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Date: April 6, 2007
Refer To: EP2007-0200

James P. Bearzi
Bureau Chief
Hazardous Waste Bureau
New Mexico Environment Department
2905 Rodeo Park Drive East, Building 1
Santa Fe, NM 87505-6303

Subject: Submittal of Addendum to the Work Plan for Sandia Canyon and Canada del Buey Work Plan, Revision 1

Dear Mr. Bearzi:

Enclosed please find two hard copies with electronic files of the "Addendum to the Work Plan for Sandia Canyon and Canada del Buey Work Plan, Revision 1." This revision is submitted in response to a requirement of the New Mexico Environment Department's (NMED's) approval with direction, dated March 5, 2007, of the Laboratory's initial submittal on January 30, 2007. All three changes required in NMED's approval with direction are addressed in the revision. An additional change that describes use of field x-ray fluorescence in the sediment investigation has also been incorporated, and the manufacturer's instructions for using the instrument are included as an attachment to this submittal.

If you have questions, please contact Danny Katzman at (505) 667-6333 (katzman@lanl.gov) or Mat Johansen at (505) 665-5046. (mjohansen@doeal.gov).

Sincerely,

Carolyn A. Mangeng, Acting Associate Director
Environmental Programs
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Sincerely,

George J. Rael, Assistant Manager
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Los Alamos Site Office

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Enclosure: Two hard copies with electronic files – "Addendum to the Work Plan for Sandia Canyon and Canada del Buey Work Plan, Revision 1" (EP2007-0200)

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Addendum to the Work Plan for Sandia Canyon and Cañada del Buey, Revision 1

Scope (New, modified, or ongoing activity)	Work Description	Rationale
Archival Work (ongoing)	A review of solid waste management units and areas of concern in Los Alamos, Sandia, and Mortandad canyons was conducted to identify the potential source(s) of chromium (including the mass associated with each source) as part of the "Interim Measures Investigation Report for Chromium Contamination in Groundwater" (LANL 2006, 094431). Additional review will further constrain the mass of chromium and volume of effluent released to these canyons. This review will also improve estimates of other contaminants released to Sandia Canyon, such as zinc, phosphate, molybdenum, and arsenic.	Knowledge of groundwater flow paths and operations at Los Alamos National Laboratory (LANL or the Laboratory) support a focus on Los Alamos, Sandia, and Mortandad canyons as the likely sources of chromium in the regional aquifer in and around R-11 and R-28. The likely sources are related to usage of chromate-containing compounds to inhibit corrosion in cooling-tower systems that discharged to each of the three watersheds. Lesser sources include electroplating and photo processing facilities. Information about sources and release histories will be incorporated into models evaluating fate and transport of chromium and other mobile constituents. Delineation of transport rates and flow paths within Sandia Canyon will be improved by tracking contaminants that were released concurrent with and following chromium releases. Model simulations will incorporate bounding estimates of released contaminant masses to quantify the level of resolution in mass estimates required to evaluate potential remedial options.
Sediment Investigation (modified)	The Laboratory proposes to modify the scope of the Sandia Canyon sediment investigation described in the work plan (LANL 1999, 064617) by (1) modifying the locations of Phase 1 investigation reaches, (2) modifying Phase 1 numbers of sample and analytical suites, adding supplemental analyses to evaluate chromium geochemistry, and (3) using field x-ray fluorescence (XRF) measurements to help in the selection of samples submitted for off-site analysis and to enhance the robustness of the nature and extent determination of chromium in sediments. These three actions are described below in detail: (1) The Laboratory proposes to add an additional upcanyon reach closer to the primary contaminant source ("S-3 West") and to eliminate two downcanyon reaches (S-4 Central and S-5 West) from the initial sampling phase. (2) The Laboratory proposes five changes to the Phase 1 sediment analyses, as presented in the work plan. First, chromium(VI),	The modification of the list of priority reaches specified in Table 7.1.2-1 of the work plan (LANL 1999, 064617, p. 7-9) will better focus work on evaluating the nature of chromium contamination. The new reach, S-3 West, will be roughly midway between reaches S-2 and S-3, in a portion of the canyon relatively close to the primary contaminant source that is expected to be important for understanding the chromium inventory in the Sandia watershed. The two reaches eliminated from this first phase of investigation, S-4 Central and S-5 West, are in an area of expected relatively low chromium contamination, based on sample data from reaches S-3 and S-5 Central collected in 2000. In addition, any contamination in S-4 Central and S-5 West will be adequately bounded by data from the nearby reaches, S-4 West, S-4 East, and S-5 Central. S-4 Central and S-5 West could be further investigated in later phases if such work is required. The modifications to the analyte suites and sample numbers will provide better Phase 1 characterization than presented in the work

Scope (New, modified, or ongoing activity)	Work Description	Rationale
	molybdenum, isotopic thorium, perchlorate, polycyclic aromatic hydrocarbons, and volatile organic compounds will be added to the analytical suite in all full-suite samples. Second analyses in S-3 West will be restricted to chromium and other metals, chromium(VI), molybdenum, and polychlorinated biphenyls as key contaminants. Analyses in the remaining Phase 1 reaches would include the full suite specified in the work plan. Third, approximately 40 samples will be collected in the wetland (reach S-2) for chromium and other metals to define the total chromium inventory better. Chromium(VI) analyses would also be obtained from these samples. Fourth, additional analyses will be conducted on approximately 20 samples to help evaluate the geochemical stability of chromium species. In addition to total chromium, chromium(VI), and other metals, the analyses will include iron(II), manganese(IV), phosphorus, nitrate, sulfate, sulfide, and total organic and inorganic carbon. Five, analyze approximately eight samples of cattails in reach S-2 for metals to evaluate potential biologic uptake. (3) The Laboratory will use a hand-held Innov-X XRF instrument to obtain field measurements of chromium concentration in sediment deposits in each reach prior to sampling. Measurements will be made for each stratigraphic interval at each geomorphic characterization location (stream banks, hand-dug holes, or hand-auger holes). These measurements will be used to determine variations in chromium concentration between different geomorphic units, sediment facies (fine-grained or coarse-grained sediment), and depth intervals. The XRF measurements will be used to provide preliminary estimates of chromium inventory in each reach and will be used to guide sampling. Samples selected for off-site analysis will include the location in each reach with the highest chromium concentration and also a range in concentration to provide representative average concentrations. The manufacturer's instruction manual is attached to this revision (Attachment 1).	plan, including: potential identification of a larger number of chemicals of potential concern; a better estimate of the inventory of total chromium, chromium(VI), and other analytes in sediment in the Sandia watershed; an improved evaluation of the geochemical stability of chromium; and an evaluation of the uptake of chromium and other metals into plants in the Sandia wetland. The results of field XRF measurements will provide a more robust determination of the spatial variations in chromium concentration and will help ensure that the locations with highest chromium concentration in each reach are sampled. Sampling for off-site analyses based on the field XRF data will also provide improved estimates of average chromium concentration and total chromium inventory in each reach.
Water Balance Investigation (new)	This activity will constrain the location and amount of surface water and alluvial groundwater loss in the narrow bedrock-dominated portion of Sandia Canyon between surface-water gaging stations E123 and	Areas of surface-water and alluvial-groundwater loss probably coincide with those parts of the canyon floor that contributed recharge to deeper perched zones and to the regional aquifer during and after

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	E123.5. The infiltration investigation conducted as part of the "Interim Measures Work Plan for Chromium Contamination in Groundwater" (LANL 2006, 091987) will be expanded by installing one or two new temporary stream gages, as allowed by the topography. Together with discharge data from outfalls and flow data from existing stream gages, the new stream gages will allow division of upper Sandia Canyon into three or four stream segments, providing better delineation of the areas of surface-water loss. Streambed infiltration losses will be determined by comparing surface-water flow between gages.	the period of chromium release from the Technical Area 3 (TA-03) power plant. A water-balance investigation, reported in the "Interim Measures Investigation Report for Chromium Contamination in Groundwater" (LANL 2006, 094431), showed that approximately 35% of the current TA-03 effluent volume to upper Sandia Canyon is lost along the approximately 3.5-km (2.2-mi) reach of canyon between the Sandia wetland and temporary gage E123.5. Further refinement of the water-loss estimates along this portion of Sandia Canyon will identify vadose-zone pathways and potential entry points for historical chromium contamination into the regional aquifer. These data will be applied in models of groundwater flow and chromium transport. These data will also help constrain the nature and extent of chromium and other contaminants.
Surface Water and Groundwater Sampling (modified)	Analytical suites for ongoing sampling conducted in Sandia Canyon, as described in the Interim Facility Groundwater Monitoring Plan (IFGMP), will be modified by adding stable isotopes of chromium (δ^{53}Cr) at the proposed locations. An initial sampling round will be conducted to determine if isotopic variations in δ^{53}Cr occur along surface water and groundwater flow paths within the Sandia and Mortandad watersheds. Samples from some of these locations may be at or below the lower concentration limit necessary for accurate and precise chromium isotope measurement, and/or the presence of high levels of organic matter may interfere with chromate extraction and analysis (Heikoop 2007, 095189). Based on results from the first round of sampling, some of these locations may be eliminated from future rounds of sampling if they are not suitable for analysis by this technique. In addition, variations in δ^{53}Cr for the initial round of data will be evaluated to determine if isotopic fractionation from the reduction of chromium(VI) to chromium(III) occurs along the surface water and groundwater flow paths. If evidence for isotopic fractionation is present, sampling will be conducted for three more quarters in the Sandia watershed and as approved in the next IFGMP for Mortandad Canyon.	Water-quality (geochemical) data obtained from one or more sampling rounds provide information about the occurrence and distribution of contaminants in surface and groundwater in the Sandia and Mortandad watersheds. Chemical and isotopic data will fingerprint potential contaminant sources and improve geochemical and hydrologic-transport numerical models that quantify processes controlling fate and transport of chromium and other contaminants of relevance (arsenic, molybdenum, and zinc). Ultimately, these data support the evaluation of potential remedial options. Analysis of δ^{53}Cr should enhance the geochemical conceptual model for chromium, including quantitative evaluation of chemical-biochemical reduction of chromium(VI) to chromium(III), followed by adsorption of chromium(III) onto ferric (oxy)hydroxide and/or precipitation of insoluble $Cr(OH)_3$. Analytical results for δ^{53}Cr are anticipated to provide quantitative information on the fate and transport and the natural attenuation of chromium in surface water and groundwater environments at the Laboratory.

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	Following completion of first phase of the sediment investigation, further consideration will be made to evaluate the potential usefulness of conducting chromium isotope analyses directly on sediment samples collected during the second phase. Stable chromium isotopes are proposed for addition to the current monitoring suites at the following locations: <u>Sandia Canyon</u> Surface Water – E121, E122, adjacent to SCA-1, E123, at new temporary gage station, and at E123.5 Alluvial Groundwater – SCA-1, SCA-3, SCA-5 Intermediate Groundwater – SCI-1, R-12 (screens 1 and 2) Regional Groundwater – R-11, R-10, R-10a, and R-35(a and b) when online <u>Mortandad Canyon</u> Surface Water – E1FW, E1E, E200 Alluvial Groundwater – MCO-0.6, MCO-2, MCO-4.5, MCO-5, MCO-7.5 Intermediate Groundwater – MCOBT-4.4, MCOI-5, MCOI-6 Regional Groundwater – R-1, R-15, R-13, R-28, R-33, R-14, R-34	
Fate and Transport Modeling (new)	This activity includes the development of a site-scale groundwater-flow and contaminant-transport model that incorporates historic chromium sources in Sandia, Mortandad, and Los Alamos watersheds, and existing water-supply wells in the vicinity (PM-3 and O-4). The model will include the vadose zone and the regional aquifer and will be based on all available hydrogeological data and supporting information, including the distribution of chemicals such as molybdenum and phosphate that replaced the use of chromium. The model will include detailed documentation of key input parameters and conceptual models and address uncertainties in the model results. The Laboratory will engage NMED in incorporating aspects of the conceptual model and plausible ranges and distributions for model input parameters. At a minimum, conceptual models to be considered	Numerical models are useful tools for integrating available knowledge about the fate and transport of chromium. Combining a quantitative treatment of all uncertainties with calibration and weighting of results based on their agreement with chromium measurements provides a defensible approach to providing quantitative answers to key questions such as: (1) What is the nature and extent of chromium? (2) What is the uncertainty in the nature and extent of chromium? (3) Is the current monitoring-well network capable of intercepting all potential chromium pathways? (4) What is the long-term fate of chromium in the vadose zone and regional aquifer? (5) What is the most effective and efficient remedial option for chromium? (6) What additional characterization is necessary to reduce model uncertainty sufficient to allow the selection of a remedial option?

Scope (New, modified, or ongoing activity)	Work Description	Rationale
	will include those recently suggested, such as (1) the fault system that crosses the wetland as a fast pathway for chromium transport; (2) a vadose zone pathway that includes down-dip lateral spreading of perched intermediate groundwater above perching horizons; and (3) spatial and temporal changes to infiltration in Sandia Canyon from past erosion of canyon-bottom alluvium. Model results will be compared to available monitoring data as well as to estimates of the total mass of chromium in the system. If necessary, simulation results will be weighted based on agreement between simulated and measured concentrations. The model results will include, but will not be limited to, maps showing the expected concentration of chromium, the probability that chromium concentrations exceed maximum contaminant levels, and chromium concentrations associated with confidence limits agreed upon by the Laboratory and NMED.	
R-28 Testing (new)	R-28 will be pumped for at least 30 hr at a constant rate of approximately 5 gal. per minute while water levels, chemical constituents, and other indicator parameters are measured. In addition, water levels will be monitored continuously in R-35(a and b), R-11, R-13, R-15, and R-8A during the test. The 30-hr test duration should produce water that is currently located outside the “zone of influence” of the potential residual drilling fluids. The Laboratory will work with NMED before the test to define a set of potential test responses, in terms of water levels, contaminant concentrations, and indicator parameters. These potential responses will be based on conceptual models of flow and transport such as one model assuming that the chromium concentrations are affected by drilling fluids and another model assuming they are not affected by drilling fluids. These hypothetical responses will seek to identify unique responses of each conceptual model and ensure test design and sampling protocols that can observe these unique responses.	This test will evaluate the effects of possible residual drilling fluid contamination on measured chromium concentrations, provide insight into the nature of the chromium plume, and provide information on aquifer parameters to be used in conceptual and numerical models of chromium fate and transport.

Scope (New, modified, or ongoing activity)	Work Description	Rationale
	Water samples will be collected at R-28 before pumping and during pumping at the following frequency: every hour during zero to 12 hr; every 4 hr from 12 to 24 hr; and at 30 hr at the end of pumping. Field parameters, including oxidation-reduction potential, temperature, pH, turbidity, specific conductance, and dissolved oxygen, will be measured during pumping and sampling. For all samples collected, filtered waters will be analyzed for metals and anions (including perchlorate), and nonfiltered waters will be analyzed for metals, total organic carbon, acetone, and 2-proponal (isopropyl alcohol). In addition, at the 4- and 6-hr intervals, filtered water samples will be analyzed for stable isotopes of nitrogen, and nonfiltered waters will be analyzed for low-level tritium (using electrolytic enrichment at the University of Miami) and stable isotopes of oxygen and hydrogen. To evaluate chromium reduction by potential residual drilling fluids, stable isotopes of chromium will be analyzed in samples collected at 0, 2, 4, 8, 12, 18, 24, and 30 hr. A field implementation plan (FIP) will be prepared to describe the details of the test at R-28. The FIP will include details of the test and the waste management plan for the purge water.	
PM-3 Zonal Sampling (new)	The Los Alamos County Utilities Department informed the Laboratory that access to PM-3 will not be granted until at least the fall of 2007.	Zonal sampling may provide insights into depth-dependant variations in the quality of water entering the well screen at PM-3. The Laboratory proposes that the need for zonal sampling at PM-3 be assessed after results for the R-35 and numerical modeling activities are evaluated.
Vadose Zone Boreholes (new)	NMED requested that one to two vadose-zone characterization core holes be installed approximately a mile upstream of SCC-1, if accessible. Field visits indicate that there are no accessible sites close enough to the stream channel in this portion of the canyon for installing core holes.	Proposed fate and transport modeling studies can evaluate different infiltration scenarios for this part of the canyon. In addition, geochemical data provided by sediment and water samples collected from the Sandia wetland, surface water, alluvial groundwater, intermediate groundwater, and regional groundwater will support evaluation of chromium migration pathways. The need for additional deep core hole investigations will be evaluated in the context of numerical modeling results.

REFERENCES

Heikoop, J., January 11 and 25, 2007. Chromium Isotopes. Personal communication between J. Heikoop and T. Johnson, Los Alamos, New Mexico. (Heikoop 2007, 095189)

LANL (Los Alamos National Laboratory), September 1999. “Work Plan for Sandia Canyon and Cañada del Buey,” Los Alamos National Laboratory document LA-UR-99-3610, Los Alamos, New Mexico. (LANL 1999, 064617)

LANL (Los Alamos National Laboratory), March 2006. “Interim Measures Work Plan for Chromium Contamination in Groundwater,” Los Alamos National Laboratory document LA-UR-06-1961, Los Alamos, New Mexico. (LANL 2006, 091987)

LANL (Los Alamos National Laboratory), November 2006. “Interim Measures Investigation Report for Chromium Contamination in Groundwater,” Los Alamos National Laboratory document LA-UR-06-8372, Los Alamos, New Mexico. (LANL 2006, 094431)

Attachment 1

Instruction Manual

INSTRUCTION MANUAL

INNOV-X SYSTEMS
ALPHA SERIES™
X-RAY FLUORESCENCE
SPECTROMETERS

August 2005
Version 2.1

Innov-X Systems, Inc.
100 Sylvan Road, Suite 100
Woburn, MA 01801

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Web-Site: www.innov-xsys.com
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INNOV-X SOIL QUICK START GUIDE

INNOV-X Systems, Inc.
10 Gill Street, Suite Q
Woburn, MA 01801

THIS INSTRUMENT PRODUCES IONIZING RADIATION.

Only Users with Manufacturer Authorized Training should operate INNOV-X Instruments
Before use, read and understand instruction manual
Review radiation safety– Chapter 3 in the Operator's Manual
Maintain Possession & Control of the Analyzer at all Times
Never Point the Instrument at Yourself or Others

Start-up preparation for new units:

- Charge main batteries for analyzer using Innov-X battery charger.
- Charge iPAQ. *The iPAQ consumes battery power even when it is turned off. If the instrument is not used for a period of many days, the iPAQ may discharge completely, or may not have enough power to run a test. If instrument will not be used for several days, charge the iPAQ during down time.*

Daily Start up procedure: ***(More detailed information can be found in Section 5.1 of the User's manual)***

1. Place a battery in the analyzer.
2. Power on the Analyzer Main Power (On/Off switch located on the back of the 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 Soil Mode.
7. 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.
9. When standardization is complete, remove the standardization clip. ***(Manual section 4.4 Standardization)***
10. Tap the "lock" icon in the lower right corner of the iPAQ screen to release software trigger lock
11. Test a soil standard to verify instrument performance.
12. Results will display on screen. Subsequent tests may be started from either Results or Analysis screens.

- If necessary, tap the X in the upper right hand corner of the Results screen to return to the "Ready to test" Analysis screen at any time.
- Test information can be entered from the analysis screen by tapping the Info button. ***(Manual 4.6)***
- Tests can be stopped by either pulling the trigger, or tapping Stop on the iPAQ screen.

HP IPAQ Pocket PC® Basics:

- Start menu is found in the upper left corner of screen. If "Start" icon is not visible, tap the screen in upper left corner.
- Text is entered by tapping the keyboard icon in lower right to bring up the Virtual Keyboard or Character recognizer.
- The File Toolbar is located at the bottom of the screen.
- Pressing the right most button below the screen provides a list of applications currently running
- To Reset the iPAQ, stick the tip of the stylus into the small hole located next to the connector.

- Innov-X software uses three main screens: Main, Analysis and Results: Users select mode to use via the **Main Screen**. Most edits to testing method are made via the **Analysis** screen, which typically says "Ready to test." The **Results** screen shows all data. All data editing features are done from **here.**
- **Always exit software completely when finished using analyzer!** It may be necessary to close all three screens. Use File → Exit. Software will prompt for confirmation before closing.
- **Removing iPAQ:** Slide iPAQ towards nose of analyzer to clear connector before lifting upwards.

Sample Preparation Considerations:

- The degree of sample preparation will largely determine the accuracy obtained when using portable XRF!
- EPA Method 6200 for portable XRF is written for in-situ testing, as opposed to prepared-sample testing.
- Sample Preparation will depend on the goal of the analysis. For **Quantitative Field Screening**, it is often sufficient to analyze samples in plastic bags, or directly on the ground – *in-situ testing*. *In-situ testing* provides a high number of data points quickly in order to profile a site rapidly and find "hot spots."
- For more quantitative results, or for direct comparison to a laboratory analytical method such as AA or ICP, samples should be dried, homogenized and prepared in sample cups. For the best possible comparisons, it is best to have the laboratory analyze the same samples analyzed with the XRF.
- Sample prep recommendations can be found in EPA Method 6200, Pg. 8 "Sample Preparation."
- For laboratory analysis, be sure to request a total digestion method.

Exporting data:

Exported data is saved as comma delimited (.csv) files. These files can be opened and formatted in MS Excel, or any other spreadsheet program. To transfer data to a PC, ActiveSync must be installed on the PC.

From Results Screen

1. Choose File → Export Readings
2. Select readings to be exported. Tap OK.
3. Input Filename, folder, tap OK
4. The results file will be stored in the My Documents Folder on the iPAQ. It can be copied to a PC using ActiveSync. If ActiveSync is set up to automatically synchronize files, this file will be copied when the iPAQ is connected to the PC.

Erasing data:

The software loads stored data when first result is taken in each mode after software is started. As more readings are stored, the analyzer will take longer to display the results of the first reading. If this period of time becomes too long, erase the data stored in memory.

From Results Screen:

1. Select File →Erase Readings
2. Enter administrative password
3. Select readings to be deleted and tap OK.
4. Select "Yes" to confirm deletion.

Manual: Section 4.7.2 Erasing readings

Changing Test Settings from Analysis screen:

Test time: Select Options →Setup Testing

- Min Time must elapse in a reading for results to be reported on screen. Tests stopped before Min Time will be considered to be aborted tests.
- Max Test time is maximum length of test if a manual test end condition is selected.

Turn live updates on/off: Select View → Live Updates

Soil mode options and test end conditions
Are described in section 5.4 of the users manual

Test end conditions:

Select Options → Soil Mode Testing Setup

- Manual: Test ends when test is stopped
- Maximum Time: Test ends when Max time has elapsed
- Rel. Std. Dev.: Must choose element and standard deviation. Test ends when error is within specified limits
- Action Level: Must choose element and desired action level. Test ends when analyzer can determine if elements concentration is above or below action level.

Table 1. Certified Values

Element	Mass Fraction (%)			Element	Mass Fraction (mg/kg)		
Aluminum	6.44	±	0.08	Antimony	38.4	±	3
Calcium	1.25	±	0.03	Arsenic	626	±	38
Iron	3.38	±	0.10	Barium	707	±	51
Magnesium	0.853	±	0.042	Cadmium	21.8	±	0.2
Manganese	1.01	±	0.04	Copper	2950	±	130
Phosphorus	0.106	±	0.015	Lead	5532	±	80
Potassium	2.11	±	0.11	Mercury	32.6	±	1.8
Silicon	28.97	±	0.18	Nickel	14.3	±	1.0
Sodium	1.14	±	0.06	Silver	35.3	±	1.5
Sulfur	0.240	±	0.006	Vanadium	76.6	±	2.3
Titanium	0.283	±	0.010	Zinc	6952	±	91

Noncertified Values: Noncertified values shown below are provided for information only. An element concentration value is not certified if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available.

Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Carbon	3	Bromine	6
		Cerium	57
		Cesium	107
		Chromium	39
		Cobalt	10
		Dysprosium	5.4
		Europium	1
		Gallium	34
		Gold	0.6
		Hafnium	3.2
		Holmium	0.6
		Indium	5.1
		Lanthanum	34
		Molybdenum	19
		Neodymium	23
		Rubidium	120
		Samarium	7.8
		Scandium	8.7
		Strontium	330
		Thallium	1.3
		Thorium	13
		Tungsten	93
		Uranium	25
		Ytterbium	1.3
		Yttrium	23

Table 1. Certified Values

Element	Mass Fraction (%)			Element	Mass Fraction (µg/g)		
Aluminum	6.53	±	0.09	Antimony	19.4	±	1.8
Calcium	2.88	±	0.08	Arsenic	105	±	8
Iron	2.89	±	0.06	Barium	726	±	38
Magnesium	1.05	±	0.03	Cadmium	41.70	±	0.25
Phosphorus	0.086	±	0.007	Copper	114	±	2
Potassium	2.45	±	0.08	Lead	1162	±	31
Silicon	30.44	±	0.19	Manganese	638	±	28
Sodium	1.14	±	0.03	Mercury	6.25	±	0.19
Sulfur	0.042	±	0.001	Nickel	20.6	±	1.1
Titanium	0.306	±	0.023	Selenium	1.52	±	0.14
				Silver	4.63	±	0.39
				Strontium	245.3	±	0.7
				Thallium	2.47	±	0.15
				Vanadium	81.6	±	2.9
				Zinc	350.4	±	4.8

Noncertified Values: Noncertified values, shown below, are provided for information only. An element concentration value may not be certified, if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available.

Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (µg/g)
Carbon	2	Bromine	5
		Cerium	69
		Cesium	6.1
		Chromium	47
		Cobalt	10
		Dysprosium	5.6
		Europium	1.1
		Gallium	15
		Gold	.03
		Hafnium	7.3
		Holmium	1
		Indium	1.1
		Iodine	3
		Lanthanum	40
		Molybdenum	1.6
		Neodymium	31
		Rubidium	110
		Samarium	5.9
		Scandium	9
		Thorium	14
		Tungsten	3
		Uranium	2.6
		Ytterbium	2.7
		Yttrium	25
		Zirconium	230

Table 1. Certified Values

Element	Mass Fraction (%)			Element	Mass Fraction (µg/g)		
Aluminum	7.50	±	0.06	Antimony	7.9	±	0.6
Calcium	1.89	±	0.05	Arsenic	17.7	±	0.8
Iron	3.50	±	0.11	Barium	968	±	40
Magnesium	1.51	±	0.05	Cadmium	0.38	±	0.01
Phosphorus	0.062	±	0.005	Chromium	130	±	4
Potassium	2.03	±	0.06	Cobalt	13.4	±	0.7
Silicon	29.66	±	0.23	Copper	34.6	±	0.7
Sodium	1.16	±	0.03	Lead	18.9	±	0.5
Sulfur	0.089	±	0.002	Manganese	538	±	17
Titanium	0.342	±	0.024	Mercury	1.40	±	0.08
				Nickel	88	±	5
				Selenium	1.57	±	0.08
				Silver	0.41	±	0.03
				Strontium	231	±	2
				Thallium	0.74	±	0.05
				Vanadium	112	±	5
				Zinc	106	±	3

Noncertified Values: Noncertified values, shown below, are provided for information only. An element concentration value may not be certified if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available.

Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (µg/g)
Carbon	1.2	Cerium	42
		Cesium	5.3
		Dysprosium	3.5
		Europium	0.9
		Gallium	14
		Gold	0.3
		Hafnium	3.7
		Holmium	0.54
		Iodine	5
		Lanthanum	23
		Molybdenum	2.0
		Neodymium	19
		Rubidium	96
		Samarium	3.8
		Scandium	12
		Thorium	11
		Tungsten	2
		Uranium	3
		Ytterbium	1.6
		Yttrium	18
		Zirconium	160

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TABLE OF CONTENTS

Topic	Page
Chapter 1: INTRODUCTION	
1.0 Inspecting the Analyzer	1-1
1.1 Components Included with the Analyzer	1-1
1.2 Quick Start Instructions	1-2
1.3 Introduction to XRF	1-3
Chapter 2: USAGE AND ASSEMBLY OF ACCESSORIES	
2.0 Accessories	2-1
2.1 Analyzer Battery	2-1
2.2 Changing a Battery	2-1
2.3 Battery Charger	2-2
2.4 HP iPAQ Battery	2-3
2.5 Removing the iPAQ from Analyzer	2-3
2.6 Standardization Cap and/or Weld Analysis Mask	2-4
2.7 Testing Stand (optional accessory)	2-5
Chapter 3: RADIATION SAFETY	
3.0 Important Safety Information	3-1
3.1 General Safety Precautions	3-1
3.2 Recommended Radiation Safety Training Components	3-2
3.3 Innov-X Safety Features	3-3
3.4 Performing a Test Following Appropriate Radiation Safety Procedures	3-3
3.5 Correct and Incorrect Instrument Usage	3-4
3.6 Radiation Warning Lights and Labeling	3-7
3.6.1 Main Power Switch and Indicator Light	
3.6.2 Probe Light and Probe Label	
3.6.3 Testing Light on Back of Analyzer	
3.6.4 Label Behind iPAQ	
3.7 Radiation Levels from Analyzer	3-8
3.8 Radiation Doses for Several Scenarios	3-9
3.9 Common Questions and Answers Regarding Radiation Safety	3-12
3.10 Safeguards and Emergency Response	3-13
3.11 Dosimeter Badges	3-14
3.12 Typical Registration Requirements	3-14
Chapter 4: OPERATION	
4.0 Operation - General	4-1
4.1 Working with HP iPAQ Pocket PC®	4-1
4.2 Operation - Main Software Screens	4-1
4.2.1 Innov-X Main Menu	
4.2.2 The Analysis Screen	
4.2.3 The Results Screen	
4.3 Passwords	4-3
4.4 Standardization	4-3
4.4.1 Standardization Procedure	
4.4.2 Standardization Errors	
4.4.3 Battery Replacement and Initialization/Standardization	
4.5 Software Trigger Lock	4-7
4.6 Test Information—Label Input	4-8
4.6.1 Modifying the Test Info Template	

4.6.2 Entering Test Information
4.6.3 Editing Test Info from the Results Screen
4.7 Exporting and Erasing Data 4-14
4.7.1 Installing ActiveSync
4.7.2 Exporting Results
4.7.3 Customizing Results Export
4.7.4 Exporting Spectra
4.7.5 Erasing Readings

Chapter 5: ALLOY ANALYSIS

5.0 Alloy Analysis Introduction 5A-1
5.1 Alloy Analysis – Starting the Instrument and Taking a Measurement Using the Standard Library 5A-1
5.2 Selecting a Library 5A-6
5.3 Setting the Analysis Time 5A-7
5.4 Live Update Display 5A-8
5.5 Sample Considerations 5A-9
5.5.1 Coated or Painted Samples
5.5.2 Mixed Samples, Heterogeneous Materials
5.5.3 Small and Irregularly Shaped Samples
5.5.4 Invisible Elements

Chapter 6: ALLOY ANALYSIS—FASTID MODE

6.0 Introduction to FastID Mode 6A-1
6.1 Analyzing a Sample 6A-1
6.1.1 Troubleshooting
6.2 Results Display 6A-2
6.2.1 Results Screen
6.2.2 Chemistry Screen
6.2.3 Spectrum Screen
6.2.4 Test Info Screen
6.3 Advanced Features 6A-5
6.3.1 Specific Regions
6.3.2 Modifying the Fingerprint Cutoff

Chapter 7: ALLOY ANALYSIS—PASS/FAIL MODE

7.0 Introduction to Pass/Fail Mode 7A-1
7.1 Using Pass/Fail 7A-1
7.1.1. Selecting a Reference Standard
7.1.2. Select Pass/Fail Method
7.2 Analyzing a Sample 7A-4
7.2.1 Use Fingerprint
7.2.2.Use Chemistry
7.3 Advanced Features 7A-6

Chapter 8: ALLOY ANALYSIS—ANALYTICAL MODE
8.0 Introduction to Analytical Mode 8A-1
8.1 Using Analytical Mode 8A-1
8.2 Results Display 8A-2
8.2.1 Results Screen
8.2.2 Chemistry Screen
8.2.3 Spectrum Screen
8.2.4 Test Info Screen

8.3 Advanced Features 8A-6
8.3.1 Nearest Match/Exact Match
8.3.2 *Smartbeam* (optional feature)
8.3.3 Light Element Analysis

Chapter 9: ALLOY ANALYSIS—LIBRARIES
9.0 Libraries 9A-1
9.1 Selecting a Library 9A-1
9.2 Editing Library Functions 9A-2
9.3 Changing Library Names 9A-3
9.4 Changing the Names of Individual Fingerprints or Grade Entries 9A-4
9.5 Deleting a Grade or a Fingerprint 9A-6
9.6 Adding a Grade 9A-7
9.7 Analytical Mode – Editing a Grade 9A-10
9.8 Adding a Fingerprint 9A-12
9.9 Editing Fingerprint Libraries 9A-16

Chapter 5S: SOIL ANALYSIS
5.0 Check Standards 5S-1
5.1 Sample Presentation 5S-1
5.2 Testing in Soil Mode 5S-1
5.3 Soil Results Screen 5S-2
5.3.1 Results View Menu
5.3.2 Spectrum Screen
5.3.3 Test Info Screen
5.4 Soil Mode Options 5S-3
5.4.1 Set Testing Times
5.4.2 Soil Mode Test End Condition
5.5 LEAP Mode (Light Element Analysis Program) 5S-6
5.5.1 LEAP Settings
5.5.2 Testing Times

Chapter 6E Dust Wipe analysis
6.1 Sample Preparation 6E-1
6.2 Positioning the Sample 6E-2
6.3 Testing in Dust Wipe Mode 6E-3
6.4 Dust Wipe Results Mode 6E-4
6.4 Dust Wipe Mode Options 6E-7

Chapter 7E Filter and Thin Film Analysis
7.1 Testing in Filter Mode 7E-1
7.2 Filter Results Screen 7E-3
7.3 Filter Mode Options 7E-6

Appendix 1A: Reference Standards in Factory-Installed Fingerprint Library

Appendix 2A: List of Alloys in Factory-Installed Grade Library

Appendix 3A: Troubleshooting

Appendix 4A: X-ray Registration Information

Appendix 1S: Troubleshooting Guide—Soil Analysis

Appendix 2S Metals in Soil Analysis Using Field Portable X-Ray Fluorescence

Chapter 1 Introduction

1.0 INSPECTING YOUR INNOV-X ANALYZER

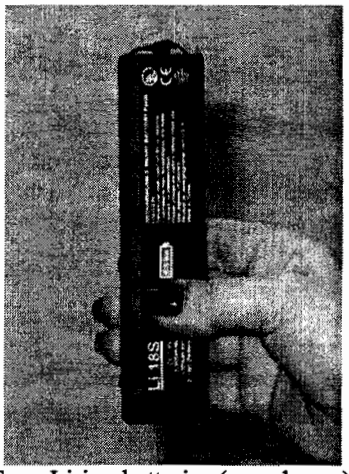
Upon receipt:

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.

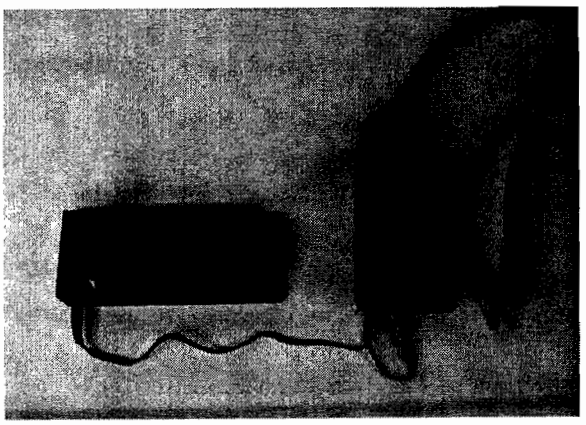
1.1 COMPONENTS INCLUDED WITH THE ANALYZER

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

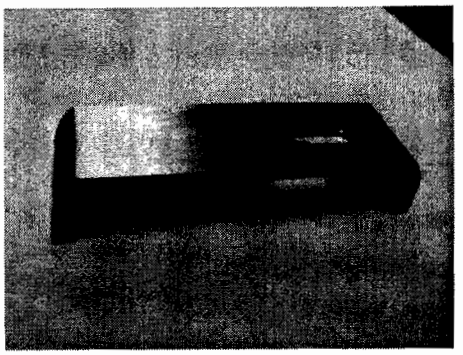
Analyzer, with iPAQ attached.



Two, Li-ion batteries (one shown).

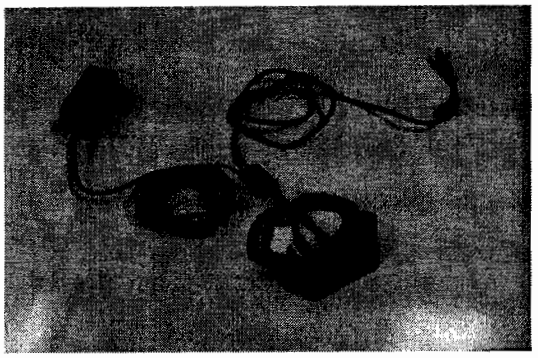


Battery charger and an AC adaptor. Battery shown mounted in charging system.

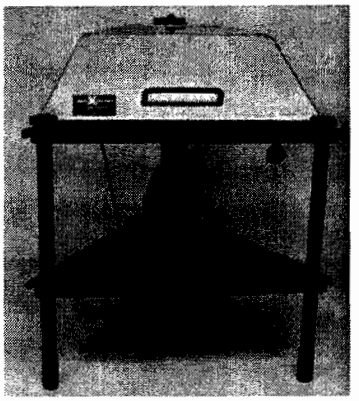


Standardization cap and weld mask (optional)

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.

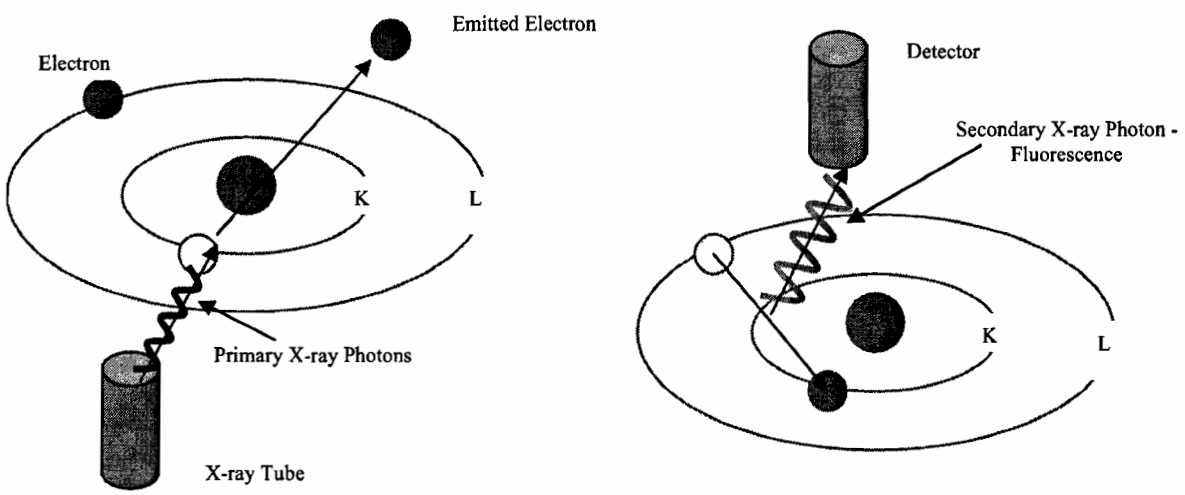
1.3 INTRODUCTION TO XRF: X-RAY FLUORESCENCE SPECTROMETRY OVERVIEW

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.



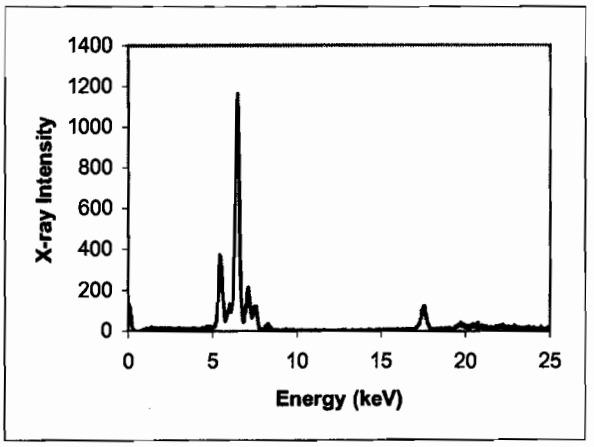
In XRF Spectrometry, high-energy primary X-ray photons are emitted from a source (X-ray tube) and strike the sample. The primary photons from the X-ray tube have enough energy to knock electrons out of the innermost, K or L, orbitals. When this occurs, the atoms become ions, which are unstable. Electrons 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.

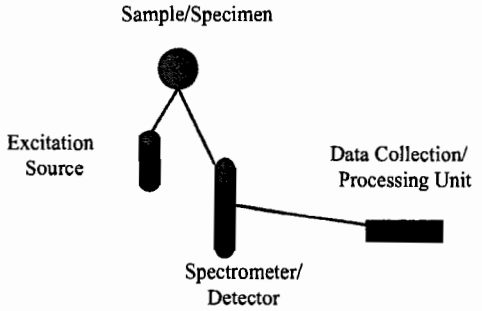
XRF Spectrometry easily and quickly identifies and quantifies elements over a wide dynamic concentration range, from PPM levels up to virtually 100% by weight. XRF Spectrometry does not destroy the sample and requires little, if any, specimen preparation. It has a very fast overall sample turnaround 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.

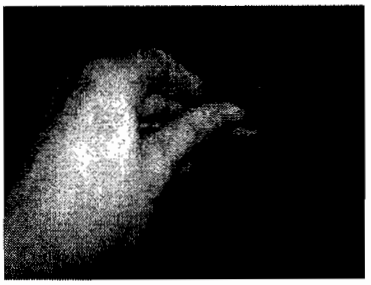


Figure 2.2a. Instrument handle. Pull the rubber latch and lift door. Reach into opening and remove battery.

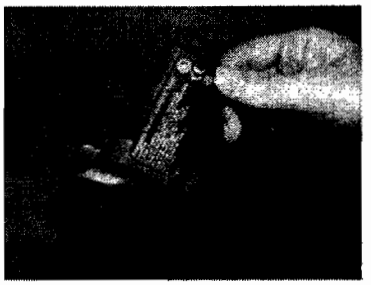


Figure 2.2b Insert new battery into opening.

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.

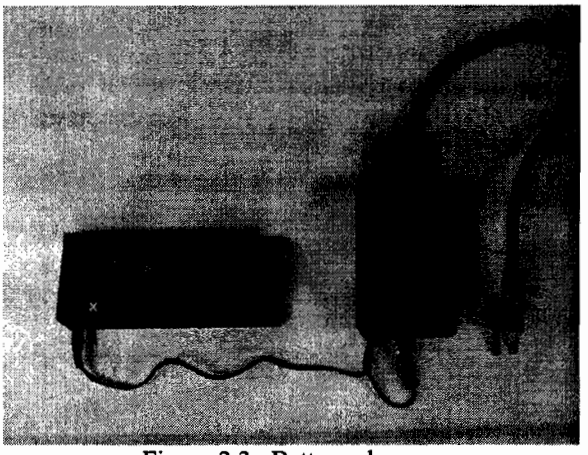


Figure 2.3. Battery charger.

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.

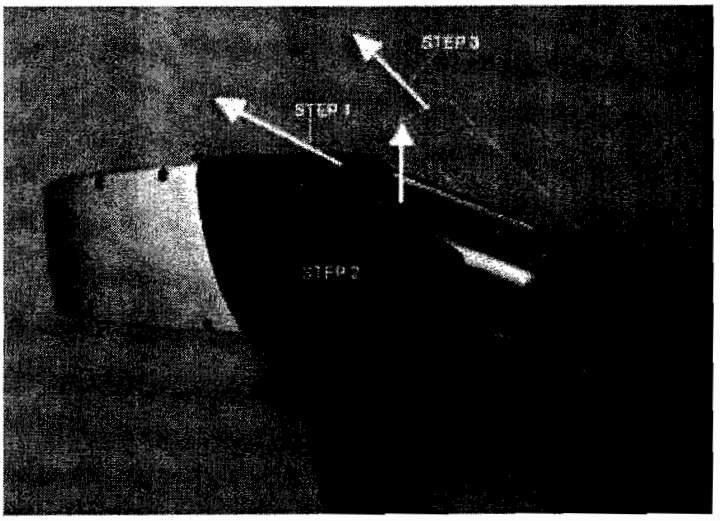


Figure 2.4. Removing the iPAQ from the analyzer.

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 ¼" 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 is 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.

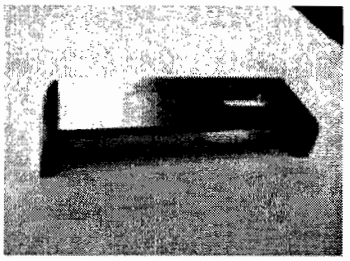


Figure 2.5 Standardization cap and welding mask. (Optional accessory)
The standard standardization cap does not have the welding slit.

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:

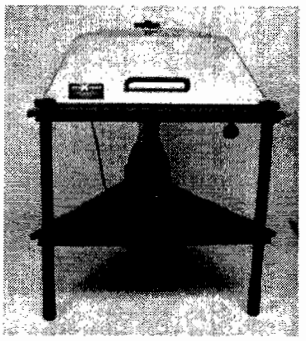


Figure 2.6. Assembled Testing Stand

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).

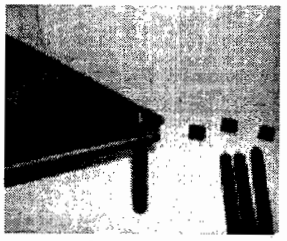


Figure 2.7. Mounting Lower Stand onto Short Legs.

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).

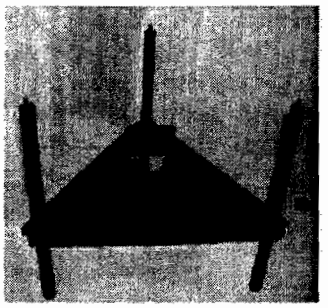


Figure 2.8. Mounting Long Legs onto Lower Stand and inserting analyzer.

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).

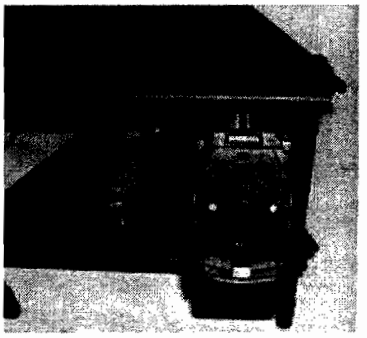


Figure 2.9. Mounting Upper Stand onto Testing Stand.

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.

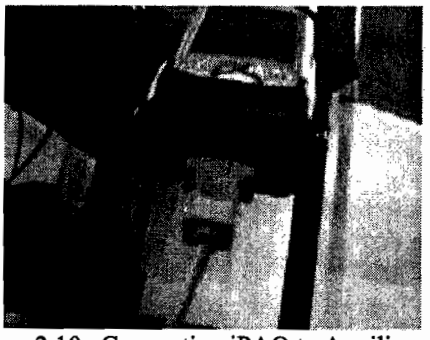


Figure 2.10. Connecting iPAQ to Auxiliary Port on analyzer.

Chapter 3 Safety Information

3.0 IMPORTANT SAFETY INFORMATION

THE XRF SHOULD NOT BE POINTED AT ANYONE OR ANY BODY PART, ENERGIZED OR DE-ENERGIZED! The safe and proper operation of the Innov-X XRF instruments is the highest priority. These instruments produce ionizing radiation and should **ONLY** be operated by individuals, who have been trained by Innov-X Systems, Inc. and received a manufacturer's training certificate. Innov-X recommends that operators and companies implement a written Radiation Safety Program, with safety components specific to the site and application of use of the instrument. The Radiation Safety Program should be reviewed annually and revised appropriately by a competent individual.

Innov-X analyzers must be used by trained operators, according to the instructions presented in this manual. Improper usage may circumvent safety protections and could potentially cause harm to the user. Pay attention to all warning labels and messages.

Important Notice for all Canadian Users:

Canadian Federal Regulations (Radiation Emitting Devices Act) require that all Canadian users must be certified according to NRC Standard CAN/CGSB-48.97/2-2000 in order to use this device.

For this certification contact: Natural Resources Canada, Manager Nondestructive Testing Certification, CANMET, 568 Booth St., Ottawa, ON, K1A 0G1; Tele: (613) 943-0583; Fax(613) 943-8297.

Users are advised to contact their appropriate federal/provincial./territorial radiation protection agency for applicable rules of operation.

The Innov-X analyzer is a very safe instrument when used according to manufacturer's recommended safety procedures as detailed in this chapter.

Radiation levels during testing are < 0.1 mR/hr on all surfaces of the analyzer except at or near the exit port for the radiation. This means that if an operator follows standard operating procedures, they will not obtain any detectable radiation dose above naturally occurring background radiation, on their hand while holding the analyzer, or on any area of their body.

This chapter details specifics of the radiation levels. It covers both standard (safe) and un-safe methods of operation, it provides radiation emission information, and also provides dose estimates for unsafe operations.

3.1 GENERAL SAFETY PRECAUTIONS AND INFORMATION:

Retain and follow all product safety and operating instructions. Observe all warnings on the product and in the operating instructions. To reduce the risk of bodily injury, electric shock, fire and damage to the equipment, observe the following precautions:

Heed service markings. Except as explained in this documentation, do not service any Innov-X product yourself. Opening or removing covers may expose you to electric shock. Service needed on components inside these compartments should be done only by Innov-X Systems, INC.

Damage requiring service:

- The power cord, plug or battery contacts for the battery charger are damaged.

- Liquid has been spilled or an object has fallen onto the instrument.
- The instrument has been exposed to rain or water.
- The instrument has been dropped or damaged.
- There are noticeable signs of overheating.
- The instrument does not operate normally when you follow operating instructions.

Safety Precautions:

Use the correct external power source: Ensure that the voltage is appropriate (100V-240 V/ 50-60 Hz) for charging the battery packs. Do not overload an electrical outlet, power strip, or convenience receptacle. The overall load should not exceed 80% of the branch circuit rating.

Use cables and power cords properly:
Plug the battery charger into a grounded electrical outlet that is easily accessible at all times. Do not pull on cords and cables. When unplugging the cord form the electrical outlet, grasp and pull the cord by the plug.

Handle battery packs properly; do not: disassemble, crush, puncture, short external contacts, dispose of in fire or water, or expose a battery pack to temperatures higher than 60 °C (140 °F).Do not attempt to open or service a battery pack.

WARNING: Danger of explosion if battery is incorrectly substituted. Replace only with Innov-X specified batteries. Used batteries may be returned to Innov-X Systems for disposal.

3.2 INNOV-X SYSTEMS – RECOMMENDED RADIATION SAFETY TRAINING COMPONENTS

Individual Companies and States have specific regulations and guidelines for the use of X-ray tube generated ionizing radiation. The purpose of the recommendations below is to provide generic guidance for an ALARA - best practice - approach to radiation safety. These recommendations do not replace the requirement to understand and comply with the specific policies of any state or organization.

1. **Proper Usage.** Never point the instrument at another person. Never point the instrument into the air and perform a test. Never hold a sample in your hand and test that part of the sample.
2. **Establish Controlled Areas.** The location of storage and use should be of restricted access to limit potential exposure to ionizing radiation. In use, the target should not be hand held and the area at least three paces beyond the target should be unoccupied.
3. **Specific Controls.** The instrument should be stored, in a locked case, or locked cabinets when not in use. When in use, it must remain in the direct control of a factory trained, certified operator.
4. **Time - Distance - Shielding Policies**. Operators should minimize the time around the energized instrument, maximize the distance from the instrument window, and shoot into high density materials whenever possible. Under no circumstances should the operator point the instrument at themselves or others.
5. **Prevent Exposure to Ionizing Radiation**. - All reasonable measures, including labeling, operator training and certification, and the concepts of time, distance, & shielding, should be implemented to limit radiation exposure to ***as low as reasonably achievable*** (ALARA).
6. **Personal Monitoring.** Radiation control regulations may require implementation of a radiation monitoring program, where each instrument operator wears a film badge or TLD detector for an initial period of 1 year to establish a baseline exposure record. Continuing radiation monitoring after this

period is recommended, but may be discontinued if accepted by radiation control regulators. Please refer to Sect. 3.10 for a list of providers of film badges.

3.3 INNOV-X SAFETY FEATURES

The Innov-X analyzer is very safe when used correctly, however the analyzer does emit radiation through the analyzer window, and all precautions must be taken to reduce exposure to this radiation. In order to minimize the possibility of accidental exposure, the following safety features are standard in all Innov-X analyzers.

1. "Deadman" trigger. The trigger must be held for the duration of the test. This requires that the user consciously depress the trigger whenever x-rays are emitted, and ensures that the analyzer is attended at all times while x-rays are emitted.

   Upon completion of safety training, an INNOV-X certified trainer may deactivate this feature upon request. The deactivation of the trigger is recommended only if long tests are required (such as for soil mode) and if the unit is used primarily by only 1 or 2 users who utilize it frequently, in a very controlled environment. In situations where multiple users are sharing the unit, it is recommended that the deadman trigger remain active.

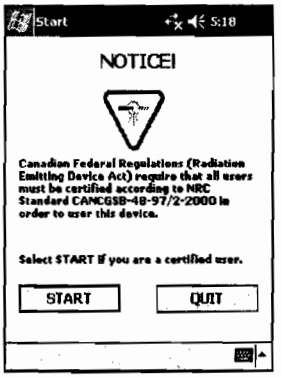
   Note: Canadian Regulations <u>require</u> that the deadman trigger be used at all times. This feature will not be disabled for usage in Canada.

2. Software Trigger lock. Before using the trigger, the user must tap on a lock icon located in the lower right hand corner of the iPAQ screen. The user must then confirm that they wish to unlock the trigger. If the instrument is used continuously, the software trigger lock will remain off. If five minutes elapse between tests, the trigger will lock automatically.

3. Software Proximity sensor. The software requires that a sample be present in front of the analyzing window. This prevents the accidental exposure of bystanders to an open beam. If the analyzer detects that a sample is not present, it will abort the test and shut off x-rays two seconds after the test is started.

3.4 PERFORMING A TEST FOLLOWING APPROPRIATE RADIATION SAFETY PROCEDURES

Starting the Analyzer:



When an operator opens the Innov-X software on the iPAQ, he or she will be presented with one of the displays shown to the right. Provided an operator has received training from an authorized Innov-X trainer, he/she should tap the START button to begin using the analyzer.

In Canada, INNOV-X ANALYZERS MUST BE OPERATED BY CERTIFIED USERS ONLY!

From this point the operator is presented with the main menu of the analyzer to choose an operating mode and begin testing (described in Chapter 4). The remainder of this section is dedicated to operational and safety aspects that pertain to safe use and storage of the analyzer.

Starting a test using the trigger.

When the trigger is depressed, the analyzer supplies power to the x-ray tube and opens the shutter to emit x-rays.

If deadman trigger is enabled, the trigger must be depressed for the duration of the test. Releasing the trigger will close the shutter and immediately end the test. If deadman trigger is disabled, pulling the trigger once will start a test, pulling it again will stop it.

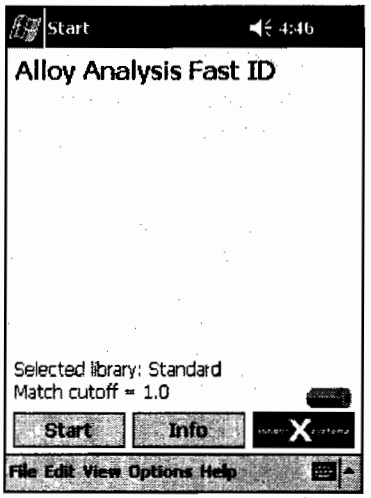
Figure 3.1 Handle of analyzer. Trigger is located at top of handle.

Starting a Test Using the "Start" Icon on the iPAQ Screen

This feature is disabled in all units shipped. It will become active only if the "deadman" trigger is disabled.

An operator may also begin a test by pressing the **Start** button on the touch screen, as shown at the right. The **Start** button, rather than the trigger, is generally used when the analyzer is docked into the testing stand.

This Feature is not available in Canada. All tests must be started via the trigger.



3.5 CORRECT AND INCORRECT INSTRUMENT USAGE:

The Innov-X XRF analyzer can be used in several different testing configurations. Safety guidelines are described for each configuration.

Configuration 1: Usage as a Handheld Alloy Analyzer:

In this configuration the analyzer is held in the hand, placed on various types of samples and a test is performed. Samples include pipes, valves, large pieces of scrap metal, basically any sample large enough to be tested in place, rather than held in the operator's hand. Point the instrument at a metal sample such that no part of your body including hands and/or fingers is near the aperture of the analyzer where x-rays are emitted.

Using the analyzer in this manner assures that the operator will not obtain a radiation dose to any body part or extremity in excess of naturally occurring background radiation. The radiation at any surface of the analyzer is < 0.1 mR/hr except at the exit port and the immediate area around the exit port.

The user should take care that personnel are not located within 3' (1 m) of the front end of the analyzer during testing, in the direction of the x-ray beam. Provided the analysis window is completely covered, there is virtually no radiation being emitted around the area of the sample. However, if a small component or curved surface is being analyzed, some radiation will be detectable.

Configuration 2: Usage in the Testing Stand

Innov-X strongly recommends that testing small pieces or small samples (rod, fasteners, turnings, XRF sample cups, bagged samples, etc.) be analyzed using the Innov-X Testing Stand. This allows the sample to be placed onto the analysis window of the analyzer without requiring the sample to be held by the operator. See figure below titled "Testing Stand Operation."

Note for Canadian Usage: The testing stand is not available for use in Canada at this time because it has not received regulatory approval yet. When an interlocked version of the testing stand has received regulatory approval, it will be available for sale into Canada. Please contact Innov-X Systems for an update on this process at 781-938-5005.

Figure 3.2 Testing Stand Operation. Please refer to **Section 2.7: Testing Stand** for assembly instructions.

Warning: Innov-X strongly recommends that operators do NOT hold samples in their hand for testing. Never hold a small sample in your hand, and test that sample, such that your hand is exposed to the x-ray beam being emitted from the analyzer. This type of testing produces a small but non-negligible radiation dose to the operator's hand. Please see **Section 3.7: Radiation Doses for Several Scenarios** for dose levels. Also, see **Figure 3.4** for an example of incorrect usage.

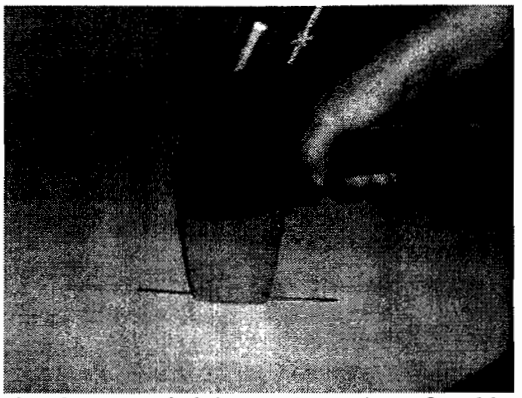
Figure 3.2.

Testing of Small Components:

Operators often are required to test small components, particularly in the field of alloy analysis. Examples of small samples include turnings, weld rod, wires, fasteners, nuts and/or bolts.

There are specific procedures to test small components. These procedures should be followed at all times. **Never hold a small part with your fingers or in the palm of your hand and perform a test. Doing so may deliver a significant dose of radiation to your fingers or hand.** Please refer to the Examples of Mis-use below.

Method 1: Testing a sample lying on a flat surface.



Figures 3.2.: Performing a testing for a sample lying on the surface of a table. This is a good way to test small samples, rather than holding them in your hand.

To analyzer small sample:

- Place the sample onto a flat surface.
- Place the window of the analyzer onto the sample and begin the test.

Safety Precautions:

Do not test samples in this manner at a desk or table where the operator is sitting. If the desk is made of wood or another non-metallic material, some radiation will penetrate the desk and may provide exposure to legs or feet if the operator is sitting at the desk or table.

Analytical Precautions:

If the sample does not completely cover the window, be sure the surface used does not contain metals or even trace levels of metals, as this may affect the accuracy of the XRF result. The XRF may report the presence of additional metals in the surface material. For this type of testing, it is good to place the sample onto a piece of 1100series aluminum alloy and perform the analysis. The operator should disable the aluminum analysis capability (See Section 8.3.3 in the manual for instructions).

Method 2: Use the testing stand as described above (see also Fig. 3.2).

Examples of Incorrect and Possible Unsafe Operation:

Improper Operation, DO NOT TEST SAMPLES LIKE THIS:

Exposure to the operator's hand/fingers will likely be minimal for this type of a testing, because the operator's hands and fingers are not in the primary beam. However, Innov-X believes that this type of the analyzer sets a poor safety precedent in that any operation where the operator places their fingers or hands near the window should not be permitted.

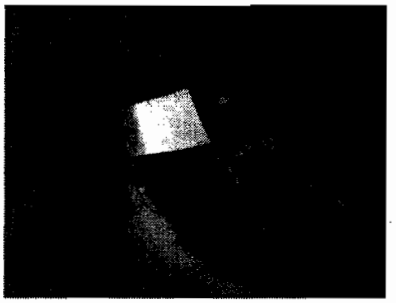


Figure 3.3. Incorrect Usage. While the dose to the operator's fingers/hand is negligible, testing this way sets a poor safety example for other operators, possibly encouraging other unsafe usage. Innov-X strongly recommends against this type of testing.

DO NOT TEST SAMPLES LIKE THIS:
Never hold a sample in your hand such that any part of your body or appendages are exposed to the x-ray beam. Testing samples in this way may generate significant radiation exposure (up to 27 R/hr) to the operator's fingers.

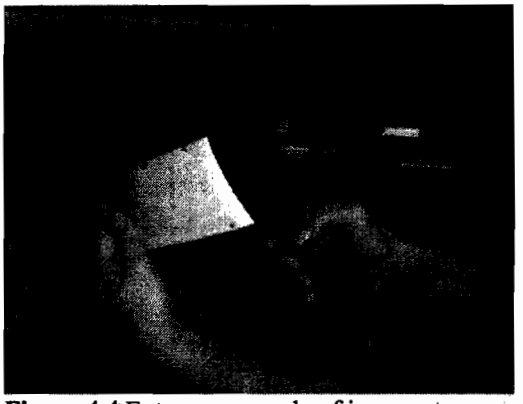


Figure 4.4 Extreme example of incorrect usage. An operator should NEVER hold small samples by hand

3.6 RADIATION WARNING LIGHTS AND LABELING:

3.6.1 Main Power switch and Indicator Light:

The main power switch is found on the rear of the unit and is shown in the figure to the left. Pressing the switch for several seconds will turn on the main power. A green LED indicates the main power is on. The main power must be turned on in order to operate the unit however, this switch DOES NOT turn on the x-ray tube. No power will be supplied to the x-ray tube unit the Innov-X software is started.

3.6.2 Probe Light and Probe Label:

The Innov-X analyzer is equipped with warning lights that alert the operator when the tube is receiving power, and when x-rays are being emitted from the analyzer. Please see Fig. 3.5.

When the red light on the front nose of the analyzer is ON continuously (not blinking), this indicates the x-ray tube is receiving a low level of electrical power and the shutter is closed. The system is producing a low level of x-rays internally in this condition, but the shutter is providing adequate shielding to keep x-ray levels below levels of detection. The instrument is safe to be carried around or set down in this configuration.

When the red light is blinking, this indicates the tube is powered, the shutter is open and the analyzer is emitting x-ray radiation out of the analysis window. The analyzer should only be pointed at a sample, or be in the testing stand with a sample resting on the window, in this configuration.

3.6.3 Display on Back of Analyzer:

The display on the back of the analyzer, shown in Fig. 3.6, provides a "testing" message to indicate that the x-ray tube is energized and the shutter is open. This display is for testing conditions (i.e. overhead) where the operator cannot see the Probe Light or the iPAQ display.

3.6.4 Label Behind iPAQ:

The analyzer also has a label just below the iPAQ indicating, as shown in Figure 3.7:

CAUTION: Radiation. This Equipment Produces Radiation When Energized.

This label is required by most regulatory agencies. The term "When Energized" refers to the condition where the tube is fully energized and the shutter is open. This condition is also indicated by the red blinking light on the probe.

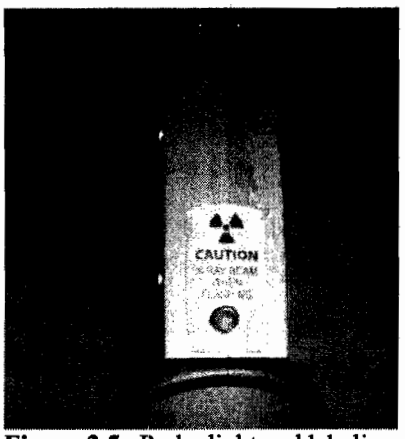


Figure 3.5. Probe light and labeling. When the light is on continuously, the x-ray tube is receiving minimal power and it is producing a minimum level of x-rays. The shutter is also closed so there is no radiation exposure to the operator or bystanders.

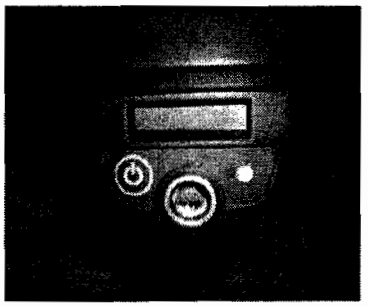


Figure 3.6. Back light on analyzer.

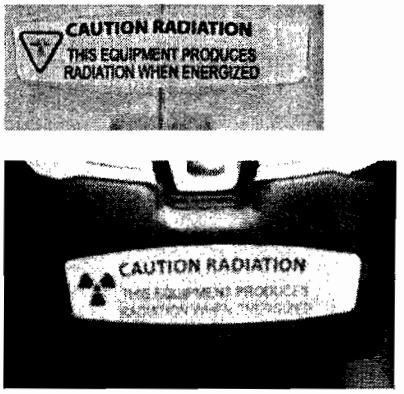
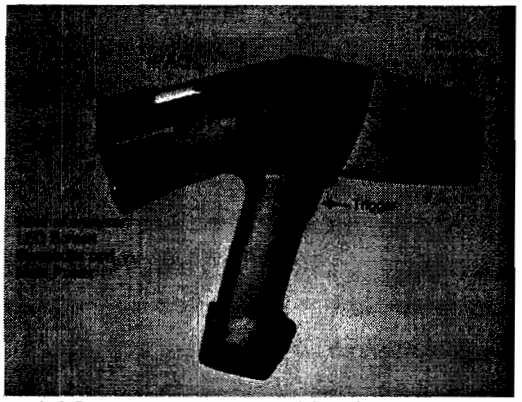


Figure 3.7. Label behind iPAQ. Top version is used in Canada

3.7 RADIATION LEVELS FROM ANALYZER

Two pictures of the analyzer are shown below. In the first picture, all the relevant components referenced in this radiation safety section are displayed and labeled. The second picture shows a close-up of the front end of the window. The four sides A, B, C and D are indicated on this picture because they are referenced in terms of radiation levels output by the analyzer. The measured radiation levels for standard operating conditions are shown in the figures and tables below. Standard operating conditions are tube voltage operating at 35 kV, tube current of 5 uA, and 2 mm aluminum filtration.



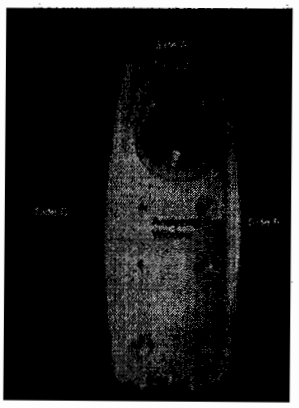


Figure 3.8 Innov-X Analyzer, Side View

Figure 3.9 Innov-X Analyzer, Front View

Radiation Levels (mrem/hr) for Alloy Analysis, Standard Beam Conditions: 35kV, 5 uA, 2mm aluminum filtering:

Sample at Window	Trigger	Location A (Top)	Location B (Right Side)	Location C (Bottom)	Location D (Left Side)
Blank (Air)	<0.1	<0.1	<0.1	<0.1	<0.1
Metal	<0.1	<0.1	<0.1	<0.1	<0.1

Table 3.1. Dose rates (units of mrem/hr) at various locations with a metal sample covering the window and with no sample present. For "no sample" the analyzer is shooting the x-ray beam into air.

As shown in the Table 3-1, the dose to the operator's hand is negligible. The radiation levels at the side surfaces of the instrument snout (aluminum housing) are all <0.1 mrem/hour. Despite these low levels of radiation, there is no reason for any body part to be in the locations denoted A, B, C and D!

Table 3-2 shows the radiation levels directly in the x-ray beam that is emitted from the analyzer. Radiation levels at the exit aperture (or "port") are substantial. There is no reason for the operator or any personnel to be exposed by the direct beam. Operators should never hold samples in their fingers or cupped in their hands, as this may generate a significant radiation exposure.

Operations should never point the analyzer at another person and start a test, as this may also provide significant exposure to the person if they are within a few inches of the port of the instrument.

Radiation Levels in the Primary Beam Versus Distance from Port:
For Alloy Analysis, Standard Beam Conditions: 35kV, 5 uA, 2mm aluminum filtering:

Tube Conditions	At Trigger, or any part of operator's body.	At Window	4 inches	12 inches	36 inches	48 inches
35 kV, 5 uA, 2 mm Al filtering	<0.05	28,160	2,080	186	24	14
15 kV, 25 uA, thinner filter material	< 0.05	27,780	1,620	145	19	11

Table 3.2. Dose rates (units of mrem/hr) in the direct x-ray beam being emitted from the analyzer

3.8 RADIATION DOSES FOR SEVERAL SCENARIOS

In this section we provide data, concrete examples of use and misuse of the analyzer and common questions and answers we encounter when training personnel on the safe use of the Innov-X analyzer. The goal is to explain scenarios of safe versus improper usage of the analyzer.

The table below presents radiation doses for normal operating conditions and also for examples of misuse of the analyzer and even extreme misuse. Innov-X provides installation training that includes detailed radiation safety training and documentation designed to prevent misuse of the analyzer

Example of Instrument Usage	Radiation Exposure and Comments
Normal Operation - Dose to Hand: User analyzes samples according to standard operating procedures described in this manual. Assumption: Operator using system with x-ray tube ON for 8 hours/day, 5 days/week, 50 weeks/year. (Practically constant usage).	Maximum exposure is to operator's hand, at the trigger. Exposure is < 0.1 mrem/hr. Annual exposure to hand is then < 200 mrem (2mSv). US: Maximum exposure under OSHA regulations is 50,000 mrem annually. Thus continuous operation provides a dose that is at least 250 times lower than maximum allowed by OSHA. Canada: Maximum exposure under ICRP regulations is 500 mSv for radiation workers and 50 mSv for the general public. Thus continuous operation provides a dosage 250 times lower for a radiation worker and and 25 times lower for the general public.
Normal Operation – Dose to Torso: Analyzer is used under the same operating conditions described above.	Exposure to Torso is so low it cannot be measured. To be conservative we use the same figure as the trigger, <0.1 mrem hr. Annual exposure using operating conditions above is < 40 mrem. (0.4 mSv) Maximum allowed is 5,000 mrem under OSHA and 20 mSv under ICRP for radiation workers (1 mSv for general public).

For the x-ray energy emitted by portable XRF analyzers (10-60 keV region), the bone in the fingers will absorb radiation about 3-5 times more than soft tissue, so the bone would be at an elevated radiation risk compared to soft tissue. For this reason no person shall hold a test specimen in front of the window with the fingers in the direct beam, or direct the beam at any part of the human body. Reference: Health Physics 66(4):463-471;1994.

Misuse Example 1: Operator holds samples in front of window with fingers, such that fingers are directly in the primary beam. Do not do this!.	For fingers at the port, in the primary beam, the maximum dose to the fingers is 28,160 mrem/hr. Assume an operator performs a 10 sec test (typical). The dose to the operator's fingers or hand is 28,160 x (10/3600) = 78 mrem. If the operator did this 641 times/year they would exceed the allowable annual dose of 50,000 mrem to an extremity. In Canada, the maximum allowed dose is 500 mSv/year (Canada ICRP radiation worker) or 50 mSv/year (Canada ICRP general public). If the test time was 30 seconds instead of 10 seconds, the operator would receive a dose of 234 mrem for each exposure, and thus would exceed the annual safe limit of 50,000 mrem after 213 tests. Even though it is unlikely to make this mistake so many times in a year, do not even do it once. Take the extra time to test a sample on a surface or use a testing stand. **Note: If the operator takes an average of only two shortcuts per week and places his/her fingers within the primary x-ray beam at the window, they will exceed the annual dose rate.**

Misuse Example 2:
Operator places analyzer against body and pulls the trigger to start a test. Analyzer tests to preset testing time (usually 10 seconds) unless operator pulls trigger again to stop test. This applies to analyzer being in contact with operator or with bystander.

Dose at exit of sampling window is 28,160 mrem/hr.

Dose for a 10 second exposure with analyzer in contact with Torso: 78 mrem (.78 mSv).
US: If an operator did this act 64 times in a year, the operator would exceed the annual safe dosage to the torso of 5,000 mrem/year. The maximum dose of 5,000 mrem/year is a whole body limit, which does not truly apply in this case because the x-ray beam size is small (about 2 cm^2 area – 1.5 cm x 1 .3 cm – at the port). Applying correction factors for the beam size is complex and beyond the scope of this manual. The important point is that for proper operation there is no reason to ever exposure any part of the human body directly to the x-ray source. This example serves to provide estimated exposure in the event this occurs.

If the testing time was 30 seconds instead of 10 seconds, thus the operator placed the port against his body or that of a bystander and performed a 30 second test, the dose would be 234 mrem. This is about the same as a mammogram. Repeating this gross mis-use 22 times would exceed the annual allowable limits.

Canada: Radiation worker would have to repeat this example (234 mrem exposure) of gross misuse 8 times to achieve the ICRP level of 20 mSv. (general public 1.3 times to achieve limit of 1mSv)

Misuse Example 3:
Operator manages to initiate a test for 10 seconds and exposes a bystander that is standing 12" away from analyzer port. What is exposure to bystander?

Note: The proximity sensor would automatically shut down the x-ray tube after 2 seconds, so this is an extremely improbable occurrence.

Note 2: Equations to scale these to other scenarios involving longer or shorter tests, and bystander being at distances other than 12" are provided at right.

Dose to bystander at 1 foot is 350 mrem/hr. For a 10 second exposure dose is 1 mrem. This is 5,000 times lower than the allowable dose to a worker in a year. This would have to happen 5,000 times to for that worker or bystander to obtain the maximum allowable dose.

Formula for calculating other scenarios:

$$Dose = 1\,mrem\left(\frac{13.25}{D+1.25}\right)^2 x\left(\frac{t}{10}\right)$$

D = distance from port in inches
T = testing time

Example: Bystander is 3' away from port for a 30 second test. In this case the dose is calculated as:

$$Dose = 1\,mr\left(\frac{13.25}{36+1.25}\right)^2 x\left(\frac{30}{10}\right) = 0.38\,mrem$$

US OSHA: Maximum allowable level is 5,000 mrem assuming bystander's torso is exposed. Thus, this misuse would have to occur 12,500 times in a year to the same bystander before that bystander achieved his maximum allowed dose.
ICRP: 5000 times for rad worker, 250 for general public

Comparative: Radiation Doses from Typical Exposures to Ionizing Radiation

Common medical and/or dental x-rays:	20-30 mrem each.
Mammogram:	100-200 mrem
Flying in a commercial jet coast to coast (6 hrs.):	1-2 mrem.
Daily exposure from background radiation: * depends on geographic location	0.3 to 0.5 mrem/day

Table 3.3 Radiation Doses from Typical Exposures to Ionizing Radiation

From the above table, a single case of analyzer misuse, thus producing a one-time exposure of 70-250 mrem, is comparable with single-event common medical x-ray procedures such as an annual chest x-ray or mammogram, or 25-50 airline flights in a year, and thus is not considered harmful. Regular misuse, such as taking safety shortcuts twice weekly, produces radiation exposure that greatly exceeds these typical levels and should be avoided entirely.

3.9 COMMON QUESTIONS AND ANSWERS REGARDING RADIATION SAFETY

Question: When I'm shooting a piece of pipe or valve on a rack or on a table top, is there any exposure to people standing in other locations, or standing several feet away from the analyzer?

> Answer: Even a thin amount of metal sample (1-2 mm thickness) is enough to completely attenuate the x-ray beam emitted from the Innov-X analyzer. Shooting a piece of material that covers the sampling window on the analyzer will completely shield any bystanders from radiation exposure. However, good practice recommends that the area for at least 4-5 feet in front of the analyzer is clear of people.

Question: If I forgot to switch the safety on the trigger to "ON", I pick up the analyzer and accidentally pull the trigger, is that dangerous to nearby personnel?

> Answer: No, this example of misuse is not dangerous, but it may produce a non-negligible radiation exposure to nearby personnel. For an exposure to occur, the following things must happen. First, you must be holding the analyzer so that a bystander is actually standing in the x-ray beam being emitted. Just being near the analyzer is totally safe otherwise. Second, the bystander must be within 1-3 feet from the nose of the analyzer in addition to being in the beam path, to receive any appreciable dose. If all of these conditions are true, the dose received by a bystander is still extremely low. It ranges between 0.1 to 0.5 mrem depending on the exact location of the bystander. This dose is 10,000 to 50,000 times less than the allowed dose. Please see Misuse Example 4 in the table above.

Question: Do I need to create restricted areas where I am using the analyzer?

> Answer: No, provided you are following normal operating procedures there is no reason to restrict access to an area where the analyzer is in use. The operator should take precautions to keep any personnel more than 3 feet away from the sampling window of the analyzer in the event of accidental misuse as detailed above. Should the operator also elect to test small components like weld rod as shown in Figure 3.3, the operator should also be sure that no personnel are standing within about 4-5 feet of the sampling window.

Question: How does the x-ray tube in the Innov-X system compare to a radiography system used for taking images of metal parts.

Answer: The x-ray tube used in the Innov-X system produces between 1,000 and 10,000 times lower power than most radiography systems (0.5-1 watt for Innov-X versus kW for radiography systems). This is because a portable XRF is designed to perform surface analysis of alloys and other samples, whereas radiography systems are designed to shoot x-rays entirely through metal components in order to obtain an image on the other side of the object being bombarded with x-rays. For example, many tube-based radiography systems use a 300-400 kV tube and currents in the tens or hundreds of milliamps (mA). The Innov-X analyzer uses a tube operating at 35 kV and 5-30 micro-amps. The radiation levels produced are therefore thousands or tens of thousands times lower with the Innov-X system.

Question: Should we use dosimeter badges with the Innov-X analyzer.

Answer: Dosimeter badges are required by some states, and optional by other states. Innov-X recommends that operators wear badges, at least for the first year of operation, as a general precaution to flag any misuse of the analyzer. Dosimeter badges are available for the torso (generally worn on the belt loop or shirt pocket) and are available as "ring" badges. The best single badge to obtain is a ring badge that is worn on a finger, on the opposite hand used to hold the analyzer. This will record accidental exposure for the most likely case – an operator grabbing a small sample and holding it in one hand while analyzing it. Note: these badges generally have a threshold of 10 mrem, and are renewed monthly. So it will take several cases of misuse even to obtain a reading on a typical badge. When purchasing a badge, obtain the type used for x-ray and low energy gamma ray radiation.

3.10 SAFE GUARDS AND EMERGENCY RESPONSE

The main safeguards to use as an owner of an Innov-X portable XRF are really intended to restrict access to properly trained operators:. **Note: Canadian regulations require certified personnel to use the device, refer to section 3.0 in this chapter.**

1. Keep the system in a controlled location, where only authorized users are likely to have access to the analyzer at any given time.
2. Make a simple sign that is kept with the analyzer indicating that an operator must have completed a training class provided by your company or must have attended an Innov-X training course in order to use the analyzer. Note that when the Innov-X system is turned on, the screen displays a message indicating that the system should only be used by authorized personnel.

Emergency Response:

Because the Innov-X system is a battery operated, x-ray tube based analyzer, the emergency response plan is very simple. If the operator believes the analyzer is locked up in an "OPEN" position, they should do two things:

1. Press the On/Off switch on the base to power the analyzer off. The green LED indicator will turn off, indicating system power is off. At this point it is not possible for the analyzer to be producing x-rays.
2. As an additional precaution, the operator may remove the battery trap door at the bottom of the analyzer (have the nose pointing away from personnel), and pull out the battery. Even if the operator has failed to properly power the system off in Step #1, removing the battery guarantees that no x-rays can be produced. There is no electrical power being provided to the x-ray tube.

Note: It would be highly unusual for an operator to somehow lock up the analyzer with the x-ray tube powered on. This would require the operator to crash the iPAQ during an analysis. If this happens the analyzer will shut off the x-ray tube 10 seconds after the last communication with the iPAQ. However, if at any time the operator believes the x-ray tube is on and no test is in progress, powering off the analyzer and

restarting will automatically shut down the x-ray tube and close the shutter. It will no longer be possible to produce x-rays at this point.

3.11 DOSIMETER BADGES

Dosimeter badges are provided as a monthly service by several companies, listed in this section (see below). The badges are generally provided monthly, and the operator returns the previous month badges to the company for analysis. The operator receives a monthly report showing any personnel with readings higher than typical background radiation.

Dosimeter badges are required by some states, and optional by other states. Innov-X recommends that operators wear badges, at least for the first year of operation, as a general precaution to flag any misuse of the analyzer. Dosimeter badges are available for the torso (generally worn on the belt loop or shirt pocket) and are available as "ring" badges. The best single badge to obtain is a ring badge that is worn on a finger, on the opposite hand used to hold the analyzer. This will record accidental exposure for the most likely case – an operator grabbing a small sample and holding it in one hand while analyzing it. Note: these badges generally have a threshold of 10 mrem, and are renewed monthly. So it will take several cases of misuse even to obtain a reading on a typical badge. When purchasing a badge, obtain the type used for x-ray and low energy gamma ray radiation.

Dosimeter Companies:

Here are two companies that provide badges as a regular service. There are certainly many more.

Landauer Inc. Glenwood, IL 708-755-7000	AEIL Houston, TX 713-790-9719

3.12 TYPICAL REGISTRATION REQUIREMENTS

Innov-X maintains a database of the registration requirements for every state, including sample registration forms. Most states require some form of registration, and generally they require the registration to be received within 30 days of receipt of the instrument. Some states require no registration, while a few require notification in advance. Please contact Innov-X for specific questions regarding the state where the instrument will be used, or for copies of registration forms.

In general a company will have to provide the following information regarding the device:

1. Purpose of device. Generally this is "Analytical" or "Industrial." Be sure to inform the state registration office that the device will NOT be used for radiography or for medical uses.
2. Radiation Safety Officer – Monitors training, safe use, and controls access to the instrument.
3. Authorized Users – Trained by Innov-X Factory Authorized Representatives in the safe and proper use of the XRF.
4. Operating parameters of the analyzer – 35 kV, 5-30 micro-amps.
5. Type of system, either fixed, mobile or portable. Generally the correct choice is "Portable."
6. User Training Specified – Indicate that only individuals receiving manufacturer training, documented by a manufacturer's training certificate will operate the instrument.
7. Personal Monitoring. This may be required by radiation control authorities. Many registration forms will ask that you indicate whether or not you intend to perform dosimeter monitoring.
8. Copy of Registration & Manual at the Job Site

If you have any questions regarding the type of registration form or filling out the form, please contact Innov-X Systems. Many states may confuse a portable XRF system that uses a tube with medical or

industrial radiography systems. This is because of the relative newness of portable tube-based systems. In all likelihood, Innov-X personnel have experience providing the necessary documentation to the state in question, and can readily assist the customer in this process.

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 size 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:

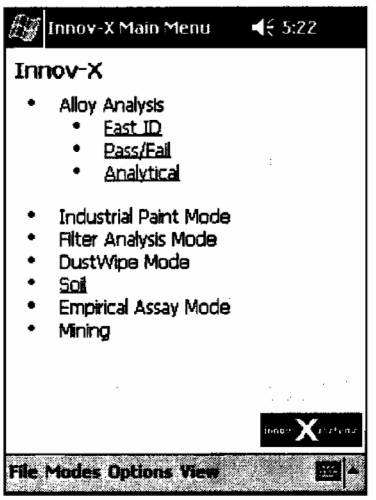
- **Main Menu screen:** Used to select the analysis mode, open the results screen, and change the administrator password.
- **Analysis Screen:** Used to change settings, edit libraries, and perform tests.
- **Results Screen:** Displays results from current reading, allows scrolling back to previous test 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.

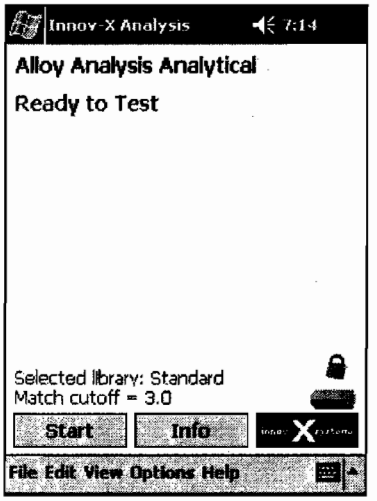


4.2.2 The Analysis Screen

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.

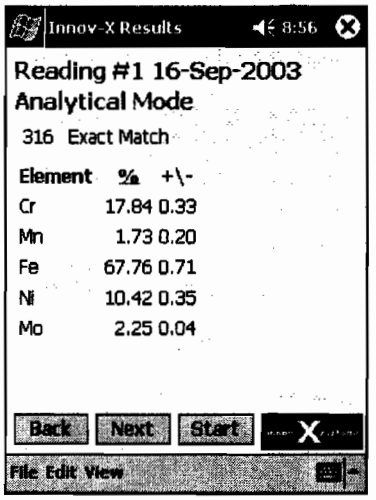
The analysis screen for Analytical mode is shown to the right. Screens from other modes are similar and will be described in later in this manual. The analysis screen shows the name of the mode that is currently active, a start/stop button (which is inactive in most cases), an info button that is used 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.

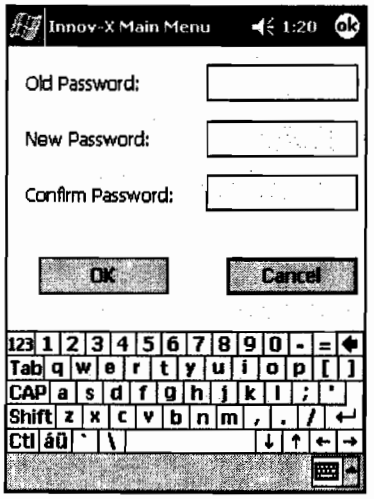


4.3 PASSWORDS - ABOUT PASSWORD PROTECTION

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

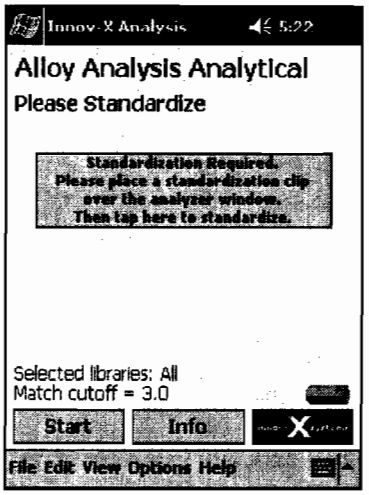
4.4.1 Standardization Procedure

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.

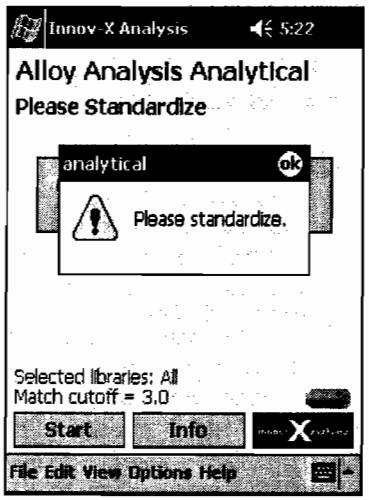
The standardization procedure takes about 1 minute. Standardization must be done any time the analyzer hardware is initiated or restarted and must be repeated if the instrument is operating for more than 4 hours.

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.

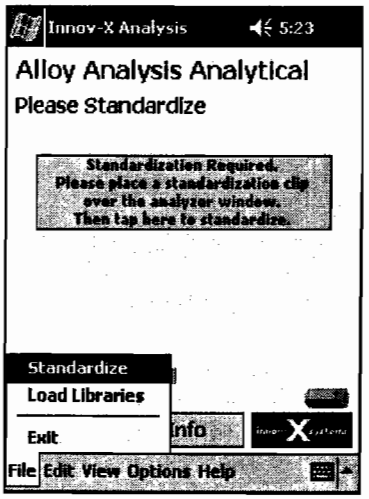
If the analyzer is restarted, you will be required to standardize the instrument before performing any measurements. This is indicated by the message "**Standardization Required. Please place a standardization clip over the analyzer window. Then tap here to standardize.**" on the analysis screen



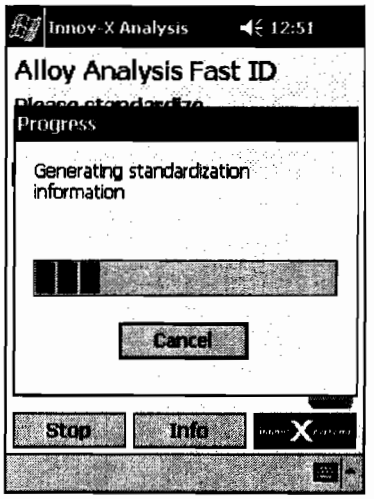
It is not possible to start a test before standardization. If the trigger is pulled before the standardization procedure is completed, a message box will appear. Press **ok** to acknowledge and clear the message.



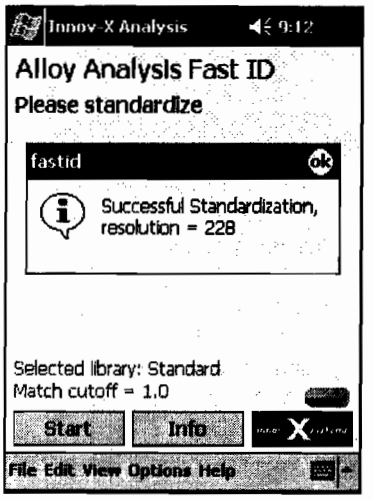
To initiate the standardization procedure, snap the standardization piece on the front of the instrument. Verify that it completely covers the analyzer window. When using a standardization mask with a weld collimator, be sure that the solid portion of the mask covers the analyzer window. Tap the grey 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.

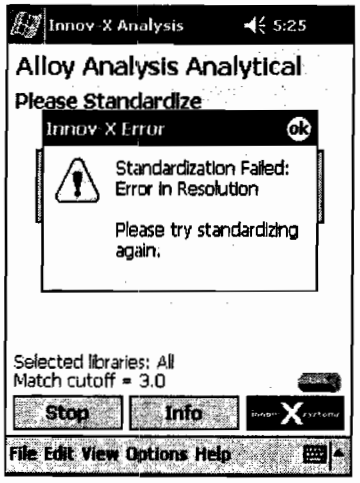


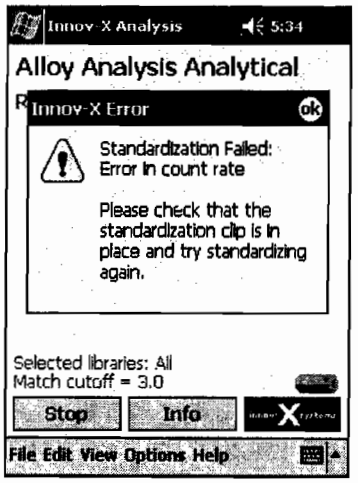
When standardization is complete, the message "Successful Standardization" will appear, along with the resolution of the instrument. Tap **ok** to acknowledge and clear the message. The instrument is ready for testing.



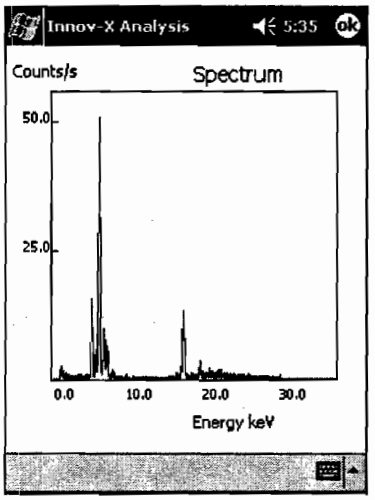
4.4.2 Standardization Errors

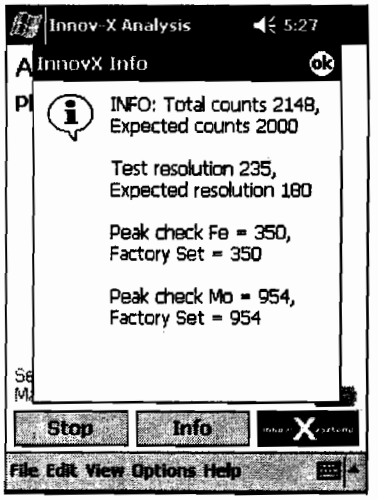
The analyzer performs several diagnostic checks during the standardization process. If the standardization fails, the instrument will prompt the user regarding the next step. Several errors could occur while standardizing: "Wrong Standardization Material," "Error in Resolution" or "Error in Count Rate"





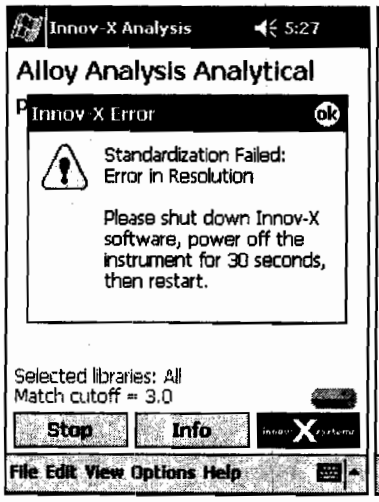
After closing the Standardization Failed message, two additional screens will appear. The first is a picture of the spectrum generated during the standardization. The second is a summary comparing factory set values for resolution, count rate, and peak positions to values calculated during the standardization.

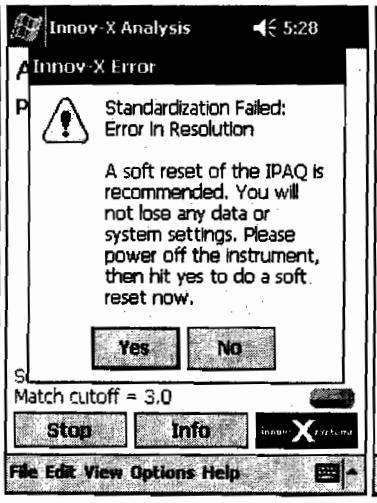


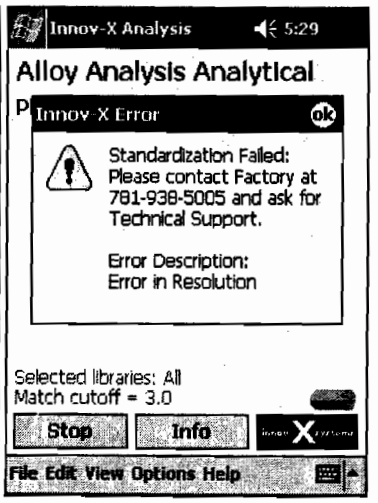


When standardization fails, verify that the standardization mask is in place, and attempt standardization again. To restandardize after a failure, tap the grey box in the center of the display, or choose ***File→Standardize***. If you are using a weld collimator, make sure that the solid part of the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

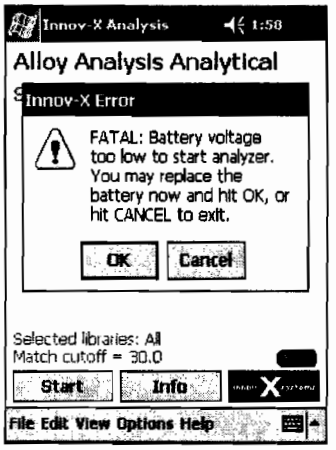






4.4.3 Battery Replacement and Initialization/Standardization

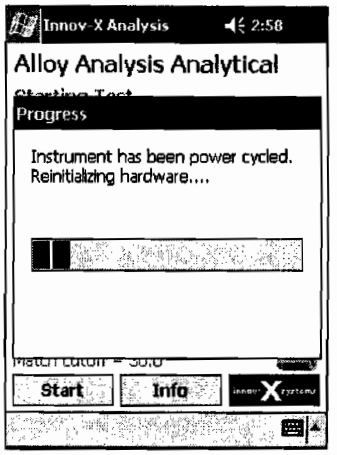
When the battery is too low to take a measurement, an error message will appear:



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.

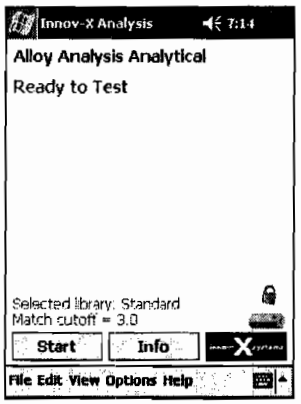
After re-initialization is completed, testing can continue.

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.

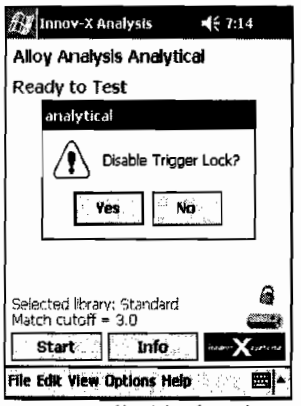


4.5 THE SOFTWARE TRIGGER LOCK

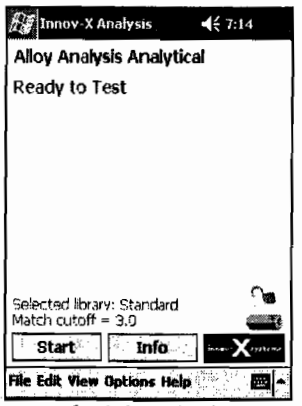
Innov-X analyzers are equipped with a software trigger lock which prevents the trigger from being actuated unintentionally. The lock is released by tapping an icon on the iPAQ screen. Once the lock is released, it will remain unlocked for subsequent tests, until more than five minutes has elapsed between 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

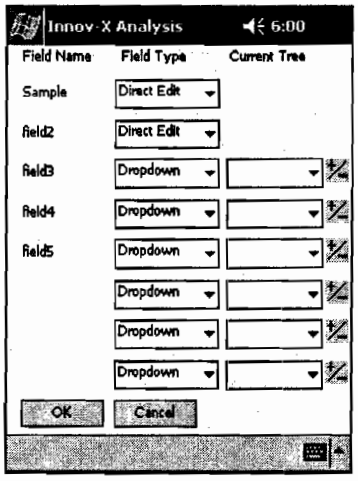
Information such as sample name, and identifying characteristics can be stored with each measurement. This is done from the test information (Test Info) screen which can be accessed from the **Analysis Screen** of any mode by tapping the **Info** button, or selecting ***Edit→Edit Test Information***.

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

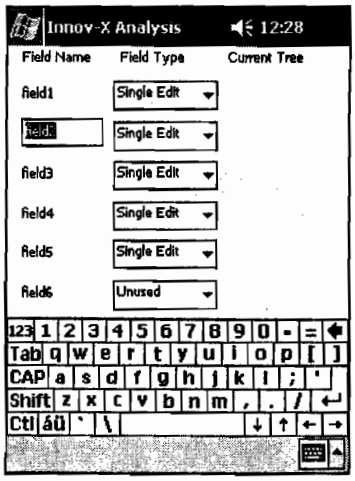
Test Info fields are modified via the Modify Test Info Template option found in the edit menu on the analysis screen in every software mode. Each field can be designated to be Direct Entry, Drop-down, or Tree. Direct entry fields allow users to enter characters directly from the virtual keyboard, or 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.



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.

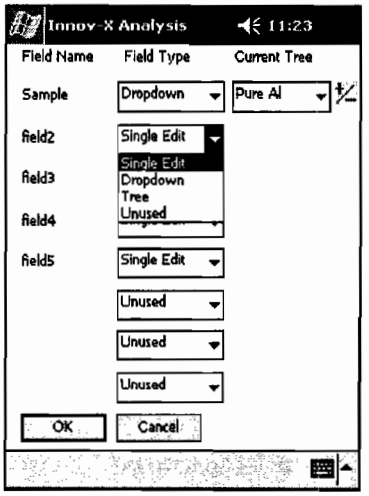


4.6.1b Selecting Field Type

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.

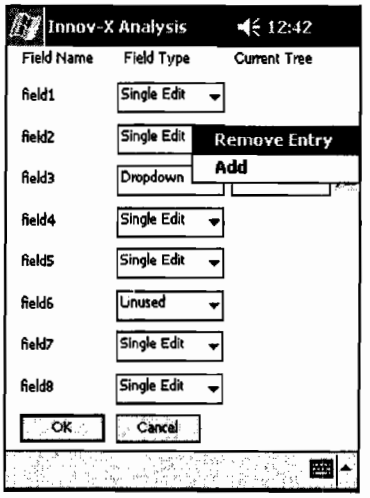
- Select **Direct Edit** for a text field which will accept data from the virtual keyboard, or a bar code scanner.
- Select **Drop-down** for a drop-down list
- Select **Tree** for a Drop-down menu with many choices, some of which may be grouped into categories and subcategories.

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

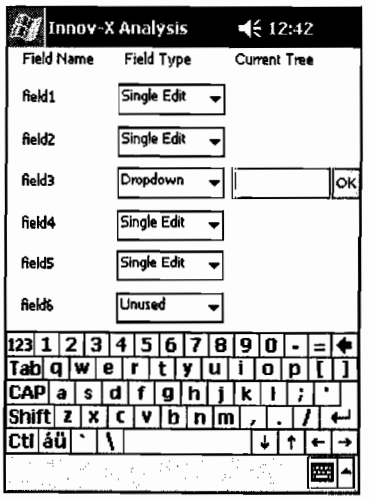


4.6.1c Changing Drop-down Menu Entries

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**.

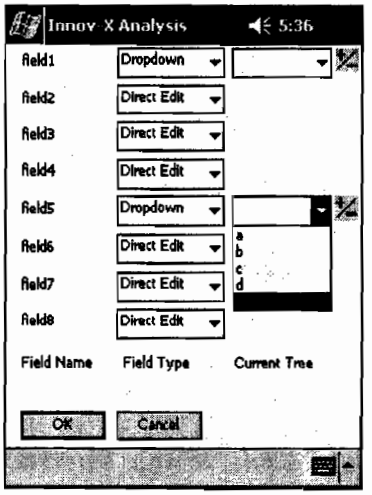


To add an entry from a drop-down list, tap the +/- symbol next to appropriate field, and select **Add**. Type the new info into the blank text box that appears. Select **OK** and the entry will be added to the drop-down menu.



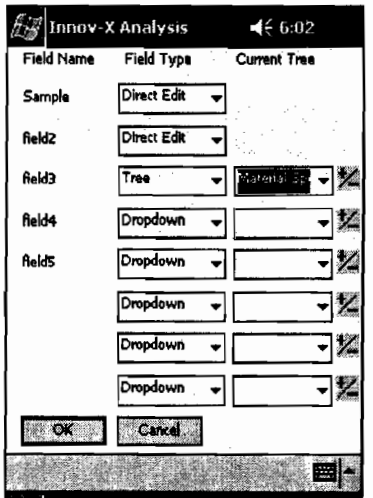
Repeat the process above to complete the complete drop-down list.

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.

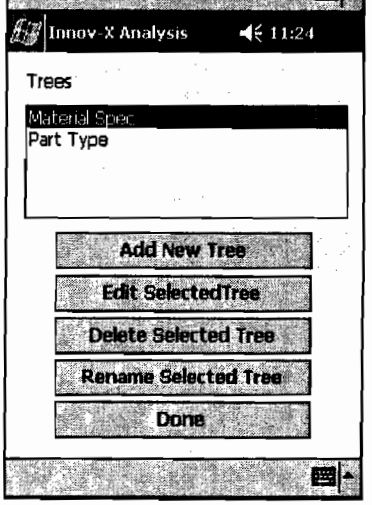
Once a field has been designated a tree, modifications to the contents of the tree can be made by tapping the +/- symbol to the right of the tree.



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.

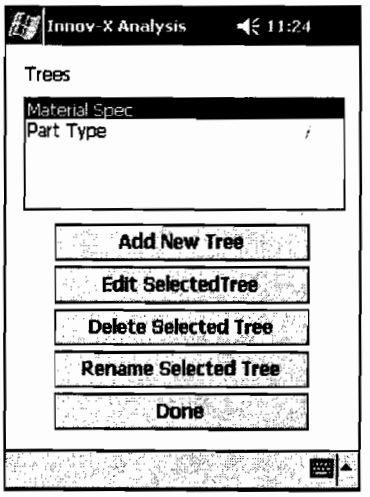
When you have finished creating/editing your tree, highlight it and select **Done.**



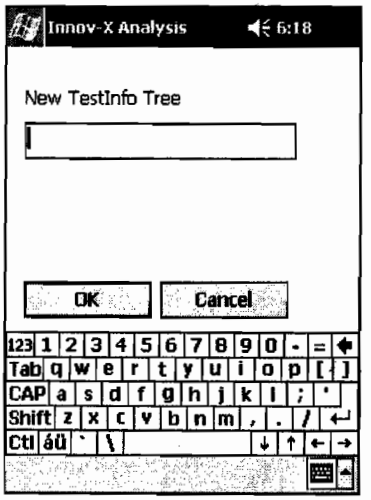
The following is an example of how a user might create a tree: A manufacturer of tubes and valves tests all parts to ensure that they're made of the proper material. The company's QC procedure involves labeling each test with the part number of the item. Rather than forcing operators to look through 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:

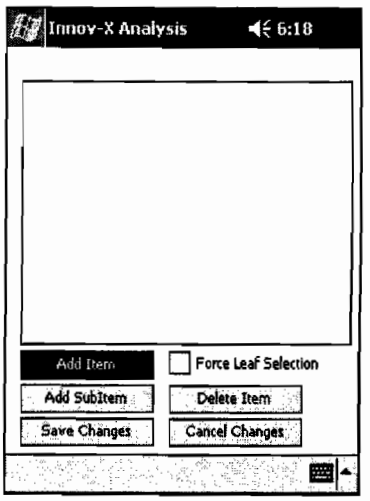
Select: Add New Tree:



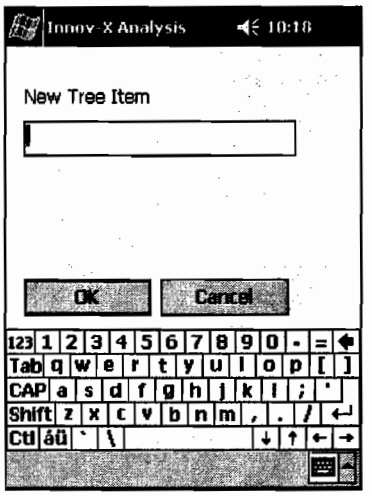
Enter the Name of the Tree in the text box and select OK.



Tap Add to add the first item

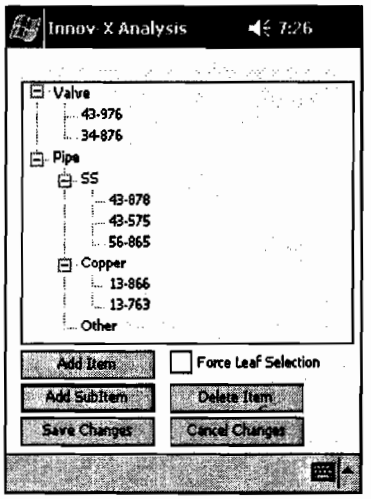


Enter the name of the item



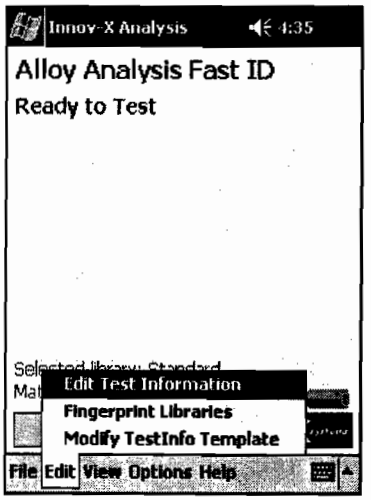
Once the tree is started, continue to Tap Add Item to add a top level menu item, or select an item and tap Add SubItem to link a subcategory to the item. Continue until all items have been added.

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

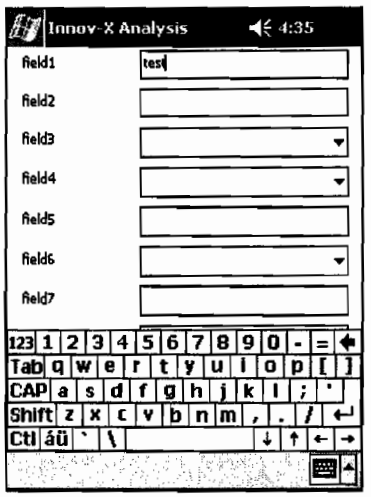


4.6.2 Entering Test Information

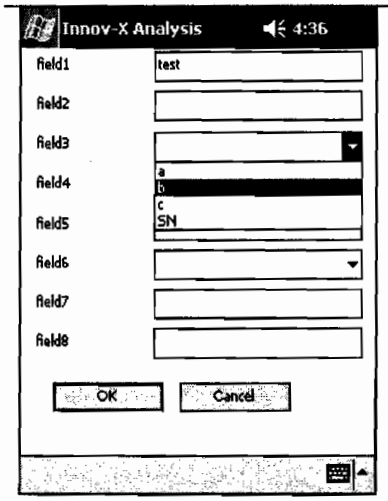
1. To enter the Test Info screen, you must be in the Analysis Screen. If the Results Screen is open, tap the ⊗ in the upper right hand corner to return to the Analysis Screen. From the Analysis Screen, select ***Edit→Edit Test Information,*** or tap the **Info** icon.



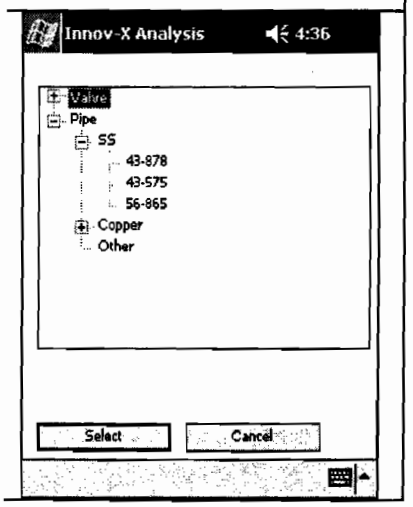
2. To enter a unique sample name or number, select a direct entry field by tapping anywhere within the field. Use the virtual keyboard to enter the information.



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**.

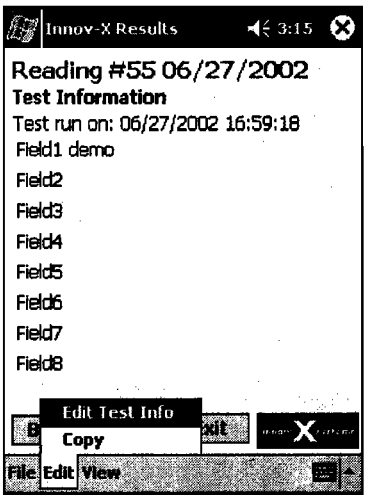


5. When all the necessary data have been entered. select OK
6. The information entered in the test info screen will be saved with each reading until the test info screen is modified again.

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

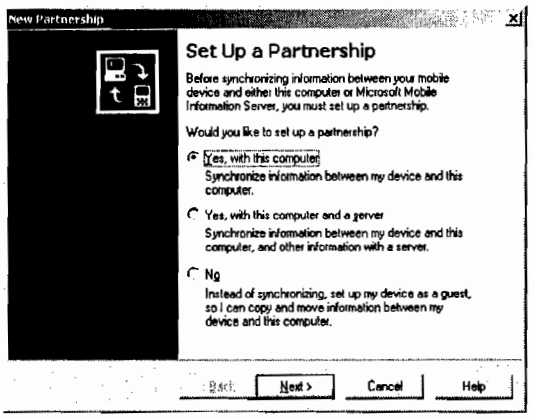
In order to copy files between the iPAQ and a desktop PC, Microsoft Active Sync Software must be installed on the desktop PC. Innov-X strongly recommends that you download the latest version of ActiveSync from the internet. ActiveSync v3.7 may be downloaded from
http://www.microsoft.com/windowsmobile/resources/downloads/pocketpc/activesync37.mspx

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.

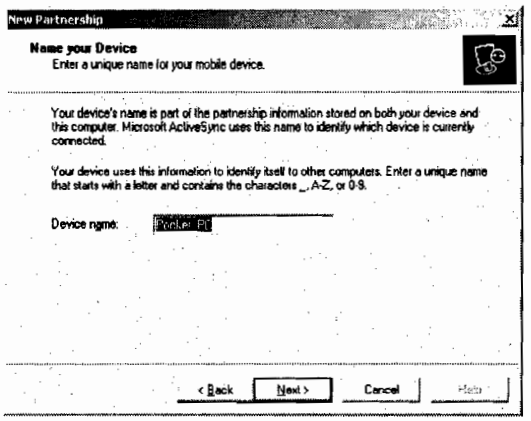
The iPAQ cradle should be hooked up to the USB port on the desktop computer before installing software.

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

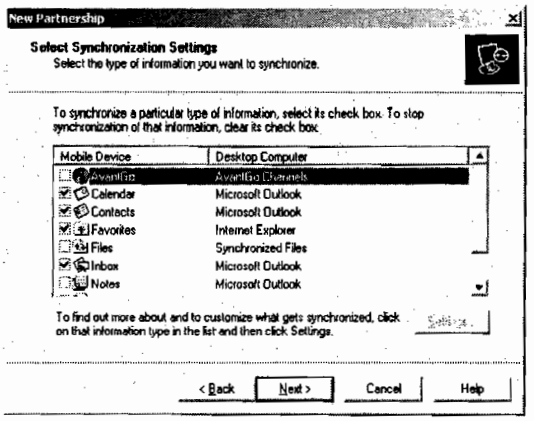
1. Insert the ActiveSync CD in your CD Drive. It will start automatically. The CD contains information about Getting Started with Your Pocket PC. This changes periodically, so it's difficult to describe exactly what the screens will look like. Step through the screens until you see the option "Install 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

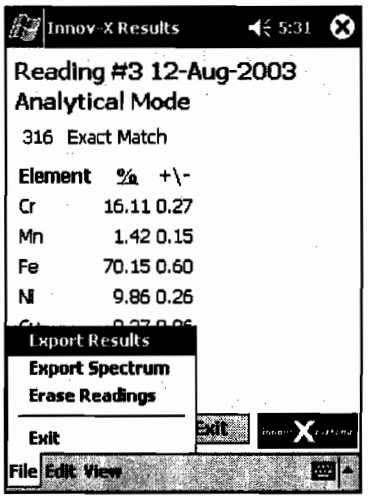
a. Exporting results on the iPAQ. (described in section 4.6.2)
b. Synching the iPAQ to the computer
c. Opening the results in Excel for viewing, or printing.

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.

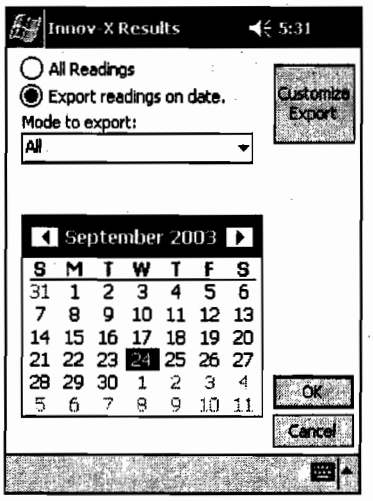
From the results screen, select ***File→Export Results***



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.

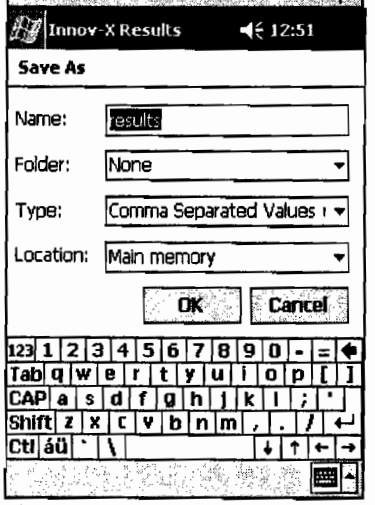
The customize export option allows users with administrative password privileges to customize the format in which data is exported. This is described in **Section 4.7.3: Customizing Results Export**.



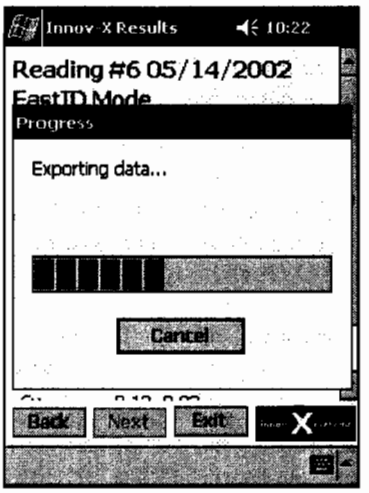
After choosing which readings to export, you may choose to export all data, or just data from a specific mode. Selecting the arrow to the right of the mode to export will open a drop-down menu. Select the mode for which you want to export data.

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."

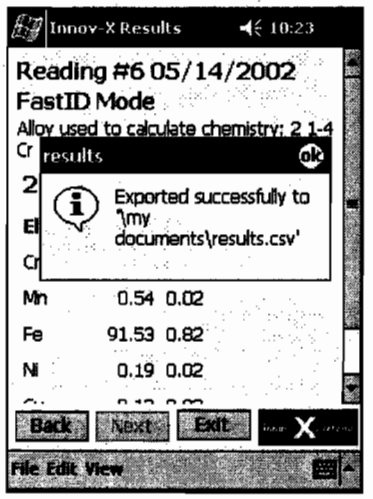
When the proper selections have been made, select **OK**. A **Save As** box will appear. Select the folder in which you want to save the data, and name the file. The file Type will always be **Comma Separated Values**. The recommended Location is Main memory and Folder is **None.** This will export files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



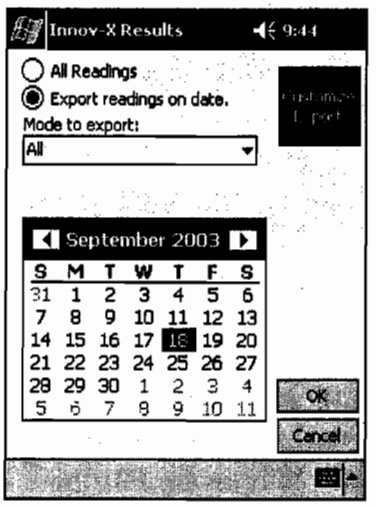
4.7.3 Customizing Results Export

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.



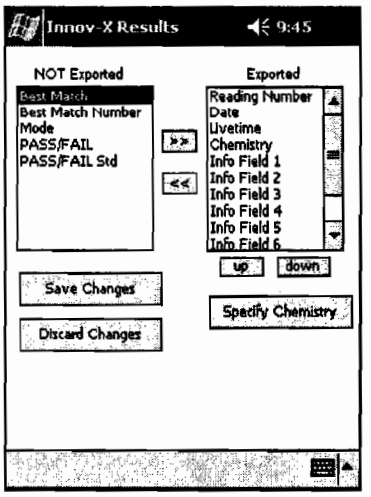
Two columns appear on the screen; the column on the left lists fields which will NOT be exported, and the right-hand column lists fields which will be exported.

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

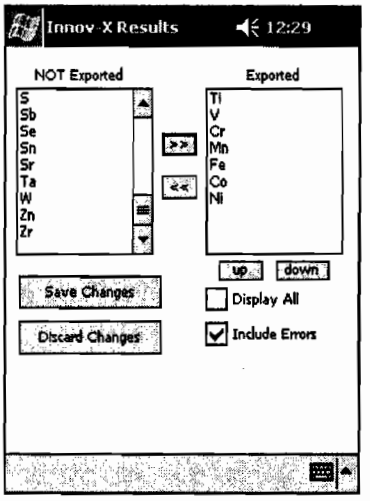
Once all changes have been made, choose **Specify Chemistry** if changes need to be made to the list of exported elements.

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



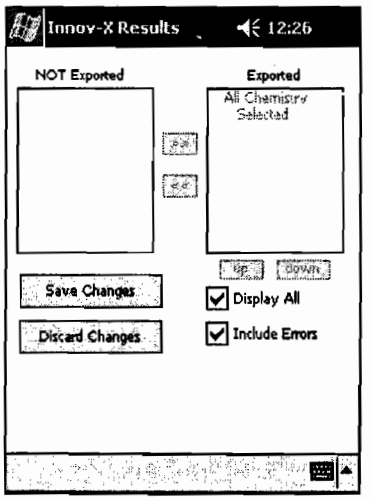
The Specify Chemistry screen resembles the previous screen. Move elements to the appropriate column, depending on whether or not an element should appear in exported files.

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



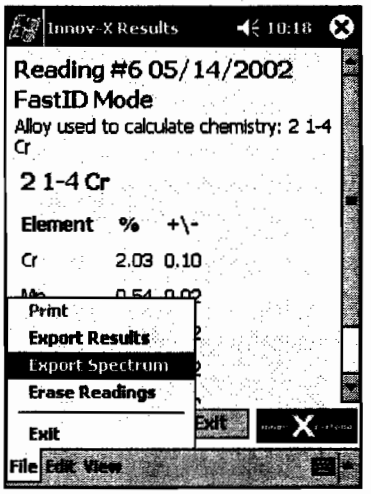
Select **Display All** to include all measured elements. *This setting is recommended, as it will ensure that all data measured with the instrument is exported.*

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

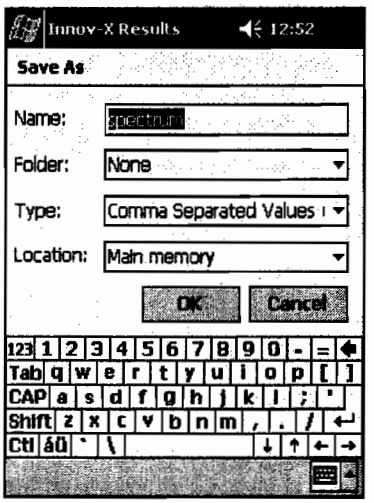


4.7.4 Exporting spectra

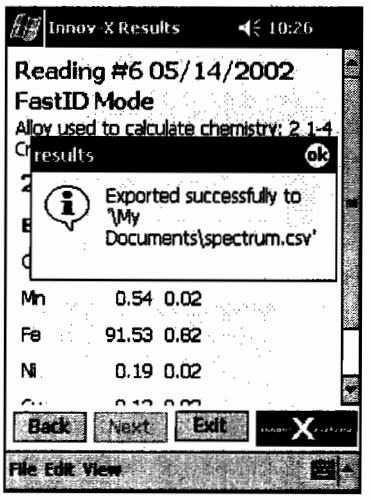
Only one spectrum may be exported at a time. In the results screen, scroll to the reading for which you wish to export the spectrum, and ***Select File→Export Spectrum.***



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.



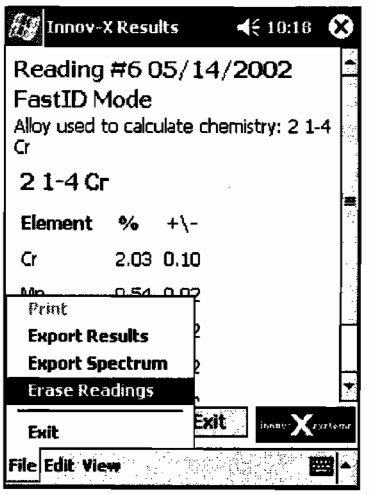
A message will appear indicating a successful export. Tap **ok** to acknowledge and clear the window.



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 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 .

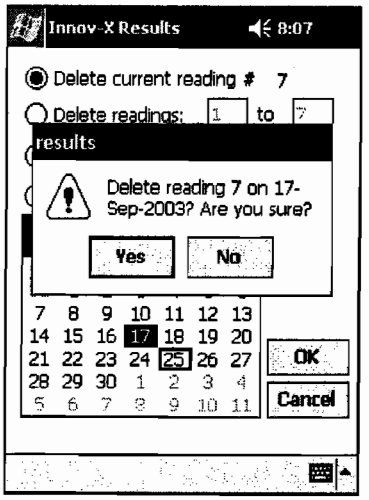
From the results screen, select ***File→Erase Readings.***

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:

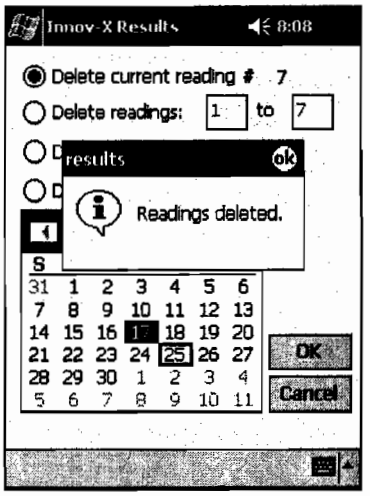
- Selecting **Delete current reading** will delete the reading that is currently open.
- Choosing **Delete readings XX to XX** will delete a range of readings from the date of the reading that is currently open.
- **Delete all readings on date** deletes all readings from a specific day.
- **Delete readings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

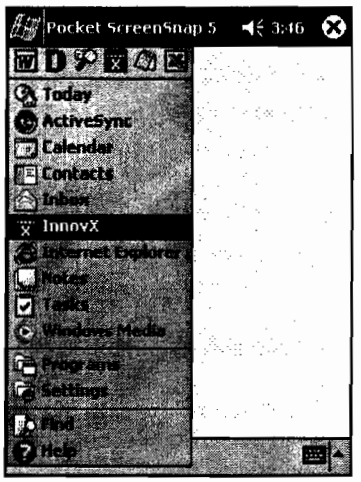
The basic startup and testing procedure is described below. Most screen shots were taken using **FastID Mode**; however, the basic procedure is the same for all three alloy modes.

BASIC OPERATION

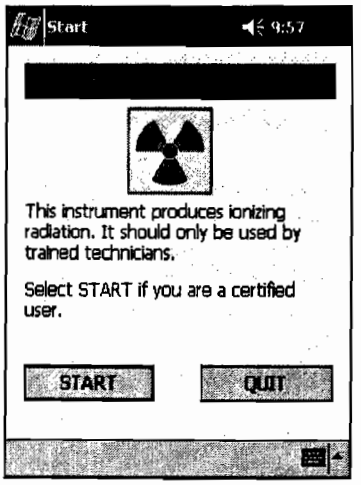
All Innov-X Systems Analyzers are shipped with a standard set of reference alloy standards that makes it possible to identify approximately 200 common alloys (35 in FastID). A list of the references in the library is provided in Appendix III. When you first receive your analyzer, it is recommended that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

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

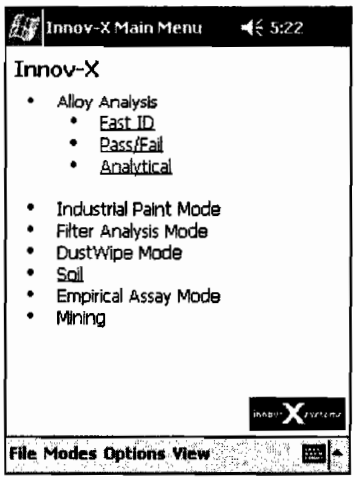
1. Install a freshly charged battery in the instrument.
2. Turn on the analyzer by pressing the power switch located at the back of the analyzer.
3. Verify that the iPAQ is correctly seated on the top of the unit. If the iPAQ is properly connected, the amber light on the upper right side of the iPAQ 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.



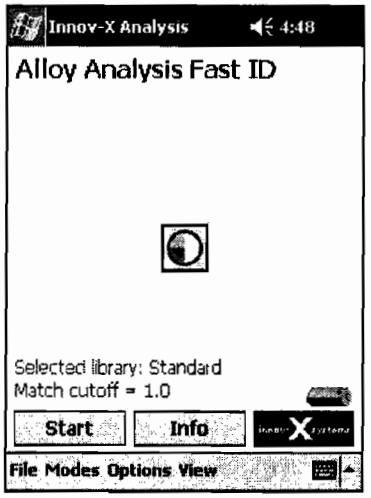
6. A notice will appear reminding the user that this instrument produces ionizing radiation and requires a trained user. . Select **START** to start the Innov-X Systems Software package. Selecting **QUIT** will exit the Innov-X Software.



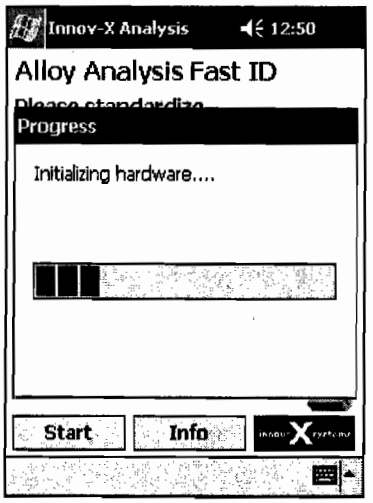
7. The Main Menu will open. Tap the name of the Mode you will be using to open it. First time users should Analytical Mode.



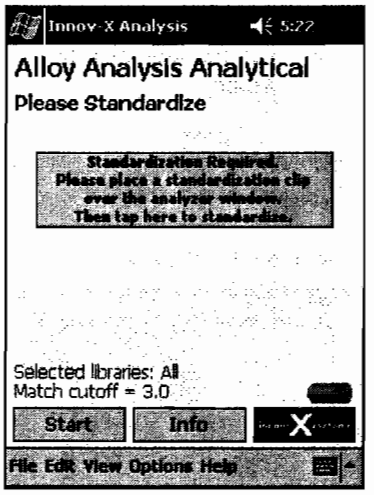
8. There may be a brief pause while the instrument loads the various parameters needed for operation. While this occurs, an icon will appear in the center of the iPAQ screen.



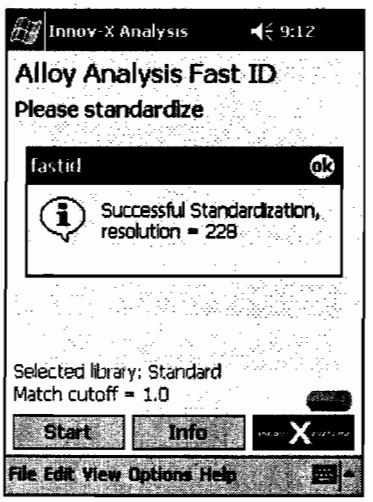
9. Once the analysis mode has been selected, the instrument will go through a 1 minute hardware initiation during which the electronics will stabilize and the detector cooling will be initialized.



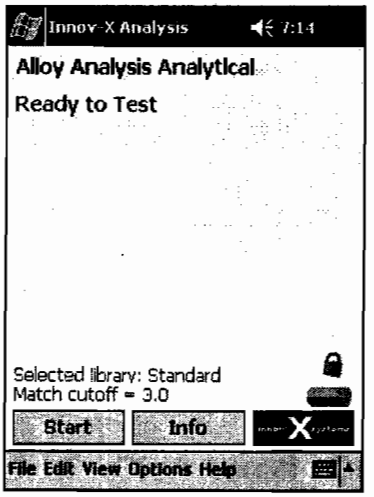
10. The message "**Standardization Required. Please place a standardization clip over the analyzer window. Then tap here to standardize.**" will appear. Standardization is required before testing can begin. Place the standardization clip in front of the analyzer window. Tap the message box. Standardization 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.**



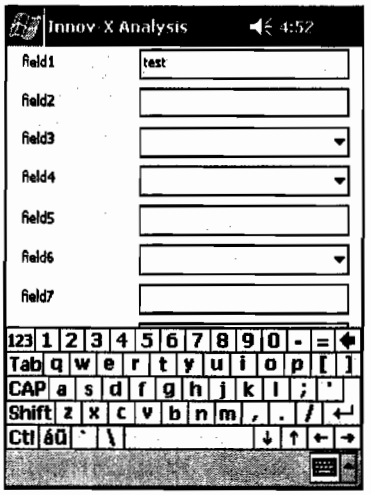
11. When standardization is complete, the resolution of the analyzer will be displayed. Tap **ok** to acknowledge and clear this screen.



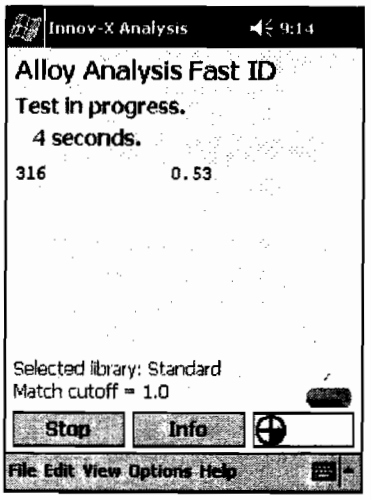
12. The analyzer is now ready to take a measurement. The Trigger lock must be unlocked before pulling the trigger will start a test.



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.

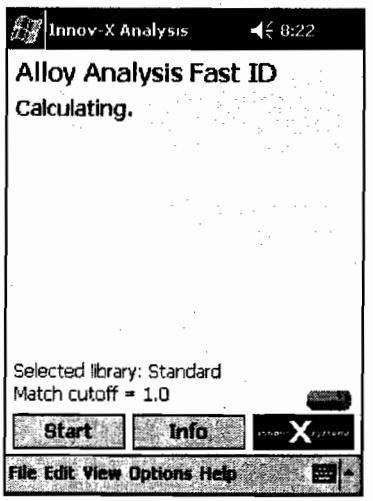


14. Hold the analyzer to the sample to be analyzed. Make sure the sample is as flush against the analyzing window as is possible. You may start an analysis by pulling and holding the trigger. Releasing the trigger will abort the test.

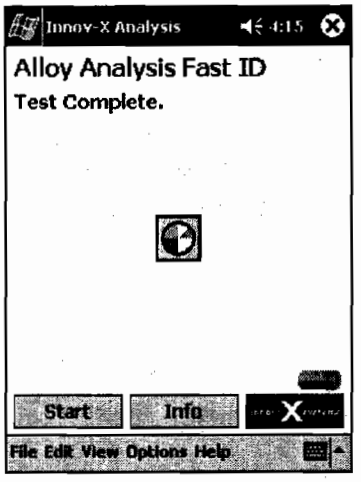


a. After an analysis is started, the message "**Test in Progress.**" will appear, followed by the number of seconds elapsed during the measurement. For the duration of the test, the red light on top of the instrument will blink, and the "testing" icon will appear in the lower right corner of the IPAQ.

b. 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.

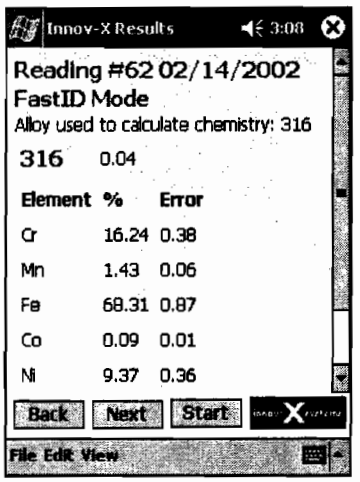


c. When the calculations are complete, there will be a slight delay the first time the results screen is opened. An icon will appear in the center of the screen during this delay. This indicates that the results program is loading and re-indexing all saved results.



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.

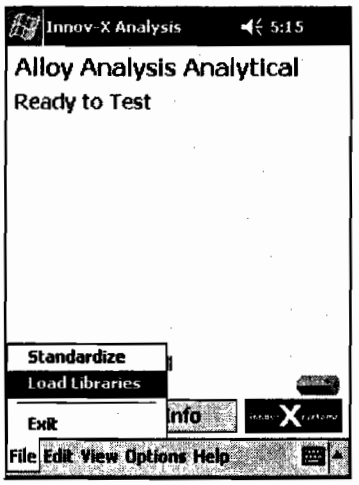
16. Once the results screen is open, subsequent readings may be started by depressing the trigger. If at any point, you wish to return to the analysis screen, select ***File→Exit*** or tap the X in the upper right hand corner of the screen.



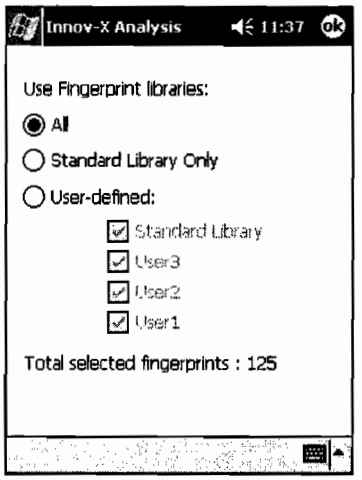
5.2 SELECTING A LIBRARY

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*** .



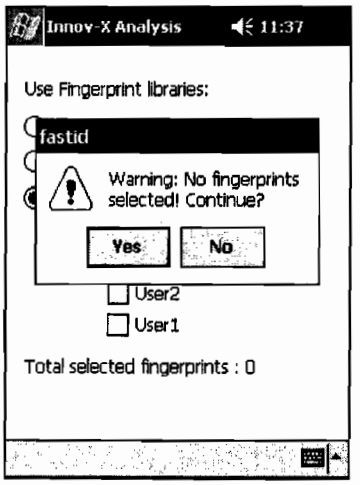
A menu appears. The first line will read **Use Grade Libraries** for Analytical Mode and **Use Fingerprints** for FastID Mode



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.

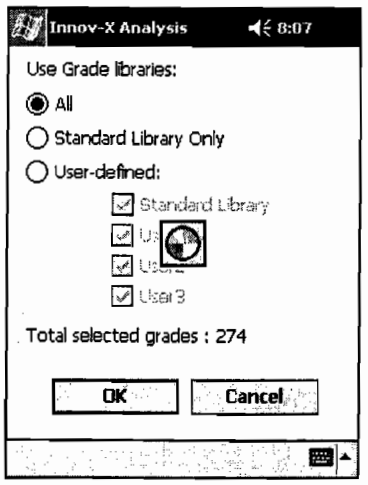


When loading libraries from the ***File→Load Libraries*** Menu, the number of fingerprints or grades in the selected libraries is displayed at the bottom of the screen. For best results especially when measuring complete unknowns, a large number of fingerprints should be selected because the analyzer 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.

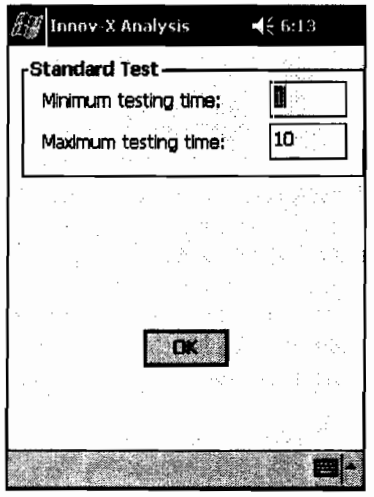
The test will end automatically when the maximum testing time is reached. A test can be ended manually at any time by releasing the trigger.

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.

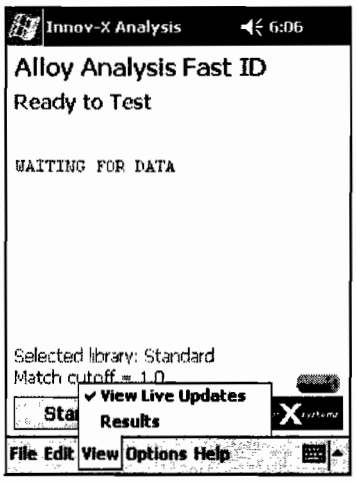
The maximum testing time will determine the length of a test. The analysis will automatically stop if the maximum testing time is reached. Normal maximum testing times will range from 5 to 20 seconds. Fundamentally, if the goal of the analysis is primarily grade identification, shorter analysis times 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.

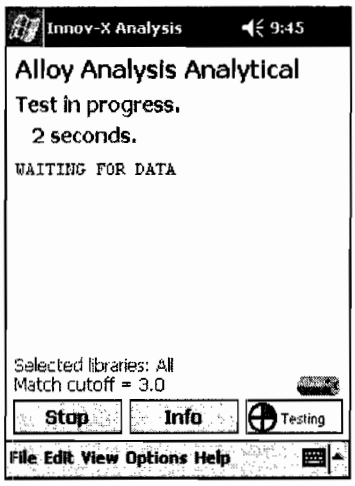
5.4 LIVE UPDATE DISPLAY

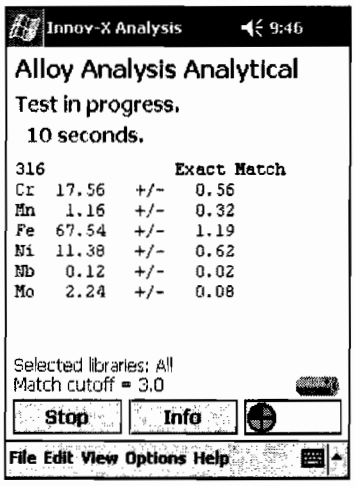
In addition to viewing completed tests in the results screen, the analyzer will display screen updates as the results are calculated during a test. This allows users to decide when to stop tests based on the precision of the reading.

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 then 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 6
Alloy Analysis—FastID Mode

6.0 INTRODUCTION TO FastID MODE

FastID mode is designed to quickly identify an alloy by comparing and matching the spectral signature of an unknown sample to spectral signatures of reference standards stored in a library. This mode can provide alloy chemistry if concentration data is entered for the standards. These chemistry data are a linear extrapolation from standard intensity data.

The system is typically shipped with a library of approximately 35 stored spectral "fingerprint" reference standards and chemical assays for these standards. Some systems may be shipped with additional fingerprints, or with no fingerprints, depending on the details of the order. To identify one of the alloys contained in the standard library, no user calibration or input is required. The operator simply tests a sample, and the instrument determines the correct alloy grade by matching to the library of spectral fingerprints. The instrument then calculates the chemistry by using the stored elemental assays using the identified grade as a reference standard. The user may easily add up to 300 additional reference standards and assays if desired.

Warning: Because **FastID** Mode performs a spectral match to a library of reference standards, it is very important that a stored reference standard be in the FastID library that is being searched. For this reason, this mode is best suited for alloy verification, where in general the operator is checking that the alloy is the expected type. Thus, the operator can assure before commencing testing that a reference standard for that alloy is in the library.

FastID is ideal for most PMI, in-service PMI and QA/QC applications. This mode offers the best combination of speed and precision. By matching the sample spectra to a stored spectrum from a certified standard, the analyzer is able to make its chemistry calculation based on a calibration file created especially for the matching grade. This spectral matching technique offers the best combination of speed and accuracy. Some of the advantages of **FastID** mode include:

- Provides a grade and chemistry in as little as 5 seconds. This is the best precision for shortest test time, when compared to other modes. Matching the sample's spectral fingerprint first allows FastID to select from hundreds of certified spectra and use the single best set of calibration data for that sample. The benefit is the fastest AND most precise chemistry calculation.
- Operator may add up to 300 additional alloy grades and assays (alloy chemistries) — password protected.
- Instrument offers 3 separate grade libraries for multiple users, or multiple departments. User can choose to search one or more libraries.

6.1 ANALYZING A SAMPLE

Once the instrument is standardized (see **Section 4.4: Standardization**), your analyzer is ready for routine measurement.

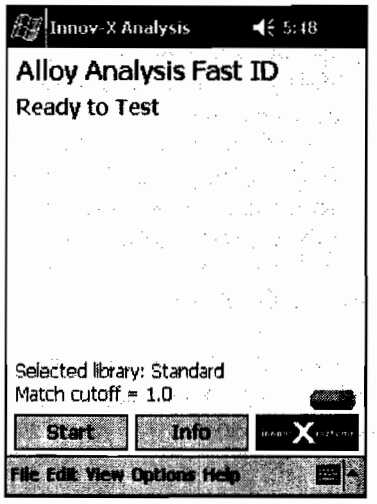
1. Hold the analyzer up to the sample to be tested. Make sure the part of the sample you wish to analyze is in contact with the window on the front of the probe.
2. Unlock the trigger by tapping on the icon located on the iPAQ Screen directly above the battery indicator. Select Yes when prompted.
3. Pull the trigger to start the measurement. The trigger must remain depressed for the duration of the test.

4. While the analysis is in progress, the red LED on top of the instrument will blink, and the screen will display **Test in Progress.** In addition a "testing" icon will appear in the lower right hand corner of the iPAQ screen. All these indicators show that the X-ray tube is energized and the shutter is open. During the testing time, it is important to keep the analyzer flush with the sample surface.
5. A test can be aborted at any time by releasing the trigger.
6. Once the measurement is complete, the results screen will automatically open. The results will be displayed in one of three forms, depending on the view that is selected.
7. Subsequent measurements can be started from the Results screen by pulling and holding the trigger.

6.1.1 Troubleshooting

There are certain parameters that must be correct in order for the analyzer to take a measurement. The instrument must be set to search a library containing fingerprints, and the match number must be valid. The Analysis Screen displays the match number cutoff and selected libraries. This information is shown in green lettering, at the bottom of the screen.

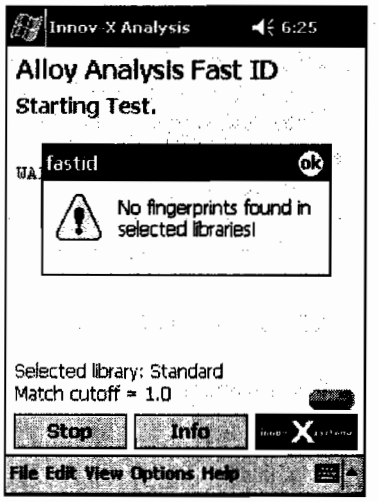
The selected library(ies) are listed. This is designed to allow the user to quickly verify that the correct library is being searched. The display will show "All" if all libraries are selected, or the names of the selected libraries. To change to Selected Libraries, choose ***File→Load Libraries***. It is recommended that all libraries are selected, unless it is necessary to search only a subset of stored fingerprints or grades.



The Match number is displayed to provide a quick check that the match number is set correctly. The factory set value for match numbers in FastID is 1. To change the match number, select ***Options→Fingerprint Settings***. More information about Match numbers can be found in the Advanced Features at the end of this section.

No Fingerprints Selected

It is impossible to start a test if no fingerprints are selected. If this is attempted, the following error message will result. Tap **ok** to acknowledge and clear the message box and select ***File→Load Libraries*** to choose libraries containing a valid number of fingerprints. See **Section 9.1.2: Loading Libraries** for more information.



Match cutoff too low

If the match cutoff is set too low, the instrument may fail to match alloys. Typically, the FastID match cutoff is set to 1. See the advanced features section at the end of this section for more information.

6.2 RESULTS DISPLAY

There are 3 possible ways to view information in the results screen; a user can view the Grade ID, the Grade ID and Chemistry, or the Raw Spectral Data. In addition, any test information entered for a reading may be viewed.

Regardless of what information is viewed, all results screens have similar characteristics. The **Date** and **Reading number** are shown at the top. Reading Numbers are useful for identifying readings. The first reading of a day will always be reading #1, thereafter; all readings within that day are labeled sequentially. Below the Date and Reading Number, the **Mode** used to acquire that reading will be listed.

Three buttons appear at the bottom of the screen: **Back, Next** and **Start**. **Back** and **Next** are used to scroll through stored data. The Start and Stop buttons are not active in most cases, and are normally only used in conjunction with a testing stand.

The results screens will show one of three possible results:

Successful Match
If an unknown alloy is a match to one of the grades contained in the Fingerprint libraries, a Grade ID will be shown.

Multiple Matches
In some cases, more than one grade will be shown as possible matches. This indicates that there was not enough statistical information to definitively separate two or more alloys, or the alloys are simply too similar to separate with portable XRF. The actual identification of the unknown alloy may be any one of the two grades listed.

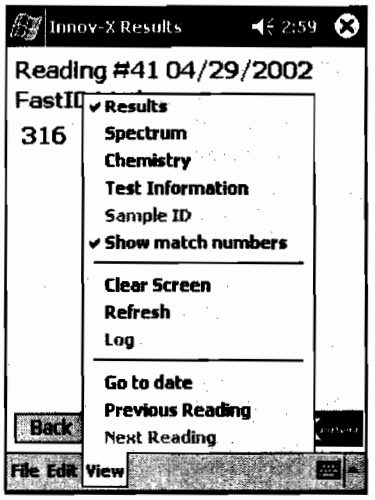
There are two procedures that may make it possible to separate the alloys:

1. Use a longer testing time. This will improve the measurement precision and may allow the alloys to be separated.
2. Define a specific element or elements that are different in the two alloys, which the analyzer can use to refine the identification. Please see Advanced Features later in this section for instructions to do this.

No Match
If no matches are found to the library, the words "no match" will be shown on the screen. If this occurs there may be several causes:

a. The alloy is not contained in the fingerprint library. Try using Analytical Mode to test the sample. Analytical Mode can provide chemistry information without requiring a library match. Alternatively, it is possible to add a standard to a library. This is described in Chapter 5.
b. The alloy may be coated. Try grinding or filing or sanding away any coating and repeat the test.
c. The testing time was too short. Trying increasing the testing time and measuring the sample again.
d. The match number is too low. See "Setting the Match cutoff" in the Advanced Features section of this chapter.

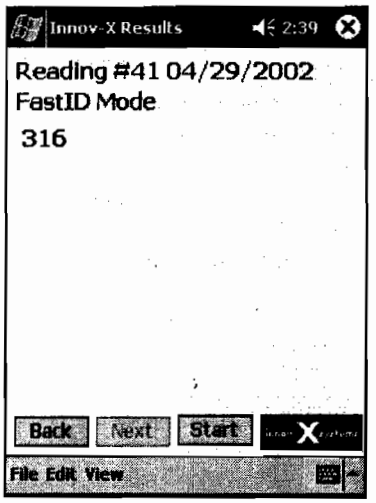
To change the appearance of the Results Screen click *View*, and select one of four choices: **Results, Chemistry, Spectrum** or **Test Information**. The active view will be denoted with a check mark.



6.2.1 Results Screen

This is the simplest screen. It displays only the Grade ID, and no extraneous information. This view is recommended if the primary goal of the analysis is grade identification, and it is not necessary to examine the chemistry results for every test.

Typically, one or more Grade IDs will be shown, or the words "No Match." In some cases, a number may be displayed after the grade identification. This is known as the match number. This number can be eliminated by Selecting ***View→Show Match Numbers***. This selection acts as a toggle. If ***View→Show Match Numbers*** is checked, match numbers will be displayed. Match numbers are described in greater detail in the Advanced Features part of this Chapter.

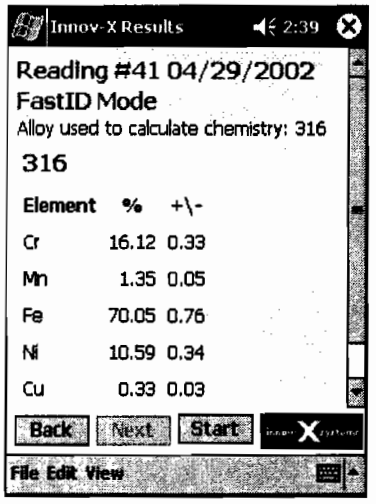


6.2.2 Chemistry Screen

This screen is the most versatile as it displays the Chemistry values, the uncertainty in the measurement and the Grade Identification. The Grade is listed at the top of the screen. The concentration (as a %) for all detected elements is then listed. After each concentration, the uncertainty in the measurement is shown. This is calculated as the 1-sigma error on the counting statistics.

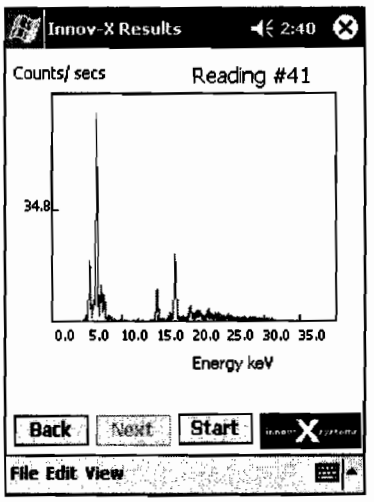
In cases where two or more matches shown, the results are based only on the first match listed. As a result there is some uncertainty associated with the chemistry values; however, given that two alloys are too close to separate, the chemistries should be fairly accurate.

Since **FastID** mode calculates chemistry results from standard assays, it cannot calculate chemistries for alloys that do not have assays stored in the fingerprint library. If no chemistries values have been entered in the library for any particular match, the message "Chemistry Values not calculated" will appear on the screen.



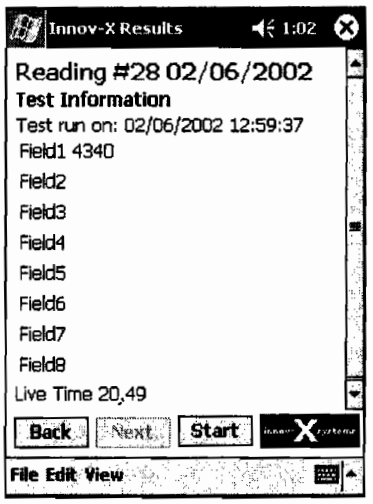
6.2.3 Spectrum Screen

This screen displays a plot of the x-ray fluorescence spectrum for an individual test, plotting the number of counts on the y axis versus the energy of the fluorescence x-rays on the x-axis. This spectrum is a unique energy signature resulting from the unique chemistry of the sample being tested.



6.2.4 Test Info Screen

The test information screen shows any information that was entered before starting a test.



6.3 ADVANCED FEATURES

This section we reviews two advanced features:

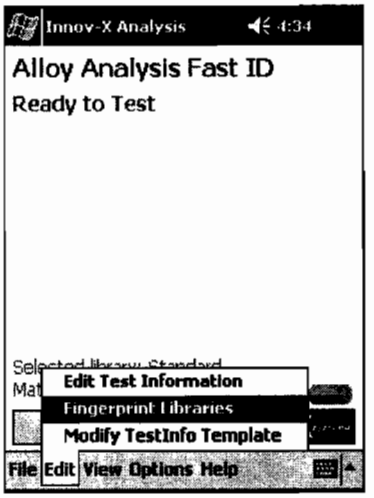
- Using Specific Regions to Separate Similar Alloys:
- Modifying the Fingerprint Cutoff Value

6.3.1 Specific Regions

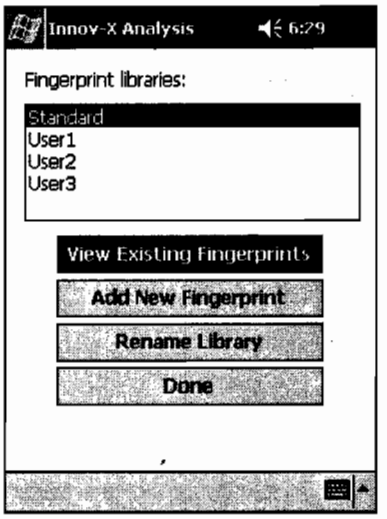
Some alloys only differ by a small concentration of a single element. For these alloys it may be necessary for the analyzer to examine the specific element or elements when performing a spectral match. In most cases, alloys that are difficult to separate when looking at all the elements may be easily separable if the analyzer is told to focus on one or more specific regions.

In order to instruct the analyzer to use one or more specific elemental regions, perform the following steps. This procedure can also be performed when reference standards are added by the user. For these instructions please refer the section on Adding a Fingerprint in Chapter 9. It is important to define the same specific elemental regions for all close alloys.

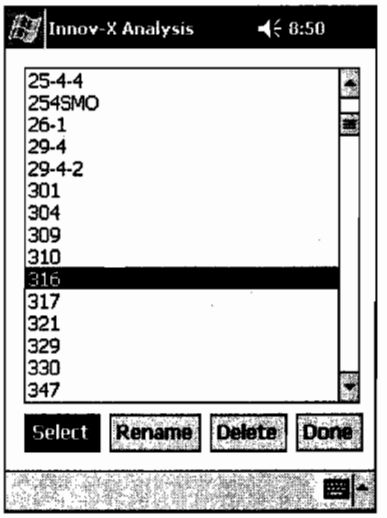
1. From the Analytical Menu, Select ***Edit→Fingerprint Libraries*** from FastID.



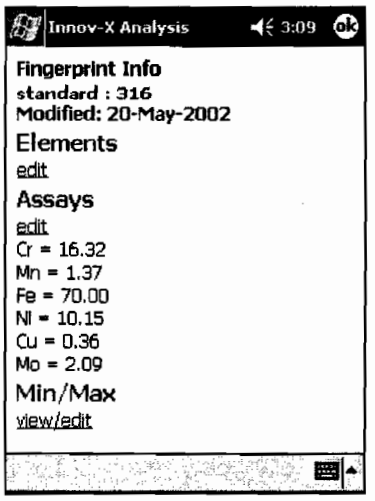
2. Select the library containing the alloy and click **View Existing Fingerprints.**



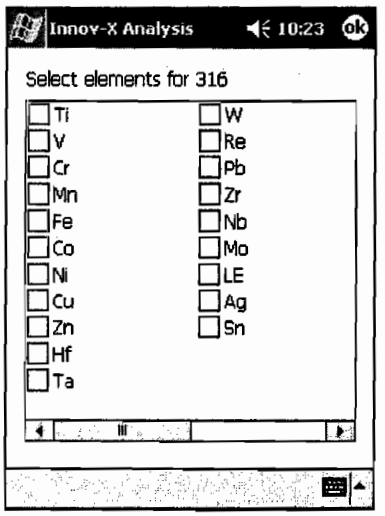
3. A list of the entries contain in the selected library will appear. Select the desired fingerprint.



4. Click the **edit** option underneath the heading **Elements**.



5. Tap the box for the element or elements you want to use for specific regions. A check will appear in that box. To disable this element, tap the element box again. The check will be removed. Select **ok**.



6.3.2 Modifying the Fingerprint Cutoff

In FastID Mode, the alloy matching is performed by comparing the x-ray spectrum of the sample to a library of reference spectra. Provided the sample spectrum is a close enough match to one or more reference spectra, the alloy grade(s) of the reference spectra is (are) displayed as the match. To determine a good match, a fingerprint match number is calculated by the analyzer by comparing the sample spectrum to the reference spectra. If the match number is below a pre-set cutoff value, then the sample is displayed as a good match to that particular reference standard. Ranges of match numbers are:

< 0.2	Excellent match
0.2-0.5	Good match
0.5-1.0	Reasonable match
1.0-2	Poor match
> 2.0	Sample is very different from reference standard.

Note: Factory default value = 1

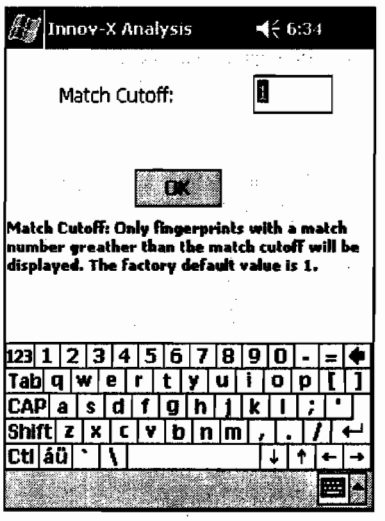
Generally, alloys within the same grade produce either excellent or good matches. Variations in match number occur because the reference standard has a slightly different chemistry than the sample being tested, even if they are the same grade. This is due to the usual grade-to-grade variations in the alloy chemistry. Testing small parts or parts with some surface oxidation may also elevate the match number.

Reasons to change the Fingerprint Cutoff:

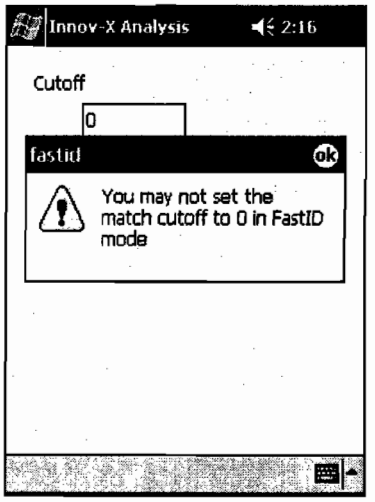
1. For sorting easily separated alloys such as 304 and 316, or alloys from different alloy bases, it is generally ok to raise the cutoff. This may be desirable if there is surface contamination like dirt, oil or mild oxidation, very small components like weld wire or turnings, or other things that may elevate the match number. It is advisable only to do this if the alloy sort being performed is known to be very easy for XRF technology.
2. The match cutoff may have been set to an extremely low number for a specific separation. If the match cutoff is too low, only very close matches will pass. Otherwise the result NO MATCH will be displayed.

How to Modify the Fingerprint Cutoff Value:

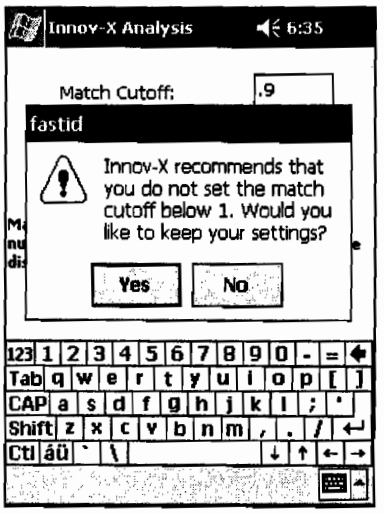
To change the Match number cutoff, from the Analysis Screen, select ***Options→Fingerprint Settings.*** The screen shown at the right will appear. Highlight the number shown in the box, tap the keyboard icon to bring up a keyboard, and enter a new cutoff value. Then tap OK.



If zero is entered for a match number, an error message will appear. Select ok to clear the message box, and select a valid match number. It will not be possible to close the Set Testing Times window with zero selected as a match number.



If the Match number is set to be a value below 1, a warning message will appear. Selecting **Yes** will keep the setting. Selecting **No** will allow the user to choose a new match number. Remember, the factory recommended setting for Match Number in FastID mode is 1.



Chapter 7 Alloy Analysis—Pass/Fail

7.0 INTRODUCTION TO PASS/FAIL MODE

Pass/Fail mode is designed for high-throughput alloy sorting and quality control. All sorting is done by comparison to a reference standard which is chosen by the operator.

Analyzed samples are compared to the reference samples, and results are displayed as a PASS or a FAIL, depending on whether they match the reference standard. Pass/Fail criteria may be based on the quality of fit to the spectral fingerprint or on elemental chemistry. Pass/Fail ranges may be implemented for one or more elements. This mode offers a full range of options from the simplest sorting of mixed loads in a recycling facility to QC on specific element(s) in even the most complex superalloys.

Two options exist for Pass/Fail mode: **Fingerprint** and **Chemistry**. Fingerprint matching should be used when the goal of the analysis is to determine whether or not analyzed samples are a specific grade. Chemistry should be used when it is important to determine whether the chemistries for specific elements fall within specified min/max grade specifications.

Fingerprint Pass/Fail uses the same method used in FastID to determine matches. Data from analyzed samples are compared to the fingerprint of a reference standard. If the differences between the fingerprints are small enough, the sample is judged to be of the same grade as the reference sample. This method requires only that a valid fingerprint for the reference standard is contained in the library.

Chemistry Pass/Fail. In addition to requiring that an alloy matches the fingerprint of a specific alloy, it requires that all detected elements fall within specified ranges.

The process followed in chemistry Pass/Fail is as follows:

1. Analyzer uses the fingerprint method to determine whether the sample matches the reference sample. If it does not, it automatically fails.
2. In the case of a match in step one, the chemistry of the alloy is calculated from assays stored for the standard fingerprint.
3. The calculated chemistry for each element is compared to the values stored in a Grade Table. In order for a sample to pass, all the chemistries must be within n standard deviations of the min and max values specified in the grade table, where n is a number which can be specified by the user.

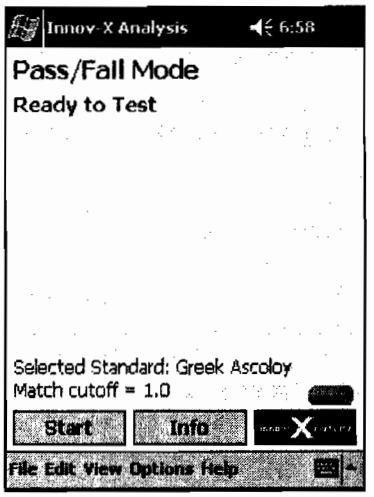
This method requires that a valid fingerprint, assays for that fingerprint, and Min/Max values are saved in the library.

7.1 USING PASS/FAIL

Before starting Pass/Fail mode, make sure that a signature for the reference sample you will be using is stored in the Fingerprint Library. You may use a fingerprint stored in the standard library, or you may add your own standard. To add a standard to the fingerprint library, follow the procedure listed in Chapter 9.

To start Pass/Fail mode, select Pass/Fail from the Main Menu. As with all modes, you will be informed if you must standardize the instrument before you can proceed with a measurement. Refer to the Chapter 4 for information on starting the analyzer and taking a measurement.

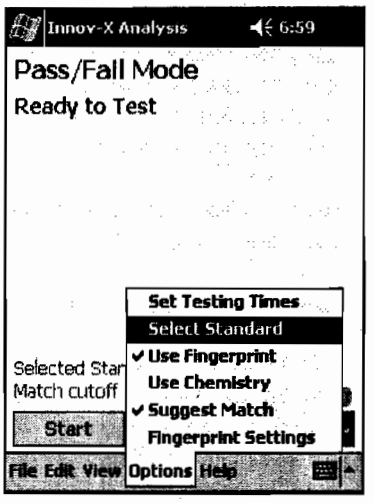
The Pass/Fail screen always shows the Selected Standard. This is the reference sample to which all other samples will be compared. Upon opening the mode, verify that the correct standard is selected.



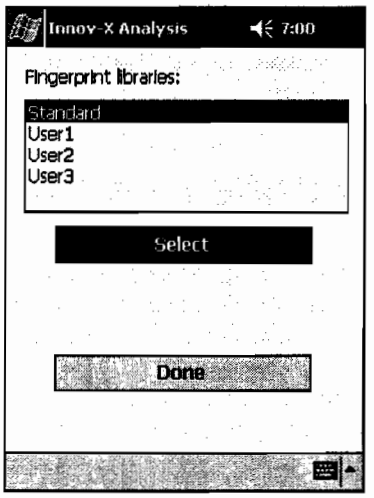
7.1.1. Selecting a Reference Standard

Once the standard you want to use is stored in a fingerprint library, select ***Options→Select Standard.***

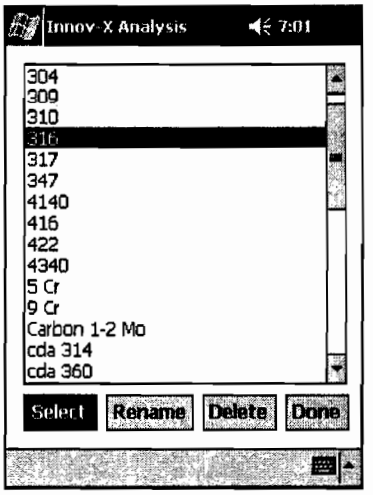
Enter your administrative level password when prompted.



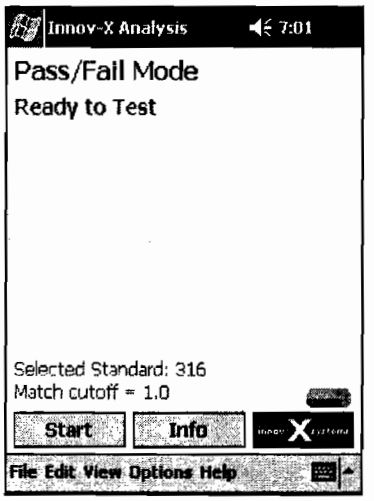
Select the library in which the fingerprint of your standard sample is stored. Choose the library and click **Select**.



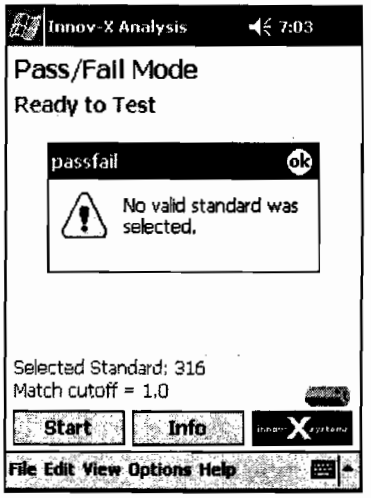
Select the name of your reference standard and click **Select**.



Verify that the proper standard is on displayed on the screen.



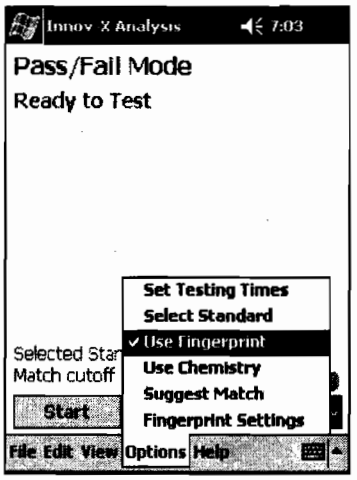
If you fail to select a standard, the instrument will always default to the last standard used. If a valid standard has not been selected, a warning message will appear. It will be impossible to start a test until a standard has been selected.

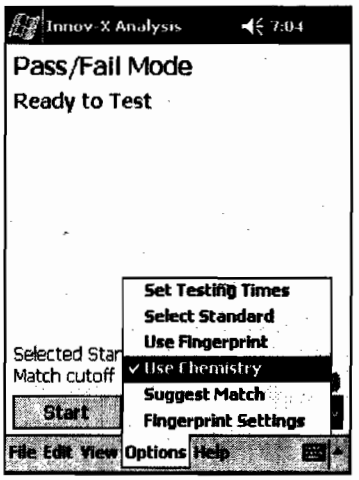


7.1.2. Select Pass/Fail Method

To use the fingerprint method and simply compare unknowns to a stored fingerprint, select ***Options → Use Fingerprint***.

Select **Options → *Use Chemistry*** from the menu if your Pass/Fail criteria will be chemistry-based.





7.2 ANALYZING A SAMPLE

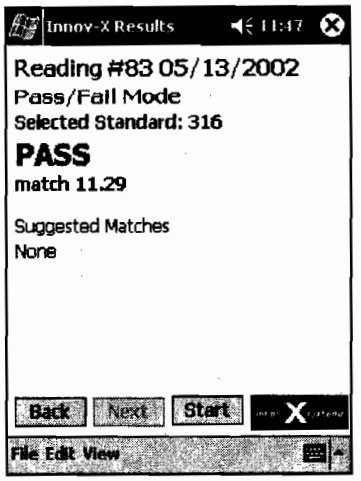
Once the instrument is successfully standardized as is described in section 4.4, your analyzer is ready for routine measurement.

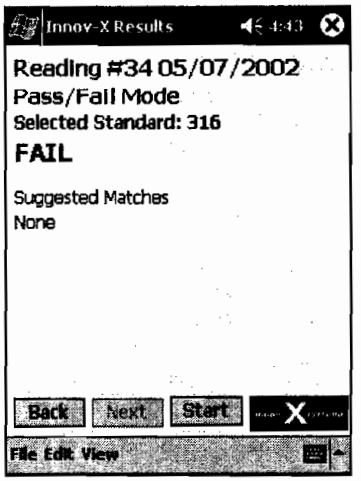
1. Hold the analyzer up to the sample to be tested. Make sure the part of the sample you wish to analyze is in contact with the window on the front of the probe.
2. Unlock the trigger by tapping on the icon located on the iPAQ Screen directly above the battery indicator. Select Yes when prompted.
3. Pull the trigger to start the measurement. It is necessary to hold the trigger for the duration of the measurement.
4. While the analysis is in progress, the red LED on top of the instrument will blink, and the screen will say "Test in Progress." In addition a "testing" icon will appear in the lower right hand corner of the iPAQ screen. All these indicators show that the X-ray tube is energized, and the shutter is open. During the testing time, it is important to keep the analyzer in contact with the sample surface, and to keep all body parts away from the measuring window.
5. A test can be aborted at any time by pulling the trigger, or by tapping **Stop** on the iPAQ screen.
6. Once the measurement is complete, the results screen will automatically open. The results will be displayed in one of two formats, depending on the view that is selected.

7.2.1 Use Fingerprint

Since Pass/Fail by fingerprint requires only that a valid fingerprint is selected, the analysis is very straightforward. Taking a measurement will open the results screen which will display the results of the measurement.

The results for Pass/Fail using Fingerprint comparison is very simple. Choosing ***View→Results*** will show only the reading, number, date, mode, selected standard and the result of the test; either the word **PASS** or **FAIL**. In the event of a failure, if the Advanced Feature: **Suggest Match** is enabled; possible Grade IDs will be displayed on the screen.

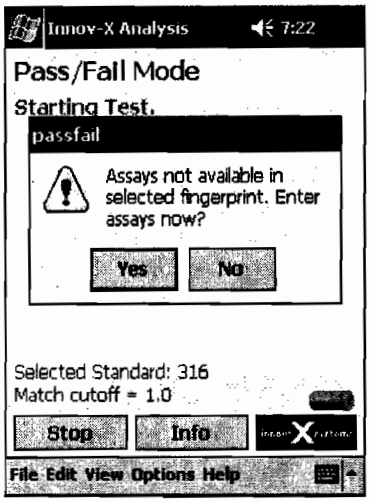


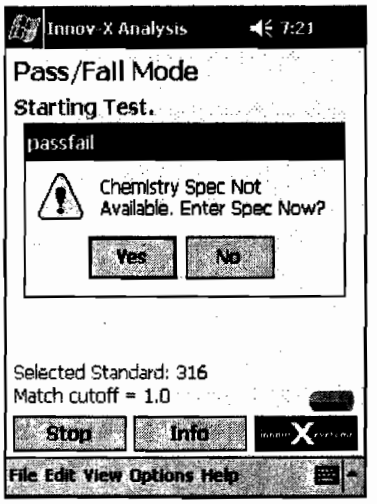


7.2.2. Use Chemistry

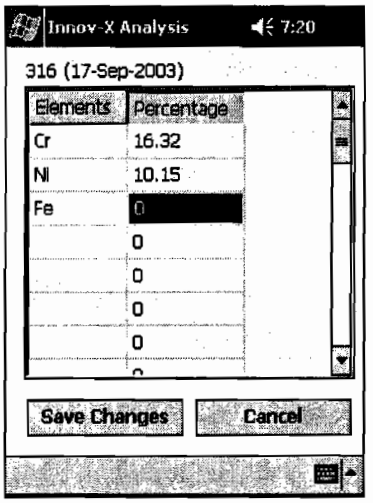
In order to get a valid reading with Use Chemistry selected, a fingerprint with an assay, and a grade table must exist. Since multiple parameters are required, it is strongly suggested that a known alloy grade is tested to make sure all parameters are set up correctly.

If a parameter is missing for a selected standard, a warning message will appear when the test is started indicating what needs to be added:

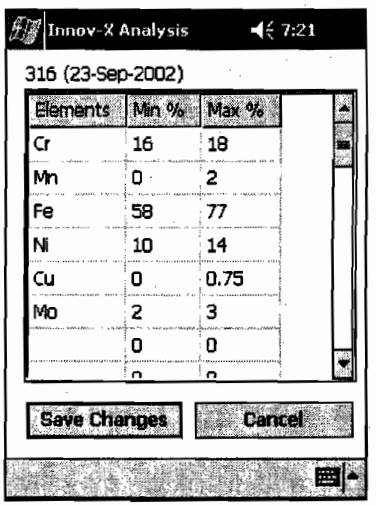




Selecting **Yes** to either of these screens will bring up either the Assays, or the Grade tables screen. Enter standard values in the assay table, and allowable Min/Max specifications in the Grade Table screen. Further information on either of these screens can be found in Chapter 9.

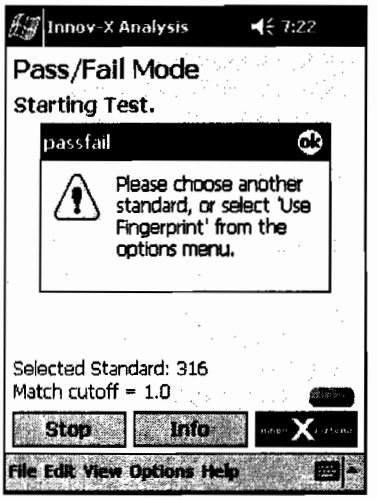


Elements	Percentage
Cr	16.32
Ni	10.15
Fe	0
	0
	0
	0
	0



Elements	Min %	Max %
Cr	16	18
Mn	0	2
Fe	58	77
Ni	10	14
Cu	0	0.75
Mo	2	3
	0	0

If you do not want to enter assays or grade libraries select No in response to the error message. You will not be able to take a reading until you either choose another standard or select Use Fingerprint.

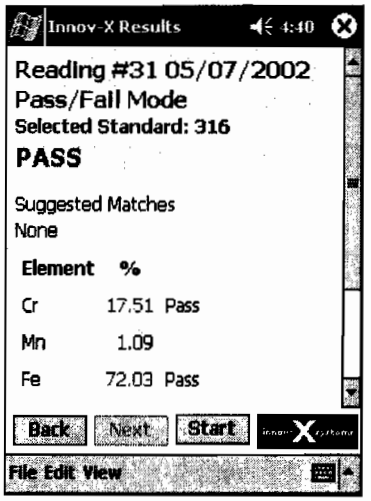


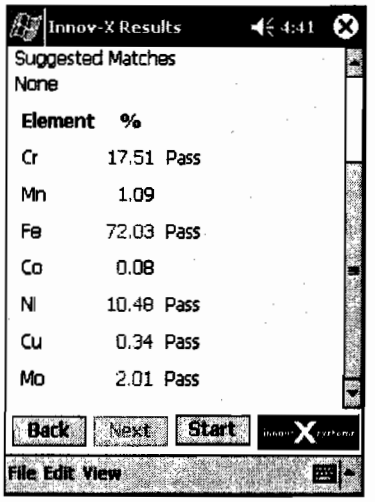
It is important that an assay is entered for every element that is listed in the grade table. If an assay is not listed for an element, the chemistry will not be calculated for that element. If this is the case, there will be no value to compare to the Min/Max spec. This will result in an automatic failure for every sample. Make sure that a standard sample is measured to ensure it is possible to get a PASS result.

Results—Use Chemistry

In addition to the PASS or FAIL results, the Chemistry results screen will show the calculated chemistry results. The percentage reading for each element will be color coded to show whether the value is within spec. A green color indicates that the reading is within specifications; a red color indicates the element is out of spec. If no Min/Max specification is provided for an element, the concentration will be shown in black. Any elements that don't have Min/Max specs will not be used as criteria for determining Pass or Fail.

If the list of elements exceeds the room available on the screen, it will be necessary to scroll down to see the complete list.

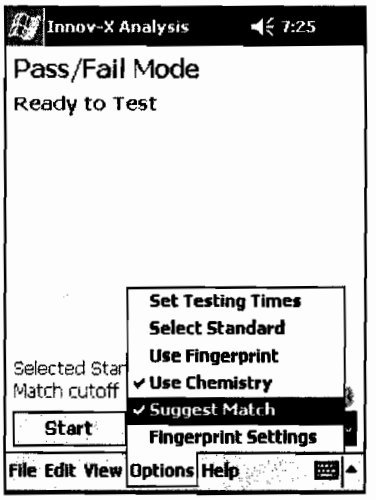




7.3 ADVANCED FEATURES

Suggest Match: Suggest Match is designed to provide information about a sample in the event of a **FAIL** result. When suggest match is enabled, the software will search the library and provide a list of one or more possible Grade IDs if the sample fails to match the selected standard.

To activate Suggest Match, Select ***Options→Suggest Match***. Once suggest match has been selected, it will remain active until it is deselected. A checkmark before the menu selection will indicate that the option is enabled.



If suggest match is enabled, the analyzer will search all active libraries for the sample(s) with the lowest match number. The match number is an indication of the degree to which the analyzed sample matches a library entry. The number is unit-less, and will vary depending on a variety of parameters. In general, the closer a match number is to zero, the better the match.

Suggested results will appear only in the results screen, not in the chemistry screen. The display will indicate a no-match, or display the suggested match. In many cases, a single match will appear. However, in cases where it is impossible to statistically separate two or more matches; all close matches will be displayed on the screen. Keep in mind that a suggested match is not necessarily a correct grade identification. It is simply the closest matching grade found in the fingerprint library.

Chapter 8
Alloy Analysis—Analytical Mode

8.0 INTRODUCTION TO ANALYTICAL MODE

Analytical mode utilizes a Fundamental Parameters (FP) algorithm to determine elemental chemistry. This method calculates chemistry from the spectral data, without the requirement of stored fingerprints. The Analytical FP calibration is done at the factory, and requires no user set-up or recalibration. The software also searches an alloy grade library to produce a grade match to calculated chemistry, based on the calculated chemistry. Analytical mode can provide a grade ID and chemistry in as little as 2-3 seconds, with increased precision for longer test times.

The FP method utilized in Analytical mode is ideal for applications that require analysis of proprietary or uncommon alloys, for monitoring chemistry of tramp elements, or for monitoring chemistry during processing. The fundamental parameters method is also ideal for obtaining an average chemistry of turnings, especially mixed turnings.

In many ways, analytical mode is the easiest to use of all the modes, since it is possible to acquire good solid chemistry data and grade identifications by using only the instrument default setup. Users who are analyzing complete unknowns or samples from a wide variety of alloy families may find this to be the most appropriate mode for their needs.

Determination of Grade Identification:

Analytical mode utilizes a Grade Library consisting of a set of minimum and maximum values for each element in an alloy. The factory installed standard grade library contains 250 alloy grades. The Operator may add up to 300 additional alloys. These alloys may be added to any of the 3 user libraries or to the standard Grade Library. For the sake of simplicity, we recommend any alloys added be added to the user libraries and not the Standard (Factory) library. The libraries may be searched individually or together. All libraries, including the standard library, may be edited by the user.

Analytical mode calculates chemical composition using a fundamental parameters algorithm. Once these values are calculated, they are compared to the grade tables stored in the grade library. The software calculates a number, called a Match Number, which is an indication of how close the chemistry of a measured alloy is to the library values. The lower this number, the better the match. A match number of 0 is an exact match, meaning that the calculated chemistry for all elements falls within the grade table specifications.

The presence of tramp elements, as well as uncertainty associated with any measurement, makes it possible for a valid match to fail to register as an Exact Match. Therefore, a cutoff value is set to determine whether or not a grade is considered a match. If the Match Number is below the cutoff value, it is considered to be a good match. The default cutoff value is set at a value that works for a wide range of alloys, but it may need to be adjusted for certain specialty applications.

8.1 USING ANALYTICAL MODE

A detailed startup procedure is described in chapter 4. The procedure is summarized here:

1. From the main menu, select Analytical Mode by tapping on the word "Analytical".
2. Hold the analyzer up to the sample to be tested. Ensure that the sample is as flat as possible against the window, and is covering as much of the window as possible.

3. Unlock the trigger by tapping on the icon located on the iPAQ Screen directly above the battery indicator. Select Yes when prompted.
4. Start the measurement by pulling the trigger. When a test is in progress, the words "Test in Progress" will be displayed, along with the elapsed time of the measurement and a "testing" icon. The LED on the front of the instrument will blink, indicating that the X-ray tube is on, and the shutter is open.
5. Make sure the sample maintains contact with the analysis window for the entire duration of the test.
6. When the test is complete, the words "Test Complete" will appear. For the first reading of the day, there will be a slight delay while the results screen is opened. This will be indicated by the appearance of a rotating icon in the center of the screen.
7. After taking the initial measurement, tests can be started from the results screen by pulling the trigger. This will close the results screen and display the analysis screen, which will show the progress of the test.

8.2 RESULTS DISPLAY

There are 3 possible ways to view information in the results screen; a user can view the Grade ID, the Grade ID and Chemistry, or the Raw Spectral Data. In addition, any test information entered for a reading may be viewed.

Regardless of what information is viewed, all results screens have similar characteristics. The **Date** and **Reading number** are shown at the top. Reading Numbers are useful for identifying readings. The first reading of a day will always be reading #1, thereafter; all readings within that day are labeled sequentially. Below the Date and Reading Number, the Mode used to acquire that reading will be listed.

Three buttons appear at the bottom of the screen: **Back, Next** and **Start**. **Back** and **Next** are used to scroll through stored data. The **Start** button is inactive in most cases, but may be used in conjunction with the test stand.

The results screens will show one of three possible results:

Successful Match
If an unknown alloy is a match to one of the grades contained in the Fingerprint libraries, a grade ID will be shown.

Multiple Matches
In some cases, more than one grade will be shown as possible matches. This indicates that there was not enough statistical information to definitively separate two or more alloys. The actual identification of the unknown alloy may be any one of the grades listed. Increasing the testing time may make it possible to separate the alloys.

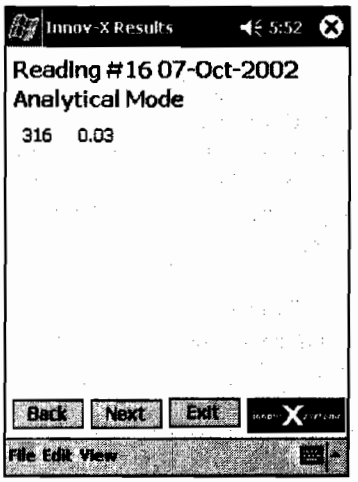
No Match
If no matches are found to the library, the words "no match" will be shown on the screen. If this occurs there may be several causes:

a. The alloy does not meet any of the specifications in the Grade Library.
b. The alloy may be coated. Try grinding or filing or sanding away the coating and repeat the test.
c. The testing time was too short. Trying increasing the testing time and measuring the sample again.
d. The match number is too low. See **Section 8.3.1: Nearest Match/Exact Match** for more information.

To change the appearance of the Results Screen click ***View***, and select one of four choices: **Results, Chemistry, Spectrum or Test Information**. The active view will be denoted with a check mark.

8.2.1 Results Screen

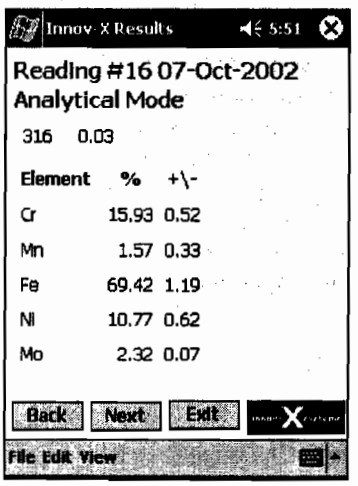
This is the simplest screen. It displays only the Grade ID, and no extraneous information. This view is recommended if the primary goal of the analysis is grade identification, and it is not necessary to examine the chemistry results for every test.



Typically, one or more Grade IDs will be shown, or the words "No Match." In some cases, a number may be displayed after the grade identification. This is known as the match number. This number can be eliminated by Selecting ***View→Show Match Numbers***. This selection acts as a toggle. If ***View→Show Match Numbers*** is checked, match numbers will be displayed. Match numbers are described in greater detail in the Advanced Features part of this Chapter.

8.2.2 Chemistry Screen

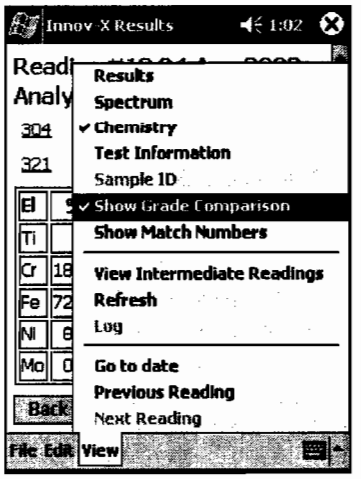
This screen is the most versatile as it displays the Chemistry values, the uncertainty in the measurement and the Grade Identification.



The Grade is listed at the top of the screen. The concentration in percent for all detected elements is then listed. After each element's chemistry, the uncertainty in the measurement is shown. This is calculated as the 1-sigma error on the counting statistics.

8.2.2A Grade Comparison

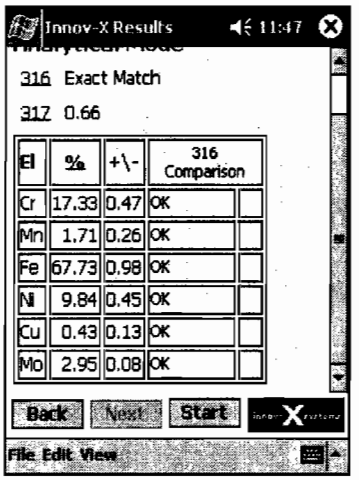
The Show Grade Comparison feature allows users to see at a glance which elements are outside the concentration range specified for a Grade Match. When this feature is activated the results will show up in a table format. If the chemistry of an element, plus or minus the error on the reading, is within the specified range for a specific grade, the word "OK" will appear next to that element. If the chemistry is out of range, the screen will indicate whether the measured elemental value is "low" or "high" and list the corresponding grade specification.



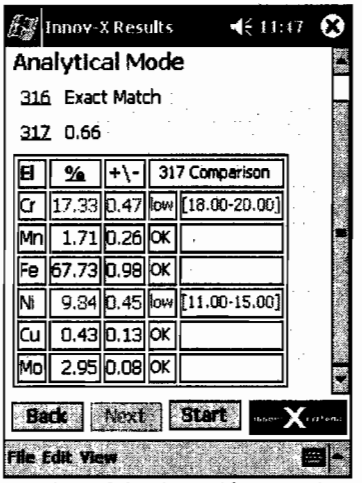
The show Grade Comparison function works only in the Chemistry view. If the results screen is not displaying chemistry results, select **View→Chemistry** from the results screen. Show Grade Comparison can be turned on or off by selecting **View→Show Grade Comparison** from the Results screen. A check mark will appear on the menu if the feature is active.

When Show Grade Comparisons is active, it is possible to show the comparison for any Grade that appears as a match for the analyzed sample. Select which grade comparison to view by tapping on the name of that grade.

The figures show the results for the measurement of 316. Both 316 and 317 show up as matches, however, 316 shows up as an "exact match" while 317 has a higher match number. The grade comparison shows that all the elemental concentrations are within specification for 316, while the Cr and Ni are both lower than specified for 317. Obviously, 316 is a better match and is the correct grade identification for the alloy.



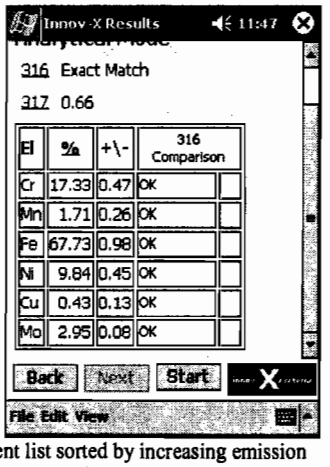
Grade comparison for 316. Select grade to compare by tapping on the name of the grade.



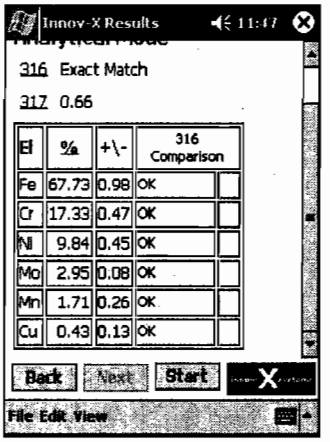
Comparison of chemistry with 317 grade spec.

8.2.2B Changing the order in which elements are displayed

Users may order detected elements in two ways; by emission line energy, or in order of decreasing concentration. To change the order, simply tap on the blue "%" header above the column displaying elemental chemistry. Tapping this header will switch the order. Tapping again will return the list to its original order.



Element list sorted by increasing emission line energy.

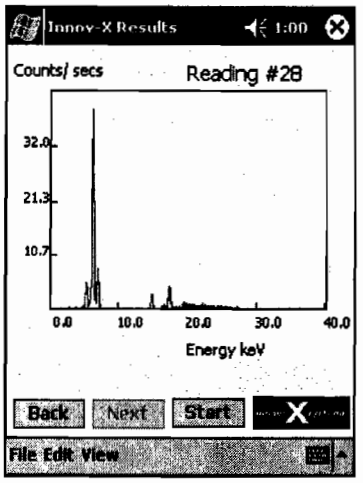


Element list sorted in order of decreasing composition.

8.2.3 Spectrum Screen

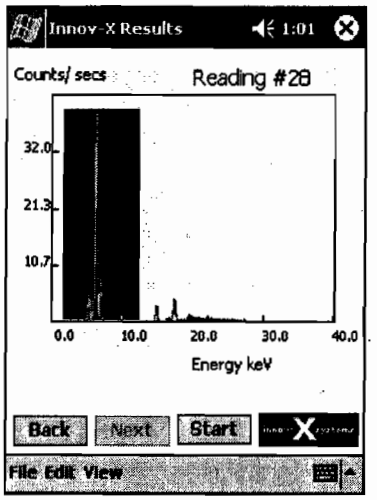
This screen displays a plot of the x-ray fluorescence spectrum for an individual test, plotting the intensity on the y-axis versus the energy of the fluorescence x-rays on the x-axis.

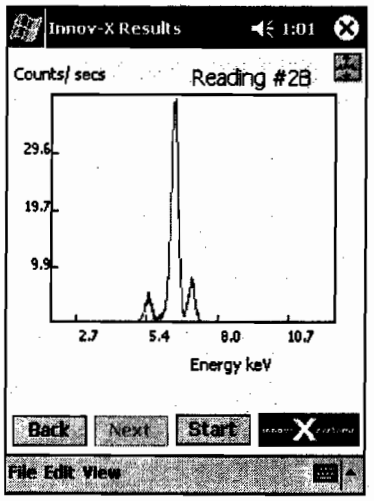
Tapping on the spectra will show the energy scale and count rate at the selected point.



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

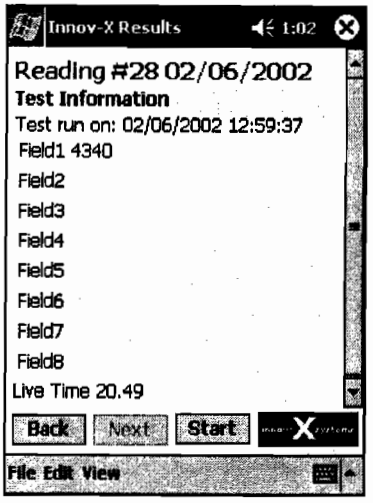
Tapping the symbol in the upper right hand corner beneath the X will restore the graph to full scale.





8.2.4 Test Info Screen

The test information screen shows any information that was entered in **Test Information** before the test was started.



8.3 ADVANCED FEATURES

8.3.1 Nearest Match/Exact Match

After calculating chemistry with the Fundamental Parameters algorithm, the analyzer software searches the Grade Library for alloy specifications which are close to the calculated chemistries. In order to evaluate which alloy is the best match for the measured sample, a number is calculated which compares the measured samples to the Grade Table. This number, referred to as a match number, is a measure of the difference between the measured chemistry and Minimum and Maximum specifications for the various alloys. A Match number of zero (0) indicates that measured chemistry is an "EXACT MATCH;" the measured chemistry for all detected elements falls within specifications. It is possible to have good matches that are not "EXACT," as there are uncertainties associated with any measurement. The presence of tramp elements (small amounts of unspecified elements) may also elevate the match number on valid matches.

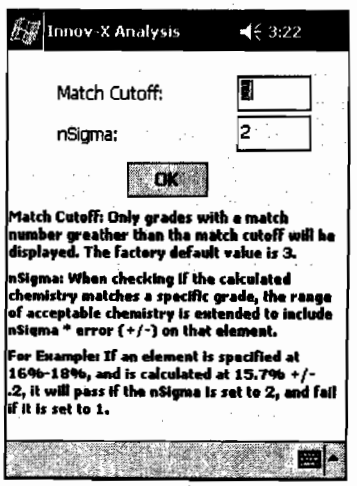
It is possible to specify whether the analyzer will report all possible matches, or just Exact Matches. These two options are referred to as Exact Match and Nearest Match. To switch between Exact and Nearest Match modes, select ***Options***. Clicking on ***Exact Match*** works as a toggle. If a checkmark appears before the words "Exact Match," Exact match is enabled. Otherwise, Nearest Match is the active method.

- **Nearest Match** When the analyzer is in Nearest Match mode, it calculates chemistries using the fundamental parameters algorithm, and searches the grade libraries and determines which alloy(s) is the closest match to the calculated results. The analyzer determines whether a grade is considered a match by comparing the calculated Match Number for that alloy to a cutoff value. This cutoff value is user modifiable. Typically this cutoff value is set at 3 and should not be changed, except in very special circumstances.

- **Exact Match** Exact match requires that all chemistry values are within a user settable error band of the min/max values specified in the Grade Libraries. The allowable error band can be changed by modifying the "nSigma" value. If nSigma is set as 0, all chemistry values must fall within the Grade Specifications. If nSigma is set as 2, for example, the chemistry value +/- 2*precision must fall within the Grade Specifications.

  Both the nSigma and Match Cutoff values are changed by selecting ***Options→Grade Library Settings.***

  A brief description of each field appears on this screen

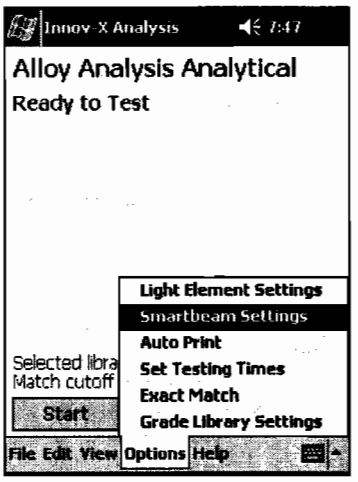


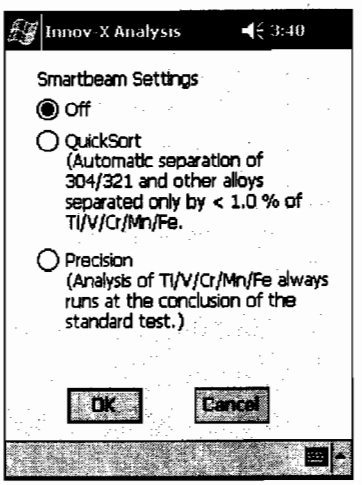
8.3.2 *Smartbeam* (optional feature)

Smartbeam adjusts the voltage, current and filtering to optimize detection of Ti and V in alloy samples. This feature is designed to provide quick, accurate separations of alloys which contain low concentrations (<1.0 %) of Ti and V, and to provide accurate and precise measurements of these elements.

Users can configure ***Smartbeam*** in two different ways: **Quicksort** mode provides automatic separation of alloys separated by a small amount of Ti or V. This mode *automatically* turns on ***Smartbeam*** and does a test with a second beam condition; if an alloy is found to match two alloy grades that only differ by a small amount of Ti or V. **Precision** mode forces ***Smartbeam*** on for every test in order to get precise chemistry on Ti and V at low levels. ***Smartbeam*** can also be disabled when it is not required.

Smartbeam settings:
To configure ***Smartbeam***, Select **Options→Smartbeam Settings** from the Analytical "Ready to Test" screen. A screen will appear providing three options.





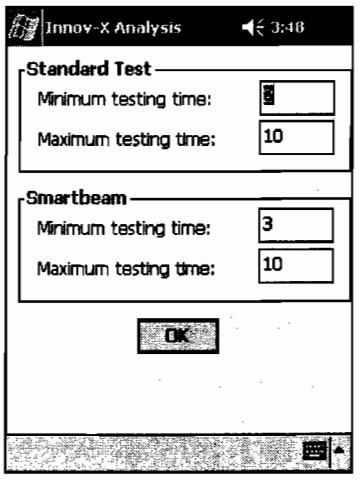
***Smartbeam* Off:**
If ***Smartbeam*** is disabled, the analyzer will perform a single, one beam analysis. This will separate most alloys; however a short test will not be sufficient to separate alloys which differ by a small amount of Ti or V. This is the recommended mode if the separation of alloys such as 304 and 321 is not required.

Quicksort:
Quicksort is the recommended ***Smartbeam*** setting for most applications. It allows for the quickest throughput of samples while maintaining the ability to separate alloys which require ***Smartbeam***. The analyzer automatically switches to a second beam condition when presented with a sample which closely matches two grades that differ by a small amount of Ti and/or V.

Precision:
This mode does a two beam test on all samples. Precision mode is only recommended for users who require precise chemistry on low concentrations of Ti and V. This is the most infrequently used ***Smartbeam*** option.

8.3.2A Setting *Smartbeam* Testing time

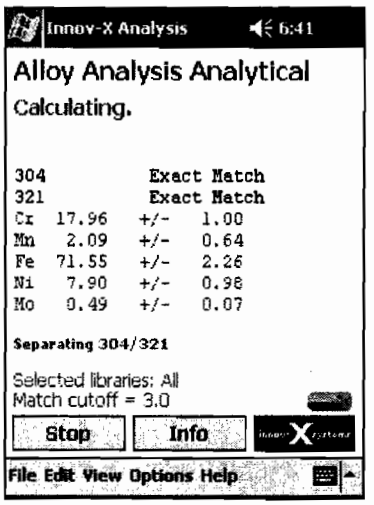
The Options **Set→Testing Times** screen for instruments equipped with ***Smartbeam*** allows users to set the smart beam testing times, as well as those for the Standard Test. Typical Testing times are shown.



8.3.2B Testing with *Smartbeam* Enabled

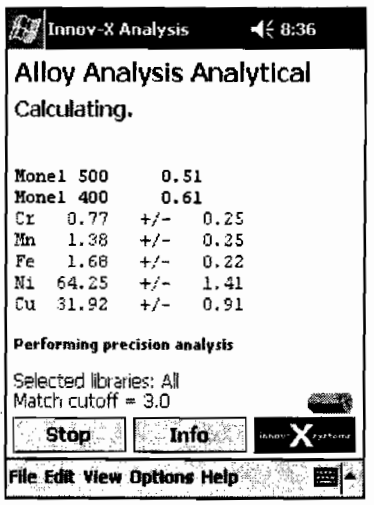
Once ***Smartbeam*** is enabled, users will operate the instrument exactly as they do when ***Smartbeam*** is disabled. Tests are started and stopped via the trigger. The analyzer will start by testing using standard beam conditions, then switch to the second beam condition automatically, if appropriate.

If Quicksort is active, the instrument will start a test using the standard beam settings. If a sample is found to match two alloys which differ by only a small amount of Ti or V, the instrument will switch to a 2nd beam condition. It will test using the second beam until the maximum testing time for beam 2 has elapsed. When the analyzer switches to 2nd beam conditions, it will display the name of the two alloys being separated.



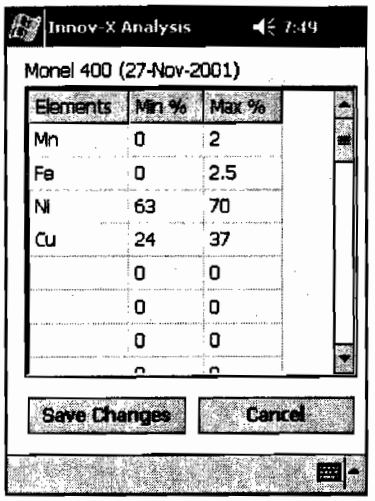
If the alloy has a unique ID, or if the best matches differ by something other than a small amount of Ti or V, the analyzer will function as it does with Smartbeam deactivated.

If Precision mode is active, the instrument will automatically switch to the second beam condition after the set time has elapsed for the standard beam. When the second beam is active the words "Performing precision analysis" will appear on the screen. The second beam will be active for the amount of time specified in the **Set Testing Times** screen, under the ***Smartbeam*** heading. At the completion of the test, a final result which reflects information from both beam settings will be displayed.

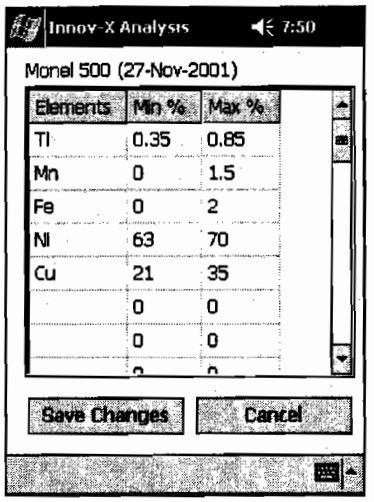


8.3.2C Creating alloy grades which utilize Quicksort *Smartbeam* Separation Mode.

When Quicksort is activated, the software uses the grade library to determine which alloys require a second beam analysis for separation. If the grade specifications for two alloys overlap completely, with the exception of Ti and/or V, Quicksort will use a second beam test. For example, the grade specifications for Monel 400 and 500 are shown below. They do not completely overlap, however, there are numerous concentrations which fall within the specifications of both grades. A second beam test will be required if a sample matches both of these alloys because Ti is specified in 500, but not 400.



Elements	Min %	Max %
Mn	0	2
Fe	0	2.5
Ni	63	70
Cu	24	37
	0	0
	0	0
	0	0



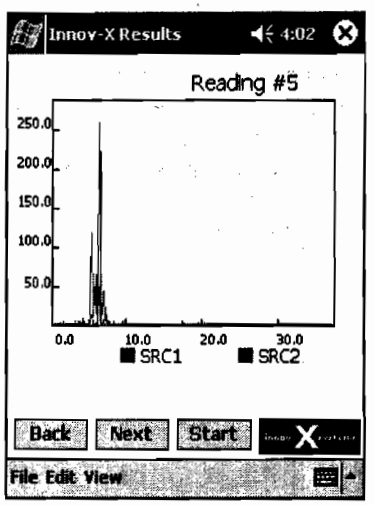
Elements	Min %	Max %
Ti	0.35	0.85
Mn	0	1.5
Fe	0	2
Ni	63	70
Cu	21	35
	0	0
	0	0

The most common grades requiring ***Smartbeam*** for quick separation; 304/321 and Monel 400/Monel 500 are included in the library. Users may add alloys to the library simply by entering the grade specifications. Quicksort will automatically use the second beam if a sample matches 2 alloys whose grade library specifications differ by <1% of Ti or V.

8.3.2D ***Smartbeam*** Results

The Chemistry results screen for ***Smartbeam*** tests is identical to that for standard tests. A single result is shown which includes information from both beams.

The Spectrum screen shows spectral overlays of data from both beams.



8.3.3 Light Element Analysis.

Innov-X Portable analyzers, like all hand-held XRF analyzers, cannot directly detect "light" elements such as Al, Si and Mg. In this manual, and in the software, light elements will be referred to as "LE" for light element. In practice Al is the most common light element of concern in alloys measured, so most of this discussion focuses on how aluminum is handled.

Innov-X analyzers handle light elements in two different ways.

1. **Use of nominal value.** All Analytical results are normalized to 100%. Since Al is invisible to the analyzer; the presence of several percent of Al can cause the other elements to appear too high after normalization. To correct for this, the software can normalize to 100% minus a nominal value for LE.

- Users should enter a nominal value for Al (or any other light element) as LE. If LE is specified for a grade, and the grade is found to be the best match for an alloy, the analyzer will use the average of the min and max specification as a nominal value. The measured results for all other elements will be normalized to 100% minus the LE nominal value.
- This is most useful for compounds which have less than 10% aluminum. This is typically used for titanium alloys and some copper alloys.

This method does not measure LE. It relies on data in the grade libraries. It's designed primarily to reduce the bias on other elements that can be caused by a significant amount of Al or other light elements. The nominal LE value will appear in blue to indicate that it is calculated, not measured.

2. **Calculation of LE using scatter lines.** A portion of x-rays from the x-ray tube are scattered back to the detector. The amount of scatter can provide additional information about the sample. In particular the amount of inelastic scatter, called Compton scatter is increased as the density decreased. As a result, the scatter lines can be used to indirectly calculate the LE concentration in low density samples, such as Aluminum alloys.
   - This method is recommended mainly for aluminum alloys, which typically contain more than 85% aluminum
   - The software will calculate the LE from the scatter lines, and all other elements from their x-ray emission lines. LE will be treated as any other element and included in the grade library search. Thus, Al alloys need to be included in the library for proper identifications.
   - Although this method can determine the amount of LE in an aluminum alloy, it is only sufficient to sort major grades of Al alloys. Many aluminum alloys cannot be separated because they differ by small amounts of Si, Mg or other light elements which cannot be measured. Please contact Innov-X System for more information on the capability of the analyzer for testing Al alloys.

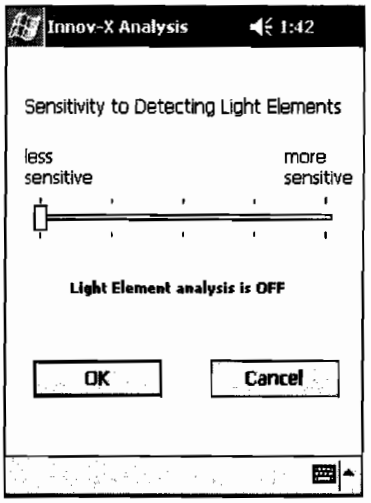
Since the calculation of LE is done indirectly, LE can be reported erroneously in some conditions if the amount of scatter is increased. For example, some customers who produce specialty alloys wrap the samples in plastic before testing. This scatter from the plastic will be erroneously reported as LE. Likewise, irregularly shaped or heterogeneous samples can, on occasion, produce false LE readings.

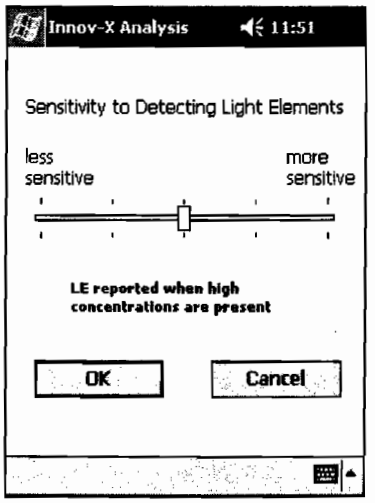
For these reasons, Innov-X allows customers to adjust their analyzer's sensitivity to Light Element detection. To change the LE sensitivity, select ***Options→Light Element Settings***.

If aluminum alloys are never measured, the slider bar should be positioned all the way to the left. This will ensure that Light elements are not erroneously detected. The default setting for all analyzers disables *Light Element analysis.*

Customers who measure Al alloys should set the slider to the middle of the screen.

The most sensitive setting should be used only in special cases, when recommended by Innov-X Application staff.





Chapter 9 Alloy Analysis—Libraries

9.0 LIBRARIES

All reference data for alloy identification is stored in a standard database, or library. Libraries are all completely editable by a user with password privileges.

There are two types of library entries: Fingerprints and grade tables. Fingerprints are used in both FastID and Pass/Fail modes. Fingerprint files contain a variety of information about a standard sample, including a spectral fingerprint, assay information, and a link to a grade library entry. Grade tables contain minimum and maximum specifications for each element present in an alloy. Grade tables are used in Analytical mode, as well as in Pass/Fail if use chemistry is selected.

A library entry may contain a variety of information about the sample. This information includes:

1. Fingerprint files (editable through FastID or Pass/Fail Modes). These files may contain:
   - Spectral signatures. Signatures or fingerprints are the basis for identifying samples in both FastID and Pass/Fail Modes.
   - Specific Elements—This information is used by the FastID Specific regions Advanced feature. This is used to separate alloys that are very close and differ by only one or two elements. Certain elemental regions are selected for comparison.
   - Assays—These assays are used by FastID and Pass/Fail mode to calculate chemistry.
   - Min/Max Ranges. These are used in Pass/Fail mode for matching by chemistry. Creating Min/Max tables in Fingerprint mode results in a grade table that can be used and edited in analytical Mode.

2. Grade tables (editable through analytical mode)
   - Grade tables include a list of minimum and maximum specifications for use in grade matching.

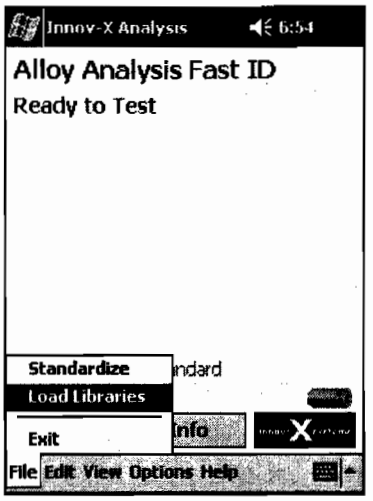
The spectral signature is automatically stored when the user adds a fingerprint to a library in FastID mode. The remaining information can be entered by the user if it is relevant. In order to calculate chemistry in either FastID or Pass/Fail, an assay must be entered. A grade table must exist to use Pass/Fail chemistry, or to get a grade ID in Analytical mode.

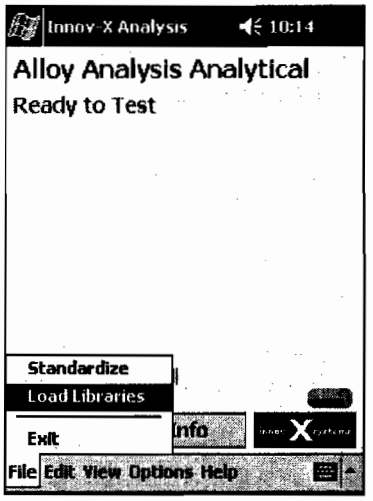
9.1 SELECTING A LIBRARY.

There are four libraries available. Each library can contain both Fingerprints and Grade Tables. The first library is the Standard Library which contains up to 300 fingerprints or grades and is supplied with every analyzer. Typically 35 FastID, and 200 Grade Tables are supplied with new analyzers. The remaining three libraries are empty, and are available for users to use create their own custom libraries. Each of these libraries can contain up to 100 samples, for a total of 300 custom samples. Users may add standards to any of the custom libraries, as well as to the standard library, however, it is good practice to make changes only to the user libraries.

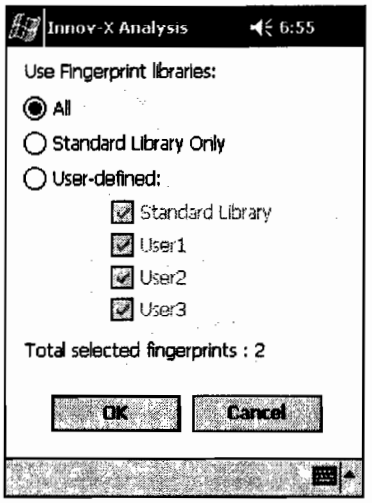
Each of the libraries may be searched individually, or in combination with any or all of the other libraries. The default setup searches all four libraries. It is possible to search any library in FastID. Pass/Fail will use only one fingerprint, so it is not necessary to select a library to search.

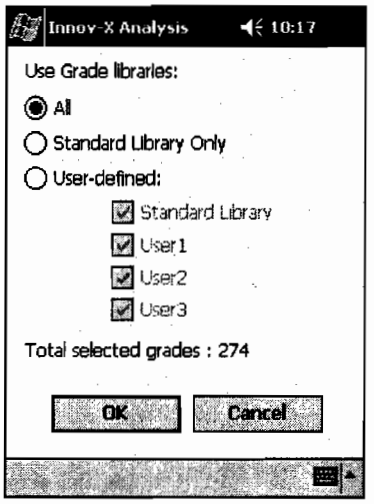
To select which library to search, select ***File→Load Libraries*** from either the FastID or the Analytical Analysis Screen.





A menu will appear. The first line reads **Use Grade Libraries** for Analytical Mode or will read **Use Fingerprints** for FastID Mode.





Choose the Fingerprint or Grade Libraries you wish to search. For the most general search, select **All** libraries. This will search the entire Standard Library, as well as any fingerprints 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**.

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.

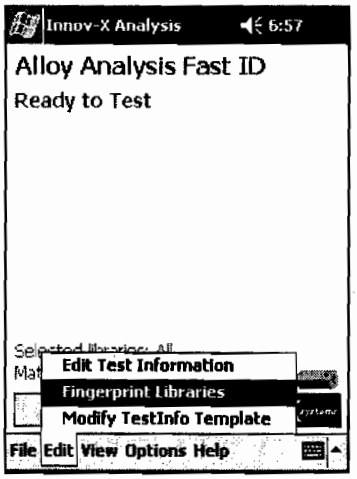
9.2 EDITING LIBRARY FUNCTIONS

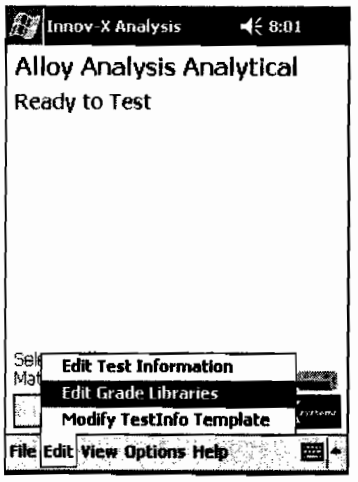
Innov-X Systems libraries are completely user editable. Library entries may be added, deleted, renamed, and the information contained within them, such as assays and min/max grade tables, can be modified.

The information contained in libraries is essential to the proper functioning of your analyzer. Care should be taken when editing libraries as incorrect settings may result in incorrect grade identification.

It is recommended that the standard library is not modified. All new alloys should be added to a user library.

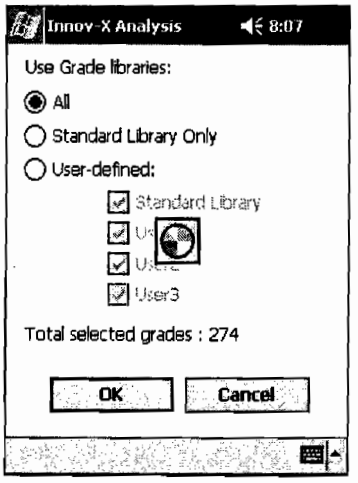
The edit libraries menu is reached by selecting either ***Edit→Grade Libraries*** from Analytical Mode, or by selecting ***Edit→Fingerprint Libraries*** from FastID or Pass/Fail modes.





All library functions require an administrative level password. The password needs to be entered only once during each editing session. The default administrator password is a lowercase **z**. This password can be changed from the main menu.

When you are finished with any edits, there may be a slight pause while the analyzer loads and re-indexes the library. This will be indicated by the following graphic, which will rotate in the center of the screen.

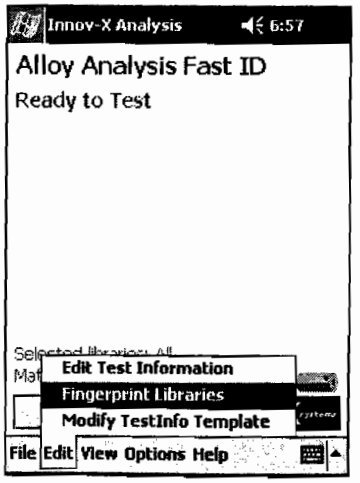


Functions such as renaming and deleting libraries are the same for both Fingerprint Libraries and Grade Libraries. They will therefore be described together in the following sections. Editing library contents and adding new library entries require different procedures for different library types and will be described separately at the end of the chapter.

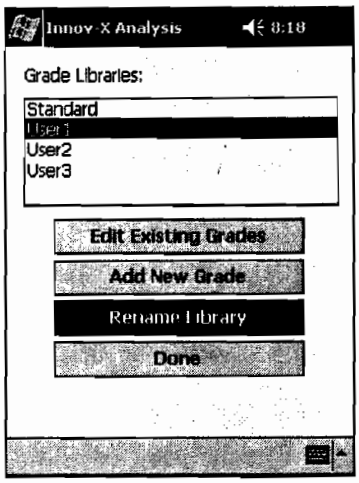
9.3 CHANGING LIBRARY NAMES

The default names for the libraries are Standard Library, User1, User2 and User3. It is possible to change the name of each of the User Libraries; however, the Standard Library can not be renamed. When a library is renamed, the new name will be used for all alloy modes, regardless of the mode that was active when the name was changed.

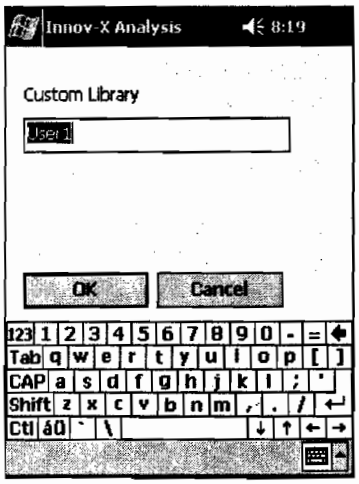
1. Library names can be changed either by selecting ***Edit→Grade libraries*** from Analytical Mode or by selecting ***Edit→Fingerprint libraries*** from Pass/Fail or FastID Mode.



2. To rename a library, select it and tap **Rename Library.**
3. Enter the administrative password when prompted.



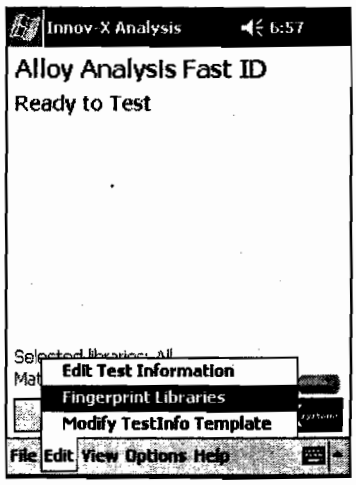
4. Type in the new name. The name may include any combination of letters or numbers, but may not include the characters "/", "." , "+", "_" or "?" . Enter the new name via the virtual keyboard. Tap **OK** to save the new name. If you do not wish to modify the name, select **Cancel.**



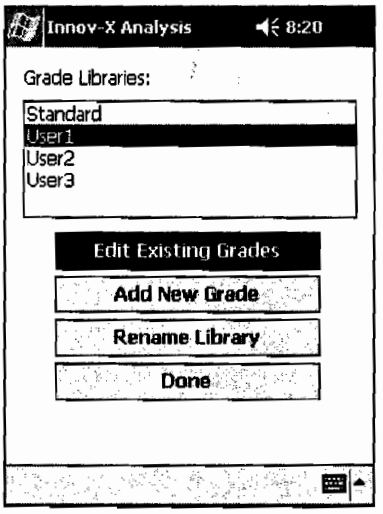
9.4 CHANGING THE NAMES OF INDIVIDUAL FINGERPRINTS OR GRADE ENTRIES.

If typographical errors are made when entering the name of a library entry, the name may be edited. Care should be taken when renaming a library entry. The name must correspond to the data saved in the file. An incorrect Fingerprint or Grade name will result in a wrong Grade ID

1. From the Analytical Menu, Select ***Edit→Grade Libraries,*** or select ***Edit→Fingerprint Libraries*** from FastID or Pass/Fail.

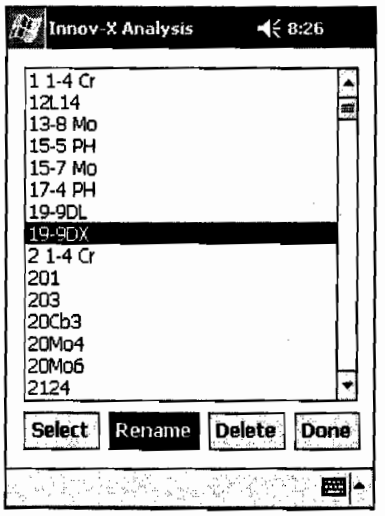


2. Select the library containing the entry you wish to rename and tap **Edit Existing Grades.**

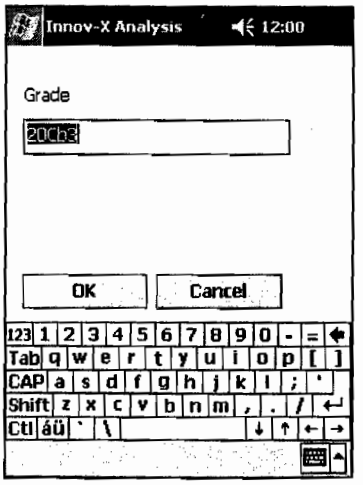


3. A list of the entries contain in the selected library will appear. Select the name to be changed, and click **Rename.** . The name may include any combination of letters or numbers, but may not include the characters /, . , +, _ or ?.

4. You will be prompted for your password.



5. Type the new name in the dialogue box. Select **OK** to accept the new name, or **Cancel** to exit without saving changes.

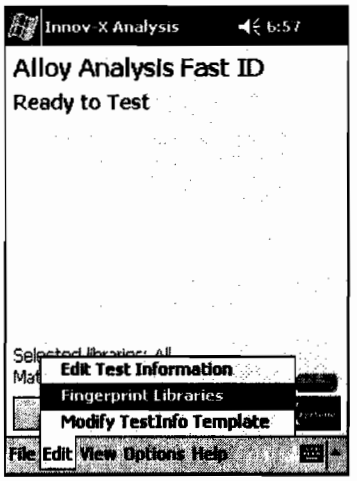


Note, because Grade Table information is stored in Fingerprint files, renaming a fingerprint file will also rename the corresponding Grade Table. However, the reverse is not true. Renaming a Grade Table entry will not rename the fingerprint with the same name. If a grade name is changed, that grade will not be useable in Pass/Fail Mode if **Use Chemistry** is selected.

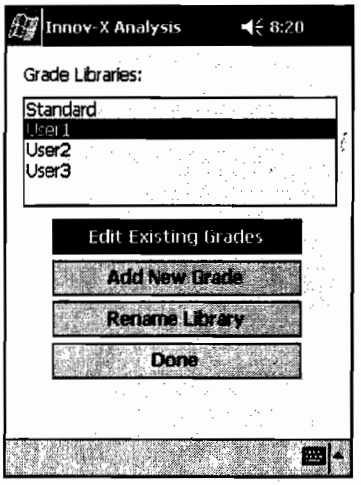
9.5 DELETING A GRADE OR A FINGERPRINT

Caution: Deleting a library entry will permanently delete the Fingerprint and Assay (FastID/PassFail) or the Grade Table (Analytical). Proceed with caution.

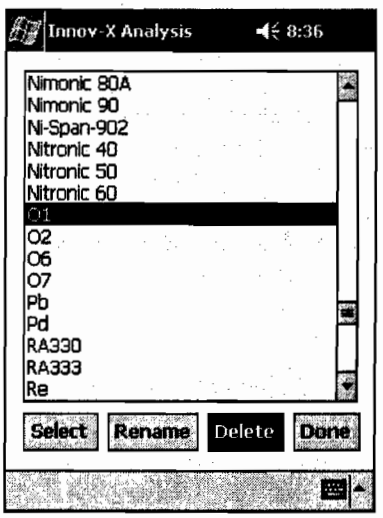
1. From the Analytical Menu, Select ***Edit→Grade Libraries,*** or select ***Edit→Fingerprint Libraries*** from FastID or Pass/Fail.



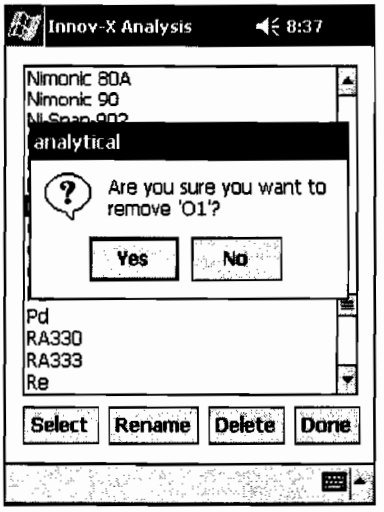
2. Select the library containing the entry you wish to delete and tap **Edit Existing Grades.**



3. Select the sample to be deleted and tap **Delete**.
4. Enter password when prompted.



5. You must select **Yes** to confirm deletion. Otherwise select **No**.

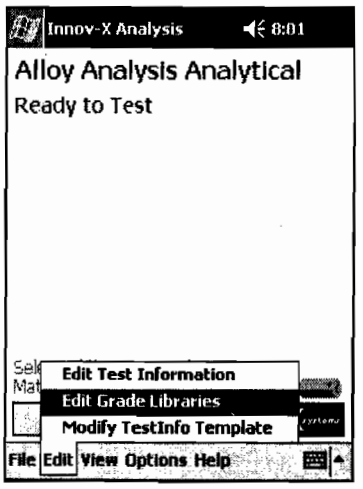


9.6 ADDING A GRADE

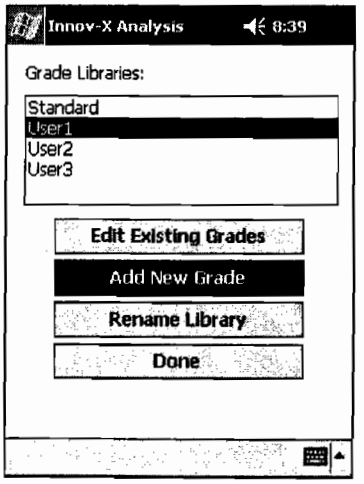
If you wish to add a grade to a library, you must have a list of the minimum and maximum allowable concentrations of each element in the library. It is important to include ALL elements that may be present in an alloy, including the balance element. Pay special attention to tramp elements that may not be specified but may be contained in an alloy of that grade. For example, many stainless alloys may contain small amounts of Molybdenum. If Mo is not included in the grade table, but is detected in sample, the sample may not be correctly identified. In a case like this, it is recommended that a maximum allowable specification is entered, such as 0-.5%.

Follow this simple procedure to add a grade:

1. From Analytical Mode, Choose **Edit→Grade Libraries**.

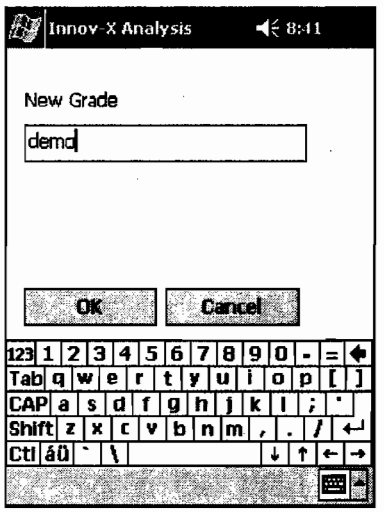


2. Select the Library to which you want to add an entry, and tap **Add New Grade**. Enter your password when prompted.

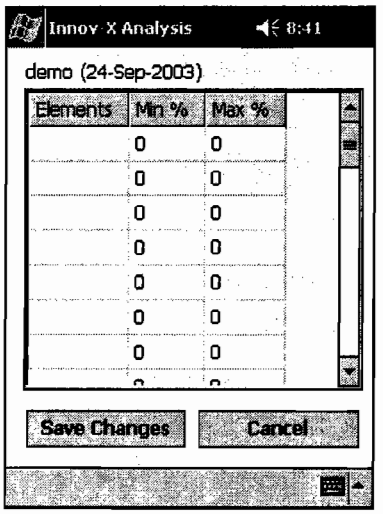


There is a limit of 100 entries in each of the libraries. If you attempt to add a grade to a library which already contains 100 entries, the message "Maximum number of user-defined libraries already exist" will appear. You must either select another library or delete a grade from the current library.

3. Enter the name of the new Grade, and tap **OK**.

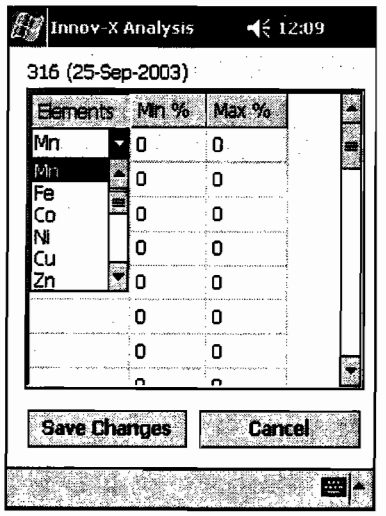


4. A new Min/Max Table will appear. It will contain no elements. A value of zero will be entered for all Min and Max values.



5. To select an element name, click twice in a box in the "Elements" Column of the table. This will bring up a drop down menu.

   You may scroll down an element, or you may advance to a specific element by pressing the first letter of the element on the virtual keyboard. Each time you press a letter, you will go to the next element in the list beginning with that letter. For example, the first time you select M, you will advance to Mn. Selecting M again will advance to Mo.

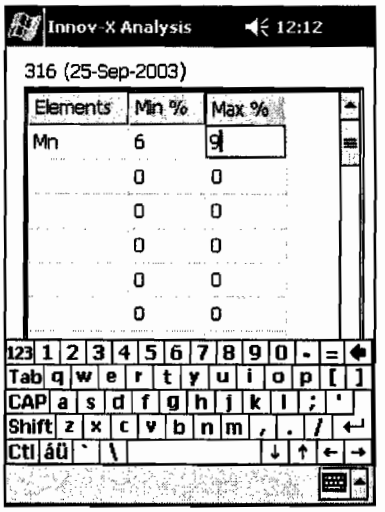


6. To enter a minimum or maximum value, tap in the appropriate space to select it, then enter the desired value.

   a. If an element is listed as being between 6 and 9 %, enter a 6 in the Min % column and a 9 in the Max % column.

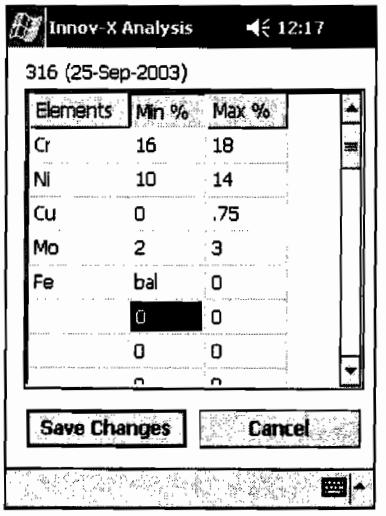
   b. If a maximum value is specified for an element, enter 0 in the Min % column and the maximum specification in the Max % column.

   c. To enter a spec that has only a minimum value specified, enter the value in the Min %column and 100 in the Max %column.

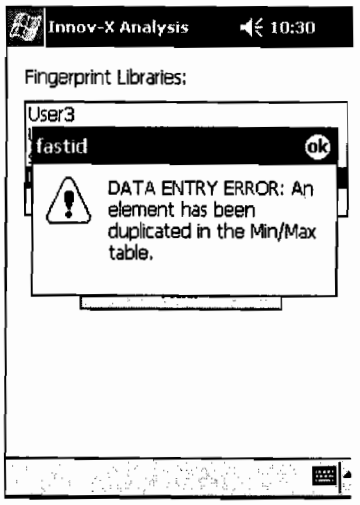


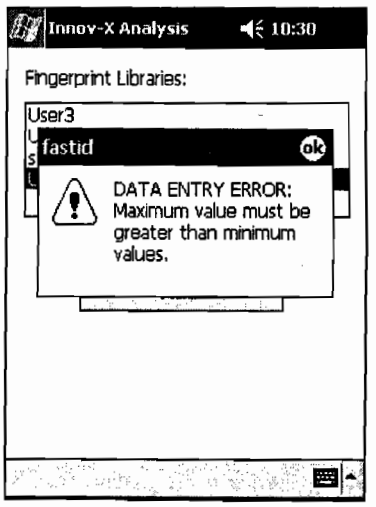
7. Repeat for all relevant elements. Bal may be entered for the balance element.

8. Tap **Save Changes.**



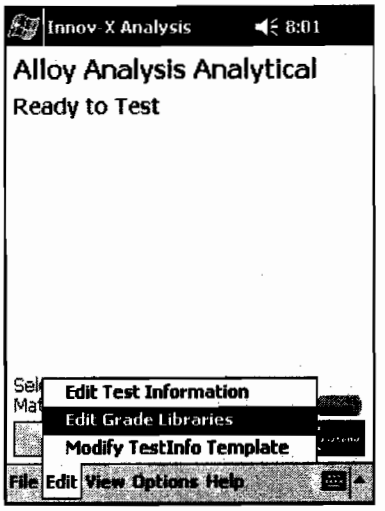
The software requires that the minimum value is less than the maximum value for each element, and that each element is only entered once. If there is a problem with the data entered, a warning message will appear. Click **ok**. The Min/Max table will appear. You must correct any errors before you are allowed to save the data.



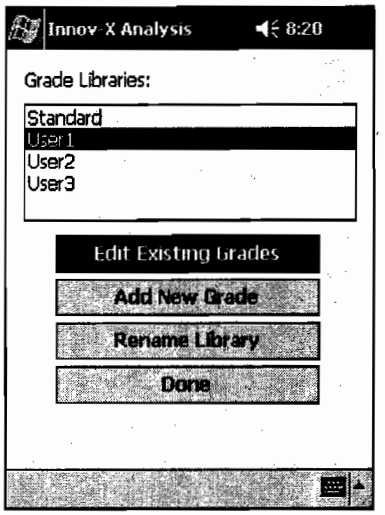


9.7 ANALYTICAL MODE--EDITING A GRADE

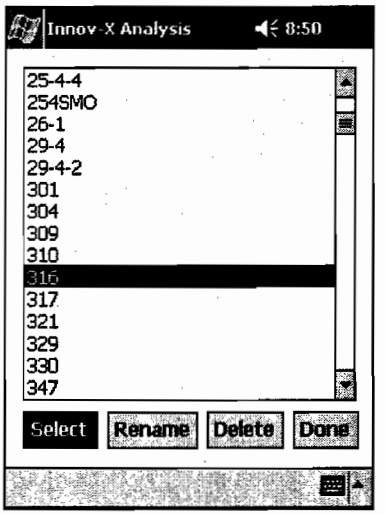
1. From Analytical Mode, Choose ***Edit→Grade Libraries***.



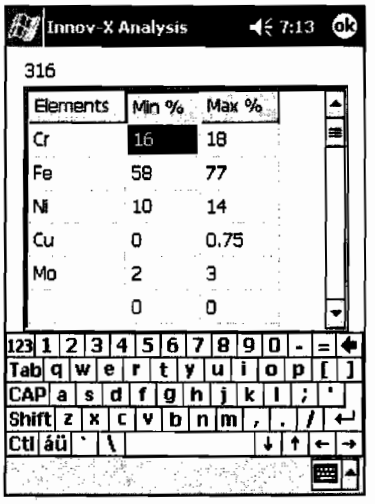
2. Select the library containing the entry you wish to rename and tap **Edit Existing Grades**.



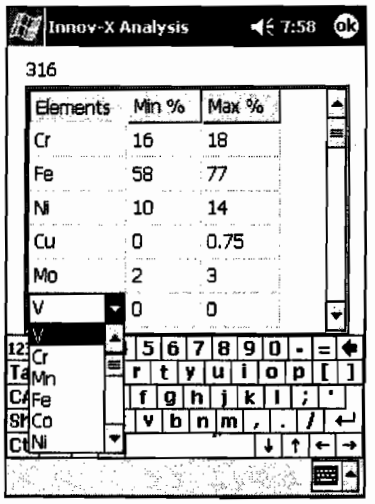
3. A list will appear showing the entries in the library. Tap on the grade name you wish to modify and tap **Select** to bring up the specifications.



4. Element specific minimum and maximum values are shown. Only elements with specified values are shown on the list. To modify min or max values for any listed element, select the appropriate field and enter the new value using the virtual keyboard.

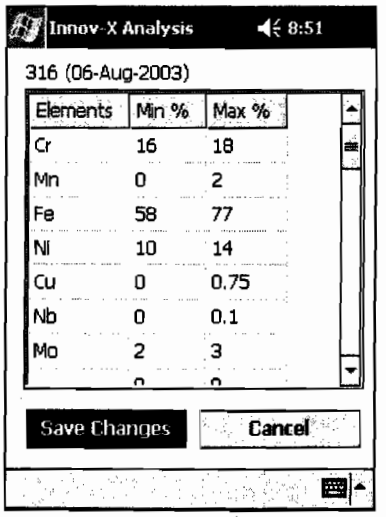


5. To add specifications for an element that is not present, tap twice in an empty box in the Elements column. Use the drop-down menu to select a new element in the field directly below the last listed element.



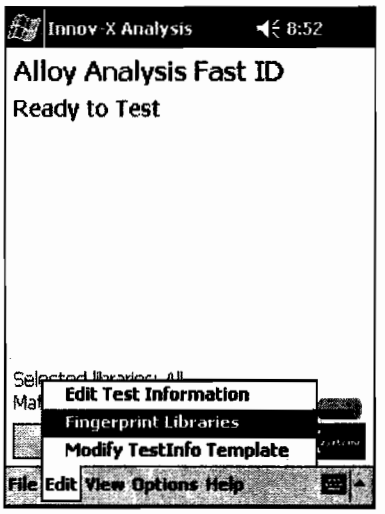
6. Repeat for all relevant elements.

7. Tap **Save Changes**.

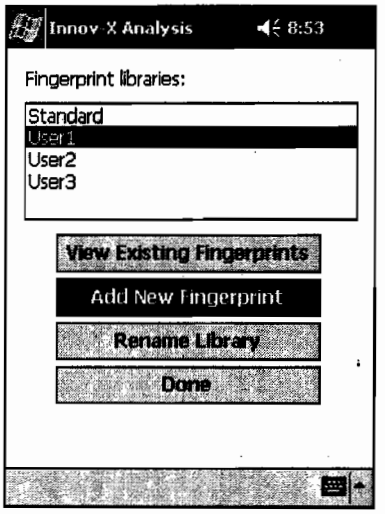


9.8 ADDING A FINGERPRINT

Adding a standard to a fingerprint library requires a sample of the material to use to create a fingerprint. In order to calculate chemistry for this type of alloy, assay values must be available and must be entered into the software. Since assay values are used to calculated chemistries for other alloys, it is important that this information is very accurate. When possible, it is strongly recommended that a certified standard be used.

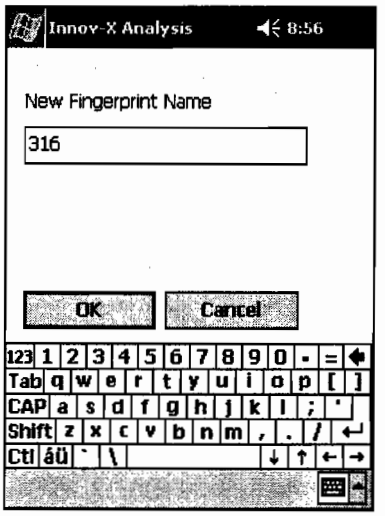


1. From the FastID Analysis Screen, Choose ***Edit→Fingerprint Libraries***.



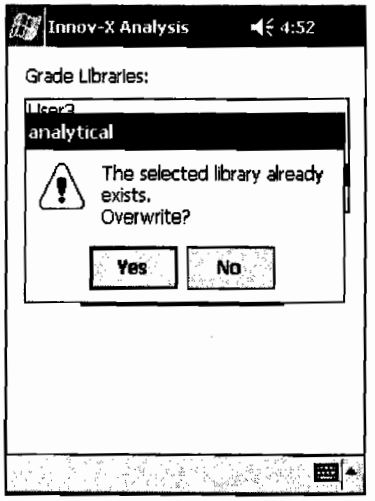
2. Select the Library to which you want to add an entry, and tap **Add New Fingerprint.** Enter your password when prompted. Remember that your password is the lowercase letter **z** unless it has been changed.

The size of the libraries is limited to 300 entries in the standard grade library, and 100 in each of the custom libraries. If the library is full, a message will appear telling you to either delete a grade, or choose a different library.

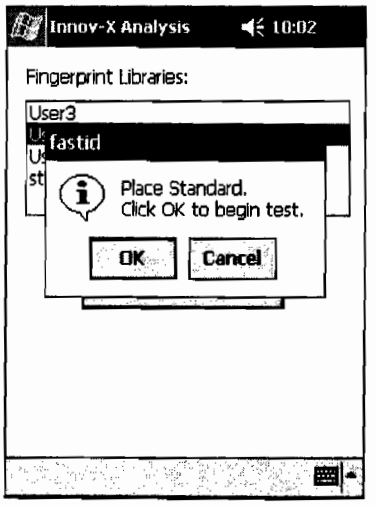


3. Enter the name of the new Fingerprint, and tap **OK**. The alloy name may be any combination of letters or numbers but may not include symbols such as ?, /, ., or +.

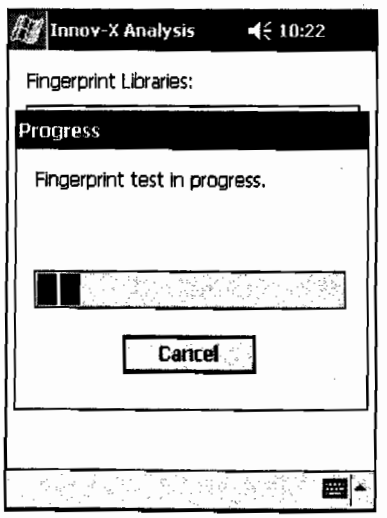
4. If you enter a sample name that is already stored in the library, you will receive a warning message. If you wish to overwrite the existing file tap **Yes,** otherwise select **No** and choose another name.



5. The analyzer will prompt you to place the sample.



6. Place the sample in front of the analyzer window and tap **OK**. The analyzer will acquire a spectrum for storage in the library. The fingerprinting process will take 30 seconds. It is very important that the analyzer window remain in contact with the standard for the entire duration of the fingerprint measurement.

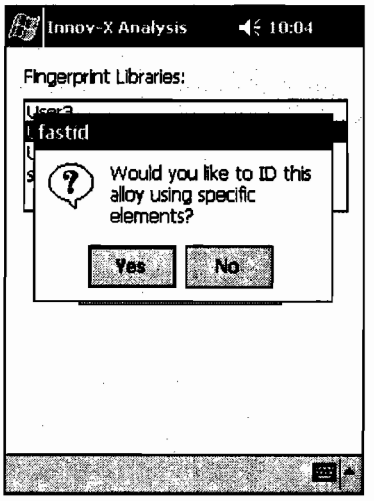


7. When the fingerprint has been stored, you will be asked if you want to enter specific elements.

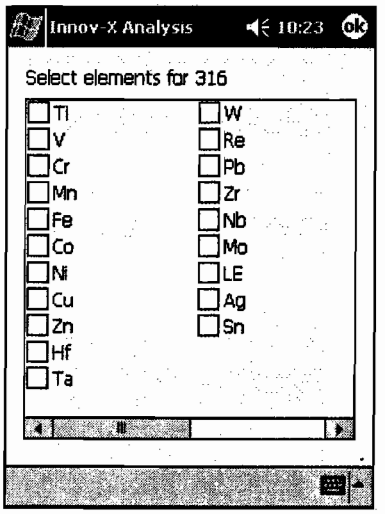
   Using specific elements is described in more detail in the FastID section of this manual. It is useful in situations where you are trying to separate two or more similar alloys that differ only in the composition of one or two elements. Note that this feature is not often used. Most users will select **No**

   If you wish to utilize this function, select **Yes**, otherwise, select **No**.

   If **No** is selected, skip to step number 10.



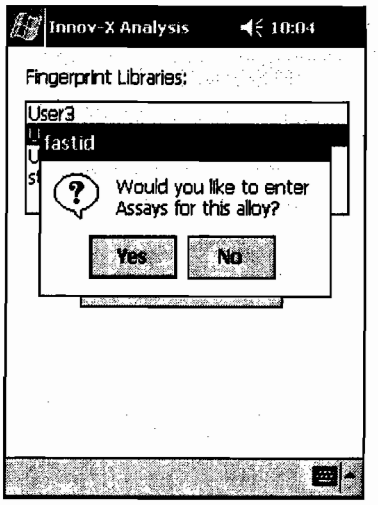
8. To enter specific elements, select the elements you wish to use by checking the box to the left of the element name. Usually, only one or two elements will be selected. Tap **ok** when you are finished



9. After specific elements have been selected, or after selecting **No** in step 8, you will be asked whether you want to enter assays.

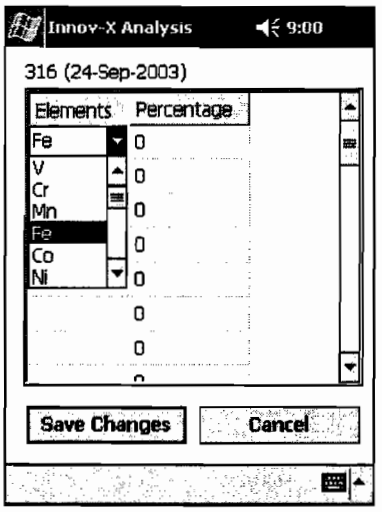
   In order for the Innov-X Systems Software to calculate chemistries in FastID or Pass/Fail Modes, assays must be entered.

   If you do not wish to enter assays, or they are not available, select **No**. If you select **No**, you will be able to use this Fingerprint for Grade Identification in FastID, and using the **Fingerprint** method in Pass/Fail. However, it will not be possible for the instrument to calculated chemistries for alloys matching this fingerprint. Skip to step 12 if **No** is selected.



10. A blank table will appear. Enter the element name and assay into this table.

    a. To select an element name, click twice in a box in the "Elements" Column of the table. This will bring up a drop down menu. Scroll down to the element you wish to select, or select the first letter in the element symbol using the keyboard.

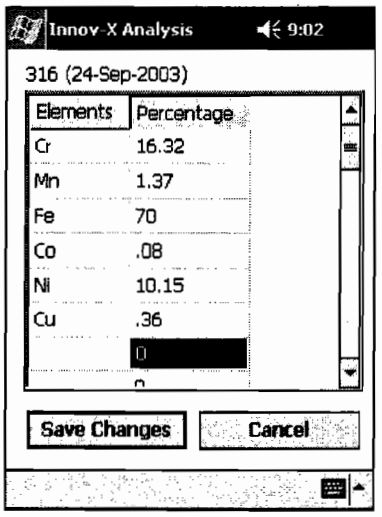


    b. Enter the assay for the selected element in the next column.

    The software will automatically calculate the value of a balance component if "bal" is typed into the percentage column.

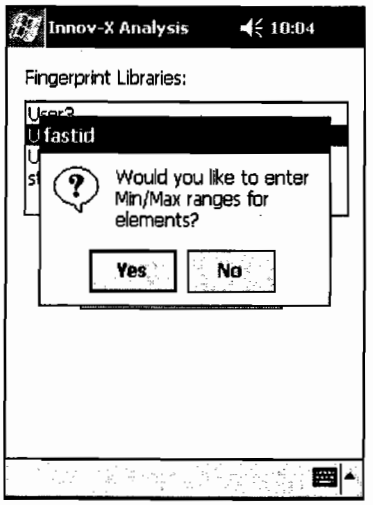
    Keep in mind typical detection limits of the instrument when entering assays. As a general rule, do not enter assays which are below 0.3%.

    c. When all assays have been entered, tap **Save Changes**.

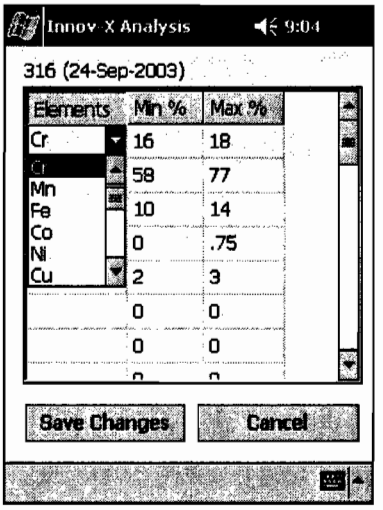


11. After entering assays (or if no was selected in step 10), the system will ask if you want to enter Min/Max ranges. This is recommended only if you will be using both Analytical and FastID to identify this standard alloy, or if you will be using Pass/Fail mode with **Use Chemistry** activated. The vast majority of users will select **No** in answer to this question.

    If you wish to enter Min/Max ranges, select **Yes;** otherwise select **No**. If you select **No**, the process of entering a fingerprint is complete.



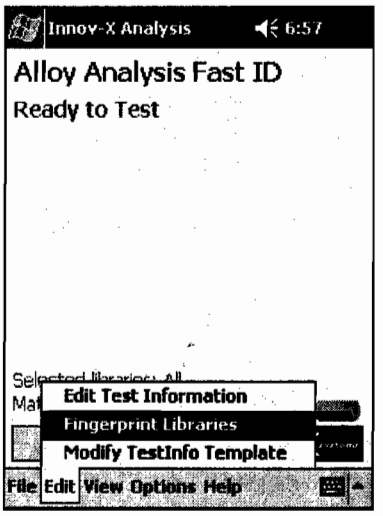
12. Enter your Min and Max ranges for each of the elements in your sample. If you have any questions about this procedure, you can refer to the description in **Section 9.6: Adding a Grade**.



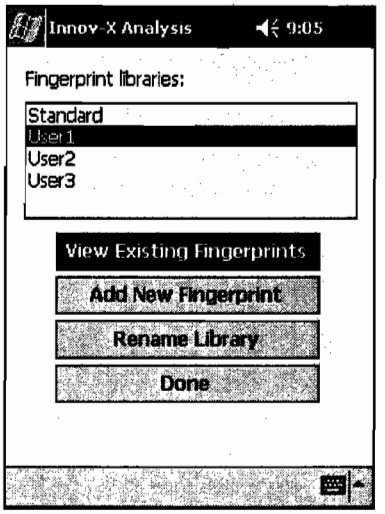
9.9 EDITING FINGERPRINT LIBRARIES

It is possible to edit Assays, Specific Regions and Min/Max (Grade Tables) for existing elements.

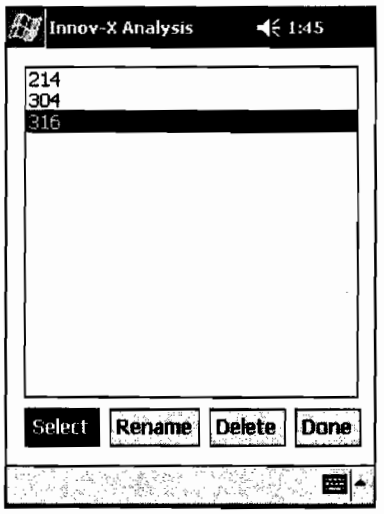
1. From the Fast ID Analysis Screen, Choose ***Edit→Fingerprint Libraries***.



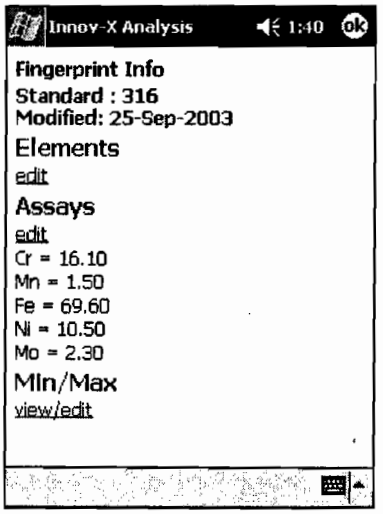
2. Select the Library containing the fingerprint you want to modify and select **View Existing Fingerprints**.



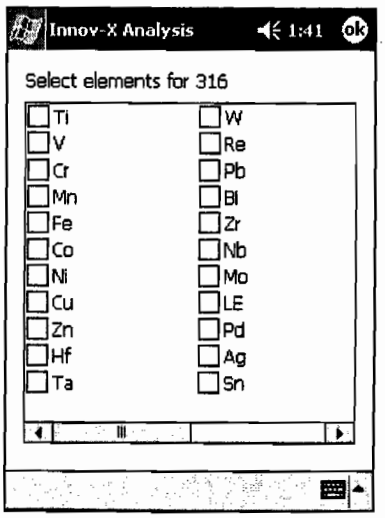
3. A list will appear showing the entries in the library. Tap on the grade name you wish to modify and tap **Select** to bring up the specifications. Refer to the **Section 9.8: Adding a Fingerprint** for more information on any of these features.



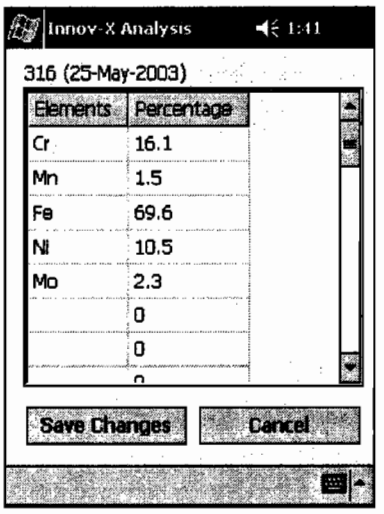
4. The fingerprint info screen will be selected. If any assays are stored with the fingerprint, they will be shown here. To edit, tap the words **edit** or **view/edit** below the information you wish to enter.



5. Editing elements brings up the specific elements screen. Select elements you wish to use. Press **ok** to select.

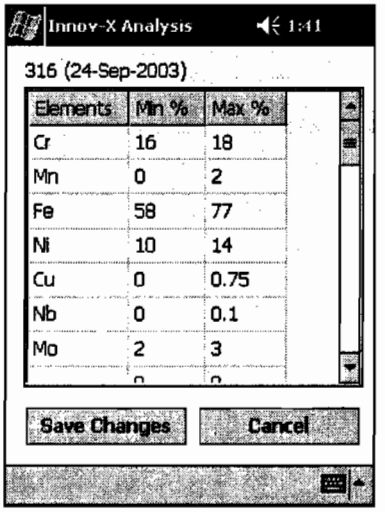


6. If you wish to enter assays, enter the elements contained in the sample and the certified values.



Elements	Percentage
Cr	16.1
Mn	1.5
Fe	69.6
Ni	10.5
Mo	2.3
	0
	0

7. Min/Max **View/Edit** allows you to enter minimum and maximum specifications for each element.



Elements	Min %	Max %
Cr	16	18
Mn	0	2
Fe	58	77
Ni	10	14
Cu	0	0.75
Nb	0	0.1
Mo	2	3

Chapter 5 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.

5.0 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.

5.1 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:

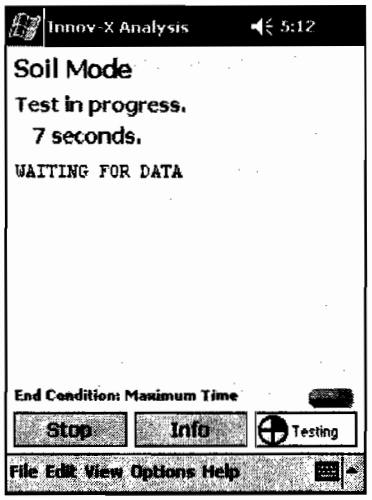
It is strongly recommended that all prepared samples be analyzed in the testing stand. Samples should be placed on top of the testing stand, completely covering the window. **Never hold prepared or bagged samples while testing,** as this could expose the operator to the x-ray beam.

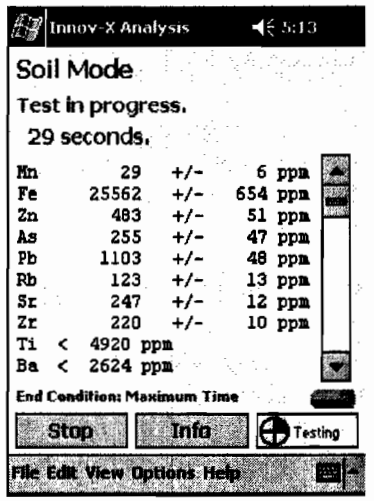
Avoid measuring very thin samples, as this can affect results. Prepare samples cups to contain at least 0.5 inches of packed samples. When analyzing bagged samples, make sure that sufficient sample exists in the bag to completely cover the window with a sample thickness of a minimum of 0.5 inches.

5.2 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.

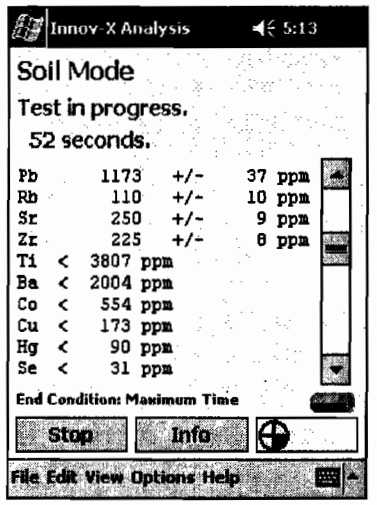
After a minimum time has elapsed, intermediate results will be displayed on the screen. Until this minimum time has elapsed, the words "WAITING FOR DATA" will appear instead. This minimum time can be set by the user by selecting ***Options→Set Testing Times***, which is described in **Section 5.4: Soil 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, 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.

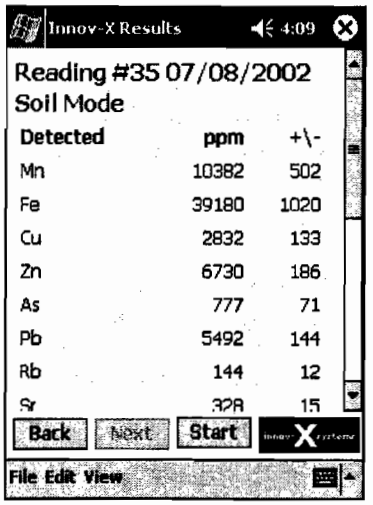
When the measurement is complete the results screen will open, displaying the final results of the measurement.

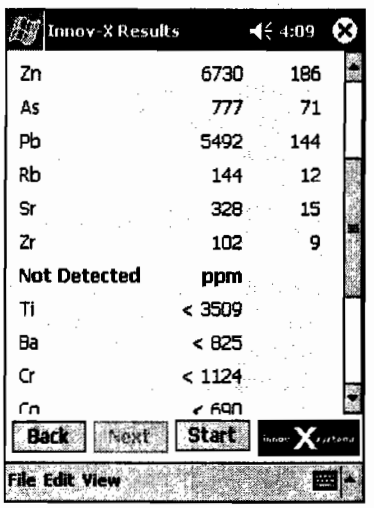


5.3 SOIL RESULTS SCREEN

5.3.1 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***.



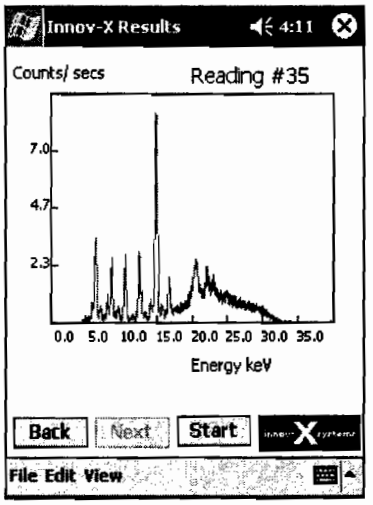


The standard soil chemistry display can be modified by using the View Menu. As with all Innov-X analytical modes, it is possible to view spectra and Test Information.

5.3.2 Spectrum Screen

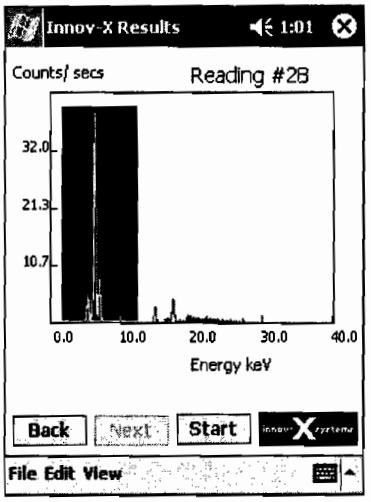
This screen displays a plot of the x-ray fluorescence spectrum for an individual test, plotting the intensity on the y-axis versus the energy of the fluorescence x-rays on the x-axis.

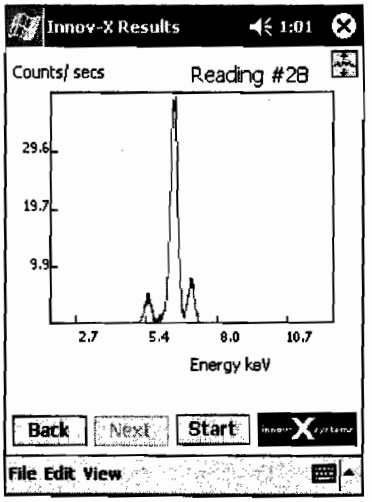
Tapping on the spectra will show the energy scale and counts rate at the selected point



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.





5.3.3 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.**

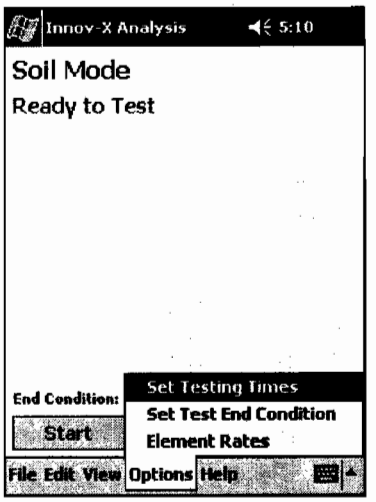
5.4 SOIL MODE OPTIONS

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.

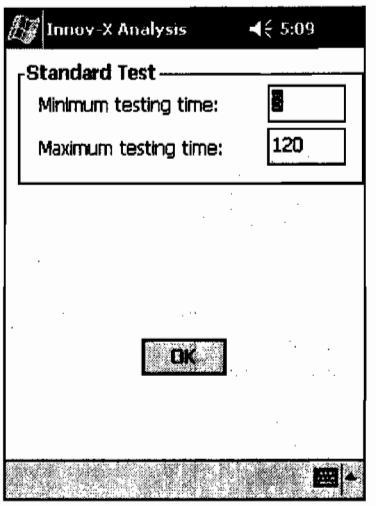
The options related to test time are contained in two menus: ***Options→Set Testing Times***, and ***Options→Set Test End Condition. Set Testing Times*** contains minimum and maximum testing time information, while ***Set Test End Condition*** allows the user to select test end conditions.

5.4.1 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".

5.4.2 Soil Mode Test End Condition

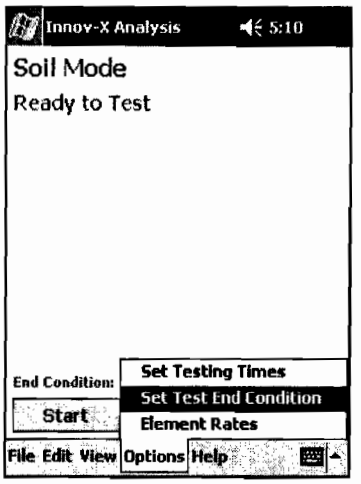
Four options exist for the test end criteria in soil mode. Depending on your application, you may choose to end the test manually, at a preset testing time, or when the uncertainty in the measurement is within a

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.

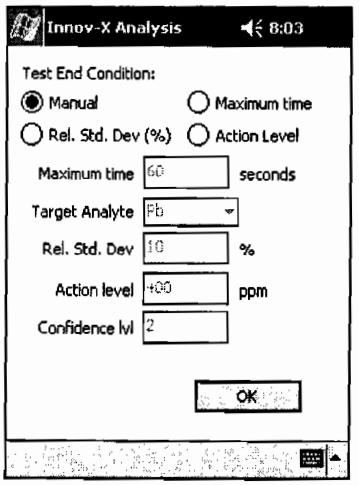
Changes to the test end condition are made by selecting ***Options→Set Test End Condition***

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



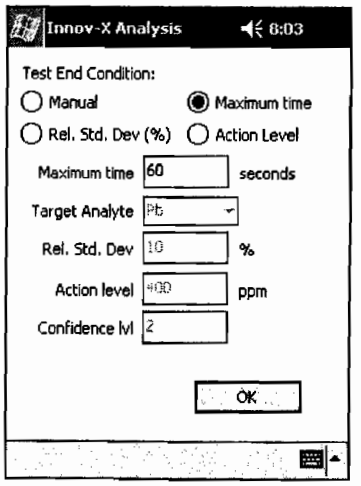
Manual: This option allows you to look at the results which are being continually updated on the screen and determine when the results look satisfactory. The test will continue until the trigger is pulled, or **Stop** is tapped on the iPAQ screen. Results will be calculated if the testing time has exceeded 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.



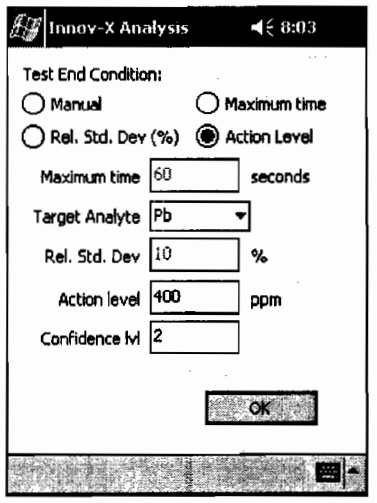
Maximum Time: If Maximum Time is selected, the test will continue until the preset time is reached. This is useful if you wish to do a set of measurements with the same testing time.

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.



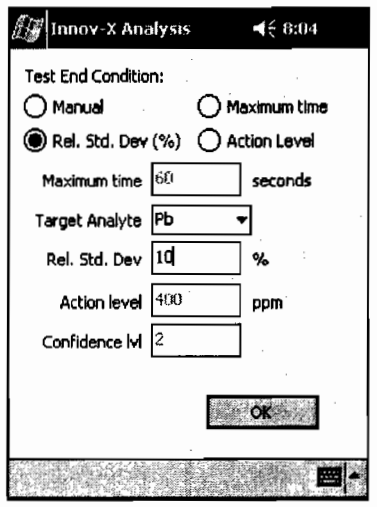
Action Level: System ends test when result for target analyte including chosen precision level is above or below pre-set action level.

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.



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.



5.5 LEAP Mode (Light Element Analysis Program):

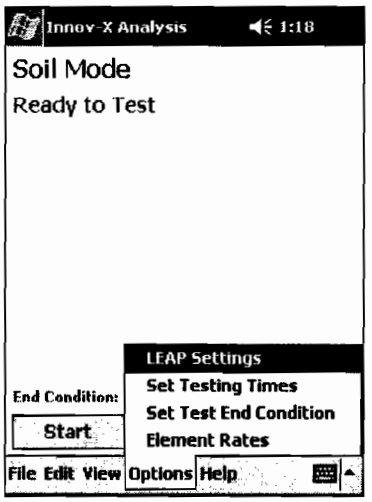
This is a factory installed optional module. Instruments can be upgraded to LEAP capabilities. Please contact the Innov-X Systems Sales department for information and pricing.

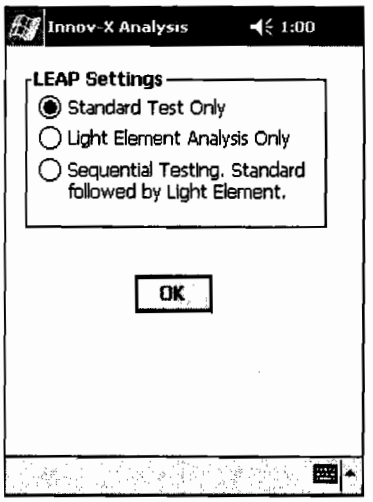
The LEAP module provides the lowest possible detection limits for elements lighter than iron. The standard LEAP package includes the elements Ti, Ba and Cr. Elements as low as Phosphorus can be detected with the Advanced LEAP package which includes a thin window detector.

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 then 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.

5.5.1 LEAP Settings

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



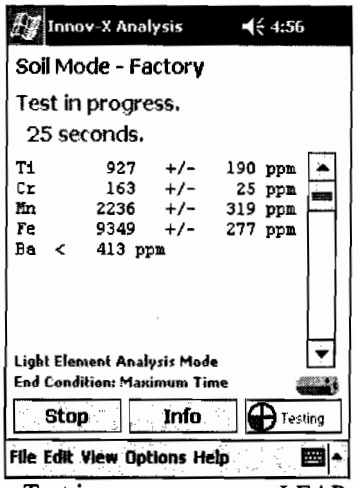


Standard Test Only: The analyzer will provide analysis for the standard suite of elements.

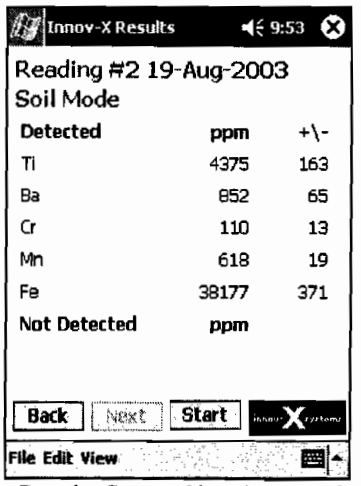
Light Element Analysis Only: The analyzer will provide analysis for elements in the LEAP suite (Typically Ti, Ba and Cr)

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.

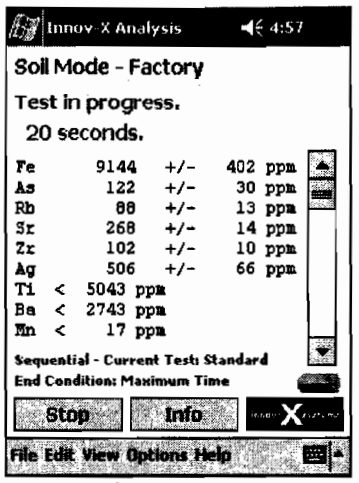


Test in progress screen, LEAP Only, Live Updates on

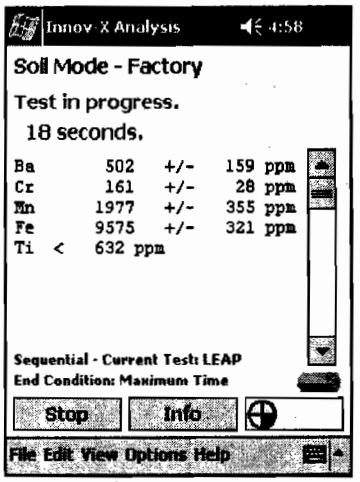


Results Screen Showing LEAP results

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.

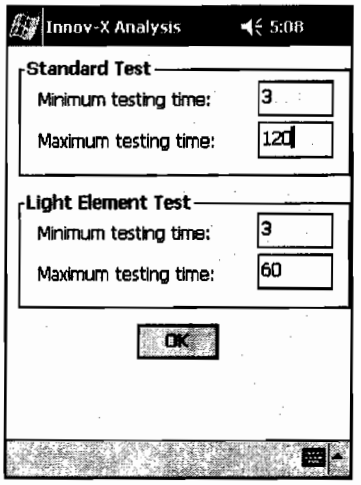


Test in progress screen, Sequential.
Second Test – LEAP Analysis

5.5.2 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

Stainless	*Chrome-Moly*	*Nickel Base*
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	*Cobalt Base*	Inco 825
347	F-75	Hast C-276
410/416	HS-6	Hast X
	HS-25	Monel 400
Low Alloy		Monel 500
CS	*Copper Base*	Waspaloy
C ½Mo	70-30	
4140, 4130	90-10	*Ti Base*
4340	CDA 836	CP Ti
		Ti 6-4

Appendix 2: Standard Grade Library

Iron Base Alloys

201	Alnico VIII	**Tool Steels**
203	AL6XN	
301	AMS 350	A2
304	AMS 355	A6
309	CD4MCU	A7
310	Custom 450	A10
316	Custom 455	D2, D4
317	Duplex 2205	D7
321	Elgiloy	H12
329	Ferallium 255	H13
330	Greek Ascoloy	L6
347	Hy Mu 80	O1
410/416/420	Kovar	O6
410 Cb	Invar 36	O7
422	Maraging C200	M1
430/440	Maraging C250	M2
431	Maraging C300	M42
434	Maraging C350	M4
441	N-155	S1
446	Ni-hard #	S7
12L14	Ni-hard #	T1
13-8 Mo	Nitronic 40	
15.5 PH	Nitronic 50	**Low-Alloy Cr-Mo Steels**
17-4 PH	Nitronic 60	
19-9DL	RA333	
19-9DX	RA330	
20Cb3		Carbon steel
20Mo4		4140
20Mo6		1 1/4 Cr
25-4-4		2 1/4Cr
254SMO		5 Cr
21-6-9		9 Cr
26-1 (Ebrite)		C - 1/2 Mo
29-4		
29-4-2		
904 L		
A-286		
Alloy 42		
Alloy 49		
Alnico II		
Alnico V		

Ni-base Alloys

Ni	Inco 718
80-20	Inco 722
B-1900	Inco 738
B-1900 Hf	Inco 750
Inco 617	Inco 792
Inco 625	Inco 800
C-1023	Inco 801
GMR 235	Inco 825
GTD 222	Inco 901
Hast B	Inco 903
Hast B2	Inco 909
Hast C-4	Mar M 002
Hast C-22	Mar M 200
Hast C-276	Mar M 246
Hast C-2000	Mar M 247
Hast F	Mar M 421
Hast G	Monel 400
Hast G-2	Monel 411
Hast G-3	Monel 500
Hast G-30	MP35N
Hast N	Mu Metal
Hast R	Nichrome V
Hast S	Nickel 200
Hast X	Nim 101
Hast W	Nim 263
Haynes 25	Nimonic 75
Haynes 36	Nimonic 80A
Haynes 214	Nimonic 90
Haynes 230	Ni-Span 902
Haynes 188	Rene 41
Haynes 556	Rene 77
HR-160	Rene 80
IN 100	Rene 95
Inco 600	Rene 125
Inco 601	Supertherm
Nim 101	Udimet 500
Nim 263	Udimet 520
Inco 690	Waspaloy
Inco 702	
Inco 706	
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 2, 4	Re
HS-25 (L605)	CDA 706	10-2-3	Zr	Sb
HS-31	CDA 836	Ti-8		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
Software won't start: Software will not start when the Innov-X Systems Icon is tapped.	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.
Software won't start: 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"	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.
IPAQ locks up: iPAQ screen "locks up" and doesn't respond when screen is tapped or buttons are pressed	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. .
Analyzer will not standardize	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. 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: • 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.
Incorrect Alloy ID in FAST ID Mode: Analyzer does not correctly ID sample that was just added to the library in FastID.	Verify that the Alloy was saved in a library which is being searched. ***Use File→Load libraries*** to change the library being search. ***Edit→Fingerprint libraries*** "Show/Modify" can be used to check that the fingerprint was saved in the proper library.

Analyzer gives "No Match" for every sample.	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.
All tests yield incorrect match	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.
Results screen doesn't show new readings after a test is completed	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.
Serial Communication Error Message: Serial Communication error occurs because iPAQ has been removed from instrument or cradle, with the software open and the instrument standardized.	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.
Serial communication error on startup, or while testing.	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.
Trigger will not start test.	Reset the instrument. If this fails, call Innov-X Systems Technical Support at 781-938-5005.

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
Software won't start: Software will not start when the Innov-X Systems Icon is tapped.	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.
Software won't start: 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"	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.
IPAQ locks up: iPAQ screen "locks up" and doesn't respond when screen is tapped or buttons are pressed	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. See Page 4 of the Compaq "Getting Started" manual for an illustration showing the location of the reset button.

Analyzer will not standardize	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. 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: 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.
Results screen doesn't show new readings after a test is completed	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.
Serial Communication Error Message: Serial Communication error occurs because iPAQ has been removed from instrument or cradle, with the software open and the instrument standardized.	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.
Serial communication error on startup, or while testing.	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.

Results take a very long time to display on the first test of the day.	There may be too many readings stored in memory. Erase readings from the results screen by selecting ***File → Delete Readings***.
Trigger will not start test.	Verify that the trigger lock is off. Reset the instrument. If this fails, call Innov-X Systems Technical Support at 781-938-5005.
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.
Results screen shows message "Error in calculation: No Results"	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. Make sure the sample being analyzed is a soil sample, if it is and this message occurs repeatedly; call Innov-X technical support.

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 the 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 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 SiO_2 (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 SiO_2 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 SiO_2 once very 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 sub-sample 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"

ω_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 commons 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:

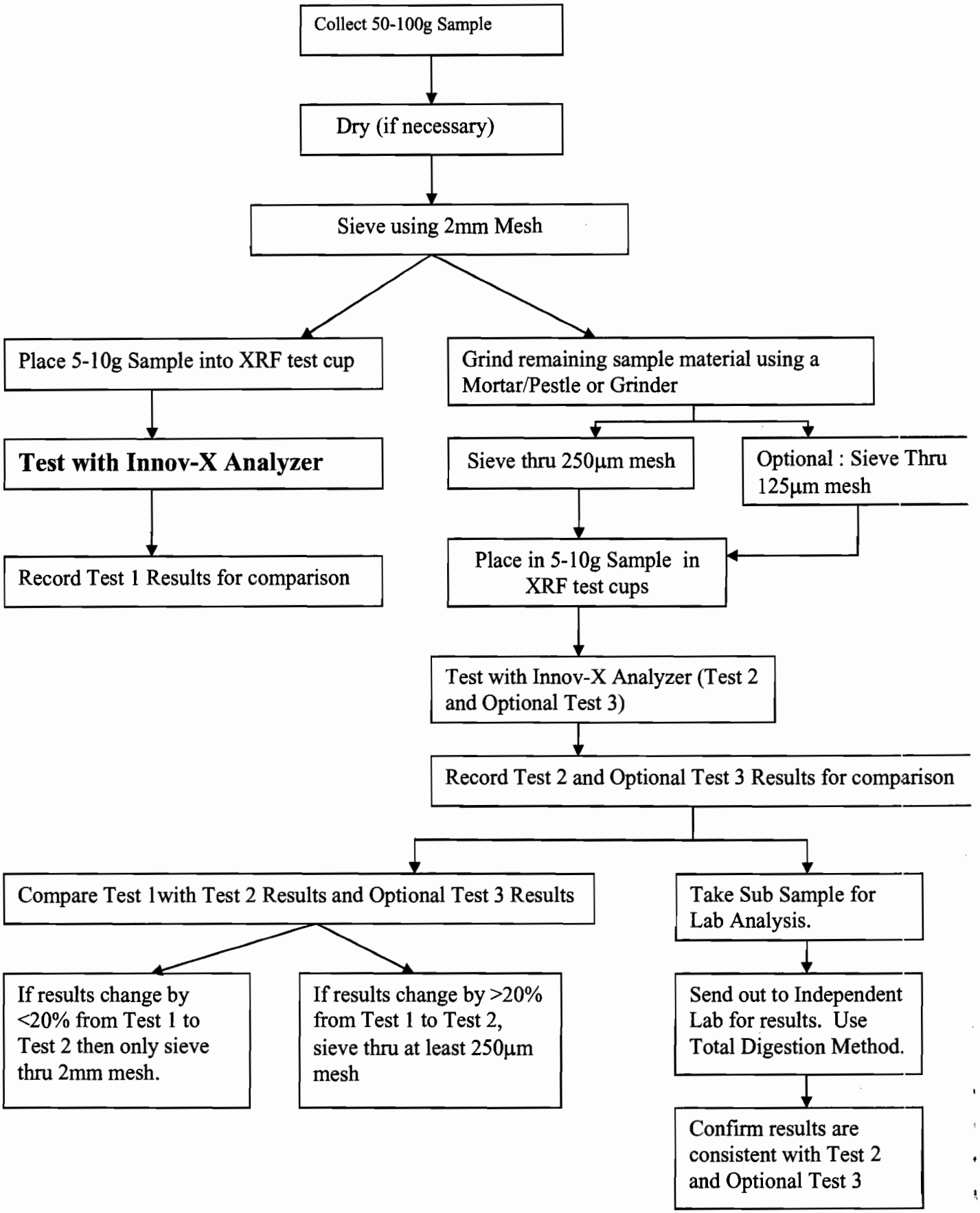
An interference occurs when the spectral peak from one element overlaps either partially or completely with the spectral peak of another. If the XRF is calibrated for both elements (CASE 1) i.e. the one causing the interference and the one being interfered with, it is generally capable of correctly handling 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.


Collect 50-100g Sample
Dry (if necessary)
Sieve using 2mm Mesh
Place 5-10g Sample into XRF test cup
Test with Innov-X Analyzer
Record Test 1 Results for comparison
Grind remaining sample material using a Mortar/Pestle or Grinder
Sieve thru 250µm mesh
Optional : Sieve Thru 125µm mesh
Place in 5-10g Sample in XRF test cups
Test with Innov-X Analyzer (Test 2 and Optional Test 3)
Record Test 2 and Optional Test 3 Results for comparison
Compare Test 1with Test 2 Results and Optional Test 3 Results
If results change by <20% from Test 1 to Test 2 then only sieve thru 2mm mesh.
If results change by >20% from Test 1 to Test 2, sieve thru at least 250µm mesh
Take Sub Sample for Lab Analysis.
Send out to Independent Lab for results. Use Total Digestion Method.
Confirm results are consistent with Test 2 and Optional Test 3

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

General Appendix 1

Technical Specifications

Description:

Innov-X Systems analyzers are hand-held, battery operated energy dispersive x-ray fluorescence analyzers. They are utilized for the detection and quantification of elements ranging from phosphorus (atomic number 15) though uranium (atomic number 92). Measurable concentrations of elements range from ppm to 100%.

Weight:	2.625 lbs (Base wt.) 3.375 lbs (1.6 kg) with batteries
Excitation Source:	X-ray tube, Ag or W anode, 10-40 kV, 5-50 uA, 5 filter positions
Detector:	Si PiN diode, thermoelectrically cooled, resolution < 280 eV.
Power:	Li-ion batteries, or AC power with Testing Stand
Battery Life:	4-8 hours, depending on duty cycle.
Display:	Color, high-resolution touch screen with variable backlighting on analyzer. Software available for PC/laptop operation also.
Data Storage:	10,000 tests with spectra minimum, expandable to 100,000+ with 1 Gb flash card.
Computer:	HP iPAQ with Intel processor, 64 Mb minimum memory, Windows CE operating system (unless operated from PC).
Optional Accessories:	Bluetooth wireless printing and data transfer, integrated bar-code reader, wireless LAN, other standard PDA accessories.

Operating Conditions

Temp 0 – 40° C
Humidity 10 – 90 % RH, no condensation
Altitude rating 2000 meters

Innov-X Analyzer Limited Warranty

General Terms

EXCEPT AS EXPRESSLY SET FORTH IN THIS LIMITED WARRANTY, INNOV-X SYSTEMS, INC. (INNOV-X) MAKES NO OTHER WARRANTIES OR CONDITIONS, EXPRESSED OR IMPLIED, INCLUDING ANY IMPLIED WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE. INNOV-X EXPRESSLY DISCLAIMS ALL WARRANTIES AND CONDITIONS NOT STATED IN THIS LIMITED WARRANTY. ANY IMPLIED WARRANTIES THAT MAY BE IMPOSED BY LAW ARE LIMITED IN DURATION TO THE LIMITED WARRANTY PERIOD.

This Limited Warranty applies to Innov-X analyzers sold or leased from Innov-X its affiliates, authorized resellers, or country distributors (collectively referred to in this Limited Warranty as ("Innov-X")).

Innov-X warrants that the analyzer and all its internal components that you have purchased are free from defects in materials or workmanship under normal use during the Limited Warranty Period. The Limited Warranty Period starts on the date of shipment by Innov-X. You may be required to provide proof of purchase or lease as a condition of receiving warranty service. You are entitled to warranty service according to the terms and conditions of this document if a repair to your Innov-X analyzer is required within the Limited Warranty Period.

During the Limited Warranty Period, Innov-X will repair or replace the defective component parts. All component parts removed under this Limited Warranty become the property of Innov-X. In the unlikely event that your Innov-X analyzer has a recurring failure, Innov-X, at its discretion, may elect to provide you with a replacement unit of Innov-X's choosing that is at least equivalent to your Innov-X analyzer. This is your exclusive remedy for defective products. The repaired or replacement analyzer is warranted for the remainder of the limited Warranty Period.

YOU SHOULD MAKE PERIODIC BACKUP COPIES OF THE DATA STORED ON YOUR ANALYZER AS A PRECAUTION AGAINST POSSIBLE FAILURES, ALTERATION, OR LOSS OF THE DATA. BEFORE RETURNING ANY UNIT FOR SERVICE, BE SURE TO BACK UP DATA AND REMOVE ANY CONFIDENTIAL, PROPRIETARY, OR PERSONAL INFORMATION. INNOV-X IS NOT RESPONSIBLE FOR DAMAGE TO OR LOSS OF ANY PROGRAMS, OR DATA. INNOV-X IS NOT RESPONSIBLE FOR THE RESTORATION OR REINSTALLATION OF ANY PROGRAMS OR DATA OTHER THAN SOFTWARE INSTALLED BY INNOV-X WHEN THE ANALYZER IS MANUFACTURED.

Innov-X does not warrant that the operation of this analyzer will be uninterrupted or error-free. Innov-X is not responsible for damage that occurs as a result of your failure to follow the instructions that came with the Innov-X analyzer.

This Limited Warranty does not apply to expendable parts. This Limited Warranty does not extend to any analyzer from which the serial number has been removed or that has been damaged or rendered defective (A) as a result of accident, misuse, abuse, or other external causes; (b) by operation outside the usage parameters stated in user documentation that shipped with the product; (c) by modification or service by anyone other than (i) Innov-X, or(ii) a Innov-X authorized service provider, (d) installation of software not approved by Innov-X.

These terms and conditions constitute the complete and exclusive warranty agreement between you and Innov-X regarding the Innov-X analyzer you have purchased or leased. These terms and conditions supersede any prior agreements or representations --- including representations made in Innov-X sales literature or advice given to you by Innov-X or any agent or employee of Innov-X --- that may have been made in connection with your purchase or lease of the Innov-X analyzer. No change to the conditions of this Limited Warranty is valid unless it is made in writing and signed by an authorized representative of Innov-X.

Limitation of Liability

IF YOUR INNOV-X ANALYZER FAILS TO WORK AS WARRANTED ABOVE, YOUR SOLE AND EXCLUSIVE REMEDY SHALL BE REPAIR OR REPLACEMENT. INNOV-X'S MAXIMUM LIABILITY UNDER THIS LIMITED WARRANTY IS EXPRESSLY LIMITED TO THE LESSER OF THE PRICE YOU HAVE PAID FOR THE ANALYZER OR THE COST OF REPAIR OR REPLACEMENT OF ANY COMPONENTS THAT MALFUNCTION IN CONDITION OF NORMAL USE.

INNOV-X IS NOT LIABLE FOR ANY DAMAGE CAUSED BY THE PRODUCT OR THE FAILURE OF THE PRODUCT TO PERFORM INCLUDING ANY LOST PROFITS OR SAVINGS OR SPECIAL, INCIDENTAL, OR CONSEQUENTIAL DAMAGES. INNOV-X IS NOT LIABLE FOR ANY CLAIM MADE BY A THIRD PARTY OR MADE BY YOU FOR A THIRD PARTY.

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Software

This Limited Warranty does not warrant software products. The Innov-X software installed on your analyzer is covered by the Innov-X Software License.

Warranty Period

The warranty period for a Model XT-245 or Model XT-260 Innov-X analyzer is two years or four thousand hours of use, whichever occurs first. The warranty for all other analyzers is one year or two thousands hours of use whichever occurs first. This warranty does not extend to expendable parts. Extended warranties are available from Innov-X.

Warranty Returns

A Return Material Authorization (RMA) Number must be obtained from the INNOV-X Service Department before any items can be shipped to the factory. Returned goods will not be accepted without an RMA Number. Custorner will bear all shipping charges for warranty repairs. All goods returned to the factory for warranty repair should be properly packed to avoid damage and clearly marked with the RMA Number.

Warranty Repairs

Warranty repairs will be done either at the customer's site or at the INNOV-X plant, at our option. All service rendered by INNOV-X will be performed in a professional manner by qualified personnel.

Contacting Innov-X

Be sure to have the following information available before you call Innov-X:

- Analyzer serial number, model name, and model number
- Applicable error messages
- Description of problem
- Detailed questions

Methods of Contact

- Phone: 781-635-5005
- Fax 781-938-0128
- Email service@Innov-Xsys.com
- Mail & Shipping Address: Innov-X Systems, Inc. 100 Sylvan Road, Suite 100 Woburn MA 01801