing time: 3

Maximum testing time: 10.

However, these values may be changed depending on the application and the desired results.

Alloy system equipped with Analytical **Smartbeam** will have an option for setting the test time in this screen as well. **Smartbeam** is discussed in chapter 8.



The minimum testing time is required to be no less than 1 second. An error message will appear if the time is set to be less than 1 second. Clear this message by selecting **ok**. The time will default to a minimum time of 1 second. This value may be used, or another value may be entered. Select **OK** to close the window.

You will not be allowed to exit the **Set Testing Times** window unless a valid minimum testing time has been entered.

Recommended Testing Times:

For most alloys, the recommended testing time is 5-10 seconds to obtain a unique grade ID and good alloy chemistry. For some alloys that only differ by small amounts of one or two elements, it may be necessary to perform longer tests. Examples include Low alloy steels 4140 and 4340. Alloys which differ by less than 1% of Ti or V require the optional **Smartbeam** feature for quick separation.

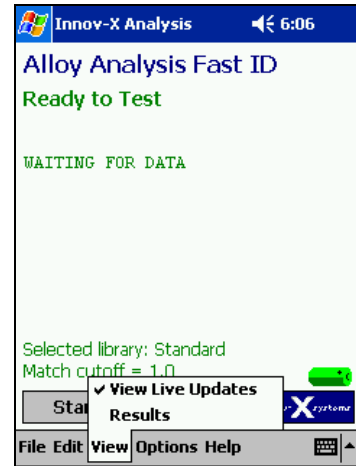
The maximum testing time will determine the length of a test. The analysis will automatically stop if the maximum testing time is reached. Normal maximum testing times will range from 5 to 20 seconds. Fundamentally, if the goal of the analysis is primarily grade identification, shorter analysis times are used. If greater precision is required in the calculation of chemistry or if an alloy separation is particularly difficult, longer test times may be used.

It should be noted that the pre-set time refers to the length of time the analyzer is actively collecting data from the detector. The total analysis time can be slightly longer than the maximum test time due to the small amount of time required to process the data and calculate the results.

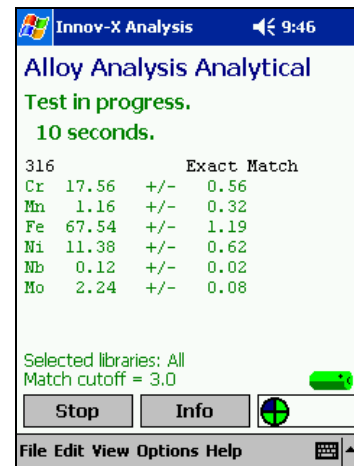
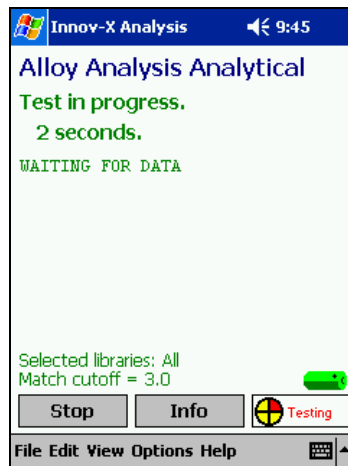
5.4 LIVE UPDATE DISPLAY

In addition to viewing completed tests in the results screen, the analyzer will display screen updates as the results are calculated during a test. This allows users to decide when to stop tests based on the precision of the reading.

To switch Live Updates on or off, select **View**→**Live Updates**. A check mark will indicate if Live Updates are turned on.



When the Live Updates feature is on, and a test is in progress, the screen will show the message “WAITING FOR DATA” until the minimum testing time set in **Options** → **Set Testing Times** has elapsed. After that, chemistry and the errors on the chemistry will be displayed. This feature allows the user to stop the test as soon as the desired precision is reached.



If too short a maximum test time is set, the test may end before the desired precision is reached. As a result, some users who prefer to end tests based on the screen display may choose to set long maximum test times (60 seconds or so) and manually end all tests.

5.5 SAMPLE CONSIDERATIONS

5.5.1 Coated or Painted Samples

Innov-X Alloy Analyzers are capable of analyzing a wide variety of sample shapes and types. However, it is important to understand that XRF is fundamentally a surface analysis technique. X-rays penetrate a very short distance into most alloy samples. Therefore, the analyzer will detect what is on the surface of an alloy, rather than what comprises the bulk of the material. If a material has been coated, plated, painted, or has had some sort of surface treatment, such as heat treating, it may be misidentified. For example, a steel piece that has been painted grey will show high concentrations of titanium from the paint, and may be misidentified as a titanium alloy. In addition, large amounts of metal dust or turnings on a surface may be detected by the analyzer.

To ensure proper identification of coated materials, an area slightly larger than the analyzing window should be ground to remove the coating. This will allow the analyzer to measure the alloy rather than the coating. It is not necessary to completely clean and polish all materials, however, obvious metal dust should be removed.

5.5.2 Mixed samples, Heterogeneous materials

Often finished metal pieces may consist of more than one type of metal. In addition, some users may wish to measure mixed turnings, or an assortment of small pieces. In these cases, the user should remember that the analyzer will measure the entire area covered by the analyzing window and report an average chemistry. For turnings, this is useful, as the analyzer will provide an average composition. However, if two or more pieces of metal cover the window, the results will also be just an average reading, and may tell very little about the composition of one piece or the other. When shooting metal pieces, or welds, it is important to make sure that only the metal of interest is covering the analyzing window. It may be possible to use a welding mask to narrow in on the area of interest.

Keep in mind, that a welding mask should only be used in Analytical mode, unless fingerprints have been taught in FastID using the mask.

5.5.3 Small and irregularly shaped samples

All Innov-X Systems alloy software modes are able to measure parts that are smaller than the analyzing window; however, it is usually necessary to increase the testing time. The precision on measurements of small parts is reduced; since the signal from smaller samples is less than it is for samples that completely cover the window. It is also a good idea to try to maximize the material in contact with the window. If possible, analyze the largest flattest side of an irregularly shaped object.

5.5.4 Invisible elements

Since the Innov-X Systems Alloy Analyzer cannot directly analyze light elements such as carbon, aluminum and silicon, samples containing large amounts of these elements may not read correctly in Analytical Mode, depending on certain instrument settings. These settings are described in **Section 8.3.3: Light Element Analysis**.

Please read this section and familiarize yourself with the issues pertaining to Light Element analysis before attempting to analyze Aluminum alloys or other alloys containing significant amounts of non-detectable elements.

Chapter 10S Soil Analysis

The Innov-X analyzer can be used to analyze in situ (directly on the ground), bagged or prepared soil samples. A guide to Soil analysis using field portable X-ray fluorescence is found in the appendix. This document summarizes EPA Method 6200 which is the standard protocol for field screening. It also provides information on prepared sample testing.

10.0 CHECK STANDARDS

It is recommended that a check standard is measured after each standardization, and periodically throughout the day. Innov-X provides several NIST certified standards for verification. The certified values for these samples are provided in the appendix. At least one standard should be measured for a minimum of 1 minute. Elemental concentrations for elements of interest plus or minus the error on the reading should be within 20% of the standard value. The Field screening guide in the appendix describes in more detail recommended quality assurance considerations.

The standards provided with the XRF analyzer are contained in XRF sample cups with a Mylar window (through which the soil can be viewed) on one side, and a solid cap on the other side. Samples should be measured in the sample cup, through the Mylar window. The best way to measure a prepared sample is using the test stand. If this is not available, the sample may be placed on the ground, and the analyzer may be pointed downwards in full contact with the soil cup. Do not hold the soil cup in your hand while measuring.

10.1 SAMPLE PRESENTATION

In situ testing:

In situ testing is performed by pointing the analyzer at the ground. Any grass or large rocks should be cleared away and the analyzer should be held such that the front of the probe head is held flush to the ground.

Since dirt can accumulate on the analyzer window, it is recommended that the window is wiped clean after each analysis. The window should also be checked to ensure it is not ripped or punctured. Instructions for replacing the window are found in the appendix.

Bagged or prepared sample testing:

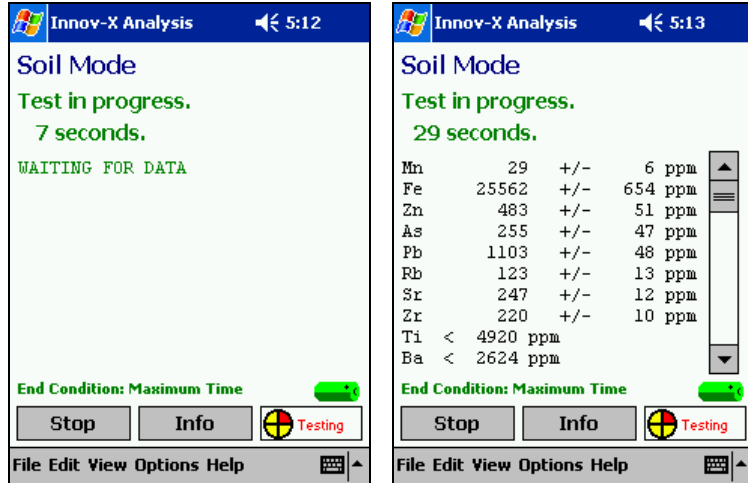
It is strongly recommended that all prepared samples be analyzed in the testing stand. Samples should be placed on top of the testing stand, completely covering the window. **Never hold prepared or bagged samples while testing**, as this could expose the operator to the x-ray beam.

Avoid measuring very thin samples, as this can affect results. Prepare samples cups to contain at least 0.5 inches of packed samples. When analyzing bagged samples, make sure that sufficient sample exists in the bag to completely cover the window with a sample thickness of a minimum of 0.5 inches.

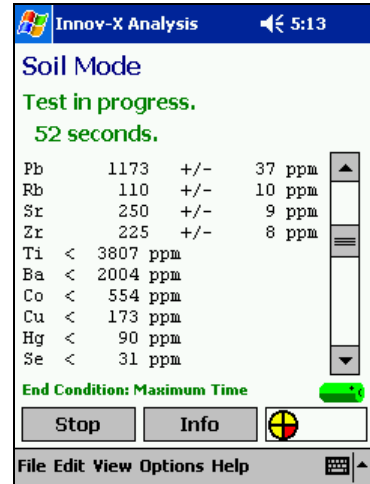
10.2 TESTING IN SOIL MODE

After the instrument has been standardized, testing can begin. Simply pull the trigger or press **Start** on the iPAQ screen to begin the test. The red warning light on the top of the instrument will blink, indicating X-rays are being emitted. The screen will display the words “Test in progress” and the time elapsed. The word “Testing” will blink on and off in the low right hand corner of the screen.

After a minimum time has elapsed, intermediate results will be displayed on the screen. Until this minimum time has elapsed, the words “WAITING FOR DATA” will appear instead. This minimum time can be set by the user by selecting *Options*→*Set Testing Times*, which is described in **Section 10.4: Soil Mode Options**. Each line of the results display shows the name of an element, its calculated concentration and the error on the measurement. This error is the 1 sigma error on the counting statistics of the measurement. The error will decrease with increased testing time.



Too many elements are measured in soil mode to display them at one time. However, it is possible to use the scroll bar located to the right of the chemistry display to view other elements. The complete display shows detected elements first, listed in order of emission line energy, from lowest to highest. Following the detected elements are the elements which are below the detection limit of the instrument. These elements are shown as less than a calculated LOD. This LOD is defined as three times the error on the counting statistics of the measurement.

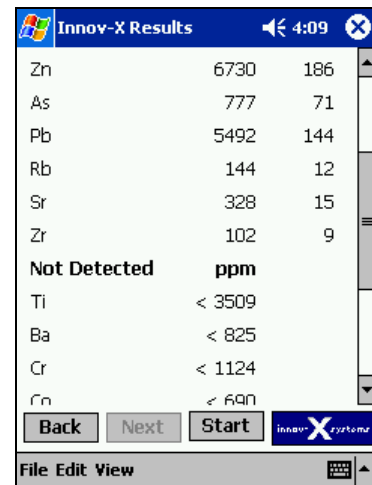
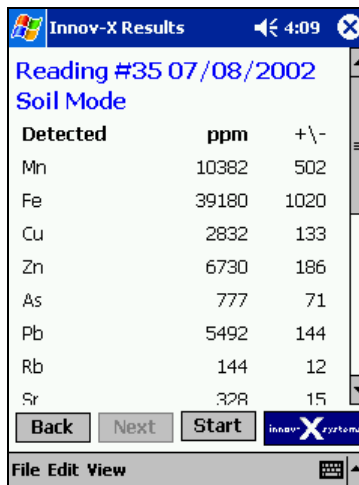


When the measurement is complete the results screen will open, displaying the final results of the measurement.

10.3 SOIL RESULTS SCREEN

10.3.1 Results View Menu

The standard Soil Mode results screen displays the concentration (in ppm) and error in measurement for detected elements, followed by the list of non-detected elements with the calculated limit of detection for each element for that test. If the display does not show soil chemistry results, change the display by selecting *View*→*Results*.

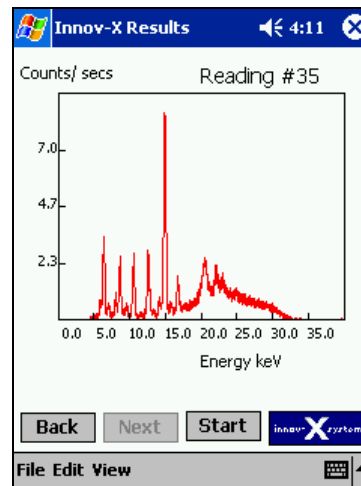


The standard soil chemistry display can be modified by using the View Menu. As with all Innov-X analytical modes, it is possible to view spectra and Test Information.

10.3.2 Spectrum Screen

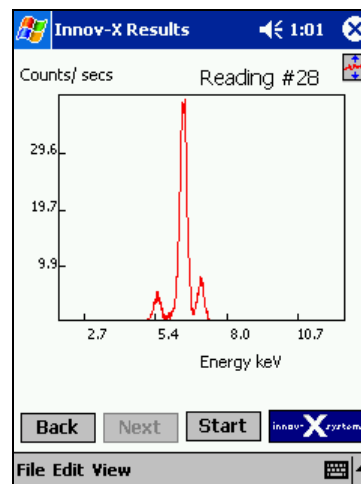
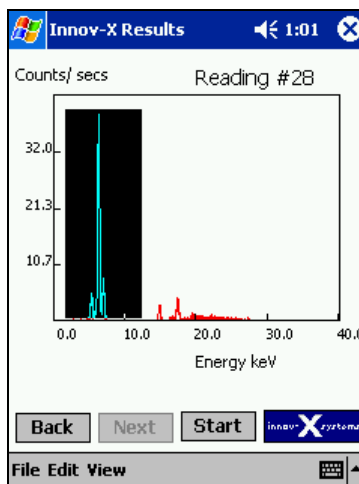
This screen displays a plot of the x-ray fluorescence spectrum for an individual test, plotting the intensity on the y-axis versus the energy of the fluorescence x-rays on the x-axis.

Tapping on the spectra will show the energy scale and counts rate at the selected point



It is possible to zoom in on certain areas of the graph by selecting one corner and drawing out the region

Tapping the symbol in the upper right hand corner beneath the X will restore the graph to full scale.



10.3.3 Test Info Screen

The test information screen shows any test information that was entered prior to the start of the test. Changes to that test information can be made by selecting **Edit**→**Test Information**.

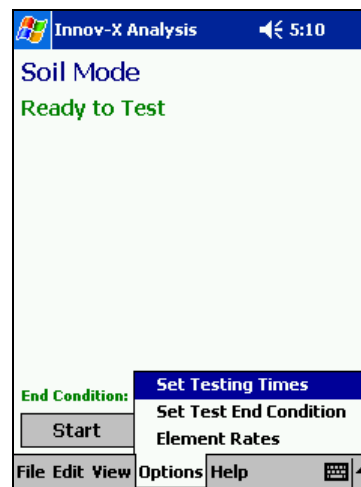
10.4 SOIL MODE OPTIONS

The length of tests in Soil Mode is user settable. Users may select a minimum testing time, and as well as choose from a variety of test end conditions.

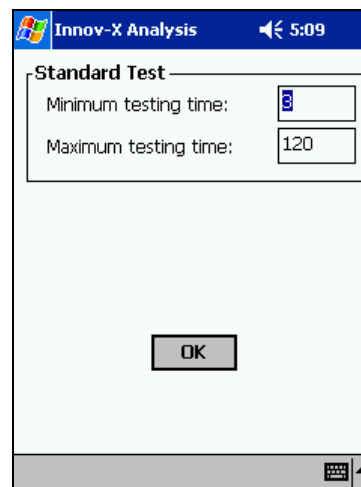
The options related to test time are contained in two menus: **Options**→**Set Testing Times**, and **Options**→**Set Test End Condition**. **Set Testing Times** contains minimum and maximum testing time information, while **Set Test End Condition** allows the user to select test end conditions.

10.4.1 Set Testing Times

To set the minimum and maximum test lengths, select **Options**→**Set Testing Times**



A screen appears prompting you to enter a Minimum and Maximum Testing times. Instruments equipped with the optional LEAP package will be able to set Light Element Testing times in this screen, as well.



The minimum testing time is the required time that must elapse before results can be calculated. Live Update results will not be displayed on the screen until the minimum has elapsed, likewise a test must complete the minimum time before any test end condition can be used. If a test is stopped before the minimum testing time has elapsed, the test will be aborted, and no results will be calculated.

Maximum testing time is relevant only if “Maximum Testing Time” is selected from **Set Test End Condition**. This will automatically end the test at a preset testing time. Typically, the maximum testing time will be in excess of 30 seconds, and may be 1 or 2 minutes, depending on detection limits and desired precision.

It should be noted, that all testing times in this section refer to “Real Time,” the time the measurement takes when timed on a normal clock. The time stored with each analytical result (accessible by selecting **View**→**Test Information** from the Results screen), refers to the test’s “Live Time”. This is the amount of time that the analyzer hardware was collecting spectra. Since there is some detector dead time associated with a measurement, the live time of a test will be slightly shorter than the preset “Real time”.

10.4.2 Soil Mode Test End Condition

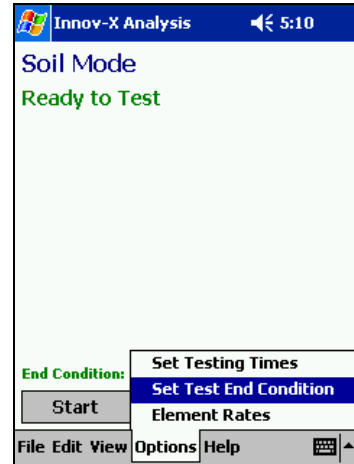
Four options exist for the test end criteria in soil mode. Depending on your application, you may choose to end the test manually, at a preset testing time, or when the uncertainty in the measurement is within a

specified relative standard deviation of the reading. Additionally, you can set up an action level for a single element. As soon as the measuring statistics are good enough to ensure that that the reading is above, below or at the action level, the test will end automatically. This allows for very rapid tests for elements that are well above or below an action level.

In all modes, pressing Stop, or pulling the trigger will end the test. If the minimum testing time has elapsed, results will be calculated. Otherwise the test will be aborted without calculating results.

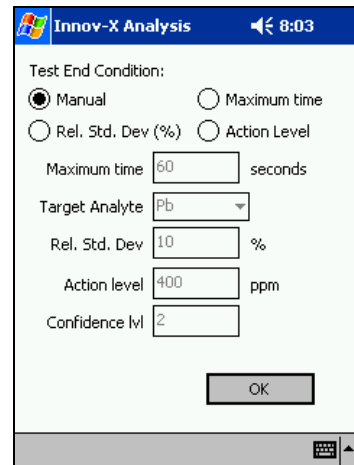
Changes to the test end condition are made by selecting **Options**→**Set Test End Condition**

The currently selected end condition will be displayed at the bottom of the screen above the Start button on the Ready To Test screen.



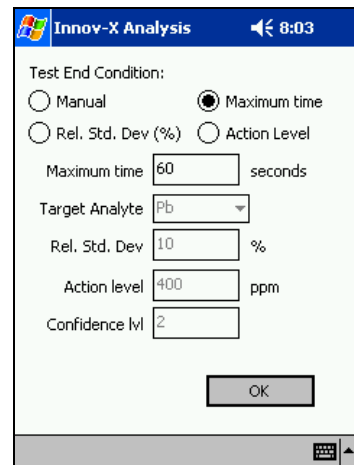
Manual: This option allows you to look at the results which are being continually updated on the screen and determine when the results look satisfactory. The test will continue until the trigger is pulled, or **Stop** is tapped on the iPAQ screen. Results will be calculated if the testing time has exceeded the Minimum Test time which is set up in **Options**→**Set Testing Times**. In order to preserve battery life, the software will stop if the testing time exceeds 300 seconds, since there is little to no advantage to continuing a test beyond 300 seconds.

To use Manual Test End Condition, simply choose **Options**→**Set Test End Condition** and select **Manual**. Press **OK** to return to the analysis screen.



Maximum Time: If Maximum Time is selected, the test will continue until the preset time is reached. This is useful if you wish to do a set of measurements with the same testing time.

To choose to end test based on a maximum time, select **Options**→**Set Test End Condition** and select **Maximum Time**. Enter the desired testing time in the appropriate box. Tap **OK** to save your selections.



Action Level: System ends test when result for target analyte including chosen precision level is above or below pre-set action level.

To choose to end a test based on an Action Level, select **Options**→**Set Test End Condition** and select **Action Level**. Select a target analyte, specify an action level in ppm, and a confidence level. This confidence level refers to the number of sigma required for the precision. This should typically be set to 2. Tap **OK** to save your selections.

Innov-X Analysis 8:03

Test End Condition:

Manual Maximum time

Rel. Std. Dev (%) Action Level

Maximum time 60 seconds

Target Analyte Pb

Rel. Std. Dev 10 %

Action level 400 ppm

Confidence lvl 2

OK

Relative Standard Deviation (RSD): When RSD is selected as a test end criteria, the system will end a test when the relative standard deviation on a target analyte reaches a pre-set level. This standard deviation is specified as a percentage of the reading. For example, if the measured value for an analyte was 1000 ppm, and the RSD was set to 10, the reading would stop when the error reached 100 ppm, or 10% of 1000.

To choose to end a test based on a Relative Standard Deviation, select **Options**→**Set Test End Condition** and select **Rel. Std. Dev (%)**. Select a target analyte and the desired Relative Standard Deviation. Tap **OK** to save your selections.

Innov-X Analysis 8:04

Test End Condition:

Manual Maximum time

Rel. Std. Dev (%) Action Level

Maximum time 60 seconds

Target Analyte Pb

Rel. Std. Dev 10 %

Action level 400 ppm

Confidence lvl 2

OK

10.5 LEAP Mode (Light Element Analysis Program):

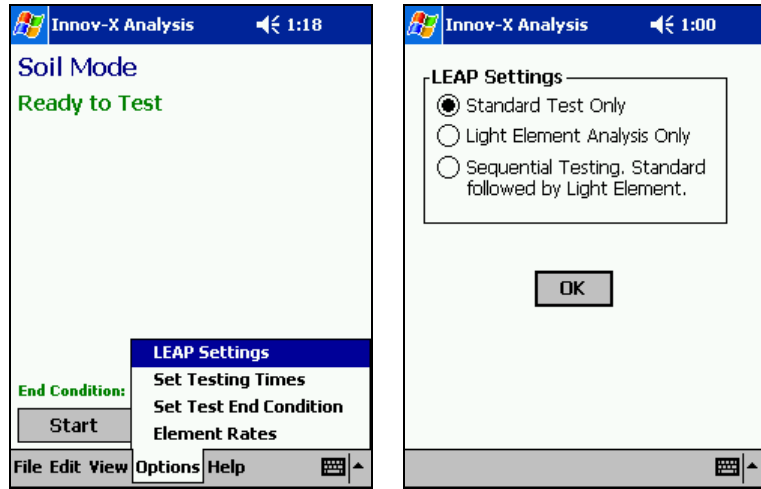
This is a factory installed optional module. Instruments can be upgraded to LEAP capabilities. Please contact the Innov-X Systems Sales department for information and pricing.

The LEAP module provides the lowest possible detection limits for elements lighter than iron. The standard LEAP package includes the elements Ti, Ba and Cr. Elements as low as Phosphorus can be detected with the Advanced LEAP package which includes a thin window detector.

The standard x-ray beam conditions used by Innov-X environmental analyzers are designed to provide good excitation for a wide range of detected elements. However it is not possible to select one beam condition which provides the absolute best excitation conditions for all elements of interest. Elements such as Chromium produce lower energy x-rays than other elements analyzed. These lower energy x-rays are not as effectively excited by the standard conditions. LEAP works by changing the X-ray tube beam conditions to settings which are optimized for the detection of elements lighter than iron. Instruments are factory calibrated with the LEAP beam conditions for all applicable elements.

10.5.1 LEAP Settings

To activate LEAP, select **Options**→**LEAP Settings** from the Soil analysis screen. This brings up the menu shown below on the right.

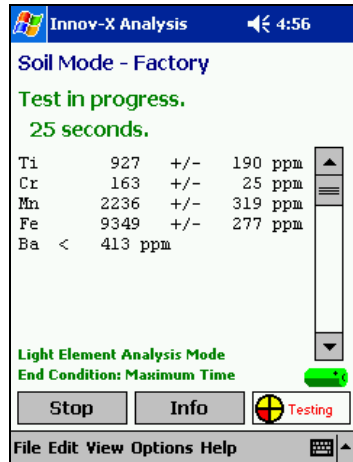


Standard Test Only: The analyzer will provide analysis for the standard suite of elements.

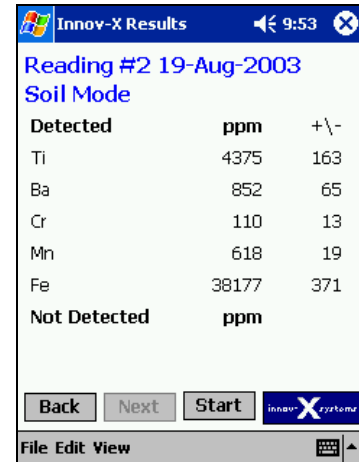
Light Element Analysis Only: The analyzer will provide analysis for elements in the LEAP suite (Typically Ti, Ba and Cr)

Sequential Testing: When sequential testing is selected, all tests will start with an analysis of elements in the standard suite. If that test ends due to reaching the selected end condition of Maximum Test Time, Action Level, or RSD, then the analyzer will immediately begin a second test analyzing the LEAP suite of elements. At the conclusion of this test, the Results screen will open with two new entries. The first summarizes the standard test results, while the second summarizes the LEAP results. For safety reasons, the second test will not begin if the test ends due to user intervention (pulling the trigger or hitting Stop). In this case, the Results screen will open with only one reading.

If Light Element Analysis Only is activated, the words “Light Element Analysis Mode” will appear above the currently selected End Condition. Instrument operation in this mode is identical to Standard (Non-LEAP) analysis. Tests can be started or stopped either by pulling the trigger, or by tapping the Start/Stop button on the iPAQ screen. The results screen for a test will show results for all elements analyzed with the LEAP mode.

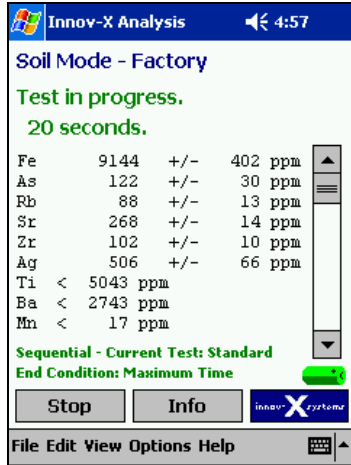


Test in progress screen, LEAP Only, Live Updates on

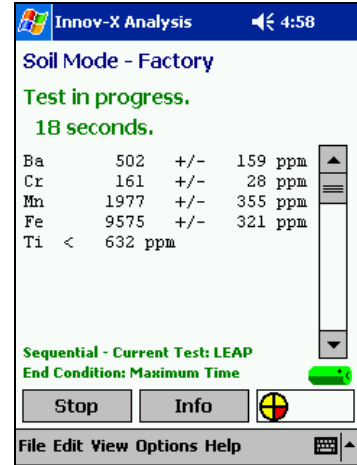


Results Screen Showing LEAP results

If Sequential testing is selected, the words “Sequential – Current Test: Standard” will appear above the currently selected End Condition. When a test is started, the instrument will appear to operate in the same manner as a Standard test. However, if the test ends according to the specified end condition (excluding Manual), the results screen will not open. Instead, the timer will reset to 0, and the description of the current test will change from “Standard” to “LEAP”. The live update screen will begin to show analysis for all LEAP elements.



Test in progress screen,
Sequential.
First Test – Standard Analysis.

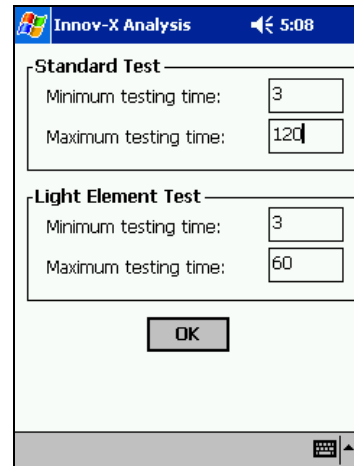


Test in progress screen,
Sequential.
Second Test – LEAP Analysis

10.5.2 Testing Times

To set the minimum and maximum test lengths for LEAP analysis, select **Options**→**Set Testing Times**.

The testing time screen includes an extra section labeled “Light Element Test” that is not found on non-LEAP systems. These are the minimum and maximum test lengths for any LEAP tests.



As with standard tests, the minimum testing time is the required time that must elapse before results can be calculated. Live Update results will not be displayed on the screen until the minimum has elapsed, likewise a test must complete the minimum time before any test end condition can be used. If a test is stopped before the minimum testing time has elapsed, the test will be aborted, and no results will be calculated.

Appendix 1: Standard Fingerprint Library

Standard Fast ID Library		
<i>Stainless</i>	<i>Chrome-Moly</i>	<i>Nickel Base</i>
304	1 1/4 Cr	Inco 600
309	2 1/4Cr	Inco 625
310	5 Cr	Inco 718
316	9 Cr	Inco 750
317		Inco 800
321	<i>Cobalt Base</i>	Inco 825
347	F-75	Hast C-276
410/416	HS-6	Hast X
	HS-25	Monel 400
<i>Low Alloy</i>	<i>Copper Base</i>	Monel 500
CS	70-30	Waspaloy
C ½ Mo	90-10	<i>Ti Base</i>
4140, 4130	CDA 836	CP Ti
4340		Ti 6-4

Appendix 2: Standard Grade Library

Iron Base Alloys			Ni-base Alloys		
201	Alnico VIII	Tool Steels	Ni	Inco 718	
203	AL6XN		80-20	Inco 722	
301	AMS 350		A2	B-1900	Inco 738
304	AMS 355		A6	B-1900 Hf	Inco 750
309	CD4MCU		A7	Inco 617	Inco 792
310	Custom 450		A10	Inco 625	Inco 800
316	Custom 455		D2, D4	C-1023	Inco 801
317	Duplex 2205		D7	GMR 235	Inco 825
321	Elgiloy		H12	GTD 222	Inco 901
329	Ferallium 255		H13	Hast B	Inco 903
330	Greek Ascology		L6	Hast B2	Inco 909
347	Hy Mu 80		O1	Hast C-4	Mar M 002
410/416/420	Kovar		O6	Hast C-22	Mar M 200
410 Cb	Invar 36		O7	Hast C-276	Mar M 246
422	Maraging C200		M1	Hast C-2000	Mar M 247
430/440	Maraging C250		M2	Hast F	Mar M 421
431	Maraging C300		M42	Hast G	Monel 400
434	Maraging C350		M4	Hast G-2	Monel 411
441	N-155		S1	Hast G-3	Monel 500
446	Ni-hard #1		S7	Hast G-30	MP35N
12L14	Ni-hard #4		T1	Hast N	Mu Metal
13-8 Mo	Nitronic 40		Low-Alloy Cr-Mo Steels	Hast R	Nichrome V
15.5 PH	Nitronic 50			Hast S	Nickel 200
17-4 PH	Nitronic 60	Hast X		Nim 101	
19-9DL	RA333	Hast W		Nim 263	
19-9DX	RA330	Carbon steel		Haynes 25	Nimonic 75
20Cb3		4140		Haynes 36	Nimonic 80A
20Mo4		1 1/4 Cr		Haynes 214	Nimonic 90
20Mo6		2 1/4Cr		Haynes 230	Ni-Span 902
25-4-4		5 Cr		Haynes 188	Rene 41
254SMO		9 Cr		Haynes 556	Rene 77
21-6-9		C - 1/2 Mo		HR-160	Rene 80
26-1 (Ebrite)				IN 100	Rene 95
29-4				Inco 600	Rene 125
29-4-2				Inco 601	Supratherm
904 L				Nim 101	Udimet 500
A-286				Nim 263	Udimet 520
Alloy 42				Inco 690	Waspaloy
Alloy 49				Inco 702	
Alnico II				Inco 706	
Alnico V				Inco 713	

Co-base Alloys	Cu-base Alloys	Ti-base Alloys	Misc. Alloys	Pure Elements
Co	Cu	CpTi	97-3	Ag
F-75	70-30	Cp Ti Pd	Cb 103	Cr
FSX 414	80-20	6-4	CP Ta	Hf
HS-1	90-10	6-6-2	Densalloy	Mn
HS-4	CDA 110	6-2-4-2	Tungsten	Mo
HS-6	CDA 314	6-2-4-6	Carbide	Nb
HS-12	CDA 360	3-2.5	Zir 702	Pb
HS-19	CDA 544	5-2.5	Zir 705	Pd
HS-21	CDA 630	15-3-3-3	Zircaloy	Re
HS-25 (L605)	CDA 706	10-2-3	2, 4	Sb
HS-31	CDA 836	Ti-8	Zr	Sn
Haynes 188	CDA 863	Ti-12		V
Jetalloy	CDA 875	Ti-17		W
Mar M 302	CDA 903	Ti 6-22-22		Zn
Mar M 509	CDA 932	Ti 13-11-3		Fe
MP 35N	CDA 937	Beta C		
Star J	CDA 954	Ti 6-2-1-1		
Ultimet	CDA 955			
	CDA 8932			

The Standard Grade library holds 250 alloys with specifications. Three additional user libraries are available, each hold 100 alloys. Users may edit all libraries entries and may add or delete grades and fingerprints.

Appendix 3: Troubleshooting Guide—Alloy Analysis

Problem	Possible Solutions
<p>Software won't start:</p> <p>Software will not start when the Innov-X Systems Icon is tapped.</p>	<p>The flash card or the iPAQ may not be correctly seated in the black external sleeve. Remove the flash card and press it firmly into its holder. Press the iPAQ down into the black sleeve.</p>
<p>Software won't start:</p> <p>Software doesn't start when the Innov-X System icon is tapped; instead, the following error message occurs: "Cannot find 'startup' (or one of its components). Make sure the path and filename are correct and all the required libraries are available"</p>	<p>The flash card or the iPAQ may not be correctly seated in the black external sleeve. Remove the flash card and press it firmly into its holder. Press the iPAQ down into the black sleeve.</p>
<p>iPAQ locks up:</p> <p>iPAQ screen "locks up" and doesn't respond when screen is tapped or buttons are pressed</p>	<p>Remove the iPAQ from the analyzer and perform a soft reset by pressing the tip of the stylus into the small indentation found on the bottom of the iPAQ. If the iPAQ is lying flat on a table with the screen facing upwards, the reset button is found to the extreme right of the side containing the power plug and connector.</p> <p>.</p>
<p>Analyzer will not standardize</p>	<p>Try again. Choose File -> Standardize to attempt a new standardization. Also be sure the standardization cap is on correctly, and that the solid half is in front of the window. It is OK to try this 2-3 times in the event of a failure.</p> <p>If a repeat attempt fails: Change the battery. In some cases the battery may be too low to provide enough power for tube startup. Follow this procedure:</p> <ul style="list-style-type: none"> • Reset the iPAQ; • Turn off the analyzer and remove the battery. • Verify that the battery is completely charged. If it is not, replace it with a fresh battery. Even if the battery has been recently recharged, remove it, and replace it in the analyzer. • Restart the analyzer and software. Wait several minutes after the software has initialized before attempting standardization.
<p>Incorrect Alloy ID in FAST ID Mode:</p> <p>Analyzer does not correctly ID sample that was just added to the library in FastID.</p>	<p>Verify that the Alloy was saved in a library which is being searched. <i>Use File→Load libraries</i> to change the library being search. <i>Edit→Fingerprint libraries</i> "Show/Modify" can be used to check that the fingerprint was saved in the proper library.</p>

<p>Analyzer gives “No Match” for every sample.</p>	<ol style="list-style-type: none"> 1. Verify that Match Numbers are set to 1 for Fast ID and 3 for Analytical. Change values by selecting Options→Fingerprint Setup from the Analysis screen for FastID mode, or Options→Grade Library Settings from Analytical mode. 2. Verify that you are searching the correct library. Use File→Load Libraries to change the library being searched. Most users should search all libraries. 3. Check to make sure that the analyzing window is clean.
<p>All tests yield incorrect match</p>	<p>Check the date and time on the last result shown. It should show the current date. If it doesn't, check the date on the iPAQ. The Innov-X Systems software indexes stored results by date. If the date is incorrect, results may not be displayed in the correct order.</p>
<p>Results screen doesn't show new readings after a test is completed</p>	<p>Check the date on the iPAQ. The Innov-X Systems software indexes stored results by date. If the date is incorrect, results may not be displayed in the correct order.</p>
<p>Serial Communication Error Message:</p> <p>Serial Communication error occurs because iPAQ has been removed from instrument or cradle, with the software open and the instrument standardized.</p>	<p>This error reflects the temporary loss in communications when the iPAQ was removed. To avoid this problem, always use the File→Exit command to exit the software properly. Try simply removing and reseating the iPAQ to solve this problem. If that fails, see steps 1 – 4 below.</p>
<p>Serial communication error on startup, or while testing.</p>	<ol style="list-style-type: none"> 1. If the analysis screen is still open, attempt another test. 2. Verify that the iPAQ is correctly seated in the analyzer by removing and replacing it. 3. Remove the iPAQ and perform a soft reset. Replace iPAQ and restart software. 4. Turn the analyzer off and restart it.
<p>Trigger will not start test.</p>	<p>Reset the instrument. If this fails, call Innov-X Systems Technical Support at 781-938-5005.</p>

Broken Kapton Window

The window is designed as a barrier to dust and dirt. If it is damaged, it should be replaced.

To change the window:

Turn off the analyzer

Remove the screws holding the front plate in place.

Remove the old kapton and adhesive, replace with new kapton and replace front plate.

Important Note: It is very important to avoid getting dirt and sharp objects within the probe, due to the close proximity of the detector. Do not use the analyzer without a kapton window for any length of time. Also, be very careful when removing/replacing screws in face plate so as to not accidentally damage the detector. If the detector is damaged, the instrument will require factory service.

Appendix 4: Guide to Product Registration

Generally, the Innov-X portable XRF system must be registered in the state of usage. Registration requirements are somewhat state dependent, but there are many similarities. You may contact Innov-X at 866-4-Innov-X (781-938-5005) to receive specific registration information. Innov-X also maintains sample registrations for every state that we can forward to you. Outside the United States, our local sales agents provide guidance in the proper registration of the analyzer.

Common Registration Features:

Most states require the following for registering an x-ray emitting device that does NOT use radioactive sources:

1. Registration within 30 days of receipt of the analyzer.
2. Annual fee in the \$25 to \$200 range, depending upon the state.
3. Basic registration form with main information described below.

Common information required on Registration Form, and responses:

Company name, address, phone/fax numbers.

Name of responsible person:	Generally the person designated as the Radiation Safety Officer (RSO).
Name of the manufacturer:	Innov-X Systems, Inc., Woburn, MA
Model of Analyzer:	XT-220 or XT-260.
Tube Operating Parameters:	35 kV, 20 uA current.
Type of Analysis:	Choose Analytical or Industrial (as opposed to radiography, medical, dental, veterinarian, etc.)
Utilization Mode:	Portable or Mobile assuming you will carry system to different locations. Fixed or stationary ONLY if you will always use the analyzer in the docking station

Appendix 1: Troubleshooting Guide—Soil Analysis

Problem	Possible Solutions
<p>Software won't start:</p> <p>Software will not start when the Innov-X Systems Icon is tapped.</p>	<p>The flash card or the iPAQ may not be correctly seated in the black external sleeve. Remove the flash card and press it firmly into its holder. Press the iPAQ down into the black sleeve.</p>
<p>Software won't start:</p> <p>Software doesn't start when the Innov-X System icon is tapped; instead, the following error message occurs: "Cannot find 'startup' (or one of its components). Make sure the path and filename are correct and all the required libraries are available"</p>	<p>The flash card or the iPAQ may not be correctly seated in the black external sleeve. Remove the flash card and press it firmly into its holder. Press the iPAQ down into the black sleeve.</p>
<p>iPAQ locks up:</p> <p>iPAQ screen "locks up" and doesn't respond when screen is tapped or buttons are pressed</p>	<p>Remove the iPAQ from the analyzer and perform a soft reset by pressing the tip of the stylus into the small indentation found on the bottom of the iPAQ. If the iPAQ is lying flat on a table with the screen facing upwards, the reset button is found to the extreme right of the side containing the power plug and connector.</p> <p>See Page 4 of the Compaq "Getting Started" manual for an illustration showing the location of the reset button.</p>

<p>Analyzer will not standardize</p>	<p>Try again. Choose File -> Standardize to attempt a new standardization. Also be sure the standardization cap is on correctly, and that the solid half is in front of the window. It is OK to try this 2-3 times in the event of a failure.</p> <p>If a repeat attempt fails: Change the battery. In some cases the battery may be too low to provide enough power for tube startup. Follow this procedure:</p> <p>Reset the iPAQ;</p> <p>Turn off the analyzer and remove the battery.</p> <p>Verify that the battery is completely charged. If it is not, replace it with a fresh battery. Even if the battery has been recently recharged, remove it, and replace it in the analyzer.</p> <p>Restart the analyzer and software. Wait several minutes after the software has initialized before attempting standardization.</p>
<p>Results screen doesn't show new readings after a test is completed</p>	<p>Check the date on the iPAQ. The Innov-X Systems software indexes stored results by date. If the date is incorrect, results may not be displayed in the correct order.</p>
<p>Serial Communication Error Message:</p> <p>Serial Communication error occurs because iPAQ has been removed from instrument or cradle, with the software open and the instrument standardized.</p>	<p>This error reflects the temporary loss in communications when the iPAQ was removed. To avoid this problem, always use the File / Exit command to exit the software properly. Try simply removing and reseating the iPAQ to solve this problem. If that fails, see steps 1 – 4 below.</p>
<p>Serial communication error on startup, or while testing.</p>	<ol style="list-style-type: none"> 1. If the analysis screen is still open, attempt another test. 2. Verify that the iPAQ is correctly seated in the analyzer by removing and replacing it. 3. Remove the iPAQ and perform a soft reset. Replace iPAQ and restart software. 4. Turn the analyzer off and restart it.

<p>Results take a very long time to display on the first test of the day.</p>	<p>There may be too many readings stored in memory. Erase readings from the results screen by selecting <i>File</i> → <i>Delete Readings</i>.</p>
<p>Trigger will not start test.</p>	<p>Verify that the trigger lock is off.</p> <p>Reset the instrument. If this fails, call Innov-X Systems Technical Support at 781-938-5005.</p>
<p>Broken Kapton Window</p>	<p>The window is designed as a barrier to dust and dirt. If it is damaged, it should be replaced.</p> <p>To change the window:</p> <p>Turn off the analyzer</p> <p>Remove the screws holding the front plate in place.</p> <p>Remove the old kapton and adhesive, replace with new kapton and replace front plate.</p> <p>Important Note: It is very important to avoid getting dirt and sharp objects within the probe, due to the close proximity of the detector. Do not use the analyzer without a kapton window for any length of time. Also, be very careful when removing/replacing screws in face plate so as to not accidentally damage the detector. If the detector is damaged, the instrument will require factory service.</p>
<p>Results screen shows message “Error in calculation: No Results”</p>	<p>The soil mode calculation is only valid for “soil-like” samples which contain primarily light elements such as carbon, oxygen and silicon. If a dense, highly metallic sample is analyzed, the calculation fails.</p> <p>Make sure the sample being analyzed is a soil sample, if it is and this message occurs repeatedly; call Innov-X technical support.</p>

Appendix 2:

Metals in Soil Analysis Using Field Portable X-ray Fluorescence

A guideline to using portable XRF according to EPA Method 6200, basic overview of the technique of x-ray fluorescence (XRF), appropriate data quality assurance protocols and sample preparation steps for operators analyzing prepared soil samples.

Prepared by:

**Innov-X Systems, Inc.
January, 2003**

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Section 1: Regulatory Status for Field Portable XRF

EPA Reference Method 6200 has been incorporated into SW486 under RCRA, and is now available for field portable XRF analysis of soils and sediments. Please call or email Innov-X Systems for a copy of Method 6200.

Method 6200: Field Portable XRF Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment.

Features of this method:

1. It is a field screening method, for analysis of *in-situ* or bagged samples.
2. The method provides basic quality assurance methods, including calibration verification, determination of instrument precision, accuracy and limit of detection.
3. The method recognizes that some XRF instruments do not require site-specific calibrations by the operator, that is, the factory calibration provides appropriate data quality.
4. The method recommends that a minimum of 5-10% of samples tested by XRF be confirmed by an outside laboratory using a total-digestion EPA analytical reference method.

The purpose of EPA Method 6200 is NOT to replace laboratory analysis. There are two primary sources of error in assessing a site for metal concentration: **Analytical error** and **Sampling error**. Analytical error is the error in the analysis of any one sample by whatever technique is used, for example XRF, ICP, or AA. Sampling error arises when too few samples are collected and tested. In this case an incomplete picture of the extent of metals contamination may be obtained. Although any one sample may be analyzed with very high analytical accuracy, measuring too few samples may result in contamination plumes being mis-judged in size, or depth into the soil. In extreme cases contamination may be missed entirely.

EPA Method 6200 was developed to reduce Sampling Errors by increasing the number of samples measured. In general, a large number of screening-level measurements provide a better characterization of contamination than a small number of measurements produced by sample removal and analytical analysis. Portable XRF is an ideal tool to make a large quantity of measurements in a short period of time. A large number of in-situ samples provides detailed data on contamination profiles, depth (provided surface soil is moved aside), and approximate contamination levels. Portable XRF also can provide results with a high degree of analytical accuracy on any given sample. Please see Section 2 “Overview of Field Usage” for this discussion.

Section 2: Overview of Field Usage:

Field portable XRF is generally used in three ways to test for metals in soil:

- ❑ **In-situ soil testing:** The XRF is placed directly onto the ground for soil testing. Operators remove any plant growth and foreign objects so that the analyzer probe is flush to the soil.
- ❑ **Bagged soil sample testing.** A soil sample is collected in a thin plastic bag (i.e. a “Baggie”) and testing directly through the Baggie. Except for a few elements – namely Cr, V and Ba – testing through the thin plastic used for a plastic bag has little effect on the test result. Results for Cr, V and Ba will be lower by 20-30%.
- ❑ **Prepared soil sample testing.** Prepared sample testing assures the operator of the maximum possible accuracy. Prepared sample tests require a sample to be collected, dried if necessary, sieved and ground into a powder. The prepared sample is then placed into a baggie or XRF cup for analysis. **A complete Soil Preparation Guide is provided in Appendix 1.**

All analytical methods require a uniform, homogenous sample for the best results. **XRF is no different!** The methods described in EPA Method 6200, namely In-situ and bagged sample testing, are considered *field-screening methods*. Although a field-screening method, in-situ testing is a valuable technique because it generates a great deal of data very quickly. Prepared soil samples generally offer the best accuracy, albeit with several minutes of sample preparation required per sample.



Figure 1. Use of a field portable XRF for in-situ soil testing.

Subsection 2-A: Data Quality Objectives.

The objectives of the testing generally determine the mixture of in-situ versus prepared sample testing. It is important to understand your data quality objectives (DQO) in order to determine the appropriate mix of field screening and prepared sample testing.

In-situ testing usually provides only screening-level data quality. This is because analytical testing always requires a uniform, homogeneous sample matrix. A laboratory achieves this by digesting the sample into a hot acid before analysis. Testing directly on the ground does not ensure uniformity is met. Preparing a sample provides a uniform sample and likely better analytical data quality, although several minutes of testing time is required.

Most portable XRF operators use a mixture of in-situ and prepared sample testing. Several examples are described below. The exact mixture of in-situ and prepared sample testing depends upon the goals of the soil testing. The examples below serve as guidelines. Please contact Innov-X (1-866-4 Innov-X or 866-446-6689) to discuss your specific testing requirements.

Example 1: Initial site investigation to provide detailed contamination data with efficient use of laboratory analysis costs.

Problem: Site needs to be assessed for metals contamination. Little information is available about what metals are present, likely contamination levels or geographic profile of contamination.

The goal of testing is to determine what metals are present at what levels, both in area and in depth into soil. Additionally, testing will locate possible contamination plumes and/or possible sources of contamination.

Recommended Testing Plan: This example uses predominately in-situ testing. The analyst will perform in-situ testing, and gather samples into plastic bags for XRF analysis. A testing grid should be established in two or three dimensions, every several feet. XRF tests can be taken at each location or bagged samples can be collected from each location for later analysis. The in-situ data for each element analyzed may be plotted in a 2-dimensional grid (X, Y coordinates versus elemental concentration) to profile a site. These concentration profiles are ideal for showing contamination patterns, boundaries and plumes. Combining this data with historical use data from the site often allows the operator to deduce sources of contamination. Obtaining this level of geographic data with purely laboratory analysis would produce excessive analytical costs.

Prepared sample analysis should also be done to confirm the regions where in-situ data indicates low or non-detected levels of metal contaminant. There is little need to prepare areas where in-situ testing indicates high concentration levels. Innov-X recommends the same procedure as EPA Method 6200. For locations where in-situ tested indicate low or non-detected concentrations, calculate the total number of in-situ tests, collect 5% of this number of tests from the various locations, and prepare these samples according to Appendix 1. Use these prepared samples to confirm the findings of the in-situ testing. Send a subset of these prepared samples to a laboratory for confirmatory results.

Cost Justification. To adequately characterize a site may require 100-200 samples/acre to be sure the contaminated areas are firmly established. This work may be done with in-situ testing to generate laboratory savings of \$5,000 - \$10,000/acre depending upon the number of elements being analyzed. The cost reduction in off-site analysis often justifies the price of the XRF.

Example 2: Monitor remediation efforts and assure site meets clearance levels before contractors leave the site.

Goal: Minimize remediation costs by only treating contaminated soil, and obtain immediate verification that various site locations meet clearance objectives.

Recommended Testing Plan: This type of project uses a lot of both in-situ and prepared sample testing. Use in-situ testing to thoroughly delineate contamination regions in both area and depth. To determine depth profiles, test surface soil, remove at least 1-2 inches, and retest. Repeat this step as necessary to profile contamination depth to guide remediation activities. (XRF is a surface technique and only analysis the first few mm of soil sample). As part of clearance, collect several samples from “cleared” area. Prepare samples according to Appendix 1 and test with portable XRF.

If XRF indicates that concentration levels are in excess of clearance requirements, then continue remediation efforts.

If XRF indicates that concentration levels are below clearance requirements, then discontinue remediation efforts, and send a subset of the samples to an analytical laboratory to confirm results. Most operators safely assume that the cleanup requirements have been met for the elements in question, but await final analysis from the laboratory.

If XRF lists concentration levels as non-detected, but the detection level reported exceeds clearance requirements, send samples to a laboratory for final results.

Cost Justification: In-situ results are used to guide remediation efforts, in order to obtain maximum efficiency. Efficiency is produced because contamination boundaries are firmly established, thus avoiding remediation efforts with “clean” soil. Prepared sample testing is used to assure that clearance requirements are met on-site in near real-time (pending laboratory confirmation). Costs savings are generated by avoiding clearance failures. The contractors can leave the site earlier and will not be called back to the site for additional cleanup.

Important Note: Never clear a site based solely on in-situ testing. Always use well-prepared samples to make a clearance decision.

Example 3: Minimize volume of hazardous waste for treatment or disposal.

Goal: For some cleanup projects, the cost of soil disposal in a hazardous waste landfill is much greater than disposal in a standard landfill. Testing soil samples with XRF may minimize the amount of “clean” soil that is inadvertently shipped to a hazardous-waste landfill.

Recommended Testing Plan: This example is almost entirely prepared sample testing. Representative samples are removed from the soil being hauled to landfill. Obtaining an accurate analysis of the samples is crucial for making a hazardous versus non-hazardous determination. For this reason, prepared sample testing is strongly recommended.

Important Note: These types of samples are subject to TCLP procedures for the landfill determination. In general, 20 times the XRF result should be less than the allowable limit for the metal in question. Please contact Innov-X Systems for more details on testing samples versus TCLP regulatory requirements.

Section 3: Quality Assurance.

Quality assurance is detailed for both the proper use of the analyzer (which is also provided in Method 6200) and for verifying the data quality of in-situ testing. All operators should perform the QC procedure, regardless of their data quality objectives. Method 6200 has strict requirements about quality assurance. Additionally, Innov-X recommends that operators verify the data quality of in-situ test results, if they are using in-situ data to guide their reporting or remediation decisions. Procedures are listed below:

3.1: Proper verification of instrument operation

These procedures are taken from EPA Method 6200 and updated to be specific to the Innov-X analyzer. Quality assurance here consists of testing known standards to verify calibration, as well as testing blank standards to determine limits of detection and to check for sample cross-contamination or instrument contamination. EPA Method 6200 provides a detailed procedure, which is provided here in abbreviated form.

Components of instrument QC:

1. An energy calibration check sample at least twice daily
2. An instrument blank for every 20 environmental samples
3. A method blank for every 20 prepared samples
4. A calibration verification check sample for every 20 samples
5. A precision sample at least one per day.
6. A confirmatory sample for every 10 environmental samples

Energy Calibration Check: The Innov-X analyzer performs this automatically; this is the purpose of the standardization check when the analyzer is started. The software does not allow the analyzer to be used if the standardization is not completed.

Instrument Blank: The operator should use the SiO₂ (silicon dioxide) blank provided with the analyzer. The purpose of this test is to verify there is no contamination on the analyzer window or other component that is “seen” by the x-rays. Method 6200 recommends an instrument blank at least once per day, preferably every 20 samples. For either in-situ or prepared-sample testing, the operator should just test the SiO₂ blank to be sure there are no reported contaminant metals.

Method Blank: The purpose of the method blank is to verify that cross-contamination is not introduced into samples during the sample preparation process. Method 6200 recommends following the sample preparation procedures with clean SiO₂ once every 20 prepared samples. This QC step is not required if the operator is not preparing samples.

Calibration Verification: Innov-X provides NIST standard reference samples for calibration check by operator. The operator should perform a 2-minute test on a NIST standard. The difference between the XRF result for an element and the value of the standard should be 20% or less. Calibration Verification should be performed upon instrument startup and periodically during testing. Note: Innov-X recommends a calibration check every 4 hours. EPA Method 6200 recommends a calibration check every 20 samples NIST reference standards are generally applicable for Pb, As, Cr, Cu, Zn. Innov-X provides additional reference standards for other RCRA or Priority Pollutant metals including Cd, Se, Ag, Hg, Ag, Ba, Sn, Sb, and Ni.

Precision Verification: Quoting from EPA “A minimum of one precision sample should be run per day by conducting from 7 to 10 replicate measurements of the sample. The precision is assessed by calculating a relative standard deviation (RSD) of the replicate measurements for the analyte. The RSD values should be less than 20 percent for most analytes, except chromium, for which the value should be less than 30 percent.

Confirmatory Sample: It is recommended that one confirmatory sample is run for every 10 samples collected. According to EPA Method 6200: “Confirmatory samples are collected from the same sample material that is analyzed on site, but are sent to an off-site laboratory for formal analysis. The purpose of a confirmatory sample is to judge the accuracy of the data obtained by analysis on site and to allow corrections, if necessary.”

Important Notes about confirmatory samples:

Innov-X always recommends that customers compare prepared-sample results to laboratory results. To do this, collect and prepare a sample following the protocols of Appendix 1. Take a subsample and submit to the laboratory for analysis. The single largest error in XRF analysis is lack of sample preparation. For the best comparison, always use prepared samples.

3.2: Determining data quality of in-situ testing:

For operators relying extensively on in-situ testing, it is important to determine the data quality of this testing at a given site. *This protocol is not intended for every sample, but rather for a small percentage of samples considered representative of the site.* If the operator can demonstrate that quantitative data is achieved with little or no sample preparation, then the site characterization will be completed much more quickly but correctly.

For example, an operator may be able to demonstrate that the XRF result changes considerably when samples are passed through a 2 mm sieve, but that XRF results do NOT change appreciably upon finer sieving. In this case the operator can conclude that good XRF data is achievable with only 2 mm sieving. Sieving only to this level requires far less time than a more robust sample preparation. A protocol to determine the appropriate level of sample preparation is the following:

1. Delineate a region of soil approximately 4" x 4".
2. Perform several in-situ tests in this area, or collect the top (approximately) quarter inch of soil from this region, bag the soil, test through the bag. In either case, average the results.
3. If you did not bag the in-situ test sample, collect the top (approximately) quarter inch of soil from this region and sieve through the 2 mm sieve provided. Otherwise sieve the bagged sample used for the in-situ test. Thoroughly mix the sieved sample, and place some of the sieved material into an XRF cup, and perform a test of this sample.
4. If the results of this prepared sample differ less than 20% with the average in-situ result, this indicates the soil in this region is reasonably homogeneous. The data quality in this case is probably at the semi-quantitative level, rather than just screening data.
5. If the results differ by more than 20%, this indicates the soil is not very homogeneous, and there are serious particle size effects affecting your in-situ measurements.
6. In this case, sieve the sample through the 250 ~m sieve. Mix this sample and place a subsample into an XRF cup for testing. If this result differs from the previous by less than 20% then this indicates that at a minimum the 2mm sieving is necessary to achieve higher data quality.

7. If this result differs by more than 20% from the sample sieved through 2 mm, then particle size effects are still affecting the XRF result. In this case samples should be sieved through 125 μm to assure data quality at the quantitative level.

Section 4: Calibration for Innov-X Portable XRF

The Innov-X analyzer may run three different calibration methods, described below. In nearly all cases, customers use the Compton Normalization method. This method (recognized in EPA 6200) offers speed, ease of use, and generally good accuracy for concentration ranges from the ppm level up to 2-3% concentrations. As most field-testing is seeking to remediate or locate environmental contaminants, the upper limit of the calibration (2-3%) is generally not a limitation. If customers do require a calibration up to 100% concentration (i.e. a pure element) then Innov-X recommends they also include the Fundamental Parameters (FP) software module with the analyzer. The FP module may be added at time of purchase or as an upgrade at any later date.

Note: In general customers do not need to calibrate the Innov-X analyzer for soil testing. The analyzer is delivered with a factory calibration, generally based upon the Compton Normalization (CN) method. The CN method has been proven over the past several years to provide a robust calibration generally independent of site-specific soil matrix chemistry. The operator may calibrate the Innov-X system if desired, but calibration is not required to use the analyzer effectively. All customers should follow the QC procedure described in Section 3, which includes a check of the calibration.

The final model is the empirical calibration. In this case, customers run standards to generate calibration curves for various elements in specific soil matrices. Provided the sample is well-prepared, the empirical method generally yields the most accurate result. In our experience, the accuracy gains going from Compton Normalization to Empirical Mode are small and not worth the extra effort in setting up calibration curves. (The greatest source of error for in-field XRF analysis of soil is lack of adequate sample preparation, thus there is little gained in developing a sophisticated empirical calibration if the operator does to grind and homogenize the all measured samples). The empirical calibration module is an optional software package, available for an upgrade fee at the time of purchase, or as an upgrade at any later date.

Calibration Requirements:

The concentration of an element in a soil sample is well-described by the formula:

$$w_i = \frac{k_i}{M(Z, i)} I_i$$

k_i = calibration constant for element “i”

w_i = concentration of element “i” – the quantity being measured.

I_i = measured x-ray intensity from element “i”

$M(Z, I)$ = Soil matrix value

The factory calibration determines the value of the calibration constants k_i for each element, and a typical value $M(Z,I)$. The calibration method – either CN, fundamental parameters, or empirical – performs the necessary corrections to the value $M(Z,I)$ that are important for the site-specific soil chemistry. The XRF analyzer uses the measured intensity of each element's fluorescence from the sample, and the calibration data, to produce elemental concentrations.

Compton Normalization:

The Compton Normalization method calibration consists of the analysis of a single, well-characterized standard, such as an SRM or SSCS. The standard data are normalized to the Compton peak. The Compton peak is produced from incoherent backscattering of X-ray radiation from the excitation source and is present in the spectrum of every sample. The matrix affects the way in which source radiation is scattered off the samples. This scatter is directly related to the intensity of the Compton peak. For that reason, normalizing to the Compton peak can reduce problems with matrix effects that vary among samples. Compton normalization is similar to the use of internal standards in analysis for organic analytes.

Fundamental Parameters Calibration:

The fundamental parameters (FP) calibration is a "standardless" calibration. Rather than establishing a unit's calibration curve by measuring its response to standards that contain analytes of known concentrations, FP calibration relies on the known physics of the spectrometer's response to pure elements to set the calibration. Built-in mathematical algorithms are used to adjust the calibration for analysis of soil samples and to compensate for the effects of the soil matrix. The FP calibration is performed by the manufacturer, but the analyst can adjust the calibration curves (slope and y-intercept) on the bases of results of analyses of check samples, such as SRMs which are analyzed in the field.

Empirical Calibration:

The empirical calibration method requires that a number of site-specific calibration standards (SSCS) are used to establish calibration parameters. The instrument response to known analytes is measured and used to create calibration curves. Empirical calibration is effective because the samples used closely match the sample matrix. SSCSs are well-prepared samples collected from the site of interest in which the concentrations of analytes have been determined by inductively coupled plasma (ICP), atomic absorption (AA), or other methods approved by the US Environmental Protection Agency (EPA). The standards should contain all the analytes of interest and interfering analytes. Manufacturers recommend that 10 to 20 calibration samples be used to generate a calibration curve. The empirical method is the least desirable calibration method as it requires that new standards and curves are generated for each site that is analyzed.

Section 5: Effects of Moisture on XRF Results:

Sample moisture has two effects on XRF results:

- ❑ It alters the soil chemistry, since water is another chemical compound that comprises the soil matrix.
- ❑ Moisture impedes the ability to properly prepare samples.

- ❑ Laboratory results are provided on a “dry weight” basis.

Effect on Soil Chemistry:

While the presence of significant moisture does impact the soil chemistry, modern XRF analyzers all perform automatic corrections for variations in soil chemistry from site to site. Indeed, such variations are expected, and that is the reason analyzers use Compton Normalization or fundamental parameters, in order to correct for moisture content changes as well as other differences in soil geochemistry.

EPA Method 6200 states “Moisture content above 20 percent may cause problems, since moisture alters the soil matrix for which the FPXRF has been calibrated.” However, the Compton Normalization or fundamental parameters methods are implemented in order to automatically correct results for changes to the soil matrix. Thus, we believe that soil moisture is not a significant effect on accuracy due to effects of soil matrix, except for the “dilution” effect that can cause discrepancies with laboratory results which is described below.

Sample preparation issues:

The inability to adequately prepare a wet sample is, we believe, the single biggest contributor to errors when testing wet samples. It is very difficult to grind or sieve a wet sample. The highest quality XRF results are generally obtained from prepared samples. If the operator is unwilling to dry the sample to prepare it, comparisons to the laboratory may yield poorer correlation since the samples are not homogeneous.

Laboratory Tests on Dry-Weigh Basis:

Laboratories always dry samples prior to analysis. They report percent weight content based upon a dry sample basis. Portable XRF may often be used to analyze wet samples in the field, and results are thus reported that include the moisture content. Thus, with all other factors the same, the laboratory will report results higher than portable XRF. The results will be higher by the amount of moisture content in the sample. For example laboratory results will be 10% higher compared to XRF results, if the sample contained 10% by weight water when it was tested with XRF. Recall, this applies to samples where other possible sources of error are the same or negligible.

Section 6: Comparing XRF Results to Laboratory Results:

Innov-X strongly recommends that operators compare prepared sample results to laboratory results. This is because prepared-sample results yield the best possible accuracy with portable XRF. Moreover, the most common source of error is due to non-uniform samples. The XRF technique, nor can any analytical technique, properly account for non-uniform sample types.

To perform a comparison between XRF results and laboratory:

1. Collect a sample and prepare it according to the sample preparation guide in Appendix 1.

2. Take a sub-sample (5-10 grams) of the fully-prepared sample, place it into an XRF cup and perform at least a one-minute test on that sample.
3. Send the same sample to the laboratory for wet chemistry analysis.
4. Require the laboratory to use a total-digestion method. If the laboratory does not use a total digestion method, they may not extract all of the elemental metal from the sample. In this case, the lab result will be lower than the XRF result. Incomplete sample digestion is one of the most common sources of laboratory error, thus it is very important to request a total digestion method.

Example of Error: The operator collects a bag of sample, performs XRF analysis on one part of the bag, and sends the bag, or part of the bag of sample to a laboratory for analysis. The laboratory reports a very different value than the operator obtained with the XRF.

Problem: Since the sample is very non-homogeneous, the operator did not obtain a result that was representative of the entire bag of sample. The lab analyzed a different part of the sample and obtained a very different result due to the non-uniformity of the sample. The solution to this problem is, at a minimum, to test several locations in the bag of sample and report the average value. Also note the differences between the tests, as this is indicative of the non-uniformity of the sample. Operator should send entire bag of sample to the lab, and instruct lab to prepare the sample before removing sub-sample for lab analysis.

Best Practice: The operator should homogenize and prepare the entire bag of sample, and then collect a sub sample for XRF testing. After testing, the same sample should be sent to the lab.

Section 7: Common Interferences:

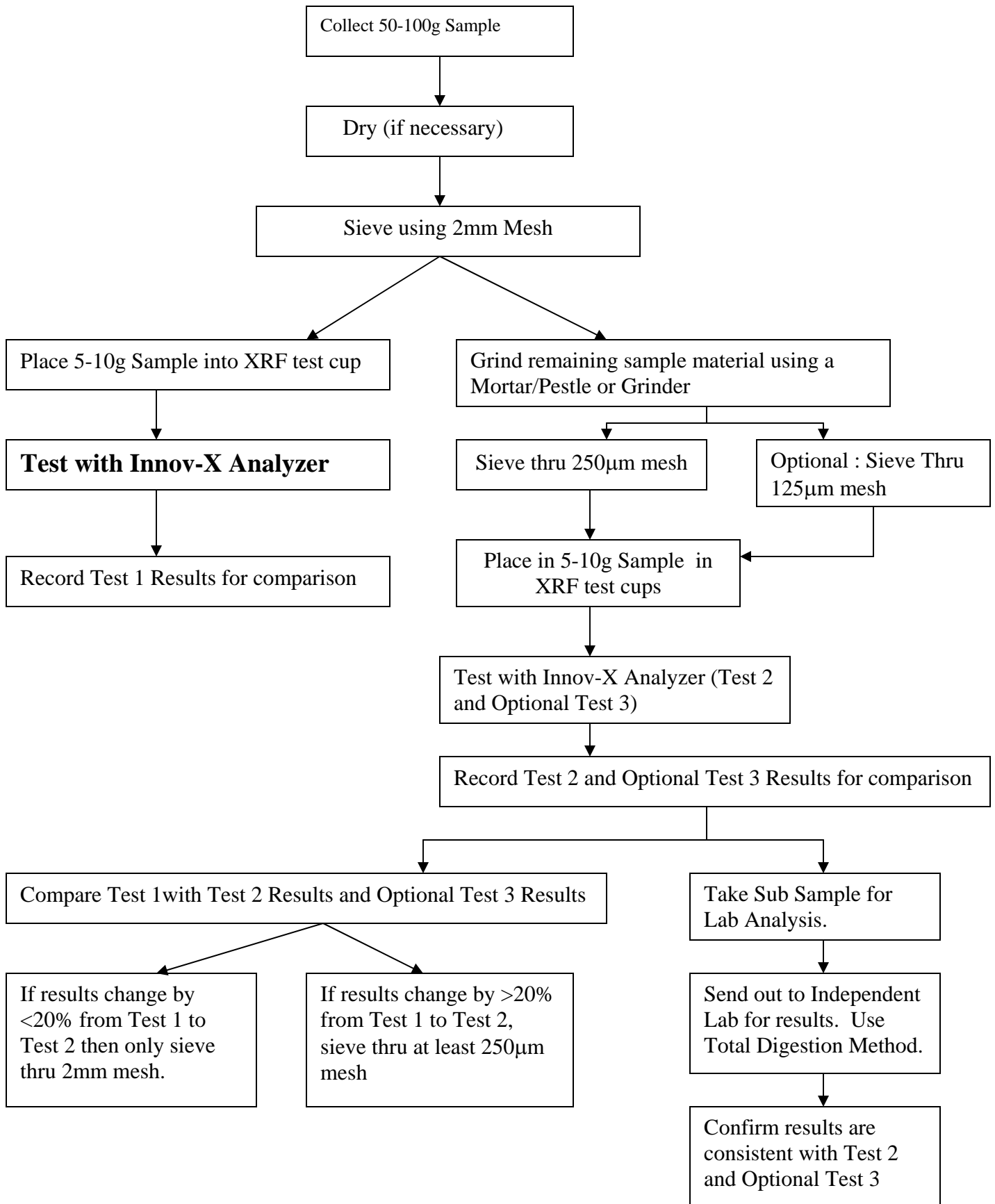
An interference occurs when the spectral peak from one element overlaps either partially or completely with the spectral peak of another. If the XRF is calibrated for both elements (CASE 1) i.e. the one causing the interference and the one being interfered with, it is generally capable of correctly handling the interference. In this case, the element being interfered with may be measured with a poorer detection limit or poorer precision, but the analytical results should still be acceptable for field-portable XRF. If the XRF is not calibrated for the element causing the interference (CASE 2), then the XRF may report the presence of elements not in the sample, or greatly elevated concentrations of elements in or not in the sample.

Example CASE 1: Lead and arsenic. Most XRFs are calibrated for lead and arsenic. Lead interferes with arsenic (not vice-versa though). The net effect is a worsened detection limit for arsenic, and poorer precision. The XRF handles the correction automatically, but the precision is affected. The loss of precision is also reported by the XRF. (Please refer to Innov-X Applications Sheet: *In-field Analysis of Lead and Arsenic in Soil Using Portable XRF* for more detail).

Example CASE 2: Bromine in the sample, but XRF is not calibrated for bromine. Bromine, as a fire retardant, is being seen more and more in soil and other sample types. For this reason, Innov-X analyzers include Br in the calibration data. If Br is not calibrated, but is present in the sample, the analyzer will report highly elevated levels of Pb, Hg and As. The levels will depend upon the concentration of Br in the sample.

Interferences between elements can be broadly categorized into a) Z, Z-1, Z+1 interferences, and b) K/L interferences. Interference type “a” occurs when high levels of an element of atomic number Z are present. This can cause elevated levels of elements with atomic number Z-1 or Z+1. Generally, portable XRFs have good correction methods, so this interference only causes problems with very high levels of the element in question. Example: High concentrations of Fe (Z=26) in excess of 10% may cause elevated levels of Mn or Co (Z=25 or Z=27 respectively).

The type “b” interference occurs when the L-shell line of one element overlaps with the K-shell spectral line of another element. The most common example is the lead/arsenic interference where the L-alpha line of lead is in nearly the exact same location as the K-alpha line of arsenic.



Appendix 3: Guide to Product Registration

Generally, the Innov-X portable XRF system must be registered in the state of usage. Registration requirements are somewhat state dependent, but there are many similarities. You may contact Innov-X at 866-4-Innov-X (781-938-5005) to receive specific registration information. Innov-X also maintains sample registrations for every state that we can forward to you.

Common Registration Features:

Most states require the following for registering an x-ray emitting device that does NOT use radioactive sources:

1. Registration within 30 days of receipt of the analyzer.
2. Annual fee ranging from \$25 to \$100, depending upon the state.
3. Basic registration form with main information described below.

Common information required on Registration Form, and responses:

Company name, address, phone/fax numbers.

Name of responsible person:	Generally the person designated as the Radiation Safety Officer (RSO).
Name of the manufacturer:	Innov-X Systems, Inc., Woburn, MA
Model of Analyzer:	Alpha XXXX
Tube Operating Parameters:	40 kV, 20 uA current.
Type of Analysis:	Choose Analytical or Industrial (as opposed to radiography, medical, dental, veterinarian, etc.)
Utilization Mode:	Portable or Mobile assuming you will carry system to different locations. Fixed or stationary ONLY if you will always use the analyzer in the docking station