

Thermo Fisher Scientific Niton Analyzers

XL3 Analyzer

Version 7.0.1

User's Guide

Revision C

November 2010



The preferred source for instrument Rentals, Sales, Service and Supplies!

4201 B Street Anchorage, AK 99503 (907) 770-9041

www.tttenviro.com

25420 74th Avenue S. Kent, WA 98032 (253) 373-9041

info@tttenviro.com



 $\hbox{@ 2010}$ Thermo Fisher Scientific Inc. All rights reserved.

Thermo Fisher Scientific Inc. provides this document to its customers with a product purchase to use in the product operation. This document is copyright protected and any reproduction of the whole or any part of this document is strictly prohibited, except with the written authorization of Thermo Fisher Scientific Inc.

The contents of this document are subject to change without notice. All technical information in this document is for reference purposes only. System configurations and specifications in this document supersede all previous information received by the purchaser.

Thermo Fisher Scientific Inc. makes no representations that this document is complete, accurate or errorfree and assumes no responsibility and will not be liable for any errors, omissions, damage or loss that might result from any use of this document, even if the information in the document is followed properly.

This document is not part of any sales contract between Thermo Fisher Scientific Inc. and a purchaser. This document shall in no way govern or modify any Terms and Conditions of Sale, which Terms and Conditions of Sale shall govern all conflicting information between the two documents.

Release history:

For Research Use Only. Not for use in diagnostic procedures.

Chapter 0	Contact Us	1
Chapter 1	Manual Overview	1
•	Warnings, Cautions, and Notes	1
	Figures	1
	Physical Buttons	2
	Other Hardware	2
Chapter 2	Using Your Analyzer	3
	Safely and Effectively Using Your Analyzer	
	Monitoring your radiation exposure	
	Safe Handling of Samples	
	Niton XL3t Radiation Profile	
	Niton XL3t GOLDD Plus Radiation Profile.	
	Niton XL3p Radiation Profile	
	Primary Radiation	
	Secondary Radiation	
	Deep and Shallow Dose.	
	Proper and Improper Operation	
	Emergency Response Information	
	Startup Procedure	
	Battery Installation and Charging	
	The Control Panel.	
	Startup Procedure	40
	Performing a System Check	
	The Data Ports	
	Power Port	49
	Using the Navigation (NAV) Menu	
	Using the Tools Menu	
	Setting the Date and Time	
	Data Entry	60
	The Results Screen	62
Chapter 3	How to Analyze	65
Chapter 4	Metal Sample Prep	67
Chapter 5	Metals Analysis	81
•	General Metals Analysis.	
	PMI Analysis	89
	FAC Analysis	
	Coatings Analysis	
	Preparatory Tasks	
	Coatings Entries and Standardization	
	General Metals Analysis.	

Thermo Scientific manual-name 1

	PMI Analysis	
Chapter 6	Coatings Analysis	
Chapter 7	Mining Analysis	1/10
Gliaptei 1	Niton XL3 X-Ray Fluorescence (XRF) Analyzer – Version 7.0 and	173
	Higher	149
	Grade Control Samples	
	Blast Hole Samples	
	Process Control Samples	157
	Powders and Fine Aggregates	157
	Concentrates	158
	Slurry	159
Chapter 8	Soil Analysis	161
onaptor o	Niton XL3 X-Ray Fluorescence (XRF) Analyzer – Version 7.0 and	
	Higher	161
Chapter 9	Consumer Goods Analysisniton	160
Gliaptei 3	Niton XL3 X-Ray Fluorescence (XRF) Analyzer – Version 7.0 and	103
	Higher	169
	APPENDIX A.	
	Appendix B	
Chautau 10	Cuantual Financial	207
Chapter 10	Spectral Fingerprint	207
Chapter 11	Helium Purged Analysis	211
Chapter 12	Data Management	219
Onaptor 12	Viewing Data	
	Viewing Fingerprints	
	Erasing Data	
	Managing Libraries	
Chapter 13	Connectivity	220
Gliapter 13	Using Your Analyzer With Your PC	
	Starting Niton Data Transfer Software	
	Connecting the XRF Analyzer to Your PC	
	Connecting From Your Analyzer to Your PC	
	Using a USB Cable to Connect Your Analyzer	
	Downloading Data	
	Creating and Using Reports	
	Viewing the Spectra Graph	
	Viewing Different Areas of the Spectra Graph	
	Viewing the X-ray Line List	

2 manual-name Thermo Scientific

	Using the Menus Instead of the Toolbar	311
	Renaming a Report	317
	Saving Selected Readings from a Report	318
	Copying a Report Into Excel	319
	Printing Certificates of Analysis	325
	Closing a Report	330
	Deleting a Report	331
	Printing from NDT	332
	Creating a Report File from Multiple Source Files	
	Controlling Your Analyzer From Your PC	351
	Setting up Bluetooth	355
	Connecting the Bluetooth device to your XRF Analyzer	361
	Troubleshooting Your Connection	374
Chapter 14	Interfacing Thermo Scientific Niton Analyzers and the Trimble/ArcPad	
Спарсет 14	GIS System383	
	Setting Up Your Trimble Handheld	383
	Setting Up Your Analyzer	
	Installing ArcPad GIS Software To Your PC	389
	Deploying ArcPad to the Trimble and Setting Up GPS	398
	Setup the ArcPad Port for Receiving Data from the Analyzer	403
	Installing the Analyzer Applet	406
	Using the Analyzer Applet	409
	Trimble-Niton Error Messages	414
Chapter 15	Parts	417
	Parts and Accessories	
Chapter 16	Extend-a-Pole	431
-	Setting Up the Extend-a-Pole	431
Chapter 17	Test Stands	ЛЛ1
onapter 17	The Portable Test Stand	
	The Mobile Test Stand	
	Configuring the Analyzer for the Test Stand	
	The Field Mate Test Stand	
	Configuring the Analyzer for the Test Stand	
	Reference Samples	
	Electronics Metals Reference (Standard) Sample Care	
	Lead Paint Reference (Standard) Sample Care	
	Small Spot Reference (Standard) Sample Care	
	oman opol reference (otanuaru) oampie Cale	4/4

Thermo Scientific manual-name 3

Chapter 18	Learning More, Service, and Support	475
•	Tips and Troubleshooting	
	Registration and Licensing FAQ	
	Storing and Transporting Your Niton XL3 Analyzer	
	Advanced Settings	489
	Tools Menu Options	
	NDF Files: User Data Structuring	
	Pseudo-Elements	
	TestAll Geo	
	Safety Settings	557
	Measuring the Standard Samples	
	Calculating Calibration Factors Using Excel	
	Uploading Calibration Factors from your PC	
	Camera and Small Spot Video	
	Reference Documents	
	Service	
	Warranty	

4 manual-name Thermo Scientific

Contact Us

Niton Analyzers HeadquartersEurope and AfricaAsia and Middle East900 Middlesex TurnpikeJoseph-Dollinger-Bogen 9Room 1101,Building #880807 Munich11/F Asia Standard TowerBillerica, MA 01821Germany59-65 Queen's RoadUSAPhone: +49 89 3681 380Central, Hong KongTel: 800-875-1578Fax: +49 89 3681 3830Phone: +852 2869-6669Tel: +1 978-670-7460Fax: +852 2869-6665Fax: +1 978-670-7430niton.eur@thermofisher.comniton@thermofisher.comNitonEurope.support@thermofisher.comniton.asia@thermofisher.comNitonAsia.Support@thermofisher.com	Americas	Europe	Asia
Building #8 80807 Munich 11/F Asia Standard Tower Billerica, MA 01821 Germany 59-65 Queen's Road USA Phone: +49 89 3681 380 Central, Hong Kong Tel: 800-875-1578 Fax: +49 89 3681 3830 Phone: +852 2869-6669 Tel: +1 978-670-7460 Fax: +852 2869-6665 Fax: +1 978-670-7430 niton.eur@thermofisher.com NitonEurope.support@thermofisher.com niton.asia@thermofisher.com	Niton Analyzers Headquarters	Europe and Africa	Asia and Middle East
Billerica, MA 01821 Germany 59-65 Queen's Road USA Phone: +49 89 3681 380 Central, Hong Kong Tel: 800-875-1578 Fax: +49 89 3681 3830 Phone: +852 2869-6669 Tel: +1 978-670-7460 Fax: +852 2869-6665 Fax: +1 978-670-7430 niton.eur@thermofisher.com niton.asia@thermofisher.com NitonEurope.support@thermofisher.com niton.asia@thermofisher.com	900 Middlesex Turnpike	Joseph-Dollinger-Bogen 9	Room 1101,
USA	Building #8	80807 Munich	11/F Asia Standard Tower
Tel: 800-875-1578 Fax: +49 89 3681 3830 Phone: +852 2869-6669 Tel: +1 978-670-7460 Fax: +852 2869-6665 Fax: +1 978-670-7430 niton.eur@thermofisher.com NitonEurope.support@thermofisher.com	Billerica, MA 01821	Germany	59-65 Queen's Road
Tel: +1 978-670-7460 Fax: +852 2869-6665 Fax: +1 978-670-7430 niton.eur@thermofisher.com NitonEurope.support@thermofisher.com NitonEurope.support@thermofisher.com	USA	Phone: +49 89 3681 380	Central, Hong Kong
Fax: +1 978-670-7430 niton@thermofisher.com niton@thermofisher.com NitonEurope.support@thermofisher.com NitonEurope.support@thermofisher.com	Tel: 800-875-1578	Fax: +49 89 3681 3830	Phone: +852 2869-6669
niton.eur@thermofisher.com niton@thermofisher.com NitonEurope.support@thermofisher.com niton.asia@thermofisher.com	Tel: +1 978-670-7460		Fax: +852 2869-6665
niton@thermofisher.com	Fax: +1 978-670-7430		
	niton@thermofisher.com		

Ocontact Us

Manual Overview

Warnings, Cautions, and Notes

Warnings

Warnings are extremely important recommendations, violating which may result in either injury to yourself or others, or damage to your analyzer and/or data. Warnings will always be identified as Warnings in the text, and will always be visually presented as follows:

WARNING This is a Warning.

Example Warning:

WARNING Tampering with the 5,500 ppm (Lead high) lead-in-soil standard may cause exposure to lead dust. Keep all standards out of reach of children.

Cautions

Cautions are important recommendations. Cautions will always be identified as Cautions in the text, and will always be visually presented as follows:

CAUTION This is a Caution.

Example Caution:

CAUTION Never tamper with Test Standards. They should not be used unless they are completely intact

Notes

Notes are informational asides which may help you with your analyses. Notes will always be identified as Notes in the text, and will always be visually presented as follows:

Note This is a Note.

Example Note:

Note For defensible Quality Control, keep a record of the time and precision of every calibration

Figures

Figures are illustrations used to show what something looks like. Figures will always be labelled and identified as Figures directly below the Figure itself, and will always be visually presented as follows:

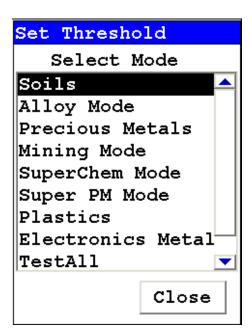


Figure 1. This is a Figure

Physical Buttons

Physical Buttons are actual buttons on the analyzer which must be pushed to activate their function. Physical Buttons will always be identified as Buttons in the text, and will always be visually presented as follows:

This is a Physical Button.

Example Physical Buttons:

On/Off/Escape Button, Clear/Enter Button, Interlock Button, and Trigger Button.

Other Hardware

Other Hardware refers to any physical part of the analyzer which performs a necessary function. Other Hardware will always be visually presented as follows:

This is an example of Other Hardware.

Example Other Hardware:

Battery, Touch Screen Display, Measurement Window, and USB Cable

Using Your Analyzer

This section discusses the basics of using your analyzer, no matter the specific type of analysis you wish to perform. First we go over analyzer safety, particularly radiation safety. Using an X-ray based analyzer safely is very important, and not difficult, provided you read, understand, and follow these guidelines. Secondly, we outline the startup procedure we recommend for daily use to ensure that your analyzer is performing properly and at its most efficient level.

Safely and Effectively Using Your Analyzer

CAUTION Niton analyzers are not intrinsically safe analyzers. All pertinent Hot Work procedures should be followed in areas of concern.

Radiation and General Safety

WARNING Always treat radiation with respect. Do not hold your analyzer near the measurement window during testing. Never point your analyzer at yourself or anyone else when the shutter is open.

Radiation and General Safety

This section covers topics related to radiation safety and general safety when using a Thermo Scientific Niton XL3 analyzer. At a minimum all operators of the analyzer should be familiar with the instructions provided in this chapter in order to handle the analyzer in a safe manner. In addition to reading the information presented on the following pages, Thermo Fisher Scientific recommends that instrument users participate in a radiation safety and operational training class.

Radiation Protection Basics

The Niton Model XL3t analyzer contains an x-ray tube which emits radiation only when the user turns the x-ray tube on. When the x-ray tube is on and the shutter is open, as during a measurement, the analyzer emits a directed radiation beam - see Figures 1 and 2. Reasonable effort should be made to maintain exposures to radiation as far below dose limits as is practical. This is known as the ALARA (As Low as Reasonably Achievable) principle. For any given source of radiation, three factors will help minimize your radiation exposure: Time, Distance, and Shielding.

The Niton Model XL3p analyzer contains a radioactive sealed source. Radiation from this source is fully contained within the device when not in use and allowed to escape through the measurement window only while the user is analyzing a sample. Radiation emission is controlled by a shutter. The analyzer emits a directed radiation beam (See Figure 1 and Figure

2 Using Your Analyzer Safely and Effectively Using Your Analyzer

2) when the shutter is open during a measurement. Reasonable effort should be made to maintain exposures to radiation as far below dose limits as is practical. This is known as the ALARA (As Low as Reasonably Achievable) principle. For any given source of radiation, three factors will help minimize your radiation exposure: Time, Distance, and Shielding.

Time

The longer you are exposed to a source of radiation the longer the radiation is able to interact in your body and the greater the dose you receive. Dose increases in direct proportion to length of exposure.

Distance

The closer you are to a source of radiation, the more radiation strikes you. Based on geometry alone, dose increases and decreases with an inverse-squared relation to your distance from the source of radiation (additional dose rate reduction comes from air attenuation). For example, the radiation dose one foot from a source is nine times greater than the dose three feet from the source. Remember to keep your hands and all body parts away from the front end of the analyzer when the shutter is open to minimize your exposure.

Shielding

Shielding is any material that is placed between you and the radiation source. The more material between you and the source, or the denser the material, the less you will be exposed to that radiation. Supplied or optional test stands are an additional source of shielding for analysis. A backscatter shield accessory is also available and may be appropriate in some applications.

Exposure to Radiation

Human dose to radiation is typically measured in rem, or in one-thousandths of a rem, called millirem (mrem), 1 rem = 1000 mrem. Another unit of dose is the Sievert (Sv), 1 Sv = 100 rem. The allowable limit for occupational exposure in the U.S (and many other countries) is 5,000 mrem/year (50 mSv/year) for deep (penetrating) dose and 50,000 mrem/year (500 mSv/year) for shallow (i.e., skin) dose or dose to extremities. Deep, shallow, and extremity exposure from a properly used Niton XL3t analyzer should be less than 200 mrem per year, (2.0 mSv per year) even if the analyzer is used as much as 2,000 hours per year, with the shutter open continuously. The only anticipated exceptions to the 200 mrem maximum annual dose are: 1) routine and frequent analysis of plastic samples without use of a test stand, backscatter shield, or similar additional protective measures, or 2) improper use where a part of the body is in the primary beam path.

Note NEVER OPERATE THE DEVICE WITH A PART OF YOUR BODY IN THE PRIMARY BEAM PATH OR WITH THE PRIMARY BEAM PATH DIRECTED AT ANYONE ELSE.

Also, consider the use of protective accessories such as a shielded test stand or backscatter shield (or equivalent) when performing routine and/or frequent analysis of any of the following:

- plastic (or similarly low density) samples,
- thin samples (such as foils, circuit boards, and wires)
- samples that are smaller than the analysis window.

Shown in Table 1 are the typical background radiation doses received by the average member of the public. The radiation dose limits for radiation workers in the US are also shown in Table 2.

Table 1. Typical Radiation Doses Received (Source: NCRP 1987)

Category	Dose in mrem	Dose in mSv
Average total dose in US (annual)	360	3.6
Average worker exposure (annual)	210	2.1
Average exposure for an underground miner	400	4.0
Exposure for airline crew (1,000 hours at 35,000 ft)	500	5.0
Additional from living in Denver at 5300' (annual)	25	.25
Additional from 4 pCi/l radon in home	1,000	10.0
Typical Chest X-Ray	6	0.06
Typical Head or Neck X-Ray	20	0.2
Typical pelvis/hip x-ray	65	0.65
Typical lumbar spine x-ray	30	0.3
Typical Upper G.I. x-ray	245	2.45
Typical Barium enema x-ray	405	4.05
Typical CAT scan	110	1.10

Table 2. Annual Occupational Dose Limits for Radiation Workers

(Source: Code of Federal Regulations Title 10, Part 20)

Category	Dose in mrem	Dose in mSv	
Whole Body	5000	50	
Pregnant Worker (during gestation period)	500	5	
Eye Dose Equivalent	15,000	150	
Shallow dose equivalent to the skin or any extremity or organ	50,000	500	
Maximum allowable dose for the general public (annual)	100	1.0	
For a Minor	500	5.0	

Monitoring your radiation exposure

Individuals can be monitored for the radiation dose they receive by use of radiation dosimetry devices (dosimeters). Monitoring dose using a dosimeter can be a way of identifying improper use and at the same time demonstrating proper use. In some locations, dosimetry is required by regulations and in others it is optional. It is normally required when the user could reasonably be expected to receive in excess of 10% of the annual dose limit. Thermo Fisher Scientific recommends that you determine and obey the local regulatory requirements concerning radiation monitoring of occupational workers.

Two common types of dosimeters are whole-body badges and ring badges. Whole body badges are often attached to the user's torso (e.g., clipped to the collar, shirt pocket, or waist as appropriate). A ring badge is worn on the finger as a measure of maximum extremity dose. When worn, the specific location of the dosimeter should be that part of the body that is expected to receive the highest dose. This location will depend on how the analyzer is used and so it may not be the same for all users. Dosimetry services are offered by many companies. Two companies offering dosimetry services in the USA and much of the world are:

Table 3. Dosimeters

Company	Global Dosimetry Solutions	Landauer, Inc.
Address	2652 McGaw Avenue	2 Science Road
City and State	Irvine, CA 92614	Glenwood, IL 60425-9979
Website	www.dosimetry.com	www.landauerinc.com
Phone Number	(800) 251-3331	(800) 323-8830

Note Wearing a dosimeter badge does not protect you against radiation exposure. A dosimeter badge only measures your exposure (at the dosimeter location).

Pregnancy and Radiation Exposure

International guidance documents (e.g., ICRP Publication 60 and NCRP Publication 116*) recommend that the radiation dose to the embryo/fetus of a pregnant woman should not exceed a total of 500 mrem (10% of normal radiation worker limit) during the gestation period. While this dose limit exceeds the dose limit to a trained operator, pregnant workers may want to take special precautions to reduce their exposure to radiation. For more information see the U.S. NRC Regulatory Guide 8.13 "Instruction Concerning Prenatal Radiation Exposure" which can be found on the resource CD.

- * The International Commission on Radiological Protection, ICRP, is an independent Registered Charity, established to advance for the public benefit the science of radiological protection, in particular by providing recommendations and guidance on all aspects of protection against ionizing radiation.
- * The National Council on Radiation Protection and Measurements (NCRP) was chartered by the U.S. Congress in 1964 as the National Council on Radiation Protection and Measurements.

How to Use the Niton XL3t Analyzer Safely

The Niton XL3t analyzer is designed to be safe to operate provided that it is used in accordance with manufacturer's instructions. Under conditions of normal use, monitored operators seldom receive a measurable dose and have not been known to receive in excess of 10% of the annual occupational dose limits (a criteria that would require monitoring under regulation in the U.S.). In addition to proper use of the XL3t, it is recommended that you follow these precautions to ensure your safety and the safety of those around you.

Know where the beam is

The primary beam is a directed beam out of the front of the analyzer that can have high dose rates. The secondary beam, or scattered beam, has much lower dose rates.

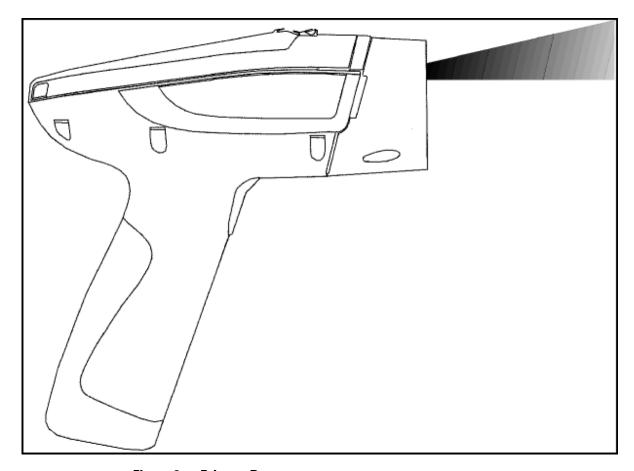


Figure 2. Primary Beam

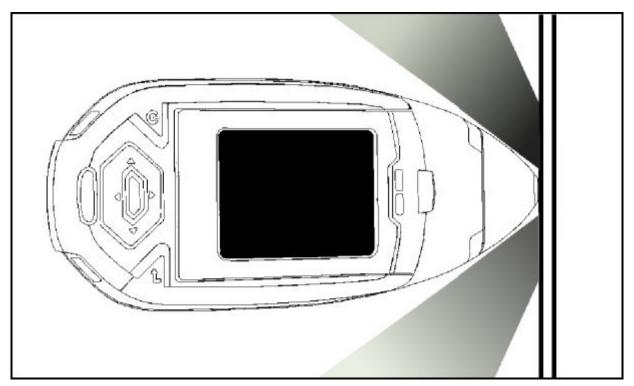


Figure 3. Secondary (Scattered) Beam

The Shutter-Open Indicator Lights

When the lights are flashing, the primary beam is on, and radiation is being emitted from the front of the analyzer.

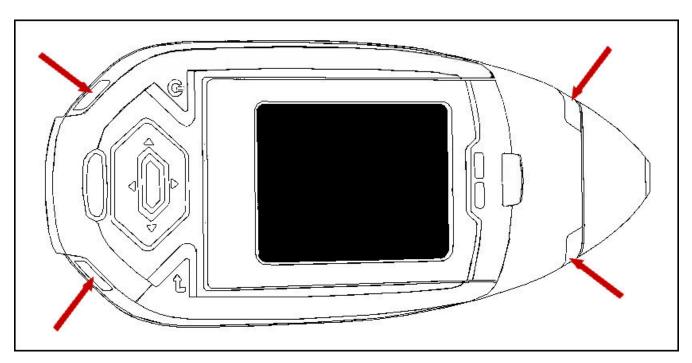


Figure 4. The X-ray Beam Indicator Lights

Handle and Use with Respect

Avoid holding the front of the analyzer when the x-ray tube is energized and the shutter is open. Never point the instrument at yourself or anyone else when the shutter is open and the x-ray tube is energized. Never look into the path of the primary beam.

Follow a Radiation Protection Program

Your organization should establish, document, and follow a Radiation Protection Program. An example of such a program can be found on the resource CD (provided with the instrument).

Take Proper Care of your Niton XL3

Keeping your analyzer maintained in good condition will help minimize the risk of accidental exposure. Mechanical malfunction of the shutter can be avoided by maintaining the measurement window, as described in the User Guide. This prevents foreign objects from entering your analyzer

Avoid Over-Exposures

Direct contact with the window could result in overexposures in the times indicated in Table 4 below.

Table 4. Potential Exposure Limit Times

Location of Dose	Limit	Time to Reach Limit
Deep Dose / Whole Body	5 rem (50 mSv)	2.1 minutes
Shallow Dose / Extremities	50 rem (500 mSv)	0.95 minutes
Member of Public (i.e. untrained operator)	0.1 rem (1 mSv)	2.5 seconds*

Extremity is defined by the NRC as the hand, elbow, arm below the elbow, foot, knee, or leg below the knee. Whole Body is defined by the NRC as the head, trunk (including male gonads), arms above the elbow, or legs above the knee.

Safe Handling of Samples

As mentioned many times in this chapter, never place any part of your body in the path of the x-ray beam. There is always a safe way to handle samples whether they are small, irregularly shaped, or of low density. Never look into the path of the primary beam.

Small Samples

A small sample would be any sample that is smaller than the measurement window. Small samples present a unique risk because they don't block the entire beam path. The difficulty with placing small samples down on a work surface to analyze them is that you may get readings from the work surface that interfere with analytical results. A test stand is an effective way of analyzing small samples accurately and safely. Never hold samples during analysis or look into the path of the primary beam.

^{*}Based on maximum deep dose rate and US exposure limit.

2 Using Your Analyzer Safe Handling of Samples

Irregularly Shaped Samples

Irregularly shaped samples may not allow the proximity button to be depressed, or they may not entirely cover the primary beam and cause additional scattering. A back scatter shield is a safe way of reducing your radiation exposure while effectively analyzing an irregularly shaped sample.

Low Density Materials (such as plastics).

X-rays are attenuated more through denser materials and less through low density materials such as plastic. This causes higher dose rates in the scattered radiation. If you are frequently handling low density samples, you should consider the use of test stands, backscatter shields, or the equivalent.

Niton XL3t Radiation Profile

Radiation Meter Information

Model: Bicron MicroRem

SN: 2057

Cal Due: 10/10/2009

Background Radiation Level

<0.01 mr/hr

Table 1-4 - Niton XL3t Radiation Profile - Scatter Measurements - mRem/hr

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
50	40	EM, GM, S, T, IP, PM, DA, M, E, P (Main Filter)	Plastic	40	3.5	2
		S, T, M, E (High Filter)				
50	40	EM, GM, S, T, IP, PM, DA, M, E, P (Main Filter)	Soil	8	0.4	0.07
		S, T, M, E (High Filter)				
20	100	S, P, T, M, E (Low Filter)	Aluminum	0.015	0.01	0.01
20	100	S, P, T, M, E (Low Filter)	Stainless	0.015	0.01	0.01
20	100	S, P, T, M, E (Low Filter)	Plastic	0.13	0.015	0.015
20	100	S, P, T, M, E (Low Filter)	Soil	0.015	0.015	0.015
15	100	IP, EM (Low Filter)	Aluminum	0.015	0.015	0.015
15	100	IP, EM (Low Filter)	Stainless	0.015	0.015	0.015

^{*} GM = General Metals, EM = Electronics Metals, DA = Dental Alloy, PM = Precious Metals, M = Mining, S = Soil, E = Exploration, IP = Industrial Paint, T = Thin Sample, P = Plastic

Scatter Measurements off various substrates - Dose Rates in mRem/hr

2 Using Your Analyzer Niton XL3t Radiation Profile

Table 1-5 - Niton XL3t Radiation Profile - Scatter Measurements - $\mu Sv/hr$

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
50	40	EM, GM, S, T, IP, PM, DA, M, E (P (Main Filter) S, T, M, E (High Filter)	Plastic	400	35	20
50	40	EM, GM, S, T, IP, PM, DA, M, E (P (Main Filter) S, T, M, E (High Filter)	Soil	80	4	0.7
20	100	S, P, T, M, E (Low Filter)	Aluminum	0.15	0.1	0.1
20	100	S, P, T, M, E (Low Filter)	Stainless	0.15	0.1	0.1
20	100	S, P, T, M, E (Low Filter)	Plastic	1.3	0.15	0.15
20	100	S, P, T, M, E (Low Filter)	Soil	0.15	0.15	0.15
15	100	IP, EM (Low Filter)	Aluminum	0.15	0.15	0.15
15	100	IP, EM (Low Filter)	Stainless	0.15	0.15	0.15

Notes:

Scatter measurements were taken at a radius of 5 or 30 cm around the nose of the analyzer with the highest scatter dose rate being recorded.

Scatter Measurements off various substrates - Dose Rates in $\mu Sv/hr$

^{*} GM = General Metals, EM = Electronics Metals, DA = Dental Alloy, PM = Precious Metals, M = Mining, S = Soil, E = Exploration, IP = Industrial Paint, T = Thin Sample, P = Plastic

Table 1-6 Niton XL3t Radiation Profile - In Beam Measurements - Rem/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30 cm Shallow
50	40	EM, GM, S, T, IP, PM, DA, M, E, P (Main Filter) S, T, M, E (High Filter)	110	410	8.4	1.3
20	100	S, P, T, M, E (Low Filter)	150	3200	0.52	0.05
15	100	IP, EM (Low Filter)	14.0	1100	0.43	0.042

In Beam Measurements - Dose Rates in Rem/hr

Reported results are based on measurement results that have been reduced to 2 significant digits by rounding up. For example, a measurement result of 1441 would be reported as 1500.

Table 1-7 Niton XL3t Radiation Profile - In Beam Measurements - mSv/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30cm Shallow
50	40	EM, GM, S, T, IP, PM, DA, M, E, P (Main Filter) S, T, M, E (High Filter)	1100	4,100	84.0	13
20	100	S, P, T, M, E (Low Filter)	1500	32000	5.2	0.50
15	100	IP, EM (Low Filter)	140	11000	4.3	0.42

Notes:

^{*} GM = General Metals, EM = Electronics Metals, DA = Dental Alloy, PM = Precious Metals, M = Mining, S = Soil,

E = Exploration, IP = Industrial Paint, T = Thin Sample, P = Plastic

In beam dose rates were measured using thermoluminescent dosimeters (TLDs) or Optically Stimulated Luminescent Dosimeters (OSL).

In Beam Measurements - Dose Rates in mSv/hr

^{*} GM = General Metals, EM = Electronics Metals, DA = Dental Alloy, PM = Precious Metals, M = Mining, S = Soil,

E = Exploration, IP = Industrial Paint, T = Thin Sample, P = Plastic

Reported results are based on measurement results that have been reduced to 2 significant digits by rounding up. For example, a measurement result of 1441 would be reported as 1500.

Niton XL3t GOLDD Plus Radiation Profile

Table 1-8 - Niton XL3t GOLDD Plus Radiation Profile - In Beam Measurements - mSv/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30cm Deep
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	440	1300	74	5.3
50	40	GM, PM, M, S, IP (Main)	1500	3800	360	22
20	100	EM, P, TA, M, S, T (Low)	690	19000	190	9.2
15	133.3	GM (Low)	240	13000	50	2.3
8	200	GM, P, M (Light)	0.30	17000	0.10	< 0.003

Notes:

*SAMPLE TYPES (MODES)

GM=General Metals, M=Mining, EM=Electronics Metals, S=Soils, PM=Precious Metals, IP=Industrial Paint (Action lead Paint & Quantify lead Paint), MC=Metal Coatings, PP=Painted Products, P=Plastics, TG=Test All Geo (soil and mining), TA=Test All (consumer products), T=Thin

Reported results are based on measurement results that have been reduced to 2 significant digits by rounding up. For example, a measurement result of 1441 would be reported as 1500.

Table 1-9 - Niton XL3t GOLDD Plus Radiation Profile - In Beam Measurements - Rem/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30cm Deep
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	44	130	7.4	0.53
50	40	GM, PM, M, S, IP (Main)	150	380	36	2.2
20	100	EM, P, TA, M, S, T (Low)	69	1900	19	0.92
15	133.3	GM (Low)	24	1300	5.0	0.23
8	200	GM, P, M (Light)	0.030	1700	0.010	< 0.0003

Notes:

*SAMPLE TYPES (MODES)

GM=General Metals, M=Mining, EM=Electronics Metals, S=Soils, PM=Precious Metals, IP=Industrial Paint (Action lead Paint & Quantify lead Paint), MC=Metal Coatings, PP=Painted Products, P=Plastics, TG=Test All Geo (soil and mining), TA=Test All (consumer products), T=Thin

Reported results are based on measurement results that have been reduced to 2 significant digits by rounding up. For example, a measurement result of 1441 would be reported as 1500.

Table 1-10 - Niton XL3t GOLDD Plus Radiation Profile - Scatter Measurements - mRem/hr

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Steel	0.14	<0.01	<0.01
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Aluminum	2	<0.01	<0.01
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Soil	2	0.04	<0.01
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Plastic	10	0.45	6
50	40	GM, PM, M, S, IP (Main)	Steel	0.3	<0.01	<0.01
50	40	GM, PM, M, S, IP (Main)	Aluminum	4	0.01	0.01
50	40	GM, PM, M, S, IP (Main)	Soil	4	0.09	<0.01
20	100	EM, P, TA, M, S, T (Low)	Soil	<0.01	<0.01	<0.01
20	100	EM, P, TA, M, S, T (Low)	Plastic	0.07	<0.01	<0.01
15	133.3	GM (Low)	Steel	<0.01	<0.01	<0.01

Table 1-10 - Niton XL3t GOLDD Plus Radiation Profile - Scatter Measurements - mRem/hr

15	133.3	GM (Low)	Aluminum	< 0.01	< 0.01	< 0.01
8	200	GM, P, M (Light)	<0.01 (no detectable scatter radiation) at any location for steel, aluminum, soil, or plastic sample types			

Notes:

GM=General Metals, M=Mining, EM=Electronics Metals, S=Soils, PM=Precious Metals, IP=Industrial Paint (Action lead Paint & Quantify lead Paint), MC=Metal Coatings, PP=Painted Products, P=Plastics, TG=Test All Geo (soil and mining), TA=Test All (consumer products), T=Thin

Table 1-11 - Niton XL3t GOLDD Plus Radiation Profile - Scatter Measurements - μSv/hr

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Steel	1.4	<0.1	<0.1
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Aluminum	20	<0.1	<0.1
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Soil	20	0.4	<0.1
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Plastic	100	4.5	60
50	40	GM, PM, M, S, IP (Main)	Steel	3	<0.1	<0.1
50	40	GM, PM, M, S, IP (Main)	Aluminum	40	0.1	0.1

^{*}SAMPLE TYPES (MODES)

Table 1-11 - Niton XL3t GOLDD Plus Radiation Profile - Scatter Measurements - $\mu Sv/hr$

50	40	GM, PM, M, S, IP (Main)	Soil	40	0.9	<0.1
20	100	EM, P, TA, M, S, T (Low)	Soil	<0.1	<0.1	<0.1
20	100	EM, P, TA, M, S, T (Low)	Plastic	0.7	<0.1	<0.1
15	133.3	GM (Low)	Steel	<0.1	<0.1	<0.1
15	133.3	GM (Low)	Aluminum	<0.1	<0.1	<0.1
8	200	GM, P, M (Light)	<0.1 (no detectable scatter radiation) at any location for steel, aluminum, soil, or plastic sample types			

Note *SAMPLE TYPES (MODES)

GM=General Metals, M=Mining, EM=Electronics Metals, S=Soils, PM=Precious Metals, IP=Industrial Paint (Action lead Paint & Quantify lead Paint), MC=Metal Coatings, PP=Painted Products, P=Plastics, TG=Test All Geo (soil and mining), TA=Test All (consumer products), T=Thin

Niton XL3p Radiation Profile

Table 1-12 - Niton XL3p Radiation Profile - In Beam Measurements

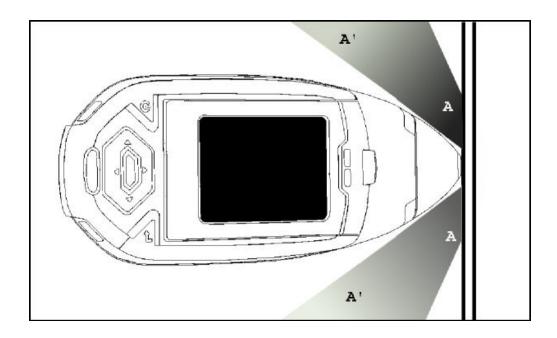
Distance From Window	Dose Rate (mSv/hr)	Dose Rate (mrem/hr)
5 cm	0.45	45
30 cm	0.03	3.0
100 cm	0.003	0.3

Table 1-13 - Niton XL3p Radiation Profile - Scatter Measurements - mSv/hr

Location	Plastic Substrate	Wood Substrate	Soil Substrate	Aluminum Substrate	Steel Substrate
Max Scatter @ 5cm from Snout (A')	0.06	0.03	0.018	0.01	0.0042
Max Scatter @ Trigger (B)	0.0038	0.002	0.0015	0.0048	0.0003

Table 1-14 - Niton XL3p Radiation Profile - Scatter Measurements - mRem/hr

Location	Plastic Substrate	Wood Substrate	Soil Substrate	Aluminum Substrate	Steel Substrate
Max Scatter @ 5cm from Snout (A')	6.0	3.0	1.8	1.0	0.42
Max Scatter @ Trigger (B)	0.38	0.2	0.15	0.48	0.03



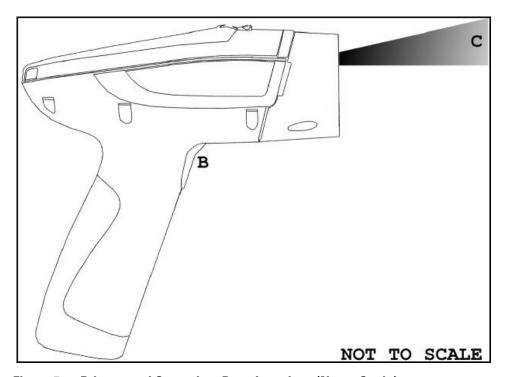


Figure 5. Primary and Secondary Dose Locations (Not to Scale)

Primary Radiation

Niton XL3t and XL3t GOLDD

Primary radiation is radiation that is produced by the analyzer and emitted out through the measurement window. Individuals should never place any part of their body in the primary beam path when the x-ray tube is on. There should always be a sample in contact with the measurement window when the x-ray tube is on. The sample will absorb most of the primary-beam radiation unless it is smaller than the instrument's measurement window or of low density and/or thickness. Caution should be taken when analyzing samples that are small, thin, and/or low in density as they may allow much more of the primary beam to escape. In-beam primary radiation dose rates are listed in Table 1-6, 1-7, 1-8, 1-9, and their location identified relative to the analyzer in Figure 4 as Dose Point C.

Niton XL3p

Primary radiation is radiation that is produced by the analyzer and emitted out through the measurement window. Individuals should never place any part of their body in the primary beam path when the shutter is open. There should always be a sample in contact with the measurement window when the shutter is open. The sample will absorb most of the primary-beam radiation unless it is smaller than the instrument's measurement window or of low density and/or thickness. Caution should be taken when analyzing samples that are small, thin, and/or low in density as they may allow much more of the primary beam to escape. In-beam primary radiation dose rates are listed in Table 1-6, 1-7, 1-8, 1-9, and their location identified relative to the analyzer in Figure 4 as Dose Point C.

Secondary Radiation

Under conditions of normal and proper use, individuals can be exposed to secondary (or "scattered") radiation. Secondary radiation is low-level radiation that emanates from the sample being analyzed as a result of primary beam radiation scattering in the sample or primary beam radiation inducing fluorescent x-rays in the sample. Dose points A, A' and B in Figure 4 are examples of where you can encounter secondary radiation. The magnitude of this secondary radiation is sample dependent. Higher density samples such as steel will emit the lowest levels as they absorb most primary and secondary radiations. Lower density samples such as aluminum, wood, and especially plastic, will produce higher levels of secondary radiation.

Niton XL3t and XL3t GOLDD

Secondary radiation dose rates are listed in Tables 1-4, 1-5, 1-10, and 1-11, for a few common sample types over a wide range of densities.

Niton XL3p

Secondary radiation dose rates are listed in Tables 1-13 and 1-14 for a few common sample types over a wide range of densities.

Holding Samples

The operator is reminded that one should never hold samples during analysis, doing so will result in higher than necessary exposure to secondary radiation and could expose the operator directly to the much higher primary-beam dose rates.

Deep and Shallow Dose

You will find in the tables that shallow dose rates are listed for some dose points. All dose rates listed in the In-Beam Primary Radiation Tables are deep dose unless they are specifically identified as shallow dose. Deep dose is dose from penetrating radiation that is delivered to both skin and underlying tissues and organs and is the type most commonly referred to when describing external radiation hazards. Occupational deep dose is limited to a maximum of 5 rem (50 mSv) per year in the United States and most countries internationally. Deep dose is measured at 1.0 cm below the skin surface.

Shallow dose is often referred to as "skin dose" because it is a result of low penetrating radiation that only interacts with the skin. Shallow dose is limited to a maximum of 50 rem (500 mSv) per year in the United States and most countries internationally. Shallow dose is listed for primary in-beam dose points only because the low penetrating radiation that causes shallow dose is nearly all absorbed by a sample and does not produce any significant secondary radiation. Shallow dose is measured at a point 0.007 cm below the surface.

Proper and Improper Operation

Storage and Transportation

Storage

Regulations in nearly all locations will require that you store your analyzer locked in a secured area to prevent access, use, and/or removal by unauthorized individuals. Storage requirements will vary by location, particularly with regard to storage at temporary job sites or away from your primary storage location such as hotels and motels and in vehicles. You should contact your local Radiation Control Authority to identify the specific storage requirements in your jurisdiction.

Transportation

Niton XL3t and XL3t GOLDD

There are no X-ray tube specific US Department of Transportation (DOT) or International Air Transport Association (IATA) radiation regulations regarding shipping the Niton XL3t analyzer. It is recommended that you ship the analyzer in its carrying case and an over-pack to protect the sensitive measuring equipment inside the analyzer. Do NOT ship the analyzer with the battery pack connected to the analyzer.

Niton XL3p

For Thermo Fisher Scientific, Niton Analyzers (Niton Analyzers), in the United States, the government agency that has primary authority and regulations which apply to transportation is the Department of Transportation (DOT) (Code of Federal Regulations 49 Parts 100 to 185).

In addition, the EPA, OSHA, and the NRC also have regulations that touch on the transportation of hazardous substances.

The International Air Transport Association (IATA) has recommendations that every major air carrier has incorporated into their policies for the air transport of hazardous substances.

The enforcement of DOTregulations is carried out by the Department of Transportation, the Federal Aviation Administration, the Federal Highway Administration, the Federal Railroad Administration, the Coast Guard, and Customs and Border Protection. State agencies may also enforce state DOT regulations.

The major aspects of the regulations include:

- "training to recognize hazards and how to safely deal with hazardous substances,
- "classification and identification of packages to inform of hazards,
- "protective packaging to safely transport hazardous materials,
- "hazard communication to inform personnel of hazards in a package,

and

• "incident reporting to inform regulators of incidents.

As part of shipping hazardous substances, you should be trained in:

- "a general awareness and a familiarity with the general provisions of the DOT and hazardous materials regulations,
- "training that is function specific and be applicable to the daily work performed,
- "the recognition and identification of hazardous substances,

- "the specific requirements for functions performed,
- "security measures to keep a package secure.
- "safety issues as related to safe handling and hazard communication.
- "knowledge of emergency response information, self-protective procedures, and accident prevention procedures.

Employers are responsible for providing the proper training (every three years for DOT) to employees, testing employee knowledge, and record keeping.

The DOT Hazardous Material Regulations set the criteria for determining the hazard class and the proper shipping name for hazardous materials. The Hazard Classes as follows:

Class

- 1. Explosives
- 2. Gases
- 3. Flammable and Combustible Liquids
- 4. Flammable solids, combustible materials, and dangerous when wet materials
- 5. Oxidizers and organic peroxides
- 6. Toxic materials and infectious substances
- 7. Radioactive Materials
- 8. Corrosive Materials
- 9. Miscellaneous dangerous goods

Niton analyzers with radioisotopes are Hazard Class 7. The Hazard Class of the material being transported tells you which Parts of the regulations are required.

A UN number is assigned to each type of hazardous material. It is the letters "UN" followed by a four digit numerical code, which allows emergency responders to identify the chemical being shipped. The UN number for Niton analyzers is "UN 2911".

There is also an official name designation for Niton Analyzers called the Proper Shipping Name. The proper Shipping Name for the Niton Analyzers is "Radioactive material, excepted package-instruments and articles".

A reportable quantity (RQ) is listed in DOT regulations for each hazardous material (e.g., each radioactive isotope). If you are shipping more than the reportable quantity in a package, that package must be marked clear and legibly with the letters "RQ". The Niton XL3p analyzer contains a 30 mCi (1.11 GBq) Am-241 source and is therefore considered a reportable quantity requiring the RQ marking when being transported.

2 Using Your Analyzer Proper and Improper Operation

Also, the quantity of Am-241 that is used in a Model XL3p Analyzer requires a special form certification for transport as an excepted package. This Special Form certificate must accompany the instrument during shipment. Thermo Fisher Scientific will provide the Special Form certificate for any analyzer with Am-241."

The type of protective packaging used is dependent on the nature of the material to be packaged. All packaging must be designed to prevent a release of hazardous material during normal transportation or storage of the material. The classification of package used for Niton Analyzers is designated as an "Excepted Package". Always ship the analyzer in its original plastic case to ensure that the packaging used meets the regulatory requirements for an Excepted Package.

Shipments of radioactive materials must have proper Labeling and Marking.

Niton analyzers have a Marking requirement (i.e., UN number and RQ if applicable), but not a Labeling requirement (i.e., diamond shaped hazmat labels), and vehicles transporting these analyzers are not required to have "Placards".

When reading the DOT regulations, you will find the following information useful.

Thermo Fisher Scientific, Niton Analyzers are shipped:

- "Under the proper shipping name "Radioactive material, excepted package-instruments or articles" in accordance with 49 CFR 173.424,
- \bullet "with the radiation level at 10 cm from the unpacked instrument surface less than 10 mrem/hr (0.1 mSv/hr) "

Note A Niton Analyzer in proper condition will be less than 0.5 mrem/hr (0.005 mSv/hr) at 10 cm.

- "with the radiation level at the package surface less than 0.5 mrem/hr (0.005 mSv/hr)" **Note** A Niton Analyzer in proper condition will be less than 0.05 mrem/hr (0.0005 mSv/hr) at the surface of the case,
- "with all radioactive sources as "solid", "sealed sources"
- "Am-241 listed in A1 column of 173.435 (270 Ci) (Special Form capsule)
- "with the package design meeting the requirements of 173.410
- "package marked with "UN2911"
- "with the Am-241 source, the package is marked with "RQ"
- "meeting the 173.424 criteria for labeling and marking requirements

For any shipment: Include in the package a current copy of the instrument Leak Test.

Include a list of emergency numbers in the package.

For Am-241, include the Special Form Certificate in the package.

Always ship in supplied plastic case, with the case secured against accidental opening. Always ship with the battery disconnected.

When shipping by air: Ship with the proper IATA marking (See IATA Dangerous Goods regulations Figure 10.7.8.A), UN 2911, and proper shipping name.

"RQ" marking and "dangerous goods declaration" are required.

When shipping by Ground: Ship under proper marking "UN2911"

"RQ" marking and shipping papers are required (Note: a dangerous goods declaration form can be used to meet the shipping paper requirement).

A "Dangerous Goods Declaration" can be obtained by the air carrier that you will be using. Instructions can also be obtained from the same source.

Carefully follow the directions given by the air carrier. Several typed copies will be required.

Shipping papers contain all of the same information as a Dangerous Goods Declaration, but do not have a specified format for that information.

At a minimum, a properly prepared shipping paper clearly identifies the hazardous substance by its proper shipping name, hazard class and division, UN identification number, packing group (if applicable), and total quantity. It also has consigner information, consignee information, and a shipper's declaration that the package is in compliance with the DOT regulations.

The elements of hazard warning information are communicated through shipping documents, packaging markings, and written emergency response information.

The DOT & FAA Hazardous Materials Regulations require the carrier to report all incidents involving hazardous materials.

An "incident" involves the unintended release of hazardous materials (Am-241), suspected radioactive contamination, if the general public is evacuated for an hour or more, or the flight pattern or routine of an aircraft is altered.

For any "incident", contact the Company Radiation Safety Officer or Responsible Party and the state radiation control program.

Any "incident" needs to reported to the:

Hazardous Materials Information Center

1-800-467-4922

Mon-Fri 9AM-5PM Eastern

Leak Tests (Niton XL3p Only)

The Niton XL3p Series analyzer contains a radioactive source that must be periodically leak tested. The purpose of leak testing is to verify the integrity of the source encapsulation. A leak test sample is obtained by wiping exterior surfaces of the device with moderate pressure using a cotton swab, filter paper, or whichever wiping media is supplied by the analysis laboratory. Leak test samples are then typically analyzed at a laboratory, although some device users have the equipment and licensed authority to perform this analysis

Unless specified otherwise by your local authority or radioactive material license, the gauge must be leak tested at intervals not to exceed 6 months. In the US, leak test samples may be acquired by any end-user, however the analysis of the sample must be performed by an organization licensed to do so. If you are using a vendor to perform the laboratory analysis of the leak test sample, they will send you a leak test kit which comes with complete instructions for performing the test. These vendors will also typically send you a reminder when it is time to perform the next leak test on your instrument. Please follow the test kit instructions carefully, and promptly mail the test samples to the laboratory. They will send you a leak test certificate soon after. Keep one copy of the leak test certificate with the device at all times (i.e., in the case) and another copy safely on file.

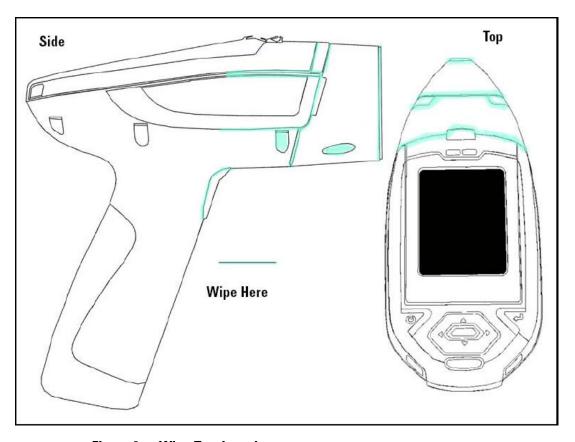


Figure 6. Wipe Test Locations

CAUTION REMOVE THE BATTERY WHILE PERFORMING A WIPE TEST TO BE SURE THAT THE SHUTTERS ARE NOT OPEN DURING THIS PROCEDURE!

Lost or Stolen Instrument

THIS PAGE CONTAINS EMERGENCY CONTACT INFORMATION THAT SHOULD BE AVAILABLE TO THE OPERATOR AT ALL TIMES.

If the Niton XL3t analyzer is lost or stolen, notify your Radiation Safety Officer (RSO) or the equivalent responsible individual at your company or institution immediately. Your company's RSO, as well as other important emergency contacts, are listed below. Your company RSO may need to notify the x-ray tube regulatory authority and the local police. It is also recommended that a notification is made to Thermo Fisher Scientific.

Damaged Instrument

Minor Damage

If the instrument is intact but there is indication of an unsafe condition such as a cracked case, a shutter mechanism failure, or the lights remain flashing after a measurement is terminated, follow these steps:

- 1. Stop using the instrument
- 2. Remove the battery. The x-ray tube can not produce radiation when the battery is disconnected. The instrument is now safe to handle.
- 3. Place the instrument securely in the holster.
- 4. Place the instrument in the carrying case that came with the instrument.
- 5. Notify your Radiation Safety Officer (RSO) or the equivalent responsible individual at your company or institution immediately.
- 6. You or your RSO should call Thermo Fisher Scientific at one of their contact numbers listed below for additional instructions and guidance.

Major Damage

If the instrument is severely damaged:

- 1. Perform the same steps as described above for minor damage. There will be no radiation hazard as long as the battery is removed from the instrument.
- 2. Place all components in a plastic bag and contact Thermo Fisher Scientific.

Emergency Response Information

Please Complete the Following Emergency Response Information and Keep with the Analyzer at All Times

at All Times
NITON ANALYZER EMERGENCY CONTACT INFORMATION
The Company RSO is:
RSO Telephone Number:
Regulatory Agency Emergency Number:
Local Fire Department:
Local or State Police Department:
Thermo Fisher Scientific's Niton Analyzer Contact Numbers
Main Number (USA): (800) 875-1578
Additional Radiation Emergency #'s: (978) 790-8269 or (617) 901-3125
Outside the USA - Local Niton Service Center:
Niton Analyzers Europe
Munich, Germany
Phone: +49 89 3681 380
Fax: +49 89 3681 3830
Email: niton.eur@thermofisher.com
Niton Analyzers Asia
Hong Kong
Phone: +852 2869-6669
Fax: +852 2869-6665
Email: niton.asia@thermofisher.com

Europe

Asia

Registration and Licensing

As a user of a Niton XL3 analyzer, you may be required to register or obtain a license with your local radiation control authority. In the US, if you intend to do work with your analyzer in states other than your own, you may be required to register there as well. See the <u>Safety and Compliance Web Hub</u> for much more information.

Regarding Safety Devices for the Open Beam Configuration:

In the US, you may be required to file for an exemption, "variance letter", with your state if there is a requirement for a safety device that would prevent entry of an extremity into the primary beam. If you need assistance with the exemption letter, you may contact the radiation safety group.

Registration and Licensing FAQ

Startup Procedure

Unpacking and Preparing Your Niton XRF Analyzer

WARNING The nose of the analyzer should not be touched during sample testing and calibration. If an ESD event occurs during measurement, the instrument may terminate the testing in progress and automatically reset to Log On screen. Any test data collected prior to reset will be lost and the testing may have to be repeated.



Figure 7. Populated Analyzer Case

- Inspect the shipping carton for signs of damage such as crushed or water damaged packaging. Immediately notify the shipping company and Thermo Fisher Scientific, in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460, if there is any visible damage to the shipping container or any of its contents.
- Open the packing carton. If your analyzer is not packed in its carrying case, please call Thermo Fisher Scientific immediately, in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460.
- Verify the contents of the shipping container against the enclosed packing list. If there are any discrepancies between the actual contents of the shipping container and the enclosed packing list, please notify Thermo Fisher Scientific immediately, in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460.
- Open the carrying case and visually inspect the analyzer for damage before removing it from the case. Contact the freight carrier and Thermo Fisher Scientific if you find any damage to the case or its contents.

• Save the shipping carton and all packing materials. Store them in a safe, dry area for reuse the next time that you ship the analyzer.



Figure 8. The Thermo Scientific Niton XL3 Analyzer Overview

Battery Installation and Charging

Installing or Replacing The Battery Pack

- 1. Slide back the catch on the bottom of your analyzer's pistol grip and drop the battery out into your hand.
- 2. Place the old battery aside and slide the new battery up into the cavity in the bottom of the pistol grip. The battery is keyed, and will only insert fully one way.



Figure 9. Rear and Side of Battery Pack Showing Key CAUTION Do not force the battery into the cavity!

3. Press in until the latch resets.

Recharging The Battery Pack

Fully recharging a battery pack takes approximately 2 hours.

- 1. Remove the battery pack from the analyzer.
- 2. Place the battery pack upside down into the charger. The battery pack is keyed, and will only fit into the charger fully one way. If your battery pack is resting on the back of the back of the charger rather than sliding all the way to the bottom, remove the battery pack, turn it around, and re-insert it into the charger.

CAUTION Do not force the battery into the charger!

3. The red light is on when the charger is plugged in. This is the power indicator light.

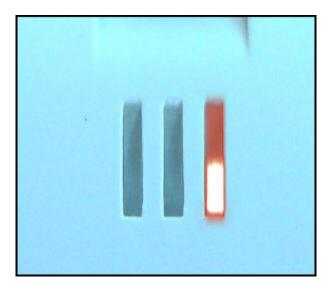


Figure 10. Power On

4. The yellow light indicates that the battery pack is currently being charged.

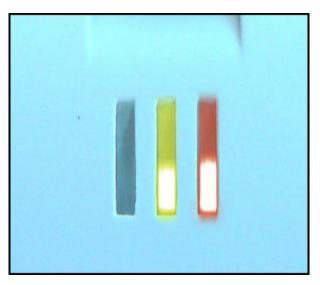


Figure 11. Charging

5. The green light indicates that the battery pack has finished charging and is ready for use.

2 Using Your Analyzer Battery Installation and Charging

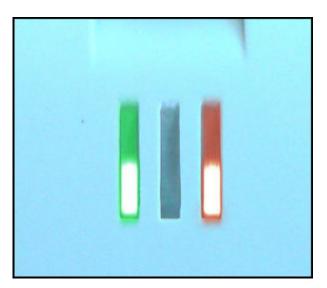


Figure 12. Charge Completed

Note If there is a fully seated battery pack in the charger and only the red light is on, there is a fault with the battery pack or charger.



Figure 13. Battery Pack in the ChargerCAUTION Do not store battery packs or charger in direct sunlight.CAUTION Do not let the battery pack recharge for excessive periods of time.

The Control Panel

The control panel is located on the analyzer's top housing, directly below the Touch Screen. The control panel consists of a 4 Way Touch Pad, an Interlock Button, and two Control Buttons, one on each side. Using either the control panel or the touch screen you may navigate through all of the analyzer's screens and menus. You can control the movement of the screen cursor by pressing the 4 Way Touch Pad in one of four directions to highlight each of the menu options. The Clear/Enter button to the right of the 4 Way Touch Pad is used to select highlighted menu options. The On/Off/Escape Button both controls the power to the analyzer and serves as an "escape" button. When the On/Off/Escape Button is pushed and immediately released, it functions as an "escape", and brings you back to the Main Menu from the current screen in the menu system.

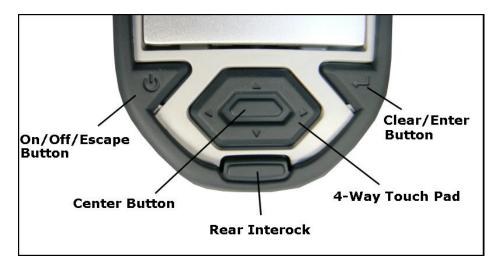


Figure 14. The Control Panel

To turn your analyzer on, push and hold the On/Off/Escape Button until the Touch Screen comes on. To turn off your analyzer, push the On/Off/Escape Button and hold it down until the Touch Screen shuts off.

CAUTION If for any reason your analyzer does not shut off normally, removing the battery will shut it down safely.

You also have the option of operating the analyzer, including navigating the menu system, by using the built in Touch Screen. To select a menu option, tap on the icon once. The touch screen icons have the same functionality as the 4 Way Touch Pad, the On/Off/Escape Button, and the Enter Button. This User's Guide will refer to the process of choosing a course of action by selecting an icon from a menu, either using the Touch Screen or using the control panel buttons, as "selecting."

Selecting the Return Icon works everywhere throughout the User Interface to bring you back to the previous menu from the current menu in the menu system. Use the On/off/escape button to return to the Main Menu.

The LCD Touch Screen

The LCD Touch Screen on your Niton XL3 Analyzer is designed to swing up and down to different angles for ease in viewing and interacting with your analyzer. The LCD Touch Screen is connected to your analyzer along the base of the screen, right above the Control panel. The screen is not designed to separate from the analyzer, but can be adjusted to any arbitrary angle between zero degrees - that is, flush with the analyzer - and 85 degrees, which is almost perpendicular. The LCD Touch Screen will stay at any given angle between these extremes until moved to a different angle. When in closed position, the screen is secured by a catch at the top center of the screen housing.



Figure 15. Niton XL3 Analyzer Showing LCD Screen Tilted.

To raise the LCD Touch Screen, disengage the catch at the top-center of the LCD Touch Screen housing and gently pull the screen towards you until it is at the best angle for your use.

2 Using Your Analyzer Startup Procedure

To close the LCD Touch Screen, gently push away from you along the top edge of the screen housing. The screen will swing down until the catch solidly engages with an audible click. **Note** The LCD Touch Screen cannot be removed from your Niton XL3 analyzer. Removing or attempting to remove the LCD Touch Screen will damage your analyzer and void your warranty.

Note Always close your LCD Touch Screen before storing or transporting your Niton XL3 analyzer.

Startup Procedure

To turn on the analyzer, depress the On/off/escape button on the control panel until the Touch Screen comes on.

On startup, the screen will show by a Start Screen which will automatically count down from 4 to 0 in increments of one second.

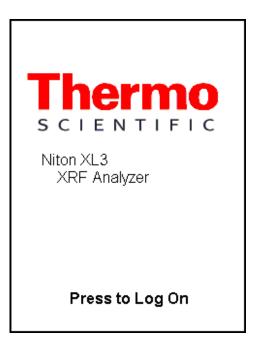


Figure 16. Logon Screen

When the startup is complete, the Start Screen will be replaced by the Logon Screen (see Figure 15). Tap anywhere on this screen to continue.

The Logon Screen will be replaced by a Warning Screen, see Figure 16, advising you that this analyzer produces radiation when the lights are flashing. You must acknowledge this warning by selecting the Yes button before logging on. Selecting the No button will return you to the Logon Screen.

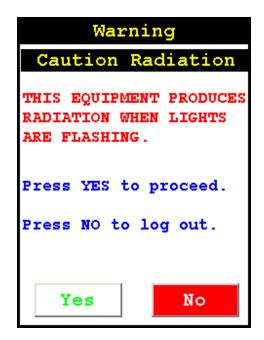


Figure 17. Warning Screen

After selecting the Yes button, the Virtual Numeric Keypad becomes available for you to log onto the analyzer.

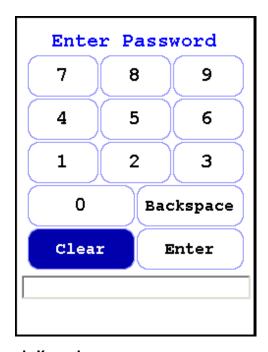


Figure 18. Virtual Numeric Keypad

2 Using Your Analyzer Performing a System Check

Select your 4 digit security code, followed by the Enter button. The default password is 1-2-3-4, followed by the Enter button. If you enter an incorrect number, you can use the Backspace button to backspace over it, or use the Clear button to clear the field. After you have completed the log on procedure, the word "USER" will appear on the bottom of the screen, then the Main Menu will appear. Note that security codes are editable. Please see Passwords and User Privileges for instructions on creating user-definable passwords.

Check the date/time. The time should be set correctly for accurate and verifiable record keeping (Setting the Date and Time).

Note Your analyzer can be stored and operated safely in temperatures from minus 5° C (23° F) to 50° C (122° F). You will not be able to take a measurement if the analyzer overheats. If it is hot to the touch, you should allow it to cool before testing.

Performing a System Check

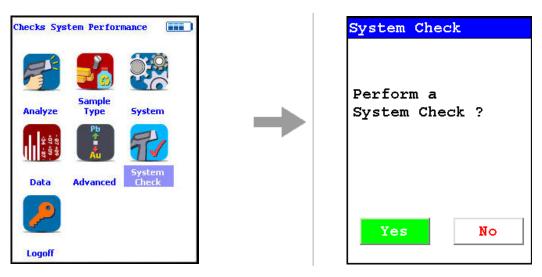


Figure 19. System Check Menu Path

Select the System Check Icon on the Main Menu to perform a system check. Thermo Scientific recommends that you perform a system check once every working day, as part of your normal startup procedure, after allowing a minute or so for the analyzer to warm up.

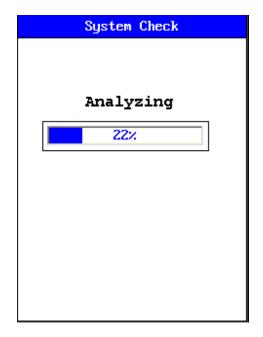


Figure 20. System Check in Progress

While performing the system check, your screen will show a progress bar indicating the progress of the check. When it is done, the screen will show a 100% completion as in Figure 20 below.

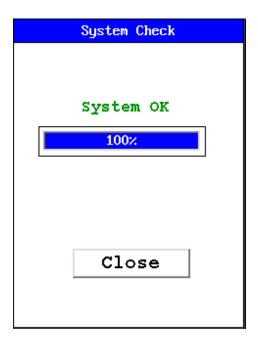


Figure 21. System Check Completed

2 Using Your Analyzer Performing a System Check

If you see any result other than "System OK", perform another system check. If the result is still not "System OK", please notify Thermo Scientific Service at 800-875-1578.

Calibrating the Touch Screen

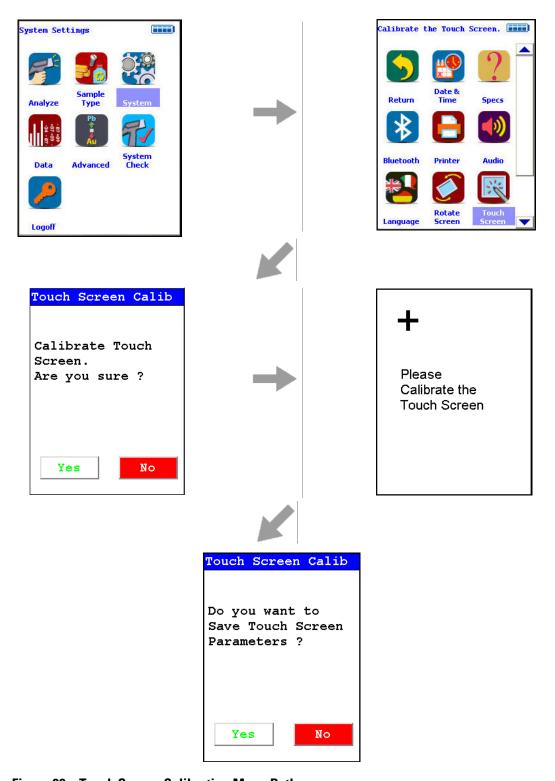


Figure 22. Touch Screen Calibration Menu Path

2 Using Your Analyzer Performing a System Check

Select the Calibrate Touch Screen icon to re-calibrate the analyzer's touch screen display. This procedure establishes the display boundaries for the touch screen interface.

- 1. Select the Touch Screen icon.
- 2. The display will show a message asking you to confirm whether or not you want to calibrate your Touch Screen. Select the Yes button.
- 3. The display will show the message: "Calibrate Touch Screen". There will be a small cross in the upper left-hand corner of the display.
- 4. Tap on this cross with the stylus, and the cross will disappear and reappear in the upper right-hand corner of the screen.
- 5. Tap on the cross again, and it will reappear in the lower right-hand corner of the screen.
- 6. Tap on the cross again and it will reappear in the lower left-hand corner of the screen.
- 7. Tap on the cross once more, and you will be presented with a Confirmation Screen.
- 8. Select the Yes Button to confirm that the parameters are good. Select the No Button to start the process again.
- 9. Once you have confirmed the parameters, the System Menu will be displayed. The screen is now calibrated.



Figure 23. The System Menu

Calibrating the Touch Screen Without Using the Touch Screen

If the touch screen becomes misaligned, you may need to use the buttons below the screen to complete this process. There are 2 single buttons and a 4 way switch located to the rear of the display screen. The button at the left is the On/Off/Escape button. The button to the right is the Enter button and the center keypad is a 4 Way Touch Pad.

The 4 Way Touch Pad has 4 positions, Up, Down, Left and Right. The Interlock Button is not used in this procedure.

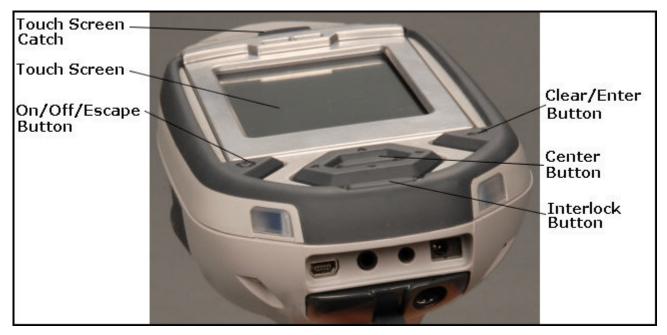


Figure 24. The Control Buttons for the Niton XL3

- 1. Please, turn on your XRF analyzer using the On/Off button. **Note** From this point please DO NOT touch the touch screen.
- 2. Press the Enter button. You are now at the Radiation Warning screen.
- 3. Using the 4 way touch pad on the on the cover of the instrument, move the cursor around the screen by pressing the appropriate up, down, left, or right button. Please move the cursor such that the "Yes" option is highlighted in green.
- 4. Press the Enter button. You are now at the Enter Password Screen.
- 5. Move the cursor on the Virtual Numeric Keypad using the 4-way Touch Pad to the appropriate first number in your password. Press the Enter button on the right it has the arrow/enter key symbol on it. The first number of your password should appear in the lower left of the screen.
- 6. Repeat step 5 until you have entered the entire password. Then move the cursor to the Enter Key on the Virtual Numeric Keypad and press the Enter button to enter it.

2 Using Your Analyzer Performing a System Check

- 7. You will now be at the Main Menu.
- 8. Again using the 4 Way Touch Pad, move the cursor to highlight the System icon, and press the Enter button to select it.
- 9. You will now be at the System Menu.
- 10. From the System Menu screen, move the cursor to highlight the Touch Screen icon and press the Enter button.
- 11. Now move the cursor to highlight the Yes Button and press the Enter button.
- 12. You are now at the Touch Screen Calibration screen.

Note You must now use the touch screen for the balance of this procedure

- 13. In the upper left hand corner you will see a crosshair like this: "+". Using the stylus or a pen, tap the center of the "+".
- 14. Repeat this for each "+" sign that appears, there should be one for each of the 4 corners.
- 15. Select the Yes Button to confirm that the parameters are good. Select the No Button to start the process again.
- 16. Once you have confirmed the parameters, the System Menu will be displayed. The screen is now calibrated.

Your touch screen should work properly after this and you may use normally it from this point forward. If it does not, please repeat the process.

The Data Ports



Figure 25. The Data Ports

USB Port

The USB Port is a communications and control port, for uploading and downloading data, configuration files, and software to the analyzer.

Remote Trigger Port

The Remote Trigger Port controls the analyzer's trigger function, for use with accessories and test stands.

Serial Port

The Serial Port is a communications and control port, for uploading and downloading data, configuration files, and software to the analyzer.

Power Port

The Power Port is used to run the analyzer under external power.

Using the Navigation (NAV) Menu

The NAV Menu enables you to move between various menus and screens directly, without going through the intervening screens. Select a destination from the drop down menu and you will be brought directly to that menu or screen.

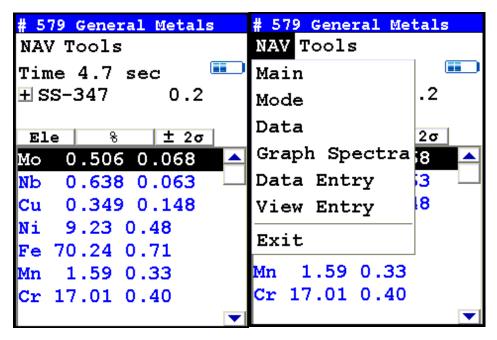


Figure 26. Accessing the NAV Menu

Using the Tools Menu

The Tools Menu enables you to perform common data-related tasks such as printing and averaging readings. Select a task from the menu to initiate that task. The options available can vary depending on the selected mode.

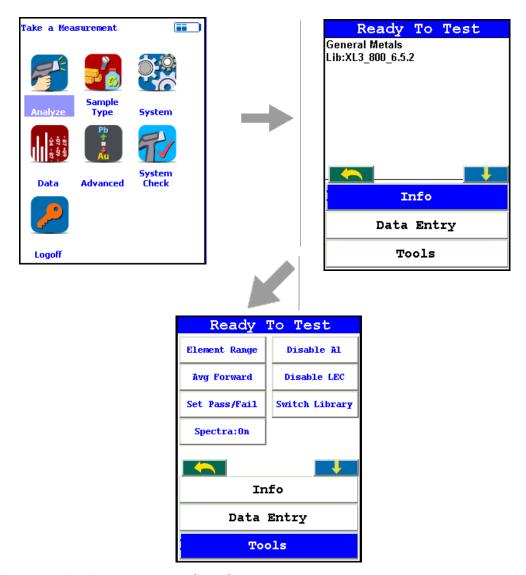


Figure 27. The Tools Menu Path (Main)

The Tools Menu can be accessed in two ways, and the options are different depending on the way you access it. The main Tools Menu is accessed by selecting the Analyze Icon from the Main Menu, then selecting the Tools button from the slide down window on the Ready to Analyze screen.

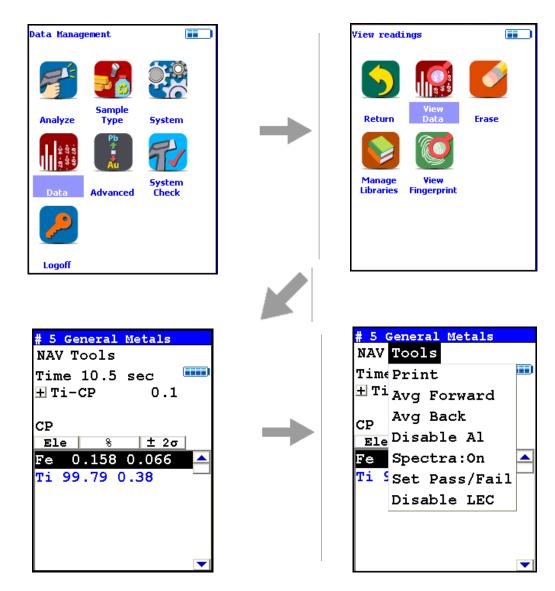


Figure 28. The Tools Menu Path (Alt)

The alternate Tools Menu can be accessed by selecting the Data Icon from the Main Menu, selecting the View Data Icon from the Data Menu, then selecting the drop down Tools Menu from the View Data screen. It can also be accessed from the Analysis Screen, after the reading is saved.

Toggling Buttons and Options

Most of the buttons and options on the Tools Menu are toggles. These buttons and options on the Tools Menu change to their opposites when selected. To stop averaging, for example, select Stop Avg Fwd or Stop Avg Back from the Tools Menu as appropriate.

Tools Menu Options (Mode Dependent)

Averaging Results

You will find the two types of result averaging here: Avg Forward

Spectra: On/Spectra: Off

You will find how to use Live Spectra here: Spectrum:On/Spectrum:Off

Alloy Pass-Fail

You will find how to use Alloy Pass/Fail here: Set Pass/Faill

Editing and Switching Alloy Libraries

You will find how to use Alloy Libraries here: Switch Library (Main)

Enable/Disable Al

You will find how to use the Enable/Diable Al option here: Enable/Disable Al

Light Element Correction

You will find how to use Light Element Correction (LEC) here: Enable LEC/Disable LEC

Coatings Method

You will find how to use Coatings Method here: Coatings Method

Thickness Correction

You will find how to use Thickness Correction here: Thickness Correction

Enable Paint

You will find how to use Enable Paint here: Enable/Disable Paint

Action Level

You will find how to use Action Level here: Action Level

Print Data

You will find how to use Print Data here: Print Data

2 Using Your Analyzer Using the Tools Menu

Element Ranges

You will find how to use Element Ranges here: Adjusting the Element Range

Toggle He

You will find how to use Toggle He here: Enable/Disable Al

Toggle Spot

You will find how to use Small Spot here: Calibrating the Spot

Setting the Date and Time

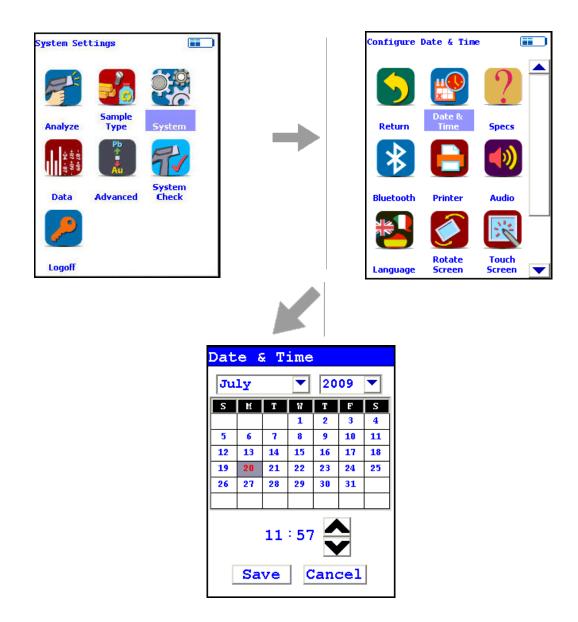


Figure 29. The Date and Time Menu Path

From the System Menu, select the Date & Time icon to set the date and time as needed for different time zones, daylight savings time, or any other reason. The date and time are factory preset prior to shipping. The clock is a 24 hour clock, so add 12 to PM hours - i.e. 1:13 PM would be 13:13.

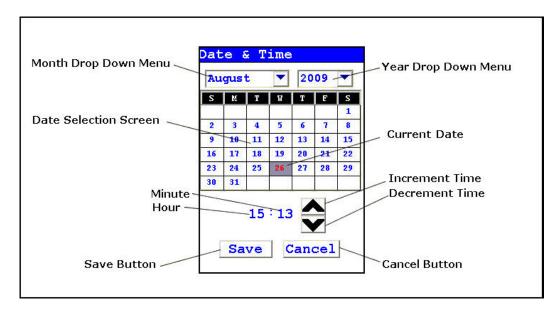


Figure 30. Setting the Date & Time

When the Date & Time icon is selected, the Date & Time Screen comes up on your analyzer's LCD Screen. You may change the Month, Year, Date, Hour, and Minute on your analyzer.

Changing the Month

To change the month, select the downward pointing triangle button next to the month displayed. A drop down menu will appear, listing the months of the year in order of appearance.

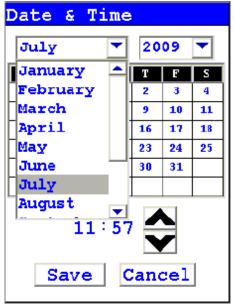


Figure 31. Month Drop Down Menu

Select the month you want from the drop down menu, using the vertical slider button to display hidden months. The display will change to show the month you selected.

Changing the Year

To change the year, select the downward pointing triangle button next to the year displayed. A drop down menu will appear, listing the years in order of appearance.

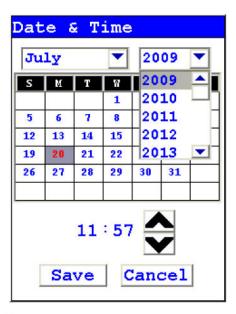


Figure 32. Changing the Year

Select the year you want from the drop down menu, using the vertical slider button to display hidden years. The display will change to show the year you selected.

Changing the Date

To change the date, select the date you want from the Date Selection Screen. The date you selected will be highlighted in red, while the old date will be shown in red numbers.



Figure 33. Selecting the Date

Changing the Hour and Minute

To change the hour, select the hour numbers. The hour numbers will be highlighted in gray. Then select the Upwards Pointing Chevron Button to increment (increase) the hour, or the Downward Pointing Chevron Button to decrement (decrease) the hour.



Figure 34. Changing the Hour

To change the minute, select the minute numbers. The minute numbers will be highlighted in gray. Then select the Upwards Pointing Chevron Button to increment (increase) the minute, or the Downward Pointing Chevron Button to decrement (decrease) the minute.

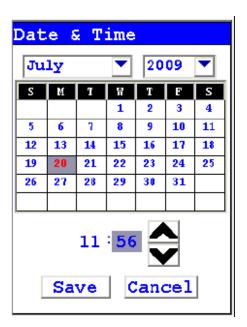


Figure 35. Changing the Minute

Saving Your Changes

To save your changes, select the "Save" screen Button. The display will return to the previous screen and the Date and Time will be saved.

Exiting Without Saving

To exit the screen without saving changes, select the "Cancel" Screen Button. The display will return to the previous screen and the Date and Time will not be saved.

Data Entry

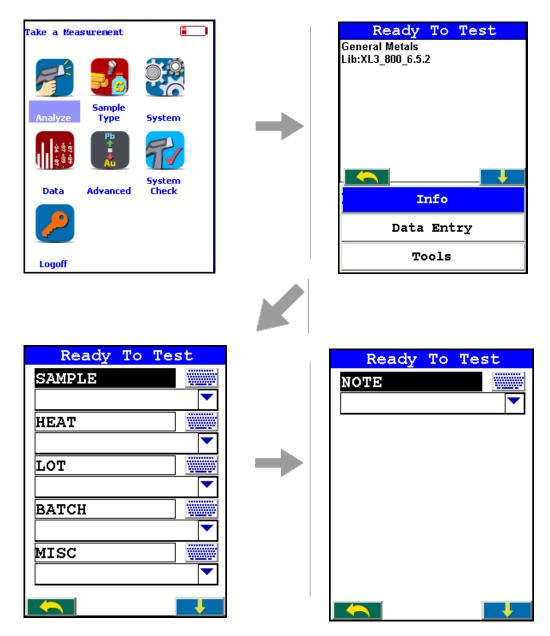


Figure 36. The Data Entry Menu Path

The Data Entry Menu can be accessed from the Ready to Test Screen, then selecting the Data Entry button from the slide down window on the Ready to Test screen. The Data Entry Menu is composed of several data fields with associated buttons. How many and exactly which fields are displayed depends on the Mode the analyzer is currently in.

Entering data into these fields enables you to keep track of details about the reading, for use in later analysis and enhancing the defensibility of the readings.

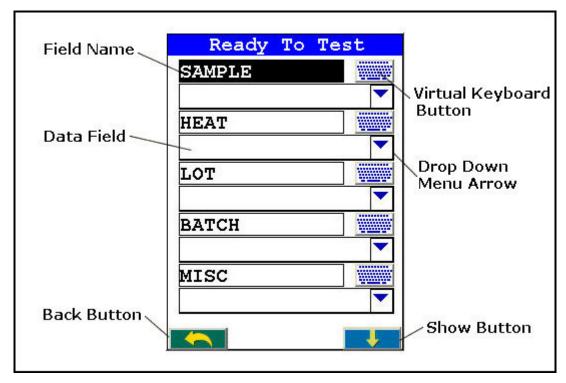


Figure 37. The Data Entry Menu Buttons

The Data Entry Menu Buttons

The Data Entry Menu Buttons each enable you to enter data into the fields in different ways.

Field Name

The Field Name is the designation of the type of data to be entered into the field. Selecting the Field Name enables you to enter data into that field.

Virtual Keyboard Button

Selecting the Virtual Keyboard Button will bring up the Virtual Keyboard, which you can use to type data into the field.

Drop Down Menu Arrow

Selecting the Drop Down Menu Arrow enables you to select data from a list you have previously entered into the analyzer.

Data Field

Your data will appear in the Data Field.

Back Button

Selecting the Back Button will return you to the Main Menu.

Show Button

Selecting the Show Button will bring the slide down window up into view for access to the Tools Button or Info Button.

The Results Screen

Your analyzer will display the Results Screen throughout the duration of each reading. The Results Screen is updated regularly throughout the reading. When the reading is complete, a final screen update will appear, and your analyzer will display the final results of the measurement which has just been completed.

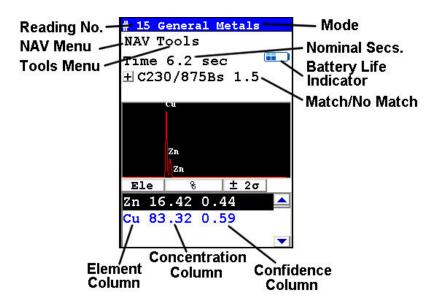


Figure 38. The Results Screen

The Results Screen displays the following information:

The Reading Number shows a number sequentially assigned by your analyzer in order to uniquely identify each reading. The reading number automatically increments with each successive reading.

The Nominal Seconds Test Duration line shows the number of nominal (source) seconds elapsing since the initiation of the reading. Nominal seconds are instrument time designed to compensate for the electronic dead-time that may occur when your analyzer is taking a measurement, and therefore tend to be a bit slower than real time.

The Mode displays the test mode in use during the measurement.

The Match/No Match line indicates whether your analyzer has found a matching alloy in it's library for the sample you have measured, and displays the name(s) of any matching alloys and the Match Number (chi squared deviation).

The Element (left) column shows the elements that have been detected in the sample.

The Concentration Level (central) column shows the concentration levels of the corresponding elements in percentages.

The Confidence (right) column displays the 2 sigma (95%) confidence interval for the corresponding elements.

If there are too many elements detected to fit onto a single screen, you can see the balance of the elements and their results (as in figure 3-16) by pressing the down arrow of the 4-way touch pad. To see previous results, use the left arrow of the 4-way touch pad. To go forward to later readings, use the right arrow of the 4-way touch pad.

Match Number

The Match Number is a rating of confidence in the identification of the alloy. The Match number ranges from 0.0 to 10.0, with 0.0 being a perfect match. Any number greater than 4.0 gives a result of No Match by default, although you can change this match threshold. Any number less than 2.0 is considered a Good Match, and can usually be brought closer to 0.0 with longer testing times. Numbers between 2.0 and 3.0 can be considered Probable Matches, and numbers between 3.0 and 4.0 as Possible Matches, often having one or more elements out of spec.

2 Using Your Analyzer The Results Screen

How to Analyze

This section contains all the Standard Operating Procedures (SOPs) we have developed to serve as frameworks for you to build your own SOPs, tailored to your unique requirements. You can also use these SOPs as is if they suit your needs. We have also included the US EPA's Method 6200 for soil testing. Electronics Metals and Plastics analysis use the same SOP as that established for Consumer Goods analysis, so the links for those analysis types will redirect there. This section also covers data management on your analyzer.

3 How to Analyze

Metal Sample Prep

Up until recently, sample preparation was not a big worry for XRF metals analysis, as the LOD of the analyzer was seldom low enough for any but the most heavy contamination to be intrusive; but recent developments such as He-purged analysis and the GOLDD technology have brought analysis to a level where even light surface contamination can skew an analysis.

You should always prepare your samples before analysis, especially when using GOLDD or He-purged analysis, as these analyzers will see even trace amounts of contaminants. Oils from fingerprints and other body contact, lint, oxidation materials, and abrasive materials used in cleaning can all skew readings if not removed. Sample preparation is simple and not time consuming, and usually well worth the effort.

The following is a list of problems that need correction before testing:

- Oxidation or Rust may produce an increase or decrease in one or more element test values unless we remove the rust or oxidation and expose the raw metal.
- Paint may contain several elements which need to be tested at lower levels within metal alloys (Ti & Zn in white paint, Fe in red paint, Cr in green paint).
- Oil, grease or lubricates may contain high levels of the following elements: lithium, aluminum, barium, strontium, molybdenum or calcium.
- Plated surfaces may have high levels of the following elements: zinc, chromium, nickel, or copper.

CAUTION Anything on the metal surface will become part of your test results!

Sample Analysis Preparation

You need to clear the surface of your samples of any paint, plating, or any oxidation such as rust or verdigris before analysis. In order to accomplish this, you need the following:

- Isopropyl alcohol not rubbing alcohol, which contains oils.
- Lint-free paper.
- Diamond paper P/N 179-1202- cut into 1 inch/2.5 cm squares. Never re-use this paper, as it may transfer contaminants to the surface of the sample from previous cleanings. Depending on the state of the sample, several squares may be needed per sample.
- A Sample Grinder for removing deeper surface contamination. Choice of grinding wheel media also may be important, depending on what you are testing for. Never re-use grinding media, as contaminants can be transferred from sample to sample on the media itself.

4 Metal Sample Prep

For light contamination on hard metal reference standards, remove the oxidation by scrubbing the dry sample lightly with the diamond paper square, using the fingers to maintain pressure. If the diamond paper begins to load up with material, discard it and use a fresh square. When the oxidation is removed, wipe the sample with lint-free paper soaked with isopropyl alcohol to remove any oils or dust. Let the sample dry before attempting analysis.

For soft metal reference standards, wipe the sample with lint-free paper soaked with isopropyl alcohol, then remove the oxidation by scrubbing the wet sample lightly with the diamond paper square, using the fingers to maintain pressure. If the diamond paper begins to load up with material, discard it and use a fresh square. When the oxidation is removed, wipe the sample again with lint-free paper soaked with isopropyl alcohol to remove any oils or dust. Let the sample dry before attempting analysis.

Oils, lint and dust can be removed by wiping the sample with lint-free paper soaked with isopropyl alcohol. Let the sample dry before attempting analysis.

Surface Oxidation

With the exception of a limited number of metal types, most metal alloys form an oxide covering on the surface when exposed to oxygen or air. This oxide covering is visible in carbon and low alloy steel as a red colored substance called rust. Other metal alloys form oxidation which is not always visible, but that does not mean that it is not present. If the test results for low concentration elements are higher or lower than expected, remove the oxide coating by grinding and retest. Follow proper safety procedures when changing discs or grinding materials.

During a recent case study the effects of sample preparation became apparent. A customer asked for low detection limits of nickel, chromium and copper in carbon steel pipe. The reported chemistry of the purchased material is listed on the first line in the chart below. The test results of a hand held XL3t 900S GOLDD instrument appears in the second line of the chart. The results from a test on the unground surface appear in the bottom line of the chart. Note the values for nickel and copper in this carbon steel alloy in the chart below. The oxidation on the surface of this pipe was not visibly egregious. We need to always be wary of the presence of even low levels of oxidation and their possible effects on analytic accuracy.

Comparative test results with and without grinding

Sample	% Mn	% Ni	% Cr	% Mo	% Cu
Reported Chemistry	0.650	0.090	0.070	0.030	0.040
Test Results with Ground Surface	0.67	0.089	0.070	0.033	0.039
Test Results with Unground Surface	0.61	0.178	0.081	0.033	0.514

Painted Surfaces

Paint is a mixture of several items that are combined into a liquid which is applied to the surface of materials such as metal. Once applied this liquid dries with time and adheres to the surface of metal. Paint is used to protect or decorate the metal item. Paint can also be used to identify or mark the metal during the manufacturing process.

Components of paint are divided into classifications of pigments, binders, solvents, additives and fillers. The inorganic elements in pigments will contribute to increases in displayed values for those elements if paint on the metal surface is not removed prior to testing. Be especially careful of the presence of heavy elements, which can also act to shield x-rays from lighter elements in the metal sample.

The following is a list of some of the most common components of paint:

White Paint

- Antimony (Sb)
- Lead (Pb)
- Titanium (Ti)
- Zinc (Zn)
- Cobalt (Co)

Red Paint

- Iron (Fe)
- Lead (Pb)
- Green Paint
- Chromium (Cr)

An experiment was conducted to determine the effect and severity of surface problems on XRF results. Results from analyses of a 1541 alloy steel sample are shown below, before and after surface grinding. The sample had painted markings, of light to medium thickness, on the surface, as well as light rust. Note the change in titanium, zinc and cobalt levels after surface grinding.

Prepped and unprepped painted metal analysis

Sample	Mn	Ni	Cr	Mo	Ti	Zn	Co
Ground Surface	1.49	0.04	0.03	0.004	0.011	0.0001	0.03
Unground Surface	1.34	0.01	0.04	0.011	2.507	1.751	0.21

Oil, Grease & Cutting Oils

Oil and grease contain a number of elements combined into a viscous substance and applied to moving parts in order to reduce friction. Grease coatings can remain on component surfaces after it has been removed from service. Grease can also be applied to a metal's surface by accidental contact with other materials coated in heavy grease. Metals can also be coated in oil as a result of cutting and machining processes in manufacturing.

Grease and oil may contain the following elements:

- Aluminum (Al)
- Zinc (Zn)
- Molybdenum (Mo)
- Sodium (Na)
- Calcium (Ca)

An experiment was performed to show how grease on metal surfaces affects XRF results. A carbon steel sample was cleaned and ground as a control surface for the experiment. XRF tests were performed on the control surface, and again after light and heavier layers of automotive wheel bearing grease were applied to the surface of the steel sample. Results are shown below. Note the elevated levels of molybdenum, cobalt and zinc from the grease.

Clean and greased sample metal analysis

Sample	Mn	Ni	Cr	Mo	Cu	Co	Zn
Clean Surface	1.18	0.001	0.041	0.004	0.001	0.001	0.019
Light Grease	1.07	0.001	0.001	0.067	0.033	0.322	0.416
Heavy Grease	0.96	0.001	0.001	0.500	0.062	1.760	3.430

If a sample's surface contains lubricants or cutting oil, use a solvent and a clean towel or rag to remove them before analysis. You may then need to grind the surface to insure good results. Clean first, grind second, test last.

Remember to follow safe techniques for handling and disposing of solvents and cleaning rags

Anodized, Plated and Galvanized Surfaces

Anodizing is the process of polarizing the metal surface into a passive state which protects it against corrosion. This process is most often applied to aluminum alloys.

Galvanized steel is one of the most common of the coated surfaces. In this process, steel is passed through a molten bath of a zinc alloy. Zinc reacts with the steel metal to form a bonding layer on the steel surface. The zinc layer does not separate from the steel and forms a protective layer that protects the steel from oxidation.

Galvanized layers are relatively thick compared to other plating elements and methods. When grinding to remove the zinc coating, you will find increased zinc values even when you can see the steel surface. Grind a little further and zinc values will disappear. Zinc clings to the surface of the sanding disc, so you will need to frequently change discs.

Electroplating is another common practice of applying a coating which not only protects the surface from oxidation, but also improves the base material's wear resistance, lubricity and improves the overall aesthetics of the product. The electroplated coating is generally thinner and more evenly applied than galvanizing. Electroplating has a wide range of elements and in some situations there may be two or more different coatings on the same part.

The following is a partial list of elements that are used to plate the surface of base metals:

Ni, Cr, Cadmium (Cd), Tin (Sn), Zn, Al

Cordless Right Angle Drill

This style of drill is recommended for most surface preparation in the field because it gives the operator the greatest amount of control, and thus safety, when grinding samples. When moving a sanding disc on a conventional drill over a sample, forces tend to produce movement the operator may find difficult to control. Control and stability are important in grinding from effectiveness and safety perspectives.

A cordless right angle drill similar to the one pictured below is recommended for light to medium surface removal. For materials with heavy oxidation such as carbon and low alloy steel, an angle grinder, explained in the next section, is recommended. A kit with the drill, batteries and charging units, can be purchased from ThermoFisher, or companies such as DeWalt, Hitachi, Makita, Milwaukee or Ryobi.



Figure 39. Example of Right Angle Drill

A disc holder is needed with the drill to hold the sanding disc. (In the US, we recommend a 3.0 inch disc holder. It has a 0.25 inch shank to insert into the chuck of the drill.) If sanding discs are ordered from a local supplier, attention should be paid to the method of attaching the sanding disc to the disc holder. There are three types of connections: metal snap-on, plastic twist and plastic snap-on.

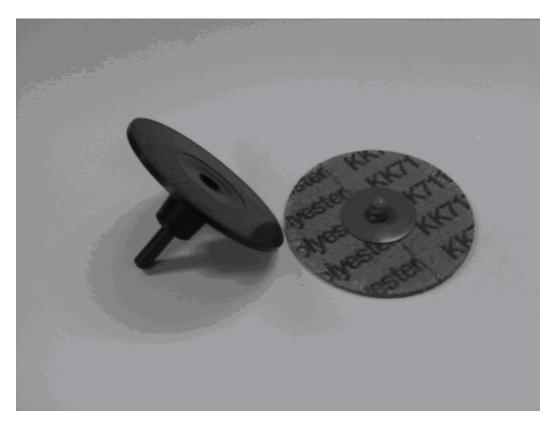


Figure 40. Sanding Disc

Before attaching the grinder and sanding disc as pictured below, first remove the battery to disable the grinder. Then insert the shaft of the disc holder into the drill and securely tighten the chuck. Next, attach the appropriate sanding disc. The method of attachment will vary depending upon the type of fastener on the sanding disc (snap-on or twist connectors). Reinstall the battery and prepare for use.

4 Metal Sample Prep





Figure 41. Attaching the Sanding Disc 1

Figure 1-3. Attaching the Sanding Disc 2

Cordless Angle Grinder

A cordless angle grinder similar to the one pictured below will successfully remove medium to heavy oxidation or paint. This grinder (which uses a 4.5 inch sanding disc with a rubber backup pad) which can be purchased from ThermoFisher or industrial tool manufactures like DeWalt, Makita or Milwaukee.

4 Metal Sample Prep



Figure 42. Cordless Angle Grinder Kit

A grinder kit typically contains the grinder, a battery, and charging unit. If the kit contains a grinding stone wheel, remove and dispose of it. Grinding stones are not to be used for XRF sample preparation. A rubber backup pad and a retaining nut are needed to use with sanding discs. (See picture below).



Figure 43. Rubber Backing Pad and Nut

In the US, sanding discs are 4.5 inch diameter and can be purchased in various grit sizes of 36 to 120. The surface abrasive can be one of the following materials: aluminum oxide, silicon carbide or zirconia alumina. The selection of sanding discs is covered in the next section.



Figure 44. Assembling the Grinder

Remove the battery before assembling the grinder, backup pad and sanding disc. Start by installing the backup pad onto the drive shaft of the grinder, or, with some backup pads. You will need to screw it onto the threaded shaft.

Next place the sanding disc over the drive shaft onto the backup pad. Hold the locking button on the reverse side of the grinder while tightening the retaining nut into the hole of the sanding disc.

Once the backup pad, sanding disc and locking nut are secured, reinstall the battery. The grinder is now ready for use.

Sanding Discs

It has been well tested and determined that samples can be easily contaminated by the abrasive material contained in and on a sanding disc. An example would be the increase in aluminum content of carbon steel after grinding the sample with a new aluminum oxide sanding disc. Aluminum from the aluminum oxide disc imbeds itself in the surface of the steel sample and an XRF would show an unusually high aluminum concentration.

Aluminum oxide is the most common abrasive surface used today. For most applications it will be safe to use aluminum oxide discs. But if test results for aluminum in any metal alloy are significantly higher than expected, switch to another type of abrasive disc. Also, when grinding aluminum, aluminum oxide discs tend to trap aluminum from the metal surface into the disc surface. Once this happens, the disc looses its efficiency and cross contaminates the next sample.

Silicon Carbide

Silicon carbide discs are a good alternative for aluminum oxide and the cost of a disc is only slightly higher than aluminum oxide. This adhesive type is best for grinding aluminum, copper and other soft metals.

Zirconia Alumina

Zirconia alumina discs are more expensive than aluminum oxide or silicon carbide but they last much longer and so may be the best investment. Few metal alloys have low additive levels of zirconium, so it is one of the safest abrasive types for general use.

One exception is the Aluminum alloy Al 7050 which is a near twin to alloy Al 7075 except for the ~0.1% Zr in 7050. Therefore, if 7075 is ground with Zr grinding paper it may be erroneously identified as Al 7050 due to the Zr transferred from the grinding disk to the surface of the Al 7075. s

Diamond Sanding Paper

Do not use diamond sanding paper for surface preparation in the field. Even after extensive and aggressive sanding with diamond paper, a metal surface will not be prepared properly. Diamond sanding paper is only recommended for removal of very light oxide coatings on flat surfaces such as analytical reference standards.

- Nickel, cobalt, and steel alloys should be ground using 36, 40, 50 or 60 grit discs. The selection of a grit size of 100 or larger will produce an acceptable metal surface but will result in very short disc life. Even a slightly used 100 grit disc will stop cutting and will start to just smear a sample's surface. Although a 50 grit size will produce a rough surface, it will have a much longer useful life.
- Aluminum, copper alloys, and other softer metals should be ground using 60 or 80 grit discs.
- Grinding stones are not recommended because they will absorb surface material and transfer them onto the next surface ground.

Safety Rules

When using a grinder, follow these safety rules:

4 Metal Sample Prep

- When changing sanding discs, always remove the grinder battery to prevent accidental activation of the grinder.
- Allow the grinder to stop spinning before placing it on a flat surface.

Replace any damaged or torn sanding discs immediately.

- Always wear impact eye protection to prevent eye damage from flying debris.
- Place small samples or standards in a clamping device when grinding to prevent accidental contact between the spinning disc and your hand.
- Use proper techniques and safety precautions when grinding beryllium, beryllium copper, lead, or titanium alloys.
- Always follow the safety instructions outlined by the grinder's manufacture as mentioned in the instruction manual.

Metals Analysis

To analyze metal samples, from the main menu select sample type, and then click on the Metals icon. Once in the Metals Selection Screen you have between 1 and 4 metal modes to select (depending on how your instrument is calibrated).



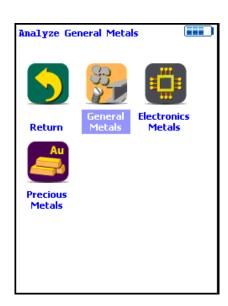


Figure 45. The Metals Analysis Menu Path (Main)

Select General Metals for most typical alloy analysis, such as iron (Fe), nickel (Ni), cobalt (Co), chromium (Cr), aluminum (Al), titanium (Ti), copper (Cu) and zinc (Zn) base metals. This is the ideal mode for any general metals analysis, such as PMI, incoming / outgoing inspections, QA / QC, and scrap metal sorting. This mode includes most common alloying constituents.

General Metals Analysis Standard Operating Procedure

PMI Analysis Standard Operating Procedure

FAC Analysis Standard Operating Procedure

Select **Electronics Metals** for analysis of metal samples typically found in electronic goods (such as printed circuit boards PCBs, wires and other manufactured components). This mode includes many common alloying constituents plus some elements used for bonding and soldering (such as Au and In).

Consumer Goods Analysisniton Standard Operating Procedure

Select **Precious Metals** for the analysis of precious alloys such as those found in jewelry, gold and silver coins, and archaeological and historical samples.

Select **Metal Coatings** for the determination of the thickness of 1 to 3 layers (coatings) on a metal substrate. For example, this mode may be used for the analysis of the thickness of a coated surface to ensure the material is plated consistently, or to determine that a conversion coating is of sufficient thickness. This mode does not determine the chemical composition of the sample.

Coatings Analysis Standard Operating Procedure

Element Ranges and Lists

From the Element Range Screen, select the Element List Button to display the Element List for the Range you want to use. This list shows the elements that the Range is best designed to detect. See Adjusting the Element Range for details.

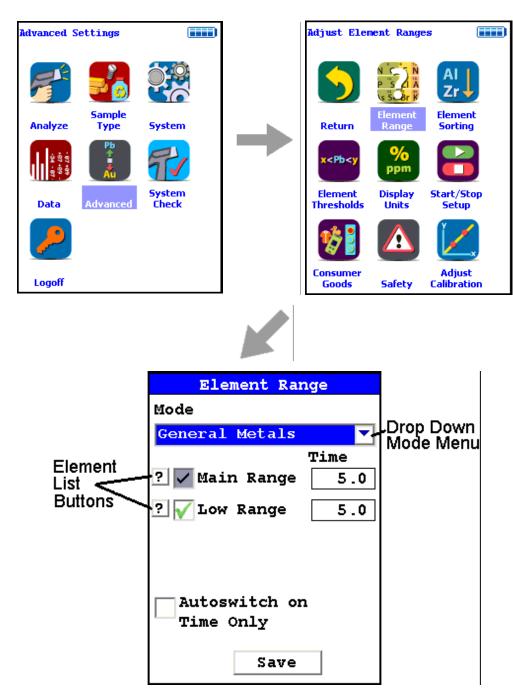


Figure 46. The Element Range Menu Path

General Metals Analysis

Analyzing General Metal (Alloy) Samples Using the Thermo Scientific Niton XL3 800 Series X-Ray Fluorescence (XRF) Analyzer – Version 7.0 and Higher

Standard Operating Procedure

October 2009

Note Each user should read the Thermo Scientific Niton XL3 User's Guide carefully before initiating measurements with the system. Users are strongly urged to attend the Thermo Scientific Niton XRF Analyzer Radiation Safety and Operations Training courses offered regularly, or the web-based trainings. For more information, visit www.thermo.com/niton.

PREPARATORY TASKS

- 1. Attach a charged battery to the analyzer and turn it on. Follow the screen instructions and "Log On" as the operator using either the default password or a custom one as designated by the user in an NDU file. See Passwords and User Privileges for details.
- 2. Wait five (5) minutes before using the analyzer, allowing the instrument electronics to stabilize.
- 3. Verify that the date is set properly for data tracking purposes.

From the Main Menu, select the System icon, then the Specs icon. The date will be displayed for verification. If the date is incorrect, correct it prior to proceeding. This can be done by "Closing" out of the Specs screen and selecting the Date & Time icon. Detailed information on this procedure is available in Setting the Date and Time.

- 4. (Optional) Connect the analyzer to a computer via the included serial cable, USB cable, or Bluetooth™ wireless module. (Consult Connecting the XRF Analyzer to Your PC for details, if necessary.)
- 5. During analysis and detector calibrations, it is important to ensure that the analyzer is not exposed to strong electromagnetic fields, including those produced by computer monitors, hard drives, cellular telephones, walkie talkies, etc. Keep a minimum two (2) feet (0.7 meters) distance between the analyzer and electronic devices.
- 6. From the Main Menu, select System Check icon then the Yes button. (Figure 1.)
- 6.1. System Check calibrates the detector and verifies it is operating to specifications. After starting the process, no further user interaction is required during this operation. When the instrument is finished performing the check, the unit will show either "System OK" or one of the failure errors.

6.2. If the unit shows a failure error, then perform a second System Check by clicking Recheck. If the unit still does not show a "System OK," please contact Thermo Scientific Niton Analyzers toll-free in the USA at (800) 875-1578, +1 978 670-7460, niton@thermofisher.com, or contact your local Niton Analyzers representative for assistance.

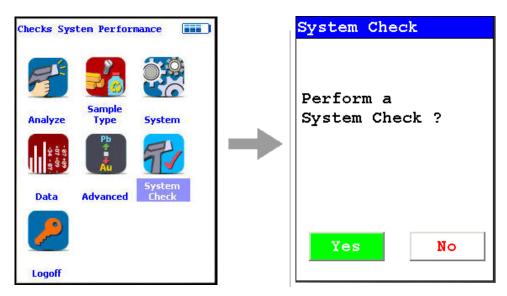


Figure 47. System Check Menu Path

7. Verify and/or change Range time.

Thermo Scientific Niton XL3 analyzers are equipped with excitation filters that optimize the analyzers' sensitivity for various elements. The "Main Range" filter provides optimum sensitivity for the elements manganese (Mn) through bismuth (Bi). The "Low Range" filter is used to optimize the sensitivity for the elements from titanium (Ti) through chromium (Cr). Note that the main range filter can be used to analyze Ti, V and Cr, but the sensitivity is not as good as when using the low filter. The "Light Range" filter is available only with He-purged and GOLDD technology analyzers, and is typically used in light element analysis. The amount of time that the analyzer spends in each filter position is user definable. Please note that the analyzer will continue alternating excitation filters until the user selectable maximum analysis time is reached or the operator terminates the measurement. To verify or change the length of time each filter will be active, do the following:

7.1 From the Main Menu select the Advanced icon, then the Element Range icon (Figure 2).

In the drop-down menu, select the mode that is intended for use (i.e., General Metals, Electronics Metals).

- 7.2 Select the box next to Main Range and Low Range so that a check mark appears in each box.
- 7.3 Enter the desired length of time for each filter. For example, set values of 5 seconds for the Main Range and 5 seconds for the Low Range as shown in Figure 3.

7.4 Select Save and return to the Main Menu by clicking Return.

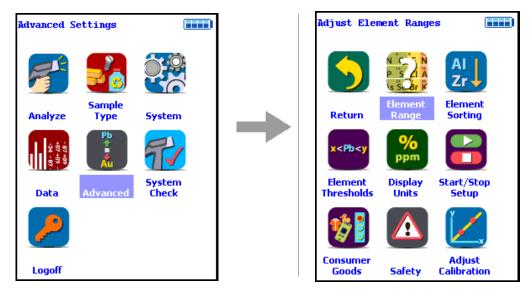


Figure 48. Element Range Menu Path

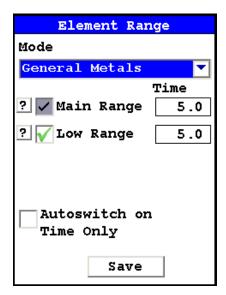


Figure 49. Setting Element Ranges

- 8. Verify instrument measurement accuracy using the 1½Cr ½Mo reference material (RM) supplied with the analyzer.
- 8.1 Test the factory-supplied 1¼Cr-½Mo alloy standard (or other approved check sample) based on a 30s measurement using main range filter only. If the sample is correctly identified and all major elements read within calculated acceptance limits (within the low and high values of factory readings found on QC sheet, proceed to General Testing Protocol section

- 8.2 If the analyzer reports values outside the acceptance tolerance ranges specified in the tables, repeat the detector calibration described in step 7, then repeat the reference sample analysis in step 8.1
- 8.3 If the analyzer again fails to meet the acceptance tolerance ranges specified in the tables, please contact Thermo Scientific Niton Analyzers or your local representative for assistance.

GENERAL TESTING PROTOCOL

1. Good surface preparation is essential for obtaining accurate test results. All non-representative material (e.g., paint, coating, scale) must be removed prior to testing. An approximately 2-inch-square section of surface should be cleaned down to fresh or "white"/"red"/"yellow" metal using an abrasive wheel. To prevent cross contamination, wheels that have been used on one type of alloy should not be used on a different alloy type. For example, a wheel used on stainless steel should not be used on carbon steel. The area to be sampled should be wiped clean or blown with air to remove any grinding dust before testing.

For the Thermo Scientific Niton XL3 Series, test times of 5 seconds on each filter (smart library will determine if low filter is needed) will generally give good identification of grade for most alloys. Total test time will be 5s for most samples but 10s if 2nd filter is needed.

Test times of 10 seconds on each filter will generally provide accurate chemistry analysis for unknown samples. Since there are two Ranges (Main and Low), the total test time will be 20 seconds if the sample is not identified as one of the grade library samples.

The analyzer will often display a correct alloy identification and accurate chemistry result before the above specified time interval. If the accuracy meets the user's requirements, it is not necessary to measure for the full time.

Longer measurements might be necessary if low concentrations of alloy elements must be determined.

2. INSTRUMENT QC

Measure the supplied $1\frac{1}{4}$ Cr $-\frac{1}{2}$ Mo calibration check sample AT LEAST once a shift. If correct, continue work. If incorrect, redo System Check and re-take the past 2 hours of results.

- 3. UNDERSIZED OR NON-CONTACT SAMPLES (samples that do not make contact with or that do not fully cover the measurement aperture)
- 3.1. For samples that do not fully cover the measurement aperture, increase the testing time by increasing the time in inverse proportion to the decrease in percentage of aperture covered. For example: a rod only covers ½ of the aperture, so increase the measurement time by two (e.g., from 10 to 20 seconds per filter for chemistry).
- 3.2. The best procedure to measure undersized samples is to use the Thermo Scientific Niton portable test stand (optional see Parts and Accessories), which is shielded to prevent radiation exposure to the operator.

5 Metals Analysis General Metals Analysis

3.3 An undersized sample may alternately be measured while lying on another material. Results may be affected by the signal coming from the underlying material itself. Use only pure aluminum, pure plastic, or clean wood and employ the Disable Al feature. Use the Tools Menu, then select Disable Al, and check the underlying surface itself to be sure no metals are present. Be sure to use the Tools Menu and select Enable Al before testing aluminum alloys.

PMI Analysis

Field Procedures for PMI Using the Thermo Scientific Niton XL3 800 Series Alloy PMI Tool with Software Version 7.0 or Higher

Standard Operating Procedure

October 2009

1. DOCUMENT PURPOSE

1.1. This document is intended for use as a guide for performing Positive Material Identification (PMI) using the handheld Thermo Scientific Niton XL3 800 Series x-ray fluorescence (XRF) alloy analyzer in the field to ensure compliance with the refinery procedures for ensuring Positive Material Identification.

2. SAFETY

- 2.1. Any persons, including refinery personnel and their representatives, using approved alloy analyzers at the refinery will be given a safety orientation by the PMI coordinator (or his /her approved instructor) prior to using any PMI device.
- 2.2. The safety orientations will include the following as a minimum:
- 2.2.1. Radiation safety for x-ray fluorescence analyzers covering safe operation of the device and emergency procedures in case of malfunction or unit damage during field use.
- 2.2.2. Personnel will demonstrate and acknowledge satisfactory understanding of safe operation by passing and signing a written test for each analyzer to be used. This documentation is to be kept on file by the PMI coordinator or his/her designee.
- 2.3. PMI coordinator will periodically audit field use of instruments for compliance with safety procedures.
- 3. PERSONNEL AUTHORIZED TO CONDUCT PMI INSPECTIONS
- 3.1. Refinery inspection personnel who have completed safety-training requirements.
- 3.2. Refinery's designated contractor representatives. These representatives are people who have demonstrated to the PMI coordinator or his/her designee that in addition to the safety requirements, they also have a firm understanding of the Niton® XL3 800 Series alloy analyzer operation, auto-calibration frequency requirements, and PMI job goals and objectives.
- 3.3. All authorized PMI personnel will be familiar with and have copies of the following documents available to them:
- 3.3.1. Thermo Scientific Niton XL3 800 Series User Guide

5 Metals Analysis PMI Analysis

- 3.3.2. API Recommended Practice 578 Material Verification Program for New and Existing Alloy Piping Systems.
- 3.3.3. PMI Field Procedures (e.g., this document)
- 3.3.4. Other documentation as specified
- 4. PRE-INSPECTION Niton XL3 800 ANALYZER START UP AND CALIBRATION
- 4.1. Startup and Stabilization
- 4.1.1. Turn on, accept the radiation warning (if you are trained), and log into (enter password) the Niton XL3 800 Series analyzer. Allow 5 minutes for the analyzer to stabilize BEFORE proceeding.
- 4.1.2. Calibrate the analyzer by pressing the "System Check" icon. This will start the detector auto calibrate process. When the Niton XL3 instrument completes the auto calibration, press the "Sample Type" icon, press the "Metals" icon and then the "General Metals" icon. Measure the check sample as directed in 4.2.1. If it reads correctly, testing may start (see 4.5).
- 4.2. Verify the Calibration.
- 4.2.1. Test the factory supplied 1¼Cr-½Mo alloy standard (or other approved check sample). If the sample is correctly identified and all major elements (Cr and Mo primarily) read within calculated acceptance limits (see factory QC readings), testing may start (see 4.5).
- 4.2.2. If the check sample does not read correctly, then re-calibrate the analyzer by pressing the "System Check" icon. This will start the auto calibration. When the Niton XL3 instrument completes the auto calibration, return to "Test" and re-measure the check sample as directed in 4.2.1. If it reads correctly, testing may start (see 4.5). If the check sample still does not read correctly contact your local Thermo Scientific Niton Service center.
- 4.3. The need for a hot surface adapter should be determined at this time. If surface temperatures of the components to be tested will exceed 315°C/600°F, the operator must install the HotfootTM hot surface adapter. If sample temperatures will exceed 315°C/600°F but are below 800°F, the user may operate the unit with ~6 mm (~1/4 inch) gap between the front measuring plate and the sample. When testing hot surfaces with the weld adapter in place, the operator should double the measuring time to maintain precision (compensates for the lower signal due to the gap created by the weld adapter and the 3 mm (reduced size) aperture. Accuracy compensation is handled automatically by the Niton XL3 software.
- 4.4. If measurements will be taken on vibrating surfaces, hold the analyzer a few millimeters (~1/8 inch) away from the vibrating surface and double the measuring time to maintain precision. All concentration accuracy compensation (for the gap) is handled automatically by the Niton XL3 software (up to maximum of 6 mm).

4.5. Check the analyzer for material-specific performance prior to any actual testing. Typically, this should be done using a Certified Reference Material (CRM) or Reference Material (RM) such as an ARMI CRM. The CRM(s) used in this procedure should represent each of the materials expected to be encountered during inspection. A kit of 15 PMI refinery-specific CRM alloys is available upon request. Contact Thermo Scientific Niton Analyzers or your representative for a quote.

5. FIELD TESTING REQUIREMENTS

- 5.1. Prior to beginning testing, the specific PMI testing and documentation requirements for the job should be discussed with the PMI coordinator or his designee. Data storage can be accomplished in real time at the exact test site by utilizing optional data download button storage devices and position indicators that affix directly to the measured piece or location. Contact Thermo Scientific Niton Analyzers or your representative for a quote.
- 5.2. Good surface preparation is essential for obtaining accurate test results. All non representative material (e.g., paint, coating, scale) must be removed prior to testing. This can be accomplished by using hand files, grinders, pneumatic rotary files, etc. to obtain a clean metal area on which to conduct the test. Test areas on welds may have to be ground flat. Note: The battery powered Makita 9500D cordless angle grinder with several extra batteries has been found to provide good service with rapid surface preparation.
- 5.3. Test time for the Niton XL3 Series will be a minimum of 5 seconds for grade identification and 10 seconds for chemical analysis. If used in the Spectral Fingerprints mode, testing time may be reduced to 3-5 seconds for identification. Although the analyzer may display a result before the specified time interval, the operator should continue to measure until the specified time has elapsed. Note sections above and below covering special cases that require longer measuring times.
- 5.4. One test point for each piping component (pipe, tee, ell, etc.) is required. Weld test requirements vary according to size. See Section 6.
- 5.5. Minimum documentation for field work will include:
- 5.5.1. An isometric drawing or sketch of the as-installed and tested piping or equipment. This drawing should include equipment ID and piping line number, or other documentation reference(s) as specified.
- 5.5.2. Alloy chemistry documentation for the primary elements required to provide PMI of the alloy grade for all units tested.
- 5.5.3. Documentation must be signed and dated by the personnel that performed the tests; the signed documentation is then given to the PMI coordinator or designee.
- 5.5.4. The PMI coordinator or designee may require additional documentation, which will be specified prior to testing. Electronic copies of the encrypted Niton Data Transfer (NDT©) files should be maintained in addition to paper copies.
- 6. WELDS NEW AND EXISTING INSPECTION

5 Metals Analysis PMI Analysis

- 6.1. Prior to welding, consumables should be tested as indicated in 6.6 below.
- 6.2. Number of test locations per weld:
- 6.2.1. 34" to 1-1/2" butt welds One test point.
- 6.2.2. 34" to 2" socket welds One test point.
- 6.2.3. 2" to 4" butt welds Two test points.
- 6.2.4. 6" and larger weld At least four test areas evenly distributed around the circumference of the weld should be inspected. If no discrepancy is found, the average of the primary identifier element chemistries for all areas tested on that weld can be used for documentation.
- 6.3. At the discretion of the inspector, as in the case of large diameter welds or at any other time deemed necessary to ensure correctness of the weld material, additional locations on the weld may be tested.
- 6.4. When testing welds, care must be taken to position the analyzer window to read only the weld, with no false reading from the adjacent piping component.
- 6.4.1. If weld is smaller than the measuring aperture (smaller than 10 mm), insert the weld adapter and activate the Weld Mode with the Weld Icon. If your analyzer did not come with the optional weld adapter, there will not be a Weld Mode. Contact Thermo Scientific Niton Analyzers or your representative for a quote.
- 6.4.2. The weld adapter should reduce the measurement aperture width to a size smaller than the width of the weld to be tested, or
- 6.4.3. If the optional weld adapter is not an acquired accessory, it is possible to isolate the weld by masking adjacent metal with a non readable material, such as several layers of pure aluminum tape. Care should be taken that no readable elements are contained in the mask material and that the mask material is thick enough to prevent read through from the underlying material.
- 6.4.4. Double the measuring times when using the weld adapter (e.g., increase from 5 source seconds to 10 source seconds for ID, and from 10 to 20 seconds for chemistry), to compensate for the added gap and reduced surface area being tested.
- 6.5. Testing of welding consumables shall be performed as follows:
- 6.5.1. Each and every box of weld rod to be used for the job will be prepared by first puddling a sample using at least two passes and grinding smooth for testing.
- 6.5.2. Documentation for the weld rod tested should include:
- 6.5.2.1. Rod designation
- 6.5.2.2. Manufacturer's name
- 6.5.2.3. Box lot number

- 6.5.2.4. Element composition of samples
- 6.5.3. Rod box will be marked PMI CONFIRMED with:
- 6.5.3.1. Name of authorized person who performed the testing
- 6.5.3.2. Date tested
- 6.5.3.3. Other information as specified (e.g., vendor or contractor ID)
- 6.5.4. GTAW (TIG) wire can often be tested directly from the box either individually with a slot aperture or by using several pieces to cover more of the aperture window of the analyzer. If satisfactory results cannot be obtained, then puddling and grinding of the wire should be performed as specified in 6.5.1.
- 7. UNDERSIZED OR NON-CONTACT SAMPLES (samples that do not make contact with or that do not fully cover the measurement aperture)
- 7.1. For samples that do not fully cover the measurement aperture, increase the testing time by increasing the time in inverse proportion to the decrease in percentage of aperture covered. For example: a rod only covers ½ of the aperture, so increase the measurement time by two. (e.g., from 5 seconds to 10 seconds for ID and from 10 to 20 seconds for chemistry).
- 7.1.1. Undersized samples should be placed in the Thermo Scientific portable alloy test stand (optional), which is shielded to prevent radiation exposure to the operator. (NOTE: An undersized sample one that does not cover the entire measuring aperture measured while lying on another material such as, e.g., a stainless steel table top, will be affected by the signal coming from the table top itself.) Be sure to employ the Disable Al feature if measuring on an aluminum table or any plastic or wood table. Use "Tools," then select "Disable Al." Be sure to use Tools to "Enable Al" before testing aluminum alloys.
- 7.2. Non-contact sample measurements include those measured with a gap of up to a maximum of ¼ inch (~6 mm) from the Niton XL3 measurement aperture opening. Such measurements require double the exposure time. (e.g., from 5 seconds to 10 seconds for ID and from 10 to 20 seconds for chemistry).
- 7.3. Inside pipe measurements on pipes that are less than a few feet in diameter will result in non-contact measurements. Follow recommendations in 7.2 above.
- 8. INSTRUMENT QC
- 8.1. Measure the supplied $1\frac{1}{4}$ Cr $-\frac{1}{2}$ Mo calibration check sample AT LEAST once for every 2 hours of use time. If correct, continue work. If incorrect, recalibrate the Niton XL3 detector and re-take the past 2 hours of results.
- 9. STAMPS
- 9.1. All stamping is done with a specified proprietary stamp.

5 Metals Analysis PMI Analysis

- 9.2. All stamps purchased for special marking should be marked with a control number. A list indicating the number and location of these stamps is held by the PMI coordinator.
- 9.3. Typically, a stamp is assigned to each individual analyzer and stays in the respective analyzer's field carry bag.
- 9.4. Report any loss of a stamp immediately to the PMI coordinator or designee.

10. WHEN TO USE AN APPROVED STAMP

- 10.1. Materials and components should be tested prior to fabrication. This will help find any discrepancies before final field inspection and prevent costly field cut outs.
- 10.2. Items checked prior to fabrication and before the final as-installed condition will only be marked with approved paint pen as "PMI CONFIRMED."
- 10.3. Chemistry documentation may not be necessary for this phase of the PMI inspection.
- 10.4. The number of components and type tested should be documented for the job.
- 10.5. Discrepancies should be noted and handled as specified in the appropriate procedures document.
- 10.6. Shop made spool pieces may be stamped, providing that their final field location in the piping system can be tracked and documented. Final field installation must be confirmed by visual inspection for the stamp.
- 10.7. Use of any approval stamp should be in accordance with the appropriate company procedures document for PMI on existing equipment.
- 10.8. Use of an approval stamp does not negate the need for the written documentation.

11. RESOLUTION OF DISCREPANCIES

- 11.1. When analysis indicates a wrong material, component, or weld, then take the following steps:
- 11.2. Test several other areas on the same component. For welds, check adjacent components.
- 11.3. Check the analyzer calibration against the check sample or other known sample.
- 11.4. If performing pre-fabrication testing, components having reached this point must be rejected according to established procedures.
- 11.5. Discrepancies on field installed or in-place existing materials requires notifying the responsible inspector and completing the following items:
- 11.5.1. Complete chemistries should be taken for all elements present in the suspect material to help identify the material.

- 11.5.2. Review the test data for the suspect component, and, if possible, confirm the results using a second analyzer.
- 11.5.3. If conclusive results cannot be obtained with field analyzers, the inspector may request a lab analysis on a small sample material removed from the component.

FAC Analysis

Field Procedures for Measurement of FAC Predictor Elements Cr, Cu, Mo Using the Thermo Scientific Niton XL3 800 Series Alloy Analyzer with Software Version 7.0 or Higher

1. DOCUMENT PURPOSE

1.1. This document is intended for use as a guide for using the handheld Thermo Scientific Niton XL3 800 Series x-ray fluorescence (XRF) alloy analyzer as a tool in performing Flow Accelerated Corrosion (FAC) data modeling to ensure that proper measurement parameters are followed to reach indicative low-level concentrations of Cr, Cu, and Mo.

2. SAFETY

- 2.1. Any persons, including power plant personnel and their representatives, using approved alloy analyzers at the plant will be given a safety orientation by the FAC coordinator (or his/her approved instructor) prior to using any XRF inspection device.
- 2.2. The safety orientations will include the following as a minimum:
- 2.2.1. Radiation safety for x-ray fluorescence analyzers covering safe operation of the device as well as emergency procedures in case of malfunction or unit damage during field use.
- 2.2.2. Personnel will demonstrate and acknowledge satisfactory understanding of safe operation by passing and signing a written test for each analyzer to be used. This documentation is to be kept on file by the FAC coordinator or his/her designee.
- 2.3. FAC coordinator will periodically audit field use of instruments for compliance with safety procedures.
- 3. PERSONNEL AUTHORIZED TO CONDUCT MEASUREMENT OF FAC PREDICTOR ELEMENTS
- 3.1. Power plant inspection personnel who have completed safety-training requirements.
- 3.2. Power plant's designated contractor representatives. These representatives are people who have demonstrated to the FAC coordinator or his/her designee that in addition to the safety requirements, they also have a firm understanding of the Niton® XL3 800 Series alloy analyzer operation, auto-calibration frequency requirements, and FAC program goals and objectives.
- 3.3. All authorized inspection personnel will be familiar with and have copies of the following documents available to them:
- 3.3.1. Thermo Scientific Niton XL3 800 Series User Guide
- 3.3.2 Thermo Scientific Niton NDT Data Transfer User's Guide
- 3.3.3 Flow Accelerated Corrosion in Power Plants, EPRI Publication, 1996
- 3.3.4. Other documentation as specified

- 4. PRE-INSPECTION Niton XL3 800 ANALYZER START UP AND CALIBRATION
- 4.1. Startup and Stabilization
- 4.1.1. Turn on, accept the radiation warning (if you are trained), and log into (enter password) the Niton XL3 800 Series analyzer (see Figures 1 & 2).

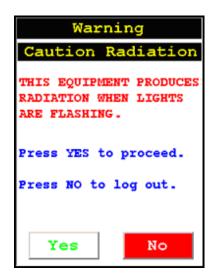


Figure 50. Warning Screen

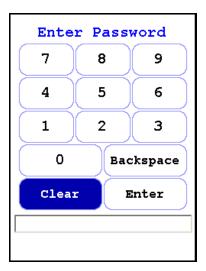


Figure 51. Virtual Numeric Keypad for Log On

5 Metals Analysis FAC Analysis

4.1.2. Ensure that the filters are properly set up for the measurement: From "Main Menu" select "Advanced" -> "Element Range" (see Figure 1-3). In the drop-down box select "General Metals". Ensure that the boxes are checked for "Main Range" and "Low Range". Ensure that the "Time" boxes show "10.0" in both the "Main Range" and "Low Range" boxes (see Figure 1-4). If not, -> "Main Range Time" -> "C" to clear existing entry -> "1", "0", "E" to enter the desired time of 10 seconds. Repeat for the "Low Range Time" if so required (see Figure 1-5). Ensure that the "Filter Change on Time Only" is checked. Then press "Save".

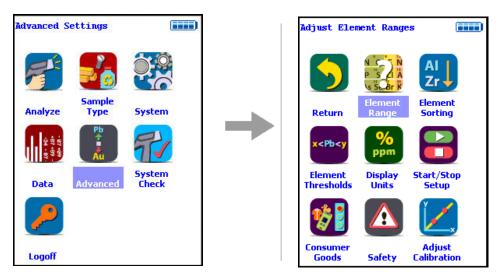


Figure 52. The Element Range Menu Path

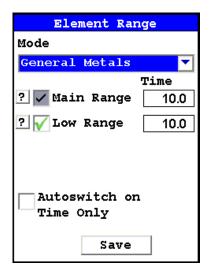


Figure 53. Range Checkboxes, Range Time, and Time Only Checkbox

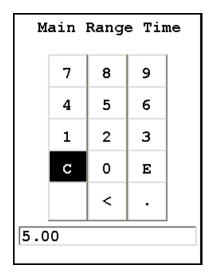


Figure 54. Range Time Editor

4.1.3. Set up the screen display so that Cr, Cu, and Mo are always displayed, and are shown prominently at the top of the displayed element list: From "Main Menu" select "Advanced" -> "Element Sorting" (see Figure 1-6). In the drop down box select "Alloy Mode" (see Figure 7); in the element box scroll down and highlight Cr; in the "Display Options" box select "Always Display"; then using the blue arrow keys at the right of the screen use the up arrow to move Cr to the top of the element table; repeat this procedure for Cu and Mo respectively, first marking them as "Always", then moving them up the element table to immediately below Cr. Press "Save" -> "Close" -> "Return" -> "Return" to reach the Main Menu. See Figure 1-8.

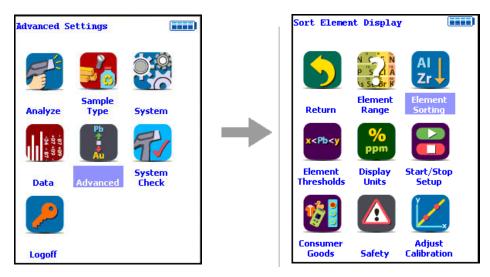


Figure 55. Sort Element Display Menu Path

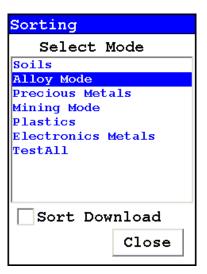


Figure 56. Sort Element Display

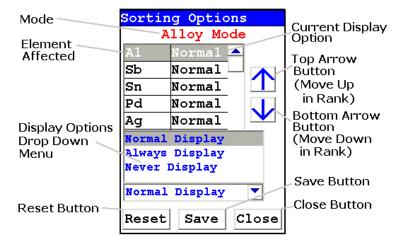


Figure 57. Sorting Options Screen

4.1.4. Calibrate the analyzer by pressing the "System Check" icon, (see Figure 9). This will start the detector auto-calibrate function. When the Niton XL3 instrument completes the auto-calibration, return to "Test" and measure the check sample as directed in 4.2.1. If it reads correctly, testing may start (see 5.0).

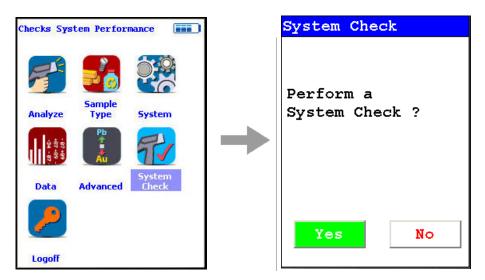


Figure 58. System Check Menu Path

- 4.2. Verify the Calibration.
- 4.2.1. Test the factory supplied 1¼Cr-½Mo alloy standard (or other approved check sample). If the sample is correctly identified and all major elements (Cr and Mo primarily) read within calculated acceptance limits (within the low and high values of factory readings found on QC sheet), testing may start (see 5.1).
- 4.2.2. If the check sample does not read correctly, then re-calibrate the analyzer by pressing the "System Check" icon (see Figure 9). This will start the auto-calibration. When the Niton XL3 instrument completes the auto-calibration, return to "Test" and re-measure the check sample as directed in 4.2.1. If it reads correctly, testing may start (see 5.0). If the check sample still does not read correctly, contact your local Thermo Scientific Niton service center.
- 4.3. If a weld of less than 10 mm is to be analyzed, it will be necessary to reduce the analysis area from the 10 mm normal aperture size to a size below that of the weld bead. See section 6.4.1.
- 4.4. Check the analyzer for sensitivity performance for Cr, Cu, and Mo prior to any actual testing. Typically, this should be done using a Certified Reference Material (CRM) such as an ARMI CRM. The CRM(s) used in this procedure should represent each of the materials expected to be encountered during inspection.

5. FIELD TESTING REQUIREMENTS

5.1. Prior to beginning testing, the specific testing and documentation requirements for the job should be discussed with the FAC coordinator or his designee. Data storage can be accomplished in real time at the exact test site by utilizing optional data download button storage devices and position indicators (compact memory button – CMB) that affix directly to the measured piece or location. Contact Thermo Scientific Niton Analyzers or your local representative for a quote.

5 Metals Analysis FAC Analysis

- 5.2. Good surface preparation is essential for obtaining accurate test results. All non-representative material (e.g., paint, coating, scale) must be removed prior to testing. An approximately 2-inch-square section of surface should be cleaned down to fresh or "white" metal using an abrasive flapper wheel. The cleaning should not be performed using the same wheel that was used to prepare the component for ultrasonic testing (UT), but should be performed using a fresh wheel, designated for this operation only. Wheels that have been used on high chromium alloyed components (e.g., 300 series chromium-nickel stainless steels, or chromium steels) should not be used to clean carbon steel components. The area to be sampled should be wiped clean or blown with air to remove any grinding dust before testing.
- 5.3. Test time for the Niton XL3 Series will be a minimum of 10 seconds on each filter for accurate chemistry analysis. Since two filters (Main and Low) are required for the targeted low concentrations, the total test time shall be a minimum of 20 seconds. Although the analyzer may display a result before the specified time interval, the operator should continue to measure until the specified time has elapsed. Note sections above and below covering special cases that require longer measuring times.
- 5.4. One test point for each piping component (pipe, tee, ell, etc.) is required. Weld test requirements vary according to size. See Section 6.
- 5.5. Minimum documentation for field work will include:
- 5.5.1. An isometric drawing or sketch of the as-installed and tested piping or equipment. This drawing should include equipment ID and piping line number, or other documentation reference(s) as specified.
- 5.5.2 Documentation must be signed and dated by the personnel that performed the tests; the signed documentation is then given to the FAC coordinator or designee.
- 5.5.4. The FAC coordinator or designee may require additional documentation, which will be specified prior to testing. Electronic copies of the encrypted Niton Data Transfer (NDT©) files should be maintained in addition to paper copies.
- 6. WELDS NEW AND EXISTING INSPECTION
- 6.1. Prior to welding, consumables should be tested as indicated in 6.5 below.
- 6.2. Number of test locations per weld:
- $6.2.1. \frac{3}{4}$ " to $1-\frac{1}{2}$ " butt welds one test point.
- 6.2.2. ³/₄" to 2" socket welds one test point.
- 6.2.3. 2" to 4" butt welds two test points.
- 6.2.4. 6" and larger weld At least four test areas that are evenly distributed around the circumference of the weld should be inspected. If no discrepancy is found, the average of the primary identifier element chemistries for all areas tested on that weld can be used for documentation.

- 6.3. At the discretion of the inspector, as in the case of large diameter welds, or at any other time deemed necessary to ensure correctness of the weld material, additional locations on the weld may be tested.
- 6.4. When testing welds, care must be taken to position the analyzer window to read only the weld, with no false reading from the adjacent piping component.
- 6.4.1. If weld is smaller than the measuring aperture (smaller than 10 mm), insert the weld adapter and activate the Weld Mode with the Weld Icon. If your analyzer did not come with the optional weld adapter, there will not be a Weld Mode. Contact Thermo Scientific Niton Analyzers or your local representative for a quote.
- 6.4.2. The weld adapter should reduce the measurement aperture width to a size smaller than the width of the weld to be tested, or
- 6.4.2. If the optional weld adapter is not an acquired accessory, it is possible to isolate the weld by masking adjacent metal with a non-readable material, such as several layers of pure aluminum tape. Care should be taken that no readable elements are contained in the mask material and that the mask material is thick enough to prevent read through from the underlying material.
- 6.4.4. Double the measuring times when using the weld adapter (e.g., increase from 10 to 20 seconds per filter for chemistry) to compensate for the added gap and reduced surface area being tested.
- 6.5. Testing of welding consumables shall be performed as follows:
- 6.5. Testing of welding consumables shall be performed as follows:
- 6.5.1. Each and every box of weld rod to be used for the job will be prepared by first puddling a sample using at least two passes and then grinding smooth for testing.
- 6.5.2. Documentation for the weld rod tested should include:
- 6.5.2.1. Rod designation
- 6.5.2.2. Manufacturer's name
- 6.5.2.3. Box lot number
- 6.5.2.4. Element composition of samples
- 6.5.3. Rod box will be marked FAC CONFIRMED with:
- 6.5.3.1. Name of authorized person who performed the testing
- 6.5.3.2. Date tested
- 6.5.3.3. Other information as specified (e.g., vendor or contractor ID)

5 Metals Analysis FAC Analysis

- 6.5.4. GTAW (TIG) wire can often be tested directly from the box either individually with a slot aperture or by using several pieces to cover more of the aperture window of the analyzer. If satisfactory results cannot be obtained, then puddling and grinding of the wire should be performed as specified in 6.6.1.
- 7. UNDERSIZED OR NON-CONTACT SAMPLES (samples that do not make contact with or that do not fully cover the measurement aperture)
- 7.1. For samples that do not fully cover the measurement aperture, increase the testing time by increasing the time in inverse proportion to the decrease in percentage of aperture covered. For example: a rod only covers ½ of the aperture, so increase the measurement time by two (e.g., from 10 to 20 seconds per filter for chemistry).
- 7.1.1. Undersized samples should be placed in a Thermo Scientific Niton approved alloy test stand (optional), which is shielded to prevent radiation exposure to the operator. (NOTE: An undersized sample one that does not cover the entire measuring aperture measured while lying on another material, such as a stainless steel table top, will be affected by the signal coming from the table top itself.) Be sure to employ the Disable Al feature if measuring on an aluminum table or any plastic or wood table. Use "Tools," then select "Disable Al" Be sure to use Tools to "Enable Al" before testing aluminum alloys.
- 7.2. Inside pipe measurements on pipes that are less than a few feet in diameter will result in non-contact measurements. Follow recommendations in 7.2 above.

8. INSTRUMENT QC

8.1. Measure the supplied $1\frac{1}{4}$ Cr $-\frac{1}{2}$ Mo calibration check sample AT LEAST once for every 2 hours of use time. If correct, continue work. If incorrect, recalibrate the Niton XL3 detector and re-take the past 2 hours of results.

9. RESOLUTION OF DISCREPANCIES

- 9.1. When analysis indicates an undesirable or anomalous result, take the following steps:
- 9.2. Test several other areas on the same component. For welds, check adjacent components.
- 9.3. Check the analyzer calibration against the check sample or other known sample.
- 9.4. If performing pre-fabrication testing, components having reached this point must be rejected according to established procedures.
- 9.5. Discrepancies on field-installed or in-place existing materials require notification of the responsible inspector and completing the following items:
- 9.5.1. Review the test data for the suspect component, and, if possible, confirm the results using a second analyzer.

Coatings Analysis

Coating Thickness Measurement Using the Thermo Scientific Niton XL3 800 Series X-Ray Fluorescence (XRF) Analyzer – Version 7.0 and Higher

Standard Operating Procedure

October 2009

Note Each user should read the Thermo Scientific Niton XL3 User's Guide carefully before initiating measurements with the system. Users are strongly urged to attend the Thermo Scientific Niton XRF Analyzer Radiation Safety and Operations Training courses offered regularly, or the web-based trainings. For more information, visit www.thermo.com/niton.

Preparatory Tasks

- 1. Attach a charged battery to the analyzer and turn it on. Follow the screen instructions and Log On as the operator using either the default password or a custom one as designated by the user in an NDU file.
- 2. Wait five (5) minutes before using the analyzer, allowing the instrument electronics to stabilize.
- 3. Verify that the date is set properly for data tracking purposes.

From the Main Menu, select the System icon, then the Specs icon. The date will be displayed for verification. If the date is incorrect, correct it prior to proceeding. This can be done by returning to the System Menu and selecting the Date & Time icon. See Setting the Date and Time for detailed information on this procedure.

- 4. (Optional) Connect the analyzer to a computer via the included serial cable, USB cable, or Bluetooth™ wireless module. See Connecting the XRF for more information.
- 5. During analysis and detector calibrations, it is important to ensure that the analyzer is not exposed to strong electromagnetic fields, including those produced by computer monitors, hard drives, cellular telephones, walkie talkies, etc. Keep a minimum two (2) feet (0.7 meters) distance between the analyzer and electronic devices.
- 6. From the Main Menu, select the System Check icon, and select the Yes Button. (Figure 1-1.)
- 6.1. System Check calibrates the detector and verifies it is operating to specifications. After starting the process, no further user interaction is required during this operation. When the instrument is finished performing the check, the unit will show either "System OK" or one of the failure errors.

5 Metals Analysis Preparatory Tasks

6.2. If the unit shows a failure error, then perform a second System Check by selecting the Recheck Button. If the unit still does not show a "System OK," please contact Thermo Scientific Niton Analyzers toll-free at (800) 875-1578, +1 978 670-7460, niton@thermofisher.com, or contact your local Niton Analyzers representative for assistance.

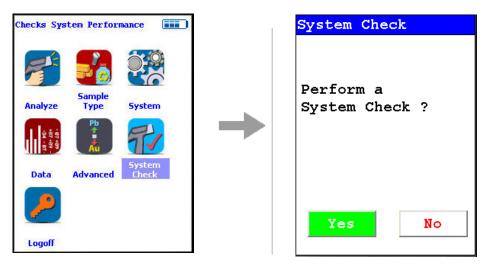


Figure 59. System Check Menu Path

7. Verify and/or change filter position time.

Thermo Scientific Niton XL3 analyzers are equipped with 2 excitation filters that optimize the analyzers' sensitivity for various elements. The "Main Range" filter provides optimum sensitivity for the elements manganese (Mn) through bismuth (Bi). The "Low Range" filter is used to optimize the sensitivity for the elements from titanium (Ti) through chromium (Cr). Note that the main range filter can be used to analyze Ti, V and Cr, but the sensitivity is not as good as when using the low filter. The amount of time that the analyzer spends in each filter position is user definable. Please note that the analyzer will continue alternating excitation filters until the user selectable maximum analysis time is reached or the operator terminates the measurement. To verify or change the length of time each filter will be active, do the following:

7.1 From the Main Menu select the Advanced icon, then the Element Range icon (Figure 22)

In the drop-down menu, select the mode that is intended for use (i.e., General Metals, Electronics Metals).

- 7.2 Select the box next to Main Range and Low Range so that a check mark appears in each box.
- 7.3 Enter the desired length of time for each filter. For example, set values of 5 seconds for the Main Range and 5 seconds for the Low Range as shown in Figure 23.
- 7.4 Select the Save Button and return to the Main Menu by selecting the Return icon.

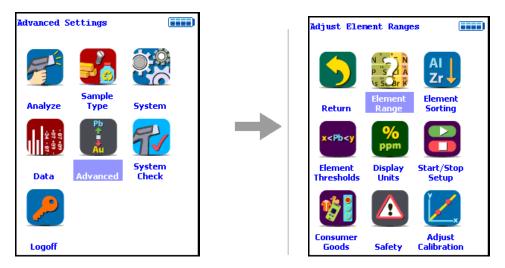


Figure 60. Element Range Menu Path

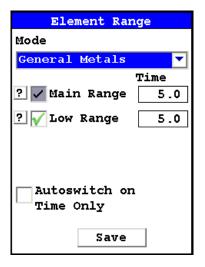


Figure 61. Setting Element Ranges

- 8. Verify instrument measurement accuracy using the 1½Cr ½Mo certified reference material (CRM) supplied with the analyzer.
- 8.1 Test the factory-supplied 1¼Cr-½Mo alloy standard (or other approved check sample) based on a 20s measurement using main range filter only (see Figure 1-3). If the sample is correctly identified and all major elements read within calculated acceptance limits (within 3 sigma of the average value of factory readings, proceed to General Testing Protocol section.
- 8.2 If the analyzer reports values outside the acceptance tolerance ranges specified in the tables, repeat the System Check described in step 7, then repeat the reference sample analysis in step 8.1.

8.3 If the analyzer again fails to meet the acceptance tolerance ranges specified in the tables, please contact Thermo Scientific Niton Analyzers or your local representative for assistance.

Coatings Entries and Standardization

How to Access Metal Coatings Mode

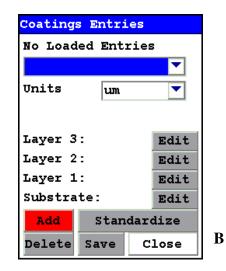
To access Metal Coatings mode follow the sequence shown in Figure 24.



Figure 62. Metal Analysis Menu Path

If no coatings method entries have been loaded, screen A in Figure 25. will appear. Press the Add Button, which brings up a Virtual Keyboard, and enter a method name, which will bring you to screen B. Follow the steps in <u>Creating a Metal Coatings Entry</u> to create a new entry.

If method entries have been loaded, screen C will appear. Select desired method from drop down menu. If you need to add more methods, select the Edit Methods Button to open screen A.



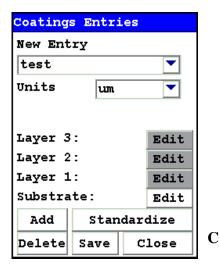




Figure 63. Screens A, B, and C

Creating a Metal Coatings Entry

A

Before coating thickness can be measured, a Coatings System Entry must be made defining the coatings system (see Figures 26-29). Note that the Coatings Method Menu can be accessed via the steps outlined in Figures 24 and 25 above or via the Tools Menu. See <u>Using the Tools Menu</u> for more information on the Tools Menu.

Metals AnalysisCoatings Entries and Standardization

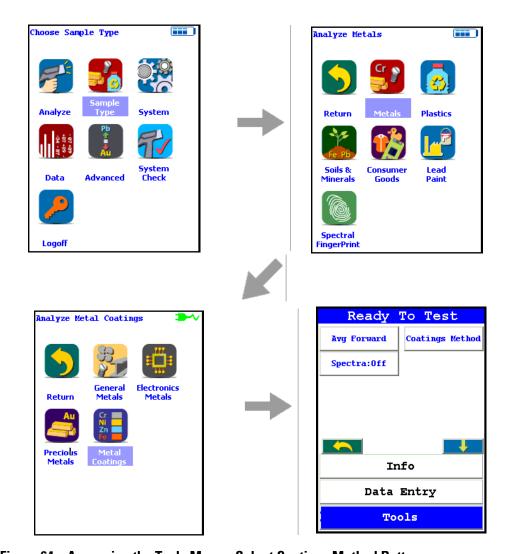


Figure 64. Accessing the Tools Menu - Select Coatings Method Button

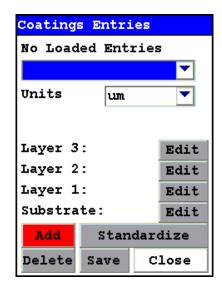
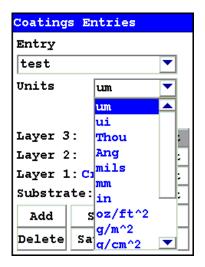
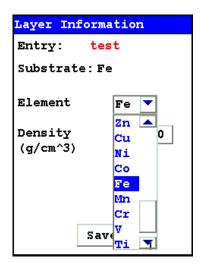


Figure 65. Coating System Screen

When creating a coating system, select the Add Button to activate the Virtual Keyboard and enter the name of a coating system (e.g. test). Once the name is created, select the coatings thickness units, substrate (type of metal to be coated) and layers (metal coatings) from the individual pull-down menus. See Figure 28.





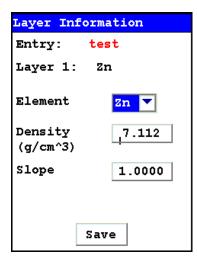


Figure 66. Entering Units, Substrate, and Layer Information

Standardize Feature

The Standardize feature is a standardization routine that corrects for any bias in thickness measurement when compared to a known thickness sample or thickness standard.

5 Metals Analysis Coatings Entries and Standardization

To standardize, follow the sequence shown in Figure 29. From the Coating Entries screen select the Standardize Button. Enter known thickness of Zn layer, e.g. 20.0, and initiate a reading. Select the Yes Button to save the standardization.

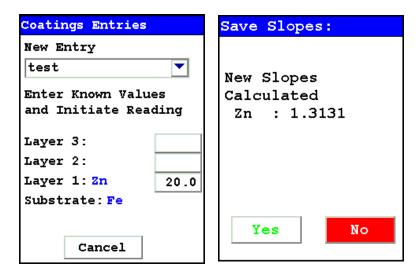


Figure 67. Standardizing the Coating

Coatings Testing Protocol

Once a methods are created and standardized the analyzer is ready for routine analysis. All non-representative material (e.g. dust, oil) must be removed prior to testing. Test times of 10 s will generally provide accurate results

Figure 30. shows a typical measurement of Zn thickness over steel.

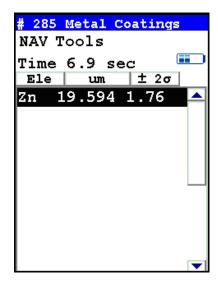


Figure 68. Example Coating Measurement

There is a maximum coating thickness that can be measured. This maximum value, know as "infinite thickness", varies with coating element. If a coating above this value is measured it will give the message Saturated as shown in Figure 31.



Figure 69. Example Saturated Measurement

Typical infinite thickness values are given the fowllowing table.

5 Metals Analysis Coatings Entries and Standardization

Coating Infinite Thickness

Element	Substrate	Microns
Au	Cu	10
Cd	Al	75
Cd	Fe	75
Co	Fe	50
Co	Ti	50
Cr	Fe	15
Cu	Fe	20
Mo	Fe	50
Mo	Ti	50
Ni	Fe	20
Pt	Cu	15
Sn	Fe	100
Ti	Fe	50
Zn	Fe	50
Zr	Al	70

General Metals Analysis

Analyzing General Metal (Alloy) Samples

Using the Thermo Scientific Niton XL3 800 Series X-Ray Fluorescence (XRF) Analyzer – Version 7.0 and Higher

Standard Operating Procedure

October 2009

Note - Each user should read the Thermo Scientific Niton XL3 User's Guide carefully before initiating measurements with the system. Users are strongly urged to attend the Thermo Scientific Niton XRF Analyzer Radiation Safety and Operations Training courses offered regularly, or the web-based trainings. For more information, visit www.thermo.com/niton.

PREPARATORY TASKS

- 1. Attach a charged battery to the analyzer and turn it on. Follow the screen instructions and "Log On" as the operator using either the default password or a custom one as designated by the user in an NDU file. See Passwords and User Privileges for details.
- 2. Wait five (5) minutes before using the analyzer, allowing the instrument electronics to stabilize.
- 3. Verify that the date is set properly for data tracking purposes.

From the Main Menu, select the System icon, then the Specs icon. The date will be displayed for verification. If the date is incorrect, correct it prior to proceeding. This can be done by "Closing" out of the Specs screen and selecting the Date & Time icon. Detailed information on this procedure is available in Setting the Date and Time.

- 4. (Optional) Connect the analyzer to a computer via the included serial cable, USB cable, or Bluetooth™ wireless module. (Consult Connecting the XRF Analyzer to Your PC for details, if necessary.)
- 5. During analysis and detector calibrations, it is important to ensure that the analyzer is not exposed to strong electromagnetic fields, including those produced by computer monitors, hard drives, cellular telephones, walkie talkies, etc. Keep a minimum two (2) feet (0.7 meters) distance between the analyzer and electronic devices.
- 6. From the Main Menu, select System Check icon then the Yes button. (Figure 1.)
- 6.1. System Check calibrates the detector and verifies it is operating to specifications. After starting the process, no further user interaction is required during this operation. When the instrument is finished performing the check, the unit will show either "System OK" or one of the failure errors.

6.2. If the unit shows a failure error, then perform a second System Check by clicking Recheck. If the unit still does not show a "System OK," please contact Thermo Scientific Niton Analyzers toll-free in the USA at (800) 875-1578, +1 978 670-7460, niton@thermofisher.com, or contact your local Niton Analyzers representative for assistance.

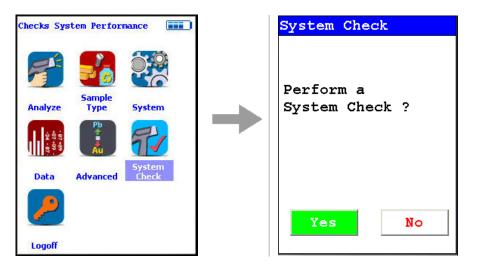


Figure 70. System Check Menu Path

7. Verify and/or change Range time.

Thermo Scientific Niton XL3 analyzers are equipped with excitation filters that optimize the analyzers' sensitivity for various elements. The "Main Range" filter provides optimum sensitivity for the elements manganese (Mn) through bismuth (Bi). The "Low Range" filter is used to optimize the sensitivity for the elements from titanium (Ti) through chromium (Cr). Note that the main range filter can be used to analyze Ti, V and Cr, but the sensitivity is not as good as when using the low filter. The "Light Range" filter is available only with He-purged and GOLDD technology analyzers, and is typically used in light element analysis. The amount of time that the analyzer spends in each filter position is user definable. Please note that the analyzer will continue alternating excitation filters until the user selectable maximum analysis time is reached or the operator terminates the measurement. To verify or change the length of time each filter will be active, do the following:

7.1 From the Main Menu select the Advanced icon, then the Element Range icon (Figure 2).

In the drop-down menu, select the mode that is intended for use (i.e., General Metals, Electronics Metals).

- 7.2 Select the box next to Main Range and Low Range so that a check mark appears in each box.
- 7.3 Enter the desired length of time for each filter. For example, set values of 5 seconds for the Main Range and 5 seconds for the Low Range as shown in Figure 3.
- 7.4 Select Save and return to the Main Menu by clicking Return.

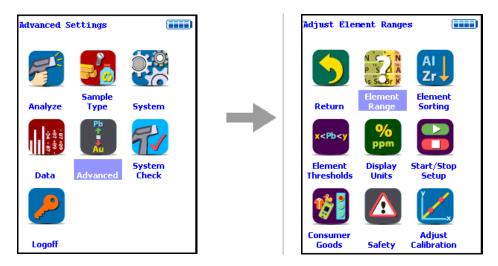


Figure 71. Element Range Menu Path

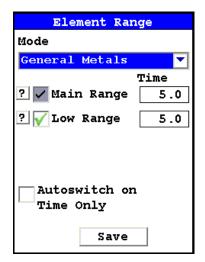


Figure 72. Setting Element Ranges

- 8. Verify instrument measurement accuracy using the 1½Cr ½Mo reference material (RM) supplied with the analyzer.
- 8.1 Test the factory-supplied 1¼Cr-½Mo alloy standard (or other approved check sample) based on a 30s measurement using main range filter only. If the sample is correctly identified and all major elements read within calculated acceptance limits (within the low and high values of factory readings found on QC sheet, proceed to General Testing Protocol section
- 8.2 If the analyzer reports values outside the acceptance tolerance ranges specified in the tables, repeat the detector calibration described in step 7, then repeat the reference sample analysis in step 8.1

8.3 If the analyzer again fails to meet the acceptance tolerance ranges specified in the tables, please contact Thermo Scientific Niton Analyzers or your local representative for assistance.

GENERAL TESTING PROTOCOL

1. Good surface preparation is essential for obtaining accurate test results. All non-representative material (e.g., paint, coating, scale) must be removed prior to testing. An approximately 2-inch-square section of surface should be cleaned down to fresh or "white"/"red"/"yellow" metal using an abrasive wheel. To prevent cross contamination, wheels that have been used on one type of alloy should not be used on a different alloy type. For example, a wheel used on stainless steel should not be used on carbon steel. The area to be sampled should be wiped clean or blown with air to remove any grinding dust before testing.

For the Thermo Scientific Niton XL3 Series, test times of 5 seconds on each filter (smart library will determine if low filter is needed) will generally give good identification of grade for most alloys. Total test time will be 5s for most samples but 10s if 2nd filter is needed.

Test times of 10 seconds on each filter will generally provide accurate chemistry analysis for unknown samples. Since there are two Ranges (Main and Low), the total test time will be 20 seconds if the sample is not identified as one of the grade library samples.

The analyzer will often display a correct alloy identification and accurate chemistry result before the above specified time interval. If the accuracy meets the user's requirements, it is not necessary to measure for the full time.

Longer measurements might be necessary if low concentrations of alloy elements must be determined.

2. INSTRUMENT QC

Measure the supplied $1\frac{1}{4}$ Cr $-\frac{1}{2}$ Mo calibration check sample AT LEAST once a shift. If correct, continue work. If incorrect, redo System Check and re-take the past 2 hours of results.

- 3. UNDERSIZED OR NON-CONTACT SAMPLES (samples that do not make contact with or that do not fully cover the measurement aperture)
- 3.1. For samples that do not fully cover the measurement aperture, increase the testing time by increasing the time in inverse proportion to the decrease in percentage of aperture covered. For example: a rod only covers ½ of the aperture, so increase the measurement time by two (e.g., from 10 to 20 seconds per filter for chemistry).
- 3.2. The best procedure to measure undersized samples is to use the Thermo Scientific Niton portable test stand (optional see Parts and Accessories), which is shielded to prevent radiation exposure to the operator.

3.3 An undersized sample may alternately be measured while lying on another material. Results may be affected by the signal coming from the underlying material itself. Use only pure aluminum, pure plastic, or clean wood and employ the Disable Al feature. Use the Tools Menu, then select Disable Al, and check the underlying surface itself to be sure no metals are present. Be sure to use the Tools Menu and select Enable Al before testing aluminum alloys.

5

General Metals Analysis

PMI Analysis

Field Procedures for PMI Using the Thermo Scientific Niton XL3 800 Series Alloy PMI Tool with Software Version 7.0 or Higher

Standard Operating Procedure October 2009

1. DOCUMENT PURPOSE

1.1. This document is intended for use as a guide for performing Positive Material Identification (PMI) using the handheld Thermo Scientific Niton XL3 800 Series x-ray fluorescence (XRF) alloy analyzer in the field to ensure compliance with the refinery procedures for ensuring Positive Material Identification.

2. SAFETY

- 2.1. Any persons, including refinery personnel and their representatives, using approved alloy analyzers at the refinery will be given a safety orientation by the PMI coordinator (or his /her approved instructor) prior to using any PMI device.
- 2.2. The safety orientations will include the following as a minimum:
- 2.2.1. Radiation safety for x-ray fluorescence analyzers covering safe operation of the device and emergency procedures in case of malfunction or unit damage during field use.
- 2.2.2. Personnel will demonstrate and acknowledge satisfactory understanding of safe operation by passing and signing a written test for each analyzer to be used. This documentation is to be kept on file by the PMI coordinator or his/her designee.
- 2.3. PMI coordinator will periodically audit field use of instruments for compliance with safety procedures.
- 3. PERSONNEL AUTHORIZED TO CONDUCT PMI INSPECTIONS
- 3.1. Refinery inspection personnel who have completed safety-training requirements.
- 3.2. Refinery's designated contractor representatives. These representatives are people who have demonstrated to the PMI coordinator or his/her designee that in addition to the safety requirements, they also have a firm understanding of the Niton® XL3 800 Series alloy analyzer operation, auto-calibration frequency requirements, and PMI job goals and objectives.
- 3.3. All authorized PMI personnel will be familiar with and have copies of the following documents available to them:
- 3.3.1. Thermo Scientific Niton XL3 800 Series User Guide

- 3.3.2. API Recommended Practice 578 Material Verification Program for New and Existing Alloy Piping Systems.
- 3.3.3. PMI Field Procedures (e.g., this document)
- 3.3.4. Other documentation as specified

4. PRE-INSPECTION Niton XL3 800 ANALYZER START UP AND CALIBRATION

- 4.1. Startup and Stabilization
- 4.1.1. Turn on, accept the radiation warning (if you are trained), and log into (enter password) the Niton XL3 800 Series analyzer. Allow 5 minutes for the analyzer to stabilize BEFORE proceeding.
- 4.1.2. Calibrate the analyzer by pressing the "System Check" icon. This will start the detector auto calibrate process. When the Niton XL3 instrument completes the auto calibration, press the "Sample Type" icon, press the "Metals" icon and then the "General Metals" icon. Measure the check sample as directed in 4.2.1. If it reads correctly, testing may start (see 4.5).
- 4.2. Verify the Calibration.
- 4.2.1. Test the factory supplied 1¼Cr-½Mo alloy standard (or other approved check sample). If the sample is correctly identified and all major elements (Cr and Mo primarily) read within calculated acceptance limits (see factory QC readings), testing may start (see 4.5).
- 4.2.2. If the check sample does not read correctly, then re-calibrate the analyzer by pressing the "System Check" icon. This will start the auto calibration. When the Niton XL3 instrument completes the auto calibration, return to "Test" and re-measure the check sample as directed in 4.2.1. If it reads correctly, testing may start (see 4.5). If the check sample still does not read correctly contact your local Thermo Scientific Niton Service center.
- 4.3. The need for a hot surface adapter should be determined at this time. If surface temperatures of the components to be tested will exceed 315°C/600°F, the operator must install the HotfootTM hot surface adapter. If sample temperatures will exceed 315°C/600°F but are below 800°F, the user may operate the unit with ~6 mm (~1/4 inch) gap between the front measuring plate and the sample. When testing hot surfaces with the weld adapter in place, the operator should double the measuring time to maintain precision (compensates for the lower signal due to the gap created by the weld adapter and the 3 mm (reduced size) aperture. Accuracy compensation is handled automatically by the Niton XL3 software.
- 4.4. If measurements will be taken on vibrating surfaces, hold the analyzer a few millimeters (~1/8 inch) away from the vibrating surface and double the measuring time to maintain precision. All concentration accuracy compensation (for the gap) is handled automatically by the Niton XL3 software (up to maximum of 6 mm).

4.5. Check the analyzer for material-specific performance prior to any actual testing. Typically, this should be done using a Certified Reference Material (CRM) or Reference Material (RM) such as an ARMI CRM. The CRM(s) used in this procedure should represent each of the materials expected to be encountered during inspection. A kit of 15 PMI refinery-specific CRM alloys is available upon request. Contact Thermo Scientific Niton Analyzers or your representative for a quote.

5. FIELD TESTING REQUIREMENTS

- 5.1. Prior to beginning testing, the specific PMI testing and documentation requirements for the job should be discussed with the PMI coordinator or his designee. Data storage can be accomplished in real time at the exact test site by utilizing optional data download button storage devices and position indicators that affix directly to the measured piece or location. Contact Thermo Scientific Niton Analyzers or your representative for a quote.
- 5.2. Good surface preparation is essential for obtaining accurate test results. All non representative material (e.g., paint, coating, scale) must be removed prior to testing. This can be accomplished by using hand files, grinders, pneumatic rotary files, etc. to obtain a clean metal area on which to conduct the test. Test areas on welds may have to be ground flat. Note: The battery powered Makita 9500D cordless angle grinder with several extra batteries has been found to provide good service with rapid surface preparation.
- 5.3. Test time for the Niton XL3 Series will be a minimum of 5 seconds for grade identification and 10 seconds for chemical analysis. If used in the Spectral Fingerprints mode, testing time may be reduced to 3-5 seconds for identification. Although the analyzer may display a result before the specified time interval, the operator should continue to measure until the specified time has elapsed. Note sections above and below covering special cases that require longer measuring times.
- 5.4. One test point for each piping component (pipe, tee, ell, etc.) is required. Weld test requirements vary according to size. See Section 6.
- 5.5. Minimum documentation for field work will include:
- 5.5.1. An isometric drawing or sketch of the as-installed and tested piping or equipment. This drawing should include equipment ID and piping line number, or other documentation reference(s) as specified.
- 5.5.2. Alloy chemistry documentation for the primary elements required to provide PMI of the alloy grade for all units tested.
- 5.5.3. Documentation must be signed and dated by the personnel that performed the tests; the signed documentation is then given to the PMI coordinator or designee.
- 5.5.4. The PMI coordinator or designee may require additional documentation, which will be specified prior to testing. Electronic copies of the encrypted Niton Data Transfer (NDT©) files should be maintained in addition to paper copies.

6. WELDS – NEW AND EXISTING INSPECTION

- 6.1. Prior to welding, consumables should be tested as indicated in 6.6 below.
- 6.2. Number of test locations per weld:
- 6.2.1. $\frac{3}{4}$ " to $1-\frac{1}{2}$ " butt welds One test point.
- 6.2.2. 34" to 2" socket welds One test point.
- 6.2.3. 2" to 4" butt welds Two test points.
- 6.2.4. 6" and larger weld At least four test areas evenly distributed around the circumference of the weld should be inspected. If no discrepancy is found, the average of the primary identifier element chemistries for all areas tested on that weld can be used for documentation.
- 6.3. At the discretion of the inspector, as in the case of large diameter welds or at any other time deemed necessary to ensure correctness of the weld material, additional locations on the weld may be tested.
- 6.4. When testing welds, care must be taken to position the analyzer window to read only the weld, with no false reading from the adjacent piping component.
- 6.4.1. If weld is smaller than the measuring aperture (smaller than 10 mm), insert the weld adapter and activate the Weld Mode with the Weld Icon. If your analyzer did not come with the optional weld adapter, there will not be a Weld Mode. Contact Thermo Scientific Niton Analyzers or your representative for a quote.
- 6.4.2. The weld adapter should reduce the measurement aperture width to a size smaller than the width of the weld to be tested, or
- 6.4.3. If the optional weld adapter is not an acquired accessory, it is possible to isolate the weld by masking adjacent metal with a non readable material, such as several layers of pure aluminum tape. Care should be taken that no readable elements are contained in the mask material and that the mask material is thick enough to prevent read through from the underlying material.
- 6.4.4. Double the measuring times when using the weld adapter (e.g., increase from 5 source seconds to 10 source seconds for ID, and from 10 to 20 seconds for chemistry), to compensate for the added gap and reduced surface area being tested.
- 6.5. Testing of welding consumables shall be performed as follows:
- 6.5.1. Each and every box of weld rod to be used for the job will be prepared by first puddling a sample using at least two passes and grinding smooth for testing.
- 6.5.2. Documentation for the weld rod tested should include:
- 6.5.2.1. Rod designation
- 6.5.2.2. Manufacturer's name

- 6.5.2.3. Box lot number
- 6.5.2.4. Element composition of samples
- 6.5.3. Rod box will be marked PMI CONFIRMED with:
- 6.5.3.1. Name of authorized person who performed the testing
- 6.5.3.2. Date tested
- 6.5.3.3. Other information as specified (e.g., vendor or contractor ID)
- 6.5.4. GTAW (TIG) wire can often be tested directly from the box either individually with a slot aperture or by using several pieces to cover more of the aperture window of the analyzer. If satisfactory results cannot be obtained, then puddling and grinding of the wire should be performed as specified in 6.5.1.

7. UNDERSIZED OR NON-CONTACT SAMPLES (samples that do not make contact with or that do not fully cover the measurement aperture)

- 7.1. For samples that do not fully cover the measurement aperture, increase the testing time by increasing the time in inverse proportion to the decrease in percentage of aperture covered. For example: a rod only covers ½ of the aperture, so increase the measurement time by two. (e.g., from 5 seconds to 10 seconds for ID and from 10 to 20 seconds for chemistry).
- 7.1.1. Undersized samples should be placed in the Thermo Scientific portable alloy test stand (optional), which is shielded to prevent radiation exposure to the operator. (NOTE: An undersized sample one that does not cover the entire measuring aperture measured while lying on another material such as, e.g., a stainless steel table top, will be affected by the signal coming from the table top itself.) Be sure to employ the Disable Al feature if measuring on an aluminum table or any plastic or wood table. Use "Tools," then select "Disable Al." Be sure to use Tools to "Enable Al" before testing aluminum alloys.
- 7.2. Non-contact sample measurements include those measured with a gap of up to a maximum of ½ inch (~6 mm) from the Niton XL3 measurement aperture opening. Such measurements require double the exposure time. (e.g., from 5 seconds to 10 seconds for ID and from 10 to 20 seconds for chemistry).
- 7.3. Inside pipe measurements on pipes that are less than a few feet in diameter will result in non-contact measurements. Follow recommendations in 7.2 above.

8. INSTRUMENT QC

8.1. Measure the supplied $1\frac{1}{4}$ Cr $-\frac{1}{2}$ Mo calibration check sample AT LEAST once for every 2 hours of use time. If correct, continue work. If incorrect, recalibrate the Niton XL3 detector and re-take the past 2 hours of results.

9. STAMPS

- 9.1. All stamping is done with a specified proprietary stamp.
- 9.2. All stamps purchased for special marking should be marked with a control number. A list indicating the number and location of these stamps is held by the PMI coordinator.
- 9.3. Typically, a stamp is assigned to each individual analyzer and stays in the respective analyzer's field carry bag.
- 9.4. Report any loss of a stamp immediately to the PMI coordinator or designee.

10. WHEN TO USE AN APPROVED STAMP

- 10.1. Materials and components should be tested prior to fabrication. This will help find any discrepancies before final field inspection and prevent costly field cut outs.
- 10.2. Items checked prior to fabrication and before the final as-installed condition will only be marked with approved paint pen as "PMI CONFIRMED."
- 10.3. Chemistry documentation may not be necessary for this phase of the PMI inspection.
- 10.4. The number of components and type tested should be documented for the job.
- 10.5. Discrepancies should be noted and handled as specified in the appropriate procedures document.
- 10.6. Shop made spool pieces may be stamped, providing that their final field location in the piping system can be tracked and documented. Final field installation must be confirmed by visual inspection for the stamp.
- 10.7. Use of any approval stamp should be in accordance with the appropriate company procedures document for PMI on existing equipment.
- 10.8. Use of an approval stamp does not negate the need for the written documentation.

11. RESOLUTION OF DISCREPANCIES

- 11.1. When analysis indicates a wrong material, component, or weld, then take the following steps:
- 11.2. Test several other areas on the same component. For welds, check adjacent components.
- 11.3. Check the analyzer calibration against the check sample or other known sample.
- 11.4. If performing pre-fabrication testing, components having reached this point must be rejected according to established procedures.
- 11.5. Discrepancies on field installed or in-place existing materials requires notifying the responsible inspector and completing the following items:

- 11.5.1. Complete chemistries should be taken for all elements present in the suspect material to help identify the material.
- 11.5.2. Review the test data for the suspect component, and, if possible, confirm the results using a second analyzer.
- 11.5.3. If conclusive results cannot be obtained with field analyzers, the inspector may request a lab analysis on a small sample material removed from the component.

5

PMI Analysis

FAC Analysis

Field Procedures for Measurement of FAC Predictor Elements Cr, Cu, Mo Using the Thermo Scientific Niton XL3 800 Series Alloy Analyzer with Software Version 7.0 or Higher

1. DOCUMENT PURPOSE

1.1. This document is intended for use as a guide for using the handheld Thermo Scientific Niton XL3 800 Series x-ray fluorescence (XRF) alloy analyzer as a tool in performing Flow Accelerated Corrosion (FAC) data modeling to ensure that proper measurement parameters are followed to reach indicative low-level concentrations of Cr, Cu, and Mo.

2. SAFETY

- 2.1. Any persons, including power plant personnel and their representatives, using approved alloy analyzers at the plant will be given a safety orientation by the FAC coordinator (or his/her approved instructor) prior to using any XRF inspection device.
- 2.2. The safety orientations will include the following as a minimum:
- 2.2.1. Radiation safety for x-ray fluorescence analyzers covering safe operation of the device as well as emergency procedures in case of malfunction or unit damage during field use.
- 2.2.2. Personnel will demonstrate and acknowledge satisfactory understanding of safe operation by passing and signing a written test for each analyzer to be used. This documentation is to be kept on file by the FAC coordinator or his/her designee.
- 2.3. FAC coordinator will periodically audit field use of instruments for compliance with safety procedures.

3. PERSONNEL AUTHORIZED TO CONDUCT MEASUREMENT OF FAC PREDICTOR ELEMENTS

- 3.1. Power plant inspection personnel who have completed safety-training requirements.
- 3.2. Power plant's designated contractor representatives. These representatives are people who have demonstrated to the FAC coordinator or his/her designee that in addition to the safety requirements, they also have a firm understanding of the Niton® XL3 800 Series alloy analyzer operation, auto-calibration frequency requirements, and FAC program goals and objectives.
- 3.3. All authorized inspection personnel will be familiar with and have copies of the following documents available to them:
- 3.3.1. Thermo Scientific Niton XL3 800 Series User Guide

- 3.3.2 Thermo Scientific Niton NDT Data Transfer User's Guide
- 3.3.3 Flow Accelerated Corrosion in Power Plants, EPRI Publication, 1996
- 3.3.4. Other documentation as specified

4. PRE-INSPECTION Niton XL3 800 ANALYZER START UP AND CALIBRATION

- 4.1. Startup and Stabilization
- 4.1.1. Turn on, accept the radiation warning (if you are trained), and log into (enter password) the Niton XL3 800 Series analyzer (see Figures 1 & 2).



Figure 73. Warning Screen

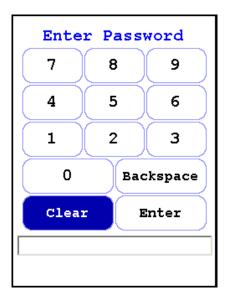


Figure 74. Virtual Numeric Keypad for Log On

4.1.2. Ensure that the filters are properly set up for the measurement: From "Main Menu" select "Advanced" -> "Element Range" (see Figure 1-3). In the drop-down box select "General Metals". Ensure that the boxes are checked for "Main Range" and "Low Range". Ensure that the "Time" boxes show "10.0" in both the "Main Range" and "Low Range" boxes (see Figure 1-4). If not, -> "Main Range Time" -> "C" to clear existing entry -> "1", "0", "E" to enter the desired time of 10 seconds. Repeat for the "Low Range Time" if so required (see Figure 1-5). Ensure that the "Filter Change on Time Only" is checked. Then press "Save".

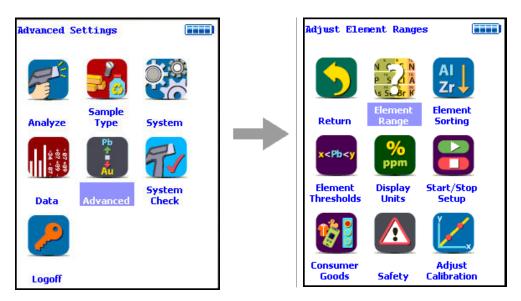


Figure 75. The Element Range Menu Path

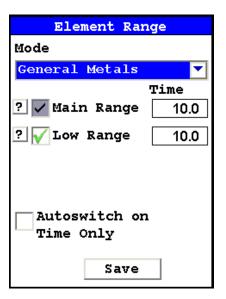


Figure 76. Range Checkboxes, Range Time, and Time Only Checkbox

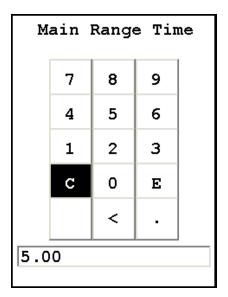


Figure 77. Range Time Editor

4.1.3. Set up the screen display so that Cr, Cu, and Mo are always displayed, and are shown prominently at the top of the displayed element list: From "Main Menu" select "Advanced" -> "Element Sorting" (see Figure 1-6). In the drop down box select "Alloy Mode" (see Figure 7); in the element box scroll down and highlight Cr; in the "Display Options" box select "Always Display"; then using the blue arrow keys at the right of the screen use the up arrow to move Cr to the top of the element table; repeat this procedure for Cu and Mo respectively, first marking them as "Always", then moving them up the element table to immediately below Cr. Press "Save" -> "Close" -> "Return" -> "Return" to reach the Main Menu. See Figure 1-8.

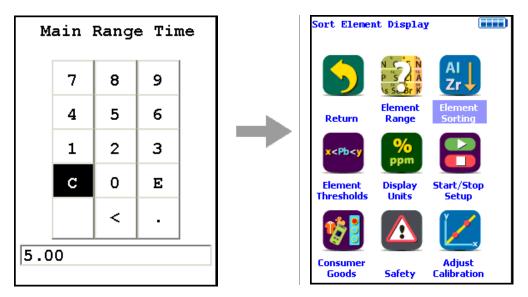


Figure 78. Sort Element Display Menu Path

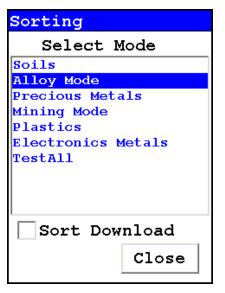


Figure 79. Sort Element Display

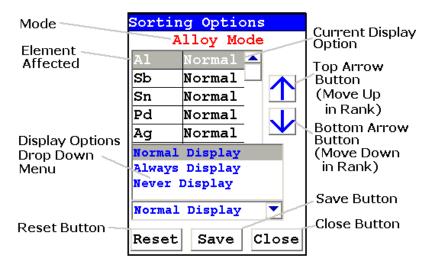


Figure 80. Sorting Options Screen

4.1.4. Calibrate the analyzer by pressing the "System Check" icon, (see Figure 9). This will start the detector auto-calibrate function. When the Niton XL3 instrument completes the auto-calibration, return to "Test" and measure the check sample as directed in 4.2.1. If it reads correctly, testing may start (see 5.0).

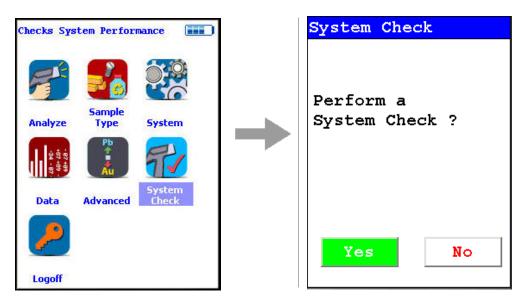


Figure 81. System Check Menu Path

4.2. Verify the Calibration.

- 4.2.1. Test the factory supplied 1¼Cr-½Mo alloy standard (or other approved check sample). If the sample is correctly identified and all major elements (Cr and Mo primarily) read within calculated acceptance limits (within the low and high values of factory readings found on QC sheet), testing may start (see 5.1).
- 4.2.2. If the check sample does not read correctly, then re-calibrate the analyzer by pressing the "System Check" icon (see Figure 9). This will start the auto-calibration. When the Niton XL3 instrument completes the auto-calibration, return to "Test" and re-measure the check sample as directed in 4.2.1. If it reads correctly, testing may start (see 5.0). If the check sample still does not read correctly, contact your local Thermo Scientific Niton service center.
- 4.3. If a weld of less than 10 mm is to be analyzed, it will be necessary to reduce the analysis area from the 10 mm normal aperture size to a size below that of the weld bead. See section 6.4.1.
- 4.4. Check the analyzer for sensitivity performance for Cr, Cu, and Mo prior to any actual testing. Typically, this should be done using a Certified Reference Material (CRM) such as an ARMI CRM. The CRM(s) used in this procedure should represent each of the materials expected to be encountered during inspection.

5. FIELD TESTING REQUIREMENTS

- 5.1. Prior to beginning testing, the specific testing and documentation requirements for the job should be discussed with the FAC coordinator or his designee. Data storage can be accomplished in real time at the exact test site by utilizing optional data download button storage devices and position indicators (compact memory button CMB) that affix directly to the measured piece or location. Contact Thermo Scientific Niton Analyzers or your local representative for a quote.
- 5.2. Good surface preparation is essential for obtaining accurate test results. All non-representative material (e.g., paint, coating, scale) must be removed prior to testing. An approximately 2-inch-square section of surface should be cleaned down to fresh or "white" metal using an abrasive flapper wheel. The cleaning should not be performed using the same wheel that was used to prepare the component for ultrasonic testing (UT), but should be performed using a fresh wheel, designated for this operation only. Wheels that have been used on high chromium alloyed components (e.g., 300 series chromium-nickel stainless steels, or chromium steels) should not be used to clean carbon steel components. The area to be sampled should be wiped clean or blown with air to remove any grinding dust before testing.
- 5.3. Test time for the Niton XL3 Series will be a minimum of 10 seconds on each filter for accurate chemistry analysis. Since two filters (Main and Low) are required for the targeted low concentrations, the total test time shall be a minimum of 20 seconds. Although the analyzer may display a result before the specified time interval, the operator should continue to measure until the specified time has elapsed. Note sections above and below covering special cases that require longer measuring times.
- 5.4. One test point for each piping component (pipe, tee, ell, etc.) is required. Weld test requirements vary according to size. See Section 6.

- 5.5. Minimum documentation for field work will include:
- 5.5.1. An isometric drawing or sketch of the as-installed and tested piping or equipment. This drawing should include equipment ID and piping line number, or other documentation reference(s) as specified.
- 5.5.2 Documentation must be signed and dated by the personnel that performed the tests; the signed documentation is then given to the FAC coordinator or designee.
- 5.5.4. The FAC coordinator or designee may require additional documentation, which will be specified prior to testing. Electronic copies of the encrypted Niton Data Transfer (NDT©) files should be maintained in addition to paper copies.

6. WELDS – NEW AND EXISTING INSPECTION

- 6.1. Prior to welding, consumables should be tested as indicated in 6.5 below.
- 6.2. Number of test locations per weld:
- 6.2.1. $\frac{3}{4}$ " to $1-\frac{1}{2}$ " butt welds one test point.
- 6.2.2. 34" to 2" socket welds one test point.
- 6.2.3. 2" to 4" butt welds two test points.
- 6.2.4. 6" and larger weld At least four test areas that are evenly distributed around the circumference of the weld should be inspected. If no discrepancy is found, the average of the primary identifier element chemistries for all areas tested on that weld can be used for documentation.
- 6.3. At the discretion of the inspector, as in the case of large diameter welds, or at any other time deemed necessary to ensure correctness of the weld material, additional locations on the weld may be tested.
- 6.4. When testing welds, care must be taken to position the analyzer window to read only the weld, with no false reading from the adjacent piping component.
- 6.4.1. If weld is smaller than the measuring aperture (smaller than 10 mm), insert the weld adapter and activate the Weld Mode with the Weld Icon. If your analyzer did not come with the optional weld adapter, there will not be a Weld Mode. Contact Thermo Scientific Niton Analyzers or your local representative for a quote.
- 6.4.2. The weld adapter should reduce the measurement aperture width to a size smaller than the width of the weld to be tested, or
- 6.4.2. If the optional weld adapter is not an acquired accessory, it is possible to isolate the weld by masking adjacent metal with a non-readable material, such as several layers of pure aluminum tape. Care should be taken that no readable elements are contained in the mask material and that the mask material is thick enough to prevent read through from the underlying material.

- 6.4.4. Double the measuring times when using the weld adapter (e.g., increase from 10 to 20 seconds per filter for chemistry) to compensate for the added gap and reduced surface area being tested.
- 6.5. Testing of welding consumables shall be performed as follows:
- 6.5. Testing of welding consumables shall be performed as follows:
- 6.5.1. Each and every box of weld rod to be used for the job will be prepared by first puddling a sample using at least two passes and then grinding smooth for testing.
- 6.5.2. Documentation for the weld rod tested should include:
- 6.5.2.1. Rod designation
- 6.5.2.2. Manufacturer's name
- 6.5.2.3. Box lot number
- 6.5.2.4. Element composition of samples
- 6.5.3. Rod box will be marked FAC CONFIRMED with:
- 6.5.3.1. Name of authorized person who performed the testing
- 6.5.3.2. Date tested
- 6.5.3.3. Other information as specified (e.g., vendor or contractor ID)
- 6.5.4. GTAW (TIG) wire can often be tested directly from the box either individually with a slot aperture or by using several pieces to cover more of the aperture window of the analyzer. If satisfactory results cannot be obtained, then puddling and grinding of the wire should be performed as specified in 6.6.1.

7. UNDERSIZED OR NON-CONTACT SAMPLES (samples that do not make contact with or that do not fully cover the measurement aperture)

- 7.1. For samples that do not fully cover the measurement aperture, increase the testing time by increasing the time in inverse proportion to the decrease in percentage of aperture covered. For example: a rod only covers ½ of the aperture, so increase the measurement time by two (e.g., from 10 to 20 seconds per filter for chemistry).
- 7.1.1. Undersized samples should be placed in a Thermo Scientific Niton approved alloy test stand (optional), which is shielded to prevent radiation exposure to the operator. (NOTE: An undersized sample one that does not cover the entire measuring aperture measured while lying on another material, such as a stainless steel table top, will be affected by the signal coming from the table top itself.) Be sure to employ the Disable Al feature if measuring on an aluminum table or any plastic or wood table. Use "Tools," then select "Disable Al" Be sure to use Tools to "Enable Al" before testing aluminum alloys.

7.2. Inside pipe measurements on pipes that are less than a few feet in diameter will result in non-contact measurements. Follow recommendations in 7.2 above.

8. INSTRUMENT QC

8.1. Measure the supplied $1\frac{1}{4}$ Cr $-\frac{1}{2}$ Mo calibration check sample AT LEAST once for every 2 hours of use time. If correct, continue work. If incorrect, recalibrate the Niton XL3 detector and re-take the past 2 hours of results.

9. RESOLUTION OF DISCREPANCIES

- 9.1. When analysis indicates an undesirable or anomalous result, take the following steps:
- 9.2. Test several other areas on the same component. For welds, check adjacent components.
- 9.3. Check the analyzer calibration against the check sample or other known sample.
- 9.4. If performing pre-fabrication testing, components having reached this point must be rejected according to established procedures.
- 9.5. Discrepancies on field-installed or in-place existing materials require notification of the responsible inspector and completing the following items:
- 9.5.1. Review the test data for the suspect component, and, if possible, confirm the results using a second analyzer.

Coatings Analysis

Coating Thickness Measurement Using the Thermo Scientific Niton XL3 800 Series

X-Ray Fluorescence (XRF) Analyzer – Version 7.0 and Higher

Standard Operating Procedure

October 2009

Note Each user should read the Thermo Scientific Niton XL3 User's Guide carefully before initiating measurements with the system. Users are strongly urged to attend the Thermo Scientific Niton XRF Analyzer Radiation Safety and Operations Training courses offered regularly, or the web-based trainings. For more information, visit www.thermo.com/niton.

Preparatory Tasks

- 1. Attach a charged battery to the analyzer and turn it on. Follow the screen instructions and Log On as the operator using either the default password or a custom one as designated by the user in an NDU file.
- 2. Wait five (5) minutes before using the analyzer, allowing the instrument electronics to stabilize.
- 3. Verify that the date is set properly for data tracking purposes.

From the Main Menu, select the System icon, then the Specs icon. The date will be displayed for verification. If the date is incorrect, correct it prior to proceeding. This can be done by returning to the System Menu and selecting the Date & Time icon. See Setting the Date and Time for detailed information on this procedure.

- 4. (Optional) Connect the analyzer to a computer via the included serial cable, USB cable, or Bluetooth™ wireless module. See Connecting the XRF Analyzer to Your PC for more information.
- 5. During analysis and detector calibrations, it is important to ensure that the analyzer is not exposed to strong electromagnetic fields, including those produced by computer monitors, hard drives, cellular telephones, walkie talkies, etc. Keep a minimum two (2) feet (0.7 meters) distance between the analyzer and electronic devices.
- 6. From the Main Menu, select the System Check icon, and select the Yes Button. (Figure 1.)
- 6.1. System Check calibrates the detector and verifies it is operating to specifications. After starting the process, no further user interaction is required during this operation. When the instrument is finished performing the check, the unit will show either "System OK" or one of the failure errors.

6.2. If the unit shows a failure error, then perform a second System Check by selecting the Recheck Button. If the unit still does not show a "System OK," please contact Thermo Scientific Niton Analyzers toll-free at (800) 875-1578, +1 978 670-7460, niton@thermofisher.com, or contact your local Niton Analyzers representative for assistance.

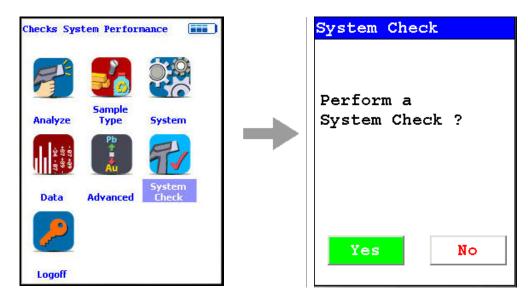


Figure 82. System Check Menu Path

7. Verify and/or change filter position time.

Thermo Scientific Niton XL3 analyzers are equipped with 2 excitation filters that optimize the analyzers' sensitivity for various elements. The "Main Range" filter provides optimum sensitivity for the elements manganese (Mn) through bismuth (Bi). The "Low Range" filter is used to optimize the sensitivity for the elements from titanium (Ti) through chromium (Cr). Note that the main range filter can be used to analyze Ti, V and Cr, but the sensitivity is not as good as when using the low filter. The amount of time that the analyzer spends in each filter position is user definable. Please note that the analyzer will continue alternating excitation filters until the user selectable maximum analysis time is reached or the operator terminates the measurement. To verify or change the length of time each filter will be active, do the following:

7.1 From the Main Menu select the Advanced icon, then the Element Range icon (Figure 1-2)

In the drop-down menu, select the mode that is intended for use (i.e., General Metals, Electronics Metals).

- 7.2 Select the box next to Main Range and Low Range so that a check mark appears in each box.
- 7.3 Enter the desired length of time for each filter. For example, set values of 5 seconds for the Main Range and 5 seconds for the Low Range as shown in Figure 1-3.
- 7.4 Select the Save Button and return to the Main Menu by selecting the Return icon.

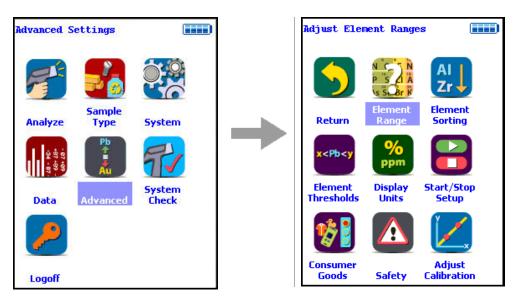


Figure 83. Element Range Menu Path

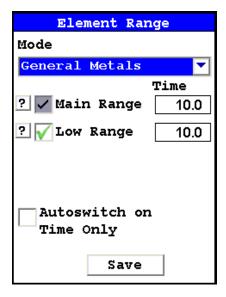


Figure 84. Setting Element Ranges

- 8. Verify instrument measurement accuracy using the 1½Cr ½Mo certified reference material (CRM) supplied with the analyzer.
- 8.1 Test the factory-supplied 1¼Cr-½Mo alloy standard (or other approved check sample) based on a 20s measurement using main range filter only (see Figure 3). If the sample is correctly identified and all major elements read within calculated acceptance limits (within 3 sigma of the average value of factory readings, proceed to General Testing Protocol section.

8.2 If the analyzer reports values outside the acceptance tolerance ranges specified in the tables, repeat the System Check described in step 7, then repeat the reference sample analysis in step 8.1.

8.3 If the analyzer again fails to meet the acceptance tolerance ranges specified in the tables, please contact Thermo Scientific Niton Analyzers or your local representative for assistance.

Coatings Entries and Standardization

How to Access Metal Coatings Mode

To access Metal Coatings mode follow the sequence shown in Figure 4.



Figure 85. Metal Analysis Menu Path

If no coatings method entries have been loaded, screen A in Figure 1-5. will appear. Press the Add Button, which brings up a Virtual Keyboard, and enter a method name, which will bring you to screen B. Follow the steps in Creating a Metal Coatings Entry to create a new entry.

If method entries have been loaded, screen C will appear. Select desired method from drop down menu. If you need to add more methods, select the Edit Methods Button to open screen A.

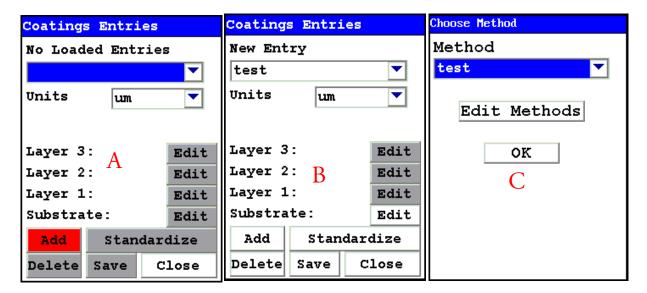


Figure 86. Screens A, B, and C

Creating a Metal Coatings Entry

Before coating thickness can be measured, a Coatings System Entry must be made defining the coatings system (see Figures 7-11). Note that the Coatings Method Menu can be accessed via the steps outlined in Figures 1-4 and 1-5 above or via the Tools Menu. See Using the Tools Menu for more information on the Tools Menu.

6 Coatings Analysis

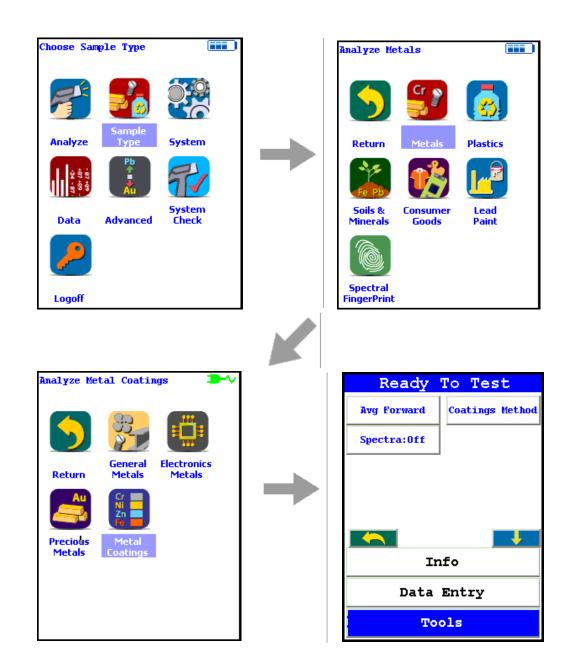


Figure 87. Accessing the Tools Menu - Select Coatings Method Button

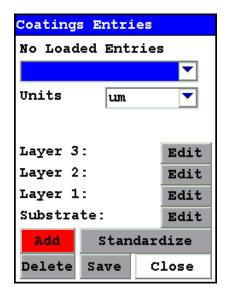
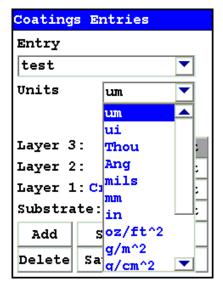
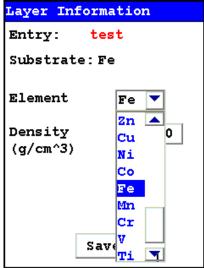


Figure 88. Coating System Screen

When creating a coating system, select the Add Button to activate the Virtual Keyboard and enter the name of a coating system (e.g. test). Once the name is created, select the coatings thickness units, substrate (type of metal to be coated) and layers (metal coatings) from the individual pull-down menus. See Figure 7.





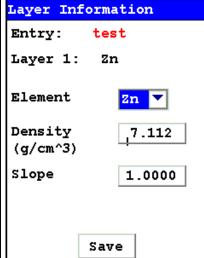


Figure 89. Entering Units, Substrate, and Layer Information

Standardize Feature

The Standardize feature is a standardization routine that corrects for any bias in thickness measurement when compared to a known thickness sample or thickness standard.

To standardize, follow the sequence shown in Figure 1-8. From the Coating Entries screen select the Standardize Button. Enter known thickness of Zn layer, e.g. 20.0, and initiate a reading. Select the Yes Button to save the standardization.

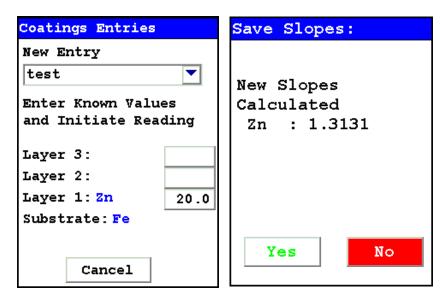


Figure 90. Standardizing the Coating

Coatings Testing Protocol

Once a methods are created and standardized the analyzer is ready for routine analysis. All non-representative material (e.g. dust, oil) must be removed prior to testing. Test times of 10 s will generally provide accurate results

Figure 9. shows a typical measurement of Zn thickness over steel.

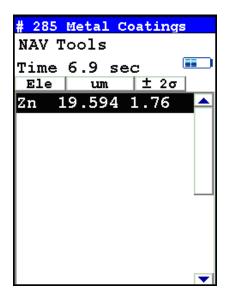


Figure 91. Example Coating Measurement

There is a maximum coating thickness that can be measured. This maximum value, know as "infinite thickness", varies with coating element. If a coating above this value is measured it will give the message Saturated as shown in Figure 1-10.

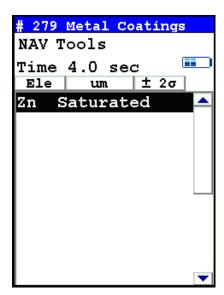


Figure 92. Example Saturated Measurement

Typical infinite thickness values are given in Table 1.

Table 1: Coating Infinite Thickness

Element	Substrate	Microns
Au	Cu	10
Cd	Al	75
Cd	Fe	75
Со	Fe	50
Со	Ti	50
Cr	Fe	15
Cu	Fe	20
Mo	Fe	50
Mo	Ti	50
Ni	Fe	20
Pt	Cu	15
Sn	Fe	100
Ti	Fe	50
Zn	Fe	50
Zr	Al	70

Mining Analysis

Niton XL3 X-Ray Fluorescence (XRF) Analyzer — Version 7.0 and Higher

Analyzing Grade and Process Control Mining Samples Using the Thermo Scientific Niton® XL3 Series X-Ray Fluorescence (XRF) Analyzer with Software Version 7.0 and Higher

Standard Operating Procedure

Note Each user should read the Thermo Scientific Niton XL3 Series User's Guide carefully before initiating measurements with the system. Users are strongly urged to attend the Thermo Scientific Niton XRF Analyzer Radiation Safety and Operations Training courses offered regularly (US only), or contact their local representative for product and safety training. For more information, visit www.niton.com.

PREPARATORY TASKS

- 1. Insert a charged battery into the analyzer and turn it on. See Battery Installation and Charging for detailed instructions. Follow the screen instructions and "Log On" as the operator using either the default password or one designated by the user in an NDU file.
- 2. Wait five (5) minutes before using the analyzer, allowing the instrument electronics to stabilize.
- 3. Verify that the date is set properly for data tracking purposes. From the Main Menu, select the System icon then the Specs icon. The date will be displayed for verification. If the date is incorrect, correct it prior to proceeding. This can be done by "Closing" out of the Specs screen and selecting the Date & Time icon from the System Menu. See Setting the Date and Time for detailed instructions. Select the Return Button to go back to the Main Menu.
- 4. (Optional) Connect the analyzer to a computer via the included serial cable, USB cable, or Bluetooth™ wireless module. See Connecting the XRF Analyzer to Your PC for more information.
- 5. During analysis and System Check, it is important to ensure that the analyzer is not exposed to strong electromagnetic fields, including those produced by computer monitors, hard drives, cellular telephones, walkie talkies, etc. Keep a minimum two (2) feet (0.7 meters) distance between the analyzer and electronic devices.
- 6. From the Main Menu, select the System Check icon, and select the Yes Button. (Figure 1.)

- 6.1. System Check calibrates the detector and verifies it is operating to specifications. After starting the process, no further user interaction is required during this operation. When the instrument is finished performing the check, the unit will show either "System OK" or one of the failure errors.
- 6.2. If the unit shows a failure error, then perform a second System Check by selecting the Recheck Button. If the unit still does not show a "System OK," please contact Thermo Scientific Niton Analyzers toll-free at (800) 875-1578, +1 978 670-7460, niton@thermofisher.com, or contact your local Niton Analyzers representative for assistance.

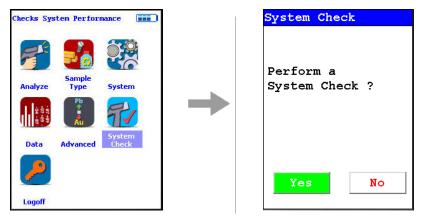


Figure 93. System Check Menu Path

7. Verify and/or change filter position time.

Thermo Scientific Niton XL3 analyzers are equipped with excitation filters that optimize the analyzers' sensitivity for various elements. The "High Range" filter is used to optimize barium (Ba) through silver (Ag). The "Main Range" filter provides optimum sensitivity for the elements manganese (Mn) through bismuth (Bi). The "Low Range" filter is used to optimize the sensitivity for the elements from titanium (Ti) through chromium (Cr). Note that the main range filter can be used to analyze Ti, V and Cr, but the sensitivity is not as good as when using the low filter. The "Light Range" filter is available only with He-purged and GOLDD technology analyzers, and is typically used in light element analysis. The amount of time that the analyzer spends in each filter position is user definable. Please note that the analyzer will continue alternating excitation filters until the user selectable maximum analysis time is reached or the operator terminates the measurement. To verify or change the length of time each filter will be active, do the following:

7.1 From the Main Menu select the Advanced icon, then the Element Range icon (Figure 1-2)

In the drop-down menu, select the mode that is intended for use (i.e., General Metals, Electronics Metals).

7.2 Select the box next to Main Range, High Range, and Low Range so that a check mark appears in each box.

- 7.3 Enter the desired length of time for each filter. For example, set values of 30 seconds for the Main Range, 30 seconds for the Low Range, and 30 seconds for the High Range as shown in Figure 1-3.
- 7.4 Select the Save Button and return to the Main Menu by selecting the Return icon.

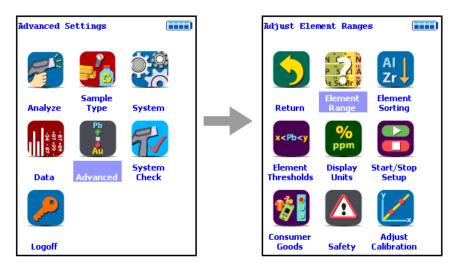


Figure 94. Element Range Menu Path

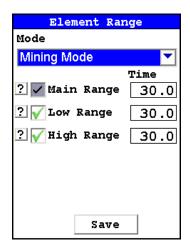


Figure 95. Setting Element Ranges

8. Verify instrument measurement accuracy. Depending on the configuration of your instrument, you will have been supplied with a set of multi-element reference samples. These samples are use to verify that the instrument is performing properly. Use CCRMP TILL-4 (180-601) to verify the instrument performance in Mining Mode and Soil Mode. The nominal element concentrations and acceptable ranges of results for each reference sample are shown below in Table 1-1

8.1 Mining

From the Main Menu, select the Sample Type icon, then the Soils & Minerals icon, then the Mining Cu/Zn icon (Figure 1-4) or Mining Ta/Hf icon (Figure 1-5) as appropriate. You can also use this procedure with TestAll Geo Mode. See GENERAL TESTING PROTOCOL below for more information.



Figure 96. Mining Cu/Zn Analysis Menu Path

-OR-



Figure 97. Mining Ta/Hf Analysis Menu Path

- 8.1.1. Place the reference sample CCRMP TILL-4 (180-601) in the test stand for analysis, or set the reference sample on a clean surface. The unlabeled side should be presented to the spectrometer. Place the front end of the instrument against the sample with the window centered on the sample.
- 8.1.2. Take a 90-second measurements on this reference sample (30 sec. with High Range, 30 sec. with Main Range, and 30 sec. with Low Range). Prior to measurement, ensure that the analyzer has the maximum test time set to 90 seconds or greater. From the Main Menu select the Advanced icon, then the Start/Stop Setup icon. Edit the Max. Time dialog box to display the proper setting. Calibration factors must be set to factory values. See Start/Stop Setup for more information on the Start/Stop Menu, and Adjusting the Calibration for setting Calibration factors.
- 8.1.3. Compare the result with the acceptable ranges listed in Table 1-1; Fe and K for Mining mode and Cu ,Ti for Soil mode. The result should fall within these specified values.

Table 1-1 Nominal Concentrations and Acceptable Ranges

Element - Mode	Certified Value	Minimum Acceptable*	Maximum Acceptable*
% Fe - Mining	3.97 %	3.50 % or 35000 ppm	5.0 % or 50000 ppm
% K - Mining	2.68 %	2.3 % or 23000 ppm	3.5 % or 35000 ppm
ppm Cu - Soil	237 ppm	0.02% or 200 ppm	0.028% or 280 ppm
ppm Ti - Soil	4840 ppm	0.41% or 4100 ppm	0.52% or 5200 ppm

^{*} Depending on whether % or ppm is selected for reporting concentration.

- 8.1.4 If the analyzer reports values within the acceptable ranges of the reference values, it is ready for measurement of unknown samples. Proceed to GENERAL TESTING PROTOCOL section.
- 8.1.5 If the analyzer reports values outside the acceptance tolerance ranges specified in Table 1-1, repeat the System Check described in step 5, then repeat the reference sample analysis in step 8.1
- 8.1.6 If the analyzer again fails to meet the acceptance tolerance ranges specified in Table 1-1, please contact Thermo Scientific Niton Analyzers or your local representative for assistance.

GENERAL TESTING PROTOCOL

Mining Grade and Production Control samples require analytical capabilities for a range of elements and concentrations, as well as sample type. Samples may come out of production fully prepared as fine aggregates or powders, or in a less prepared state such as slurry. Similarly, they may come out of the mine pit as a whole rock hand sample or in an advanced state of preparation as blast chips. Furthermore, measurements may be required in situ on a mine wall. Consider the data quality requirements when determining the number of samples and the amount of preparation to undertake. When examining the data, consider the state of the samples in deciding the number of samples and readings to average.

The proper mode must be utilized obtain useful analytical results. For mining applications there are three choices: Mining (divided into Mining Cu/Zn and Mining Ta/Hf) and Soil. Mining Mode is used for measuring concentrations of all elements that are 0.5% (5000 ppm) or greater, or high concentration samples. Soil Mode is best for low concentration elements at 0.5% (5000 ppm) or less. For samples containing high concentrations of elements other than those of primary interest, it is best to use Mining Mode for analysis. Mining Mode is a fundamental parameters (FP) calibration and will do the best job for all elements, even those at lower concentrations, as long as there are high concentrations of any elements present in the sample.

There are direct overlaps between the spectral peaks for copper (Cu) and tantalum (Ta) and zinc (Zn) and hafnium (Hf). Handheld XRF technology cannot distinguish between these sets of elements. Cu and Zn are associated with each other as are Ta and Hf. Therefore, if the samples contain Cu or Zn, choose Mining Cu/Zn; if the samples contain Ta or Hf, choose Mining Ta/Hf mode.

Representative Sample

Homogeneity of samples is extremely important when considering whether or not to average readings. Non-homogenous samples contain different concentrations of elements at different points across the sample. Averaging a few or many measurements is often the best way to determine whole rock or area concentrations. This averaging gives the user a more complete information set over a specific area than would a single measurement. The other option with inhomogeneous samples is to fully prepare them, which then homogenizes the sample in the process.

To ensure robustness of the data, two criteria must be met when samples are presented to the analyzer. The first is that the sample must cover the analysis window. The window is located on the nose of the analyzer, and it is the point at which the x-ray beam is emitted from the analyzer and fluoresced x-rays are collected by the detector. It must be as close to the sample as possible for the entire measurement time, contact with the sample is required. The second condition is that the sample needs to be "infinitely thick." Infinite thickness refers to having enough sample depth so that even if the depth of the sample were increased, no additional fluoresced x-rays would be produced and reach the detector.

Testing time affects detection limit and precision and is set by the user. For best results, each required filter should be activated for a minimum of 30 seconds. For analysis of mining samples, it is not unusual for measurement to take 60 seconds or more per filter. If concentrations of the elements of interest are high enough, and the range of acceptable values broad enough, shorter measurement times may be sufficient to achieve the desired results. For example, if it is necessary to determine whether a sample contains more or less than 1000 ppm of lead (Pb), 30 seconds on the Main Filter may be sufficient sampling time. In contrast, if the question is whether or not a sample contains 100 ppm of copper, suggested testing time is for a minimum of 60 seconds on the Main Filter. If the error is too great, increasing the testing time by a factor of four will decrease the error by 50%.

Sample Preparation

In situ samples are analyzed as is, on site, and with no sample preparation performed. Sample preparation can increase accuracy of data when compared to other analytical techniques. A fully prepared sample is one that is dried, crushed, ground, sieved, and loaded into a sample cup. Any intermediate step in the preparation process may be achieved and determined acceptable by the user.

Using He-purged and/or GOLDD analysis will both expand your element range (down to Mg) and give extremely low LODs, but can have other consequences. At these low Z elements and levels, contamination of the sample is a factor, and fingerprints, dust, oil, and other chance contaminants can give a false reading. Any samples, even in-situ, must be as clean as possible, given the nature of the sample and the circumstances of the reading. Condition of the sample should be noted if possible.

32mm Sample Cups first must be assembled if they are going to be used as part of the sample preparation process. They are assembled by placing a Mylar Film 2.5" dia. 6m thick on top of the sample cup collar. Then the sample cup cylinder is pressed into the collar so that the film is pulled tightly between the collar and cylinder, creating an enclosure. Next, the sample cup is turned over with the film down so that sample material can be placed and tamped into the cup. Place a Filter Paper 2.4cm dia. on top of the material, when the material reaches the line close to the top of the cup. Then place Polystuffing on top of the filter so that when the sample is inverted, the material is held firmly against the Mylar film. Last, place the sample cap on top of the batting and, if desired, affix a label to the cap.

There is a Portable Test Stand available for fixed bench or portable location (such as the tailgate of a truck) use.

Grade Control Samples

In-situ Measurements

In-situ Measurements – Measurements taken directly on the wall or floor of the mine are in situ. This is considered the most basic type of measurement because there is no sample preparation performed.

7 Mining Analysis Blast Hole Samples

- 1. Determine whether to average readings over a pre-determined area or to just take one spot measurement. See Avg Forward to set the averaging function for the analyzer.
- 2. Set the appropriate filters and reading times. See Adjusting the Element Range to set and save the filter settings.
- 3. If desired, set beeps to activate at the end of the predetermined measurement time. See Setting up Audio Cues for instructions to set beep times.
- 4. Grasp the analyzer by the handle, ensuring the wrist strap is properly placed on the wrist.
- 5. Position the analyzer on the desired analysis spot, ensuring that the nose of the analyzer is making contact with the rock face, and then initiate a reading. Continue the reading for the predetermined measurement time, and stop the reading when measurement is complete.
- 6. Repeat to complete averaging over an area or move to different spot and repeat for a new measurement.

Hand Sample

If analysis of hand samples is required, they can be treated much like in-situ samples when performing analysis and interpreting the data.

- 1. Determine whether to average multiple readings or to take one measurement. See Avg Forward to set the averaging function for the analyzer.
- 2. Set the appropriate filters and reading times. See Adjusting the Element Range to set and save the filter settings.
- 3. If desired, set beeps to activate at the end of the predetermined measurement time. See Setting up Audio Cues for instructions to set beep times.
- 4. Grasp the analyzer by the handle, ensuring the wrist strap is properly placed on the wrist.
- 5. Position the sample so that the analyzer may be safely operated, accounting for ALARA principals as low as reasonably achievable. Place the nose of the analyzer on the desired analysis spot, ensuring that the nose of the analyzer is touching the sample, and then initiate a reading. Continue the reading for the predetermined measurement time, and stop the reading when measurement is complete.
- 6. Repeat to complete averaging or choose a new sample and repeat.

Blast Hole Samples

Blast Hole Samples contain material sizes from fine-grained to chips and chunks. To ensure that infinite thickness is maintained and the sampling area will be covered, it is advisable to place these samples in a bag or sample container for analysis.

- 1. Determine whether or not to average multiple readings or to take one measurement per bag or cup. See Avg Forward to set the averaging function for the analyzer.
- 2. Set the appropriate Ranges and reading times. See Adjusting the Element Range to set and save Range settings.
- 3. If desired, set beeps to activate at end of predetermined measurement time. See Setting up Audio Cues for instructions to set beep times.
- 4. Grasp the analyzer by the handle ensuring the wrist strap is properly placed onto the wrist.
- 5. Position the sample so that the analyzer may be safely operated accounting for ALARA principals. Place the nose of the analyzer on the spot where analysis is desired, ensuring the nose of the analyzer is touching the sample, and initiate a reading. Continue the reading for predetermined measurement time and stop the reading when measurement is complete.
- 6. Repeat to complete averaging, or choose a new sample and repeat.

Process Control Samples

Measurements taken after the initial crushing of mine material can be treated much like Blast Hole Samples in the Grade Control section because of their similarly inconsistently sized particles.

- 1. Determine whether to average multiple readings or to take one measurement per bag or cup. See Avg Forward to set the averaging function for the analyzer.
- 2. Set the appropriate Ranges and reading times. See Adjusting the Element Range to set and save Range settings.
- 3. If desired, set beeps to activate at end of predetermined measurement time. See Setting up Audio Cues for instructions to set beep times.
- 4. Grasp the analyzer by the handle ensuring the wrist strap is properly placed onto the wrist.
- 5. Position the sample so that the analyzer may be safely operated accounting for ALARA principals. Place the nose of the analyzer on the spot where analysis is desired, ensuring the nose of the analyzer is touching the sample, and initiate a reading. Continue the reading for predetermined measurement time and stop the reading when measurement is complete.
- 6. Repeat to complete averaging, or choose a new sample and repeat.

Powders and Fine Aggregates

Samples that may come out of a ball mill or similar device will be finer-grained and contain more consistent particle sizes than any of the previously discussed sample types. Therefore, it is appropriate to use a sample cup if desired for analysis.

7 Mining Analysis Concentrates

- 1. Determine whether to average multiple readings or to take one measurement per bag or cup. See Avg Forward to set the averaging function for the analyzer.
- 2. If using 32mm Sample Cups, ensure the cup is properly assembled. Load the sample by placing a few scoops of material into the sample cup and then tamping. Repeat until the material reaches the line just below the top of the cup. Place the Filter Paper 2.4cm dia., the Polystuffing, and the cap on top of the sample, in that order. Do not forget to properly identify each sample.
- 3. Set the appropriate Ranges and reading times. See Adjusting the Element Range to set and save Range settings.
- 4. If desired, set beeps to activate at end of predetermined measurement time. See Setting up Audio Cues for instructions to set beep times.
- 5. Grasp the analyzer by the handle ensuring the wrist strap is properly placed onto the wrist.
- 6. Position the sample so that the analyzer may be safely operated, accounting for ALARA principals as low as reasonably achievable. Place the nose of the analyzer on the desired analysis spot, ensuring that the nose of the analyzer is touching the sample, and then initiate a reading. Continue the reading for the predetermined measurement time and stop the reading when measurement is complete.
- 7. Repeat to complete averaging, or choose a new sample and repeat.

Concentrates

- "Cons" contain relatively high concentrations of the elements of interest and are typically measured using Mining Mode. Samples in this stage of production are generally sized so that a bag or cup is appropriate for analysis.
- 1. Determine whether or not to average multiple readings or to take one measurement per bag or cup. See Avg Forward to set the averaging function for the analyzer.
- 2. If using 32mm Sample Cups, ensure that the cup is properly assembled. Load the sample by placing a few scoops of material in the sample cup, and then tamping it down. Repeat until the material reached the line towards the top of the sample cup. Place the Filter Paper 2.4cm dia. the Polystuffing, and the cap on top of the sample, in that order. Do not forget to properly identify each sample.
- 3. Set the appropriate Ranges and reading times. See Adjusting the Element Range to set and save Range settings.
- 4. If desired, set beeps to activate at end of predetermined measurement time. See Setting up Audio Cues for instructions to set beep times.
- 5. Grasp the analyzer by the handle ensuring the wrist strap is properly placed onto the wrist.

- 6. Position the sample so that the analyzer may be safely operated accounting for ALARA principals. Place the nose of the analyzer on the spot where analysis is desired, ensuring the nose of the analyzer is touching the sample, and initiate a reading. Continue the reading for predetermined measurement time and stop the reading when measurement is complete.
- 7. Repeat to complete averaging, or choose a new sample and repeat.

Slurry

The level of moisture in slurry samples is relatively high. Depending on the data quality needs, drying prior to analysis may be necessary. Like some of the previously discussed sample types, 32mm Sample Cups or bags may be used as long as care is taken to ensure that infinite thickness is maintained.

- 1. Determine whether or not drying samples is appropriate. If so, remove samples from the bath and place in the oven for 1 to 24 hours; then place in sample bag or cup, taking care to break up the sample if necessary.
- 2. If using 32mm Sample Cups, ensure that the cup is properly assembled. Load sample by placing a few scoops of material in the sample cup, and then tamping. Repeat until the material reaches the line towards the top of the sample cup. Place the Filter Paper 2.4cm dia., Polystuffing, and the cap on top of the sample, in that order. Do not forget to properly identify each sample
- 3. Determine whether to average multiple readings or to take one measurement per bag or cup. See Avg Forward to set the averaging function for the analyzer.
- 4. Set the appropriate Ranges and reading times. See Adjusting the Element Range to set and save Range settings.
- 5. If desired, set beeps to activate at end of predetermined measurement time. See Setting up Audio Cues for instructions to set beep times.
- 6. Grasp the analyzer by the handle ensuring the wrist strap is properly placed onto the wrist.
- 7. Position the sample so that the analyzer may be safely operated accounting for ALARA principals. Place the nose of the analyzer on the spot where analysis is desired, ensuring the nose of the analyzer is touching the sample, and initiate a reading. Continue the reading for predetermined measurement time and stop the reading when measurement is complete.
- 8. Repeat to complete averaging, or choose a new sample and repeat.

7 Mining Analysis Slurry

Results Interpretation

Data from fully prepared samples will be comparable with laboratory results for most elements from most types of instrumentation within the error limits from both instruments. Samples that are less than fully prepared may show some discrepancies depending upon the homogeneity of the material in its in situ condition. If the user determines that one sample is not representative of an area, then two or more samples from that area may be quickly analyzed and averaged to aid in data interpretation.

If results differ greatly from the user's expectations, they can always be verified by inspecting the spectrum generated by the sample. Two peaks from each element that is present in the sample will always be present in the spectrum. X-ray energies for each element can be found either on the Thermo Scientific Niton X-ray Energy Reference chart or on the right hand side of the NDT software program. In Figure 1-4 below the Ka and Kb peaks of iron (Fe) are displayed between the 5.40 and 7.20 energies.

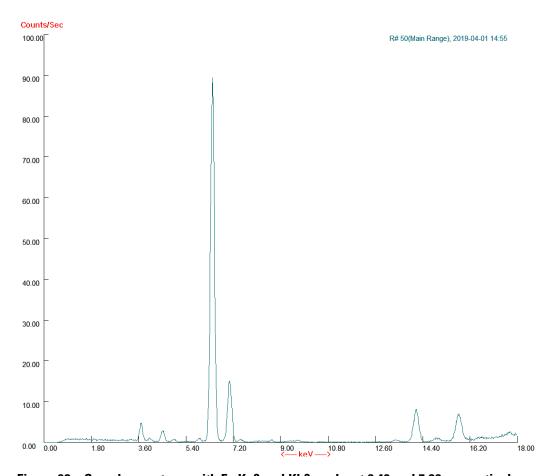


Figure 98. Sample spectrum with Fe Ka? and Kb? peaks at 6.40 and 7.06 respectively

Soil Analysis

Niton XL3 X-Ray Fluorescence (XRF) Analyzer — Version 7.0 and Higher

Analyzing Soil Samples Using the Thermo Scientific Niton® XL3 Series X-Ray Fluorescence (XRF) Analyzer with Software Version 7.0 and Higher

Standard Operating Procedure

Note - Each user should read the Thermo Scientific Niton XL3 Series User's Guide carefully before initiating measurements with the system. Users are strongly urged to attend the Thermo Scientific Niton XRF Analyzer Radiation Safety and Operations Training courses offered regularly (US only), or contact their local representative for product and safety training. For more information, visit www.niton.com.

PREPARATORY TASKS

- 1. Insert a charged battery into the analyzer and turn it on. See Battery Installation and Charging for detailed instructions. Follow the screen instructions and "Log On" as the operator using either the default password or one designated by the user in an NDU file.
- 2. Wait five (5) minutes before using the analyzer, allowing the instrument electronics to stabilize.
- 3. Verify that the date is set properly for data tracking purposes. From the Main Menu, select the System icon then the Specs icon. The date will be displayed for verification. If the date is incorrect, correct it prior to proceeding. This can be done by "Closing" out of the Specs screen and selecting the Date & Time icon from the System Menu. See Setting the Date and Time for detailed instructions. Select the Return Button to go back to the Main Menu.
- 4. (Optional) Connect the analyzer to a computer via the included serial cable, USB cable, or Bluetooth™ wireless module. See Connecting the XRF Analyzer to Your PC for more information.
- 5. During analysis and System Check, it is important to ensure that the analyzer is not exposed to strong electromagnetic fields, including those produced by computer monitors, hard drives, cellular telephones, walkie talkies, etc. Keep a minimum two (2) feet (0.7 meters) distance between the analyzer and electronic devices.
- 6. From the Main Menu, select the System Check icon, and select the Yes Button. (Figure 1.)

- 6.1. System Check calibrates the detector and verifies it is operating to specifications. After starting the process, no further user interaction is required during this operation. When the instrument is finished performing the check, the unit will show either "System OK" or one of the failure errors.
- 6.2. If the unit shows a failure error, then perform a second System Check by selecting the Recheck Button. If the unit still does not show a "System OK," please contact Thermo Scientific Niton Analyzers toll-free at (800) 875-1578, +1 978 670-7460, niton@thermofisher.com, or contact your local Niton Analyzers representative for assistance.

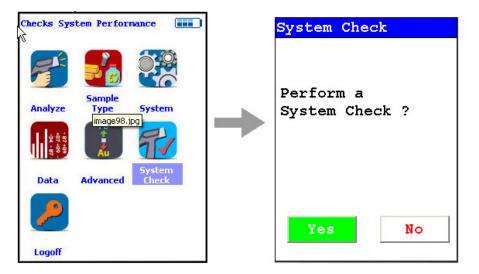


Figure 99. System Check Menu Path

7. Verify and/or change filter position time.

Thermo Scientific Niton XL3 analyzers are equipped with excitation filters that optimize the analyzers' sensitivity for various elements in different matrices. In Soil Mode, up to three filters may be selected, depending on the elements required. The amount of time that the analyzer spends in each filter position is user definable. Please note that the analyzer will continue alternating excitation filters until the user selectable maximum analysis time is reached or the operator terminates the measurement. To verify or change the length of time each filter will be active, do the following:

- 7.1 From the Main Menu select the Advanced icon, then the Element Range icon (Figure 1-2) In the drop-down menu, select Soils Mode.
- 7.2 Select the box next to High Range, Main Range, and Low Range so that a check mark appears in each box.
- 7.3 Enter the desired length of time for each filter. For example, set values of 60 seconds for the Main Range, 60 seconds for the Low Range, and 60 seconds for the High Range as shown in Figure 3.

Adjust Element Ranges

Adjust Element Ranges

All Zri

Return Element Range Sorting

Return Check

Pb

System Check

Consumer Adjust

7.4 Select the Save Button and return to the Main Menu by selecting the Return icon.

Figure 100. Element Range Menu Path

Logoff

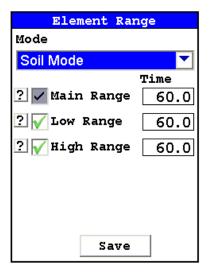


Figure 101. Setting Element Ranges

8.1 Verify instrument measurement accuracy (QC check). From the Main Menu, select the Sample Type icon, then the Soils & Minerals icon, then the Soils icon. You can also use this procedure with TestAll Geo Mode. See GENERAL TESTING PROTOCOL below for more information.



Figure 102. Soil Analysis Menu Path

- 8.1.1. Place TILL4 Reference Sample (P/N 180-601) (or any other reference sample supplied with your instrument) in the test stand for analysis, or set the reference sample on a clean surface. The unlabeled side should be presented to the spectrometer. Place the front end of the instrument against the sample with the Analysis Window centered on the sample.
- 8.1.2. Take one 180-second measurement on this reference sample (60 sec. on each Range). Ensure that the analyzer has the maximum test time set to 180 seconds or greater. From the Main Menu select the Advanced icon, then the Start/Stop Setup icon. Edit the Max. Time dialog box to display the proper setting. See Start/Stop Setup for more information on the Start/Stop Menu.
- 8.1.3. Compare the results with the acceptable ranges as listed in the original accompanying Certificate of Calibration sheet, the results should fall within these specified values. Depending on the element calibrations provided with your Thermo Scientific Niton XRF analyzer, some of the reference sample elements may not be displayed. If your instrument is not displaying all elements, check the Element Display option to determine which elements are being displayed. To force elements not displayed to display, from the Main Menu, select the Advanced icon. Select the Element Sorting icon, then select Soil Mode from the menu. Select the element you wish to modify, select Always Display from the drop down list, then select the Save Button. Repeat this process for each element you want to change, then select the Close Button, select the Close Button again, then select the Return icon.
- 8.1.4 If the analyzer reports values within the acceptable ranges of the reference values, it is ready for measurement of unknown samples. Proceed to GENERAL TESTING PROTOCOL section.
- 8.1.5 If the analyzer reports values outside the acceptance tolerance ranges specified in the accompanying Certificate of Calibration sheet, repeat the System Check described in step 5, then repeat the reference sample analysis in step 7.1

8.1.6 If the analyzer again fails to meet the acceptable tolerance ranges specified in the accompanying Certificate of Calibration sheet, please contact Thermo Fisher Scientific or your local Thermo Fisher representative for assistance.

GENERAL TESTING PROTOCOL

Soil analysis may be performed as either In-Situ Analysis (i.e., in place) or Ex-Situ Analysis and (typically) prepared. The results obtained from the XRF instrument may be different on the same sample when comparing an unprepared with a fully prepared sample. This is generally caused by sample inhomogeneity, particle size effects and moisture content. The more time and effort you spend on preparing the sample properly will allow you to obtain close-to laboratory grade data, whereas in-situ analysis may only provide "screening" type data. The decision to prepare or leave a sample unprepared may be determined by your Data Quality Objectives (DQOs).

Soil mode is a Compton normalized calibration which works over a fairly limited element concentration range. It is highly recommended that samples containing high concentrations of elements (such as ore samples) be analyzed using the Mining mode (see Mining Analysis).

In-Situ Analysis

This is the most basic type of measurement as there is little to no sample preparation performed. It is typically used for providing screening data to help rapidly define an area of contamination for example.

- 1. It is highly recommended that you read and follow the USEPA Method 6200 for soil analysis.
- 2. After performing a QC check (see above), place the In-Situ Test Guard over the front nose of the instrument. This test guard has 2 main purposes, firstly it helps to keep the front of the instrument clean by preventing soil particles from collecting on the window. Secondly it provides a solid base for the Proximity Button to engage properly, which can be difficult on very loose or powdery samples.
- 3. Set the appropriate range and analysis times. The analysis times should have been previously determined See Adjusting the Element Range to set and save the filter settings.
- 4. If desired, set beeps to activate at the end of the predetermined measurement time. See Setting up Audio Cues for instructions to set beep times.
- 5. Remove any large or non-representative debris from the soil surface before analysis. This debris includes rocks, pebbles, twigs, leaves, concrete, etc. If the surface is unlevel, it may be smoothed with a stainless steel trowel or other similar implement. If the soil is saturated with water, allow it to dry before starting the analysis.
- 6. Grasp the analyzer by the handle, ensuring the wrist strap is properly placed on the wrist.

- 7. Enter the sample name, GPS co-ordinates or any other pertinent information regarding the sample to maintain appropriate chain-of-custody. See Entering Soil Sample ID for details.
- 8. Position the analyzer on the desired analysis spot, ensuring that the nose of the analyzer is making contact with the soil, and then initiate a reading.

Continue the reading for the predetermined measurement time, and stop the reading when measurement is complete.

- 9. Confirm that the readings are appropriate and then proceed to next sampling area. Repeat steps 6 and 7.
- 10. Per USEPA Method 6200, a minimum of 1 sample for every 20 analyses (i.e. 5%) should be taken and submitted for laboratory analysis. Make sure that as best you can the sample taken is the sample that you read, samples can vary immensely over very short distances (a few cm).
- 11. After completing all the required analysis, rerun the QC standard to confirm stability of the instrument.
- 12. Download all results (unknown sample readings, QC samples, and system check) and save as an appropriately named file. The ndt file created during the download is encrypted and provides data integrity.

Ex-Situ Analysis

This is a more labor-intensive type of analysis as the sample undergoes full preparation. It is typically used to quickly provide on-site personnel with laboratory grade data without the wait for laboratory analysis, allowing more immediate and accurate decision making to occur.

- 1. It is highly recommended that you read and follow the USEPA Method 6200 for soil analysis.
- 2. Samples are procured in the field and generally taken to a fixed location such as an office or even a vehicle for preparation and analysis. Sample collection is very important as samples collected incorrectly may bias the results. As for the in-situ analysis, non-representative debris should be removed (such as twigs, leaves, rocks etc.) before sample collection begins. The sample is collected from a 4" * 4" square that is 1" deep (10cm * 10cm * 2.5cm deep). This provides enough material to fill an 8oz or 250cm3 specimen jar. Label the jar immediately to maintain chain-of-custody requirements.
- 3. Collect all samples in a similar fashion, and return samples to sample preparation area.
- 4. Sample preparation must then be performed, samples are dried and homogenized, and then ground and sieved.
- 5. Samples may be dried rapidly in either a convection or a toaster oven (if the work site has electricity), or spread out on a tray and air dried (this takes longer) if power is not available. It is generally not recommended to dry samples in a microwave oven.

6. The dried sample should then be well mixed and then split using a technique such as coning and quartering that will ensure an even particle size distribution (so you do not bias the results). This procedure should be repeated until you have approximately 1oz (25g) of representative sample. This sample is then ground with a pestle and mortar until it passes a 60-mesh (250?m) sieve.

Note - The remaining unprepared sample should be returned to the container to allow duplicate XRF or laboratory analysis if required. It is possible to fully prepare the entire sample, but this can be a lengthy process and in-field work does not always allow the analyst the time to do this.

- 7. The ground sample should then be cupped. The 32mm Sample Cups should be assembled on a clean surface (to prevent cross-contamination). Place the outer ring on a clean surface. Lay a piece of Mylar Film 2.5" dia. 6m thick or Polypropylene Film 2.5" dia. 4m thick over the top of the outer ring making sure it is evenly situated over the ring. Take the inner ring and press gently but firmly into the inner ring until it is completely together. Check the film to ensure there are no rips or tears. Fill the cup with the prepared sample and tamp down to ensure there are no air gaps and that the sample is densely packed. When the cup is filled to approximately ¼" (6mm) from the top, add a piece of Filter Paper 2.4cm dia. and then a piece of Polystuffing over the filter (this will prevent the sample moving in the cup if it is inverted). Snap on the cap and label to maintain identification of sample.
- 8. Perform instrument QC check (see above).
- 9. The sample cups may then be analyzed. It is best to analyze these in Test Stands so that the cup is held firmly against the instrument window and the operator is shielded from any back-scattered radiation.
- 10. Enter the sample name, GPS co-ordinates or any other pertinent information regarding the sample to maintain appropriate chain-of-custody. See Entering Soil Sample ID for details.
- 11. Confirm that the readings are appropriate and then proceed to analyzing next cup.
- 12. Per USEPA Method 6200, a minimum of 1 sample for every 20 analyses (i.e. 5%) should be taken and submitted for laboratory analysis. Some of the remaining split can be used for this purpose.
- 13. After completing all the required analysis, rerun the QC standard to confirm stability of the instrument.
- 14. Download all results (unknown sample readings, QC samples, and system check) and save as an appropriately named file. The ndt file created during the download is encrypted and provides data integrity.

Entering Soil Sample ID

1. From the Main Menu, select the Sample Type icon, then the Soils & Minerals icon, then the Soils icon which brings you into the Ready to Test screen.

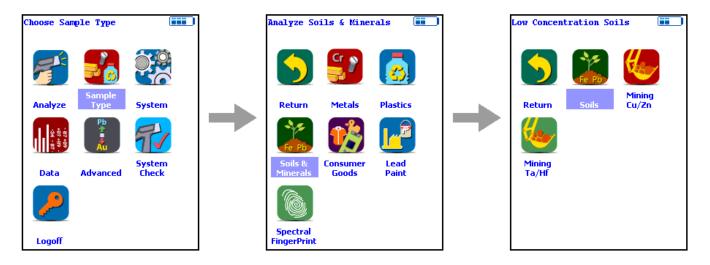


Figure 103. Soil Analysis Menu Path

2. From the Ready to Test screen, select the Data Entry button, then select the Keyboard icon next to the word Sample. This takes you to the virtual keyboard with which you can enter the sample identification. When complete, select the Return Button to take you back to the Ready to Test screen.

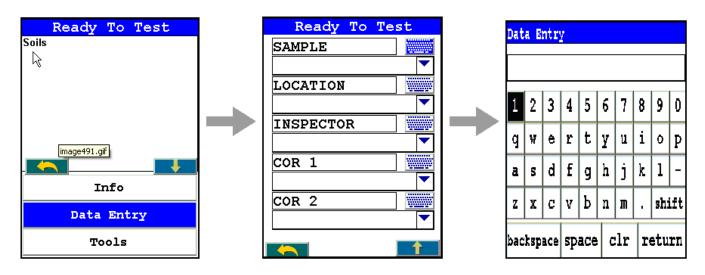


Figure 104. Soil ID Data Entry

3. Additional sample identification information may be entered into other fields if required.

Consumer Goods Analysisniton

Niton XL3 X-Ray Fluorescence (XRF) Analyzer — Version 7.0 and Higher

Screening Toys, Children's Jewelry, and Consumer Products for Lead (Pb) and Other Heavy Metals Using the Thermo Scientific Niton XL3 700 Series X-Ray Fluorescence (XRF) Analyzer – Version 7.0 and Higher

Standard Operating Procedure

Note Each user should read the Thermo Scientific Niton XL3 User's Guide carefully before initiating measurements with the system. Users are strongly urged to attend the Thermo Scientific Niton XRF Analyzer Radiation Safety and Operations Training courses offered regularly, or the web-based trainings. For more information, visit www.thermo.com/niton.

For the purposes of this document the terms testing and screening are used interchangeably. The primary goal of screening is not to accurately analyze the chemical composition of all components or parts of the product, but to weed out and eliminate from the market products containing prohibited elements.

PREPARATORY TASKS

- 1. Insert a charged battery into the analyzer and turn it on. Follow the screen instructions and "Log On" as the operator using either the default password or a custom one as designated by the user in an NDU file.
- 2. Wait five (5) minutes before using the analyzer, allowing the instrument electronics to stabilize.
- 3. Verify that the date is set properly for data tracking purposes.

From the Main Menu, select the System icon, then the Specs icon. The date will be displayed for verification. If the date is incorrect, correct it prior to proceeding. This can be done by selecting the Close Button on the Specs screen, and selecting the Date & Time icon. See Setting the Date and Time for detailed information on this procedure.

- 4. If testing will be performed on small samples, mount the analyzer in its test stand. Any sample that does not completely cover the analyzer measuring window (aperture) is considered small. Also, samples that have a form of thin sheet, such as foil, paper, fabric, etc., should also be analyzed in a test stand.
- 5. (Optional) Connect the Thermo Scientific Niton 700 Series analyzer to a computer via the included serial cable, USB cable, or Bluetooth™ wireless module. See Connecting the XRF Analyzer to Your PC

- 6. During analysis and detector calibrations, it is important to ensure that the analyzer is not exposed to strong electromagnetic fields, including those produced by computer monitors, hard drives, cellular telephones, walkie talkies, etc. Keep a minimum two (2) feet (0.7 meters) distance between the analyzer and electronic devices.
- 7. From the Main Menu, select the System Check icon, then the Yes Button.
- 7.1. System Check standardizes the detector. After starting the process, no further user interaction is required during this operation. When the instrument is finished performing the check, the unit will show either "System OK" or one of the failure errors.
- 7.2. If the unit shows a failure error, then perform a second detector calibration. If the unit still does not show a "System OK," please contact Thermo Scientific Niton Analyzers toll-free at (800) 875-1578, +1 978 670-7460, niton@thermofisher.com, or contact your local Niton Analyzers representative for assistance.
- 8. Verify and/or change Range time.

Thermo Scientific Niton analyzers are equipped with excitation filters that optimize the analyzers' sensitivity for various elements. The "Main Range" provides optimum sensitivity for the elements iron (Fe) through barium (Ba) plus the elements mercury (Hg), lead (Pb), and bismuth (Bi). The "Low Range" is used to optimize the sensitivity for the elements from titanium (Ti) through chromium (Cr). The "Light Range" filter is available only with He-purged and GOLDD technology analyzers, and is typically used in light element analysis. The amount of time that the analyzer spends in each Range position is user definable. Please note that the analyzer will continue alternating excitation filters until the user selectable maximum analysis time is reached or the operator terminates the measurement. To verify or change the length of time each filter will be active, do the following:



Figure 105. The Range Menu Path (Main)

To verify or change the length of time each Range will be active, do the following:

- 8.1. From the Main Menu select the Advanced icon, then the Element Range icon.
- 8.2. In the drop-down menu, select the mode that is intended for use (i.e., Plastics, Electronics Metals, or TestAll).
- 8.3. Select the box next to Main Range and Low Range so that a check mark appears in each box.
- 8.4. Enter the desired length of time for each filter. For example, set values of 50 seconds for the Main Range and 10 seconds for the Low Range as shown in Figure 1-2.
- 8.5. Select Save and return to the Main Menu by clicking Return.

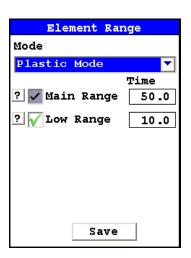


Figure 106. Filter Configuration

- 9. Verify instrument measurement accuracy. Depending on the configuration of your instrument, you will have been supplied one or more multi-element reference samples. These samples are use to verify that the instrument is performing properly. There are two polymer reference samples; P/N 180-619 and P/N 180-554. You may have also been supplied with a modified SAC 305 solder sample, P/N 180-606. Either of the polymer samples can be used to verify the instrument performance in Plastics Mode, and the solder sample is used to verify Electronic Alloys Mode. The nominal element concentrations and acceptable ranges of results for each reference sample are given below in the following tables.
- 9.1 Plastics. From the Main Menu, select the Sample Type icon, then the Plastics icon.
- 9.1.1. Place the Multi-element Reference Sample (e.g., PN 180-619) in the test stand for analysis, or set the reference sample on a clean surface. The unlabeled side should be presented to the spectrometer. Place the front end of the instrument against the sample with the window centered on the sample. Due to the length of this check measurement, it is recommended that a test stand be used. It can be difficult to hold the analyzer steady on the reference sample for the required length of time.

- 9.1.2. Take three 180-second measurements on this reference sample (120 sec. with Main Filter and 60 sec. with Low Filter). Ensure that the analyzer has the maximum test time set to 180 seconds. From the Main Menu, select the Advanced icon, then the Start/Stop Setup icon. Edit the Max. Time dialog box to the proper setting. Average the results. See Averaging Results for information on how to average results forward. Select the number of readings to be average (in this case 3) and begin measuring.
- 9.1.3. Compare the averaged results with the acceptable ranges as listed in Tables 1, 2, or 3. The averaged results should fall within these specified values. Depending on the element calibrations provided with your Thermo Scientific Niton XRF analyzer, some of the reference sample elements may not be displayed. If your instrument is not displaying all elements, check the Element Display option. From the Main Menu, select the Advanced icon, then the Element Sorting icon. Select the mode that you wish to check or modify. Then, set the elements you wish to be displayed and not displayed according to the choices in the drop-down menu at the bottom of the screen (Always, Normal, and Never).

Table 5: Multi-element Polymer Reference Sample (Niton Part Number 180-619) in Nominal Concentrations and Acceptable Ranges (Main Range 120s, Low Range 60s)

Element	Nominal Concentration* (mg/kg)**	Acceptable Range (+/- % Rel)	Acceptable Range (mg/kg)	
Ba	690	15%	585 - 795	
Pb	150	10%135-165	135 - 165	
Cr	100	15%	85 - 115	
Hg	100	15%	85 - 115	
Cd	290	16%	246 - 336	
Sb	100	15%	86 - 116	
Se	200	15%	173 - 235	
As	50	25%	37-63	

Table 6: Multi-element Polymer Reference Sample (Niton Part Number 180-554) in Nominal Concentrations and Acceptable Ranges (Main Range 100s)

Element	Nominal Concentration* (mg/kg)**	Acceptable Range (+/- % Rel)	Acceptable Range (mg/kg)		
Br	500	25%	375 - 625		
Pb	1000	10%	900-1000		
Cr	1000	15%	850 - 1150		

Table 6: Multi-element Polymer Reference Sample (Niton Part Number 180-554) in Nominal Concentrations and Acceptable Ranges (Main Range 100s)

Hg	1000	15%	850 - 1150
Cd	150	25%	112 - 188

Table 7: Multi-element Solder Reference Sample (Niton Part Number 180-606) in Nominal Concentrations and Acceptable Ranges (Main Range 30s)

Element	Nominal Concentration* (wt %)	Acceptable Range (Wt %)
Sn	Balance	93 - 98
Ag	3.05	2.50 - 3.30
Cu	0.55	0.40 - 0.80
Cd	0.34	0.27 - 0.41
Pb	0.12	0.10 - 0.18

^{*} Please refer to the certificate accompanying the reference sample for actual elemental composition data.

- 9.1.4 If the analyzer reports values within the acceptable ranges of the reference values, it is ready for measurement of unknown samples. Proceed to ANALYSIS OF UNKNOWN SAMPLES section.
- 9.1.5 If the analyzer reports values outside the acceptance tolerance ranges specified in the tables, repeat the detector calibration described in step 7, then repeat the reference sample analysis in step 9.1.
- 9.1.6 If the analyzer again fails to meet the acceptance tolerance ranges specified in the tables, please contact Thermo Scientific Niton Analyzers or your local representative for assistance.

ANALYSIS OF UNKNOWN SAMPLES

General Remarks

Toys and the majority of consumer products represent a wide variety of composition, shapes, and functionality. Plastic, wooden, and paper goods, as well as most apparel, should be tested using Plastic Mode, while those wholly made of alloys and metals, including isolated buttons and zippers, should be tested in Metals Mode. Products and components made of a combination of metallic and nonmetallic parts (materials) should be tested using Plastic Mode. Painted objects should be tested in the mode appropriate to the nature of their substrate, i.e., in Plastic or Metals Mode.

^{**} Please note that mg/kg is a unit of measure of concentration equal to ppm by mass, i.e., 1000 ppm is equivalent to 1000 (mg/kg) = 0.1%.

9 Consumer Goods Analysisniton Niton XL3 X-Ray Fluorescence (XRF) Analyzer – Version 7.0 and Higher

Inside the Consumer Goods mode, the operator can select TestAll™ (patent pending), Metals & Minerals, or Plastics depending upon the operator's knowledge of the material being tested. The TestAll calibration was developed to assist the operator when he or she is unsure of the correct calibration to use. This program will automatically determine the nature of the material being analyzed and will select the most appropriate calibration to apply to the analysis. If the operator can correctly identify the matrix of the test specimen, the best results may be obtained by selecting Metals & Minerals (alloys, ceramics, and minerals) or Plastics (PVC, polyethylene, or any polymer).

Consumer Goods mode also has a provision for an optional Painted Products calibration (calibration designator Ap). When enabled, Painted Products mode will determine if the element lead (Pb) is present in the painted coating or the substrate material. If the analyzer determines that Pb is present in the painted surface, the analyzer will report the Pb content in units of mass-per-unit-area, specifically $\mu g/cm^2$. Pb, if present, will cause the analyzer to PASS or FAIL the object undergoing testing based on the user-selectable Painted Products Action Level. The value of the action level can be set from 1 $\mu g/cm^2$ to 9 $\mu g/cm^2$. If the analyzer determines that Pb is present in the substrate of the material being tested, then the analyzer will report the Pb concentration in parts-per-million (ppm) and will be subject to the PASS/FAIL criteria designated by the user. If so desired, the Painted Products mode can be disabled within TestAll so that all data will be reported in ppm.

A more thorough discussion of the Consumer Goods mode, TestAll technology, and Painted Products is included in APPENDIX A. A more thorough discussion of Errors of Measurement, Quality of Results, and Their Interpretation for Compliance Testing Using Niton XL3 Series XRF analyzers can be found in Appendix B. The following text applies to the use of an Niton XL3 700 for testing consumer goods in general and should be followed regardless of the software version installed on your instrument.

All parts of a product should be screened for restricted elements using the appropriate analytical mode of analyzer. The primary goal of screening is not to accurately analyze the chemical composition of all components or parts of the product, but to single out and eliminate from the market products containing prohibited elements. Therefore, it is only logical to start testing the product "as is," without disassembly. This is particularly true of the products that are painted because many of the toxic elements – especially lead – are typically found in paint. Once the presence of a toxic element is confirmed during testing "as is," the product can be sequestered, and further testing may not be necessary. If, however, the testing of the product "as is" produces no evidence of toxic elements, but it is suspected that such elements may be contained in inaccessible parts of the product for direct "as is" testing, one must consider disassembly of the product for compliance testing at the component level. If the product or its components are large enough, they may be tested directly by pressing the analyzer against their surfaces. Otherwise, measurements should be performed in a test stand.

Due to the large variety of consumer products, only general rules of testing, related mainly to proper sample selection and results interpretation, are outlined here. Detailed testing protocols should be developed for specific cases by the user.

GENERAL TESTING PROTOCOL

Large Objects

These are the objects that can easily cover the measuring window of the analyzer and are typically at least as large as the analyzer.

- 1. Select the areas or fragments of the sample that are to be tested.
- 2. Based on the type of material, select appropriate analytical mode:

Plastic Mode for all nonmetallic materials and combinations of nonmetallic and metallic

Metals Mode for all metallic materials

TestAll Mode for a mixture of both metallic and nonmetallic materials

- 3. Place the measuring window of the analyzer flush within the area identified on the toy or product for analysis and pull the trigger to start measurement.
- 4. Continue the measurement until the display says PASS or FAIL. These results can vary depending on the settings that are selected. For elaboration, refer to APPENDIX A.
- 5. Figures 1-3 and 1-4 show examples of this type measurement.



Figure 107. Screening a Larger Object



Figure 108. One Possible Way of Screening a Toy

6. When testing larger surfaces, it is advisable to take several measurements at different locations. This is especially important if the surface tested is partially painted, plated, or otherwise appears different from location to location.

This will help in identifying the location of the analytes, whether in the substrate, paint, or both.

- 7. For interpretation of the results, consult Testing Time and Detailed Results Interpretation.
- 8. Continue measurements as planned.

Small Objects

These are objects that are typically smaller in size than analyzer, but still large enough to cover the measuring window of the analyzer.

- 1. Select the areas or fragments of the object that are to be tested.
- 2. Mount the analyzer in a test stand and connect the analyzer to a PC (via Serial Cable, USB Cable, or via Bluetooth). Start the NDTr Program on PC.
- 3. Place the object in a test stand in such a way that the area selected for testing covers the measuring aperture of the instrument as much as possible. If the toy cannot be manipulated into a position suitable for measurement, one may need to use some mounting assistance. Care must be taken to not allow any part of the support to be within the perimeter of the measuring window of the analyzer. Figures 4 through 13 show examples of how the object can be manipulated into proper testing position.
- 4. Based on the type of material, select appropriate analytical mode:

Plastic Mode for all nonmetallic materials and combinations of nonmetallic and metallic

Metals Mode for all metallic materials

TestAll Mode for a mixture of both metallic and nonmetallic materials

- 5. Start the measurement, preferably via a PC using the NDTr software program.
- 6. Continue the measurement until the display says PASS or FAIL. These results can vary depending on the settings that are selected. For elaboration, refer to APPENDIX A.
- 7. If possible, take more than one measurement of the sample, preferably in a different location.
- 8. For interpretation of the results consultTesting Time and Detailed Results Interpretation.
- 9. Continue measurements as planned.

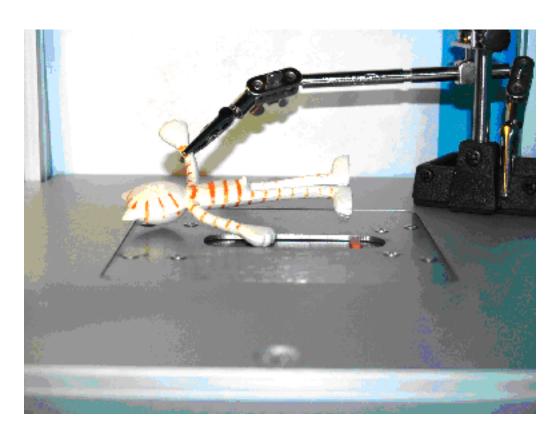


Figure 109. Positioning the Product

In Figure 1-5, the figure is positioned so that the paint on the palm of its hand is as close to the window as possible. A "helping hand" is used to assist positioning. Note: Clamps should be at least 1 inch from window.

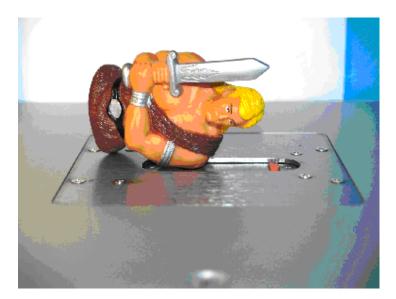


Figure 110. Analyzing the Aarm

In Figure 1-6, the figure is balanced with its arm positioned for analysis. Not all samples will require "helping hand" or other devices.



Figure 111. Analyzing Figure Base

In Figure 1-7, we analyzed the unpainted bulk of plastics at the base of the figure.



Figure 112. Analyzing the Back

In Figure 1-8, the figure is positioned to analyze its back.

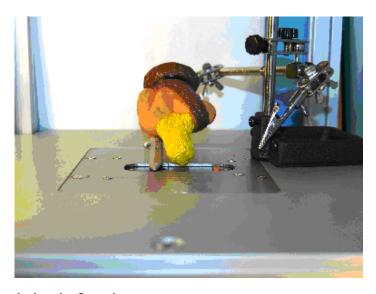


Figure 113. Analyzing the Sword

In Figure 1-9, the sword of the figure is undergoing analysis. Note use of "helping hand" to position the sword.



Figure 114. Ana; yzing the Arm Band

In Figure 1-10, again with the aid of the "helping hand," the silver arm band on the right arm is screened.

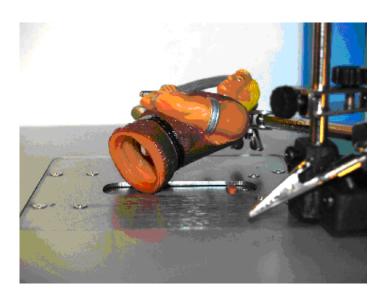


Figure 115. Analyzing the Paint

In Figure 1-11, we are analyzing the brown paint on the figure's clothing.



Figure 116. Analyzing the hair

In Figure 1-12, propped on the "helping hand," the yellow hair on the figure undergoes screening.

Very Small Objects

These are objects that are not large enough to cover the analyzer measuring window, typically less than 10 mm in any dimension, e.g., beads, small jewelry pieces, chains, etc.

- 1. Mount the analyzer in a test stand and connect it to PC (via Serial Port, USB port, or Bluetooth). Start NDTr software program on user's PC.
- 2. Place the object in a test stand in such a way that it resides in the center of the measuring window of the analyzer. If available, it is possible to take more than one identical small item and pile a small quantity of them over the measuring window of the analyzer. However, one must be certain that these individual items are identical in composition.
- 3. Based on the type of material, select appropriate analytical mode:

Plastic Mode for all nonmetallic materials and combinations of nonmetallic and metallic

Metals Mode for all metallic materials

TestAll Mode for a mixture of both metallic and nonmetallic materials

- 4. Start the measurement, preferably using NDTr.
- 5. Continue the measurement until the display says PASS or FAIL. These results can vary depending on the settings that are selected. For elaboration, refer to APPENDIX A.
- 6. For interpretation of the results consult Testing Time and Detailed Results Interpretation.

7. Continue measurements as planned.

Thin Objects, Such as Plastic, Foils, or Films

- 1. Mount the analyzer in a test stand and connect it to PC (via Serial Port, USB port, or Bluetooth). Start NDTr program on user's PC.
- 2. Place the film/foil object in a test stand in such a way that it covers the measuring window of the analyzer. If possible, take more than one layer of film and stack as many of them as are available over the measuring window of the analyzer.

It is recommended the thickness of the stack be at least 5 mm. However, either single layers or stacks can be analyzed as long as they are not less than 1 mm in thickness. Also, it should be noted for a meaningful representative result, all samples should have the exact same composition.

- 3. Based on the type of material, select appropriate analytical mode:
 - Plastic Mode for all nonmetallic materials and combinations of nonmetallic and metallic
 - Metals Mode for all metallic materials
 - TestAll Mode for a mixture of both metallic and nonmetallic materials
- 4. If Plastic Mode has been selected or if the sample is a plastic being tested in TestAll Mode, set the thickness correction on the instrument to the actual thickness of the sheet or stack of film in mm. See Thickness Correction for details.
- 5. Start the measurement, preferably using NDTr.
- 6. Continue the measurement until the display says PASS or FAIL. These results can vary depending on the settings that are selected. For elaboration, refer to APPENDIX A.
- 7. For interpretation of the results consult Testing Time and Detailed Results Interpretation.
- 8. Continue measurements as planned.

Paint Chips

Some objects may have painted surfaces that will not be accessible for direct x-ray scan. In such circumstances, it is possible to scrape a small sample of paint and analyze it directly by placing it over the measurement window of the analyzer.

- 1. Mount the analyzer in a test stand and connect it to PC (via Serial Port, USB port, or Bluetooth). Start NDTr program on user's PC.
- 2. Take a Mylar film (Part Number 187-462) and spread it on a clean, flat surface.
- 3. With a sharp razor blade or Exacto knife, scrape paint from the area to be tested and place it over the Mylar film. Use caution during this step so as not to disturb the substrate material.

- 4. Take a Prolene window used for helium flush instruments (PN 187-1454) and cover the chip of paint. Now the paint chip is secured between two thin plastic films and ready for analysis (see Figure 1-13).
- 5. Select the Plastic Mode of analysis. Set thickness correction to OFF. See Thickness Correction for details.
- 6. Place the paint sample over analyzer window and perform a measurement. Typically, a 30-second analysis time is sufficient to ascertain the presence or absence of lead in the paint.
- 7. Continue the measurement until the display says PASS or FAIL.
- 8. For interpretation of the results consult Testing Time and Detailed Results Interpretation.
- 9. Continue measurements as planned.



Figure 117. Analyzing Paint Chips Secured Between Two Plastic Films

Apparel

Screening and analyzing apparel items – fabric, appliqués, buttons, zippers, footwear – for toxic elements using XRF can pose some challenges. While these challenges can be met, it is important to keep some key concepts in mind to successfully perform the screenings and interpret the results.

9 Consumer Goods Analysisniton Niton XL3 X-Ray Fluorescence (XRF) Analyzer – Version 7.0 and Higher

By nature, fabrics, in particular, are a very low-density sample. This means that the XRF instrument effectively "sees" less sample-per-unit area than with a metallic specimen, for example. One way to compensate for the lower density is to increase the analysis time, thereby allowing the instrument to gather more data. Another option is to increase the apparent mass of the sample "seen" by the instrument by folding the fabric several times. Using the latter technique may mitigate having to significantly increase the analysis time, though increasing the time still could be necessary.

Another consideration when analyzing fabrics is that the incident x-ray beam will penetrate the material relatively unimpeded, meaning that x-rays generated from the sample-support-medium beneath the fabric can pass back up through the fabric sample to the analyzer. Simply pressing the analyzer on the garment while it is being supported on a tabletop is a common temptation. As expedient as this appears, it is possible to get erroneous results if there are elements in the table that are the same as those elements being tested for in the sample material. In addition, there may be elements present in the table that interfere with the detection of the elements of interest in the sample material. In all cases, the composition of the tabletop should be non-metallic.

Analyzing fabrics in one of the supplied test stands is always preferable, however, that may not always be practical because of the size of the sample undergoing screening or some other circumstance. If sample analysis on a tabletop is necessary, then the tabletop itself should be screened first with the analyzer to determine if it contains contaminate elements or if it contains significant concentrations of an element that could interfere with the target elements within the sample material. If the user determines, after screening, that tabletop does not pose an impediment to the analysis of the test specimens, then the fabric should be folded several times before subjecting it to analysis. Proper x-ray safety procedures should be followed, which includes avoiding having any body parts under the tabletop where the analysis is being performed.

The Niton XL3 can also be used to analyze garments on a hanger, though the user may find that it is impractical to support the weight of the instrument for the entire analysis time. Please note that when undertaking this type of analysis, it is imperative that the user give proper consideration to radiation safety procedures, ensuring that no one could possibly be exposed to the x-ray beam after it passes through the hanging garment.

Garment fasteners, such as buttons and zippers, should be centered as closely as possible over the analysis aperture of the instrument. If a signal is detected for any of the elements of concern, then the adjacent fabric should be tested to determine from where the signal is coming. Alternatively, the fastener of interest can be removed and screened separately.

Appliqués, such as decals or silk screens, can be screened directly. As in the above example, the adjacent fabric material should be screened separately if an element of concern is detected to determine whether the signal is coming from the appliqués or the fabric.

1. Mount the analyzer in a test stand, if used, and connect it to PC (via serial port, USB port, or Bluetooth TM). Start NDTr software program on the user's PC.

- 2. Place the object in a test stand in such a way that it is centered in the measuring window of the analyzer. If enough material is available, fold it over on itself several times.
- 3. Based on the type of material, select appropriate analytical mode:

Plastic Mode for all nonmetallic materials and combinations of non-metallic and metallic

Metals Mode for all metallic materials

TestAll Mode for a mixture of both metallic and nonmetallic materials

- 4. Start the measurement, preferably using NDTr.
- 5. Continue the measurement until the display says PASS or FAIL. These results can vary depending on the settings that are selected. For elaboration, refer to APPENDIX A.
- 6. For interpretation of the results consult Testing Time and Detailed Results Interpretation.
- 7. Continue measurements as planned.

Detect vs. Non-Detect Screening (GO/NO-GO Screening)

All Thermo Scientific Niton XL3 700 Series instruments can be factory set with "Detection Element Display Option" activated in the Consumer Goods Settings Screen. When "Detection Settings" are activated in the Consumer Goods Settings, the user can set a requirement for a minimum measurement time. The minimum time must be achieved before the instrument will allow a sample to PASS on screening. If the minimum time is not achieved, and the sample has not yet reached a FAIL determination, then the Message will read INCONCLUSIVE.





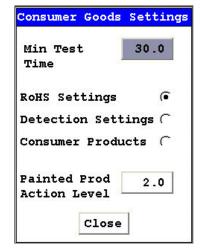


Figure 118. Consumer Goods Setting Screen

Note The minimum time threshold applies only to the main filter. Match the Main Filter time to the minimum test time for optimized Pb testing. Also, if Cr, Ti, or V analysis is not desired, shut off the Low Filter.

For better sensitivity to Cr, set Main Range to less than the minimum test time, and activate Low range for the desired Cr test time. This mode yields best results for Cr.

Factory default settings are as follows:

- Main Range = 50 seconds
- Low Range = 10 seconds
- Detection Settings minimum test time = 30 seconds

The system toggles between the two Ranges until the minimum time on Main Range is achieved. Therefore, total test time is 30 seconds. Although the minimum test time is set to 30s, the Low Range is still able to be activated by allowing the instrument to scan over 50s.

Making Determinations

The decisions for GO/NO-GO analysis will be made by checking whether or not a monitored element rises above the detection limit in a predefined measurement time.

Testing Time and Detailed Results Interpretation

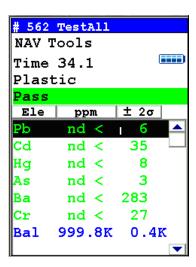


Figure 119.

In this case, the sample has been measured for the predetermined minimum amount of analysis time (30s).

• All elements are below the detection limit.

- The element display changes each element to the color green.
- "Pass" is displayed meaning that the instrument either did not detect a monitored element or detected an element within the acceptable range in the pre-determined minimum analysis time.

This sample area passes the test.

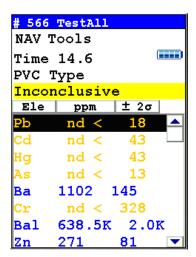


Figure 120.

In this case, the sample was measured for 6 seconds.

Pb determination:

- The instrument has already detected the presence of Pb.
- No more testing time is required for the determination of Pb presence.
- This sample area fails the test.

The other six elements of interest:

- More time is required to determine the presence of the other six elements being screened.
- If no other element is detected in time, then each of the other elements will turn to a green display color.

Pb remains red, the sample still fails

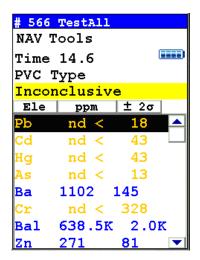


Figure 121.

Here, the sample was measured for 15 seconds.

Ba, Bal, and Zn determination:

• The color display is blue meaning that although the instrument did detect the presence of Ba, Bal, and Zn, they are not part of the Pass/Fail criteria and thus, their presence is considered neutral.

The other elements of interest:

- More time is required to determine the presence of the other elements being screened (Pb, Cd, Hg, As, Cr).
- If no other element is detected in time, then each of the other elements will turn to a green display color.

The "Inconclusive" will then change to "Pass"

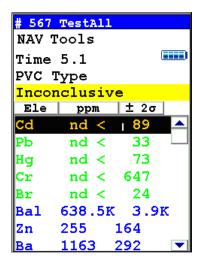


Figure 122.

In the case of RoHS or Consumer Goods, as there are valued limits set for Pass/Fail and no minimum test time it is entirely possible to see the following screen. Here the sample was measured for 5s.

For lead(Pb)

- "nd" < 33" means non-detect below 33ppm.
- Pb and its result appear in green because the instrument has determined that the sample has less than the lower set limit.

The other elements of interest:

- Hg, Cr, and Br have also been deemed to be a Pass denoted by the green color.
- The sample, itself, is inconclusive, though because Cd is showing nd below 89ppm which is above the lower Pass limit of 70ppm.

For the typical user, the system will be pre-programmed with fixed parameters. The user simply holds the trigger down through the INCONCLUSIVE period, until the display gives a result of PASS or FAIL.

The advanced user may choose to optimize testing times for a specific task. The test should never be run for less than 5 seconds. If two Ranges are activated, then measure for a minimum of 5 seconds on each filter. Normal test times are in the range of 20-120 seconds, depending on the desired detection limit. For example, it will take approximately 60 seconds on the Main Range to find 600 ppm Pb concentration in a 1 mil (25 micron) thick layer of paint.

See Thermo Scientific Niton Analyzer application notes for detailed studies into testing times for each monitored element.

9 Consumer Goods Analysisniton APPENDIX A

The results can always be verified by inspecting the spectrum generated by the sample. Whenever lead is detected, the spectrum will show two peaks of lead, approximately the same size, one at 10.5 keV and another at 12.6 keV. If lead is not detected, the spectrum will show no lead peaks. The spectra in Figure 22 below are examples of the two situations described.

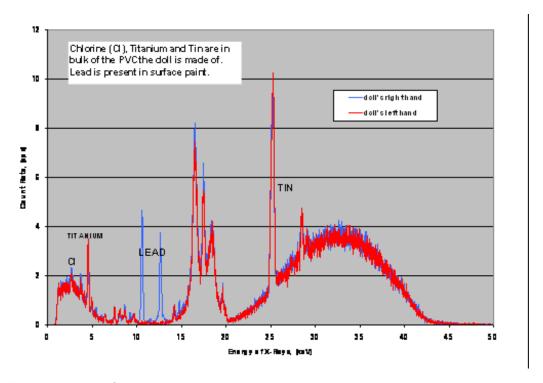


Figure 123. Paint Spectrum

Spectrum of paint as measured directly on the same color on two separate areas of a doll. One part is painted with leaded paint (blue spectrum). The other part containing the same color paint, shown with red spectrum, does not show any lead. This can be caused by separate parts being painted on separate production lines, or may be due to rework of a cosmetic defect.

APPENDIX A

Consumer Goods Mode for Screening Consumer Products

Consumer Goods mode was recently developed to aid in the analysis of the wide range of samples for regulated toxic elements that may be present in consumer products. Toys and apparel have been the subject of much concern lately, so their testing will be the primary focus of this discussion. This is not to say that the Niton XL3 700 system cannot be used to screen for toxic elements in other diverse products such as artificial turf, furniture and apparel. Recent updates to the software have allowed for the ability to determine Cl (chlorine) and Br (bromine) for the analysis of consumer goods for halogen-free acceptance.

The main features of Consumer Goods mode consists of calibrations for TestAll™ (patent pending), Metals & Minerals, Plastics and the optional Painted Products calibration.

The TestAll calibration was briefly described in the body of the attached SOP. As a review, this calibration performs a brief analysis of the sample being analyzed and then picks the most appropriate calibration to apply to this sample. The user benefits by not having to determine and then manually select the proper calibration to use. Upon starting the analysis sequence, the program will automatically select between the following matrix classifications: metal, painted metal, thin metal, plastic, PVC plastic, thin plastic, aluminum alloys, layered materials, and non-metal. The test results are evaluated for PASS/FAIL/INCONCLUSIVE based on the results for each element the user selects and compared against the decision criteria selected.

The decision criteria for PASS/FAIL/INCONCLUSIVE are set for each mode programmed into the Niton XL3 700. To view or change the criteria for the Plastic calibration, from the Main Menu select the Advanced icon, then the Element Thresholds icon, then the Plastic icon.

One of the following screens will appear:

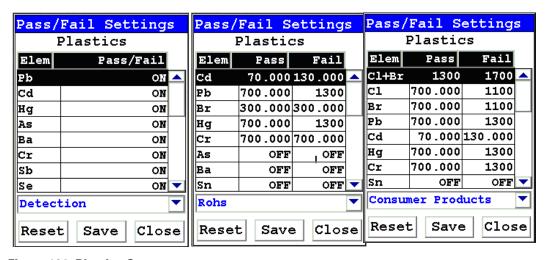


Figure 124. Plastics Screens

The screen on the left shows the eight commonly regulated elements in toys that the analyzer is monitoring. These eight elements, or any other elements, can be monitored by toggling the value under the Pass/Fail column heading to be either ON or OFF. When the analyzer is in Toy mode, the elements are being monitored (i.e., ON) based solely on whether they are detected or not. If a monitored element is detected, that element will be noted as a failed condition regardless of its measured concentration. By scrolling down the elements list, you will see that all the other elements are labeled as OFF, which means that their presence in the sample is not being considered in the PASS/FAIL determination. The user may elect to not consider any element for PASS/FAIL by clicking on the Pass and Fail columns adjacent to the element and then editing the selection to be OFF.

9 Consumer Goods Analysisniton APPENDIX A

The screen in the middle shows the Restriction of Hazardous Substances (RoHS) elements. You will notice that there are fewer monitored elements than in Toy mode and that there are ranges of concentration for pass and fail. In the RoHS mode, the actual concentration of the monitored element is taken into account before declaring the sample as a PASS/FAIL/INCONCLUSIVE. The use of the instrument for the RoHS application is discussed in the SOP that is available to you. See Consumer Product Settings for changing testing criteria.

The screen image on the right shows the Pass/Fail limits set for Consumer Products. As in RoHS, there are concentration values, rather than just ON/OFF. In addition to the RoHS settings, there is also the option to set limits for halogens, which are a function of industries that are looking to push their products to be manufactured "Green."

The analyzer will use one of the above three criteria for determining the PASS/FAIL state of a test sample based upon the selection made in the following section.

From the Main Menu select the Advanced icon, then the Consumer Goods icon.

The following screen will appear:

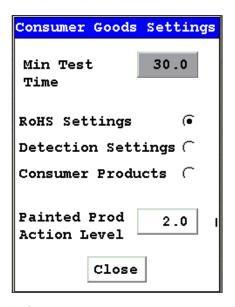


Figure 125. Consumer Goods Screens

The options are:

Minimum Test Time – This is the minimum time in seconds that must elapse before a sample will be labeled as PASS. If one of the monitored elements is detected prior to the minimum test time being reached, the sample will FAIL. The active analysis screen will indicate INCONCLUSIVE, up to the minimum test time, if a monitored element has not yet been detected. This setting is only active and valid in Detection Settings

9 Consumer Goods Analysisniton APPENDIX A

RoHS Settings – Selecting this option will make the analyzer consider the pass and fail criteria entered for each element as described above. There is no Minimum Test Time considered when the RoHS Settings are selected.

Detection Settings – This option will make the analyzer PASS or FAIL a sample based on a detect/not-detect basis for each of the selected monitored elements as described above. The minimum test time must be set to a desired value for the instrument to function up to the user's standards.

Consumer Products – This option addresses the analysis of total halogens – chlorine plus bromine. To be considered halogen free there can be no more than 900 ppm Cl and 900 ppm Br. In aggregate, the sum of Cl and Br cannot exceed 1,500 ppm. On a Niton XL3 700, chlorine is detected on the low filter, yielding a detection limit of approximately 500 ppm or better in a polymer at a measurement time of 60s.

Painted Products Action Level – If so calibrated, the analyzer will refer to the entered value, which is in $\mu g/cm^2$, to determine if the test sample is a PASS or FAIL. Currently, only the element Pb is subjected to this test.

To select the TestAll mode, perform the following steps:

9 Consumer Goods Analysisniton APPENDIX A

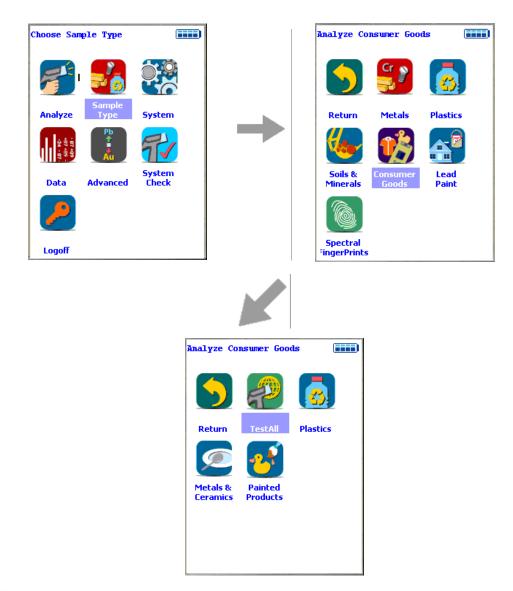


Figure 126. TestAll Menu Path

The following screen shots are from an example of analyzing a painted metal sample that has a high level of Pb content in the dried paint film. The Live Spectrum option has been selected from the Tools drop-down menu. The Painted Products output option can be toggled off and on by using the Tools drop-down menu.

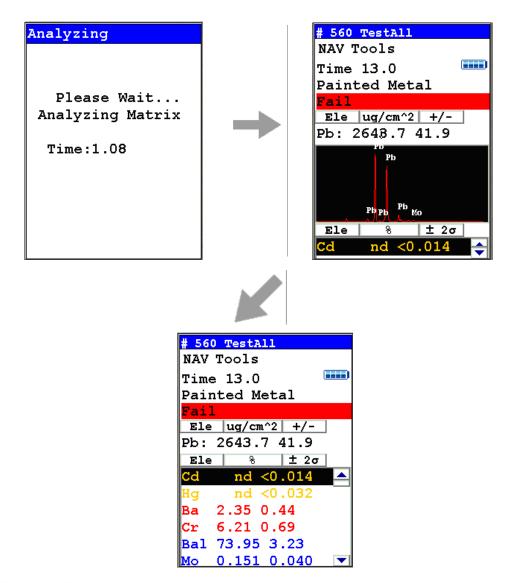


Figure 127. TestAll Analysis

The Painted Products calibration and its indication of the lead content in µg/cm² was developed in anticipation of current legislation where the use of the mass-per-unit area indication will be allowed, in certain specific instances, in addition to the historical ppm lead level concentration stipulated by 16 CFR 1303 from the Consumer Products Safety Commission and ASTM F963. For XRF to accurately give readings in paint films on products directly in ppm, two more pieces of information are required to make the calculation – the paint density and thickness. These two variables are rarely known. Presenting the lead content in mass-per-unit area allows the instrument to be calibrated with known standards and does not rely on information about the sample being tested that is difficult or impossible to know.

9 Consumer Goods Analysisniton Appendix B

Thus, this method allows the XRF instrument to be used to its full advantage, namely as a rapid and non-destructive test. XRF has already been widely used for determining the lead content in painted surfaces for years with the acceptance of OSHA and HUD for leaded paint remediation in buildings.

Appendix B

Errors of Measurement, Quality of Results, and Their Interpretation for Compliance Testing Using Thermo Scientific Niton XL3 Series X-ray Fluorescence (XRF) Analyzers

By Stanislaw Piorek, PhD, Principal Research Scientist

Author's Note: For the purposes of this document, milligrams per kilogram (mg/kg) and parts per million (ppm) are used interchangeably, with mg/kg the preferred SI unit of measure.

MEASUREMENT ERROR

Any measurement result – regardless of the analytical method used – is always accompanied by error, which is defined as the difference between the measured value of the quantity (such as mass fraction or concentration) and its value accepted as true. Error is the consequence of variability inherent in any measurement process. Every time we repeat a measurement, we obtain a slightly different result.

Depending on the specific conditions of measurement and the measurement technique itself, the error may be large or small; however, it never is equal to zero. Therefore, any measurement result must always be reported with its associated error; the results reported without error are fundamentally meaningless despite the fact that the habit of reporting measurement without error is very common.

Error of measurement is expressed in the same units as the result, and it is called an absolute error. If we relate the value of an absolute error to the result of measurement itself, the outcome will be a unit-less number, a ratio, which we call relative error. When multiplied by 100%, the relative error is called percent relative error.

For example, we have analyzed lead-tin solder for its lead content, and obtained the result of 80% lead by weight, with an absolute error of \pm 2% by weight. Now, if we want to find the relative error of this measurement, we must take the ratio of an absolute error to the result, that is: divide \pm 2% by 80%, which is equal to \pm 0.025, and which - after multiplying by 100% - may be expressed as \pm 2.5% relative. In other words, we can either say that we measured the concentration of lead in solder and obtained the result of 80% by weight with an absolute error of \pm 2% by weight, or that we obtained the result of 80% by weight with relative error of \pm 2.5%. This example shows the importance of using the term relative, especially when measuring percent concentration(s) of elements in sample.

Error of measurement has two fundamental, additive components: systematic error and random error.

Systematic error is constant in nature and is usually caused by inadequacy of calibration or gross and persistent mistakes during the measurement process (such as use of the wrong calibration curve, significant change in measurement conditions, etc.). A large systematic error is relatively easy to detect. It will manifest itself by producing an obviously erroneous result. However, a small systematic error can be detected only by measurement of a known, well-characterized standard sample(s) or certified reference materials (CRM). Systematic errors can usually be corrected during the calibration procedure. Systematic error is also called a bias or inaccuracy of measurement.

Random error, often called imprecision of measurement, varies from measurement to measurement and expresses the variability of the measurement process when repeated under the same conditions and on the same object. Random error is due to randomly fluctuating factors that cannot be controlled or eliminated from the measurement process.

Thermo Scientific Niton® analyzers are calibrated at the factory to eliminate systematic errors or at least make them insignificant. Therefore, the errors associated with measured results are predominantly random in nature and mainly reflect the imprecision of the measurement process.

All analytical results obtained with any Niton XL3 Series analyzer are assumed to be bias free and are reported with error quoted at two standard deviations (colloquially known as "two sigma"), which approximately corresponds to a 95% confidence level. This rule follows the practice commonly accepted by analytical chemists. The 95% confidence level of measurement error means that if we measure concentration of lead at, for example, 3000 mg/kg with two sigma error of ± 50 mg/kg, then, with a probability of 95%, we can expect that the true value of lead concentration will be within a range of 2950 to 3050 mg/kg. In other words, if we repeated the lead analysis 1000 times, 950 results (almost all) would fall within the range from 2950 to 3050 mg/kg and only 50 would not. Please note that error reported at one sigma corresponds roughly to a 68% confidence level. In practice, this means that almost half of the measured results would fall outside the ± one sigma band around the measured value. On the other hand, error reported at ± three sigma corresponds roughly to a 99.7% (practically 100%) confidence level that the true value of lead concentration will be between 2925 and 3075 mg/kg.

It is specific to x-ray fluorescence (XRF) spectrometry that imprecision of measurement error can be calculated from just one measurement. [All other analytical methods require multiple measurements on a sample to determine the error.] It is also specific to XRF that the imprecision error can be reduced by extending measurement time. For each fourfold extension of measurement time, one can expect a twofold reduction of imprecision error.

QUANTITATIVE VS. QUALITATIVE RESULTS AND MEASUREMENT TIME, LIMITS OF DETECTION

Quantitative or Qualitative Results

It is an accepted, practical rule to consider the measurement result as quantitative if the relative error of measurement (commonly two standard deviations, or sigmas) is not greater than 20% (or 1/5) of the reported result. If the relative error is greater than 20%, the result is considered qualitative or semi-quantitative at best, and should not be used as the basis of quantitative decisions (such as compliance with regulatory thresholds).

For example, if we measure Cd in polyvinyl chloride (PVC) for 120 seconds, and the result and error are 237 mg/kg and ±34 mg/kg respectively, this result may be considered quantitative, because two sigma, 34 mg/kg, is less than 20% of the result (here 47 mg/kg). On the other hand, if we measure for only 20 seconds, the error may be as high as ±78 mg/kg. In a case such as this, the relative error will be greater than 47 mg/kg, rendering the result semi-quantitative at best. This example illustrates how, by adjusting the measurement time, it is often possible to convert a qualitative result into a quantitative one.

The error of a single measurement is highly dependent on sample density and measurement time. As we have seen, a longer measurement time will yield a smaller reported error. To a first approximation, each fourfold extension of measurement time yields a twofold reduction of imprecision error. Therefore, when measuring an unknown sample, the user should observe the values of error reported by the instrument and continue analysis until the error reported is less than 1/5 of the result. Therefore, the answer to the commonly asked question of "How long do I need to measure a sample?" is "Measure at least as long as is needed to obtain quantitative result."

Generally, it is a good practice to take several trial measurements before attempting a meaningful measurement of an unknown material. These trial measurements help estimate the measurement time needed for quantitative results.

For Restriction of Hazardous Substances (RoHS) screening, a 30-second minimum analysis is typically required to determine whether the RoHS elements, with limits at 1000 ppm, are compliant or non-compliant. If a 30-second reading yields non-compliant results for Pb, for example, then a longer reading time is not required because it would yield a more precise, but still non-complaint result. If a 30-second analysis yields compliant results for some, but not all, of the RoHS elements, then a longer analysis time may be required to obtain a compliant result. Note that this is typically the case for Cd because it is has a RoHS action level of 100 ppm.

Limit of Detection

The Limit of Detection parameter describes the ability of the analyzer or analytical method to positively affirm the absence or presence of analyte(s) in a sample. Limits of Detection (LODs) used in Niton XL3 Series analyzers are specified following the US Environmental Protection Agency (EPA) protocol of 99.7% confidence level. That is, the LOD for an individual analyte is numerically expressed as three standard deviations (or 1.5 times reported measurement error) measured on a sample with none or only a trace amount of the analyte present. An exception is metal alloys for which LODs are expressed at the more conservative level of four standard deviations. Since the individual LODs are based on measurement error, they also improve as a function of the square root of the testing time.

Due to the density of certain matrices, the detection limits of certain elements may be higher than the RoHS limits. One example would be Cd in a lead-free tin solder like a SAC305. Because of the high density of tin and the location of its x-ray energy in relation to Cd, the detection limit for Cd in this alloy can be as high as 500 ppm.

Obviously, the LODs are important only when measured analyte concentration is very low, or close to the LOD.

Example of a Single Measurement

Meas.	Calculated			Result reported on			
time	± 2 sigma	Measured	Calculated	the instrument			Quality of
[sec]	error	result	LOD	l ine	screen	the Result	
1	111	165	167	ND	<	167	ND
2	78	126	118	126	±	78	ND
3	64	93	96	ND	<	96	ND
4	56 50	103	83	103	±	56	QUALIT
5 6	50	86	74	86 99	±	50	QUALIT
7	45	99	68		±	45	QUALIT
	42	108	63 50	108	±	42	QUALIT
8 9	39 37	109	59 50	109	±	39	QUALIT
		101	56 50	101	±	37 35	QUALIT
10 11	35 33	97	53 50	97 103	± ±	33	QUALIT
	33 32	103		103	_		QUALIT
12 13	32 31	104 99	48 46	99	±	32	QUALIT
13		l	l	100	± ±	31 30	QUALIT
	30	100	44				QUALIT
15 16	29 28	100 103	43 42	100 103	±	29 28	QUALIT
		ı	l	99	±		QUALIT
17 18	27	99	40	101	± ±	27 26	QUALIT
	26 25	101	39		_		QUALIT
19 20	25 25	97 99	38 37	97 99	±	25 25	QUALIT
21	25 24	ı	l	102	± ±	23 24	QUALIT
22	24 24	102 101	36 35	101		24	QUALIT QUALIT
23	24	100	35	100	±	23	QUALIT
24		ı	l	99	± ±	23	
25	23 22	99 102	34 33	102	±	22	QUALIT
26	22	98	33	98	±	22	QUALIT QUALIT
27	22	99	32	99	±	21	
28	21	99	31	97	±	21	QUALIT QUALIT
29	21	100	31	100	±	21	QUALIT
30	20	l	30	_			
	20 20	101	30	101 100	±	20	QUANTIT
31 32		100		99	±	20 20	QUANTIT QUANTIT
	20	99	29		±		
33	19 19	101	29 29	101 99	±	19 19	QUANTIT
34 35		99		100	±	19 19	QUANTIT
36	19	100	28	100	± ±	19	
36 37	19	102	28	102		18	QUANTIT
38	18 18	100 100	27 27	100	±		QUANTIT
38 39		l		101	±	18 18	QUANTIT
40	18 10	101	27 26	99	± ±	18	QUANTIT
40	18	99	∠ზ	99	Ι	10	QUANTIT

Figure 128. Table 1. Measurement of Cd in Polyethylene (PE). (Results reported in mg/kg)

Table 1 above illustrates the results of a single measurement of a polyethylene (PE) sample that contains 100 mg/kg of Cd. All Niton analyzers report results in real-time, updating them every data acquisition cycle, which lasts about one second. The first column in the table shows the number of seconds elapsed from the start of the measurement. The second, third, and fourth columns show the results as calculated by the analyzer's internal software, while the

fifth column (all in bold face) shows the results as displayed on the instrument screen. Note that after the first and third cycle the calculated LOD was larger than the actual measured concentration of cadmium and, therefore, the instrument screen shows only the value of < LOD. Due to statistical fluctuations, the second cycle produced the result that was greater than the LOD, and the instrument reported measured value with its ±2 sigma error. However, the total period of the first three cycles, that is the first three seconds, are interpreted as yielding a result below detection limit. Only after the fourth cycle (second) is the error of measurement, and consequently the LOD, low enough for the result to be displayed as calculated. Over the consecutive cycles, up to the 29th second, the results can be considered only as qualitative because the reported error of two sigma is larger than 1/5 of the reported result. This period of qualitative results is marked with green color. Starting with the 30th cycle, the results are of quantitative quality (measurement at the 32nd cycle is borderline and is a perfect illustration of statistical fluctuations), and, as such, may be used for assessing compliance status. Please note that LOD is not assumed as constant, but is calculated anew after each data acquisition cycle.

In summary, this example shows that if we stopped the measurement after the third second, we would be able to say only whether Cd were present in the sample and whether its concentration were below 96 mg/kg. By continuing the measurement, we have seen that the measured Cd concentration stabilized around 100 mg/kg value; by waiting until the error dropped below 1/5 of the reported result, we have obtained quantitative concentration of Cd, which now could be compared with appropriate thresholds.

INTERPRETATION OF THE RESULTS FOR COMPLIANCE

In our case, the objective of testing for compliance is to ensure that the concentrations of specified elements in the product (material) do not exceed the thresholds set by regulatory bodies. Below we see an excerpt from the Consumer Product Safety Improvement Act of 2008 (CPSIA), which in paragraph 2A states that the total lead content by weight for any part of the product may not be more than 600 mg/kg.

TITLE I—CHILDREN'S PRODUCT SAFETY

SEC. 101. CHILDREN'S PRODUCTS CONTAINING LEAD; LEAD PAINT RULE.

(a) GENERAL LEAD BAN.—

(1) TREATMENT AS A BANNED HAZARDOUS SUBSTANCE.—
Except as expressly provided in subsection (b) beginning on the dates provided in paragraph (2), any children's product (as defined in section 3(a)(16) of the Consumer Product Safety Act (15 U.S.C. 2052(a)(16))) that contains more lead than the limit established by paragraph (2) shall be treated as a banned hazardous substance under the Federal Hazardous Substances Act (15 U.S.C. 1261 et seq.).

(2) LEAD LIMIT.—

(A) 600 PARTS PER MILLION.—Except as provided in subparagraphs (B), (C), (D), and (E), beginning 180 days after the date of enactment of this Act, the lead limit referred to in paragraph (1) is 600 parts per million total lead content by weight for any part of the product.

Numerically, this condition is expressed as:

measured lead concentration, [mg/kg] ≤ 600 [mg/kg]

9 Consumer Goods Analysisniton Appendix B

We will now discuss the meaning of this condition in terms of actual measured results and making compliance decisions.

First, we will analyze the decision-making process when the results are matched against a single value threshold. Figure 25 below illustrates several possible outcomes of lead measurement.

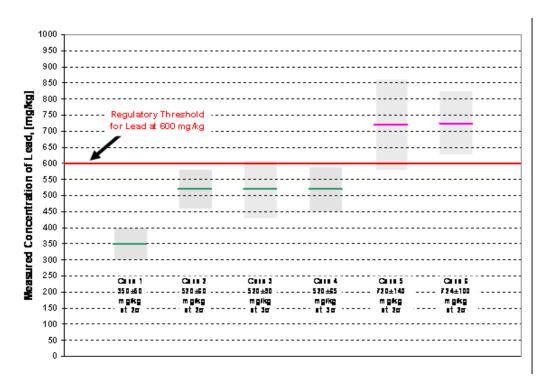


Figure 129. Results of measurement compared to a single value threshold.

Case 1 – In this case, the measured concentration of lead was equal to 350 mg/kg and the absolute error of this result at two sigma level (95% confidence) was ± 50 mg/kg. This is shown in Figure 1 with a green solid line at 350 mg/kg value and by a light green band stretching 50 mg/kg above and below the solid line. Based on the previous discussion, this result is definitely quantitative, meaning that we can say with 95% confidence that a true concentration of lead is within 300 to 400 mg/kg. As we can see, even the upper value of the range (400 mg/kg) is way below the regulatory threshold of 600 mg/kg lead, which is shown in Figure 1 with a thick red line. Therefore, we can make a statement that the object measured for lead is compliant with regulations; that is, its lead content is way below the regulatory level. The decision is PASS.

Case 2 – In this case, the measured concentration of lead is 520 mg/kg with an absolute error of two sigma equal to ± 60 mg/kg. Again, this result is quantitative, and it means that we can say with 95% confidence that the true concentration of lead is between 460 and 580 mg/kg. Therefore, since the upper limit of the error band is less than 600 mg/kg, we will consider the result as compliant. The decision is PASS.

Case 3 – This case is identical to Case 2 except that the error band around the measured result this time is equal to three sigma (three standard deviations). Therefore, this time we can say with 99.7% confidence that the true concentration of lead is somewhere between 430 and 610 mg/kg. Now the upper limit of the error band, 610 mg/kg, is greater than the threshold value of 600 mg/kg and we have to consider the object as noncompliant. The decision is FAIL.

Case 4 – This case presents another variant of Cases 2 and 3. Here, we decided to double the measurement time, which resulted in the reduction of error by the square root of 2 or by about 1.41. Now, the three sigma error is only 65 mg/kg, as shown by the light green box around the value of 520 mg/kg. The consequence of doubling of measurement time is a more precise estimate of lead concentration, which now can be found with 99.7% confidence within the range from 455 to 585 mg/kg. Here we can say that the object tested contains lead below the regulatory level of 600 mg/kg and is compliant. The decision is PASS.

Case 5 – The lead is measured at 720 mg/kg with an absolute error of two sigma equal to ±140 mg/kg. This is a quantitative result implying with 95% confidence that the true value of lead concentration is between 580 and 860 mg/kg. Note that the lower limit of the error band, 580 mg/kg lead, is now smaller than the threshold of 600 mg/kg, suggesting that perhaps the true concentration of lead is less than 600 mg/kg. While plausible, it is not very likely. In this case, it is more likely that the true concentration is greater than 600 mg/kg, and we decided that the tested object is not compliant. The decision is FAIL.

Case 6 – This is a follow-up to Case 5. Simply put, by extending the measurement time, we can make the result more precise before comparing it with 600 mg/kg threshold. As shown, now the measured result for lead is 724 mg/kg with a two sigma absolute error equal to ±100 mg/kg. Now, both the upper and lower limits of the error band are above the 600 mg/kg threshold, and the result is definitely noncompliant with 95% confidence level. In other words, in comparison to Case 5 we have obtained more precise but still noncompliant result. The decision is FAIL.

Conclusions

- 1. Whenever the result of a measurement plus its absolute error at the accepted confidence level (of two or three sigma) is not greater than the regulatory threshold, the result may be considered as compliant.
- 2. If the result of a measurement is smaller than the regulatory threshold but the upper limit of its error band is greater than the regulatory threshold, then the result may be considered as noncompliant. Alternatively, one may perform more precise measurement with a narrower error band, which will either confirm the previous decision or determine that the product compliant.
- 3. If the measured result is greater than the regulatory threshold and the lower limit of its error (at the accepted confidence level of two or three sigma) band is also greater than the threshold, the result is considered noncompliant.

9 Consumer Goods Analysisniton Appendix B

4. When the measured concentration is greater than the regulatory threshold but the lower limit of its error band is below the threshold, then the result may be labeled as noncompliant. Alternatively, performing a more precise measurement resulting in a narrower error band will either confirm the previous decision or determine the product as compliant.

By now it should be clear that to decide whether the measured concentration of an element in a sample does or does not exceed the regulatory concentration threshold, one must include the error of measurement in comparison. The decisions are then made with the confidence level adopted for the error of measurement. That is, if the error is reported at two sigma level, which corresponds to 95% confidence, the resulting compliance decisions are made with 95% confidence that they are correct. If the error is reported as three sigma, which corresponds to 99.7% confidence, we then have 99.7% confidence that the compliance decisions are correct.

Note For error reporting purposes, the user can select one, two, three, and even four standard deviations (sigmas) on their Niton XRF analyzers. However, for compliance determination (PASS/FAIL, as reported on the analyzer), the instrument software always uses three standard deviation (sigma) errors.

Next we will consider an example where the compliance threshold is specified as a range rather than as a single value. If the tested materials are homogeneous, uniform, and in all other aspects ideal samples for XRF analysis, the single value of a threshold may be adequate. However, the majority of testing is performed on finished products that rarely fulfill the conditions of perfect samples for XRF analysis. The results of tests on such non-ideal samples may affect the accuracy of results, which, in turn, may lead to wrong decisions. To prevent or at least minimize this occurrence, we can adopt more stringent criteria for rendering PASS/FAIL decisions. Instead of using the regulatory threshold for PASS, we could adopt a new, lower threshold for PASS. For example, instead of 600 mg/kg lead, we could use a value 30% smaller or 420 mg/kg. Similarly, for the FAIL criterion, we might use a value which is 30% higher than 600 mg/kg or 780 mg/kg. This would create a band of uncertainty that would make some of decisions INCONCLUSIVE, that is, neither PASS nor FAIL. This condition is illustrated in Figure 26. This "band-like" criterion has been suggested in the Test Method IEC 62321 for compliance testing under the EU RoHS Directive.

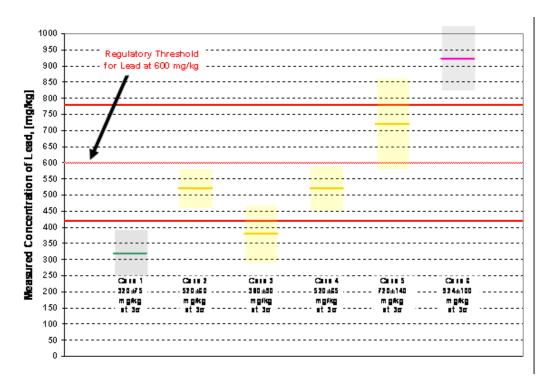


Figure 130. Acceptance band and measured results. All error bands around measured values are at three sigma (99.7% confidence).

Case 1 – The result and its 3 sigma error band never exceed the lower threshold level of 420 mg/kg. Therefore, the test result shows the tested object as compliant. Decision is PASS.

Case 2 – The result and its error band are above the lower threshold, but below the upper threshold. This makes the result INCONCLUSIVE. Since the measured value is well within a band, it is doubtful whether the more precise test would yield a conclusive result. Therefore, either we can make an arbitrary decision that the result implies noncompliance (FAIL), or we can analyze the object using another, much more accurate method to determine whether the lead concentration is below 600 mg/kg (and not below 420 mg/kg!).

Case 3 – Although the error band encroached over the lower acceptance threshold, it is warranted that we expect that a longer, more precise measurement might end with both the result and its error band falling below the lower acceptance threshold. As is, this measurement is INCONCLUSIVE.

Case 4 – This is identical to Case 2.

Case 5 – The conclusion is the same as Case 2.

Case 6 – The result and its error band are decisively above the upper acceptance threshold of 780 mg/kg. Therefore, the result is conclusive and the decision is FAIL.

9 Consumer Goods Analysisniton Appendix B

As illustrated in the above example, we could simplify the interpretation of test results by adopting only the lower threshold value as a criterion and regard all cases except for Case 1 as noncompliant. Then, the situation would be the same as we saw in Figure 1, except the comparisons would be made against the new, lower value of 420 mg/kg rather than 600 mg/kg. All conclusions based on Figure 1 would apply for here at the lower acceptance threshold.

Note that the values of lower and upper acceptance thresholds were selected arbitrarily and are not related to measurement error.

Last, there is one more situation that deserves mention, which is when the acceptance threshold is close to the detection limit. In such instances, the result is conclusive and the decision would be PASS if the measured LOD is smaller than the threshold. In all other cases, the result would be FAIL.

Spectral Fingerprint

Every instrument is supplied with the Spectral Fingerprint Mode which allows for the spectral comparison of any unknown sample with a known material. From the main menu select the Sample Type icon, then the Spectral Fingerprint icon.

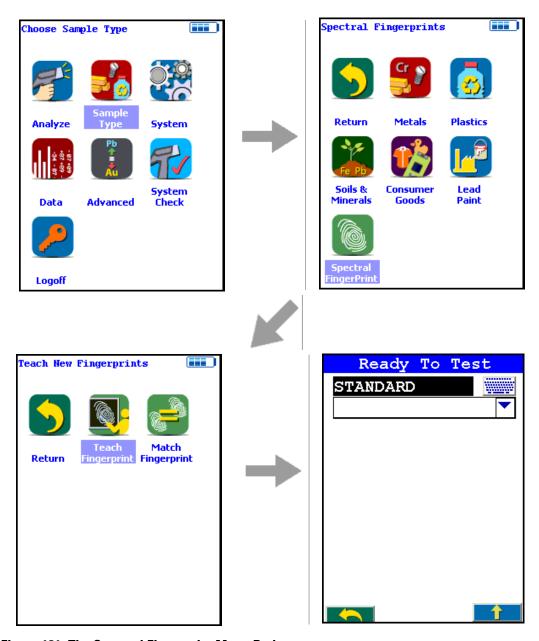


Figure 131. The Spectral Fingerprint Menu Path

10 Spectral Fingerprint

Select the Teach Fingerprint icon and then enter name or ID of the material into the Standard box. All Fingerprint names must be unique. Start analysis and run for a minimum of 60s to ensure the instrument collects a representative spectrum with good statistics. The screen will show elements and readings in cps/ μ A for a number of elements. After analysis the system will ask if you want to save the reading, answer yes. Analyze and save as many different types of material as you wish.

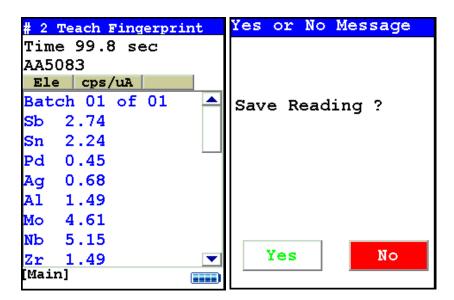


Figure 132. Teaching the Fingerprint

After saving the fingerprints of the sample you want identified, it is possible to then run unknown samples and determine if there is a correlation between them and the stored fingerprint. Click on the Match FingerPrint icon, enter sample ID if required and start analysis. At the end of the analysis, the instrument will display the FingerPrint match name, and give an indication of how close the spectrum is to the original reference material. No chemical concentrations are given in this mode, simply an indication of how close a sample is to a reference material.

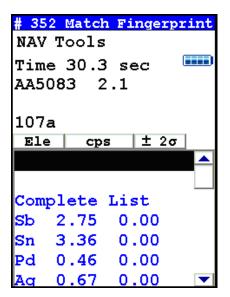


Figure 133. Matching the Fingerprint

10 Spectral Fingerprint

Helium Purged Analysis

Attaching the Helium Supply to Your Analyzer

Make sure before you start that you are using a clean Prolyne Measurement Window intended for use with He-purged analysis, rather than the standard Polypropylene Measurement Window. Securely attach the regulator accompanying your analyzer to the helium tank. This custom regulator governs the helium flow to 70 cc/min flow rate. Confirm that the helium tank contains a sufficient supply for your testing purposes.





Figure 134. The Helium Tank Setup - Inset Showing Regulator Dial

- 1. Check the line for kinks, knots, or other obstructions before turning on helium flow.
- 2. Attach the ferrule, shown in Figure 2, to the Helium Input Port, shown in Figure 3.



Figure 135. The Helium Ferrule



Figure 136. The Helium Input Port

3. Snap the Locking Clip into the Locking Groove, securing the ferrule to the instrument, as in Figure 4.

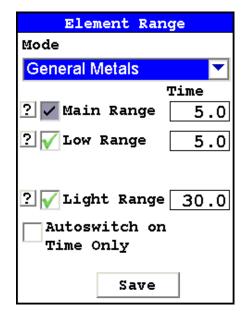


Figure 137. Ferrule Attached and Locked

4. Turn on helium flow. Allow the helium to purge the instrument's front end for a minimum of five minutes before starting analysis. Helium purging can be done at the same time as instrument warm-up.

He-Purged Analysis

Helium purge is required for optimum detection of light elements such as Mg, Al, Si and P. For analyses using helium, we recommend using the default Range setup, as shown below in Figures 5 and 6. As with other energy dispersive XRF analyzers, the longer the testing time, the better the statistical precision of the reading. See Adjusting the Element Range for more on setting Ranges.



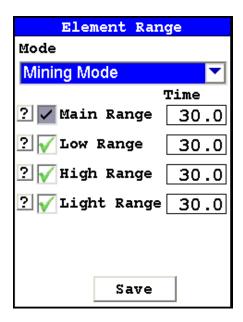


Figure 138. The Default Range Setups for Metals and Mining

The user can see the precision improving with analysis time by watching the third column on the analysis display – the 2-sigma error. This value decreases as reading time increases. Knowledge of this relationship is important, especially for light element work.

See Metals Analysis and/or Mining Analysis for more information on sample analysis. See also Avg Forward, Spectrum:On/Spectrum:Off, Enable LEC/Disable LEC, and Set Pass/Fail for more information on specific aspects and methods of analysis.

Table 1 shows the approximate limits of detection of the light elements in various matrices. If testing time is shortened, the limits of detection (which are directly based on statistical precision) will increase.

Table 8. Approximate LODs for Light Elements in Fe and Al Matrices

Element	Fe Matrix (concentration %)	Al Matrix (concentration %)
Zn	0.015	0.010
Cu	0.030	0.030
Ni	0.050	0.010
Cr	0.015	0.015
V	0.010	0.010
Ti	0.010	0.010

Table 8. Approximate LODs for Light Elements in Fe and Al Matrices

Al	0.031	-
Si	0.075	0.150
Mg	1.300	0.650

The above LODs were calculated with 60 sec per filter measurement times

Sample Requirements

- 1. Must be flat, sit directly on top of and cover the entire measurement window.
- 2. Surface must be clean and free of oxidation or other surface films.

See Metal Sample Prep for more information for metal sample preparation, and the Mining Analysis SOP for more information on preparing mining samples.

Alloy Grade Libraries

Using a library of alloy grade specifications, your alloy analyzer will attempt to identify an alloy sample being analyzed. It does this by comparing the chemistry analysis results against the minimum and maximum specifications in the library. The library is an .alb or .clb file, and is viewable, editable, and upload-able via the included NDT program. Your analyzer can store two alloy grade libraries in its memory, and automatically chooses one of the two for use while testing. See Managing Libraries for information on how to manage your Libraries.

Selecting the He Library

The 900 library will be auto loaded when the Light Range is selected. When the Light Range is de-selected, the standard 800 library will autoload. Selecting Switch Library (Main) from the Tools Menu will not work. It is disabled in favor of the autoloading. Alternately, you can select Toggle Helium from the Tools Menu. In non-GOLDD analyzers, this will select the Light Range and change to the 900 Library. Selecting it again will de-select the Light Range and change to the 800 Library.

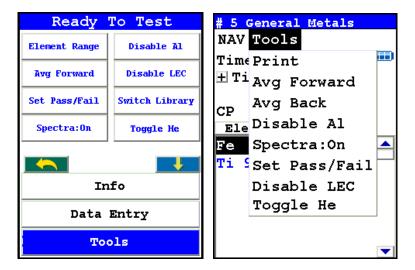


Figure 139. Selecting Toggle He from the Tools Menu and Alt Tools Menu

In GOLDD analyzers equipped with Helium Purge, selecting the Toggle Helium function from the Tools Menu will not deselect the Light Range. Instead it switches between Helium and Non-Helium applications - the Light Range remains enabled. You may also wish to use the analyzer without the Light Range activated. To do this, deselect the light range via the Tools Menu, as shown in Figure 1-7. To use the analyzer this way, be sure helium has been completely purged from the system.

Note When switching between Helium and Non-Helium applications on a GOLDD analyzer, allow the helium to dissipate out of system for at least ten minutes before beginning non-helium analysis. To expedite this process, you may remove the Measurement Window bracket for three or four minutes instead.

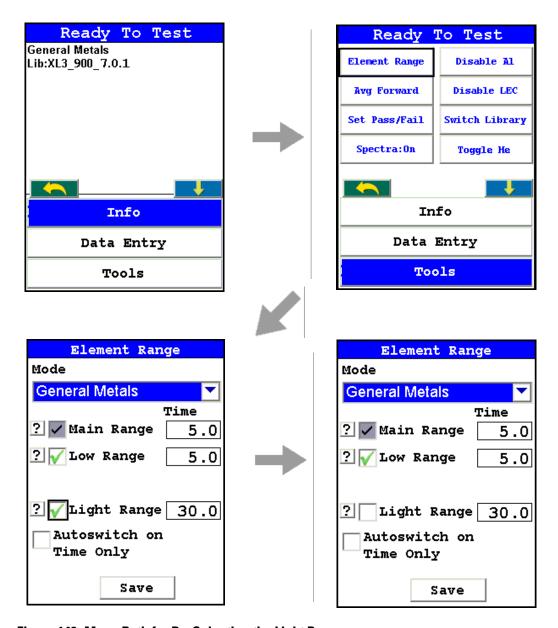


Figure 140. Menu Path for De-Selecting the Light Range

GOLDD and He-Purged Analysis Results

When using GOLDD technology analyzers and/or optional He-Purged analyzers, your results screen may look very different from the examples in this User's guide, due to the general lack of elements below the LOD. Here are two analysis screens showing GOLDD analysis, for comparison.

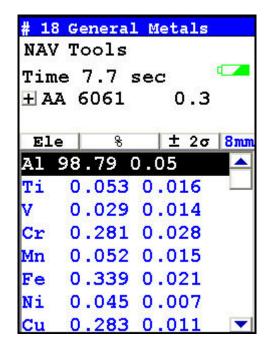


Figure 141. All Alloy Analysis of Al 6061, Showing Fast ID of Al Alloys (Light Range Disabled)

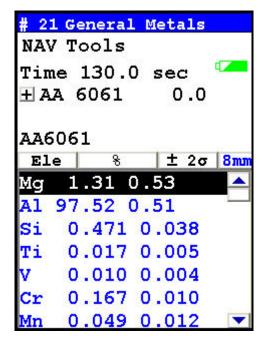


Figure 142. All Alloy Analysis of Al 6061, Showing Long Analysis of Composition (Light Range Enabled)

Data Management

Viewing Data

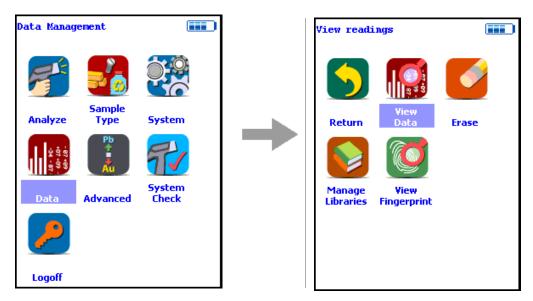


Figure 143. The View Data Menu Path

Use the Data Screen to view previously taken test result readings. When the View Data icon is selected, the Results screen of your most recent test is shown on the Touch Screen.

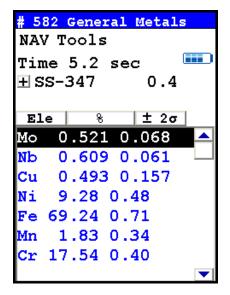


Figure 144. The View Data Screen

Using the buttons on the control panel, you may view different readings or additional data for individual readings. Your analyzer will display the standard screen analysis. Pressing the Down Button on the 4-way touch pad will display a complete scrolling elemental chemistry listing. Each press of the Down Button scrolls the screen down to the next element. You can also use the scroll bar along the right side to scroll or page through the elements.

Scrolling Down Through the Complete Listing of Elements

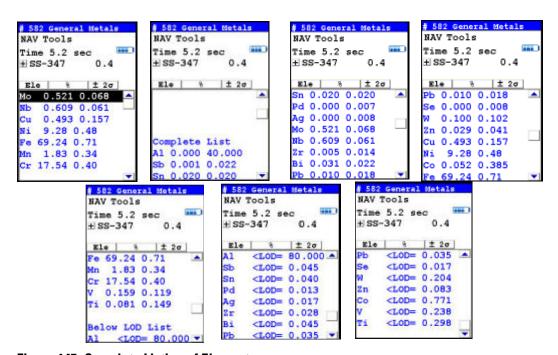


Figure 145. Complete Listing of Elements

Pressing the Left Button on the 4-way touch pad of your analyzer will display the previous reading, or if the first reading is currently displayed, the last reading. Pressing the Right Button on the 4-way touch pad will display the next reading, or if the last reading is currently displayed, the first reading in memory. Your analyzer can store up to 10,000 readings. You can also look at the complete x-ray spectra for each reading stored in the analyzer's memory.

Sorting Elements

You can sort element rows by various criteria in order to view your data in the manner you prefer. The Sort Buttons, which double as column headings, can be used to re-sort the data in different ways. The Data Screen always begins as a Standard Sort, as you have defined it. Selecting the appropriate Sort Button once toggles the sort order to High-to-Low. Selecting the Sort Button again toggles the sort order to Low-to-High. To return to the Standard Sort, select the Sort Button a third time.

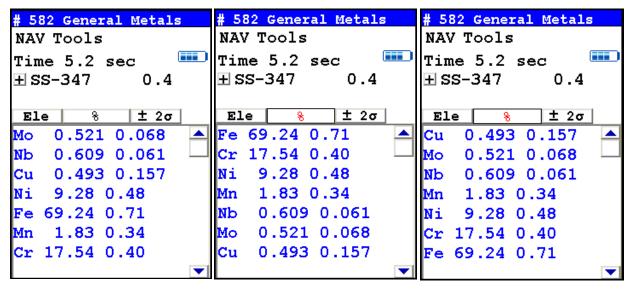


Figure 146. Standard, High-to-Low, and Low-to-High Composition Sorts

Element Sorts

Element sorts are performed alphabetically based on the element symbol.

Composition Sorts

Composition sorts are performed numerically based on the percentage of composition, i.e. from lowest to highest concentration, or by toggling again, from highest to lowest.

Error Sorts

Error sorts are performed based on the size of the error in the reading, i.e. from largest to smallest error, or by toggling again, from smallest to largest.

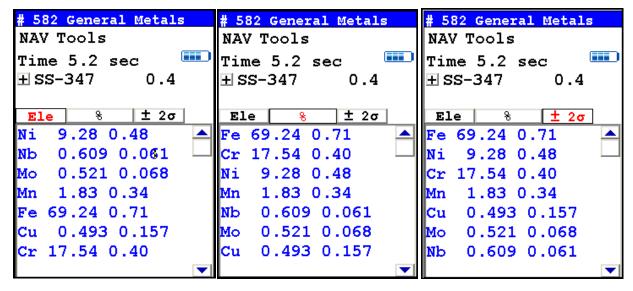


Figure 147. Element, Composition, and Error Sorts

Spectrum Graph

For any reading result, simply use the NAV Menu to gain access to the reading's spectrum graph. Selecting Spectra will show a graphed spectrum of this reading, called SpectraView. SpectraView can be a useful tool for rapid, qualitative analysis of a sample. See Viewing the Spectrum for details.

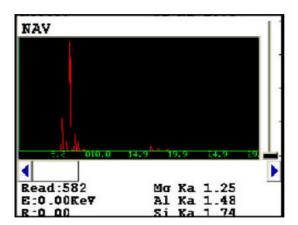


Figure 148. The SpectraView Screen

Viewing the Spectrum

SpectraView

SpectraView enables you to to visually inspect the fluorescent x-ray peaks obtained from any sample and qualitatively identify them using the on-board software. In SpectraView Mode, the spectrum is displayed using a linear energy scale along the x-axis, with the count rate autoscaled logarithmically on the y-axis so that the highest peak on the screen reaches the top of the scale.

How to Use SpectraView

You can access the SpectraView screen after taking a measurement in any mode, or while viewing a previous measurement, by selecting Spectra from the NAV Menu. Once you are in SpectraView, you can use the up and down positions of the 4-way touch pad to scroll through the spectrum, or you can tap on the spectrum display with the stylus to place the cursor at the point you tapped. The vertical cursor line indicates the current position along the spectrum.

Viewing the Information in SpectraView Mode

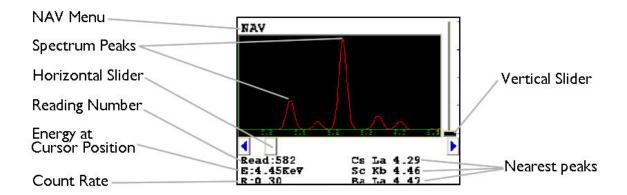


Figure 149. The SpectraView Screen

By default, the following information is shown along with the spectrum:

- The Reading number (Bottom Left) in the form "Read:x", where x is the Reading number.
- The position of the cursor on the energy scale (Bottom Left, under the Reading number), in the form "E: x.xx KeV", where KeV is kilo electron volts.
- The count rate (Bottom Left, under the energy position), in the form "R:x.xx".

• Ka, Kb, La, Lb, and/or Lg peaks of the three elements closest to where your cursor is positioned on the energy scale (Bottom Right). This information is written with the element symbol first, followed by either Ka (K shell alpha peak), Kb (K shell beta peak), La (L shell alpha peak), La (L shell beta peak), or Lg (L shell gamma peak). An example would be "Al Ka 1.48." To determine if a given element is present, look at the count rate at that cursor position.

Note SpectraView cannot be used to determine exact element percentages in a sample.

Fitting the Spectrum

By using the touch screen, you can select parts of the displayed spectrum and zoom in. Touch and hold the stylus to the screen immediately before the area of the spectrum you wish to enhance, then - still holding the stylus to the screen - sweep it across the area of the spectrum you wish to see closer, lifting the stylus from the screen when you pass the end of the area of interest. The screen will display vertical lines to either side of the area of interest, delineating the boundaries of the area.

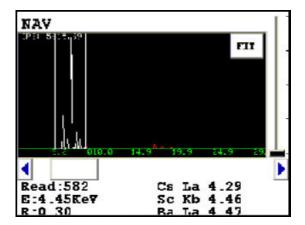


Figure 150. Delineating the Area of Interest

Select the FIT button in the upper right hand corner of the Spectrum to fit the area of interest to the display area.

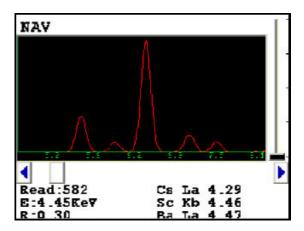


Figure 151. Area of Interest Fit to the Display

The view of the spectrum will change to show only the area of interest.

Multiple Ranges

SpectraView can display any individual spectra, including those obtained from multiple Ranges (filters) if you are using more than one Range. Use the NAV Menu to select which spectrum to view.

The Spectra1 choice will display the spectrum produced by the first Range.

The Spectra2 choice will display the spectrum produced by the second Range.

SpectraView Navigation

Use the left button on the 4-way touch pad to expand the spectrum, centered on the position of the cursor.

Use the right button on the 4-way touch pad to contract the spectrum, centered on the position of the cursor.

Viewing Fingerprints

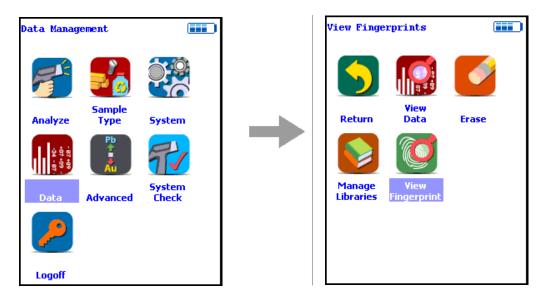


Figure 152. The View Fingerprints Menu Path

Select the View Fingerprints icon to view data saved as reference sample Fingerprints in Teach Fingerprint Mode. When the View Fingerprints icon is selected, the Results Screen of your most recent Teach Fingerprint is shown on the Touch Screen display.

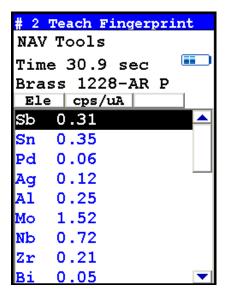


Figure 153. The View Fingerprints Screen

Erasing Data

The Erase All Data Screen

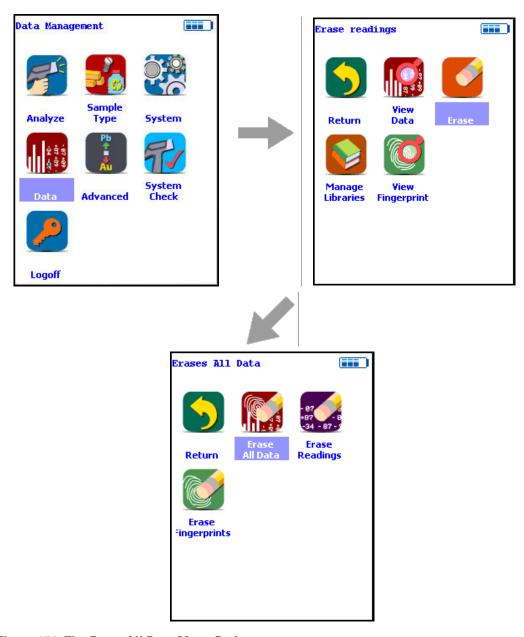


Figure 154. The Erase All Data Menu Path

Select the Erase All Data icon to erase all data, including signatures and readings, from your analyzer. Selecting the Erase All Data icon will bring up a confirmation screen asking you "Are you sure?" with options to select "YES" or "NO". Selecting the Yes Button will erase all data from your analyzer. Selecting the No Button will return you to the Erase Menu.

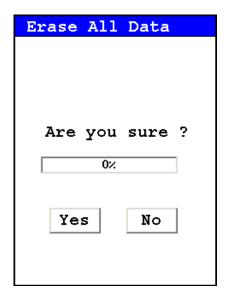


Figure 155. The Erase All Data Confirmation Screen

CAUTION Never turn off the analyzer while data is being erased!

WARNING Do not attempt to take measurements while downloading readings! This will generate an error requiring a system reset, and may corrupt your stored readings, requiring all stored readings to be erased.

The Erase Readings Screen

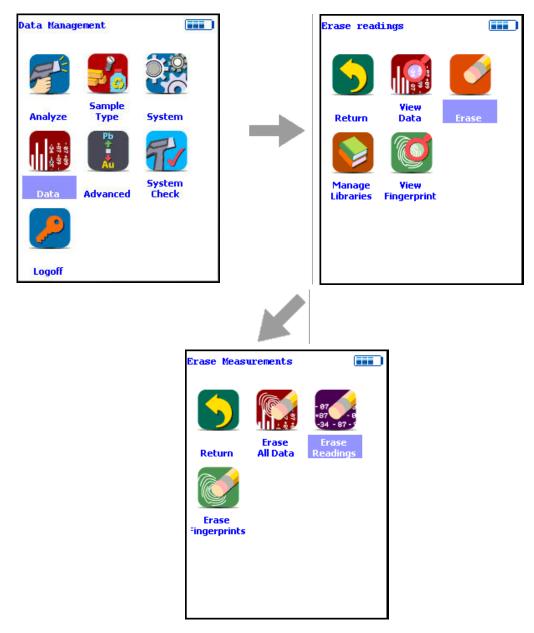


Figure 156. The Erase Readings Menu Path

Select the Erase Readings icon to erase all accumulated test readings from your analyzer. Selecting the Erase Readings icon will bring up a confirmation screen asking you "Are you sure?" with options to select "YES" or "NO". Selecting the Yes Button will erase all test reading data from your analyzer. Selecting the No Button will return you to the Erase Menu.

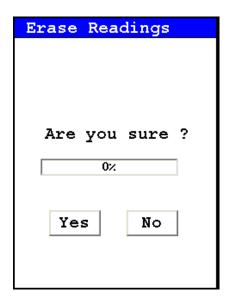


Figure 157. The Erase Readings Confirmation Screen

Note - We recommend that you download all your readings into an NDT file for recording purposes before erasing all data.

The Erase Fingerprints Screen

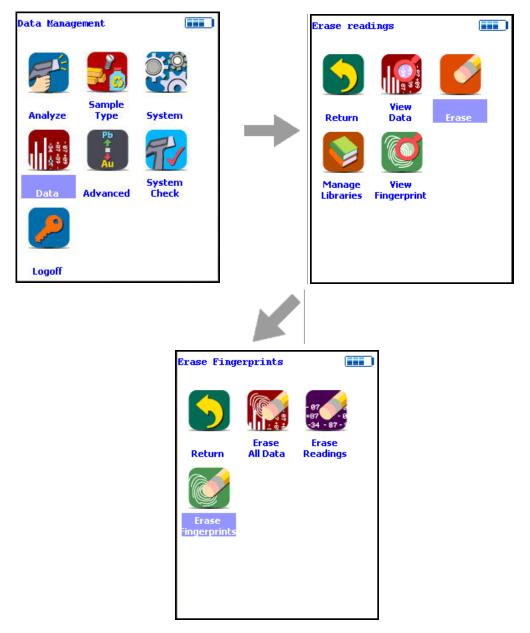


Figure 158. The Erase Fingerprints Menu Path

Select the Erase Fingerprints icon to erase all accumulated alloy fingerprints from your analyzer. Selecting the Erase Fingerprints icon will bring up a confirmation screen asking you "Are you sure?" with options to select "YES" or "NO". Selecting the Yes Button will erase all fingerprint data from your analyzer. Selecting the No Button will return you to the Erase Menu.

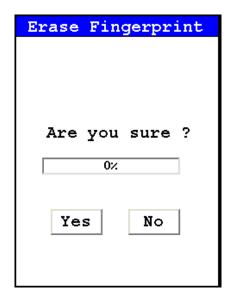


Figure 159. The Erase Fingerprints Confirmation Screen

Managing Libraries



Figure 160. The Manage Libraries Menu Path

Select the Manage Libraries icon to access the Library Management Menu. The Library Management Menu allows you to view and modify data in the Primary Library as well as the currently loaded alternate libraries. Just select the library you wish to view or editfrom the list on screen.

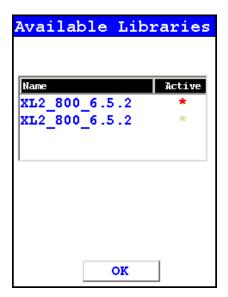


Figure 161. Viewing the Libraries

The entries in the Grade Library serve as a reference for chemistry based analysis. The library entries allow the analyzer to work properly "out of the box" without needing time-consuming pre-analysis.

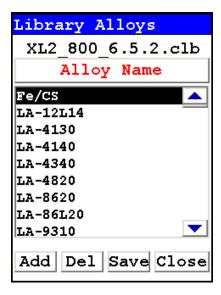


Figure 162. The Library Editor

Using the Library Editor

The Library Editor enables you to edit any library to conform to your specifications.

Alloy Name Button

Selecting the Alloy Name Button sorts the alloy list alphanumerically.

(Name in List)

Selecting the actual name of the alloy - i.e. "Fe/CS" - will bring up the Element Specification Screen.

Add Button

Selecting the Add Button will add a new alloy to the Library. First the Alloy Name Editor will appear, enabling you to enter the name of the new alloy.

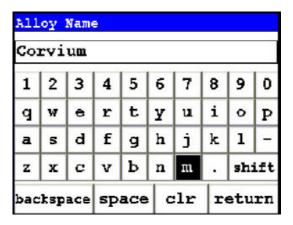


Figure 163. The Alloy Name Editor

The Alloy Name Editor is a standard Virtual Keyboard. Use it as you would any Virtual Keyboard. Hitting the return key enters the name into the alloy list. Select the name of the new alloy to bring up the Element Specification Screen and enter the specification of the alloy.

Del Button

Selecting the Del Button will delete the currently selected alloy. First a confirmation screen appears.

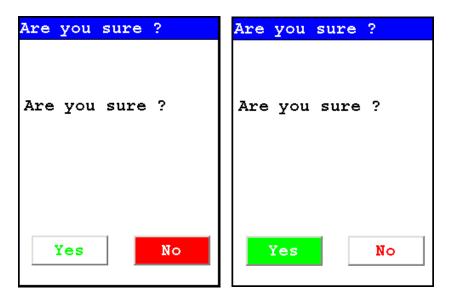


Figure 164. Confirmation Screen

Selecting the Yes Button will delete the alloy from the list. Selecting the No Button will return you to the Alloy List.

Save Button

Selecting the Save button will save the current Library.

Close Button

Selecting the Close button will close the current Library without saving it.

The Element Specification Screen

The Element Specification Screen allows you to edit the elemental content of any alloy.

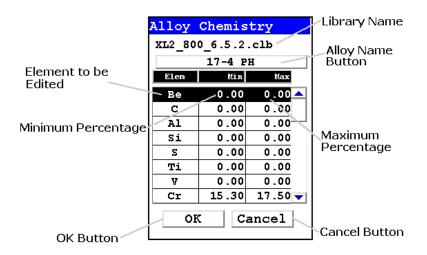


Figure 165. The Element Specification Screen

Library Name

This is the name of the library you are editing. Make sure you are editing the correct library before proceeding further.

Alloy Name

This is the name of the alloy you are editing. Make sure you are editing the correct alloy before proceeding further.

Element to be Edited

This is the element you need to edit for this alloy.

Minimum Percentage

This is the lowest amount of the element in question you want to be in the alloy. If the element in the analyzed sample is any lower, the sample will not be recognized as this alloy. Selecting the element minimum will open the Minimum Editor.

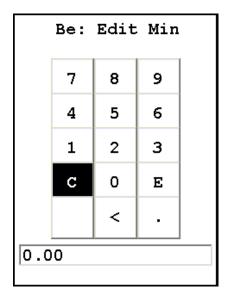


Figure 166. The Minimum Editor

This is a standard Virtual Numerical Keypad. The C Button will clear the current display, The < Button will backspace one space, and the E Button will enter this number as the minimum. After selecting the E Button, you will be returned to the Element Specification Screen.

Maximum Percentage

This is the highest amount of the element in question you want to be in the alloy. If the element in the analyzed sample is any higher, the sample will not be recognized as this alloy. Selecting the element maximum will open the Maximum Editor.

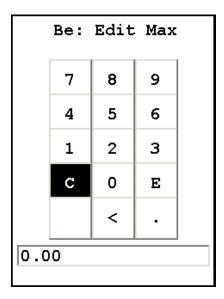


Figure 167. The Maximum Editor

12 Data Management Managing Libraries

This is a standard Virtual Numerical Keypad. The C Button will clear the current display, The < Button will backspace one space, and the E Button will enter this number as the minimum. After selecting the E Button, you will be returned to the Element Specification Screen.

OK Button

Selecting the OK button will save the edited library.

Cancel Button

Selecting the Cancel button will exit the Element Specification Screen for this alloy, returning you to the Library Editor.

Connectivity

This section discusses how to connect your computer and your analyzer, for data transfer and integration, translation to other formats, data storage and security, as well as controlling your analyzer remotely through your computer. Connection can be achieved via USB, serial, and/or Bluetooth wireless means. Your analyzer comes with software which facilitates these uses, and works together with your analyzer to increase your productivity and efficiency.

Using Your Analyzer With Your PC

Installing Niton Data Transfer Software

We recommend the following system configuration for Niton Data Transfer (NDT):

Table 9: System Configuration

	Minimum Configuration	Recommended Configuration
Processor	133 MHz	
Operating System	Windows 98, Millennium Edition, 2000, XP, Vista, Windows 7	Windows XP
Memory	64 MB	128 MB
Free Disk Space	10 MB	

The installation program installs NDT and NDTr. You may install the files in any directory on your PC.

To install Niton Data Transfer:

- 1. Close all the programs that you have open on your PC.
- 2. Insert the CD into the CD-ROM drive.

If the installation program starts automatically, follow the instructions on the screen. If the installation program does not start, go to the next step.

3. Double-click My Computer.



Figure 168. My Computer Icon

4. Double-click the Compact Disc icon:



Figure 169. Compact Disc Icon

5. Double-click the Setup Application icon.

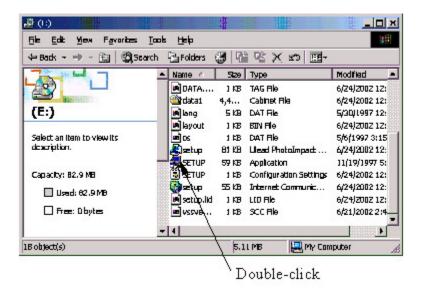


Figure 170. Setup Application Icon

6. Follow the instructions on the screen.

Starting Niton Data Transfer Software

To start Niton Data Transfer:

- 1. Click the Start button.
- 2. Select Programs.
- 3. Select Thermo Niton.
- 4. Click NDT.

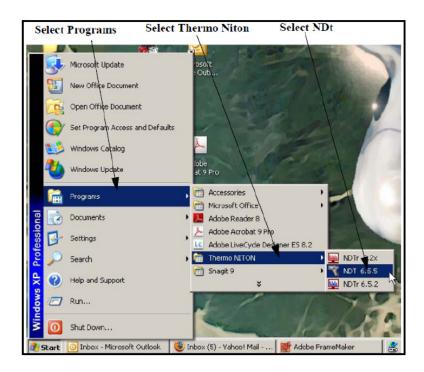


Figure 171. Starting NDT Up

Connecting the XRF Analyzer to Your PC

There are three ways to connect the XRF Analyzer to your computer:

- The serial cable provided. See <u>Using a Serial Cable to Connect the XRF Analyzer</u> for more information.
- The serial cable provided, along with a Serial-to-USB adapter. See Using a Serial-to-USB Adapter to Connect Your XRF Analyzer for more information.
- The wireless USB Adapter provided. See Using the Wireless USB Adapter to Connect the XRF Analyzer for more information.
- The USB cable provided. See Using a USB Cable to Connect Your Analyzer for more information.

Using a Serial Cable to Connect the XRF Analyzer

To connect the XRF Analyzer to your PC using the serial cable:

1. Insert the DB9 connector on the serial cable into the COM port on your computer.

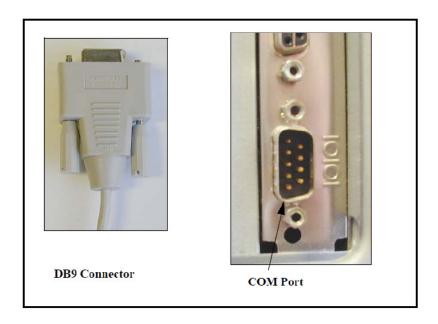


Figure 172. The DB9 Serial Connector and COM Port

Note The COM port has 2 rows of pins; 5 pins in one row, 4 pins in the other. Check that the holes in the DB9 connector align correctly with the pins in the COM port.

- 2. Tighten the screws of the DB9 connector with your fingers. You do not need to use a screwdriver.
- 3. Open the Port Cover on the XRF Analyzer.
- 4. Insert the micro stereo connector on the serial cable into the Serial port in the handle of the XRF Analyzer.

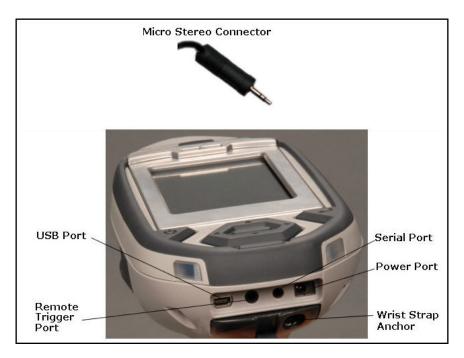


Figure 173. Micro Stereo Connector

Using a Serial-to-USB Adapter to Connect Your XRF Analyzer

If your PC has a USB port available, but does not have a COM port available, you can also use the serial cable to connect using the USB port. You will need a Serial-to-USB Adapter. This type of adapter lets you connect the DB9 connector to one end, and plug the other end into a USB port.

Contact the manufacturer of your PC to get the correct adapter. The manufacturer of your PC will be able to provide an adapter that includes the driver software that is appropriate for your PC.

Using the Wireless USB Adapter to Connect the XRF Analyzer

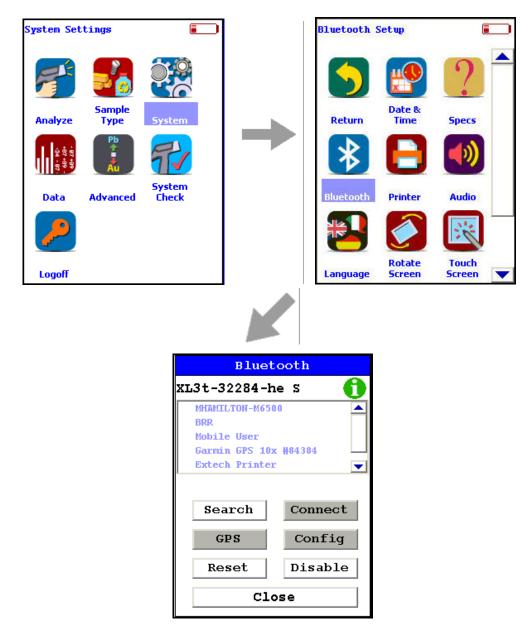


Figure 174. The Bluetooth Setup Menu Path

The USB adapter provided by Niton uses Bluetooth wireless technology. See Setting up Bluetooth to set up Bluetooth.

Select the Bluetooth icon from the System Screen to set up your analyzer for Bluetooth wireless connection.

i Icon

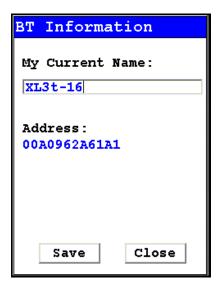


Figure 175. BT Information Screen

Selecting the i Icon in the top right of the Bluetooth Setup Screen will open the Bluetooth Information Screen. The Bluetooth Information Screen will supply the current name as well as the MAC address of your analyzer

Search Button

Selecting the Search Button will initiate a search for currently available Bluetooth devices in the area.

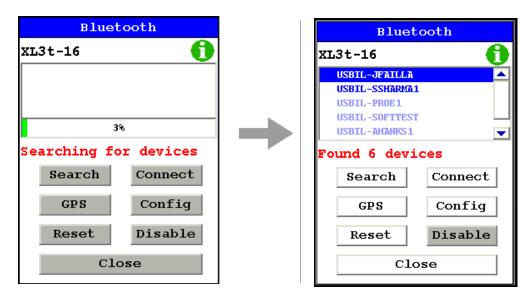


Figure 176. Bluetooth Searching

After the search, your analyzer will report which Bluetooth devices it has found in the main window of the screen.

You can connect your PC and analyzer two different ways, from the analyzer to the PC, and from the PC to the analyzer.

Connecting From Your Analyzer to Your PC

After searching, select the PC you would like to connect to from the main window of the screen.

Select the Connect Button. Your analyzer's screen will show connection progress.

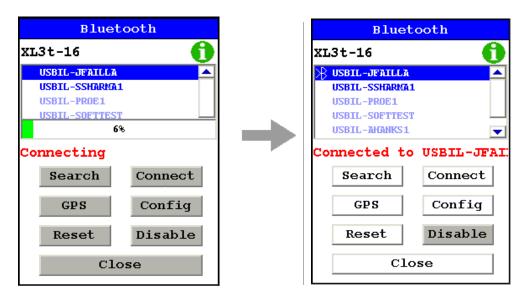


Figure 177. Connecting Via Bluetooth

Open the program you are attempting to connect to. Here we are connected to NDTr, running the analyzer remotely over COM 5



Figure 178. Connecting Via Bluetooth

Connect Button

Select a located Bluetooth device from the Search List, then select the Connect Button to connect to that device.

GPS Button

Select the GPS Button to download GPS data from a connected GPS Device.

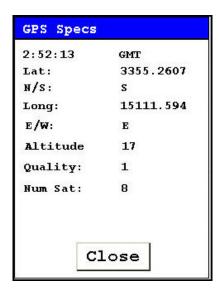


Figure 179. Bluetooth GPS Data Screen

Reset Button

Selecting the Reset Button initiates a Bluetooth reset.

Config Button

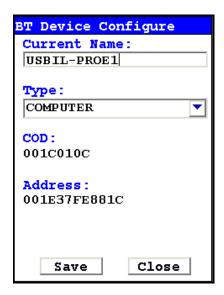


Figure 180. Bluetooth Config Screen

Selecting the Config Button will load the Config Screen. This screen enables you to se the name of the currently selected Bluetooth device, change the type of device, see that device's COD Number, and see that device's MAC Address.

Changing the Bluetooth Device Type

Select the Down Arrow button next to the Type field to change the type of device connected. Select the proper type from the drop down menu. Select the Save button to save this configuration. Select the Close Button to exit without saving.

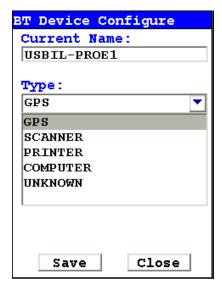


Figure 181. Changing Bluetooth Device Type

Disable/Enable Button

Selecting the Disable Button will disable Bluetooth neetworking and change the button to Enable. Selecting the Enable Button will restart Bluetooth networking and change the button back to Disable.

Close Button

Select the close button to exit from Bluetooth setup.

Using a USB Cable to Connect Your Analyzer

To connect the XL3 XRF Analyzer to your PC using the USB cable:

1. Insert the Standard USB connector on the USB cable into a USB port on your computer.

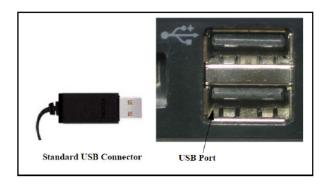


Figure 182. Standard USB Connector

- 2. Open the Port Cover on the XRF Analyzer.
- 3. Turn on the analyzer and insert the mini USB connector on the USB cable into the USB port in the handle of the XRF Analyzer.
- 4. Upon initial installation, insert the NDT disk located behind the foam in your case. Follow the prompts and install the USB driver located on the disk.



Figure 183. Mini USB Connector

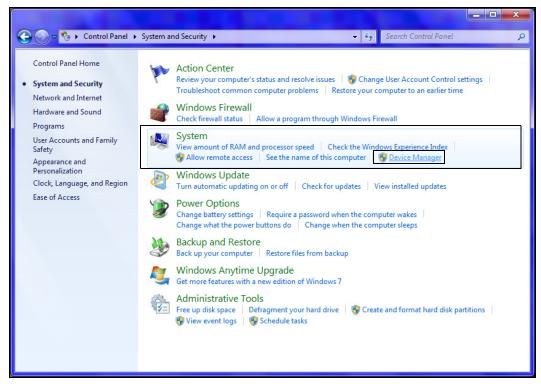
Installing the Windows 7 USB Driver

1. Insert the NDT CD and close out any dialogue box that pops up. The driver is located on this disk.

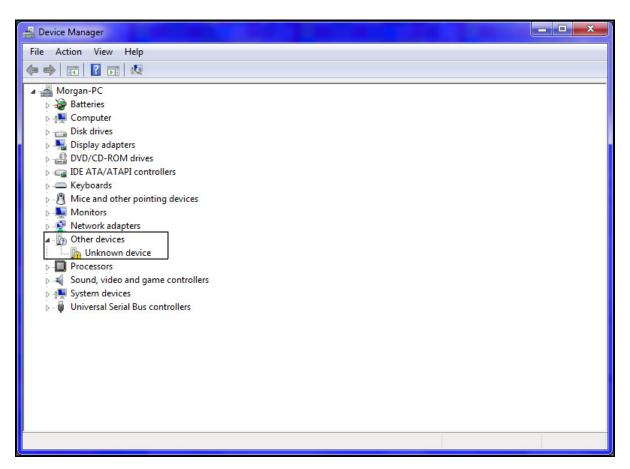


2. Click on "Control Panel" and locate the "Device Manager". If it is not available directly under "Control Panel", look under "System and Security" then "System".



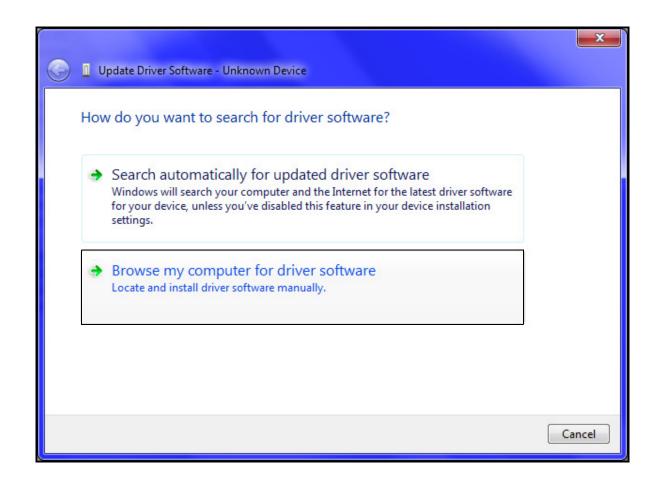


- 3. Open "Device Manager"
- 4. Plug in instrument using the USB cable provided
- 5. Message will appear "Device Driver Software Not Successfully Installed"
- 6. In "Device Manager", "Unknown Device" will appear under "Other Devices"

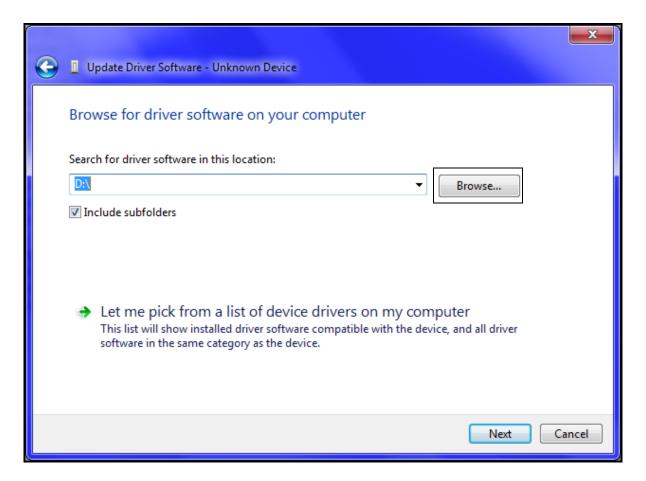


7. Right click on "Unknown Devices"; select "Update Driver Software"

8. Click on "Browse My Computer for Driver Software"



9. Click "Browse" button; select CD drive or the location of the driver if you are not installing from the NDT CD (recommended).



- 10. Click "OK"
- 11. Click "Next"

13 Connectivity Downloading Data

12. A Security Dialog Box will appear. Select "Install This Driver Software Anyway?"



13. Driver will install; close out.

Downloading Data

Standard Download

To download data you have collected offline:

- 1. Make sure that the XRF Analyzer is connected to your computer. See Connecting the XRF Analyzer to Your PC for more information.
- 2. Turn on the XRF Analyzer. See the manual for the XRF Analyzer for more information. **Note** Wait at least 30 seconds after turning on the XRF Analyzer to begin downloading files. The System Start screens do not allow downloading.
- 3. Start Niton Data Transfer. See Starting Niton Data Transfer Software for more information.
- 4. Click the Download button. The Download dialog box will open.



Figure 184. The Download Button

5. In the Download dialog box, Select the Test button to test the serial connection to the Analyzer.

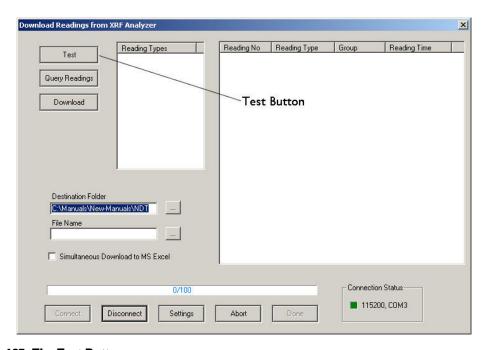


Figure 185. The Test Button

6. You should get a pop-up window informing you that the connection tested successfully. If the test fails, there is a problem with your serial port setup.



Figure 186. Connection Success Window

7. In the Download dialog box, click the Connect button.

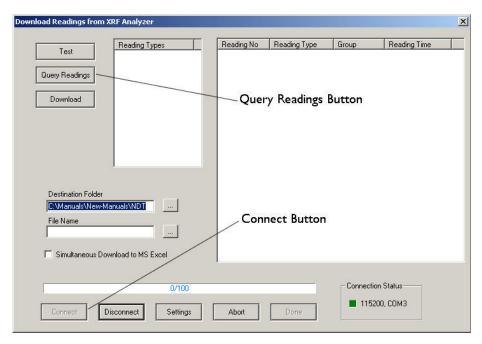


Figure 187. The Connect Button

8. Click the Query Readings button. This will return a list of all current readings on your analyzer. The list appears in the large white box in the Download dialog box.

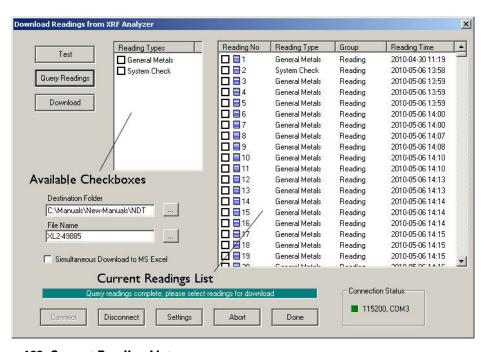


Figure 188. Current Reading List

9. Select the readings that you want to download. There are two ways to do this.

a. Click the boxes next to each of the reading numbers to select or de-select individual readings. You can select a range of readings by pressing the shift key, then selecting the first and last reading in the range. All readings from the first reading selected to the last will then be selected.

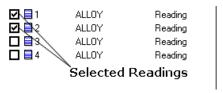


Figure 189. Selecting Readings

b. Click the boxes on the left to select or de-select all the readings of a specific type. You can also use the Shift-Click method of selecting a range of readings as described above.



Figure 190. Using Check Boxes

- 10. The download generates a data file containing the selected readings. To save the file for later use:
 - c. Enter the path for the file in the Destination Folder field. You can use the ... button to browse.



Figure 191. The Browse Button

d. Enter a name for the file in the File Name field.

13 Connectivity Downloading Data

CAUTION Some characters are not allowed in the file name. Characters such as the "#" sign will cause an error. Niton recommends using only alphanumeric characters "-", "_" and the space character when naming a file.

e. Click the Download button.

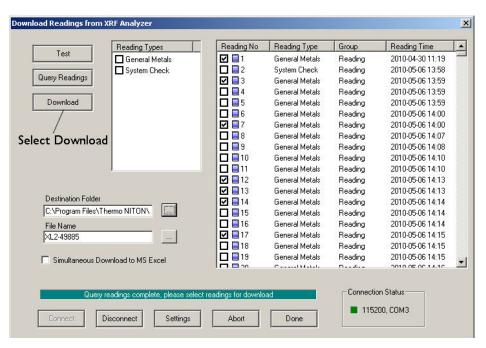


Figure 192. The Download Button

When the progress bar shows that all the readings are downloaded, click the Done button.

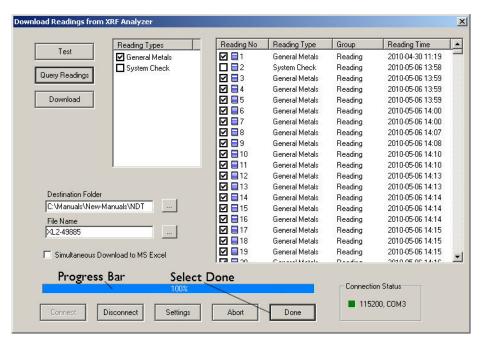


Figure 193. The Progress Bar

Error Message

You should now see the readings you selected for download displayed, one reading per horizontal line. The data has been saved to the folder and filename you indicated prior to downloading. If an error message has appeared, see the following section. See Creating and Using Reports to continue otherwise.

You can also automatically save reports in .csv format for importing into Excel or other programs.Simultaneous Save as CSV File

Action

Table 10: Error Messages while Downloading

Diror Wessage	1 Letton
Couldn't open \\.\COM7 Error Code: 2	Select another COM port.
The port \\.\COM2 is in use	Select another COM port.
Please Open the Port	Click the Connect button.

Table 10: Error Messages while Downloading

Hardware Not Responding or Hardware Not Ready	Turn on the XRF Analyzer. If you are using a serial cable, check that the cable is inserted snugly. If you are using a serial cable, select the other COM port. If you are using the wireless USB adapter, connect the serial port. See the "Installing and Using Bluetooth" manual for complete instructions on setting up the Bluetooth adapter to work with your analyzer. Check that the spare battery is fully charged.
The Serial Port connection failed: RFCOMM connection failed	Check that the battery is fully charged.
WARNING: 38400 baud rate not supported.	This indicates a potential problem. Test the serial port. If there is a problem connecting, switch baud rate on both the analyzer and the NDT software to 115200.
Incorrect Data in reading # XXX. Reading will be skipped. Error code: BOUNDARY_ERROR1.	This indicates a version mismatch between your instrument code and the NDT code running on your computer. Use a version of NDT that matches the ver?sion number of the software on your analyzer.
Incorrect Data in reading # XXX. Reading will be skipped. Error code: BOUNDARY_ERROR2.	This indicates a version mismatch between your instrument code and the NDT code running on your computer. Use a version of NDT that matches the ver?sion number of the software on your analyzer.
Incorrect Data in reading # XXX. Reading will be skipped. Error code: BOUNDARY_ERROR3.	This indicates a version mismatch between your instrument code and the NDT code running on your computer. Use a version of NDT that matches the ver?sion number of the software on your analyzer.
WARNING: 115200 baud rate not supported.	This indicates a potential problem. Test the serial port. If there is a problem connecting, switch baud rate on both the analyzer and the NDT software to 38400.
SH4 Successfully Communicating Result: SUCCESS	This indicates a normal connection.

Note When using the wireless USB adapter, if the serial port repeatedly disconnects, check that the battery is fully charged.

Live Download (Automatic Save)

If desired, your Niton XL3 analyzer has the capability to download and store each reading to the PC in real time to a file of your choice. To enable this feature, you must do the following:

- Your Niton analyzer must be turned on and connected to the PC. See Connecting the XRF Analyzer to Your PC.
- The NDTr program module must be running and connected to your analyzer. See Operating Your Analyzer Remotely.
- The Download icon in the NDTr toolbar must be selected.



Figure 194. Live Download Icon

The file created is in a format readable by the NDT program module, has an extension of .ndt, and looks identical to a file of manually downloaded readings - see Standard Download. It can also create a simultaneous .CSV file. Simultaneous Save as CSV File .

Please note the following:

- 1. When the instrument is unplugged, selecting the Download icon does nothing.
- 2. When you disconnect, then reconnect, your analyzer, Download appends future readings to same file.
- 3. Live Download does not overwrite any previous readings in the file. If you want to do this, you must first explicitly erase the file before initiating Live Download.
- 4. Live Download does not retroactively add any readings taken while your analyzer was disconnected.

Changing the Filename for Live Download

Once you have selected the Download icon, a dialog box appears as shown below:



Figure 195. File Dialog Box

You can change the destination file or folder by clicking in the appropriate text box and typing in the new file name, or by clicking on the browse button (...) to the right of the text box and selecting a different pre-existing filename. To implement these changes, click the OK button.

Your instrument serial number is associated with the file. If a different instrument is connected and Live Download is started, a message will appear saying that the connected instrument and file instrument do not match, and Live Download will not start. Saving the session as a new file will alleviate this issue

Simultaneous Save as CSV File

By clicking on the checkbox box labeled "Save the readings to CSV file as well" you can enable simultaneously saving the data as a standard CSV (Comma Separated Value) file for use with other programs.

Creating and Using Reports

Creating a New Report

Niton Data Transfer creates a new report whenever you download data. For more information. See Downloading Data for more information.

Opening an Existing Report

To open an existing report

1. Click the Open button to open the Open Existing Report dialog box.

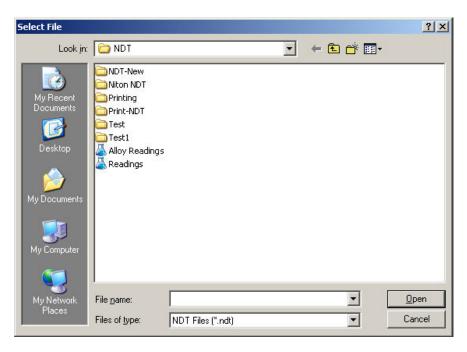


Figure 196. Open Existing Report Dialog Box

2. Click the name of the report to highlight it.

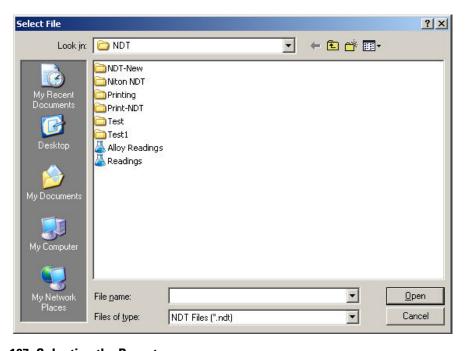


Figure 197. Selecting the Report

3. Click the Open button.

13 Connectivity Creating and Using Reports

4. The report is presented with one line per measurement. The report doesn't all fit onto one screen, but you can scroll right if the fields you are interested in are not visible. If they are still not visible read the following section on changing visible columns.

Configuring Reports for Printing.

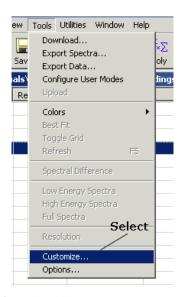


Figure 198. Select Customize from the Menu

1. Select Customize from the Tools Menu.

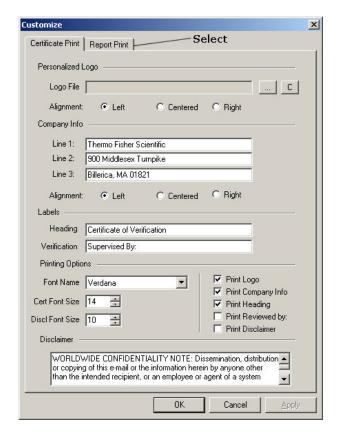


Figure 199. Select the Report Print Tab

2. Select the Report Print tab.

This brings up the Report Customization window.

13 Connectivity Creating and Using Reports

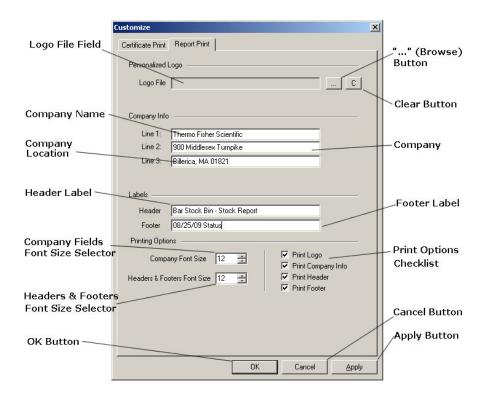


Figure 200. Customizing the Report

Like the Certificate Print option, the Report Print option adds company information, company logo, headers, and footers to a standard report printout. The data input into the various report fields is arbitrary - you can put anything you want to into any of the fields. For example, you can type test location into the company address field. The names given to the fields do not limit the data placed into the fields.

The Company Fields Font Size Selector controls the size of the font used in the Company Name, Address, and Location fields. The Headers & Footers Font Size Selector controls the size of the font used in the Header and Footer.

The report will print every optional field selected in the Print Options Checklist.

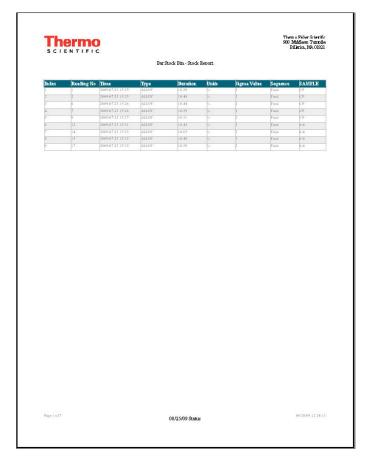


Figure 201. Sample Report

Viewing the Report Properties

The report properties are:

- Analyzer Serial Number
- Report File Name
- File Version
- Report Download Date
- Total Readings in the Report



To view the report properties, click the Report Properties button, Properties from the View Menu.

13 Connectivity Creating and Using Reports

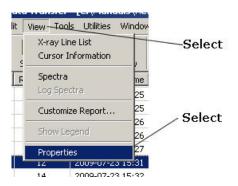


Figure 202. Select Properties from the Menu

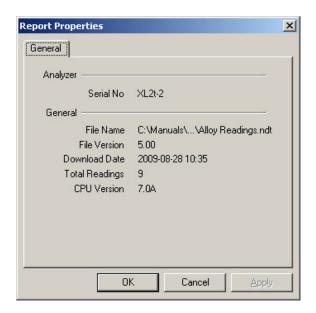


Figure 203. The General Properties Tab

Using the Customize Report Dialog Box to Add and Remove Columns

To use the Customize Report dialog box to add and remove columns:

- 1. Click the View Menu.
- 2. Click Customize Report...

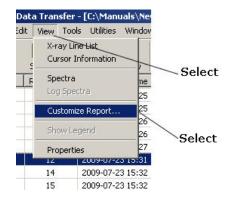


Figure 204. View Menu

2. Click the boxes next to each of the column headings to add or remove each column from the report.

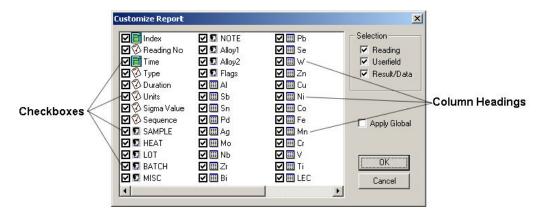


Figure 205. Select Columns for Report

3. When a checked box is selected, the check is removed and the box becomes empty.

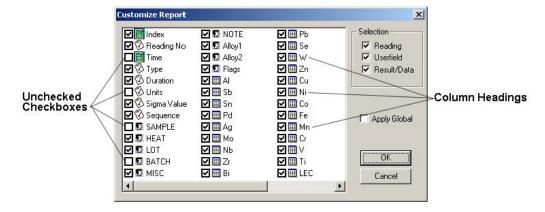


Figure 206. Deselecting Boxes

13 Connectivity Creating and Using Reports

4. If you want, you can click the boxes in the Selection area to select or de-select a group of headings.

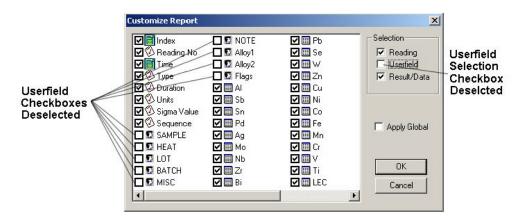


Figure 207. The Selection Area

5. If you want the changes to apply to all the reports, click the box next to Apply Global. If this box does not have a check mark, the changes only apply to the report that is currently open.

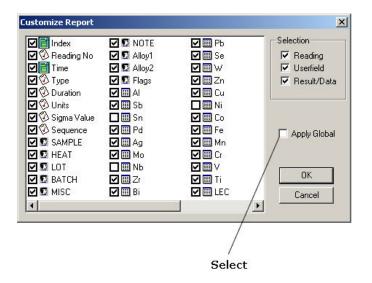


Figure 208. Applying Changes Globally

6. Click the OK Button.

The columns appear in the order in which they are listed in the Customize Report Dialog Box. If you change the position of the columns, and then use the Customize Report Dialog Box, the columns return to their original positions.

Changing the Position of the Columns in the Report

To change the position of a column, click and drag the column heading. A shadow of the heading will appear, indicating where the heading will be placed.

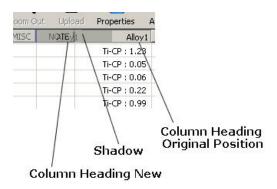


Figure 209. Switching Columns

If you want to add or remove columns, but not change their position on the report, you can also use the Customize Report Dialog Box. See Using the Customize Report Dialog Box to Add and Remove Columns for instructions.

Changing the Columns in a Report

You can remove columns from the reports, add the columns back again, or change the position of columns within the reports.

To remove a column from a report, <u>See "Removing a Column from the Report"</u>, and <u>See "Using the Customize Report Dialog Box to Add and Remove Columns"</u>.

To add a column to a report, <u>See "Adding a Column to the Report"</u>, and <u>See "Using the Customize Report Dialog Box to Add and Remove Columns"</u>.

Removing a Column from the Report

Columns can be removed from a single report, or removed globally. Removing a column from a single report removes the column only from the report that is currently open. Removing a column globally removes the column from the report that is currently open, as well as from all reports that are opened later. If you have more than one report open, and you want to remove a column from all of the open reports, you must close them and re-open them.

Removing a column does not actually delete the data, it simply removes it from the display. You can later re-display the column with the data intact.

To remove a column from the report:

1. Right-click on the heading for the column that you want to remove.

13 Connectivity Creating and Using Reports

2. Click Hide, or Hide Global, on the Pop-Up Menu.

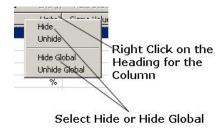


Figure 210. Hiding Columns

Note For some columns, Hide Global is not available.

Adding a Column to the Report

After you remove a column from a report, there are three ways to add it to the report again.

- Use the Hidden Columns dialog box to add a column to the same report from which you removed it. <u>See "Adding a Column with the Hidden Columns Dialog Box"</u> for more information.
- 2. Use the Options dialog box to add a column that you removed using Hide Global. See "Adding a Column with the Options Dialog Box" for more information. You can use either dialog box if you are adding a column that you removed using Hide Global, in the report from which you removed it.
- 3. Use the Customize Report Dialog Box to add or remove multiple columns at the same time. See "Adding a Column with the Options Dialog Box" for more information.

Adding a Column with the Hidden Columns Dialog Box

To add a column to the report using the Hidden Columns dialog box:

- 1. Open the report that you used to remove the column from the report.
- 2. Decide where you want the re-inserted column to appear.
- 3. Right-click on the heading for the column to the left of that.
- 4. Click Unhide on the pop-up menu to display the hidden columns

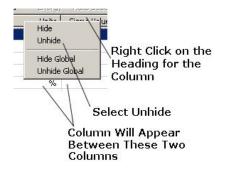


Figure 211. Displaying Hidden Columns

5. Click the column heading that you want re-insert.

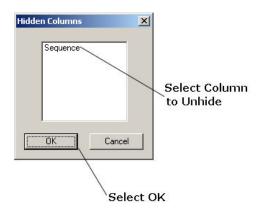


Figure 212. Selecting Column to Display

6. Click the OK button.

The column is inserted to the right of the original column.

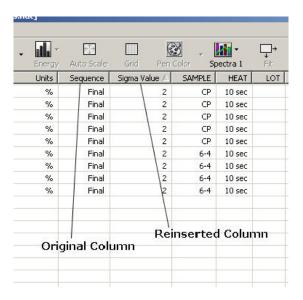


Figure 213. Reinserting Columns

Adding a Column with the Options Dialog Box

To add a column to the report using the Options dialog box:

1. Select the Tools Menu.

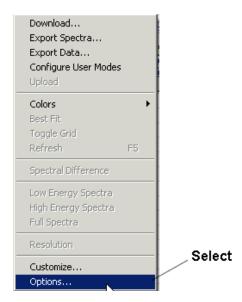


Figure 214. The Tools Menu

- 2. Select Options.
- 3. Select the Report tab.

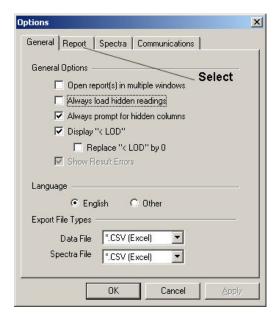


Figure 215. The Report Tab

4. Click the column heading that you want to re-insert.

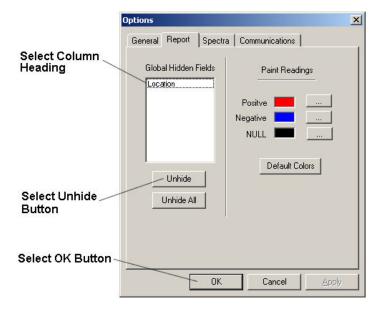


Figure 216. Unhide Button

- 5. Select the Unhide button.
- 6. Select the OK button.

Hiding a Reading in the Report

Rows can be prevented from appearing in a report. Hiding a row removes the row only from the report that is currently open.

Note Hiding a row does not actually delete the data, it simply removes it from the display. You can later re-display the column with the data intact.

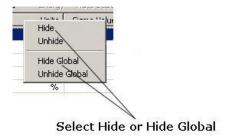
To hide a row in the report:

1. Select the row you want to hide.



Figure 217. Selected Row

2. Right-click on the row and select Hide from the pop-up menu.



3. The row is hidden from the report view.

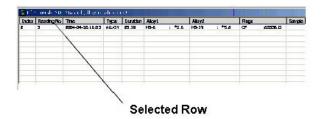


Figure 218. Hidden Row

4. You can hide multiple readings in this way.

Restoring the Row to the Report

You can restore a hidden row to the report view.

- 1. Select the readings above and below the reading you want to restore.
- 2. Right-click on the highlighted readings.
- 3. Select Unhide from the pop-up menu, and the row is restored.



Figure 219. Restoring Rows

Restoring Multiple Readings to the Report

You can restore multiple hidden readings to the report view.

- 1. Right-click on a reading or empty row, and select Unhide from the pop-up menu.
- 2. The rows are restored.

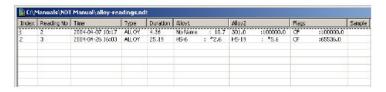


Figure 220. Restored Readings

Rows (Readings) in a Report

You can prevent rows (readings) from appearing in the reports, add the rows back again, or change the position of columns within the reports.

- To hide a row from a report view, See "Hiding a Reading in the Report".
- To restore a hidden row to a report view, See "Restoring the Row to the Report".
- To restore multiple hidden rows to a report view, <u>See "Restoring Multiple Readings to the Report"</u>.

• To reverse the sort order of the rows in a report, See "Reversing the Sort Order in a Report".

Reversing the Sort Order in a Report

You can reverse the order of the rows (readings) in a report.

1. Click on any column heading - the labels in the first row of the report.

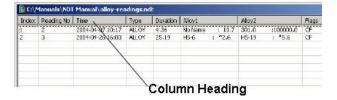


Figure 221. Column Heading

2. The rows change order from descending to ascending. The column heading you clicked displays a triangle indicating the direction of the sort.

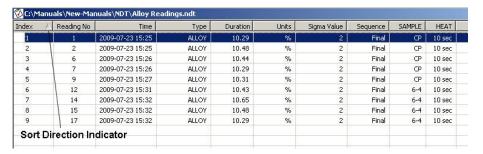


Figure 222. Sort Direction Indicator

- 3. Click the column heading again.
- 4. The rows reverse order again, with the triangle reversing orientation to indicate the new sort order.



Figure 223. New Sort Order

Note To revert reading order to the original order click on the "reading number" or "index number" column heading.

Viewing the Spectra Graph

You can access the Spectra Graph by double-clicking on any field of the report containing non-editable reading information, such as the Reading Number, Index, Time, Type, etc. .

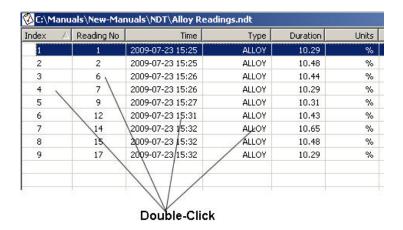


Figure 224. Accessing the Spectra Graph

Editable Fields

Double-clicking on an editable field in the report allows you to edit the contents of that cell. When the field is double-clicked, the field becomes highlighted and a text cursor appears in the cell. Editable fields contain note-type information which do not affect the reading in any way, such as SAMPLE, LOCATION, INSPECTOR, etc. Editable field names are always labelled in ALL CAPS.

Changing the Colors of the Spectra Graph

You can change the color of the pen, the text, the grid, the axis, or the background.

Changing the color of the pen changes this part of the graph:

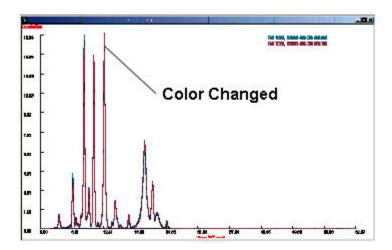


Figure 225. Changing Spectra Graph Pen Colors

Changing the color of the background changes this part of the graph:

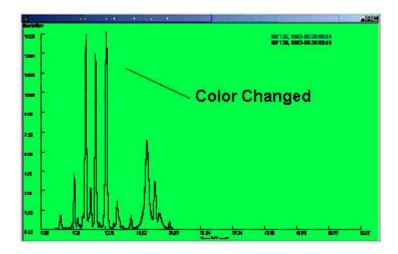


Figure 226. Changing Spectra Graph Background Color

Note Making the pen and background the same color makes the spectrum invisible.

Changing the color of the grid changes this part of the graph:

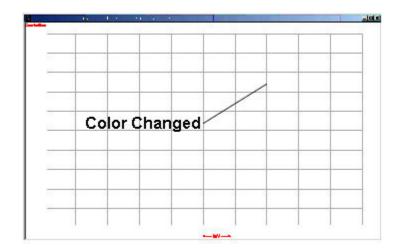


Figure 227. Changing Spectra Graph Grid Colors

Changing the color of the text changes this part of the graph:

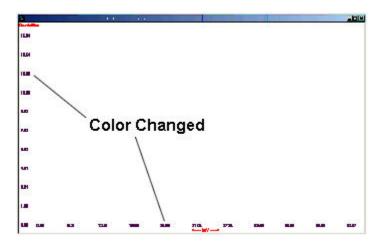


Figure 228. Changing Spectra Graph Text Colors

Changing the color of the axis changes this part of the graph:

13 Connectivity Viewing the Spectra Graph

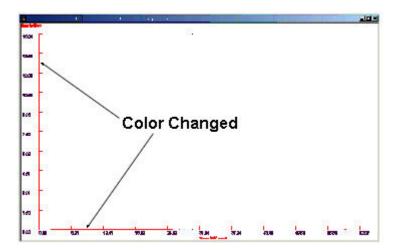


Figure 229. Changing Spectra Graph Axis Colors



To change the color of the spectra graph:



1. Click the Down Arrow button on the Pen Color button to display the Color Menu.



Figure 230. The Pen Color Button

- 2. Click the part of the graph that you want to change.
- 3. If you select Set Pen Color, the Spectrum Properties dialog box appears.



Figure 231. Spectrum Properties Dialog Box

Note This dialog box only appears if you select Set Pen Color. If you want to change the color of any other part of the spectra graph, skip to Step 6.

4. If you have selected more than one Reading, click the Reading Number that you want to set.

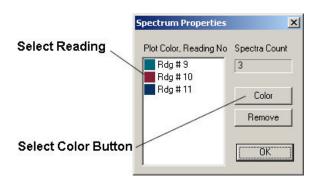


Figure 232. Selecting Readings

5. Select the Color button.

Note To select more than one Reading Number, hold down the shift key while selecting.

6. In the Color dialog box, click a color to select it.

13 Connectivity Viewing the Spectra Graph

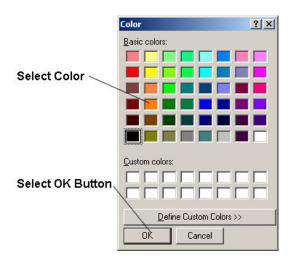


Figure 233. Selecting Colors

7. Click the OK button.

Defining Custom Colors

In addition to the forty-eight Basic Colors provided, you can also define additional colors. These are called "custom colors."

To define a custom color:



- 1. Select the Pen Color button to display the Color dialog box.
- 2. Select Define Custom Colors Button to display the custom color area.



Figure 234. Custom Colors

3. Use the panel on the right to specify a color.

Click within the large color box to specify the hue and saturation. Hue is the color, or shade of color. Saturation is whether the color is pure or mixed with gray.

The values for hue range from 0 on the left side of the color box, to 239 on the right side.

The values for saturation range from 0 at the bottom of the color box, to 240 at the top.

Notice that as you click in different places in the color box, the values for hue and saturation change.

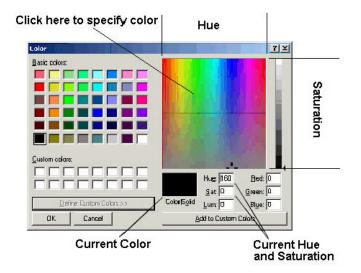


Figure 235. Selecting From the Color Box

13 Connectivity Viewing the Spectra Graph

Use the slider to specify the luminosity. Luminosity is the measure of how much light is in the color.

The values for luminosity range from 0 at the bottom of the slider, which indicates black, to 240 at the top, which indicates white.

Notice that as you move the slider up and down, the value for luminosity changes.

Notice as you move the slider up and down, the values for Red, Green, and Blue also change. These values represent a second method for specifying the same colors.

The color you have selected is shown in the smaller Color Solid box.

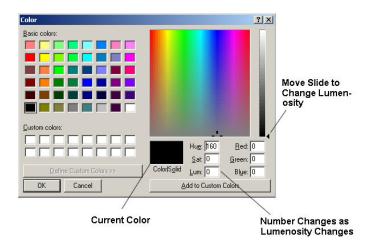


Figure 236. Current Color

Note If you know the values for the color that you want, you may type them directly in the boxes.

4. When you are satisfied with the color you have selected, click Add to Custom Colors button. The color appears on the left under custom colors. It can now be used in the spectra graph in the same manner as the Basic Colors.

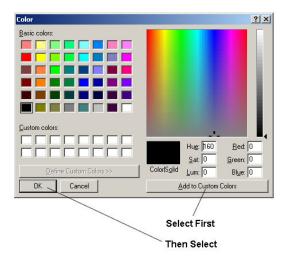


Figure 237. Closing the Dialog Box

5. Close the dialog box by doing one of the following:

To close the dialog box and replace the current color in the spectra graph, click the OK button.

To close the dialog box, but not replace the color, click the Cancel button.

Returning to the Default Colors

To return to the default pen colors, close the graph and re-open it.

To return to the default colors for the background, grid, text, axis and x-ray line:

1. From the Tools Menu, select Options.

13 Connectivity Viewing the Spectra Graph

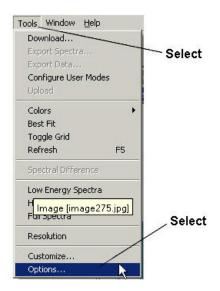


Figure 238. The Tools Menu

2. In the Options dialog box, click the Spectra tab.

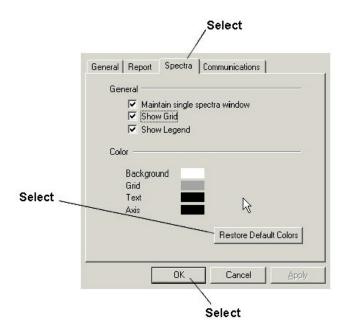


Figure 239. Restoring the Default Colors from the Spectra tab

- 3. Click the Restore Default Colors button.
- 4. Click OK.

Viewing Different Areas of the Spectra Graph

Niton Data Transfer lets you view different areas of the spectra graph. The available options are shown in the table.

Table 11. Spectra Graph Options

Option

What the Option displays

The area of the spectra from 0-40 keV.



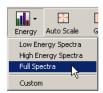
Low Energy Spectra

The area of the spectra from 40-100 keV.



High Energy Spectra

The area of the spectra from 0-100 keV.



Full Spectra

The area that you specify.



Custom Size



Max Y Limit is the maximum counts/sec?ond.

Max X Limit is the maximum keV.

Table 11. Spectra Graph Options

Auto Scale	The area varies. It begins at 0 keV. It ends wherever the spectra flattens out so that all of the peaks are less than 10% of the height of the highest peak. This option is the default.
Fit →	The area that you specify. See "Magnifying the Spectra Graph" for more information.

Magnifying the Spectra Graph

Niton Data Transfer lets you magnify a specific area of the spectra graph.

To magnify the spectra graph:

1. Display the spectra graph by double-clicking on the reading number.

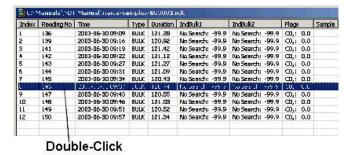


Figure 240. Displaying a Spectra Graph

2. Highlight the area that you want to magnify. To do this, click and drag over the desired area. The Cursor Information dialog box appears showing the keV of the boundaries for the area of the spectra graph that is selected. It also shows the height of the highest peak within the selected area.

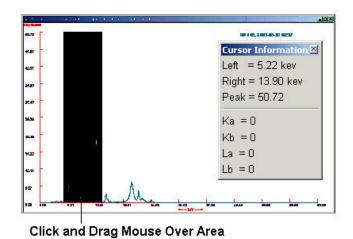


Figure 241. Selecting Spectra Graph Areas of Interest

3. Click the Fit button. The spectra graph expands to show only the previously selected area, and the Cursor Information dialog box is reset to zero.

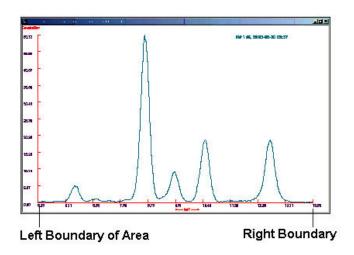


Figure 242. Spectra Graph Fit to Area Selected

Hiding or Showing the Cursor Information Dialog Box

The Cursor Information dialog box displays the keV of the left and right boundaries for the area of the spectra graph that is selected. It also shows the height of the highest peak within the selected area. To show the Cursor Information dialog box, click anywhere in the spectra graph.

To hide the Cursor Information dialog box:

1. Click the View Menu.

2. Click Cursor Information.



Figure 243. Selecting Cursor Information

The Cursor Information Box

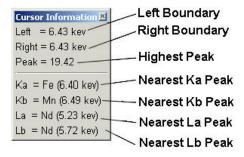


Figure 244. The Cursor Information Box

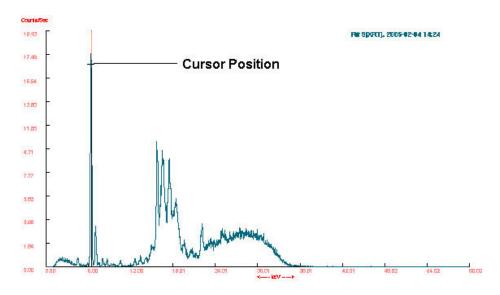


Figure 245. Cursor Positioning on the Spectra Graph

Left Boundary

This line lists the energy in keV of the Left boundary selected.

Right Boundary

This line lists the location of the Right boundary selected.

Highest Peak

This line lists the height of the peak selected in Counts/Second.

Nearest Ka Element peak

This line lists the element with the nearest Ka peak to the selected energy position, along with the energy of that peak in keV. If the selected energy position is an area - i.e. the Left and Right boundaries are different - this result will be zero.

Nearest Kb Element peak

This line lists the element with the nearest Kb peak to the selected energy position, along with the energy of that peak in keV. If the selected energy position is an area - i.e. the Left and Right boundaries are different - this result will be zero.

Nearest La Element peak

This line lists the element with the nearest La peak to the selected energy position, along with the energy of that peak in keV. If the selected energy position is an area - i.e. the Left and Right boundaries are different - this result will be zero.

Nearest Lb Element peak

This line lists the element with the nearest Lb peak to the selected energy position, along with the energy of that peak in keV. If the selected energy position is an area - i.e. the Left and Right boundaries are different - this result will be zero.

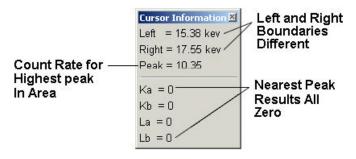


Figure 246. Cursor Information for Selected Area

Hiding or Showing the Grid

Hiding the grid hides this part of the graph:

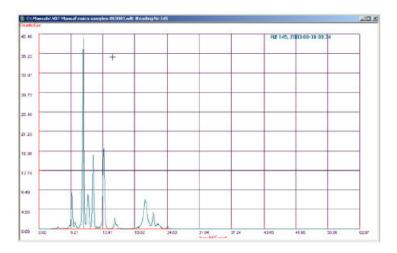


Figure 247. Hiding or Showing the grid



To hide the grid, click the Toggle Grid button.

To show the grid, click the Toggle Grid button again.

Viewing the X-ray Line List

To view the X-Ray Line List, Click on the View Menu, then Select the X-Ray Line List.

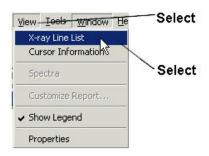


Figure 248. The X-ray Line List

The X-Ray Line List gives the energy in KeV of the peaks for various elements. This is a reference you may find handy. The list gives the Ka, Kb, and La energy peaks for the elements listed.

Below is the X-ray Line List:

Element	Ka	КЬ	La	Lb
DA	ZZ.10	24.99	2.98	3.15
Al	1.48			
Ar	2.96	3.19		
As	10.53	11.73		
Au	68.79	77.97	9.71	11.44
Ba	32.19	36.38	4.47	4.83
Bi	77.10	87.34	10.84	13.02
Br	11.91	13.30		
Ca	3.69	4.01		
Cd	23.11	26,14	3.13	3.32
Ce	34.72	39.26	4.84	5.26
cl	2.62	2,82	77,000	
Co	6.93	7.65		
Cr	5.41	5.95		
Cs .	30.97	34.98	4.29	4.62
Cu	8.04	8.91		
Fe	6.40	7.06		
Hf	55.76	63.Z1	7.90	9.02
Hq	70.82	80.26	9.99	11.82
I	28.61	32.29	3.94	4.22
In	24.14	27.38	3.28	3,45
	64.89	73.55	9.17	10.71
lir K	3.31	3.59	9.17	10.71
la	33.44	37.80	4.65	5.04
		37.60	9.05	5.04
Mg	1.25	e .m		
Mn	5.90	6.49		2 42
Mo	17.44	19.63	2.29	2,40
Nb	16.58	18.65	2.17	2.25
Nd	37.36	42.27	5.23	5.72
Ni	7.47	8.27		
Os	62.99	71.40	8.91	10.35
P	2.02	201002	100000000000000000000000000000000000000	0121112121
Pb	74.96	84.92	10.55	12.61
Pd	Z1.1Z	Z3.86	Z.84	2.99
Pr	36.02	40.75	5.03	5.49
Pt	66.82	75.74	9.44	11.07
RЬ	13.38	14.97	65,756,75	500000
Re	61.13	69.30	8.65	10,01
Rh	20.17	22.76	2.70	2.83
Ru	19.24	21.69	2.56	2.68
S	2.31	2.47		
5b	26.36	Z9.7Z	3.51	3.84
SC .	4.09	4.46		
Se	11.21	12.50		
Si	1.74			
Sn	25.27	28.48	3.44	3.66
Sr	14.14	15.85		
Ta	57.52	65.21	8.15	9.34
Te	27.47	30.99	3.77	4.03
Th			12.97	15.20
Ti	4.51	4.93		
TI	72.86	82.56	10.27	12.21
U		22/00/2006	13.61	17.22
Ÿ	4.95	5.43	mmistro	200000000
w	59.31	67.23	8.40	9.67
Y.	14.93	16.75	1.92	
Zn	8.63	9.57		

Figure 249. The X-ray Line List

You can select the element of your choice to highlight the characteristic energies for that element.

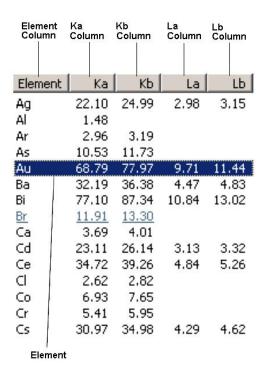


Figure 250. Close View of the X-ray Line List

Peak Labels

If you load up the X-Ray Line List while viewing a spectrum, **Peak Labelling** is activated. When you click on a peak with Peak Labeling activated, that peak is labeled with the name of the nearest elemental energy line:

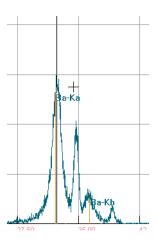


Figure 251. Peak Labelling

In the example above, clicking on the peak at 32 shows that the nearest elemental energy line is the Ka line of Ba. Simultaneously, the other lines of Ba are also labeled - in this case the Kb line. The element Ba is also highlighted on the X-ray Line List:

Ar	2.96	3.19		
As	10.53	11.73		
Au	68.79	77.97	9.71	11.44
Ba	32.19	36.38	4.47	4.83
Bi	77.10	87.34	10.84	13.02
Br	11.91	13.30		
Ca	3.69	4.01		
Cd	23.11	26.14	3.13	3.32
Ce	34.72	39.26	4.84	5.26
Cl	2.62	2.82		

Figure 252. Highlighted Element Peaks

This linking between the Line List and the Spectrum works in reverse as well. Click on an element in the line list, and the appropriate peaks in the spectrum will be labeled:

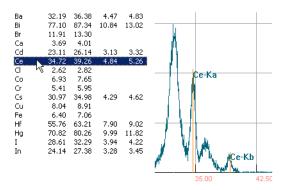


Figure 253. Selecting a Peak with the X-ray Line List

Select Options... from the Tools Menu, then select the Spectra Tag in the Options Screen.

13 Connectivity Viewing the X-ray Line List

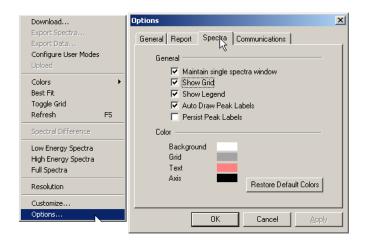


Figure 254. The Spectra tag

Then click the check box next to Persist Peak Labels.

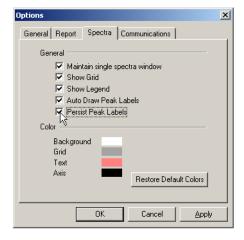


Figure 255. Making Peak Labels Persistent

This makes all the previous Peak Labels stay on screen when you select another peak.

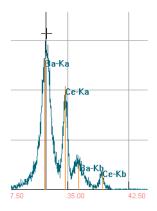


Figure 256. Viewing Multiple Peak labels

You can also do this by right clicking on the spectrum and selecting Preserve Label(s)



Figure 257. Selecting Persistent Labels Through the Menu

You can also right click on the spectrum and select "Clear Label(s)" to remove all accumulated Peak Labels from the spectrum.

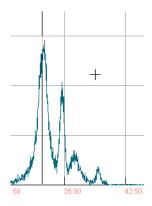


Figure 258. Clearing Labels from the Spectrum

Overlaying Spectra

You can overlay the spectra from several different readings to highlight differences and similarities between and among the different spectra.

Open a reading file.

Select the readings you want by holding down the control key and clicking on the row containing the reading. This will select the readings you clicked on, but not anything in between.

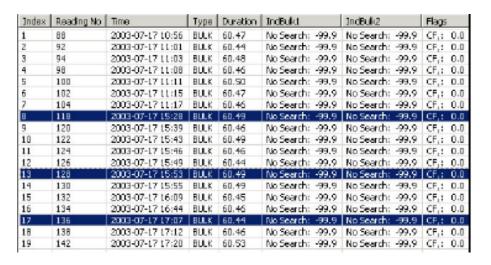


Figure 259. Selecting Multiple Spectra for Overlay

OR

Select the readings you want by holding down the shift key and click on the row containing the first reading you want, then clicking in the row containing the last reading you want. This will select the reading you clicked on and any readings in between.

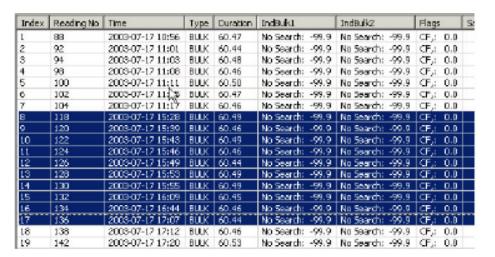


Figure 260. Selecting a Range of Spectra for Overlay

Right click on any selected file and choose Spectra from the pop-up menu.

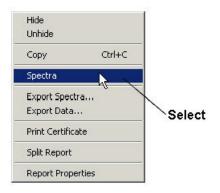


Figure 261. Selecting Spectra from the Menu

This will bring up the a window displaying the selected spectra overlain on the same graph.

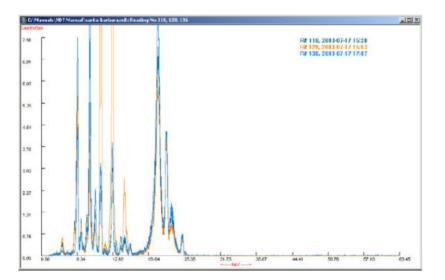


Figure 262. Overlain Spectra

Spectral Difference

To better detect subtle differences in spectra, you can subtract one spectrum from another. The difference will be displayed as its own spectrum.

Select the two samples you wish to compare, and display them as overlain spectra.

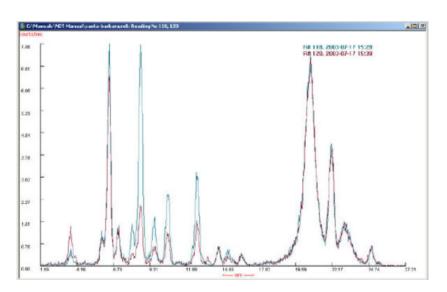


Figure 263. Two Overlain Spectra

From the Tools Menu, select Spectral Difference.

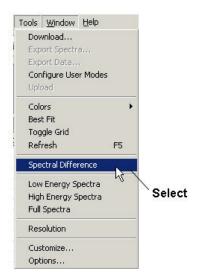


Figure 264. Selecting Spectral Difference from the Menu

The difference between the two spectra is then presented as a spectrum of its own, giving the number of counts *difference* at each energy level

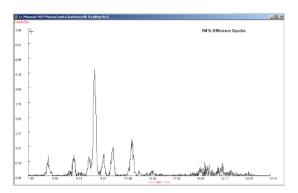


Figure 265. Difference Spectrum

When you are done with the Difference Spectrum, you can return to view the overlaid spectra by selecting Undo Spectral Difference from the Tools Menu.

13 Connectivity Viewing the X-ray Line List

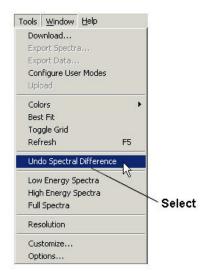


Figure 266. Undoing Spectral Difference

Paint Readings and Spectra

Paint readings are special in that they can be compared to a set quantity, known as the **Action Level**, for comparison. If a reading is below the **Action Level**, then one course of action is followed. If the reading is at or above the action level, then another course of action is followed. The NDT program allows you to see this difference visibly in the data.



Figure 267. Paint Readings

Above is a series of three paint readings. Each is a different color. The color of the row correlates to the result. In this case, the **Null** reading is black, the **Positive** reading is red, and the **Negative** reading is blue. These colors can be set to any combination of colors as your preference.

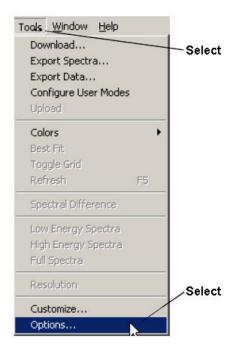


Figure 268. Selecting Options from the Menu

To change your color preferences, select Options from the Tools Menu, then select the Report tab in the Options dialog box.

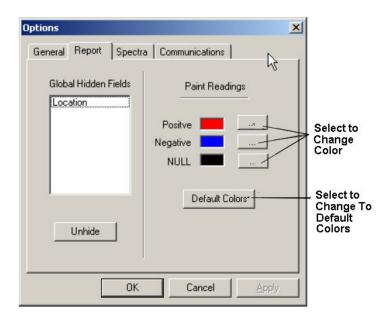


Figure 269. The Report Tab

You can select the color you want in the Color menu, or create a custom color.

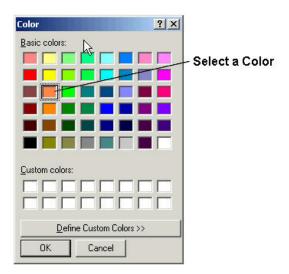


Figure 270. Selecting Your Preferred Colors

The new color will show up in the Report tab.

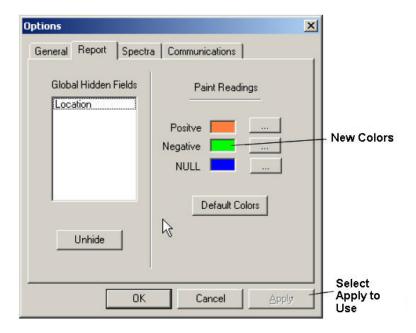


Figure 271. The Report Tab

The new colors will show up on the report.



Figure 272. New Colors in the Report

Using the Menus Instead of the Toolbar

Most of the functions that can be performed using the buttons on the Toolbar can also be performed using the menus. This is illustrated in the table.

Table 12. Using the Menus Instead of the Toolbar

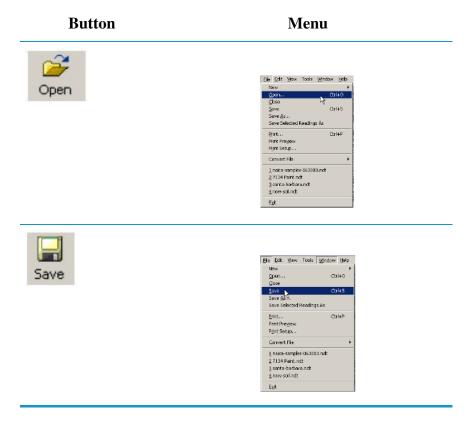


Table 12. Using the Menus Instead of the Toolbar







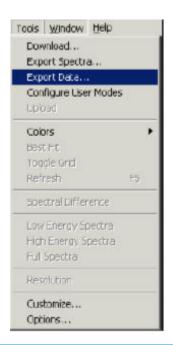






Table 12. Using the Menus Instead of the Toolbar







No Menu Equivalent



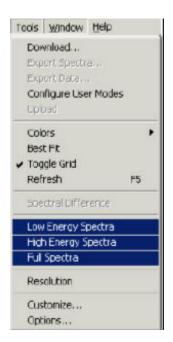


Table 12. Using the Menus Instead of the Toolbar







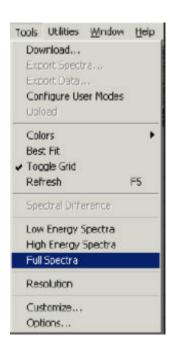


Table 12. Using the Menus Instead of the Toolbar



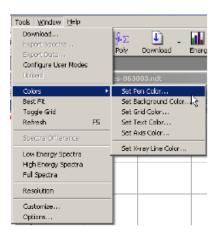






Table 12. Using the Menus Instead of the Toolbar



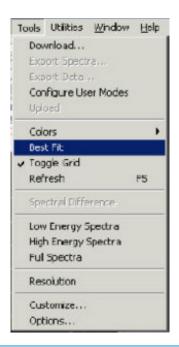
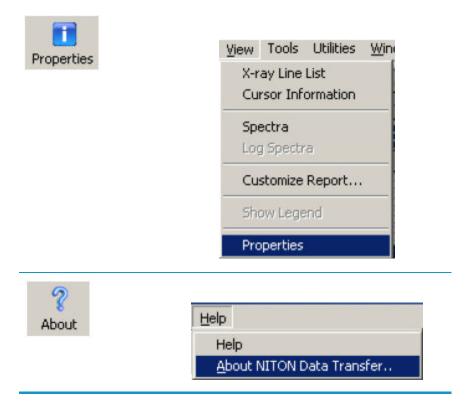






Table 12. Using the Menus Instead of the Toolbar



Renaming a Report

To give a report another name:

1. Select Save As from the File Menu.

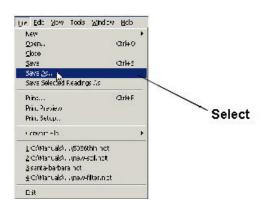


Figure 273. Saving a Report Under Another File Name

2. Type a name in the File Name field of the Save As Dialog Box.

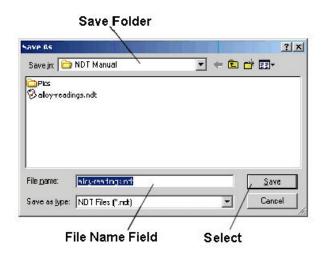


Figure 274. Renaming the File

3. Click the Save Screen Button.

Saving Selected Readings from a Report

To save part of an NDT file, select the readings you wish to save by highlighting them with the Shift or Ctrl keys and the left mouse button. Select Save Selected Readings As from the File Menu.

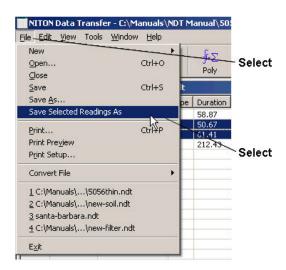


Figure 275. Saving Selected Readings As

You will then be prompted to enter a new file name. Only the selected readings will be saved to the new NDT file.

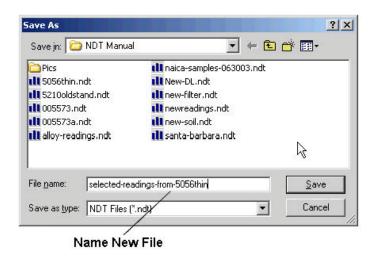


Figure 276. New File Name

Copying a Report Into Excel

Niton Data Transfer (NDT) enables you to copy report data and paste it into an Excel spreadsheet.

For information about exporting the report data as a CSV file to use in other programs, <u>See "Exporting the Report Data"</u>.

For information about exporting the data from the spectra graphs, <u>See "Exporting the Spectra Data"</u>.

To copy report data into Excel:

- 1. Open the report. For more information, See "Opening an Existing Report".
- 2. Select the readings that you want to copy to Excel.

To select a range of readings, hold down the shift key and click the first and last readings in the range.

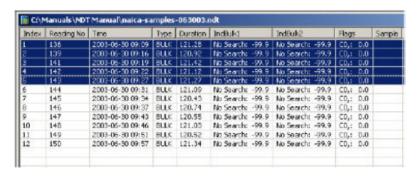


Figure 277. Selecting a Range of Readings

To select multiple readings that are not adjacent, hold down the control key and click each reading that you want to select.



Figure 278. Selecting Multiple Readings

3. Select Copy from the Edit Menu.

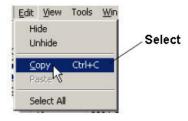


Figure 279. The Edit Menu

- 4. Open Excel. To do this:
 - a. Click the Start button.
 - b. Select Programs from the Start Menu.
 - c. Click Microsoft Excel.

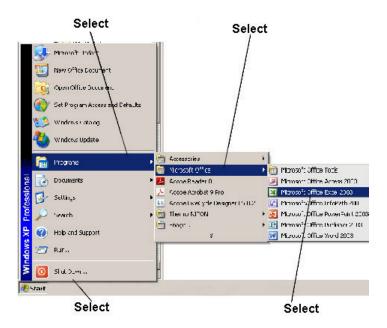


Figure 280. Loading Excel

5. Within Excel, select Paste from the Edit Menu.

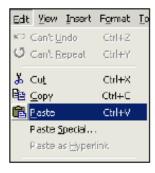


Figure 281. Pasting Data Into Excel

6. Excel shows the data from the columns that are displayed in NDT, for the rows that you selected.

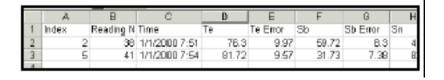


Figure 282. How NDT Data is Displayed in Excel

Note If the Time column does not display properly, click on the cell.

Exporting the Report Data

Niton Data Transfer lets you export the data from the reports to a file in Comma Separated Value (CSV) format. You can then import the data into another program to manipulate it.

You can also copy the report data directly into Excel. For more information <u>See "Copying a Report Into Excel"</u>.

This section covers exporting report data. For information about exporting spectra data, <u>See</u> "Exporting the Spectra Data".

To export the report data:

- 1. Open the report. For more information, See "Opening an Existing Report".
- 2. Select Export Data from the Tools Menu.

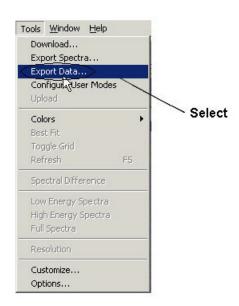


Figure 283. Exporting Data

3. The Export Data Dialog Box will appear.

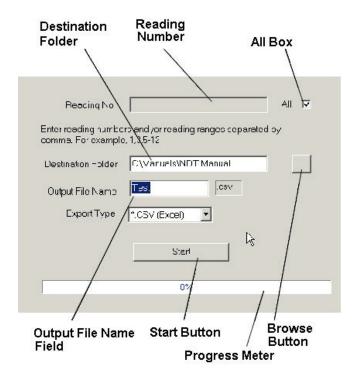


Figure 284. The Export Data Dialog Box

4. In the Export Data dialog box, specify the reading numbers that you want to export.

If you want to export the report data for all the reading numbers, you do not need to make a change for the reading numbers. Leave the All box checked and move on to the next step.

If you want to export only some of the report data:

- a. Uncheck the All box.
- b. Enter the reading numbers. You may enter individual reading numbers, or ranges of reading numbers, separated by commas. Do not use spaces. For example: 1,3,5-12

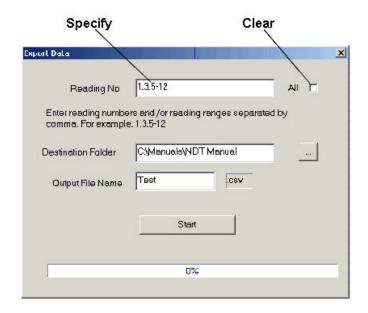


Figure 285. Exporting Data Ranges and Selected Readings

Note Be sure that you specify the reading numbers, not the index numbers.

- 5. Specify the folder where you would like to place the report files. The folder that is currently selected appears in the Destination Folder field. To change the folder, click the "..." (Browse) button.
- 6. Enter a name for the export file in the Output File Name field.

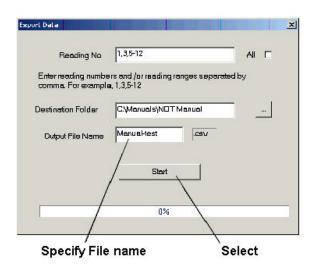


Figure 286. Naming the Export File

- 7. Click the Start button.
- 8. When the progress bar shows 100%, click the Done button.

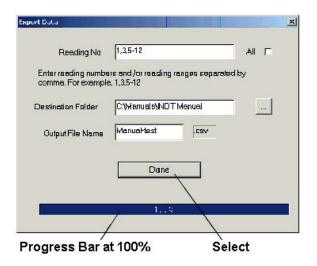


Figure 287. Finishing the Process

Note This export creates one text file that includes all of the specified readings as comma separated values. This file can be imported into any program that accepts data in CSV format.

Printing Certificates of Analysis

NDT can output certificates of analysis for any reading. To set up a default certificate for printing, select the Customize option from the Tools Menu.



Figure 288. Selecting Customize

This will bring up a pop-up window with options you can customize for your own use.

13 Connectivity Printing Certificates of Analysis

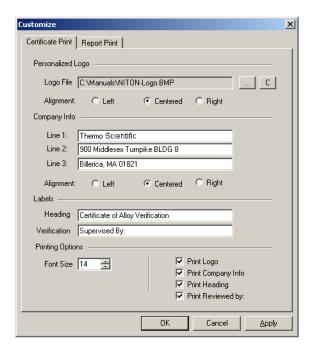


Figure 289. The Certificate Print Tab

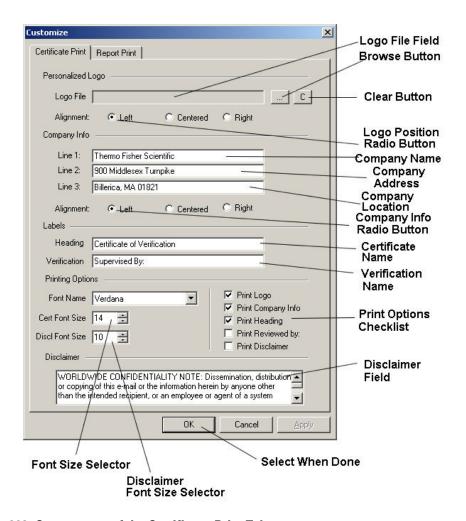


Figure 290. Components of the Certificate Print Tab

The Logo Section

In the Logo Section of the certificate pop-up window, you can add your company logo to the printed document.

Logo File Field

You can type the location of your company logo directly into the Logo File Field if you know the location of the logo on your computer or local network

"..." Browse Screen Button

Selecting this button will bring up a standard browsing pop-up window, allowing you to find the logo graphics file if you are not sure of the logo graphics file?s location. This will automatically fill that location into the Logo File Field when you confirm the selection.

Clear Screen Button

Selecting this button will clear the Logo File Field and reset it to a null (blank) condition.

Logo Position Radio Screen Buttons

Selecting one of these three buttons allows you to position your company?s logo on the certificate document. Only one of these buttons may be selected at any given time. Selecting one button will clear the other buttons.

The Company Info Section

In the Company Info Section of the certificate pop-up window, you can add your company's name and location to the printed document. There are no restrictions on what you type into these fields - the names of the fields are just suggestions. If you want the Company Address field to contain your location - i.e. "Billerica, MA USA 01821" - and the Company Location field to contain your Telephone number - i.e. "Tel: 978-670-7460" - you are free to do so.

Company Name Field

You can type your company's name - i.e. "Thermo Fisher Scientific" - directly into this field.

Company Address Field

You can type your company's street address - i.e. "900 Middlesex Turnpike, Bldg #8" - directly into this field.

Company Location Field

You can type your company's city, state, province, or country - i.e. "Billerica, MA USA 01821" directly into this field.

Company Info Radio Screen Buttons

Selecting one of these three buttons allows you to position your company's information on the certificate document. Only one of these buttons may be selected at any given time. Selecting one button will clear the other buttons.

The Heading Section

The Heading is the Certificate Name. You can use this field to enter the type of analysis certificate you are printing - i.e. "Certificate of Alloy Analysis", "Certificate of Testing", "Certificate of Analysis", or whatever else is customary for your company and the type of analysis done in the Reading.

The Printing Options

The Printing Options allows you to select which Section you want to print along with the actual analysis. Selecting an option will place a check in the box if there is none there before your selection, and remove a check if there is one already there. All Sections which are checked will be included in the printout.

Printing the Certificate

Right-click on the Reading for which you want a certificate printed, and select the "Print Certificate" option from the pop-up menu.



Figure 291. Select print Certificate from the Menu

This will send the analysis and heading information directly to your regular printer.

The Certificate of Analysis

Here is a sample C of A printed by NDT:

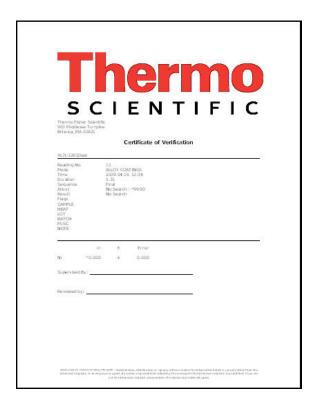


Figure 292. Example Certificate

Closing a Report

To close a report, select Close from the File Menu.

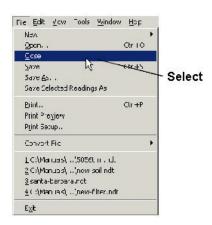


Figure 293. Closing a Report from the File Menu

Deleting a Report

To delete a report:

- 1. Click the Open button.
- 2. Right-click the name of the report to highlight it.



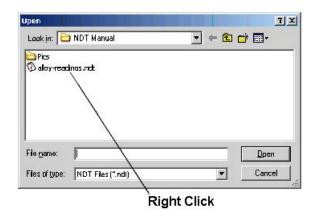


Figure 294. Selecting a Report

3. A Pop-up window will appear.

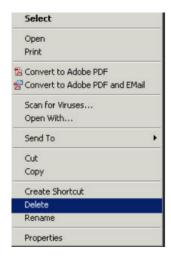


Figure 295. The Pop-up Menu

4. Choose Delete from the pop-up menu.



Figure 296. The File Deletion Pop-up Menu

5. Click the Yes button.

Printing from NDT

Niton Data Transfer lets you print both reports and spectra graphs. See <u>See "Printing Reports and Spectra Graphs from Niton Data Transfer" <XREF></u> for more information.

You can also print spectra graphs by first exporting them to Microsoft Excel. This method gives you more formatting options. See <u>"Printing Spectra Graphs from Excel"</u> for more information.

Niton NDT uses a WYSIWYG (What You See Is What You Get) approach to report printing and creation. Displayed columns and readings will print, and hidden columns and readings will not. If you find your printout is missing data you are interested in, please see the section on "Viewing Data and Creating Reports".

Printing Reports and Spectra Graphs from Niton Data Transfer

Checking Paper Size and Orientation

Before printing a report, check that the paper size and orientation are correct. There are two ways to do this:

- Click the Print Preview Screen Button. This shows how the page will be printed.
- Use the Print Setup menu item. This displays the paper size and orientation and also lets you change them.

To use the Print Setup menu item to change the paper size and orientation:

- 1. Click the File Menu.
- 2. Click Print Setup to display the Print Setup Dialog Box.



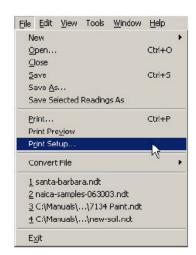


Figure 297. Select Print Setup

- 3. Select the correct paper size from the drop-down list.
- 4. Click the radio button for the correct orientation. The icon illustrates the orientation that is selected.

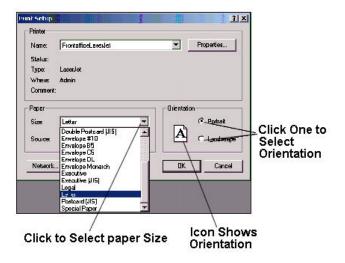


Figure 298. Print Setup Dialog Box

5. Click the OK Screen Button.

Printing from Niton Data Transfer

Note Niton Data Transfer only prints the report or spectra graph that is currently displayed. You must open the report or spectra graph before you print it.

13 Connectivity Printing from NDT

To print the report or spectra graph:



- 1. Click the Print Screen Button to display the Print Dialog Box.
- 2. Check that the correct printer is displayed in the Name field. If it is not, use the drop-down list to select the printer. If you want to change the properties of the printer, refer to the documentation for your printer for more information.
- 3. Select the pages that you want to print by doing one of the following:
 - a. To print the entire report, click the All radio button.
 - b. To print only part of the report, enter the first and last page numbers for the part that you want to print.
- 4. Specify the number of copies that you want to print.
- 5. Click OK.

Printing Spectra Graphs from Excel

Printing a spectra graph from Excel requires:

- 1. Exporting the spectra data from Niton Data Transfer.
- 2. Importing the spectra data into Excel.
- 3. Creating the graph in Excel.
- 4. Printing the graph.

These steps are explained in detail below.

Note Using this feature requires a separate copy of Microsoft Excel. Niton Data Transfer does not include a copy of Excel.

Exporting the Spectra Data

To export the spectra data from Niton Data Transfer:

- 1. Open the report. For more information, see "Opening an Existing Report".
- 2. Select Export Spectra from the Tools Menu.



Figure 299. Select Export Spectra from the Menu

3. Specify the reading numbers that you want to export.

If you want to export the spectra data for all the reading numbers, you do not need to make a change for the reading numbers. Leave the All box checked and move on to the next step.

If you want to export only some of the spectra data:

- 1. Uncheck the All box.
- 2. Enter the reading numbers. You may enter individual reading numbers, or ranges of reading numbers, separated by commas. Do not use spaces. For example: 1,3,5-12

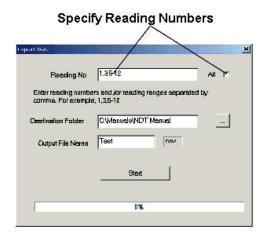


Figure 300. Specify Reading Numbers

13 Connectivity Printing from NDT

3. Specify the folder where you want place the spectra files. The folder that is currently selected appears in the Destination Folder field. To change the folder, click the "..." button.

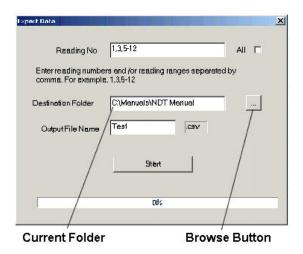


Figure 301. Changing Folders with the Browse Button

4. Enter a name for the export file in the Output Filename field.

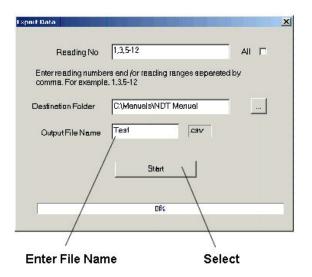


Figure 302. Naming the Export File

Note The data for each reading is in a separate file. The file name for each reading is the file name that you specified, plus the Rdg # and reading number. For example: First_1_3, First_3_14, First_5_16.

- 5. Click the Start button.
- 6. When the progress bar shows 100%, click the Done button.

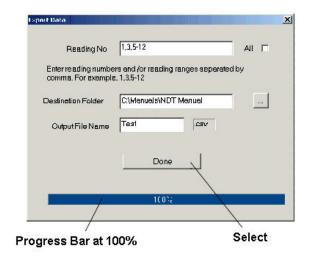


Figure 303. Finish the Export Process

Importing the Spectra Data

To import the spectra data into Excel:

- 1. Open Excel. To do this:
- 2. Click the Start button.
 - a. Select Programs.
 - b. Click Microsoft Excel.

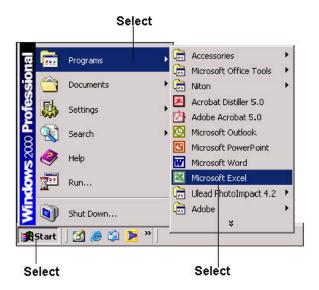


Figure 304. Opening Excel

- 3. Select Open from the File Menu.
- 4. In the Open Dialog Box, select Text Files from the drop-down list.

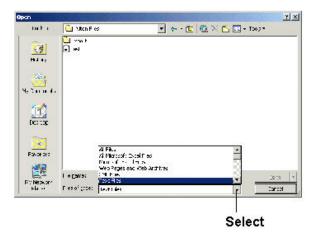


Figure 305. Selecting File Type

5. Move to the directory that contains the spectra data files. Double click on a folder to move to a lower level. Click the Up One Level button

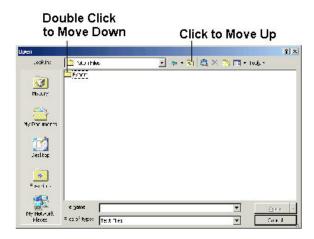


Figure 306. Navigating to the Proper Folder

6. Double-click on the name of the file that you want to import into Excel.

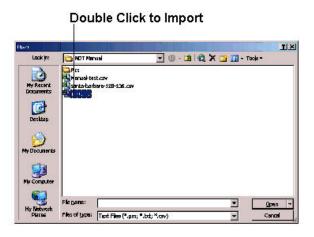


Figure 307. Importing the Data

The data is now in Excel. You should see two columns of numbers, similar to this:

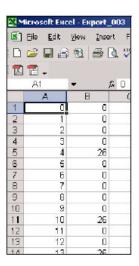


Figure 308. The Data File Displayed in Excel

The first number is the **Bin Number**, which gives the number of the bin in that row. The second number is the number of **Counts** in the **Bin**. Bins separate the number of **Counts** at different energies. Since different elements fluoresce at different energies, the number of Counts in a Bin can tell you how much of each element is in a sample. The size of the Bin depends on the testing mode used on the sample.

Creating the Graph

To create the graph:

1. Select Chart from the Insert Menu.



Figure 309. Selecting Chart from the Menu

2. Select the Scatter Chart icon with data points connected by lines without markers.

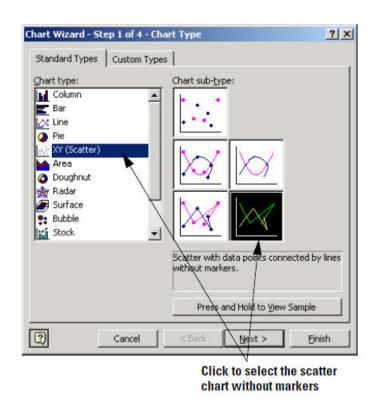


Figure 310. Selecting Scatter Chart

3. Click the Next Screen Button to move to Step 2.

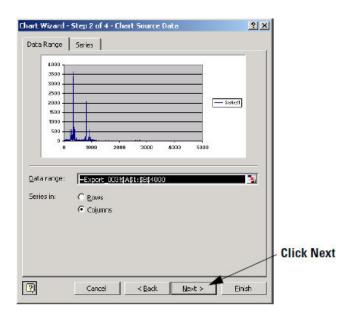


Figure 311. Selecting Next

13 Connectivity Printing from NDT

4. Click the Next Screen Button to move to Step 3.

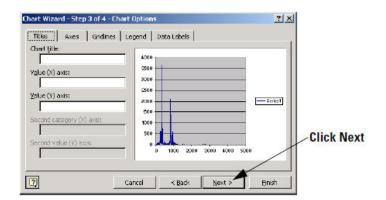


Figure 312. Selecting Next

- 5. Click the Next Button to move to Step 4.
- 6. Click the radio button for As new sheet.

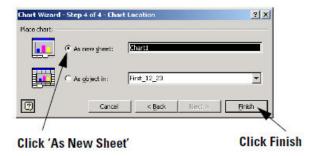


Figure 313. Selecting "As New Sheet"

7. Click the Finish Button.

Printing the Graph

Note For more information about how to format the graph before printing it, refer to the documentation for Microsoft Excel.

To print the graph:

1. Select Print from the File Menu.

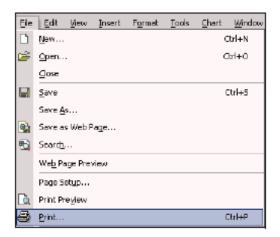


Figure 314. Selecting Print from the Menu

2. Check that the correct printer is displayed in the Name field. If it is not, use the drop-down list to select the printer. If you want to change the properties of the printer, refer to the documentation for your printer for more information.

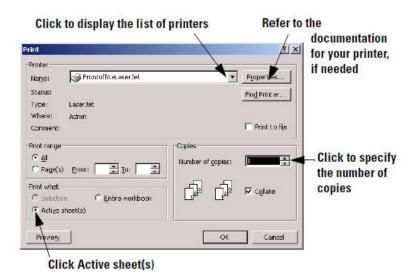


Figure 315. Print Dialog Box

3. In the Print what section, click the radio button for Active sheet(s).

Note Do not print the entire workbook. This prints the spectra data as two columns of numbers. It will require 60-70 pages to print all the data.

- 1. Specify the number of copies that you want to print.
- 2. Click the OK button.

Creating a Report File from Multiple Source Files

You can copy readings from one report file and paste them into another file, or into a new file. Select the readings you want to copy, using the shift or control keys as appropriate.

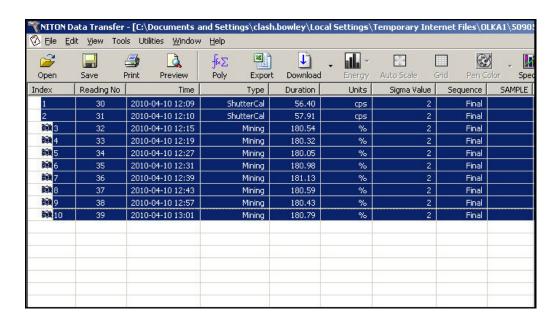


Figure 316. Selecting Multiple Readings

Right click on the selected files, and select Copy Readings (Binary) from the Pop-up Menu.

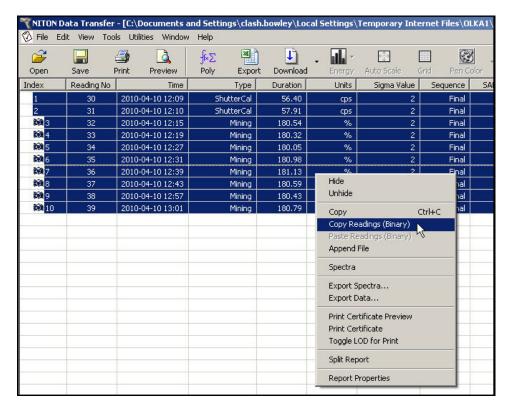


Figure 317. Copying Readings

Open a destination report file to transfer the readings into.

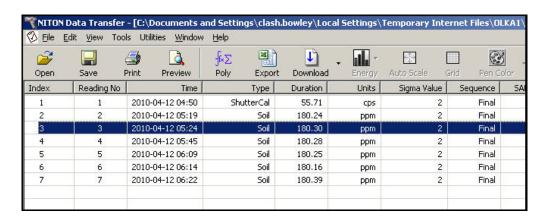


Figure 318. New Destination File

Right-click on any of the readings, and select Paste Readings (Binary) from the Pop-up Menu.

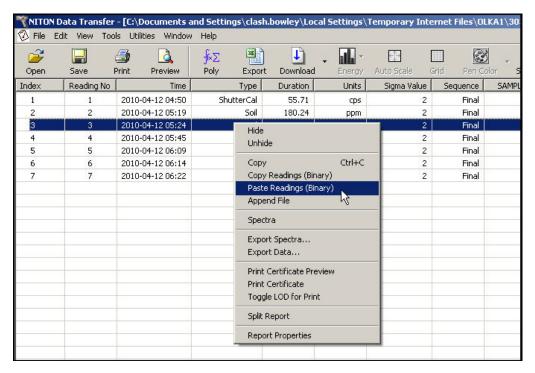


Figure 319. Pasting Multiple Readings Into New Destination File

The new readings will be highlighted in color to distinguish them from the readings already in place.

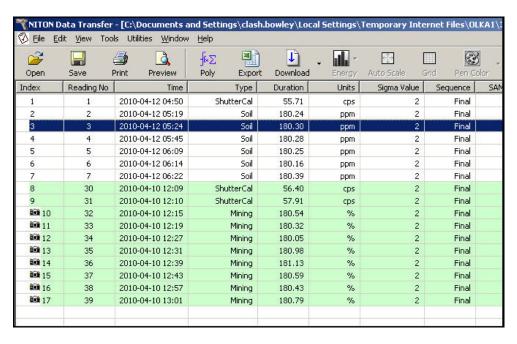


Figure 320. Multiple Readings Pasted

If you paste readings from yet another report file, they will be displayed in another color. Each group of readings copied in will have a different color.

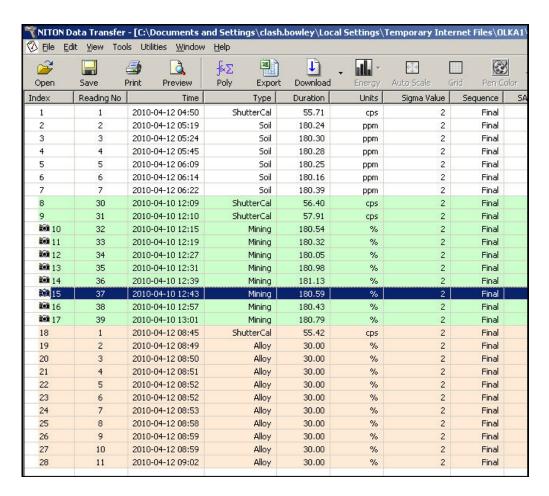


Figure 321. Multiple Readings from Multiple Files Pasted

The color distinctions will disappear when you save the file.

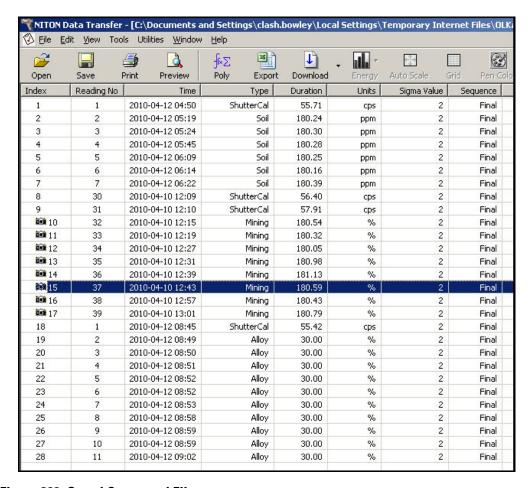


Figure 322. Saved Compound File

The original information is still there, though, and can be retrieved by right clicking on a reading and selecting Report Properties.

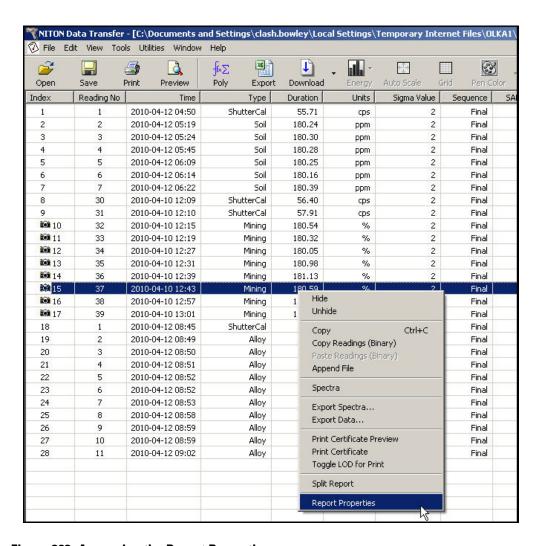


Figure 323. Accessing the Report Properties

Each analyzer whose readings have been pasted into the Compound Report will have a separate tab in properties. The tab for the analyzer which was used to take the reading you highlighted will be displayed on top.

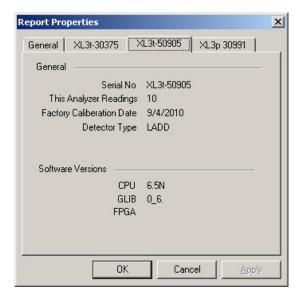


Figure 324. Reading the Properties Window

You cannot upload a compound file into an analyzer. Compound files are for data correlation only.

Controlling Your Analyzer From Your PC

The NDTr program allows you to completely control your Niton analyzer remotely, from your computer. It works over serial or USB connection over the supplied connector, or Bluetooth wireless communication. See Connecting the XRF Analyzer to Your PC for details on how to connect your Analyzer and PC.

The NDTr Toolbar

The NDTr Toolbar is a string of icons along the top of the NDTr window. It looks like this:

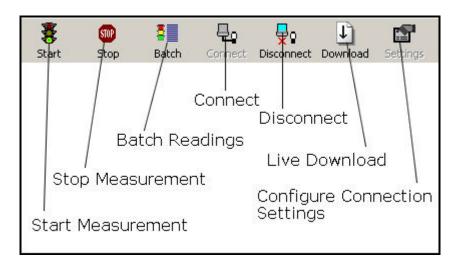


Figure 325. The NDTr Toolbar

Start Measurement

Clicking this icon will initiate a measurement in whatever mode the analyzer is in currently.

Stop Measurement

Clicking this icon will halt any ongoing measurement on the analyzer.

Configure Connection Settings

Clicking this icon will allow you to change your configuration settings.

Connect

Clicking this icon will attempt to establish a connection between your computer and your analyzer.

Disconnect

Clicking this icon will disconnect your computer from your analyzer.

Live Download

See Live Download from NDTr

Configure Connection Settings

Clicking on the Configure Connection Settings icon allows you to change the Com Port for connecting your computer to your analyzer. Once you click on the icon, a settings dialog box will appear.



Figure 326. Connection Settings Dialog Box

Selecting the Com Port

Selecting the down arrow in the "Connect Using" field will display the various Com Ports on the computer that the analyzer can connect through.

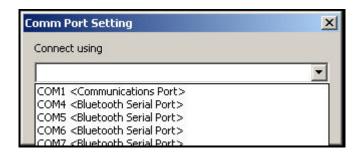


Figure 327. Selecting the Com Port

Select the proper com port from the list, then select the OK Button.

Operating Your Analyzer Remotely

NDTr version 7 and above will automatically select the proper virtual interface for you, whether you have a Thermo Scientific Niton XL2 or XL3. The virtual interface operates exactly as the analyzer would. Selecting the buttons, icons and menus with your mouse works exactly as if you were selecting them with your finger or stylus on the real analyzer.

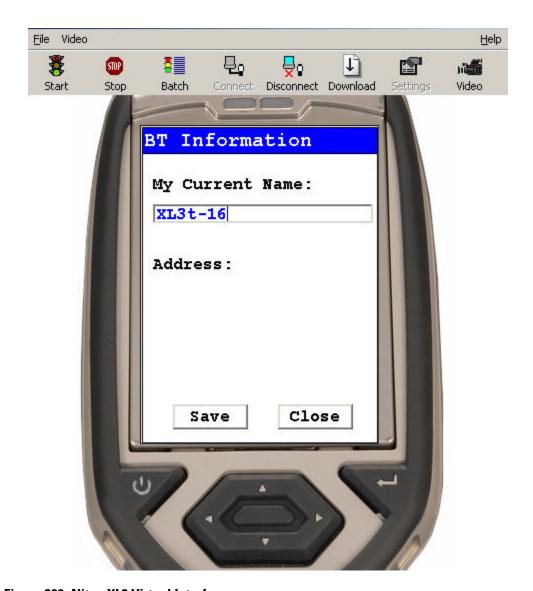


Figure 328. Niton XL3 Virtual Interface

Live Download from NDTr

Once you have connected to your analyzer using NDTr, click on the Download icon on the NDTr toolbar. When you click the Download icon, a Download dialog box will appear.



Figure 329. File Dialog Box

Dest Folder

This field shows the last used save folder, defaulting to the folder where NDT is installed.

... (Browse Folders) Button

Selecting this button enables you to select a different folder for saving the file. This will change the Dest Folder Field.

Selected File

This shows the filename the reading will be saved to unless you change it.

... (Browse Files) Button

Selecting this button enables you to select a different file name by browsing the file listing. The file extension ".ndt" will be appended to the name - i.e. File name "file" will be saved as "file.ndt" and the file will be in the NDT format.

Save the Reading to CSV File as Well Checkbox

Selecting this checkbox enables you to create a second autosave file with CSV format for importing into spreadsheets such as Excel. This file will have the same name as the NDT file above, but with the file extension ".csv" instead - i.e. "test.ndt" will be saved as "test.csv" as well. The checkbox is selected when there is a check in it, and deselected when it is empty.

Always Show this Dialog Box when the File button is Pressed Checkbox

Selecting this checkbox will enable you to change the filename whenever you want. Deselecting this checkbox will save the file under the same name in the same folder every time. The checkbox is selected when there is a check in it, and deselected when it is empty.

Setting up Bluetooth

Step 1: Insert the Bluetooth USB Adapter CD



Figure 330. The Bluetooth Setup Screen

Step 2: Click the Next > Button

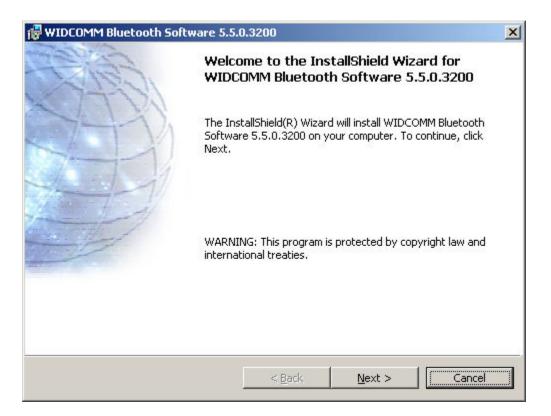


Figure 331. The Bluetooth InstallShield Wizard Screen

Step 3. Accept the Agreement and press the Next Button

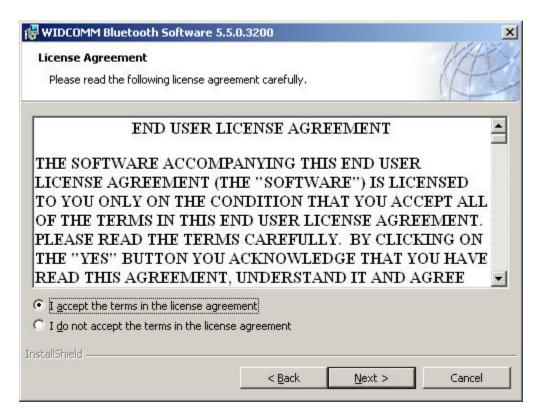


Figure 332. The Bluetooth End User License Agreement

Step 4: Accept the selected folder and press the Next Button

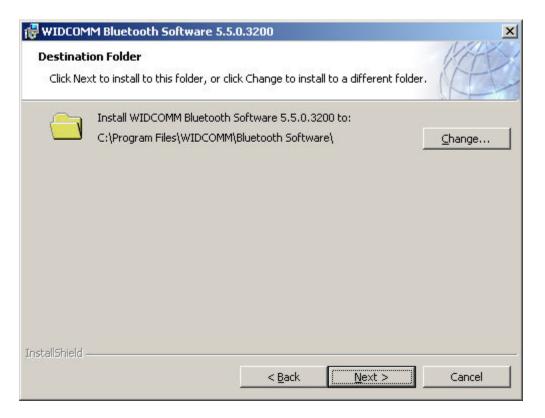


Figure 333. The Bluetooth Folder Selection Screen

Step 5: Press the Install Button to begin the Installation.

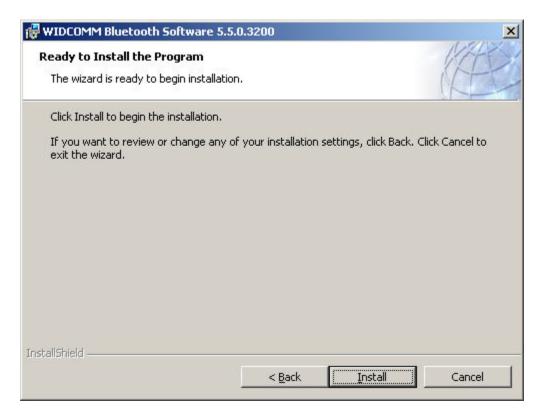


Figure 334. The Bluetooth Software Installation Screen

Step 6: The screen will show a progress bar as the software is installed.

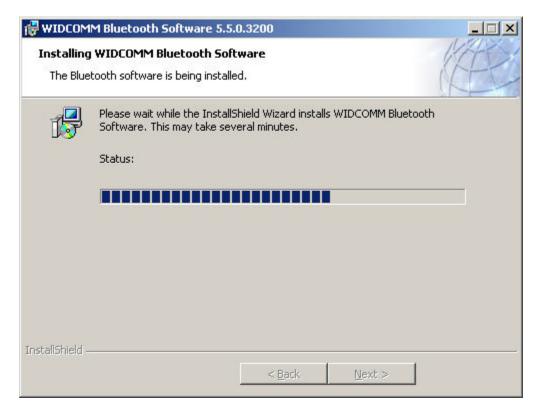


Figure 335. The Progress Bar

Step 7: When the "Bluetooth device not detected" message appears, insert your Bluetooth device into any open USB port, and select the OK Button.



Figure 336. The Bluetooth Device Missing Message

Step 8: The Install wizard will finish copying the files to the system.

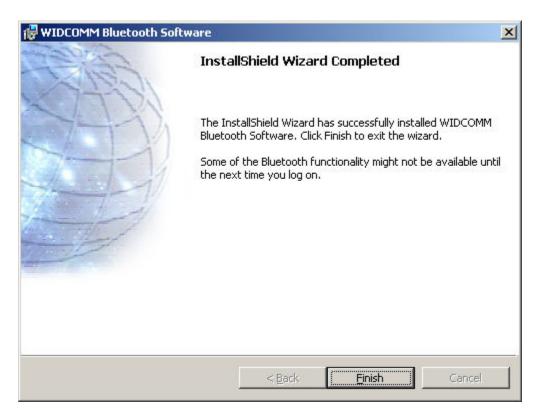


Figure 337. The InstallShield Wizard Completed Screen

Step 9: Select the Finish Button to complete the installation.

Connecting the Bluetooth device to your XRF Analyzer.

Step 1: On the System tray, Right click on the Bluetooth icon and Select "Start Using Bluetooth".



Figure 338. The Bluetooth Icon in the Setting Tray

Step 2: Select the Next Button with the following options selected.

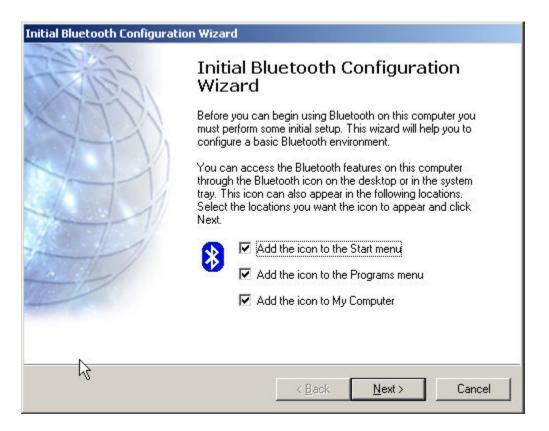


Figure 339. The Bluetooth Icon in the Setting Tray

Step 3: Select the Next Button with the default names selected by the system.

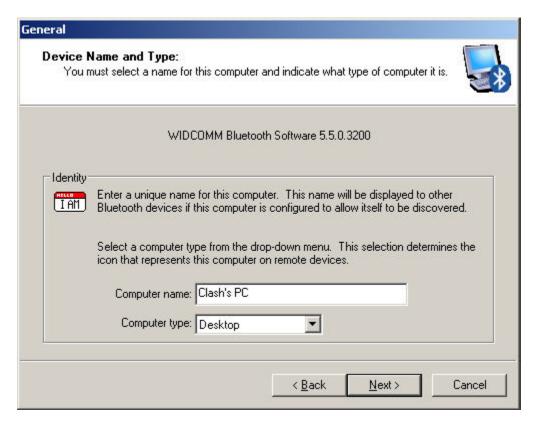


Figure 340. The Device Name and Type

Step 4: Select the Next Button.

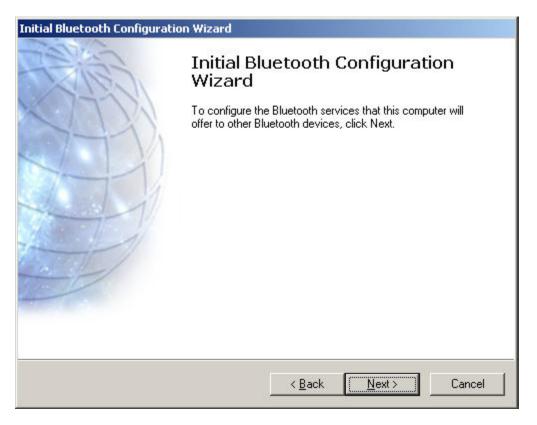


Figure 341. Configuring the Bluetooth Services

Step 5: Select Bluetooth Serial Port if it is not selected, then Select the Next Button.

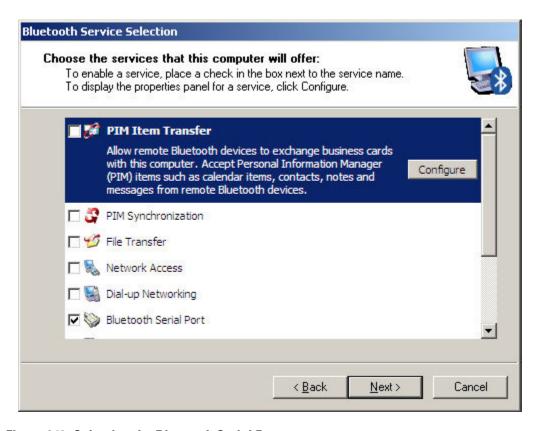


Figure 342. Selecting the Bluetooth Serial Port

Step 6: IMPORTANT: TURN ON THE XRF INSTRUMENT, then select the Cancel Button.

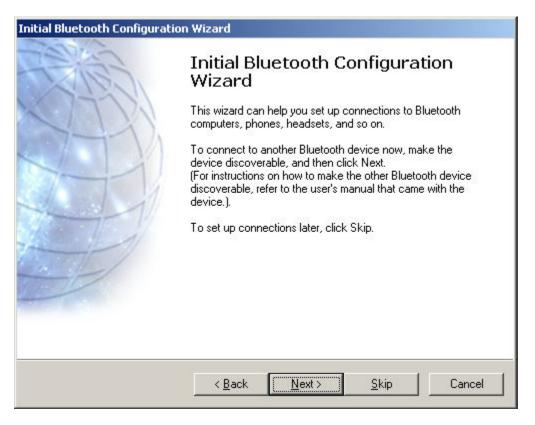


Figure 343. Stopping the Configuration Wizard

Step 7: Right Click on the Bluetooth icon in your System tray, and select Explore My Bluetooth Places.



Figure 344. Selecting My Bluetooth Places

Step 8: Select Add a Bluetooth Device

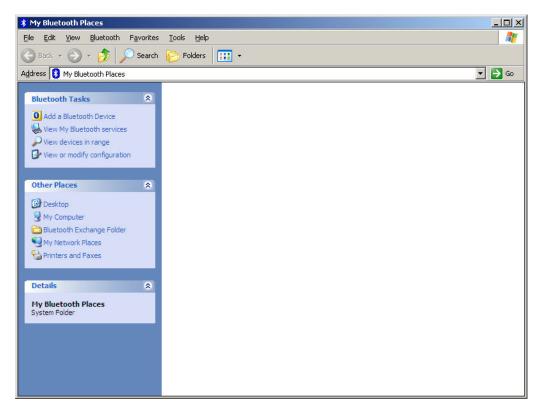


Figure 345. Adding a Bluetooth Device

Step 9: The wizard will search for the instrument. When your analyzer's icon appears, select the analyzer's icon and select the Next Button.



Figure 346. Searching for Your Analyzer

Step 10: Right-Click on the My Bluetooth Serial Port icon, and select Properties.

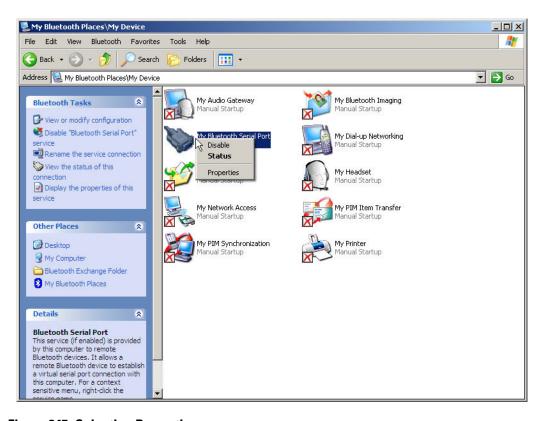


Figure 347. Selecting Properties

Step 11: The Bluetooth Device will attempt to pair with your analyzr. When it askes for the passkey, type 0000.



Figure 348. Pairing With Your Analyzer

Step 12: Select COM0, then the Next Button.



Figure 349. Selecting COM0

Step 13: Select the OK Button.

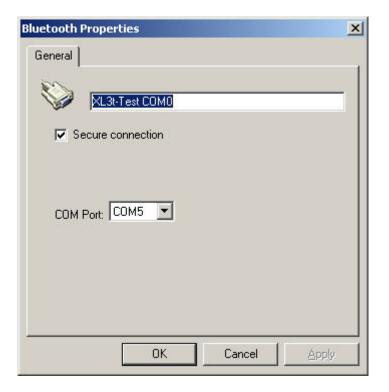


Figure 350. Establishing the Connection

Step 14: Select the Finish Button.



Figure 351. Completing the Setup

Step 14: Your analyzer is now connected. Note the COM Port assigned, as you must use this COM port when using Bluetooth with your analyzer. Select the OK Button.

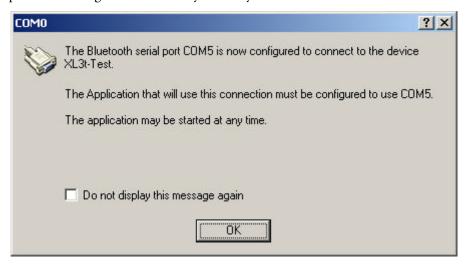


Figure 352. Note the Assigned COM Port

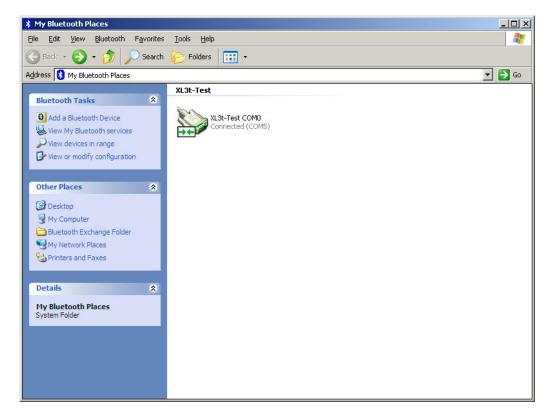


Figure 353. The Serial Connection icon

Troubleshooting Your Connection

If the XRF instrument doesn't connect with Bluetooth, load My Bluetooth Places, and check the following settings:

Step 1: Select View or Modify Configuration.

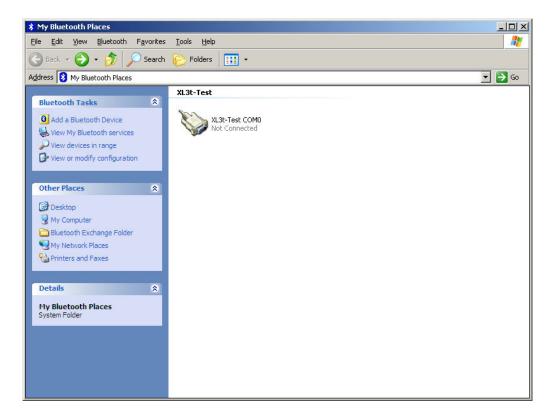


Figure 354. My Bluetooth Places Screen

Step 2: Select Client Applications.

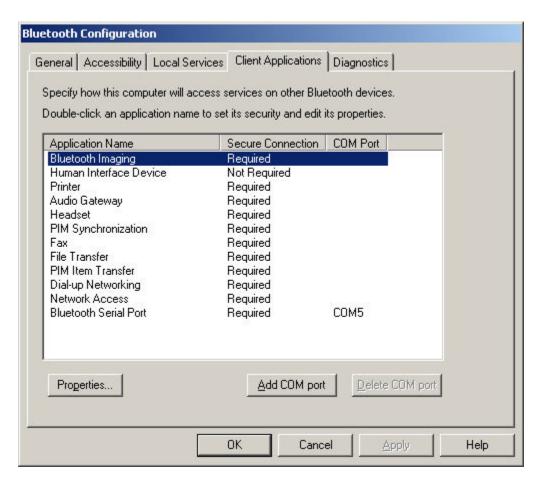


Figure 355. Client Applications

Step 3: Select Bluetooth Serial Port and then click the Properties Button.

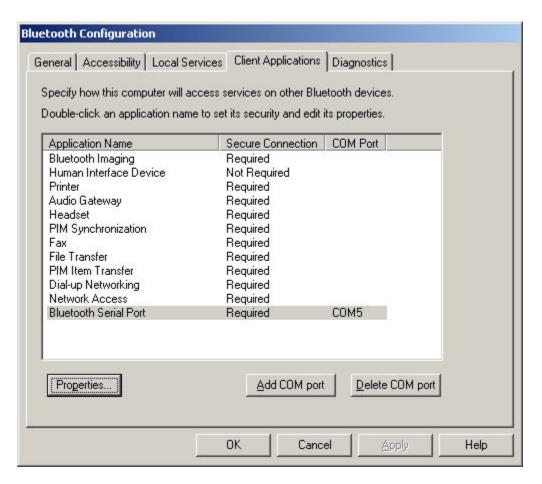


Figure 356. Selecting the Serial Port

Step 4: Uncheck Secure Connection option.

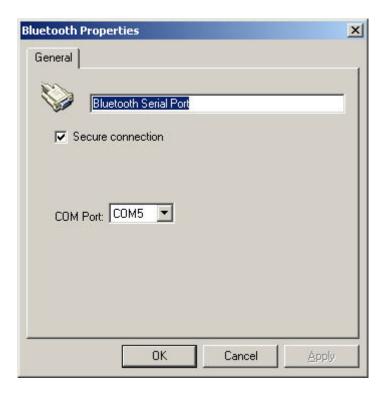


Figure 357. Deselecting Secure Connections

Step 5: Make sure the Secure Connection is unchecked.

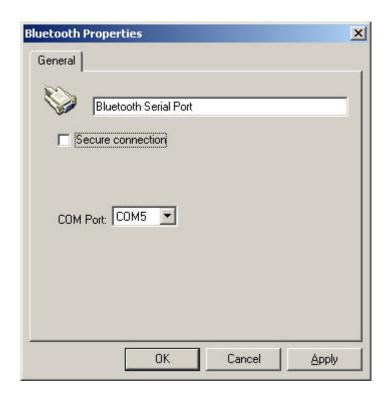


Figure 358. The Properties Screen

Step 6: You should see "Not Required" in the Bluetooth Serial Port configuration.

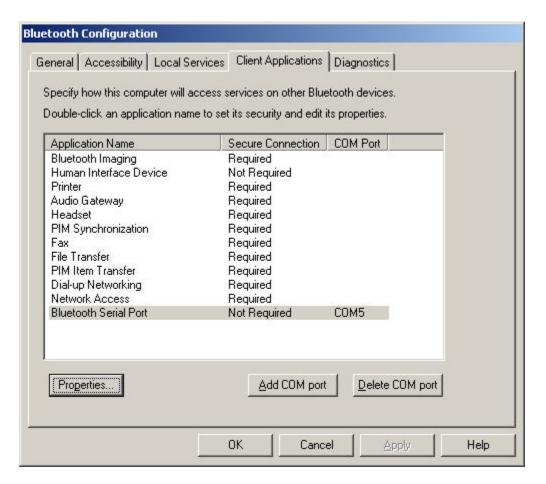


Figure 359. Secure Connection Not Required

Now you are ready to use the Bluetooth adapter. See Using the Wireless USB Adapter to Connect the XRF Analyzer for using your analyzer with a Bluetooth Connection.

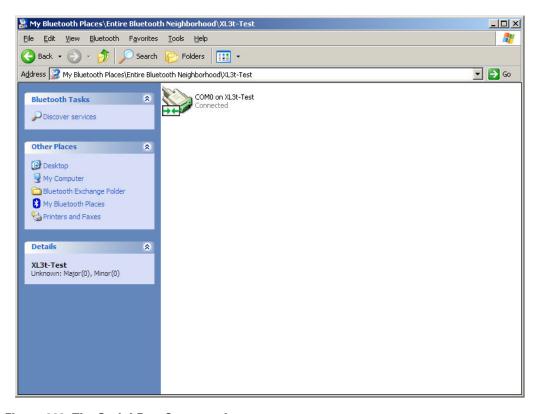


Figure 360. The Serial Port Connected

Troubleshooting Your Connection

Interfacing Thermo Scientific Niton Analyzers and the Trimble/ArcPad GIS System

Setting Up Your Trimble Handheld

Trimble Setup

Setup an incoming Bluetooth port on the handheld.

Click on Start at the upper left corner of the screen.



Figure 361. The Trimble Main Screen

Click on Settings in the pull down menu.



Figure 362. Selecting Settings

Click the connections tab at the bottom of the page.

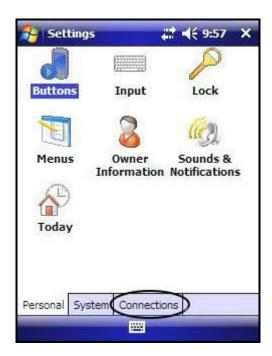


Figure 363. Selecting the Connections Tab

Select Bluetooth settings.

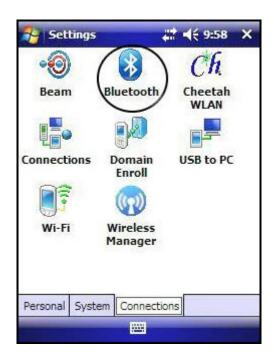


Figure 364. Selecting the Bluetooth Settings

Click the far-right tab labeled "COM Ports" in the lower right hand corner and edit the incoming port.



Figure 365. Selecting the COM Ports Tab

You may need to try this with different COM ports to find one that works properly.

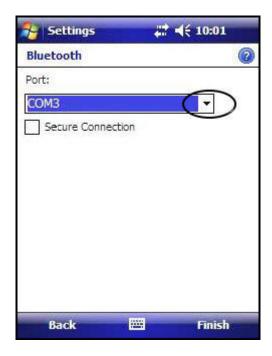


Figure 366. The COM Ports Tab

To change the port, click and hold "Incoming Port" then choose edit.

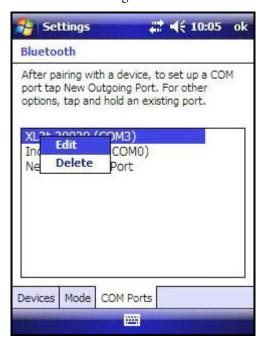


Figure 367. Selecting the Bluetooth Settings

Choose a port and be sure to remember which port you select. Select Finish when done.

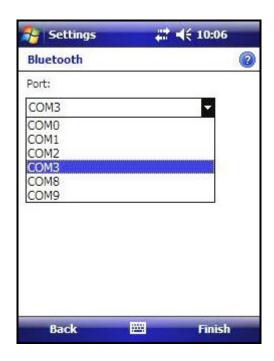


Figure 368. Selecting the Bluetooth Settings

Click the middle tab labeled Mode and select the box to the left of "Turn on Bluetooth" and the box to the left of "Make this device visible to other devices".

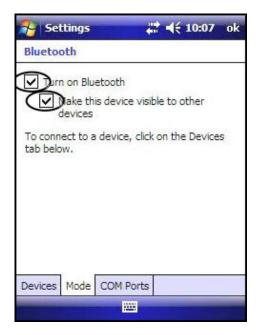


Figure 369. Turning Bluetooth Options On

14 Interfacing Thermo Scientific Niton Analyzers and the Trimble/ArcPad GIS System Setting Up Your Analyzer

When the connection is ready, a window will pop up on the Trimble asking you if you want to connect with your analyzer. Select Yes.



Figure 370. Connecting With Your Analyzer

This may connect the Trimble to the Analyzer, but not the Analyzer to the Trimble. If so, attempt to connect again to the Trimble from the Analyzer.

Setting Up Your Analyzer

From the Main Menu, select the System icon. This will load the System Menu. From the System Menu, select the Printer icon.

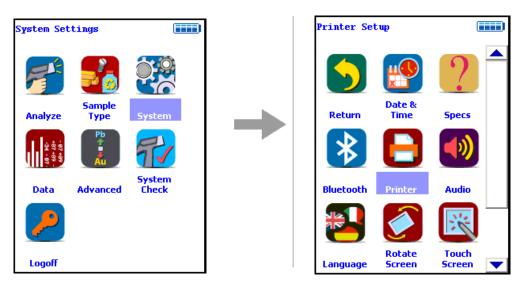


Figure 371. The Printer Setup Menu Path

Enable the automatic printing of readings by selecting the Auto Print Readings checkbox. Finally, select the Close Button.

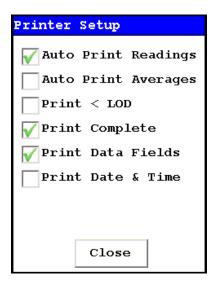
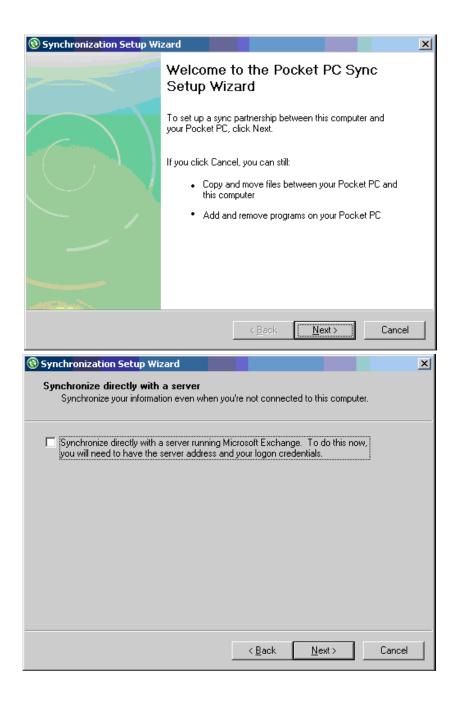


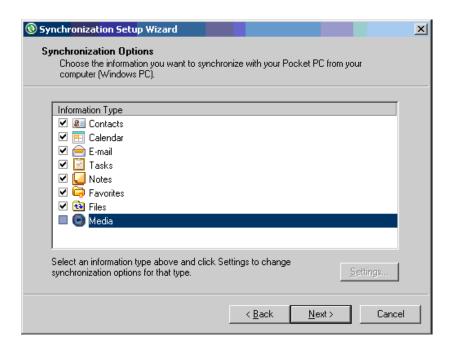
Figure 372. The Printer Setup Screen

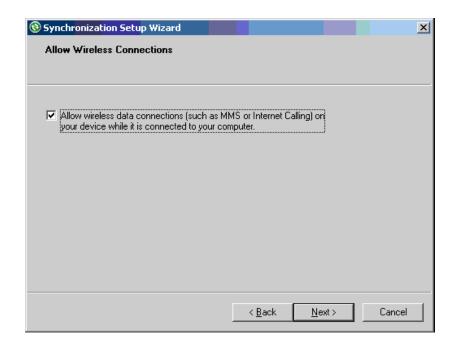
Installing ArcPad GIS Software To Your PC

Place the software DVD that came with your Trimble into the disc drive of the computer and follow the on-screen prompts to install the syncing software onto your PC.

14 Interfacing Thermo Scientific Niton Analyzers and the Trimble/ArcPad GIS System Installing ArcPad GIS Software To Your PC







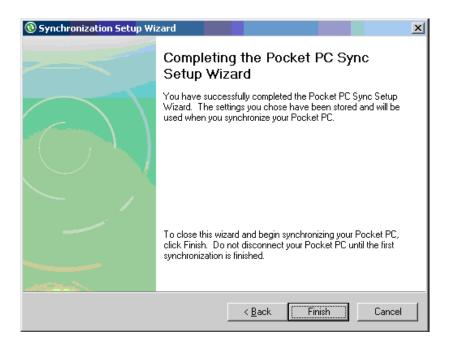


Figure 373. The PocketPC Sync Installation Sequence

Note You may not want to synchronize your email and personal folders with the Trimble device, especially if the Trimble is shared with others. The Trimble acts as a PDA, and syncing email and personal folders will share them with anyone having access to the device.

Open Microsoft ActiveSync and choose File from the tool bar and then Synchronize in the pull down menu.



Figure 374. Connecting to the Trimble

Now install the ArcGIS software onto your PC. Place the DVD into your DVD drive.

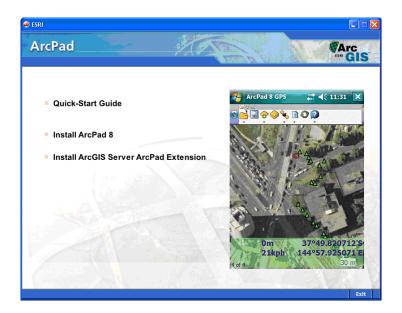


Figure 375. The ArcPad Splash Page

From the splash screen menu, select Install ArcPad 8.



Figure 376. The ArcPad Splash Page

Select Next.



Figure 377. Beginning Setup

Browse to the Folder you want to use, and select Next.

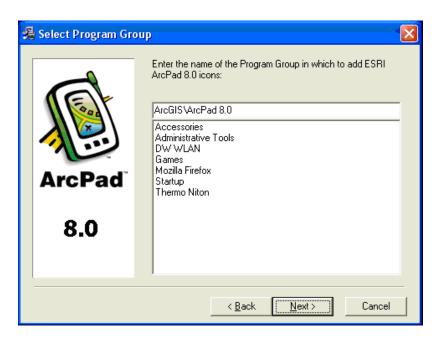


Figure 378. The ArcPad Group

If you want, change the name of the group. Otherwise select Next.



Figure 379. Ready to Install

Select the Next Button.



Figure 380. The ArcPad Splash Page

A progress bar will display as the components are installed.

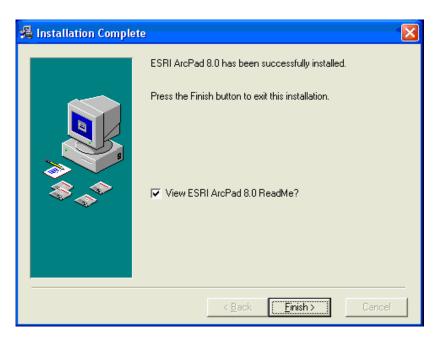


Figure 381. The ArcPad Splash Page

Select the Finish Button when the installation is done.

Select the ArcPad Deployment Manager from the ArcPad menu.

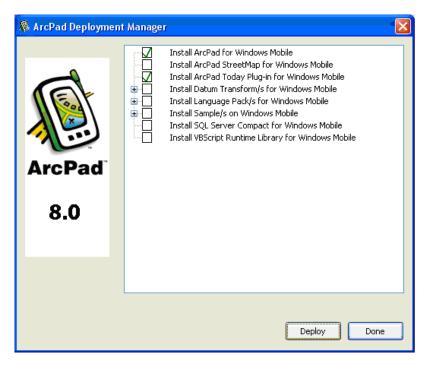


Figure 382. The Deployment Manager

Select the checkboxes next to Install ArcPad for Windows Mobile and Install ArcPad Today Plug-in for Windows Mobile.

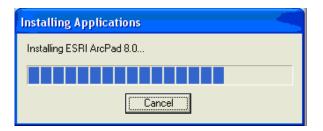


Figure 383. Progress Bar

A progress bar will display as the components are installed.

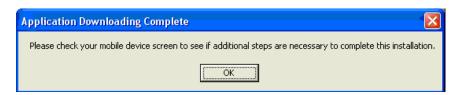


Figure 384. Deployment Complete

Select OK.

Deploying ArcPad to the Trimble and Setting Up GPS

Once the device is synchronized, Select Tools in the toolbar and choose Add/Remove Programs from the drop down menu.

Another window will open. Click boxes next to program to be added: ESRI ArcPad 8.0, ESRI ArcPad, etc.

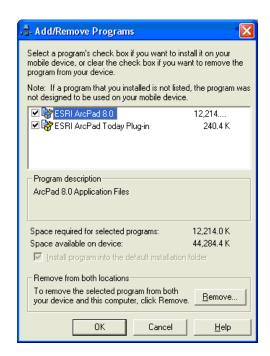


Figure 385. Adding and Removing Deployed Programs

Select the OK Button and follow the remainder of the screen prompts on the PC and mobile device.

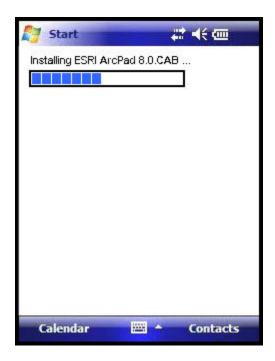


Figure 386. Progress Bar

From the home menu on the Trimble click on Start.



Figure 387. ArcPad Start Button

A pull down menu will appear, choose the ArcPad program.



Figure 388. Selecting ArcPad

When ArcPad opens, press the green OK Button in the lower left corner of the screen.

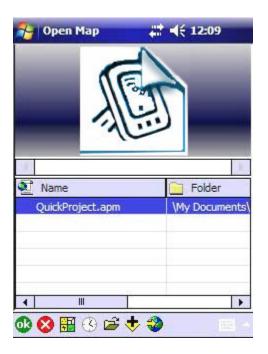


Figure 389. Select OK

14 Interfacing Thermo Scientific Niton Analyzers and the Trimble/ArcPad GIS System Deploying ArcPad to the Trimble and Setting Up GPS

The main toolbar displays an icon that looks like a satellite. Click on the icon and then select GPS Preferences from the menu.



Figure 390. Select GPS Preferences

The default protocol – NMEA 0183 - and port 4 are the correct settings for a Juno. The Port is changed by clicking on the COM# displayed and choosing COM4:GPS from the pull down menu.

Note Do not change port settings from COM 4 once initially established

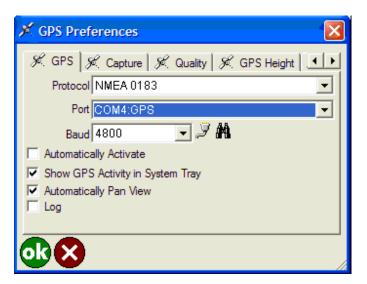


Figure 391. Setting up the GPS

Click the OK Button in the bottom left corner to return to the main page.

Setup the ArcPad Port for Receiving Data from the Analyzer

Click on the Tools icon which looks like a sheet of paper with the upper right corner folded over, then select Advanced Settings.

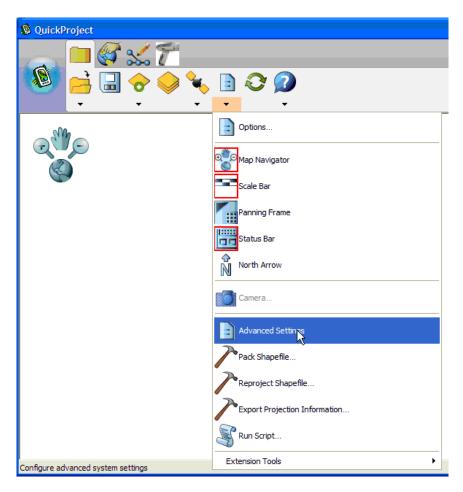


Figure 392. The Tools Menu

Click the AUX tab at the bottom of the screen. If the AUX tab does not appear, click on the right arrow at the bottom of the screen until the AUX tab appears, then click it. Select a port which is not currently being used. The Aux port must be set to the Com port of the instrument selected. In Figure 393 below, Com 4 and Com 7 are in use..

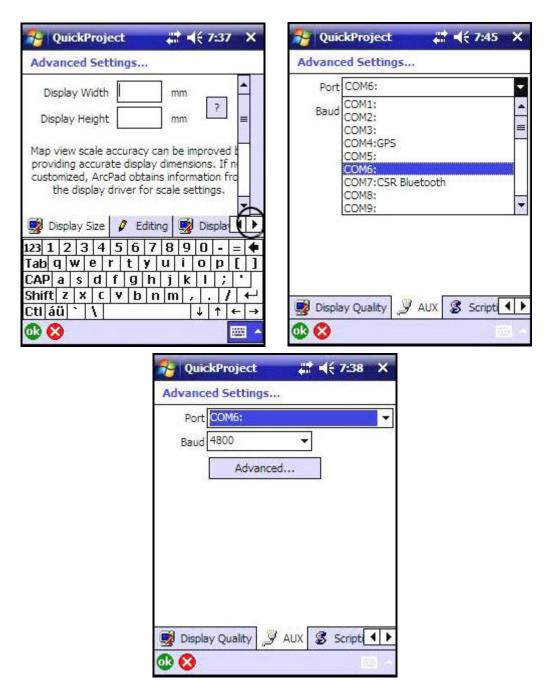


Figure 393. Selecting the AUX COM Port

Click the green OK Button at the bottom left corner of the screen.

Installing the Analyzer Applet

Extract the files from the zipped folder attached in the e-mail. Open the My Computer folder on the PC by clicking the My Computer icon on the desktop. Select the Mobile Device System Folder and double-click to open.

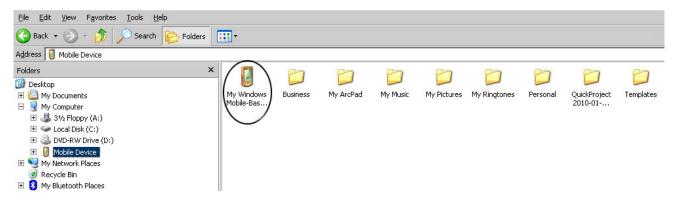


Figure 394. The Mobile Device System Folder

Double-click My Windows Mobile-Based Devices to open the folder.

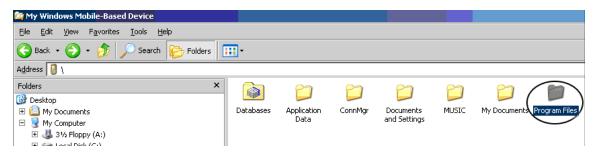


Figure 395. The My Windows Mobile-Based Device Folder

Click Program Files twice to open folder.

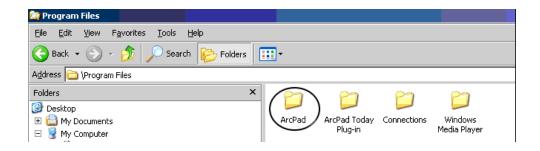


Figure 396. The Programs Folder

Click ArcPad folder twice to open.

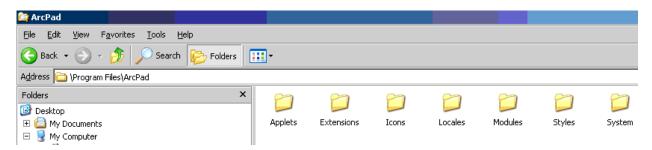


Figure 397. The ArcPad Folder

Open folder containing the ANALYZER applet. Click the ANALYZER applet and drag to the Applets folder within the Program Files.

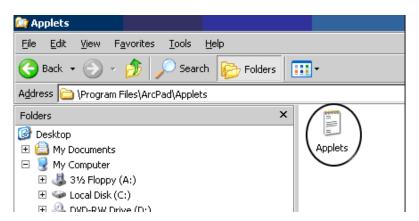


Figure 398. Copying the Applet

Drop the file in the Applets folder.

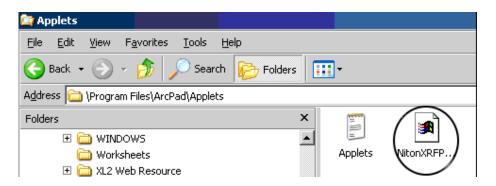


Figure 399. Dropping the Applet Into the Applets Folder

14 Interfacing Thermo Scientific Niton Analyzers and the Trimble/ArcPad GIS System Installing the Analyzer Applet

Return to Microsoft ActiveSync and click on the Sync button on the toolbar to load the Applet file onto the mobile device.

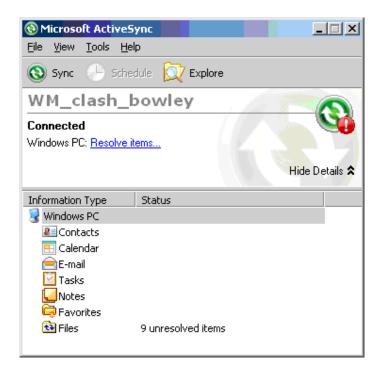


Figure 400. Syncing the Mobile Device

Restart the mobile device.

Using the Analyzer Applet

When ArcPad is started, the applet will automatically appear as an icon of an analyzer on the top toolbar. When you click that button you get a toolbar with four buttons. From left to right the buttons are 1) start collecting data, 2) view analysis, 3) save analysis to active layer, and 4) select active layer/create new layer.



Figure 401. The Applet Toolbar

To start using the software, first press the far right button to create a new layer or select a previously started layer. When you click that button you get a dialog box. The software uses ESRI shape files as its storage format, so if you add an existing layer, you will browse for existing shape files. If you create a new layer, there are a series of dialog boxes described below.

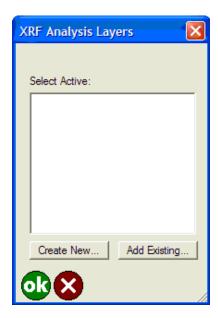


Figure 402. The Layer Dialog Box

Click the Create New button to add a new layer or click the Add Existing button to browse for a previously created layer. When you create a new layer, the dialog box in Figure 403 is shown. This allows you to choose from a set of various modes. There are checkboxes for the most commonly used elements in those modes. There is also the option of opening a textbox to enter additional elements in a space-separated list.

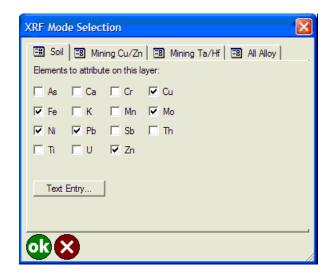


Figure 403. The Create Layer Dialog Box

Elements are selected by checking the appropriate boxes or by entering them in a list in the text entry box. The units for the analyses are assumed to be PPM for soils and percent for all other modes.

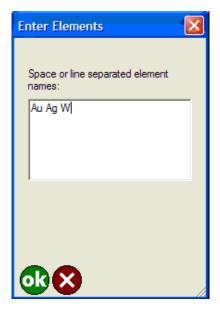


Figure 404. Entering Other Elements

After clicking ok on the mode selection box a confirmation box will appear (Figure 405). This is an important step because it is not possible to add any elements later in ArcPad. It is possible to add elements later in ArcGIS on a PC.

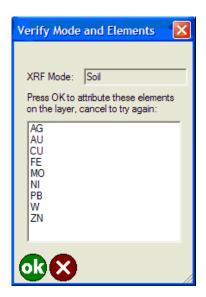


Figure 405. Mode and Element Verification

After selecting ok on the verification dialog, another window will open allowing you to choose the styles of symbols that display on the map as the data are collected (Figure 8). Choose the symbol, colors to correspond with the elements and the number of ranges that will be used for the different styles of points. Once you click ok, a dialog opens allowing you to choose the ranges and the size and color of the symbols for each range.

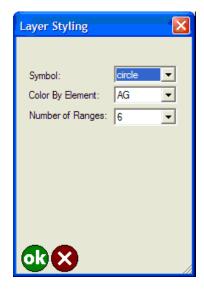


Figure 406. Selecting Symbol Size and Color

Following is the Dialog Box for choosing the ranges, size and color of symbols. The lowest value is fixed at 0 and the highest at 1,000,000. Choose the colors by clicking on the buttons labeled "...". The default is a range from blue-green-yellow-orange-red.

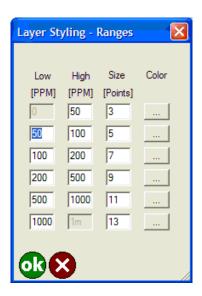


Figure 407. Symbol Dialog Box

Following is the Dialog Box for choosing the ranges, size and color of symbols. The lowest value is fixed at 0 and the highest at 1,000,000. Choose the colors by clicking on the buttons labeled "...". The default is a range from blue-green-yellow-orange-red.

Once the layer is saved, the Layer Dialog Box is shown again with the newly created file. Select it and tap the OK button. Make sure your analyzer is set to Print Complete. From the Main Menu, select the System icon, then the Printer Icon to access the proper screen. You are now ready to collect data. Tap the far left button that looks like a running man. If you get an error immediately, it means the AUX port is not setup correctly in ArcPad. Make sure the AUX port is the port that the handheld is communicating with the instrument on. Also make sure that nothing else is communicating on that port.

Now take an analysis with the Analyzer. After the analysis is complete, when connected, you will receive coordinates and analysis data as shown in the following screenshots. In Figure 408, the screen with the X, Y, and Z Coordinates is shown. When you select the View Analysis Button, the Analysis window will appear, as shown in Figure 409.



Figure 408. XYZ Coordinate Screen

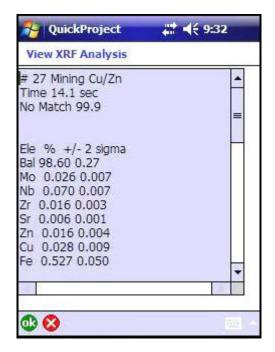


Figure 409. Analysis Window

Trimble-Niton Error Messages

"Unable to open the Niton XRF communication port. Check the ArcPad AUX configuration and try again."

This happens when the Trimble cannot communicate with the instrument. Usually means that the AUX port in ArcPad is set to a port different than the one the instrument is communicating on. It can also mean that the Bluetooth connection isn't working properly.

"XRF capture receive not progressing. Disabling data capture. Check ArcPad AUX configuration and restart ArcPad. May be configured to receive from a GPS COM device rather than a Niton XRF analyzer."

This error means that data was received by the Trimble but it is either an incomplete analysis or not a valid analysis. This differs from the next error because the maximum number of characters for an analysis is not reached in this case. This message will pop up after a delay of several seconds while the Trimble waits to see if more data is going to be received.

"XRF capture limit of 4096 characters exceeded. Disabling data capture. Check ArcPad AUX configuration and restart ArcPad. May be configured to receive from a GPS COM device rather than a Niton XRF analyzer."

This is the typical error if the AUX port in ArcPad is set to the port that the Trimble uses for the GPS signal. It means too much data was received. This box should pop up almost instantly once data capture is started if the AUX port is set to the GPS port. It shouldn't be possible for the instrument to send a valid analysis that is longer than this limit.

"Unrecognized XRF analysis received. Try transmitting the analysis again."

This means data was received from the instrument but the applet couldn't parse it. It means communication is actually working properly but either a mode that we don't support was used (i.e. alloys) or some characters got dropped during transmission. If it is a problem with characters getting dropped, hitting print on the instrument again should do the trick. If it is the wrong mode, the user will have to change the mode.

"No XRF analysis available to view. Press the capture button to receive an analysis."

This error occurs if you press the button that looks like a piece of paper but you haven't captured an analysis yet.

"No valid XRF analysis available to save."

This error happens if you click the save analysis button but have not yet captured an analysis.

"A new XRF analysis layer must contain at least one element for analysis."

This error occurs if you do not select any elements at all when setting up the layer.

"Unable to create layer file <filename>"

This happens if you don't have permission to read/write to the folder where you try to create a new layer. It could also occur if you try to replace an existing file that is already open.

"Unable to open layer file <filename>"

This indicates that you selected an existing file to save analyses to and for some reason ArcPad can't open it. Could be permissions or a corrupt file.

"<filename> does not appear to contain a layer configured for XRF analysis."

This occurs if you select an existing file to save analyses to and it isn't formatted properly (i.e. it wasn't created by the applet earlier).

"No active XRF analysis layer found. Press the XRF layers button to establish an active layer then try saving this analysis again."

This error occurs if you try to capture data before you setup a layer to store the data in.

"No element attributes were found to add to the shapefile for this analysis. The active layer is probably not configured correctly for this type of analysis."

This happens if your analysis has no elements that are in the file you selected to save analyses to.

14 Interfacing Thermo Scientific Niton Analyzers and the Trimble/ArcPad GIS System **Trimble-Niton Error Messages**

Parts

This section covers replacement parts and accessories for your analyzer, as well as the use of the reference samples included with your kit. The replacement parts and accessories section includes both a picture and the part number. The particular reference samples your kit includes depends upon the analysis modes your analyzer is configured to perform. You may not have all the reference samples referred to if your analyzer is not configured for all modes.

Parts and Accessories

Table 1. Table 1-1 - Parts and Accessories

Name	Part Number	Picture
Portable Test Stand	420-017	



The Mobile Test 430-032 Stand



The Field Mate 430-517



Holster 430-185



Battery Charger 420-011



Power
Cable/Supply 420-007
for Charger
- Power Cable



Power 420-006
Cable/Supply
for Charger
- Power Supply

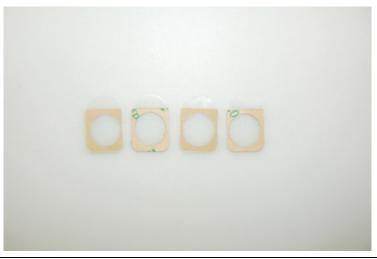


Battery 420-002



Measurement Windows

187-1555



In-Situ Test 187-1401 Guard



Mylar Window 187-813 for In-Situ Test Guard



Serial Download Cable

300-056



USB Download 179-727 Cable



Welding Mask 420-008



15 Parts

Parts and Accessories

Measurement Window Bracket 187-1255



Printer 500-933



Analyzer Case 420-013



GPS Systems GPS-01 GPS-10



Bluetooth Module

500-714



32mm Sample 187-466 Cups



15 Parts

Parts and Accessories

Mylar Film 2.5" 187-462 dia. 6m thick



Polypropylene 187-461 Film 2.5" dia. 4m thick



Filter Paper 198-014 2.4cm dia.



Polystuffing 187-468



Plastic QC Reference Sample

180-554

195-019



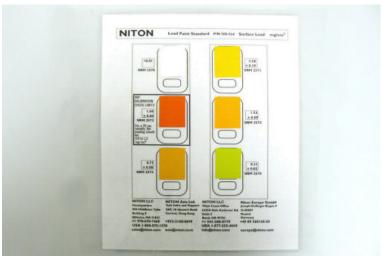
Metal QC Reference Sample



Soil/Mining QC 500-705 Reference Sample Set



Lead Paint QC 500-934 Reference Sample



Small Spot QC 180-607 Calibration Sample



Electronics Metals QC Reference Sample 180-606



Replacement Tank for He-purge



Replacement Standard Regulator for He-purge 350-001



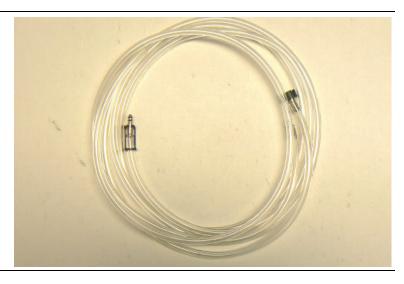
15 Parts **Parts and Accessories**

Refillable 350-008 Helium Supply Kit



He-purge Replacement Fittings

430-302



Backscatter Shield

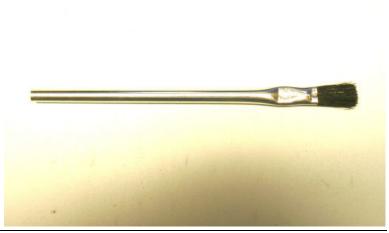
430-014



Extend-a-Pole 420-018



Acid Brush for 187-469 Soil or Mining



Whirl-Pak 187-473 Mining Sample Bag



15 Parts Parts and Accessories

Extend-a-Pole

The Extend-a-Pole accessory can be used to extend one's reach for analyzing samples, and/or to work more comfortably. The Extend-a-Pole features a remote trigger and a telescoping pole which locks securely at various lengths.

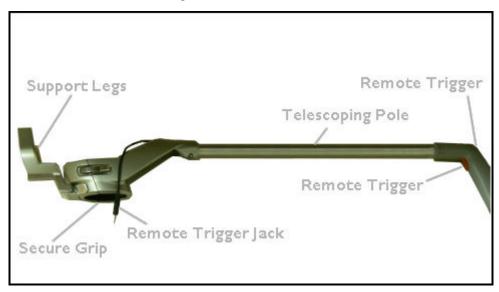


Figure 1. The Extend-a-Pole

Setting Up the Extend-a-Pole

When using the Extend-a-Pole on rough or dirty surfaces, such as with soil or mining samples, you should also use the In-Situ Test Guard. This accessory is part of the Soil Kit, and protects the nose, measurement window, and proximity button of your analyzer from wear, and facilitates use of the proximity button on soft or irregular surfaces.

16 Extend-a-Pole Setting Up the Extend-a-Pole



Figure 2. The In-Situ Test Guard



Figure 3. The In-Situ Test Guard in Place

The In-Situ Test Guard clamps firmly over the nose of your analyzer with just a push, and comes off easily. The In-Situ Test Guard does not interfere with use of the Extend-a-Pole.

Open the grip of the Extend-a-Pole. This pivots in place, allowing you to insert the handle of your analyzer.





Figure 4. The Extend-a-Pole Handle Grip - Two Views

Place the handle of your analyzer into the grip, seating the analyzer as far down into the grip as possible.

16 Extend-a-Pole Setting Up the Extend-a-Pole



Figure 5. The Extend-a-Pole Grip on the Analyzer

Close the grip, hook on the latch, and press down the lever to lock the latch in place.





Figure 6. Securing the Grip Latch

Your analyzer will be firmly held by the grip, and the trigger can be actuated through the Remote Trigger on the Extend-a-Pole.



Figure 7. The Extend-a-Pole Grip on the Analyzer

The Remote Trigger

To enable the Remote Trigger, you need to insert the Remote Trigger Jack of the Extend-a-Pole into the Remote Trigger Port of your analyzer. You can do this either before or after securing the handle in the grip.

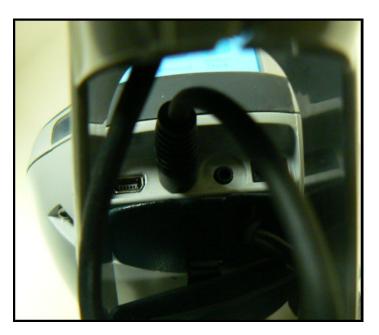


Figure 8. The Remote Trigger Jack in the Port

The screen of your analyzer can be tilted to any viewing angle you find comfortable.



Figure 9. Viewing the Touch Screen

You need to enable the Remote Trigger on the analyzer itself before using it. Go to the Start-Stop Settings and select the Remote Trigger checkbox.

16 Extend-a-Pole Setting Up the Extend-a-Pole

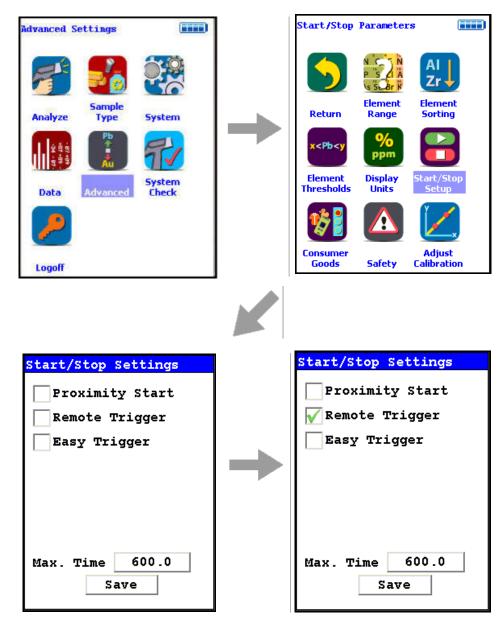


Figure 10. The Start/Stop Settings Menu Path

The Support Legs

The Support Legs can be swiveled out to make a stable, 3-point support for the Extend-a-Pole, enabling you to leave it standing without holding on.



Figure 11. Using the Support Legs

Adjusting the Pole Length

The length of the Extend-a-Pole can easily be adjusted.

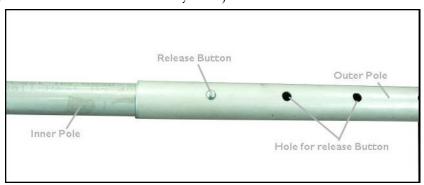


Figure 12. Adjusting the Pole

Press in the Release Button to allow the Inner Pole to slide inside the Outer Pole. The Release Button is spring loaded, and will pop up into a hole in the Outer Pole to lock the poles together in this configuration. Continue adjusting the poles until you get the length you need.

16 Extend-a-Pole Setting Up the Extend-a-Pole

Test Stands

The Portable Test Stand



Figure 1. The Portable Test Stand

Setting Up the Portable Test Stand

1. To set up the Portable Test Stand, place the folded stand on your bench top with the scissored legs down.

CAUTION When setting up and taking down the Portable Test Stand, be aware of possible pinch points.

- 2. Lift up the top part of the Portable Test Stand. As you do so, the scissored legs will rise up and come together.
- 3. Hook the notch in the back, flat part of the top over the bar between the rear legs, as in Figure 2.



Figure 2. Hooking Over the Bar

4. The Portable Test Stand is now ready for use.

Figure 3 shows a Portable Test Stand ready for use.



Figure 3. The Test Stand All Set Up

Taking down the Portable Test Stand

- 1. First, unhook all cables from the instrument and/or test stand.
- 2. To take down the Portable Test Stand, unhook the back, flat part of the top section of the Test Stand. The hood locks shut with a sliding tab for transportation.

CAUTION When setting up and taking down the Portable Test Stand, be aware of possible pinch points.

- 3. The air pistons between the scissors legs will let the top down slowly.
- 4. Support the top until the legs have fully collapsed. Figure 1-4 shows a Portable Test Stand fully collapsed.



Figure 4. The Portable Test Stand Collapsed

Connecting the Portable Test Stand to your Analyzer

Use the included Remote Trigger Cable to attach the Portable Test Stand to your Analyzer. By connecting the Test Stand, your analyzer knows whether the protective lid is open or closed, and thus whether it is safe to initiate a test, as in Figure 9. If unplugged, the instrument will not be able to initiate a reading.



Figure 5. Plugging the Remote Trigger Cable Into the Portable Test Stand

Attaching the Remote Trigger Cable

Insert one end of the Cable into the Remote Trigger Port on your Portable Test Stand, and the other end to the Remote Trigger Port on your Analyzer, as in Figures 5 and 6. Do not plug the cable into the Serial Ports.



Figure 6. Plugging the Remote Trigger Cable Into the Analyzer

Powering the Test Stand via USB

Plug the USB cable into your computer's open USB port as in Figure 7. Plug the other end into the Test Stand's USB Port. The Test Stand can draw what power it needs from your computer.



Figure 7. Plugging the USB Cable into the Portable Test Stand

Powering the Test Stand Via Separate Power Cable

You may also use the power adaptor to power up the test stand. Plug the power adapter into the power port on your Portable Test Stand, and the other end into the wall outlet.

17 Test Stands The Portable Test Stand

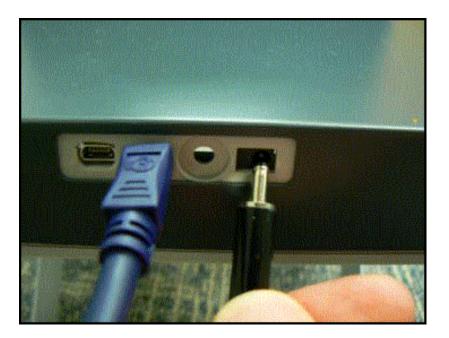


Figure 8. Plugging the Power Cable into the Portable Test Stand



Figure 9. Test Stand Open and Closed Icons

How to Use the Portable Test Stand

When the Portable Test Stand is fully set up, insert the front of your analyzer into the cone under the floor of the top part of the stand, as in Figure 1-10. Push firmly until the analyzer seats with an audible click.



Figure 10. Portable Test Stand with Analyzer Attached

Your analyzer cannot be inserted fully in any orientation but the correct orientation, with the touch screen towards the front.

To remove your analyzer, simply squeeze the tabs on either side of the cone and pull the analyzer down, gently but firmly, until it separates from the cone.

When the hood is lifted, your analyzer will display a red icon at the top of the touch screen, as in Figure 11.



Figure 11. Portable Test Stand Open Icon

17 Test Stands The Portable Test Stand

When the hood is shut, your analyzer will display a green icon at the top of the touch screen, as in Figure 12.



Figure 12. Portable Test Stand Closed Icon

Your analyzer will not take a reading unless the hood is shut and the green icon is on.

The Mobile Test Stand

How to Assemble the Mobile Test Stand

Attaching the legs to the main body

- 1. Orient the pop-up peg on the leg with the hole in the main body
- 2. Push the pop-up peg in with your thumb as shown in Figure 1-13.



Figure 13. Pushing in Peg

3. Slide the leg into the main body until the pop-up peg snaps into the matching hole as shown in Figure 14

17 Test Stands The Mobile Test Stand



Figure 14. Attaching Leg

- 4. Repeat for each leg
- 5. Adjust the height of each leg by screwing or unscrewing the feet to allow a level platform

Fitting your Niton analyzer into the Mobile Test Stand

- 6. Set the test stand so that the hinge is away from you, and you have access to the test chamber when it is open.
- 7. Place your analyzer underneath the Mobile Test Stand so that the nose points up and the touch screen is facing you, as in Figure 15



Figure 15. Inserting Analyzer into Portable Test Stand

8. Lift your analyzer firmly up into the recess until the clips snap solidly into place on both sides of the analyzer's front end.

Configuring the Analyzer for the Test Stand

The test stand contains an RFID chip, which automatically configures your analyzer to properly work with the test stand.

Proximity Start Feature

A feature you can optionally use is to set the analyzer to Proximity Start.

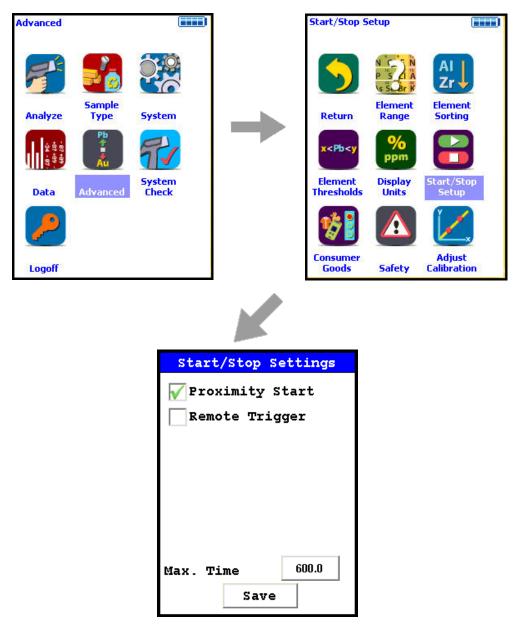


Figure 16. Menu Path to Set Proximity Start

Press the trigger to initiate the reading, and disengage the Proximity Button by opening the cover to stop the reading. With this feature enabled, you do not need to hold the trigger down while the sample is being analyzed.



Figure 17. Stopping the Analysis by Opening the Cover

You can always use standard operation, as well as remote operation via NDTr.

Measuring Small Parts and Components

To measure electronic parts and components, place the part to be measured directly on the measurement window of the analyzer, shut the hinged top firmly, and take a measurement. See Figure 16 for an example of placing electronic parts.



Figure 18. Measuring Electronic Parts

To measure small alloy parts and samples, place the part to be measured directly on the measurement window of the analyzer, shut the hinged top firmly, and take a measurement.

Measuring Bulk Samples in Sample Cups

To measure bulk materials in sample cups, place the Sample Cup Holder into the Mobile Test Stand so that it rests on the lip of the oval cutout. See Figure 17 to see how the Sample Cup Holder is oriented.



Figure 19. Inserting the Sample Cup Holder

Then place the sample cup to be analyzed into the sample cup holder, shut the hinged top firmly, and initiate a measurement. See Figure 18 for an example of placing sample cups into the Sample Cup Holder.



Figure 20. Inserting Sample Cup

Measuring Bulk Samples in Bags

To measure bulk materials in plastic bags, make sure the material to be tested is at least 1.5 cm deep over the measurement window of the analyzer. The cover of the test stand must be shut completely before taking a reading, which limits the maximum size of the bagged sample to that of the capacity of the test stand cavity.

Place the sample bag into the test stand, shut the hinged top firmly, and take the measurement.

When the hood is lifted, your analyzer will display a red icon at the top of the touch screen, as in Figure 19.



Figure 21. Mobile Test Stand Open Icon

When the hood is shut, your analyzer will display a green icon at the top of the touch screen, as in Figure 20.



Figure 22. Mobile Test Stand Closed Icon

Your analyzer cannot make an analysis unless the icon is green. To remove your analyzer from the Portable Test Stand, simply squeeze the tabs on either side of the cone and pull the analyzer down, gently but firmly, until it separates from the cone.

The Field Mate Test Stand

The Field Mate is a small test stand which can be carried about easily in the field.



Figure 23. The Field Mate

The Field Mate has a pivoting top plate which covers a shielded cavity inside which the sample is placed. There are two insets which fit inside the cavity, optimized for the two most common sample types - bagged bulk samples and sample cups.

There is a brace to support the analyzer on the underside of the base. Gently but firmly unsnap the brace by pushing down on the protruding black tab. The brace will pivot out and down 180 degrees. The brace helps compensate for the off-center weight of the analyzer when clipped into the pivoting top plate.

To place your analyzer into the Field Mate, fit your analyzer's nose firmly up into the mating cone until the clips snap solidly into place on both sides of the analyzer's front end.

17 Test Stands The Field Mate Test Stand



Figure 24. The Field Mate with Top Plate Opened

The top plate is opened by pressing the large orange latch on the base. Inserts can be placed into the cavity, and samples can be placed into the inserts. The insert for bagged bulk samples (shown in Figure 23) has a spring loaded inner plate which pushes the sample up to contact the underside of the top plate for proper analysis. The insert for sample cups (shown in Figure 1-24) holds the cup at the proper height for analysis with a soft foam support. Tilt the top plate back until it latches with an audible click for analysis. This can be done with or without your analyzer clipped into the cone.



Figure 25. The Field Mate Bagged Sample Insert

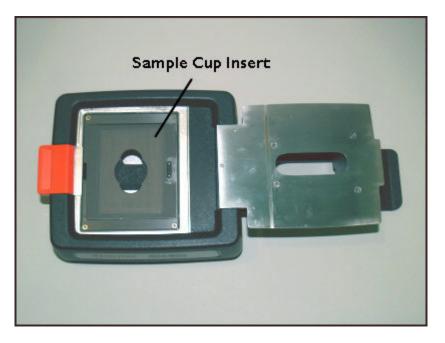


Figure 26. The Field Mate Sample Cup Insert

The top plate can also be completely removed by pressing the two small hinge tabs together towards the middle of the plate and lifting the plate from the base. This can be done with or without your analyzer mounted into the cone.

17 Test Stands The Field Mate Test Stand



Figure 27. The Top Plate Removed from the Base Unit

Removed from the base, the plate can be placed into the Test Guard, for soil screening in-situ.

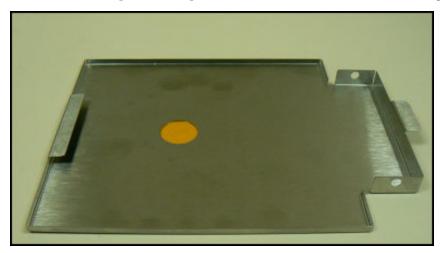


Figure 28. The Test Guard

To insert the plate into the Test Guard, slide the front tab on the plate under the tab of the Test Guard.



Figure 29. Inserting the Top Plate into the Test Guard

The interlocking tabs secure the front of the Test Guard to the plate.



Figure 30. The Front Tab

Squeezing the Hinge Tabs together, lower the back of the plate into the Test Guard. Release the tabs so the Hinge Tab Pins protrude through the holes on either side of the Test Guard. This secures the rear of the plate to the Test Guard.

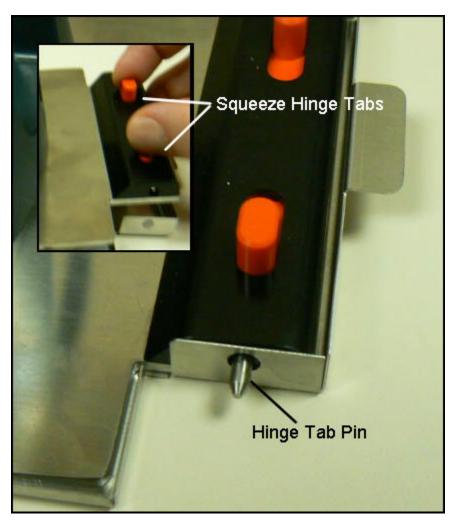


Figure 31. The Hinge Tab Pins

At this point, you can fit your analyzer into the cone. It may now be used for soil screening.



Figure 32. The Top Plate Removed from the Base Unit

When the top plate is lifted, your analyzer will display a red icon at the top of the touch screen, as in Figure 26.



Figure 33. Field Mate Open Icon

When the plate is shut, your analyzer will display a green icon at the top of the touch screen, as in Figure 27.

17 Test Stands The Field Mate Test Stand



Figure 34. Field Mate Closed Icon

Your analyzer cannot make an analysis unless the icon is green.

To remove your analyzer from the Field Mate Test Stand, simply squeeze the tabs on either side of the cone and pull the analyzer up, gently but firmly, until it separates from the cone.

Configuring the Analyzer for the Test Stand

The test stand contains an RFID chip, which automatically configures your analyzer to properly work with the test stand.

Proximity Start Feature

A feature you can optionally use is to set the analyzer to Proximity Start.

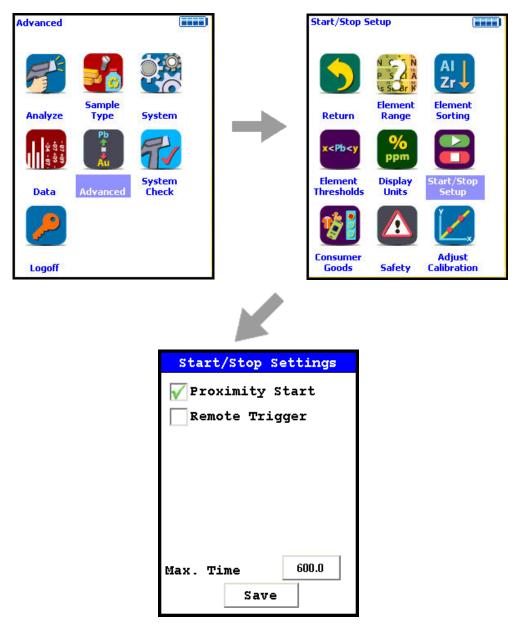


Figure 35. Menu Path to Set Proximity Start

Press the trigger to initiate the reading, and disengage the Proximity Button by pressing the Latch and opening the plate to stop the reading. With this feature enabled, you do not need to hold the trigger down while the sample is being analyzed.



Figure 36. Stopping the Analysis by Opening the Plate

You can always use standard operation, as well as remote operation via NDTr.

Reference Samples

Metal Alloy Reference (Standard) Sample Care



Figure 1. The Metals QC Reference Sample

Metal QC Reference Sample

One or more metal standard(s) are supplied with your instrument if there is an alloy calibration activated on it. This standard should be analyzed on a daily basis and the values obtained checked against the supplied certificate of analysis. For quality assurance purposes, you may also wish to keep a running chart of the values obtained and monitor it for consistency.

If one (or more) of the elements begins to change in terms of reported concentration outside of the normal expected variability, the sample may have become contaminated. This contamination can occur for multiple reasons, for example, oils and salts may transfer from hand contact onto the surface, dust or lint from the surroundings may be deposited onto the sample, or oxidation of the surface may simply occur. If this happens, the sample will need to be cleaned to provide the XRF instrumentation with a clean surface for analysis.

This may be achieved using clean, lint free paper, 2-propanol (isopropyl alcohol, IPA, (CH3)2CHOH)) and 360 grit diamond grinding paper (179-1203). Do not use rubbing alcohol as this may contain oils and other contaminants. Place the sample on a clean flat surface with the analytical side facing up. Moisten the surface with the alcohol and wipe away any surface debris or contamination. Take a square (approximately 2.5cm or 1 inch) of the diamond paper, and with the rough side against

the sample gently polish the surface to remove oxidation. Dispose of the grinding paper and clean the surface with lint free paper moistened with alcohol until the paper shows no additional residue being removed from the surface. Allow the surface to dry before analyzing.

Soil and Mining Reference (Standard) Sample Care



Figure 2. The Soil/Mining QC Reference Sample Set

Soil/Mining QC Reference Sample

One or more cupped standards are supplied with your instrument if there is a soil or mining calibration activated on it. These standards should be analyzed on a daily basis and the values obtained checked against the supplied certificate of analysis. For quality assurance purposes, you may wish to keep a running chart of the values obtained and monitor them for consistency.

If one (or more) of the elements begins to change in terms of reported concentration outside of the normal expected variability, the sample may have become damaged or contaminated. This may occur for a number of reasons, for example, oils and salts may transfer from hand contact onto the surface, dust or lint from the surroundings may be deposited onto the sample, or the thin film on the front of the cup may have pinholes or have been torn. If the cup film has torn or been damaged in any other way, it should be replaced immediately, this will prevent loss of the material and prevent contamination of the instrument and other samples. If you suspect that the film has been torn, also check the front window of the instrument and clean or replace that too if necessary.





Figure 3. The Plastic QC Reference Sample

Plastic QC Reference Sample

One or more plastic standards are supplied with your instrument if there is a plastic calibration activated on it. These standards should be analyzed on a daily basis and the values obtained checked against the supplied certificate of analysis. For quality assurance purposes, you may wish to keep a running chart of the values obtained and monitor them for consistency.

If one (or more) of the elements begins to change in terms of reported concentration outside of the normal expected variability, the sample may have become contaminated. This contamination can occur for multiple reasons, for example, oils and salts may transfer from hand contact onto the surface, and dust or lint from the surroundings may be deposited onto the sample. If this happens, the sample will need to be cleaned to provide the XRF instrumentation with a clean surface for analysis.

This may be achieved using clean, lint free paper and 2-propanol (isopropyl alcohol, IPA, (CH3)2CHOH)). Do not use rubbing alcohol as this may contain oils and other contaminants. Place the sample on a clean flat surface with the analytical side facing up. Moisten the surface with the alcohol and wipe away any surface debris or contamination. Moisten a lint free cloth with alcohol and rub harder to remove any imbedded materials. Dry the clean the surface with lint free paper moistened with alcohol until the paper shows no additional residue being removed from the surface. Allow the surface to dry before analyzing.

Electronics Metals Reference (Standard) Sample Care



Figure 4. The Electronics Metals QC Reference Sample

Electronics MetalsQC Reference Sample

One or more Electronics Metals standard(s) are supplied with your instrument if there is an Electronics Metals calibration activated on it. This standard should be analyzed on a daily basis and the values obtained checked against the supplied certificate of analysis. For quality assurance purposes, you may also wish to keep a running chart of the values obtained and monitor it for consistency.

If one (or more) of the elements begins to change in terms of reported concentration outside of the normal expected variability, the sample may have become contaminated. This contamination can occur for multiple reasons, for example, oils and salts may transfer from hand contact onto the surface, dust or lint from the surroundings may be deposited onto the sample, or oxidation of the surface may simply occur. If this happens, the sample will need to be cleaned to provide the XRF instrumentation with a clean surface for analysis.

This may be achieved using clean, lint free paper, 2-propanol (isopropyl alcohol, IPA, (CH3)2CHOH)) and 360 grit diamond grinding paper (179-1203). Do not use rubbing alcohol as this may contain oils and other contaminants. Place the sample on a clean flat surface with the analytical side facing up. Moisten the surface with the alcohol and wipe away any surface debris or contamination. Take a square (approximately 2.5cm or 1 inch) of the

diamond paper, and with the rough side against the sample gently polish the surface to remove oxidation. Dispose of the grinding paper and clean the surface with lint free paper moistened with alcohol until the paper shows no additional residue being removed from the surface. Allow the surface to dry before analyzing.

Lead Paint Reference (Standard) Sample Care

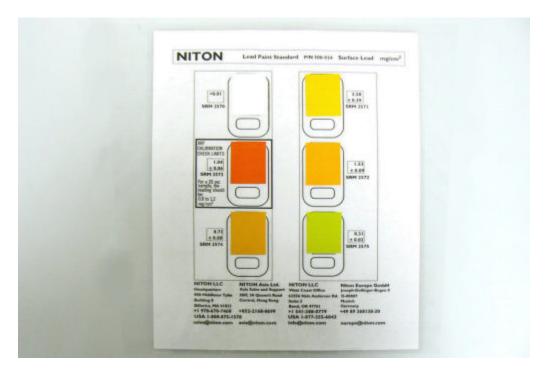


Figure 5. The Lead Paint QC Reference Sample

Lead Paint QC Metals Sample

One or more Lead Paint standard(s) are supplied with your instrument if there is a Lead Paint calibration activated on it. This standard should be analyzed on a daily basis and the values obtained checked against the supplied certificate of analysis. For quality assurance purposes, you may also wish to keep a running chart of the values obtained and monitor it for consistency.

If one (or more) of the elements begins to change in terms of reported concentration outside of the normal expected variability, the sample may have become contaminated. This contamination can occur for multiple reasons, for example, oils and salts may transfer from hand contact onto the surface, dust or lint from the surroundings may be deposited onto the sample, or oxidation of the surface may simply occur. If this happens, the sample will need to be cleaned to provide the XRF instrumentation with a clean surface for analysis.

This may be achieved using clean, lint free paper and 2-propanol (isopropyl alcohol, IPA, (CH3)2CHOH)). Do not use rubbing alcohol as this may contain oils and other contaminants. Place the sample on a clean flat surface. Moisten the surface with the alcohol and wipe away any surface debris or contamination. Clean the surface with lint free paper moistened with alcohol until the paper shows no additional residue being removed from the surface. Repeat for the other side. Allow the surfaces to dry before analyzing.

Small Spot Reference (Standard) Sample Care



Figure 6. The Small Spot QC Reference Sample

Small Spot QC Calibration Sample

One or more Small Spot standard(s) are supplied with your instrument if there is a Small Spot Camera activated on it. This standard should be analyzed on a daily basis and the values obtained checked against the supplied certificate of analysis. For quality assurance purposes, you may also wish to keep a running chart of the values obtained and monitor it for consistency.

If one (or more) of the elements begins to change in terms of reported concentration outside of the normal expected variability, the sample may have become contaminated. This contamination can occur for multiple reasons, for example, oils and salts may transfer from hand contact onto the surface, dust or lint from the surroundings may be deposited onto the sample, or oxidation of the surface may simply occur. If this happens, the sample will need to be cleaned to provide the XRF instrumentation with a clean surface for analysis.

This may be achieved using clean, lint free paper, 2-propanol (isopropyl alcohol, IPA, (CH3)2CHOH)). Do not use rubbing alcohol as this may contain oils and other contaminants. Place the sample on a clean flat surface with the analytical side facing up. Moisten the surface with the alcohol and wipe away any surface debris or contamination. Clean the surface with lint free paper moistened with alcohol until the paper shows no additional residue being removed from the surface. Allow the surface to dry before analyzing.

Small Spot Reference (Standard) Sample Care

Learning More, Service, and Support

This section of the Resource guide is about getting the most out of your analyzer. We cover troubleshooting your analyzer by using the Specs screen. We also cover advanced topics like setting thresholds, using the Tools menu, correcting for light elements in the sample composition, setting up pass/fail analysis, changing safety and start/stop parameters, and many other special situations you may need. We have also included a number of documents for reference, so you can learn more about XRF analysis if you are so inclined.

Tips and Troubleshooting

The Specs Screen

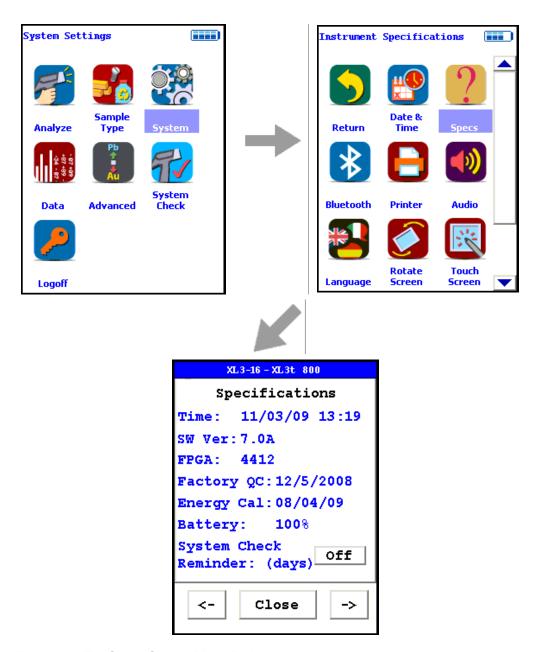


Figure 1. The Specs Screen Menu Path

Select the Specs icon from the System Menu to display the analyzer's specifications. These specifications include your analyzer's serial number, software and firmware versions, and other data. Press the Close Screen Button to return to the previous menu. Press the ">-" Screen Button to go to the Diagnostic Menu, and press the "<-" Screen Button to return to the Specifications Screen.

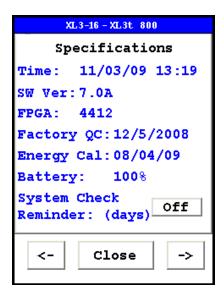


Figure 2. The Specifications Screen

On the Specs Screen, standard information on the state of your analyzer is shown for your reference. This information should be reported to Service if there is a problem.

Specs Information

The following is the information supplied on the Specs Screen:

Instrument Specific Serial Number

This is located in the left part of the blue band at the top of the screen.

Model Number

This is located in the right part of the blue band at the top of the screen.

Date And Time

This is the current Date and Time. This is particularly important for date stamping.

SW Version

This is the currently loaded software version, which should be reported to Service if there is any problem.

FPGA

This is the currently loaded FPGA software version, which should be reported to Service if there is any problem. FPGA versions are always a four digit number. Any other number of digits may be a sign of a problem in the FPGA code.

Factory QC

This is the date that the machine was QCed at the factory.

Energy Cal

This line notes the last time a System Check was performed.

Battery

This line gives the proportional charge remaining to the battery.

System Check Reminder

Select the OFF Screen Button after "System Check Reminder" to set a reminder to calibrate your analyzer. Selecting the button will open the Cal. Reminder Editor. Select the number of days you want between reminders with the numeric keys. Of the other screen buttons, C = Clear All, E = Enter, and OFF shuts off the Reminder Function. Selecting the E button will enter the current value as the reminder interval and return to the Specs Screen.

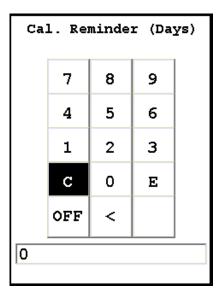


Figure 3. Cal Reminder Editor Screen

Diagnostics

Select the "->" Screen Button to load the Diagnostics Screen. The Diagnostics Screen shows Detector Temperature, Bias, Cooler Voltage, SubBias, Energy Scale, and Temperature in C and F scales.

The Diagnostics Screen can be of great utility in assuring proper operation of your analyzer.

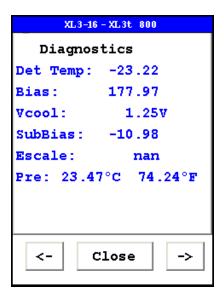


Figure 4. The Diagnostics Screen

The proper ranges of operational values on the Diagnostics Screen follow.

Det Temp:

Detector Temperature should be within this range:

- 25 + or - 5 degrees

Bias:

Bias should be within this range:

175 + or - 10

VCool:

VCool will vary with the ambient temperature.

18 Learning More, Service, and Support Tips and Troubleshooting

SubBias:

SubBias should be within this range:

-11 + or - 3

Escale:

Escale should be within this range:

6.6 through 9.0

Preamp:

Preamp value should only be noted, and reported to Service if there is a problem.

Error Messages

The analyzer will auto-detect many perceived hardware problems and may display the following error messages:

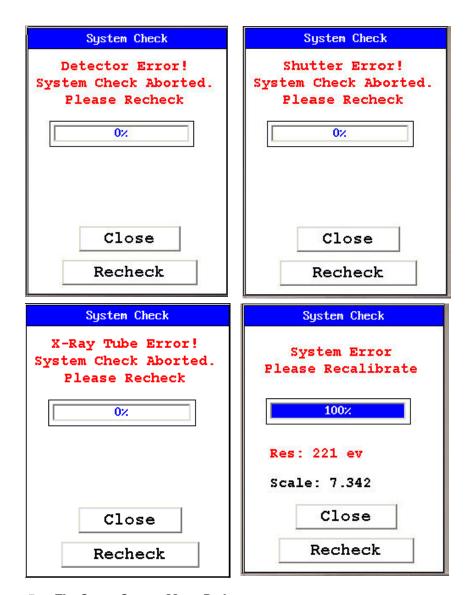


Figure 5. The Specs Screen Menu Path

Please re-run the **System Check** and if the problem persists, contact <u>customer service</u>.

Registration and Licensing FAQ

Niton XL3t and XL3t GOLDD

As a user of a Niton XL3t analyzer, you may be required to register or obtain a license with your local radiation control authority. In the US, if you intend to do work with your analyzer in states other than your own, you may be required to register there as well. Below is a list of commonly asked questions that come up when filling out registration forms.

FAQ.

Q: What is the max mA, max kVp, and max power?

A: Maximum mA is 0.1 mA

Maximum kVp is 50 kVp

Maximum power: 2 watts

Q: What is the accelerator voltage or MeV?

A: This should be filled out as not applicable N/A as it does not apply to Niton XL3t analyzers.

Q: What is the radioisotope?

A: There are no radioactive isotopes in Niton XL3t analyzers.

Q: What category is the Niton XL3t?

A: States differ greatly in their categories; the following is a list of common categories:

- X-Ray Fluorescence
- Analytical or Analytical XRF
- Open Beam or Open Beam Analytical
- Portable Gauge or Portable XRF
- Industrial Analytical or Non-Destructive Testing

When selecting the category make sure that you don't select medical or radiographic.

Q: How many tubes are in the Niton XL3t?

A: One.

Q: What is the analyzer serial number?

A: The serial number is a 5 digit number located on the yellow sticker on the underside of your analyzer.

Q: What is the tube serial number?

A: The serial number of the tube can be found on the Calibration Certificate.

Q: What is the type of X-Ray Processing?

A: None. Niton XL3t analyzers do not use film.

Q: How often do I need to perform leak tests on the Niton XL3t?

A: Never. Leak tests are only required for analyzers with radioactive isotopes. Niton XL3t analyzers do not have radioactive isotopes.

Niton XL3p

Routine Maintenance Guidelines

Maintenance, Cleaning and Repairs

To ensure the reliability, durability, and performance of your Niton Analyzer, keep it clean—especially the transparent measurement window covering the analysis window. Clean the measurement window gently with a cotton swab. Clean the body of the analyzer with a soft cloth. Never use detergents, or solvents on your analyzer, or immerse your analyzer in water. If the measurement window becomes frayed, ripped, or contaminated with metal particulates, replace it with a new window. measurement windows (Standard Window Niton P/N 187-1555 or Helium Purge Window Niton P/N 187-1454) may be ordered from Thermo Fisher Scientific's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460 or from your local Authorized Niton Analyzers Service Center.

From time to time, your touch screen will need cleaning. Niton recommends that you use a lens cleaning solution with a soft cloth. Do not use water to clean your Niton Analyzer.

WARNING All Service, except exterior cleaning and measurement window replacement, must be performed by Thermo Scientific or an Authorized Niton Analyzers Service Center. Do not attempt to make repairs yourself. Opening the case of your Niton will void the analyzer Warranty in its entirety.

CAUTION Always obtain a Return Authorization (RA) number from Thermo Fisher Scientific's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460 before returning your analyzer to the Niton Service Department or local Authorized Niton Analyzers Service Center.

Replacing the Measurement Window

1. Remove the two Phillips head screws.



Figure 6. View of Face Plate and measurement window

2. Remove the face plate and place it face down.



Figure 7. Face Plate Removed showing measurement window on Reverse

- 3. Remove the old measurement window.
- 4. Clean the back surface of the face plate and install the new Window.
- 5. Turn the face plate over and replace it on the analyzer's front end, fitting the plate carefully over the Proximity Button



Figure 8. Fitting Face Plate over Proximity Button

6. Reinstall the two screws, being careful not to over-tighten them.



Figure 9. Replacing the Screws.

Storing and Transporting Your Niton XL3 Analyzer

All Niton Analyzers are transported in waterproof, drop-resistant, fully padded carrying cases with padlocks. In most countries, Niton XRF analyzers may be transported by car or plane or shipped as an *ordinary* package. For most courier services, no special labels are required on the outside of the Niton analyzer case or on additional packaging.



Figure 10. The Niton Carrying Case

All padlocks are shipped with a default combination of "0-0-0". If you change this combination, please inform Thermo of the new combination if you return the unit for service.

To change the combination:

- 1. Dial the default combination to open the lock, and pull out the shackle.
- 2. Rotate the shackle 180 degrees and push it down as far as it can go.
- 3. While holding the shackle down, rotate it 90 degrees back in either direction and release shackle.
- 4. Change the dial settings to the desired combination, record the combination, and without disturbing the dials, rotate the shackle back 90 degrees to the position it had in step 2.
- 5. Pull shackle out and rotate it 180 degrees and secure it. Your lock now has its own secret combination.

CAUTION <u>Always</u> transport the unit in its padded carrying case, and store the Niton Analyzer in its case whenever it is not being used.

18 Learning More, Service, and Support Storing and Transporting Your Niton XL3 Analyzer

CAUTION In most cases, no notification is required if transporting within state boundaries. This may not be the case when entering federal properties.

CAUTION Within the United States, always keep a copy of the US DOT compliance statement in your Niton analyzer case at all times. A copy is included with your analyzer.

CAUTION Always follow all pertinent local and national regulations and guidelines, wherever your analyzer is transported or used.

CAUTION <u>Always</u> obtain a Return Authorization (RA) number from Thermo Fisher Scientific's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460 <u>before</u> returning your analyzer to the Service Department or to your local Authorized Niton Analyzers Service Center.

CAUTION If you return your Niton analyzer without the carrying case, you will void your warranty in its entirety. You will be billed for a replacement case plus any repairs resulting from improper shipping.

CAUTION CAUTION Always remove the battery pack when transporting or storing your analyzer.

Advanced Settings

The Set Element Threshold Menu



Figure 1. The Set Element Threshold Menu Path

Select the Set Element Threshold icon to configure pass and fail criteria for elemental analysis. Selecting the Set Element Threshold icon opens the Set Threshold Screen.

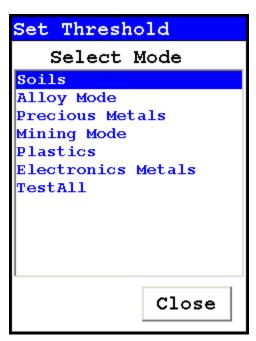


Figure 2. Set Element Threshold Screen

Select the mode you wish to work with from the scrollable list. This will open up the Pass/Fail Settings Screen for that mode.

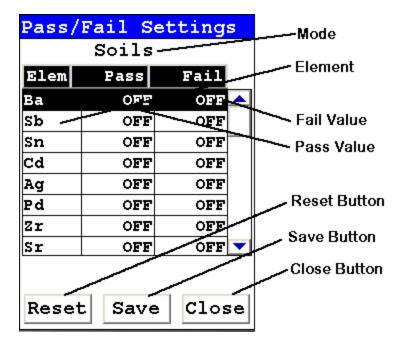


Figure 3. **Pass/Fail Settings Screen**

Selecting the Pass Value will open up the Pass Editor for the selected element.

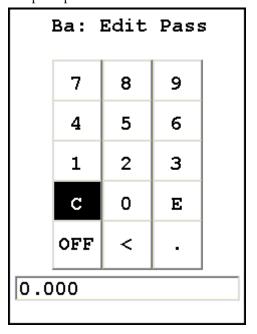


Figure 4. The Pass Editor

The Pass Editor is very similar to the Logon Screen. The C button clears the field, and the "<" button clears the last numeral. Select the numerals you want, then press the E button to enter the number. The OFF button resets the value to "OFF"

Selecting the Fail Value will open up the Fail Editor for the selected element.

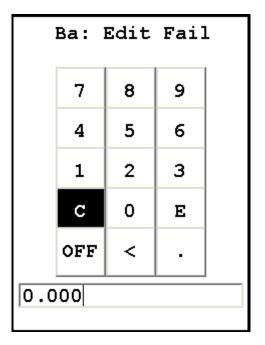


Figure 5. The Fail Editor

The Fail Editor works the same as the Pass Editor.

When you press the E button in either editor, you are returned to the Pass/Fail Settings Screen, with your new values in place.

Selecting the OFF button not only sets the value to "OFF" but also saves the new value.

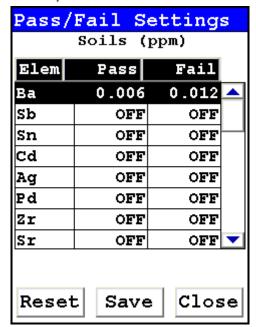


Figure 6. The Pass/Fail Setting Screen With New Parameters

Select the Reset Button to clear the current value and continue working.

Select the Save Button to save your current status as the new default. After saving, you will go back to the Element Display Menu.

Select the Close Button to exit without saving. When you select the Close Button after changing the display state of any element, a screen will open asking you if you want to save the changes you made. Selecting the Yes button will save these changes as the new default. Selecting the No button will return you to the Element Display Menu without saving the changes.



Figure 7. Save Changes Screen

Plastic Thresholds

Under Plastics Mode, selecting the triangle next to the Analysis Options Field will open a pop up menu allowing you to choose between the three Analysis Option Modes. Select the mode you wish to edit.

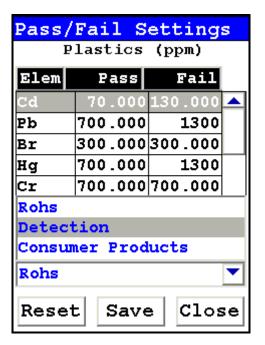


Figure 8. The Analysis Options Popup Menu

Changing the settings for one mode will not affect the settings for other modes, and the configurations can be saved independently.

RoHS Option

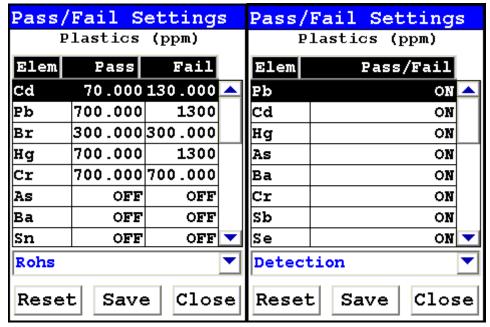
When the RoHS Option is selected, clicking on the Pass and Fail values works as it does in any other Mode.

Detection Option

When the Detection Option is selected, Selecting the Pass/Fail field for that element acts as an On/Off Toggle, which will switch Pass/Fail mode between On and Off for the selected element. Selecting it again will reverse the toggle.

Consumer Products Option

When the Consumer Products Option is selected, clicking on the Pass and Fail values works as it does in any other Mode. In addition, the total of Cl+Br is also calculated and used for Pass/Fail Testing.



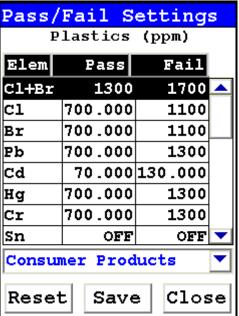


Figure 9. Pass/Fail Settings Under All Three Plastics Options

Adjusting the Element Range

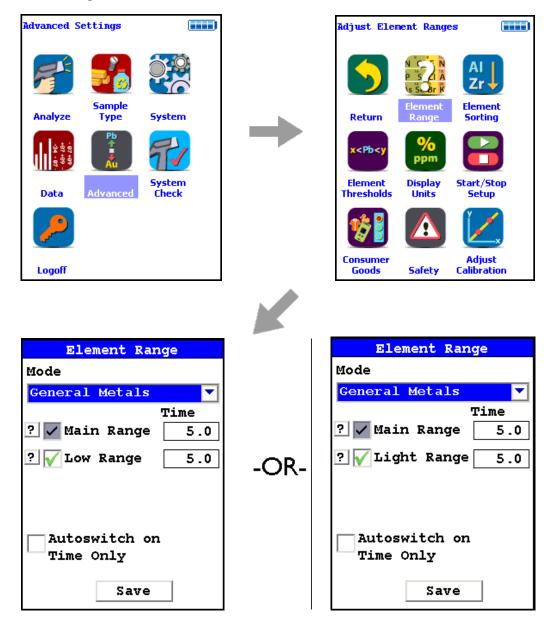


Figure 10. The Range Menu Path (Main)

Multi-Range tests are used to either preferentially excite specific elements for increased sensitivity, or to cover a wider element range than one Range alone can provide. Most modes, when enabled, will use two Ranges in sequence to produce a combined analysis result. In typical alloy analysis applications, Main Range is used for the analysis of most elements, Low Range is utilized for the subsequent high sensitivity analysis of V, Ti, and Cr, and Light Range is available only with He-purged and 900 series GOLDD technology analyzers, and is

typically used in light element analysis. Multi-Range switching can be set to activate off time alone, or, when time switching is disabled, off settings in the General Metals grade library. In most modes, Low and Light Range add the capability to analyze light elements which cannot be efficiently excited by Mid Range.



Figure 11. Selecting the Mode

Select the mode you wish to configure. You can set different configurations for different modes.

The Element Range Screen enables you to directly enable or disable any Range, or control the time that a Range alters the irradiation of the sample before auto-switching to another Range.

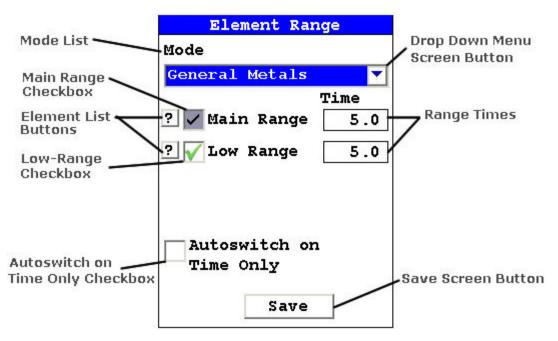


Figure 12. The Element Checkboxes

Select the checkbox next to the Range you want to use to determine exactly which of the Ranges contained in your Analyzer is used for sample testing. Selecting an empty checkbox will enable that range and place a check into the box as an indicator. Selecting a checked box will disable the Range and clear the box.

In typical alloy analysis applications, Main Range is used for the analysis of most elements. You cannot deselect the Main Range in alloy analysis

Low Range is utilized for the subsequent high sensitivity analysis of V, Ti, and Cr.

```
Element List

Mode: Alloy Mode

Filter: Main Filter
Al, Sb, Sn, Pd, Ag,
Mo, Nb, Zr, Bi,
Pb, Se, W, Zn,
Cu, Re, Ta, Hf,
Ni, Co, Fe, Mn,
Cr, V, Ti

Element List

Mode: Alloy Mode

Filter: Low Filter
Cr, V, Ti

Cr, V, Ti
```



Figure 13. The Range Element Lists

Select the Element List Button to display the Element List for that Range. This list shows the elements that the Range is best designed to detect.

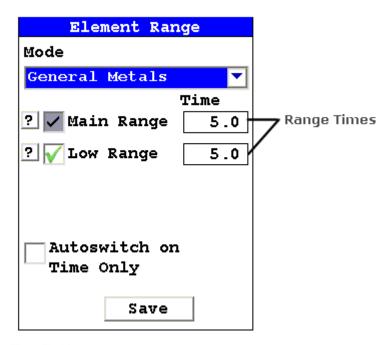


Figure 14. The Range Time Fields

Select the Range Time field for the intended range to change the switch time for that range. The Range Time Editor will appear. This enables you to set the number of seconds each enabled range is allotted before auto-switching will occur when needed during sample testing. Your analyzer will auto-switch from one range to another when the testing time for that range is greater than or equal to the time you have chosen, and the identified alloy is flagged as needing the switch in the Niton Alloy Library.

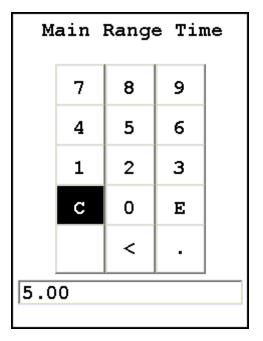


Figure 15. The Range Time Editor

Select the C button to clear the current time, then from the virtual numeric key pad, select each digit you want to input, then select the E button to enter.

The LCD Brightness Screen

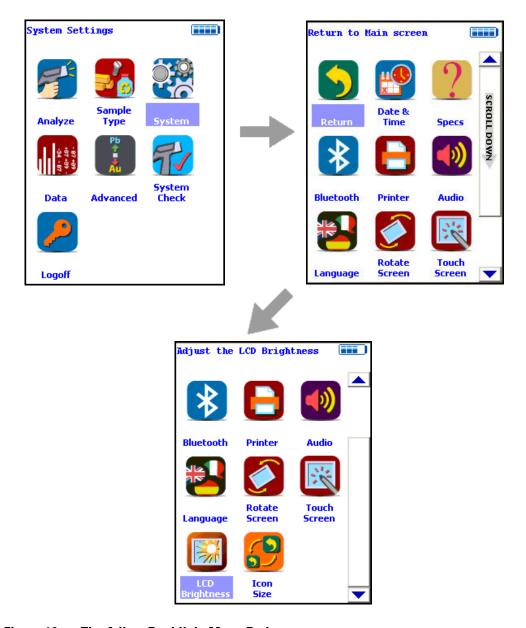


Figure 16. The Adjust Backlight Menu Path

Select the Adjust Backlight icon to adjust the brightness of the analyzer screen.

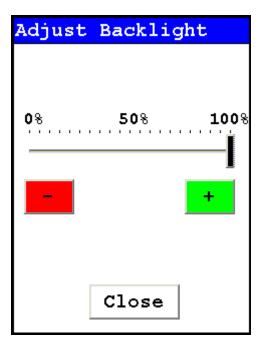


Figure 17. . The Adjust Backlight Screen

Selecting the red [-] button will cause the slider to move some to the left and the screen to dim a bit. Selecting the green [+] button will cause the slider to move a bit to the right and the screen to brighten somewhat. Find the setting most harmonious with the ambient lighting. Selecting the Close button saves the backlight setting in the current state, and returns you to the Utility Menu.

Rotate Screen

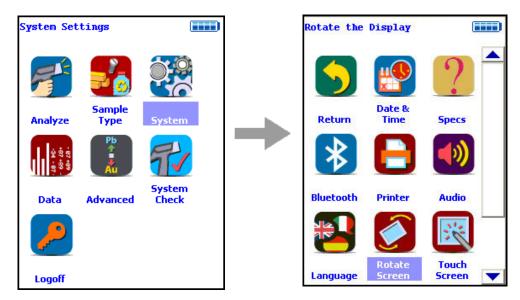


Figure 18. Rotate Screen

Select the Rotate Screen icon to toggle the orientation of the screen between right side up and upside down.

Setting Display Languages

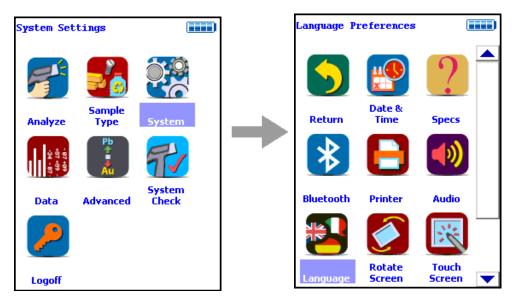


Figure 19. The Language Settings Menu Path

Selecting the Language Settings icon will load the Language Screen, allowing you to change the language from the default English to French, Spanish, Portuguese, or German.



Figure 20. The Language Screen

Select the down-pointing triangle button, and then select the language you want from the drop down menu. The menu system will now show on screen in the language you have selected.

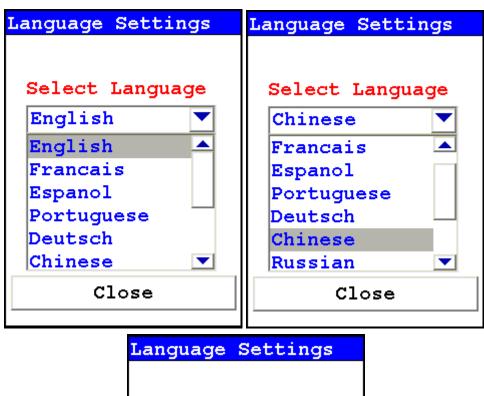




Figure 21. Select Language



Figure 22. The Radiation Warning Screen in Chinese

Setting Up the Printer

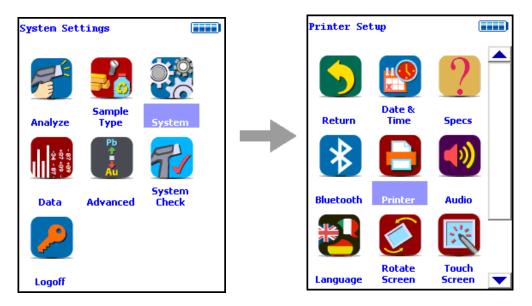


Figure 23. The Printer Setup Menu Path

The Printer Setup Screen allows you to adjust which sections of your reading data are sent to your optional portable printer. By default, your analyzer prints the detected list, reading number, reading length, reading mode and any applicable measurement data such as Alloy match grade names. You can select any combination of options on the Printer Setup Screen to change what is printed.

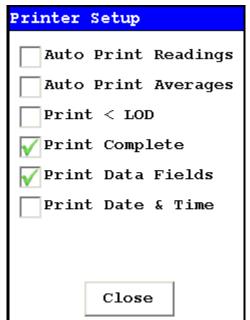


Figure 24. The Printer Setup Screen

Advanced Settings

Auto Print Readings

Selecting this option will enable automatic printing of readings when an analysis is performed.

Auto Print Averages

Selecting this option will enable automatic printing of averages when an averaging analysis is completed.

Print < LOD

Selecting this option will enable printing of readings which are lower than the Limit of Detection.

Print Complete

Selecting this option will enable printing of all the data fields in the reading.

Print Data Field

Selecting this option will enable printing of all entered data fields.

Print Date & Time

Selecting this option will enable printing of the Date and Time.

Setting up Audio Cues

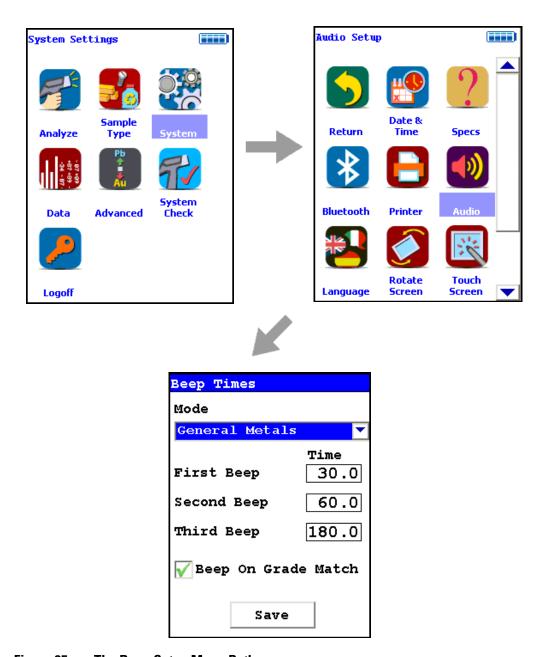


Figure 25. The Beep Setup Menu Path

Selecting the Audio icon opens the Beep Times Setup Screen, enabling changes to the beep settings for various modes. The beeps sound as follows:

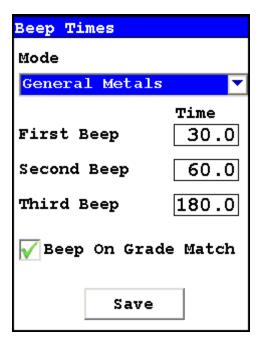


Figure 26. The Beep Setup Screen

Mode

This option allows you to change the beep settings for different modes independently. Select the Down Arrow Screen Button to access the list of modes.



Figure 27. Drop-down Mode List

First Beep

This option allows you to change the delay before the First Beep.

Second Beep

This option allows you to change the delay before the Second Beep.

Third Beep

This option allows you to change the delay before the Third Beep.

Beep on Grade Match

Selecting this option will enable a special beep when the reading chemistry matches an alloy grade.

Tools Menu Options

The following options can be performed from the Tools Menu. Options which are only on the main Tools Menu are listed as (Main). Those which can be found only on the alternate Tools Menu are listed as (Alt).

Avg Forward

Enables you to average different readings together from this analysis forward. Select the Avg Forward button to initiate future sample averaging. Avg Forward will set up an automatic personal averaging protocol to be followed until your analyzer is shut down, or this feature is disabled. To begin, select the number of readings you want to average from the virtual numeric keypad. Your analyzer will calculate an average reading after that number of tests, and continue this pattern until stopped. For example, if you select 3 on the virtual keypad, the analyzer will automatically calculate, average, and store a reading for every three tests you take, storing the individual readings along the way.

The range number is selected using a virtual numeric keypad on your analyzer similar to the keypad used for login. Select the digits in the range number from the keypad, then select the E button to enter the number. The C button will clear all, and the "<" button will clear the last digit entered. The average will automatically be displayed.

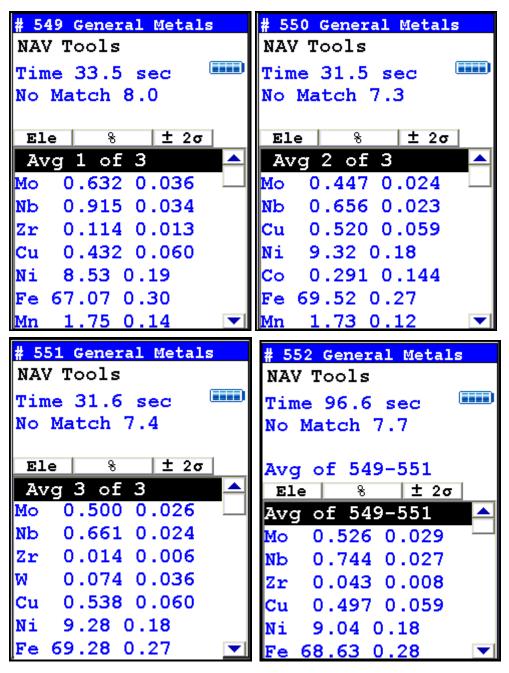


Figure 28. Example Averaging Screens

Avg Back (Alt)

Note The alternate Tools Menu is only available when viewing readings, and the menu is only accessible through the touch screen interface or NDTr.

Enables you to average different readings together from this analysis backward. Select the Avg Back option to initiate backwards sample averaging. Avg Back will take the number of readings you select and average their analytical results. The range is counted from the last reading backward by the number of readings selected. If your last reading was #15, selecting 3 would average readings #13, 14, and 15. The average is calculated, displayed, and stored into memory as the next sequential reading number, in this case, #16.

The range number is selected using a virtual numeric keypad on your analyzer similar to the keypad used for login. Select the digits in the range number from the keypad, then select the E button to enter the number. The C button will clear all, and the "<" button will clear the last digit entered. The average will automatically be displayed.

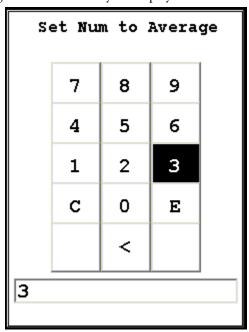


Figure 29. The Virtual Numeric Keypad

Note You cannot average readings taken in different modes. Doing this will generate an error.

Spectrum:On/Spectrum:Off

The Tools Menu contains a toggle option to display live spectra as sample analysis occurs.

Activating and Deactivating the Live Spectrum

From the Tools Menu, select the Spectra:On button to turn the Spectrum feed on. Once the spectrum is displayed, selecting Spectra:Off from the Tools Menu will stop the live spectrum display.

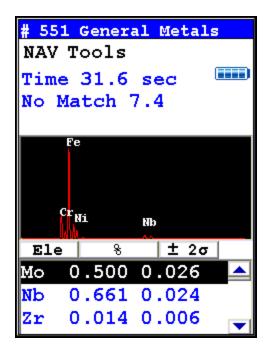


Figure 30. Example Analysis Screen Showing Live Spectrum

Enable LEC/Disable LEC

Select the Enable LEC button from the Tools Menu to enable Light Element Correction in sample analysis. Select the Disable LEC button - the toggle name - to disable Light Element Correction. Simple analysis cannot distinguish light elements such as Carbon from the background scatter, reporting only the identified elements and treating anything else as "balance". The Alloy Library, on the other hand, contains complete chemistry of the various alloys. When Light Element Correction is enabled, once an alloy is identified positively, your analyzer will report proper percentages of these light elements by reading them from the library and adjusting them to be proportional to the total "balance" of unidentified elements.

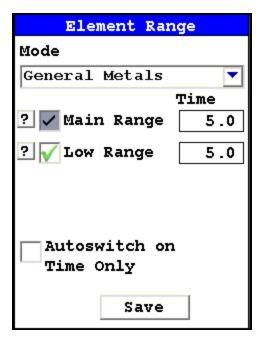


Figure 31. Autoswitch on Time Only Unchecked

Note When using Light Element Correction (LEC) do not check Autoswitch On Time Only.

Print (Alt)

Select the Print option from the Tools Menu to print the current analysis screen to any attached portable printer. If you do not have a portable printer attached to your analyzer, nothing will happen.

Set Pass/Fail

You can set up your analyzer to sort on a pass/fail basis. Pass/Fail uses the chemistry of a user-generated list of alloys in the library as a basis for comparison. If the sample analysis is entirely within the specifications for one of these alloys, a PASS result is given, otherwise a FAIL result is returned. To turn on Pass/Fail, select the Tools Menu and select Set Pass/Fail from the menu. The Pass/Fail Setup Screen will come up.

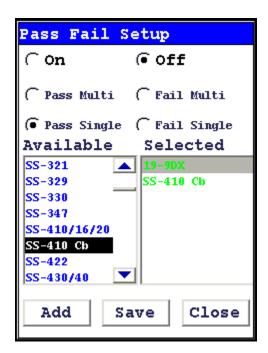


Figure 32. Set Pass/Fail Screen

0n

Select the On radio button to turn Pass/Fail on.

Off

Select the Off radio button to turn Pass/Fail off.

Add/Remove (Toggle)

Select alloys from the Available list and then the Add Button to move the alloy to the Selected List. Select alloys from the Selected list and then the Remove Button to remove the alloys from the Selected List.

Save

Select the Save Button to save these criteria.

Close

Select the Close Button to exit without saving.

Pass Single

Select the Pass Single button to initiate Pass Single Mode. Use Pass Single Mode when you have only one possible desirable match. If the alloy being analyzed matches the alloy in the selected list, the alloy will Pass the analysis.

Fail Single

Select the Fail Single button to initiate Fail Single Mode. Use Fail Single Mode when you have only one possible undesirable match. If the alloy being analyzed matched the alloy in the selected list, the alloy will Fail the analysis.

Setting the Reference Alloy for Pass Single or Fail Single

Before you use Pass Single or Fail Single mode, you need to set the Reference Alloy. Select the Data Entry Button from the slide down menu on the Ready to Test Screen, bringing up the Data Entry Screen.

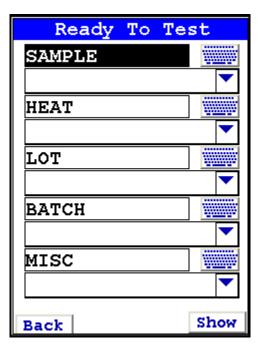


Figure 33. . The Data Entry Screen

Select the down triangle button next to the Sample Field. This will bring up the Sample Pick screen.

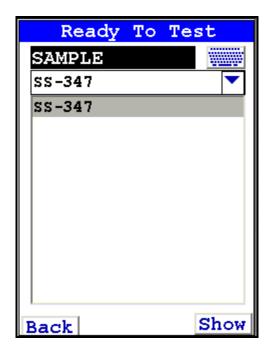


Figure 34. The Sample Pick Screen

Select the alloy you want as your Reference Alloy. In the example, we use 347 Stainless Steel.

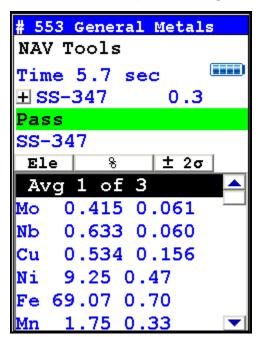


Figure 35. Example Pass Single Analysis

Pass Multi

Select the Pass Multi button to initiate Pass Multi Mode. Use Pass Multi mode when you have more than one possible desirable matches. If the alloy being analyzed matches any one of the alloys in the Selected list, the alloy will Pass the analysis.

Fail Multi

Select the Fail Multi button to initiate Fail Multi Mode. Use Fail Multi mode when you have more than one possible undesirable matches. If the alloy being analyzed matches any one of the alloys in the Selected list, the alloy will Fail the analysis.

Using Either Pass or Fail Multi

There is no need to set a Reference Alloy in Pass or Fail Multi mode. You may use it as soon as you select your alloys to test against.

How Pass/Fail Mode Works

Pass/Fail Mode compares the chemistry to that of the alloy(s) selected, using the cutoff you selected. When the sample analysis reaches a match with the chemistry of any one of the alloys on the Selected list, a PASS or FAIL notice is generated as appropriate.

Switch Library (Main)

Selecting the Switch Library button from the Tools Menu will swap the currently loaded library with the other library on the analyzer. Selecting Switch Library again will switch them back.

Enable/Disable Al

Normally, the collective amount of unquantifiable light elements in alloy analysis - the "balance" - is assumed to be aluminum and labeled as such in the analysis. Selecting the Disable Al button from the Tools Menu will delete this "aluminum" from the analysis results, showing only the quantified elements. Selecting the Enable Al button, the default state, will label this "balance" as "aluminum".

Thickness Correction

Plastics, and polymers in general, unlike metals or soil, are very weak absorbers of X rays. This is because polymers are composed mainly of very light elements such as carbon and hydrogen. While just half a millimeter of steel will completely stop 23.1 keV energy X rays of cadmium, for example, it takes at least 10mm of plasticized PVC and as much as 100mm of polyethylene (PE) to do so. Fortunately, polymers that may contain cadmium (Cd), lead (Pb) and other restricted elements would also contain considerable quantity of elements such as antimony (Sb), bromine (Br), titanium (Ti), etc. Their presence results in much stronger

absorption of X rays which means that, instead of 100mm, it takes only about 15mm of compounded PE to achieve saturation thickness for these X rays. If the thickness of analyzed polymer sample is less than 5mm for PVC or less than about 9mm for a "typical" PE, the measured intensity of X rays will be a function of both analyte concentration and sample thickness. This is why measurements performed on thin samples (less than saturation thickness) need to be corrected for thickness.

How to apply Thickness Correction.

In order for the instrument to apply thickness correction to the measured concentration results, the user must be using the Thickness Correction screen and enter the thickness of the analyzed plastic object expressed in [mm] before the measurement is initiated. The thickness may be entered with precision to the second decimal place, although in practice only one decimal place is sufficient for effective correction.

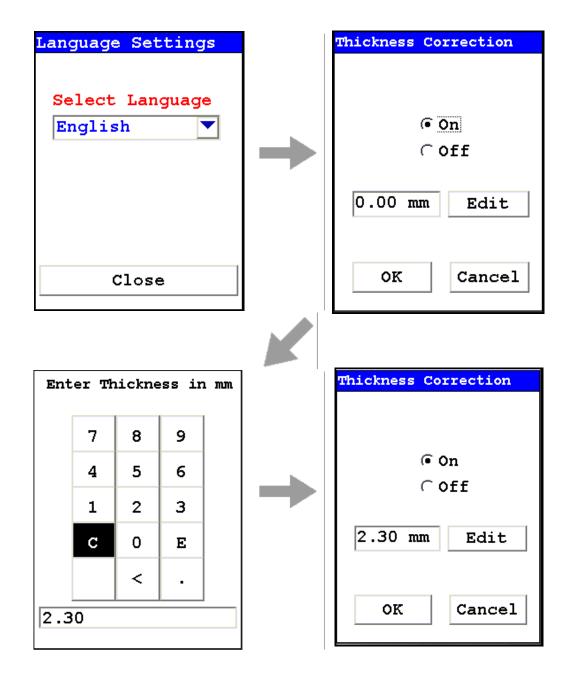


Figure 36. How to Enable and Adjust Thickness Correction for Plastics Analysis

When to use Thickness Correction

Thickness Correction should only be used during the analysis of plastic (polymer) objects. It has been experimentally verified that the correction algorithm will yield satisfactory results, for a 60 second minimum testing time, for samples as thin as 0.3mm. Nevertheless, the recommended range of use of the correction is from 1mm upwards. It is imperative that this correction is not used for thin films such as single foils and plastic membranes; analysis of thin films is performed using the Thin Sample Mode. (Contact Contact Thermo Scientific or your local Niton Analyzers representative for information on this testing mode.)

Whenever possible, one should analyze as thick a sample as available. For example, if the analyzed object is a piece of heatshrink tubing with wall thickness of 0.3mm, the best way to analyze it is to obtain several pieces of the tubing (four for example) and stack them like a flat sandwich, with the thickness correction set to 1.2mm. Doing so makes for faster and more precise analyses. While it would be possible to analyze just a single layer of the tubing with correction at 0.3 mm, by stacking several layers we reduce the relative error of measurement (by a factor approximately equal to the square root of the number of layers). Conversely, when analyzing thinner samples, we need to extend the measurement time fourfold (by the number of layers) in order to maintain the same relative error of measurement. We can see how quickly measurement time would escalate to impractical levels for thinner samples.

Examples: The most frequent instances in which thickness correction would be called for are analyses of plastic sheeting or plastic insulation on wires and/or cables and heat shrink tubing. Flat plastic sheeting or plastic enclosures pose no problems. We can either analyze an object "as is", or stack several layers of it before analysis. Plastic insulation such as that on wiring or cables requires a little more sophisticated approach. First, the wire must be removed so that only insulation is analyzed. Then, the insulation should be flattened for analysis, and a thickness correction should be applied that is equal to double the wall thickness. Alternatively, if the insulation is stiff, it should be cut lengthwise into strands which are placed on the indtrument for analysis. The applied thickness correction should be equal to the wall thickness of the sleeve. Both operations are shown in Figure 37 and Figure 38.



Figure 37. Wire Insulation Cut Into Strands

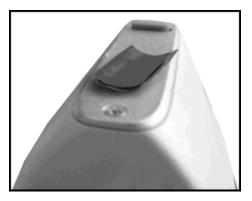


Figure 38. **PVC Wire Insulation With Conductor Removed**

A piece of large diameter heat shrink tubing presents an interesting case. It is tempting to analyze this object as is - see Figure 39. However, one needs to know that while lead or bromine or chromium X-rays from the upper wall of tubing will not contribute to the signal measured, X rays of such elements as cadmium, antimony, tin or barium in the upper wall will significantly contribute to overall signal. It is therefore imperative to either flatten the tubing for analysis or cut it in pieces and then analyze as shown in Figure 40.



Figure 39. . **Incorrect Way to Measure Heat Shrink Tubing**



Correct Way to Measure Heat Shrink Tubing WARNING Thickness correction is only for use with plastic/polymer samples.

Enable/Disable Paint

Selecting the Enable Paint option from the Tools Menu will enable the Painted Products mode and toggle the option to Disable Paint. Selecting the Disable Paint option will disable Painted Products mode and toggle the option to Enable Paint. See "APPENDIX A - Consumer Goods Mode for Screening Consumer Products" on page 145 for details on how to use this mode.

Action Level

See "APPENDIX A - Consumer Goods Mode for Screening Consumer Products" on page 145 for details on how to use Action Levels.

Print Data

Selecting the Print Data option from the Tools Menu will print the currently displayed data to the optional attached printer. See Setting Up the Printer for details.

Coatings Method

Metals are sometimes coated with various materials. If you wish to analyze the coating, select the Coatings Method. See "Coatings Analysis" on page 81 for details.

Passwords and User Privileges

- 1. Install the latest version of Niton software (NDT) on your PC, if possible. You may obtain the latest version of NDT by contacting service at 800-875-1578.
- 2. You can check the version number by opening NDT, selecting the Help menu, then selecting "About Niton Data Transfer"



Figure 41. . Selecting Help

3. Select the File menu



Figure 42. Selecting File

4. Select "New" then "New Password File". Your screen should look like this:

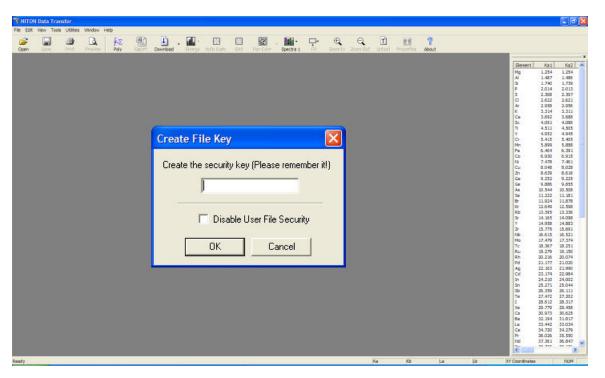


Figure 43. Creating the Security Key

5. Create a unique security key, then select the OK Button

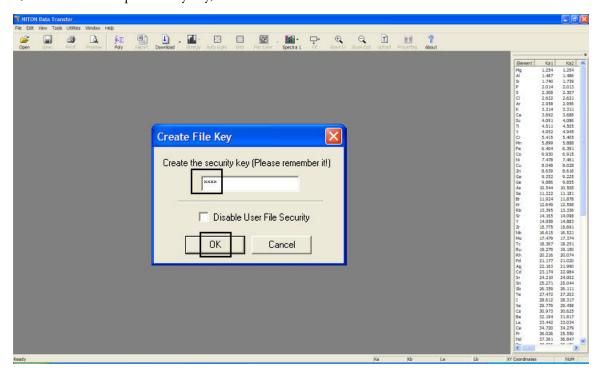


Figure 44. Security Key

6. Your screen should look like this:

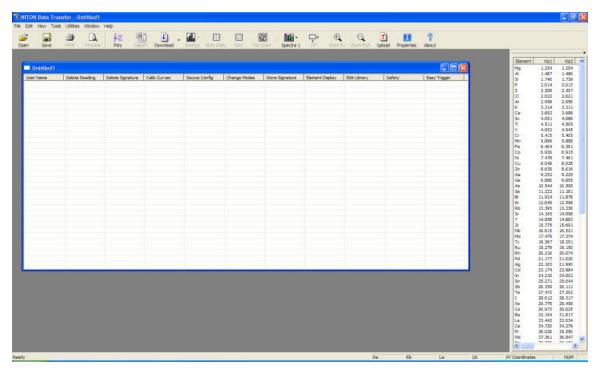


Figure 45. User Account Creation Screen

7. Right click, then select "New User"

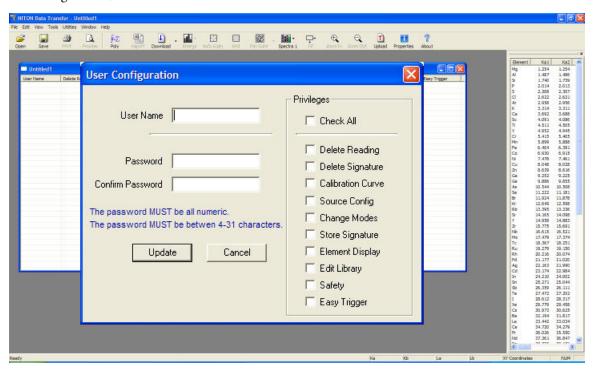


Figure 46. User Creation Dialog Box

8. Enter a user name and password, then select the privileges assigned to this user. Selecting the Check All check box will result in enabling all features.

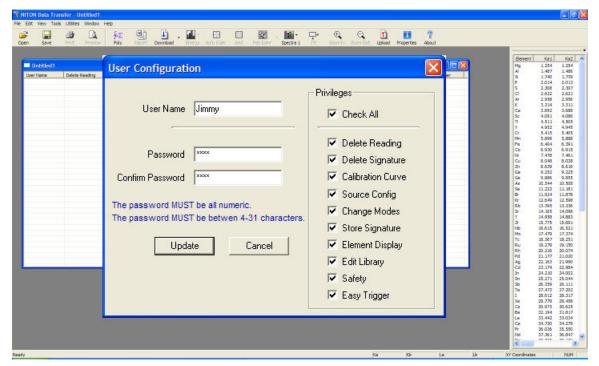


Figure 47. . Creating a User

WARNING it is recommended that only users at the highest level have access to the "Safety" feature. This should be unchecked for all other operators.

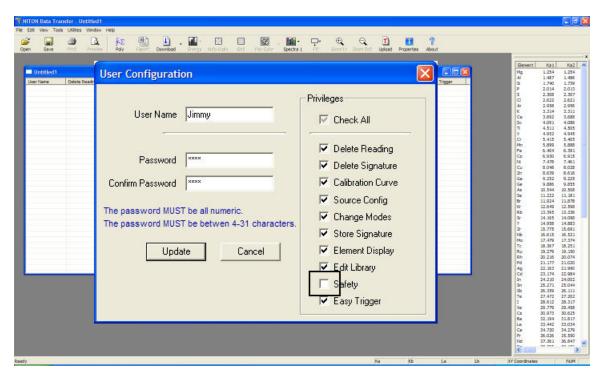


Figure 48. Unchecking Safety

9. Select the Update Button

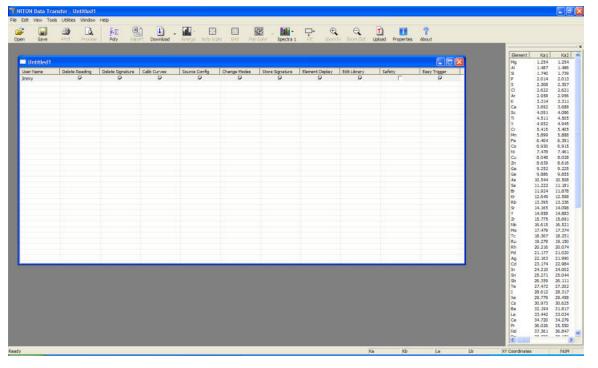


Figure 49. User is Created

a. You are now ready to upload your password file to the analyzer.

- 10. Be sure the analyzer is switched on; connect the analyzer using USB or serial connection.
- 11. Select the Upload icon.

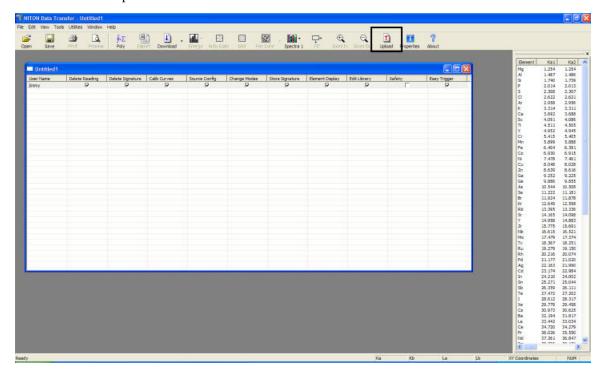


Figure 50. Selecting Upload

12. Your screen should look like this:

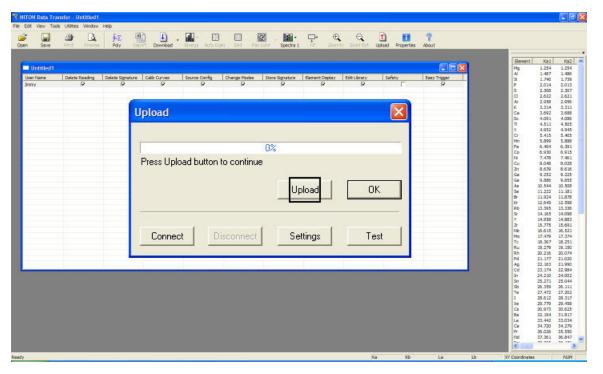


Figure 51. Selecting Upload

- 13. Select the Settings Button and choose the comm port that your analyzer is connect to.
- 14. Select the Connect Button, then the Upload Button.
- 15. Upon completion, you will receive a "File Upload Successfully Completed" message.
- 16. Click the OK Button; save your password file at this time by selecting the File icon then "Save As.

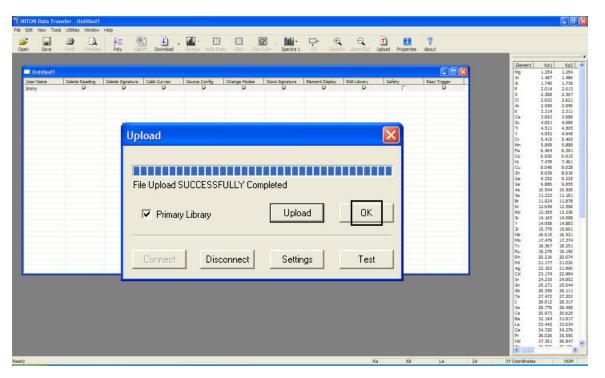


Figure 52. Successful Installation Message

17. Restart your analyzer; your password file should be successfully installed.

NDF Files: User Data Structuring

Creating New User-Defined Fields

You can create your own data entry fields for your Niton analyzer customized to your own needs and usage. These fields are saved in a special format called an NDF (Niton Data File) file. To create a new NDF file, select the File menu, then select New, then select New NDF File.

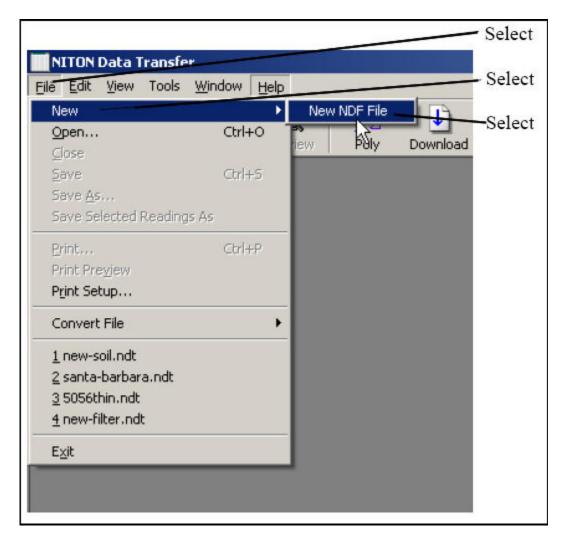


Figure 53. Creating a New NDF File

This will create a new window in which you can create your own fields, and specify their structure and parameters. The new window will appear with a single box, called "Untitled."

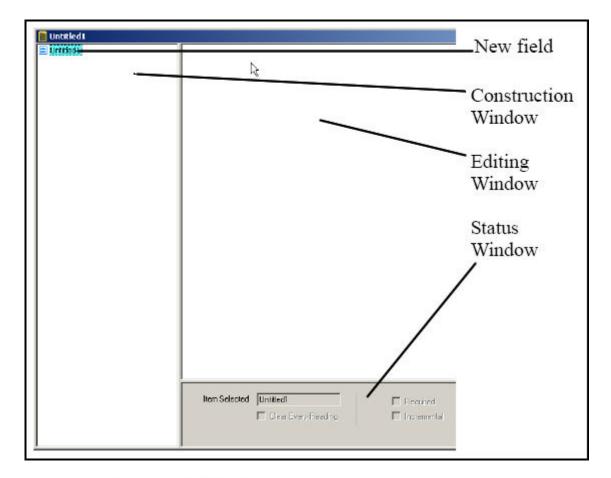


Figure 54. NDF File Work Area

By right-clicking on this box, you can access a pop-up menu allowing you to set the mode of the new data fields. Select New Mode to access the menu.

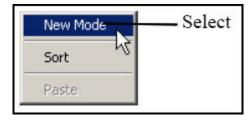


Figure 55. Selecting New Mode

The Mode you select will be the Mode within which the new data entry fields will appear. If you have multiple Modes enabled on your analyzer, the new fields will only be available from the Mode you select. Only the default fields will be available from the other Mode or Modes.

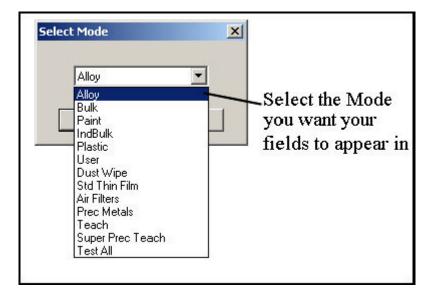


Figure 56. Selecting Mode

When you select the Mode for the new data fields, the Construction Window will change to look like this:

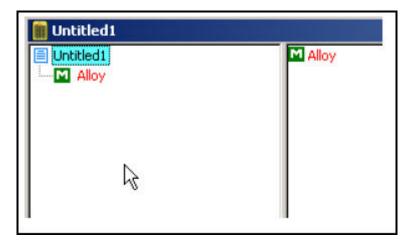


Figure 57. Working within a Mode

The "M" indicates the mode you have chosen - in this case Alloy Mode. Right click on the Mode name to access a pop-up menu.

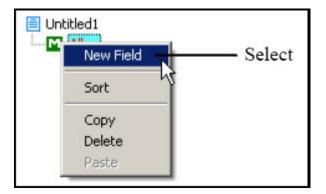


Figure 58. Mode Pop-Up Menu

Select New Field from the menu, and a blank new field will appear in the construction window.



Figure 59. Adding a New Field

Right clicking on the New Field box will bring up another pop-up menu. This menu gives you various options for using the field in your operations.

Selecting Required makes it mandatory that the new field be filled in prior to taking a measurement. This is very useful for necessary descriptors which vary from measurement to measurement, such as lot numbers, condi-tion descriptors, locations, etc.

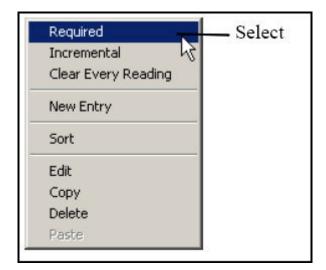


Figure 60. Making Fields Required

Selecting the Incremental option sets up a field which increments the field descriptor by one for each measurement taken. This option is handy for measuring several items with identical descriptors, such as samples within a single lot, or several instances of the same part number, because it appends the incremental number to the descriptor.

For example: P/N 455A2-1, P/N 455A2-2, P/N 455A2-3.

Another Example: Impeller-1, Impeller-2, Impeller-3.

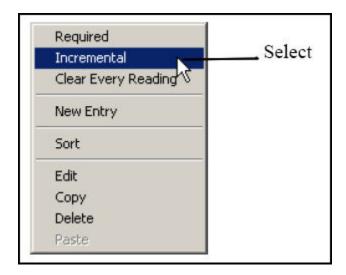


Figure 61. Making Fields Incremental

Selecting Clear Every Reading will toggle between two states. By default, the field will fill with the data which was input during the last reading. By selecting Clear Every Reading, you tell the instrument to clear the data from the field for each new reading, insuring that the person taking the reading must input new data each time. This is very useful for times when the data descriptor is expected to vary widely between readings.

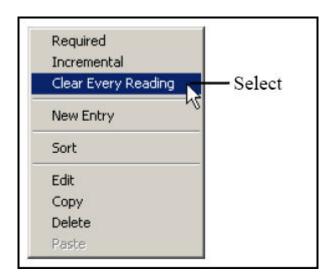


Figure 62. Clearing Data for New Readings

The state of each of these options can be seen in the Field Status Window at the bottom of the Construction Window. All options in effect for the field selected are checked.

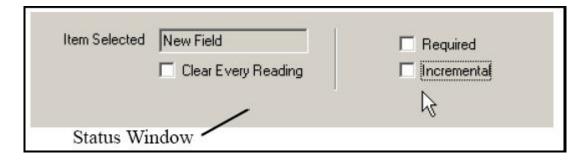


Figure 63. Field Status Window - Default

This shows a field with no options in effect, the default configuration. This is a field that will present the previous reading's data for this field - which may be changed by the user - without incrementing it, but does not require the user to input any data if there is none already there from a previous reading.



Figure 64. Field Status Window - Options Enabled

This shows a field with both Required and Clear Every Reading options in effect. This presents a field that is cleared for each reading, and must be filled in by the user before a reading is taken.

Selecting Edit from the pop-up menu allows you to edit the name of the field in the Editing Window to the right of the Construction Window.

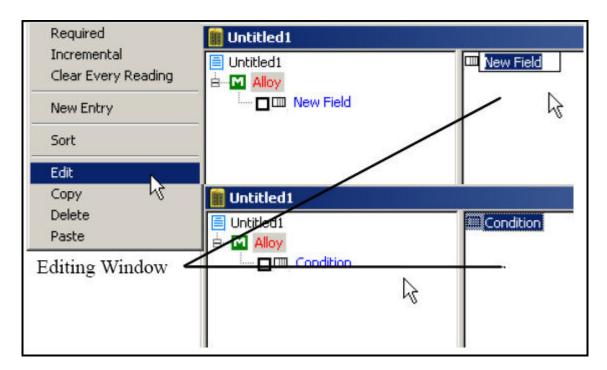


Figure 65. Editing the Field Name

Selecting the box to the left of the field toggles the Required option on or off.

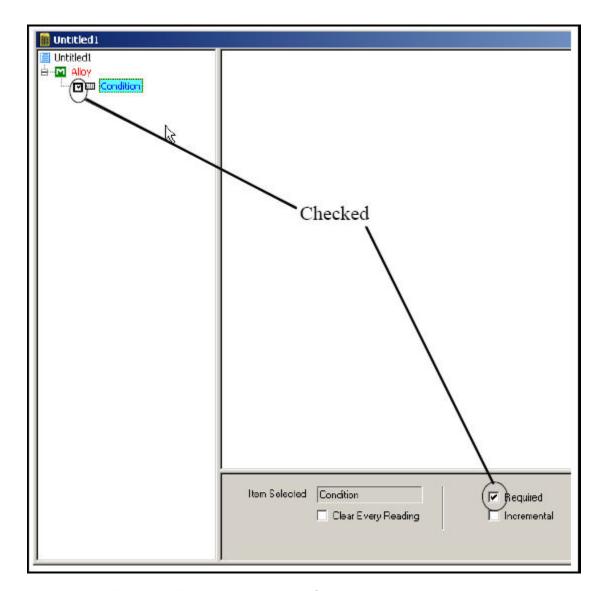


Figure 66. Toggling the Required Option

Selecting Copy from the pop-up window allows you to copy the currently selected field.

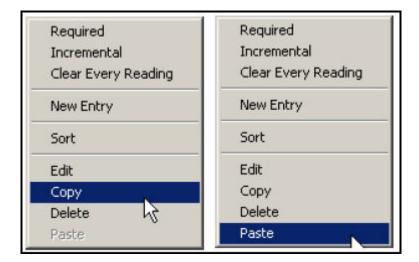


Figure 67. Copying the Current Field

Once you copy a field, the Paste option can be selected to paste the copied field into the Construction Window.

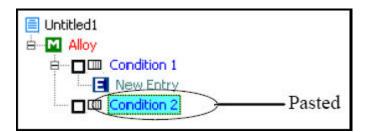


Figure 68. Pasting a Copied Field

Selecting the New Entry option from the pop-up menu allows you to define a choice for the user for this field.

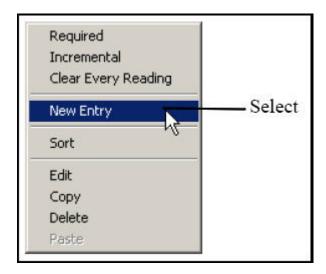


Figure 69. Creating a New Entry

This is a New Entry in the Construction Window.



Figure 70. New Entry in the Construction Window

The "E" is for "Entry." You can edit the entry once it is created, the same way as you edit the field name. Right click on the entry name, and choose Edit from the pop-up menu.

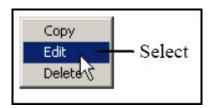


Figure 71. Editing the New Entry

You can sort your entries by name, alphanumerically, by right clicking on the field and selecting "Sort" from the pop-up menu.

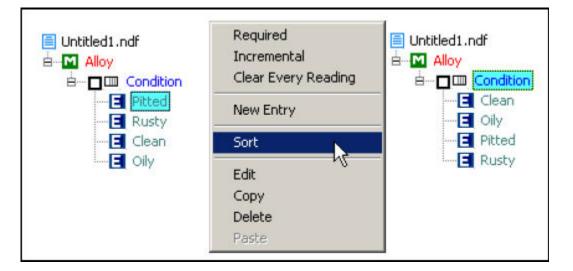


Figure 72. Sorting Entries

To delete a field or entry, just right click on the item you wish to delete, and select Delete From the pop-up menu.

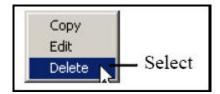


Figure 73. Deleting Fields and Entries

When you are finished creating your new NDF file, Upload it to your instrument using the Upload icon.



Figure 74. Uploading the NDF File

Make sure the instrument is connected to your computer by testing the con-nection first. Use the Test button on the Upload Window.

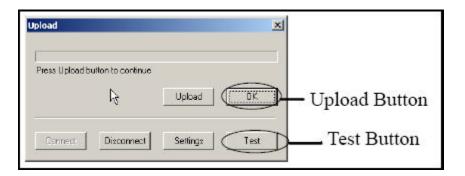


Figure 75. Testing the Upload Connection

Pseudo-Elements

Pseudo-Elements are constructs you can create which will be treated like elements in analysis, showing up in analysis as if they were actual elements. An example of this is hardcoded into the system. You can have total Halogens show up as a line item in analysis - see Consumer Goods Analysis Appendix A for details - which sums the total detected Br and Cl in the sample. In setting up a Pseudo Element, you must name it with at least 3 characters and at most 6 characters - except you cannot use * (asterisk) and space. You can set up equations with the following operands:

Add

You can Add detected levels of elements or Pseudo Elements together or to a constant. For example: sum applicable elements for FAC Analysis - (FAC=Cr+Cu+Mo), or sum elements for Residual Element Application - (REA=Cr+Ni+Cu).

Subtract

You can Subtract detected levels of elements or Pseudo Elements from each other or a constant. For example: find the level of clinker material C2S in cement - (C2S = (2.87*SiO2)-(0.754*Ca3SiO5))

Multiply

You can Multiply detected levels of elements or Pseudo Elements by a constant. For example: Convert elemental data to oxide form - (CaO=Ca*1.4).

Divide

You can Divide detected levels of elements or Pseudo Elements by a constant. For example: Convert Au concentration to Karat% - (Karat=((Au/100)*24))

Parentheses

You can use parentheses to stack order of calculation, as in the Karat and C2S examples above.

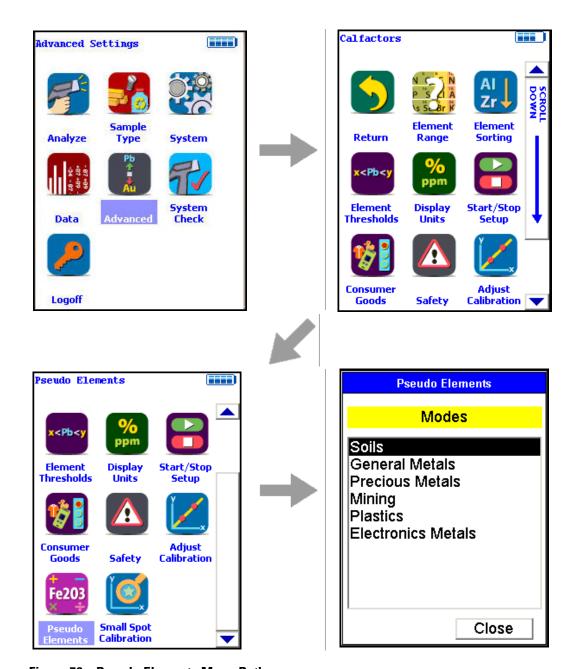


Figure 76. Pseudo Elements Menu Path

The Pseudo Elements Screen enables you to Create, Edit, and Delete Pseudo Elements in all Modes available on your analyzer. From the Pseudo Elements Screen, select the mode you wish to create the Pseudo Element for.

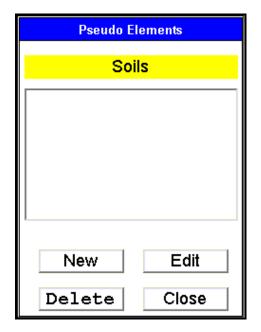


Figure 77. The Pseudo Element Mode Editor (Soils Mode)

To set up a new Pseudo Element, select the New Button. To Edit an existing Pseudo Element, select the Edit Button. To Delete an existing Pseudo Element, select the Delete Button. To Close the window and return to the previous screen, select the Close Button.

New

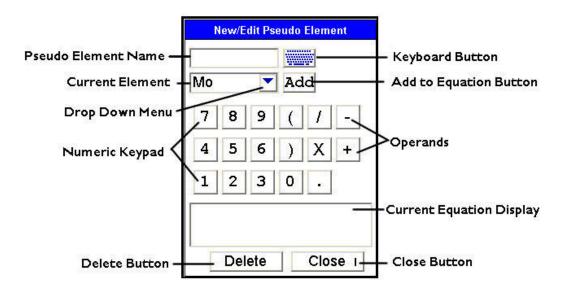


Figure 78. The Pseudo Element Equation Editor

- 1. Select the Keyboard Button to bring up the Virtual Keyboard.
- 2. Type in the name you chose to call your Pseudo Element.

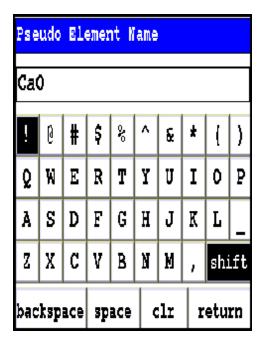


Figure 79. Using the Virtual Keyboard

- 3. Select Enter.
- 4. Select the inverted triangle button to open the Drop-Down Menu.

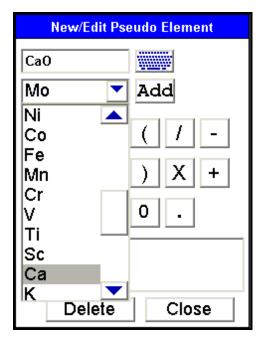


Figure 80. Selecting Elements from the Drop-Down List

- 5. Select the element you want to add to the equation. It will appear in the Current Element window.
- 6. Select the Add Button to add the Current Element to the equation.

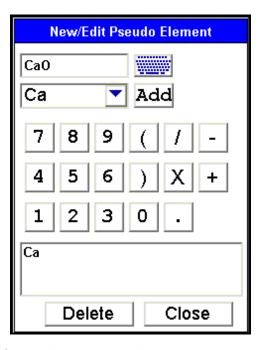


Figure 81. Adding the Current Element to the Equation

7. Using the Virtual Numeric Keypad integral to the editor, type in the other parts of the equation.



Figure 82. Adding Operands and Constants

- 8. When the equation is complete, select the Close Button.
- 9. The Pseudo Element will show up in the Mode Editor Window.

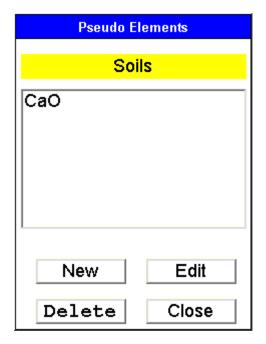


Figure 83. The Finished Pseudo Element in the Mode Editor Window

Edit

Select the Pseudo Element you want to edit from the Mode Editor window, then select the Edit Button.



Figure 84. Selecting a Pseudo Element for Editing

This will open up the Pseudo Element Equation Editor with the selected Pseudo Element pre-loaded.

Delete

1. Select the Pseudo Element you want to delete from the Mode Editor window.

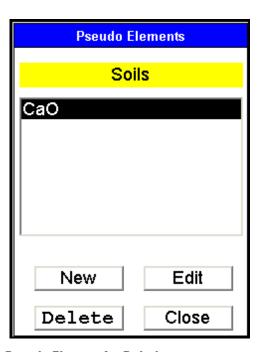


Figure 85. Selecting a Pseudo Element for Deletion

- 2. Select the Delete Button.
- 3. Select the Close Button to return to the previous screen.

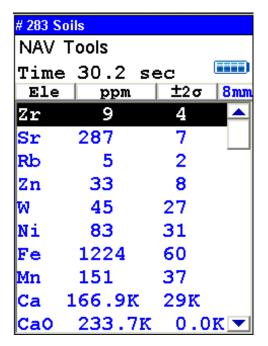


Figure 86. Pseudo Element Displayed in Reading

TestAll Geo

TestAll Geo Mode allows you to test powder, mineral, and ore samples without first determining whether the samples would best be analyzed with Mining or Soil Mode. TestAll Geo Mode uses both the Compton Normalization calibration (Soil) and the Fundamental Parameters calibration (Mining) to determine whether the soil calibration is acceptable or whether the total metal content is too high for Compton mode.

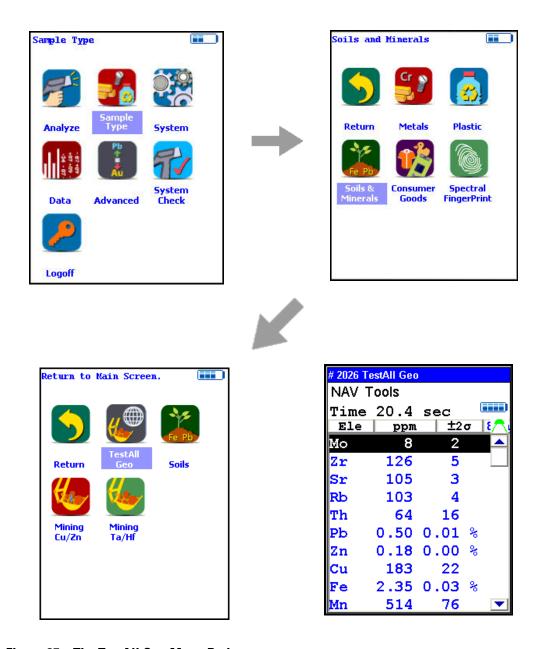


Figure 87. The TestAll Geo Menu Path

If the sample can be analyzed via soil mode, then the analyzer will display results from both Soil and Mining Modes in one unified list. If both calibrations contain the same element, then the mode that has the lower detection limit will be displayed. Elements will be displayed in both ppm (mg/kg) and wt.% with 0.10% being the cutoff point.

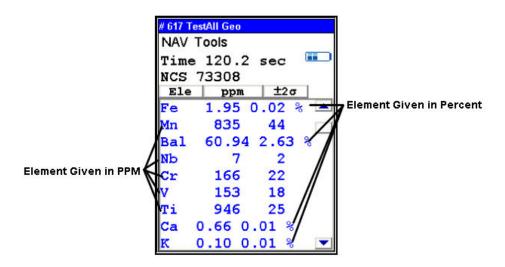


Figure 88. The TestAll Geo Analysis Screen

Notice the ppm heading, but % is listed with results where appropriate.

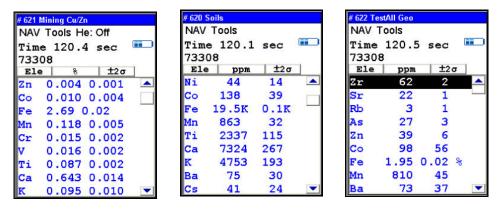


Figure 89. The Same Sample As Analyzed in Mining, Soil, and TestAll Geo Modes

TestAll Geo Mode may be used with any Standard Operating Procedure appropriate for either Mining Analysis or Soil Analysis.

Safety Settings

Access to the Safety Settings Screen is blocked unless the user logging in has explicitly been granted Safety access. The default login of 1234 does not have Safety access. See Passwords and User Privileges.

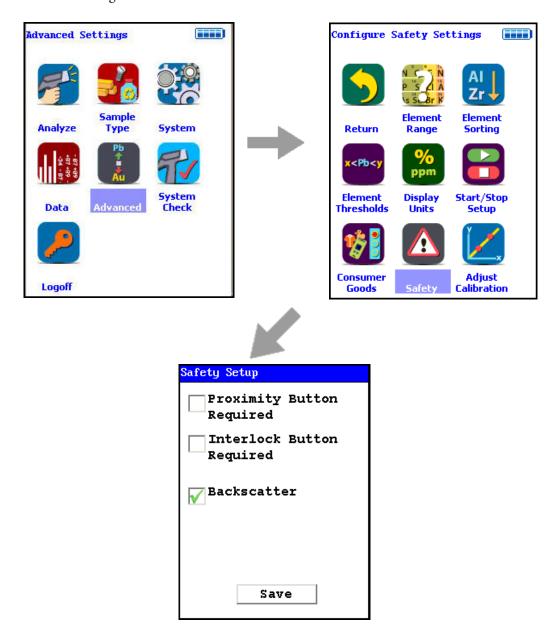


Figure 90. . Safety Settings Menu Path

The Safety Settings Screen enables you to change the Method of Operation for your analyzer. Each checkbox on the screen enables or disables the safety device named for purposes of the preconditions for operation. For example, checking the Proximity Button Required checkbox sets the engagement of the Proximity Sensor as a precondition for operation. Checking the Proximity Button Required checkbox and the Interlock Button Required checkbox sets the engagement of both the Proximity Button and the Interlock Button as preconditions for operation.

Safety settings always override start-stop settings. If your safety setting requires the use of the Proximity Button, you cannot set start-stop settings which ignore the Proximity Button. For example, the Easy Trigger start-stop setting must have the Backscatter safety setting enabled. While using Easy Trigger, you cannot disable Backscatter.

WARNING The backscatter sensor is enabled by default and acts as a recommended safety feature for most applications. Some sample types, however, cannot be analyzed when this feature is enabled. Samples that present very little mass to the analysis window, such as certain thin films, thin layers of plastic, and very thin wires, may not be of sufficient mass to allow the analysis to continue while backscatter is enabled. One should disable the backscatter feature only when necessary to analyze such low mass samples, and re-enable the feature when finished with these sample types. These samples also provide very little absorption of the primary x-ray beam so it is typically most appropriate to analyze these samples in a test stand when possible.

Start/Stop Setup

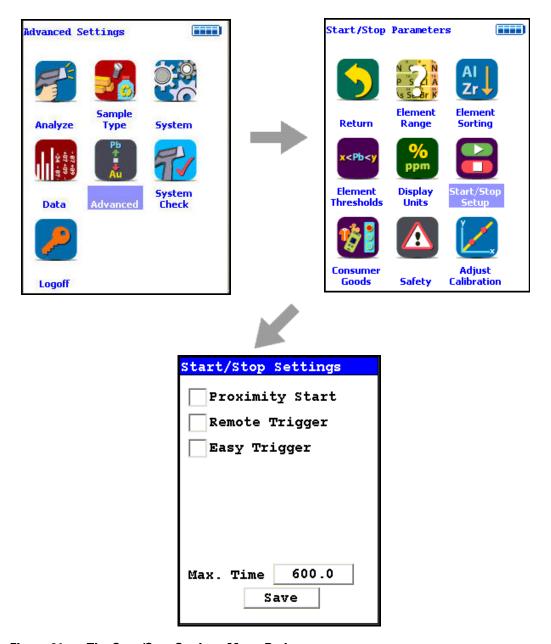


Figure 91. The Start/Stop Settings Menu Path

The Start/Stop Setting Screen enables you to change the preconditions for operation at a lower level than the Safety level. See Safety Settings for more information. Start/Stop settings cannot contradict Safety settings. If your safety setting requires the use of the Proximity Button, you cannot set start-stop settings which ignore the Proximity Button. For example, the Easy Trigger start-stop setting must have the Backscatter safety setting enabled. While using Easy Trigger, you cannot disable Backscatter.

Safety Settings

The Start/Stop parameter options are Proximity Start, Remote Trigger, and Easy Trigger. There is also a field to set the maximum time for sample analysis before the analysis stops.

Proximity Start

Select the Proximity Start checkbox to use the Proximity Start parameters. Using Proximity Start, once the reading has been started, release of the Proximity Button will immediately stop the analysis. You cannot use Proximity Start with Easy Trigger.

Remote Trigger

Select the Remote Trigger checkbox to use the Remote Trigger parameters. Remote Tigger is used with the Extend-a-Pole accessory to control the analysis. With the Extend-a-Pole's input cable connected to the analyzer's Remote Trigger port, you can initiate and stop analysis remotely from the Extend-a-Pole's handle trigger. You can use Remote Trigger with either Proximity Start or with Easy Trigger.

Easy Trigger

Select the Easy Trigger checkbox to use the Easy Trigger parameters. Easy Trigger uses a single press and release of the trigger to initiate analysis, and a second press to stop analysis. Selecting Easy Trigger will immediately disable Proximity Start.

Max Time Field

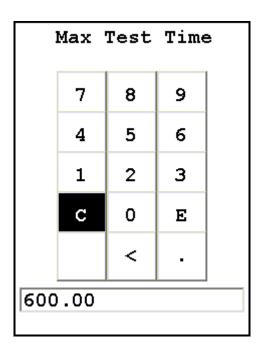


Figure 92. The Max Test Time Editor

Select the Max Time field to change the maximum analysis time parameter. Selecting the Max Time Field brings up a Virtual Numeric Keypad. To input the maximum number of seconds before automatic shutoff, select the C button to clear the current time, then from the Virtual Numeric Keypad, select each digit you want to input, then select the E button to enter. Of the non-numeric screen buttons, C = Clear All, E = Enter, and ">" will backspace over the current value. Selecting the E button will enter the current value as the Max Time, and return to the Start/Stop Settings Screen.

Save Button

Selecting the Save Button will save your current settings.

Updating System Software

You can use your computer to upload system software to your analyzer. Open the NDT application. Turn the device on and connect via USB or serial; wireless connection can be used, but is not recommended for this procedure. See Connecting the XRF for details on how to connect your analyzer and PC.

To upload CPU and FPGA code, please select the Utilities Menu then the Update Firmware option. Your computer will open a dialog box.

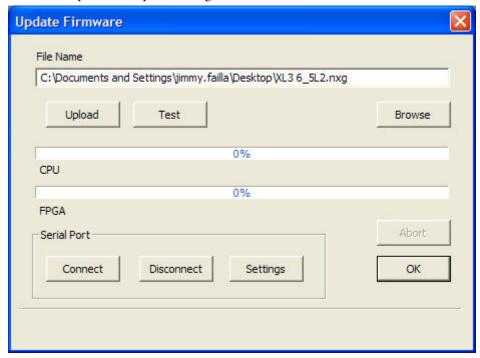


Figure 93. The Update Firmware Window

Select the Settings Button, then select your com port from the list. When you are done, select the Connect Button and the Test Button. You should see this message:

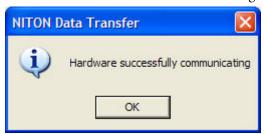


Figure 94. Successful Communications

Click on the OK Button when you see this dialog box.

Find the CPU code you wish to upload by selecting the Browse Button, then selecting the .nxg file you wish to upload. Select the Open Button when you are ready.

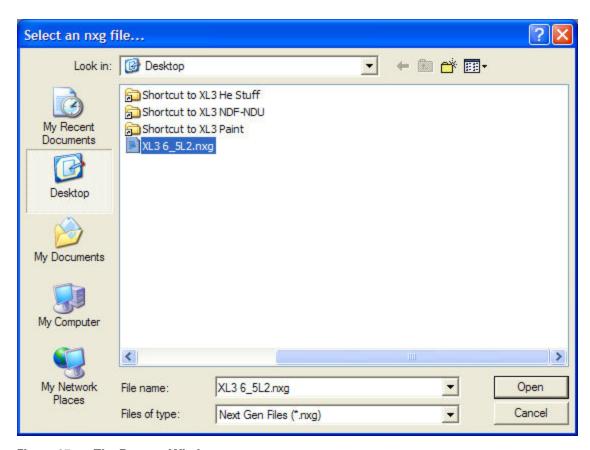


Figure 95. The Browse Window

Select the Upload Button; the progress bar will go to 100% and you will receive this message: **Note** Do not remove the battery or power supply from the instrument during this process.

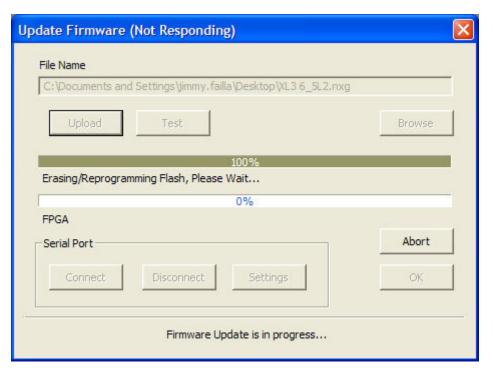


Figure 96. The Progress Bar

Once the code has finished installing, the analyzer will give you an audio signal (beeping) that it is done and NDT will display this message:

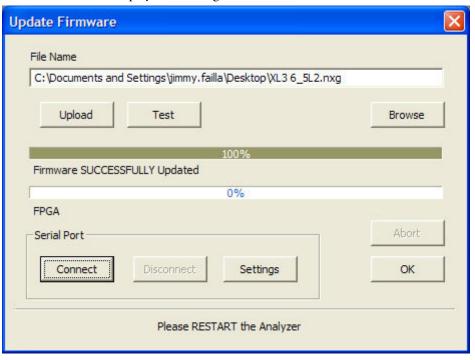


Figure 97. . Firmware Successfully Updated Message

Restart the instrument when you see this message.

Log on to the instrument. From the Main Menu, select the System icon, then the Specs icon. You should now see the updated version of code in either your "SW Ver" or "FPGA" line, depending on what you were uploading. Check the latest version of the mode specific calibration manuals for the minimum code requirements. The latest released code is always recommended when beginning a new calibration.

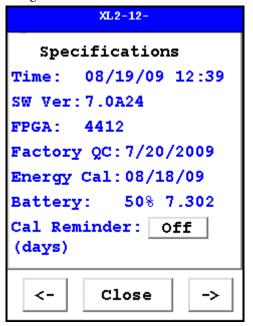


Figure 98. The Specs Screen

Loading the .dll file (Calfile)

Each device has an instrument-specific .dll file, which is generated from the Calibration file associated with it.

Note Loading this .dll file will set all settings on your analyzer back to factory specifications, and you will lose all your custom settings. Do not load the .dll file unless told to do so by Service. To obtain your instrument specific .dll file, please contact Service.

NDT may be used to upload this .dll to the analyzer. Turn the device on and connect via USB or serial; wireless connection can be used, but is not recommended for this procedure. See Connecting the XRF for details on connecting your analyzer and PC.

To upload the .dll file, please select the Utilities Menu, then the Upload Calibration Dll option. Your should see this dialog box:

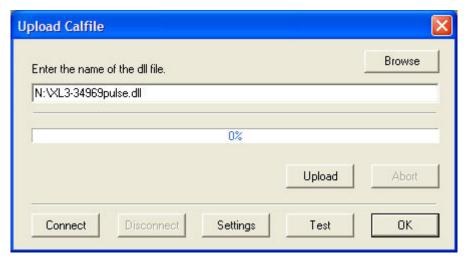


Figure 99. Loading the .dll File (Calfile)

Select the Settings Button, then select your com port from the list. When you are done, select the Connect Button and the Test Button.

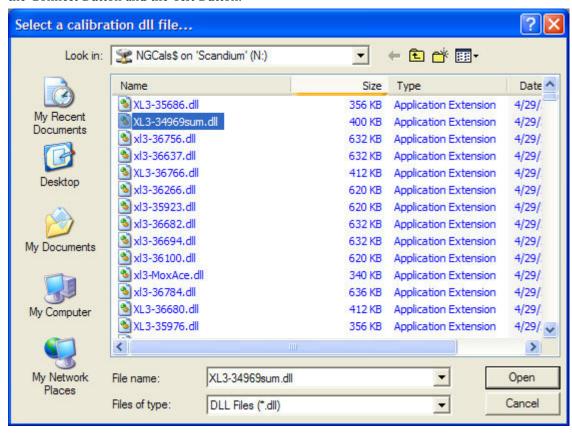
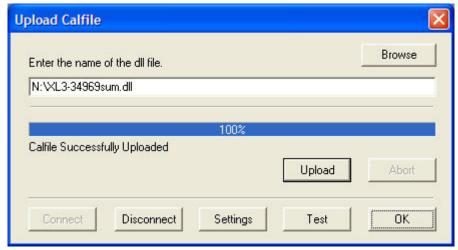


Figure 100. Browsing to Find the .dll File

Find the .dll file you wish to upload by clicking on the Browse Button and selecting the file. Select the Open Button when you are ready.



Select the Upload Button; the progress bar will go to 100% and you will receive this message:

Figure 101. The Progress Bar

Note Do not remove the battery or power supply from the instrument during this process.

Restart the instrument when you see this message.

Adjusting the Calibration

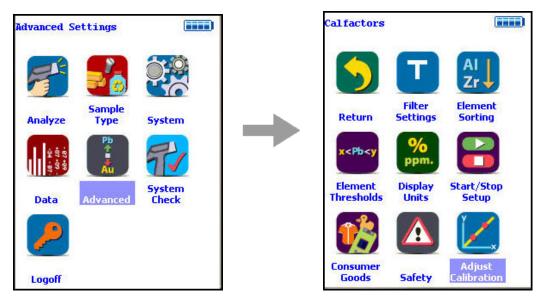


Figure 102. The Adjust Calibration Menu Path

The Adjust Calibration Screen enables you to change calibrations for Mining. Select the Mining from the list. Changing calibration factors for other modes must be enabled by download from your PC.

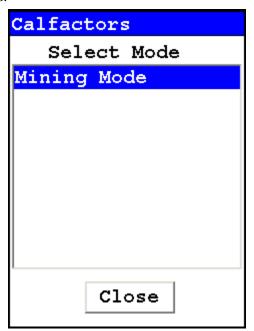


Figure 103. Select Mode

The Calfactors Screen will now appear.

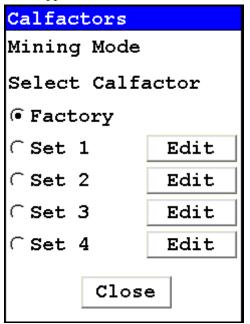


Figure 104. The Calfactors Screen

Select the radio button of the Calibration Factor Set you wish to edit, then select the appropriate Edit Button. The Calibration Edit Screen will open. You cannot edit the Factory Calibration. You may edit and store up to four alternate calibrations per Mode.

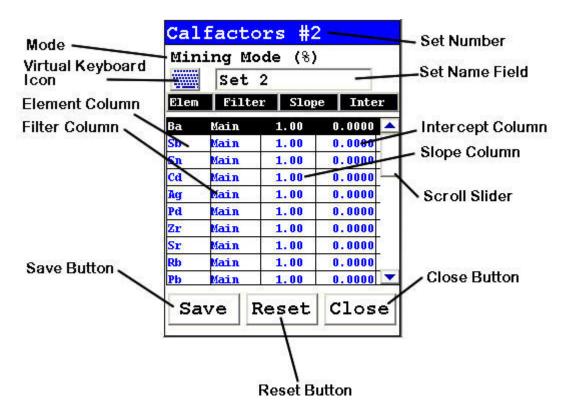


Figure 105. The Calibration Edit Screen

Select element you wish to edit in Element Column

Select intersection of Slope Column and Element Row to edit Slope.

Select intersection of Inter Column and Element Row to edit Intercept

Select Virtual Keyboard Icon to bring up the Virtual Keyboard and edit Set name in the Set Name Field.

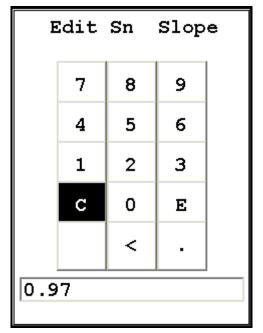


Figure 106. Changing Set Names

Editing the Calibration Factors

Selecting the intersection of the Slope Column and the Element Row will bring up the Slope Edit Screen, a variation on the standard Virtual Numeric Keypad. To enter in a new value for this slope, select the C Button to clear the field, enter in the new value for the slope, and select the E Button to enter this value. The ">" Button operates as a backspace key.

Selecting the intersection of the Intercept Column and the Element Row will bring up the Intercept Edit Screen, a variation on the standard Virtual Numeric Keypad. To enter in a new value for this intercept, select the C Button to clear the field, enter in the new value for the intercept, and select the E Button to enter this value. The ">" Button operates as a backspace key.



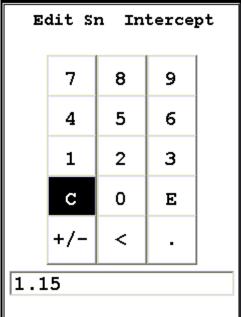


Figure 107. Slope and Intercept Edit Screens

Calibration Factors

Although the FP software automatically corrects for most inter-element matrix effects, Niton XL3 analyzers cannot currently detect elements lighter than magnesium. As a result, the oxidation state of elements can bias measurements of other elements. In many cases, this bias is small, and uncorrected results provide sufficient accuracy, especially when data is for screening purposes only. For cases when more accurate results are required, We have provided slope/intercept calibration software with the analyzer to improve the quality of the data. The calibration software is element specific, and calibration factors can be entered for individual elements, independent of the other elements. A user may choose to correct a single element or a suite of elements.

The degree of severity of the bias should be evaluated before proceeding with routine measurement. A few test samples should be carefully measured by another technique, or by an outside lab. These samples should then be analyzed using the analyzer. If the agreement is good enough to provide the accuracy required for the application, the instrument can be operated as shipped. If it is determined that a bias correction is necessary, the procedure for establishing calibration factors should be followed. As site characteristics change, it is good practice to run a few check standards to ensure that the instrument results are still within an acceptable error range.

Oxides versus elemental concentrations

Laboratories, certificates of analysis and other XRF instruments sometimes report data as oxides. The analyzer reports data only in elemental concentration. Therefore, oxide data must be converted to elemental concentration for comparison with Niton XL3 results. This may be achieved using the conversion factors below. The oxide is multiplied by this factor to convert to elemental concentration.

Oxides vs. Elemental Concentrations	
Oxide	Conversion Factor
MgO	0.6030
Al2O3	0.5292
SiO2	0.4674
SO3	0.4005
K2O	0.8301
CaO	0.7147
TiO2	0.5994
V2O5	0.5602
Cr2O3	0.6842
Mn3O4	0.7203
MnO	0.7745
Fe2O3	0.6994
FeO	0.7773
Co3O4	0.7342
NiO	0.7858
CuO	0.7989
ZnO	0.8034
PbO	0.9283
Fe2O3	0.6994
Bi2O3	0.8970

ZrO2	0.7403
MoO3	0.6665
WO3	0.7930
Ta2O5	0.8190
Nb2O5	0.6990
SnO2	0.7877

This conversion factor is obtained by taking the atomic weight of the element multiplied by the number of its atoms in the oxide, and dividing by the molecular weight of the oxide.

Worked examples:

To convert SnO2 to Sn:

Sn = concentration of SnO2 * 0.7877

To convert Sn to SnO2:

SnO2 = concentration of Sn * 1/0.7877

Measuring the Standard Samples

Use your analyzer to take readings of samples for which you already know the composition of the sample. It is important that the known composition of these samples be accurate. These samples provide the baseline to which your analyzer is adjusted. If the known composition is inaccurate, your analyzer will also be inaccurate.

For each sample, take a reading of at least 120 seconds. Make a note of the reading numbers for these samples.

For each sample, your analyzer reports the percentage by weight for the elements present in the sample. With the calibration factors set to the defaults, these percentages will differ from the known percentages, but are used to calculate the calibration factors.

Note Readings from only three samples are required for each element, but increasing the number of readings also increases the accuracy of the results.

Calculating Calibration Factors

Using the data that you collected by measuring the standard samples, you now need to plot the percentage of each element as indicated by the standard samples,

against the percentage of each element as reported by your analyzer. Then use the linear regression function to calculate the slope and intercept for a straight line drawn through a graph of those data points. The slope and intercept for this line are the calibration factors for the element.

You may use any tools that you prefer to make this calculation. This document shows one possible method, using Excel.

Note - your analyzer reports percentages of elements, and the calibration factors are based on percentages of elements. If your standard samples indicate percentages of oxides, see "Oxides versus elemental concentrations" on page 573 to convert percentages of oxides to percentages of elements.

Calculating Calibration Factors Using Excel

To calculate calibration factors:

Open Excel. To do this:

- a. Click the Start button.
- b. Select Programs.
- c. Click Microsoft Excel.

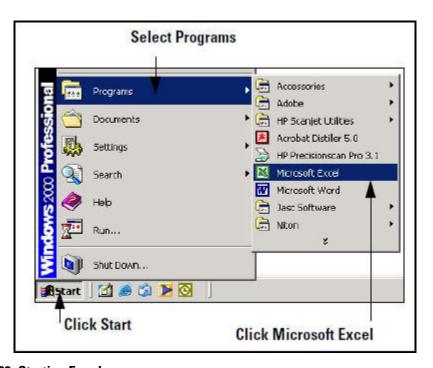


Figure 108. Starting Excel

1. In the first column, enter all the percentages by weight for the element as reported by your analyzer.

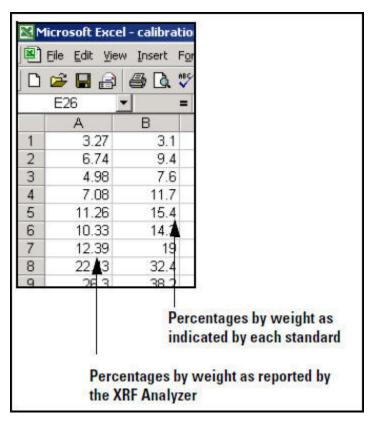


Figure 109. Adding Data

- 2. In the second column, enter all the percentages by weight for the element as indicated by each standard.
- 3. Use the cursor to highlight all the numbers in both columns.

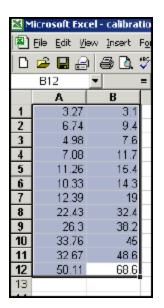


Figure 110. Setting Up the Chart

- 4. Click Chart Wizard.
- 5. Select the Scatter Chart with data points that are not connected by lines.

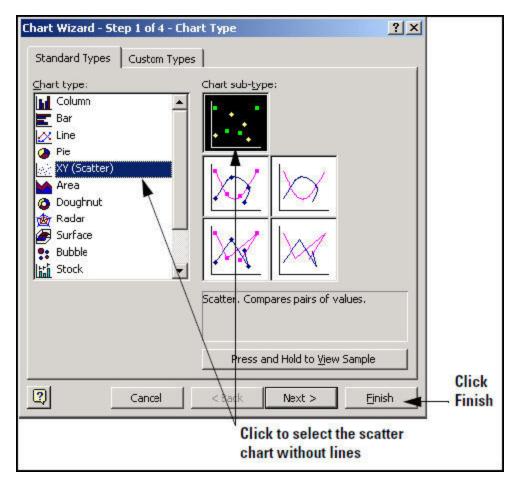


Figure 111. Selecting the Scatter Chart

- 6. Click Finish.
- 7. Right-click on one of the data points.
- 8. Click Add Trendline on the pop-up menu.

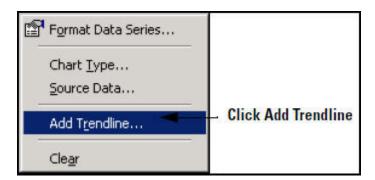


Figure 112. Adding the Trendlines

a. On the Type tab, click Linear.

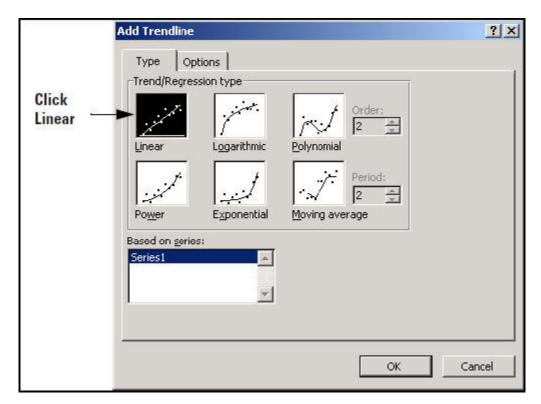


Figure 113. Selecting Linear Trendline

b. On the Options tab, check the boxes for "Display equation on chart" and "Display R-squared value on chart."

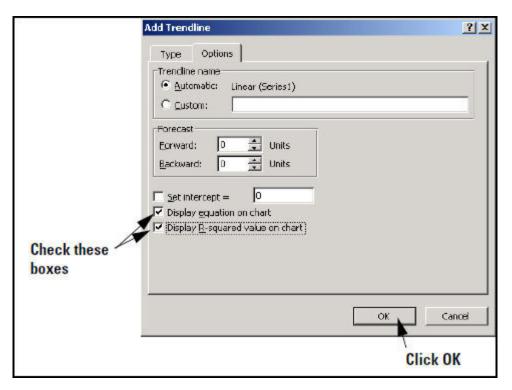


Figure 114. Selecting Options

- c. Click OK.
- d. The equation shows the slope and intercept for the trend line. These are the calibration factors that you enter into your analyzer.

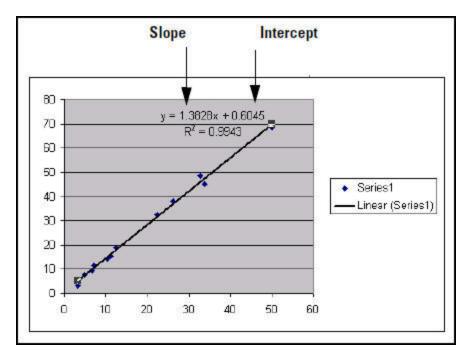


Figure 115. Slope and Intercept Displayed

Note If the intercept is negative in the equation, be sure that you enter the intercept as negative in your analyzer.

9. Repeat these steps to find the slope and intercept for every element that you are interested in.

Uploading Calibration Factors from your PC

For Precious Metals, Plastics, and Soil Modes, you need to upload the privilege to edit Calibration Factors from your PC before you can edit them. From NDT, create a New User, as described in Passwords and User Privileges. When selecting privileges, make sure that the checkbox next to Calibration Curves is checked, as in Figure 116 below. This user will have the ability to edit Calibration Factors for these modes.



Figure 116. Uploading the Calibration Curves

Display Units



Figure 117. The Set Display Units Menu Path

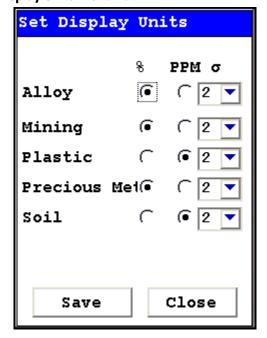


Figure 118. The Set Display Units Screen

Select the Set Display Units icon to choose between ppm (parts per million) and percentage (hundredths of whole) displays when taking readings, and to change the Sigma value you want for the reading.

Selecting the Set Display Units icon opens the Set Display Units Screen.

Setting Display Units

In the Set Display Units Screen, you can select between Percent composition and Parts per Million as the units displayed in a measurement, and you can change this setting independently for Alloy, Mining, Plastic, Precious Metal, and Soil modes. You can also change the Sigma for each of these modes independently.

When you have changed the display units to the appropriate values, select the Save button to save these settings for use. Select the Close button to return to the previous screen without saving any changes.

Changing Precision (Sigma Value)

Sigma is the symbol used for Standard Deviation, a measure of how much a set of numbers deviates from the mean. For example, each of the three data sets {0, 0, 14, and 14}, {0, 6, 8, and 14} and {6, 6, 8, 8} has a mean of 7. Their standard deviations are 7, 5, and 1, respectively. The third set has a much smaller standard deviation than the other two because its values are all close to 7. In a loose sense, the standard deviation tells us how far from the mean the data points tend to be.

The number of standard deviations between the process mean and the nearest specification limit is given in sigmas. As process standard deviation goes up, or the mean of the process moves away from the center of the tolerance, the sigma number goes down, because fewer standard deviations will then fit between the mean and the nearest specification limit.

Confidence Intervals

Confidence intervals assume that the data are from an approximately normally distributed population - generally, sums of many independent, identically distributed random variables tend towards the normal distribution as a limit. Using this assumption, about 68 % of the values must be within 1 standard deviation of the mean, about 95 % of the values must be within two standard deviations, about 99.7 % must lie within 3 standard deviations, and about 99.99% of the values must lie within 4 standard deviations.

The greater the sigma value of the test, the more confident you can be that the sample is as it appears, but the more difficult and time consuming the testing must be to verify this. That's why it's important to use the most appropriate sigma value for the test. By adjusting the sigma value for each type of test, you can optimize the process for your needs.

Adjusting the Sigma Values

The sigma values are listed in the column headed s. The default value is 2 sigma. You can change this value by selecting the down arrow next to the value, which opens up a drop-down menu from which you can select the desired sigma value by clicking on it.

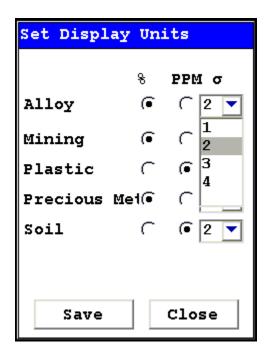


Figure 119. . Selecting the Sigma Value

When you have changed the sigma values to the appropriate number, select the Save button to save these settings for use. Select the Close button to return to the previous screen without saving any changes.

Methods of Operation

CAUTION After being powered on, your Niton Analyzer will perform an internal re-calibration before an analysis is initiated. It is recommended that you let your analyzer warm up for ten minutes after start up, before testing is begun.

There are six different methods of operation for taking a sample measurement, and your analyzer will be configured to use one of those methods for alloy samples, depending on the regulatory requirements of your locality. These methods are:

- Trigger-Only method. With the Trigger-Only method, you only need to place the
 measurement window flush with the sample to be analyzed and pull the trigger for sample
 analysis to be initiated.
- Trigger-and-Proximity-Sensor method. With the Trigger-and-Proximity-Sensor method, you must place the measurement window against the sample to be analyzed to engage the proximity sensor on the front of the analyzer, then pull the trigger for sample analysis to be initiated.
- Momentary-Trigger-Touch-and-Proximity-Sensor method. With the
 Momentary-Trigger-Touch-and-Proximity-Sensor method, you must place the
 measurement window against the surface to be analyzed to engage the proximity sensor
 on the front of the analyzer, then pull the trigger. The trigger may be released and the
 reading will continue until you release the proximity button, or other criteria (such as
 Max Time) are reached.
- Trigger-and-Interlock method. With the Trigger-and-Interlock method, you need to
 place the measurement window close to the sample to be analyzed, press and keep
 pressing the interlock button at the rear of the analyzer with your free hand, then pull the
 trigger for sample analysis to be initiated.
- Trigger-Interlock-and-Proximity-Sensor method. With the Trigger-Interlock-and-Proximity-Sensor method, you must place the measurement window against the sample to be analyzed to engage the proximity sensor on the front of the analyzer, press and keep pressing the interlock button at the rear of the analyzer with your free hand, then pull the trigger for sample analysis to be initiated.
- Easy Trigger method. With the Easy trigger method, you need only place the
 measurement window against the sample area and pull the trigger once to initiate a
 sample analysis. Your analyzer will continuously sample the backscatter, using a complex
 internal algorithm, to determine if the measurement window is against a sample or
 pointing to the empty air. If it finds that there is no sample directly against the
 measurement window, the analyzer will stop directing radiation through the window as
 soon as this determination is made.

The analyzer is constantly checking the backscatter characteristics to determine if a sample is against the measurement window, whether or not the Easy Trigger method is being used, and will shut off any radiation directed through the window if it determines that there is no sample present.

With any of these methods, analysis will stop if any one of the preconditions are violated. For example, with the Trigger-Interlock-and-Proximity-Sensor method, if the trigger or the Proximity Sensor or the Interlock is released, the reading will stop immediately, and the X-ray tube will shut down.

After your analyzer is calibrated, initiate a sample reading using the appropriate method. If you attempt to initiate a sample reading using a different method, the analyzer will inform you that one or more of the preconditions need to be met in order for sample analysis to begin.

Note The LED lights will blink whenever the x-ray tube is on.

WARNING The nose should not be touched during sample testing and calibration. If an ESD event occurs during measurement, the instrument may terminate the testing in progress and automatically reset to LogOn screen. Any test data collected prior to reset will be lost and the testing may have to be repeated.

WARNING The preconditions for operation must be continued for the duration of the reading. If the preconditions are violated, the x-ray tube will turn off, the calibration shutter will close, and the measurement will end. The LED lights will stop blinking when the measurement is ended. The flashing of the LED lights is not synchronized to minimize power consumption.

To end the test, simply release the trigger mechanism, or any other applicable preconditions.

Sorting the Element Display

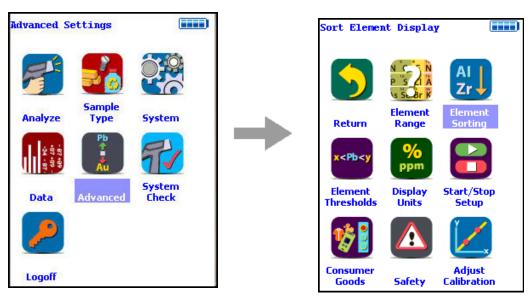


Figure 120. . The Element Sorting Menu Path

Select the Sort Element Display icon to configure sorting criteria used for analysis display. Selecting the Sort Element Display icon opens up the Sort Criteria Screen.

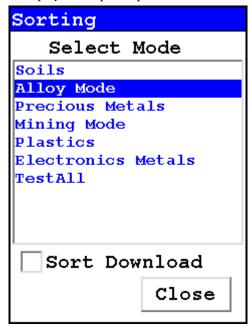


Figure 121. The Sort Criteria Screen

Select the mode you wish to change, and the Sorting Options Screen will appear.

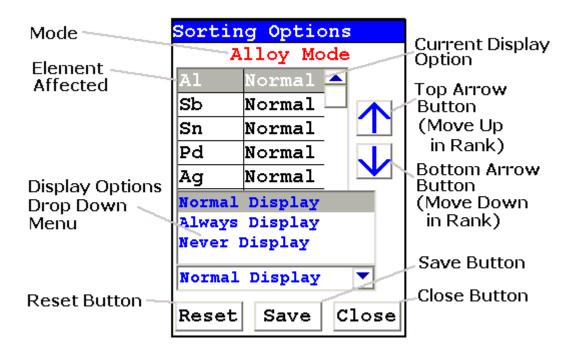


Figure 122. The Sorting Options Screen

On the left of the display are elements, each with its currently selected display option beside it to the right. The element list is ranked by importance, with the most important element on top, and each one lower down of less importance than the one above it.

By selecting an element and using the arrow buttons to the right of the list, you can change its ranking. Use the Top Arrow Button to move an element one rank closer to the top with each click. Use the Bottom Arrow Button to move an element one rank closer to the bottom with each click.

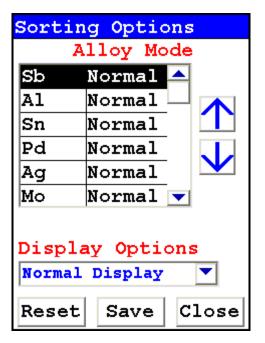


Figure 123. Changed Sort Order

Display Options

The Display Options Drop Down Menu, allows you to change the display status of any element to one of three states:

- Normal The standard state. Element displays only when the elemental value is greater than the limit of detection.
- Always Always display the results for this element. Use this state for elements critical to all of your analyses.
- Never Never display the results for this element. Use this state for elements which are unimportant to your work. This makes your instrument display less complex.

Select the element you want to change, then select the menu option corresponding to your choice of display status. The currently selected element is displayed in white on black.

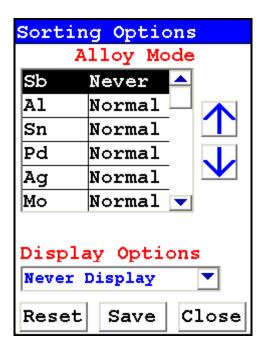


Figure 124. Changed Display Option

Select the Save Button to save your current status as the new default. After saving, you will go back to the Element Display Menu.

Close Button

Select the Close Button to exit without saving. When you select the Close Button after changing the display state of any element, a screen will open asking you if you want to save the changes you made. Selecting the Yes Button will save these changes as the new default. Selecting the No Button will return you to the Element Display Menu without saving the changes.

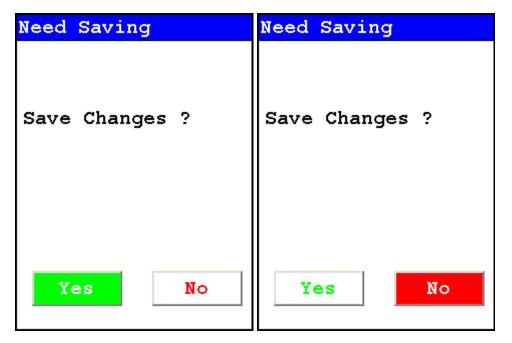


Figure 125. Save Changes Screen

Consumer Product Settings

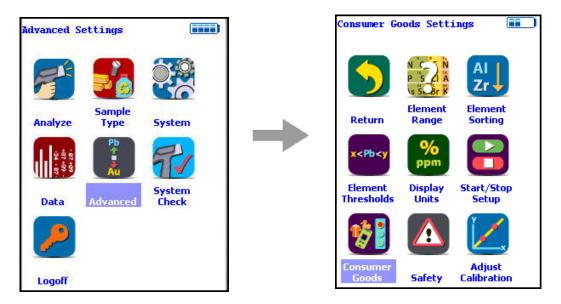


Figure 126. Consumer Products Settings Menu Path

Select the Consumer Goods Settings icon to configure Consumer Goods analysis criteria. Selecting the Consumer Goods Settings icon opens up the Consumer Goods Settings Screen. See Consumer Goods Analysis APPENDIX A for details on Consumer Goods Analysis.

In the Consumer Goods Settings Screen, you can change the Minimum Test Time in Detection Mode, and the Painted Products Action Level for all Analysis Options. You cannont change the Minimum Test Time for RoHS and Consumer Products Mode. In RoHS Settings Mode or Consumer Products Mode, the Min Test Time field is greyed-out and not editable.

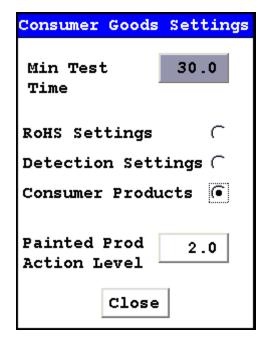


Figure 127. The Min Test Time Field Grayed Out

Editing the Min Test Time Field

To set the Minimum Test Time in Detection Mode, select the radio button next to Detection Settings. This will open the Min Test Time Field for editing.

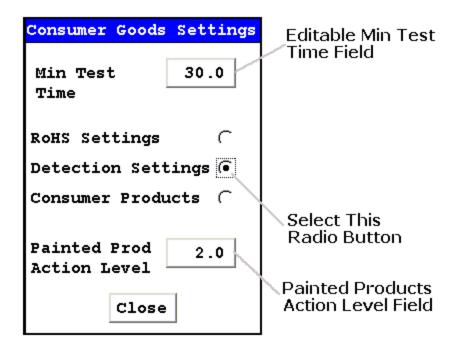
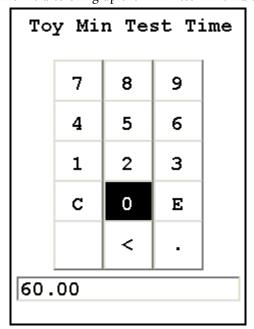


Figure 128. The Editable Min Test Time Field



Click in the Min Test Time Field to bring up the Min Test Time Editor.

Figure 129. Min Test Time Editor

Select the C Button to clear the editor's field, then type in the numbers you want for a minimum test time, using the Virtual Numeric Keypad. Select the E Button to enter this value when you are done.

Editing the Painted Products Action Level

To change the Action Level for Painted Products, click in the Painted Products Action Level Field to open the Painted Products Action Level Editor.

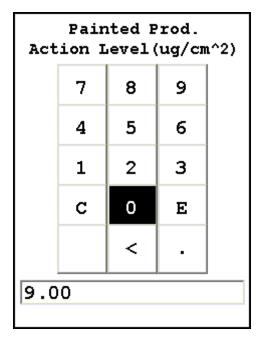


Figure 130. Painted Products Action Level Editor

Select the C Button to clear the editor's field, then type in the numbers you want for a minimum test time, using the Virtual Numeric Keypad. Select the E Button to enter this value when you are done.

Camera and Small Spot Video

The Camera feature is only usable with properly configured analyzers, and the Small Spot feature is only available on Small Spot analyzers.

If your analyzer is equipped with an internal video camera, you can turn that camera on and off, and turn the saving of images with the readings on and off through an interface. When the camera is on, the image will show in the Ready to Test screen, as in Figure 123. If the camera is off, saving of images will also be off. If the camera is on and the image saving function is also on, the images will automatically be saved with the reading. Saving images will curtail the maximum number of readings stored.

How to Use the Camera

When a Camera equipped analyzer is in the Ready to Test screen, the video feed appears live on the analyzer's touch screen. This is the image that can be saved with the sample analysis. When you take a measurement, if you choose to do so, the bitmap image will be saved on the analyzer along with the analysis results. The interface is accessible through the System menu, as in Figure 120.

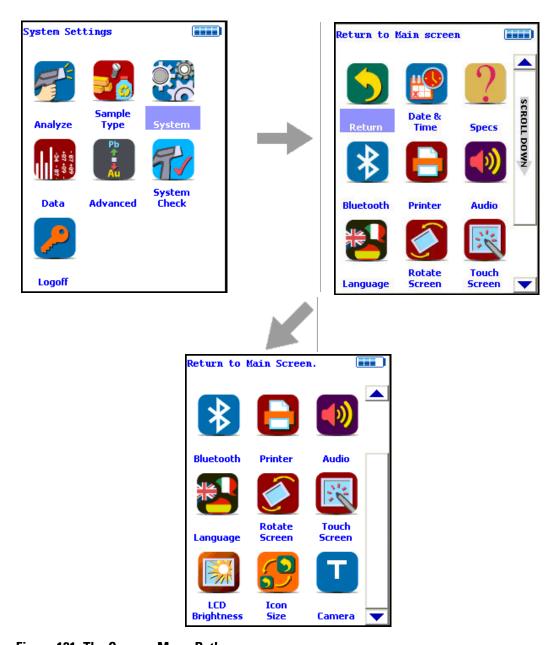


Figure 131. The Camera Menu Path

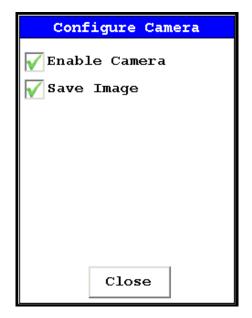


Figure 132. Setting Up the Camera View and Image Saving

Selecting the empty checkbox next to Enable Camera will turn the internal camera on, displaying the camera view in the Ready to Test screen. Selecting the checkbox again turns the camera off. Enable Camera is enabled by default.

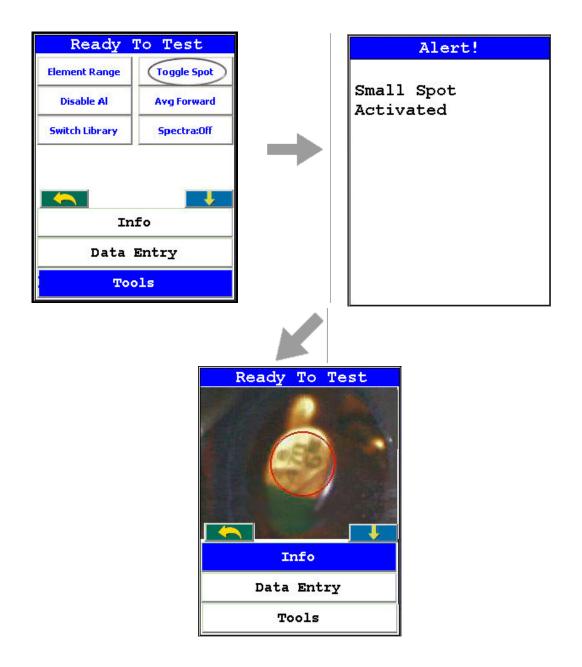
Selecting the empty checkbox next to Save Image will enable image saving with the analysis. Selecting the checkbox again will disable automatic saving of image data. Save Image is enabled by default.

Stored camera images from previous measurements can be viewed on the analyzer.

How to Use the Small Spot Technology

With a properly equipped Small Spot analyzer, you can restrict the analysis to a small spot within the camera view. You can toggle the Spot on and off from the Tools Menu as in Figure 122.

A red circle will appear on the display. The circle marks the area analyzed.



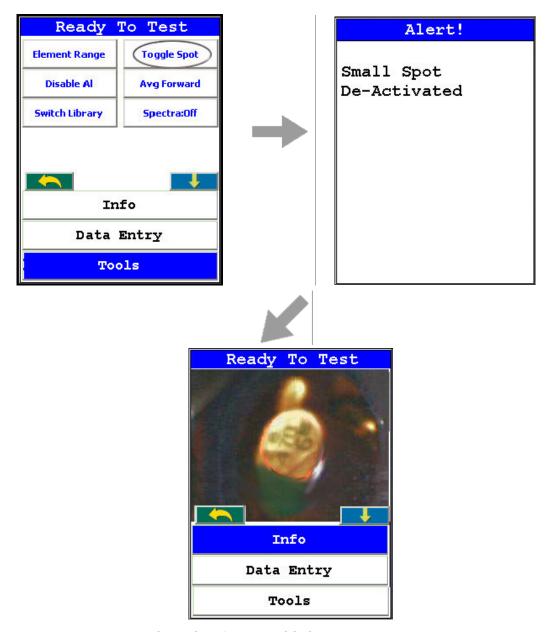


Figure 133. Toggling the Small Spot from the TOOLS Menu

Using the Small Spot

The Small Spot can be used in several different modes.

Mining

- 3mm spot size allows analysis of veins and inclusions in mineral samples
- Initiate from Tools Toggle Spot

He Purged

- Helium purge allows analysis of light elements (Mg, V and Ti)
- Small spot enables analysis of light elements in welds and inclusions
- Helium and Small Spot modes can be used simultaneously for Metals and Mining modes

Calibrating the Spot

If your instrument has small spot capability, you may calibrate the spot to optimize performance. Please follow the following procedure to calibrate the spot.

Requirements: CPU code 7.0A or higher, Small Spot QC Calibration Sample part #180-607.

- 1. Connect the instrument to NDTr. It is strongly recommended that this procedure be done in a test stand whenever possible. See Operating Your Analyzer Remotely for details.
- 2. Log on (Default "1234")
- 3. Enter a mode that has small spot capability. It is recommended that the you use either Metals or Electronics Alloy if possible. Then exit out to the main menu.
- 4. Select the Small Spot Calibration icon from the Advanced Menu

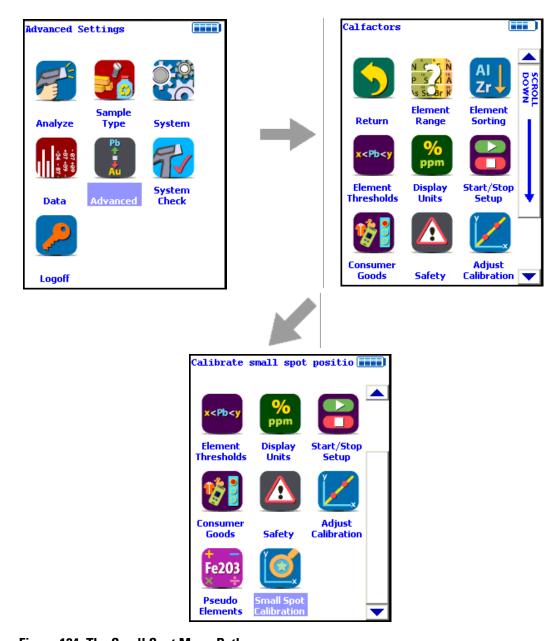


Figure 134. The Small Spot Menu Path

5. From the Calibrate Spot Screen, select the Yes Button.



Figure 135. Selecting the Yes Button

6. You will then receive a prompt asking if you want to reset the spot to the center position. It is recommended that you do this upon initial calibration. Saying "Yes" changes the position of the spot circle on the first calibration iteration to the calculated center. Saying "No" leaves this spot where it currently is for the first calibration iteration.

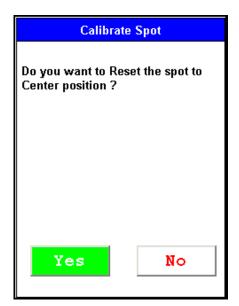


Figure 136. Centering the Spot

7. You will then see the small spot target on your instrument. Place the small spot calibration standard on the nose of analyzer so that the copper is in the center of the circle. If the copper is not properly centered for each of the five calibration shots, your calibration will not be valid.



Figure 137. Centering the Spot

- 8. After centering the standard, take a reading for 30 seconds. After the reading time ends, you will notice that the spot has changed orientation. Please follow the previous step and re-center your standard. You will be asked to do this a total of five times.
- 9. You will then receive a prompt informing you of the detected X and Y position of the spot. If you made an error along the way, please select "No" and begin again. If you would like to accept the calibration, please select "Yes".

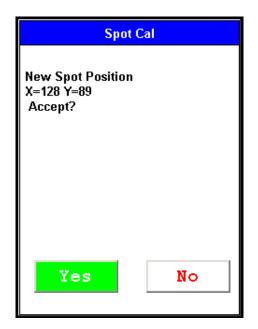


Figure 138. Spot Coordinates

10. Position the Small Spot Reference Sample over the calibrated spot and take a measurement. You should see >95% Cu in Metals or Electronics Alloy mode.

Note The 180-607 standard is unique and should only be used for spot calibration.



Figure 139. Positioning the Small Spot Reference Sample

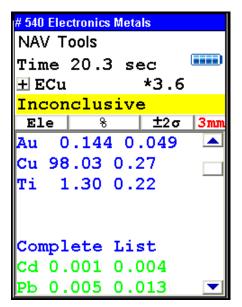


Figure 140. Small Spot Check Analysis

Reference Documents

Glossary of Terms

(including acronyms, abbreviations and symbols)

Word/Phrase	Definition
μΑ	micro-amp - one millionth of an amp, 10^{-6} A
μg	microgram -one millionth of a gram, 10 ⁻⁶ g
μg/ft ²	micrograms per square foot
μm	micrometer - one millionth of a meter, 10 ⁻⁶ M, also called a micron
%	percent
A	Ampere (often shortened to Amp), an SI base unit of electric current, equal to 6.242 x 10 ¹⁸ electrons / second
Accuracy	the closeness of a measured value to the actual (true) value
ADC	analog to digital converter
Ag	silver (element 47) a precious metal known since antiquity
AIHA	American Industrial Hygiene Association (US)
Al	aluminum (US/Canada) or aluminium (ROW), (element 13), most abundant metal in the earth's crust, first produced in 1825, it is so reactive it mostly occurs as the hydroxide in nature
ALARA	As Low As Reasonably Achievable, a method to minimize radiation exposure based on the principles of time, distance and shielding See Radiation and General Safety
Alloy	a partial or complete solid solution of two or more elements in a metallic matrix, e.g., steel (Fe + C), brass (Cu + Zn), bronze (Cu + Sn)
Alloy grade	the name or designation of an alloy with a particular composition, e.g., C377, Forging brass (Cu 58.0-62.0, Fe 0-0.3, Pb 1.5-2.5, Zn 35.2-40.5)
Alloy mode	see metals

Alpha particle	a helium nucleus, ⁴ ₂ He ²⁺ , a highly ionizing particle radiation
Am	americium (element 95), a synthetic radioactive element first manufactured in 1944
²⁴¹ Am	a specific radioisotope of americium, commonly pronounced as americium-241, used in some XRF instruments to provide the exciting radiation
Anodizing	an electrolytic passivation process used to increase the thickness of the natural oxide layer on the surface of a metal
As	arsenic (element 33), a metalloid known since antiquity, a poison
ASTM	American Society for Testing and Materials
ASIC	Application Specific Integrated Circuit (pronounced a-sick)- a custom designed chip
Au	gold (element 79), a precious metal known since antiquity, chemical symbol from the Latin aurum
Audio settings	user defined times for beep - See Setting up Audio Cues
Avg back	average back (abbreviation) See Averaging Results
Avg forward	average forward (abbreviation) See Averaging Results
Ba	barium (element 56), an alkaline earth and heavy metal, first identified 1774
Backscatter	when x-rays interact with low density materials (e.g., polyethylene) they can be scattered back towards the instrument and the operator
Backscatter characteristics	the expected x-ray scatter from a particular sample type
Backscatter shield	a device clipped onto the instrument manufactured from a dense material to absorb any backscattered radiation See Backscatter Shield
Bal	balance of elements (abbreviation), displayed in mining mode to account for light elements that are not detected by the instrument, e.g., N, O, Na
Barcode pencil	a device that reads a barcode and inputs the data electronically
Beep	a single tone made by a computer or machine, the sound made by the instrument at specific times
Beta particles	high energy electrons or positrons, β^- and β^+ -an ionizing radiation

Bi	bismuth (element 83), a metal known since ancient times, its' liquid phase is denser than its' solid phase
Binder	a material not containing any elements of interest that is used to hold together a sample in the form of a briquette, e.g., wax, cellulose or boric acid
Bq	Becquerel (abbreviation), an SI unit of activity, equal to 1 disintegration per second
Br	bromine (element 35), a halogen that is liquid at room temperature, used as a fire retardant
Brownfield	abandoned or underused industrial or commercial facility available for re-use, they may require environmental decontamination before redevelopment
Bulk analysis	the mode to select for analysis of bulk samples such as soil, mining and plastics
C	Celsius, a temperature scale, °C, also called centigrade
С	carbon (element 6), a common non-metal not detectable with the handheld XRF instrument
Ca	calcium (element 20), an alkaline earth metal, fifth most abundant element in the earth's crust
Calcination	a heat treatment process often occurring in a kiln which induces thermal decomposition. Ores may be treated in this way to give more usable materials, such as the calcination of limestone (calcium carbonate) to produce lime (calcium oxide) in the manufacture of cement
Calibrate	to undergo calibration
Calibration	a process whereby a signal is compared with a known quantity and a calculation is performed to generate values that are recognizable to the end user, e.g., ppm or %
Calibration factors	a multiplication factor that adjusts a reading to improve accuracy - See Adjusting Analyzer Calibration
Cb	columbium, see Nb (niobium)
Cd	cadmium (element 48), transition metal discovered 1817, a poison
¹⁰⁹ Cd	a specific radioisotope of cadmium, pronounced as cadmium-109, used in some XRF instruments to provide the exciting radiation
· · · · · · · · · · · · · · · · · · ·	

Ce	cerium (element 58), a lanthanoid metal, most abundant of the rare earth elements
CFR	code of federal regulations (US)
Cl	chlorine (element 17), a halogen that is a gas at room temperature, a common disinfectant
cm	centimeter
Со	cobalt (element 27), a transition metal discovered in 1735, known for its' characteristic blue color (cobalt blue) in paints and pigments
Coating	a thin covering over a substrate, usually to improve the surface properties such as corrosion resistance. Examples of coatings include anodized surfaces, electrochemical plating, chromate and phosphate coatings as well as paint and enamels
COD	Class of Device
Columbium	see Nb in glossary
COM port	communications port (abbreviation)
Compton scatter	the scattering of x-rays at lower energy than the incoming radiation
Concentration level	the amount of an element present, shown in either % or ppm
Confidence	a value indicating an estimation of probable values for a reading
Coning and quartering	a process of reducing a sample size of a powder without creating a systematic bias - See Soil Analysis
Consumer goods	
Core sample	a solid cylindrical rod of material representing changes in layers through depth. Both rock and soil cores may be analyzed. Rock cores can be hundreds of metres long and are used for exploration and mine mapping
Count rate	the number of signals (counts) per a time interval, e.g. counts per second
cps	counts per second
CPSC	Consumer Product Safety Commission (US)
CPU	central processing unit

CRM	certified reference material, a well-characterised material with a certificate which documents chemical concentrations with uncertainties generally obtained through multiple analyses. They may be used for calibration, QC or QA of an instrument
Crucible	a heat resistant container for heating samples
Cs	caesium (cesium US), element 55, discovered 1860, from the Latin caesius meaning blue-grey, first element discovered by spectrum analysis
CSV	comma separated value
Cu	copper (element 29), transition metal known since ancient times
Deep dose	sometimes referred to as "deep dose equivalent", the dose equivalent at a tissue depth of 1cm and applies to external whole body exposure (NRC) - See Radiation and General Safety
Dessicator	a sealable container usually containing a desiccant where moisture sensitive materials may be kept to prevent or minimize hygroscopic behaviour
Diagnostics	information displayed regarding the internal characteristics of the instrument
Display Units	elemental concentration units displayed on instrument, either % or ppm
Dosimeter	a device for monitoring radiation dose
DOT	Department of Transportation (US)
DSP	digital signal processor, a device that shapes and produces the spectrum in the XRF instrument
DQO	data quality objective
Dy	dysprosium (element 66), a lanthanoid metal and rare earth element, first identified 1886, from the Greek dysprositos meaning hard to get
Easy Trigger method	a way to run the instrument by placing it against the sample and pulling the trigger just once to activate the x-rays, the trigger does not need to be held down once measurement has started
EDXRF	energy dispersive x-ray fluorescence
Element	a pure chemical substance composed of only one type of atoms, e.g. iron (Fe), copper (Cu), gold (Au)

Elemental limits of detection Element a screen in the library editor which displays elements and variances for named alloys ELPAT Environmental lead proficiency analytical testing	alid
specification screen ranges for named alloys	alid
ELPAT Environmental lead proficiency analytical testing	
Environmental lead professing analytical testing	
ELV end of life vehicles directive (EU), adopted September 2000 encourage vehicle recycling and to make it more environment friendly	
electromagnetic radiation, the generic name which covers a self-propagating waves in a vacuum or in matter. It is classifint types based on the energy of these waves, so from least energetic to most energetic we have: radio waves, microway infra-red radiation, visible light, ultraviolet radiation, x-rays gamma rays	fied /es,
Energy Cal energy calibration (abbreviation), a line displayed on the specification screen indicating the last date when an energy calibration (detector calibration) was performed on the instr	ument
EPA US Environmental Protection Agency	
Er erbium (element 68) a lanthanoid metal and rare earth element discovered 1843	ent,
ESD Electrostatic Discharge, a sudden and momentary electric control that flows between two objects at different electrical potents	
Eu europium (element 63) a lanthanoid metal and most reactive rare earth elements, discovered 1890	of the
eV electron volts	
Ex-situ Latin phrase meaning off-site. Often a soil sample is remove its' original location, prepared (dried, ground, sieved and cu and then analyzed ex-situ	
FAA Federal Aviation Administration, part of the USDOT, regularization in the USA	ates
FAC Flow-assisted corrosion, a process in which steel pipe is deadue to flowing liquids or liquid-steam mixtures	graded

Factory QC	A process of running certified reference materials to check the analytical integrity of the instrument at the place of manufacture before shipment. As shown on the instrument specifications screen, this defines the actual date of this process
Fe	iron (element 26), transition metal known since ancient times
⁵⁵ Fe	a specific radioisotope of iron, pronounced as iron-55, used in some XRF instruments to provide the exciting radiation
Ferrule	a small metal object used for fastening
Filter	a mechanism built into the instrument that allows the x-ray energy to be modified to preferentially enhance the analysis of certain elements in certain matrices - See Adjusting the Element Range
Filter	a thin support mechanism for sample collection, such as a 37mm air filter
Flange	an internal or external rib which acts as a guide
Flux	a material which promotes glass formation
Fluxer	a device for rapidly heating a crucible containing a sample and a flux material, melting and mixing the sample and flux
Fingerprint ID	a method of comparing an unknown with a known sample
FP	f undamental p arameters (a calibration technique based on the physics of the instrument and sample)
FPGA	Field Programmable Gate Array, a type of ASIC chip used in the instrument. As shown on the instrument specifications screen, this defines the version number
FPXRF	field portable x-ray fluorescence
FWHM	full width half maximum, a way of determining resolution by measuring the width of a peak at half its' height
g	gram, one thousandth of a kg, i.e. 10^{-4} kg
Ga	gallium (element 31), a metal discovered in 1875, it melts slightly above room temperature
Galvanization	A process that is used to coat iron or steel with zinc, which acts as a protective coating to the underlying metal. May be done either as a hot dip (thick coating) or by electroplating (electro galvanization) which provides a thinner coating

Gamma ray	a form of electromagnetic radiation of high energy produced by sub-atomic particle interactions, i.e. produced within the nucleus of an atom
GB	gigabyte
General metal	a mode in which to read most common alloy materials
GOLDD	Geometrically Optimized Large area Drift Detector (acronym)
Grade control	the measurement and adjustment of mined materials to keep the elements of interest with a specific concentration range
Grade Library	a listing of elements describing elemental ranges for specific alloy grades
Greenfield sampling	A preliminary mineral survey of a plot of land
Grinding mill	a mechanical device for mixing and grinding samples into a more homogeneous material with a finer particle size
Gy	gray, a unit of absorbed dose equal to 1 joule of deposited energy per kilogram of tissue See Radiation and General Safety
Halogen	the generic name for a group of non-metal elements from Group 17 (formerly VII, VIIA) of the periodic table, comprising F, Cl, Br, I and At
He purge	helium purge (abbreviation)
Hg	mercury (element 80), a heavy transition metal that in its' pure form is a liquid at room temperature and pressure. Also known as quicksilver.
High Range	the Range used in the instrument to preferentially fluoresce many of the heavy elements ($Z = 47-56$)
Horizontal slider	an on-screen tool allowing a cursor or other device to be moved left to right
IATA	International Air Transport Association, an international group of airlines formed to represent, lead and serve the airline industry. It publishes the IATA Dangerous Goods manual which covers the shipping of hazardous materials (including radioactive materials)
ICRP	International Commission on Radiological Protection, an advisory board which provides recommendations and guidance on radiation protection

IDL	instrument detection limit, this is the best possible (i.e. lowest value) LOD that can be obtained, typically from pure matrices
In	indium (element 49), a rare metal first isolated in 1864, greatly used in the electronics industry, especially LCDs
Infilling	taking readings at a higher rate to more closely define the elemental composition of an area of interest, an exploration technique
In situ	Latin phrase meaning in place. The XRF instrument may be used directly on a sample without moving or preparing it, for example taking a soil reading in situ.
Ir	iridium (element 77), a transition metal discovered in 1803, a PGM, the second densest element and most corrosion resistant metal currently known
keV	kilo electron volts
kg	kilogram, an SI base unit of mass, approx equal to the mass of 1 liter of water – currently being revised by the SI committee
LCD	liquid c rystal d isplay
Library	see Grade Library
Light Range	the filter used in the instrument to preferentially fluoresce light elements where $Z < 17$
Live spectrum	a real time spectrum of the sample currently undergoing analysis
LOD	limit of detection - See Statistical Aspects of Spectrometry
LOQ	limit of quantization - See Statistical Aspects of Spectrometry
Low Range	the filter used in the instrument to preferentially fluoresce elements of $Z = 19-24$
MAC	media access control, an address mechanism in the CPU
Main Range	the filter used in the instrument to preferentially fluoresce the entire first row and some of the second row transition elements
Main menu	the main screen on the instrument through which all functions are available
Match / no match line	a line displayed on the analysis screen displaying an alloy match if found
Match fingerprint	an option on the instrument to compare spectral data from known sample to an unknown sample

Max time	maximum time (abbreviation), instrument may be programmed to run no longer than a certain time - See Start/Stop Setup
MDL	method detection limit, the limit of detection for a particular method (this includes taking into account sample matrix and preparation as well as instrument and filters used, and integration time)
Metals	the mode to select to allow analysis of metal samples such as alloys, precious metals and alloy coatings
mg/kg	milligrams per kilogram (commonly called ppm)
Mining	the process of removing material from the ground to extract the raw materials (ores or minerals) required, may be open pit or underground
Mn	manganese (element 25), a transition metal known since ancient times but not isolated until 1774
Mo	molybdenum (element 42), a transition metal discovered 1778
Mortar	a smooth bowl used for grinding samples (with a pestle)
mREM	millirem
MSv	milli-Sievert
Muffle furnace	a laboratory oven or kiln for heating and melting samples at a high temperature
Multi-filter	an instrument containing 2 or more filters which can switch during the without any prompting by the user to preferentially fluoresce specific elements (to give optimum results)
Mylar	a polyethylene terephthalate (PET) polyester material with high strength and chemical stability, used in the manufacture of thin film windows for soil cups (Part number 187-462, 6µ gauge, 2.5" diameter). Not suitable for light elements (Z<19)
N	nitrogen (element 7), a gas not detectable with the handheld XRF instrument
Na	sodium (element 11), an alkali metal discovered 1806, from the Latin Natrium, not detectable with the handheld XRF instrument
Nb	niobium (element 41), transition metal discovered 1801. Also known as columbium.
NCRP	National Council on Radiation Protection and Measurements (US)

NDT	non-destructive test
NDT	Niton Data Transfer, a software package provided with the instrument to assist in the interface between the unit and a PC
NDTr	Niton Data Transfer Remote control, a software package that allows the instrument to be run through a PC
Ni	nickel (element 28), a transition metal first isolated in 1751, a ferromagnetic element
NIOSH	National Institute for Occupational Safety and Health, the US federal agency responsible for monitoring and recommending preventative measures for work related injuries and illnesses
NIST	National Institute of Standards and Technology, a non-regulatory agency within the US Department of Commerce, previously called NBS (National Bureau of Standards), a supplier of certified reference materials
NRC	Nuclear Regulatory Commission, a US government agency overseeing the licensing and material safety of radioactive materials
О	oxygen (element 8), a common, highly reactive non-metal, the diatomic molecule $\rm O_2$ forms 20.9% of the earth's atmosphere, not detectable with FP-XRF
Os	osmium (element 76), a transition metal, also a PGM, discovered in 1803, the densest natural element
Oxidation	the process of gaining oxygen (or losing hydrogen) by an atom (or molecule) to form an oxide
Oxide	a chemical compound containing at least 1 oxygen atom and an atom of another element, e.g. Al ₂ O ₃ , aluminum oxide commonly known as alumina and Fe ₂ O ₃ , iron (III) oxide commonly known as ferric oxide or rust
P	phosphorus (element 15), a highly reactive non-metal, discovered 1669, named from the Greek for light bearer
Pb	lead (element 82), a heavy metal known since ancient times, a potent neurotoxin, chemical symbol from the Latin Plumbum (the same root word that we now get plumbing, lead was used to make pipes to carry water and waste
PC	personal computer

PCB	p oly c hlorinated b iphenyls, a class of organic compounds with 1 to 10 Cl atoms arranged on a double benzene ring with the generic formula $C_{12}H_{10-x}Cl_x$. A persistent organic pollutant
PCB	p rinted c ircuit b oard, typically a flat sheet which provides mechanical support and electrical connections to multiple components
PCS	performance characteristic sheet
Pd	palladium (element 46), a transition metal, also a PGM, discovered in 1803, an expensive precious metal
PE	polyethylene (US), polythene (UK), a common thermoplastic consisting of long chains of the ethane monomer - $(C_2H_4)_n$ -
Pearson correlation coefficient	see r
Periodic table	an organized table displaying the chemical elements, generally credited to Mendeleev
Pestle	a heavy rod with rounded end used for pounding and grinding samples in a mortar
PGM	Platinum group metal, a group of 6 precious metals all having similar chemical and physical properties, namely Ru, Rh, Pd, Os, Ir and Pt
Pistol grip	the part of the instrument to hold with your hand, the handle
Placer deposits	an accumulation of valuable minerals formed by deposition of denser materials in a trap site, e.g. inside bend of a river where gold can accumulate. (Pronounced plasser)
Plated surface	a metallic coating on a conductive surface providing a purpose, such as corrosion resistance, decorative value, increase hardness or solderability
Polypropylene	a thermoplastic polymer with reasonable strength and chemical resistance, used in the manufacture of thin film windows for soil cups (Part number 187-461, 4μ gauge, 2.5" diameter). Allows higher transmission of low energy x-rays, but is not as strong as, Mylar film.
PMI	Positive Material Identification

ppb	parts per billion, a unit of measure denoting relative proportions, and a dimensionless quantity as there are no associated units of measurement. It is not a SI term.
ppm	parts per million, a unit of measure denoting relative proportions, and a dimensionless quantity as there are no associated units of measurement. It is not a SI term.
Preamp	short version of preamplifier, an electronic amplifier used to prepare an electronic signal for further processing. In the XRF instrument, the preamp amplifies and differentiates the signal.
Precision	a measure of how close multiple readings of the same sample are to one another, also known as repeatability
Primary radiation	x-ray radiation that is emitted directly from the instrument - See Primary Radiation
Pt	platinum (element 78), a transition and noble metal, also a PGM, an expensive precious metal
PVC	polyvinyl chloride, a common thermoplastic consisting of long chains of the vinyl chloride monomer -(CH ₂ CHCl) _n -
Q	a quality factor, see RBE
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
Quicksilver	see Hg in glossary
r	Pearson correlation coefficient, a measure of the correlation between two variables used in statistics, with a value between -1 and 1 See Statistical Aspects of Spectrometry
r2	coefficient of determination (pronounced r squared) which gives an indication of the goodness of fit See Statistical Aspects of Spectrometry
rad	a unit of absorbed radiation equal to 0.01 joules of energy per kg of tissue. Now replaced with the SI unit gray, where 100 rad = 1 gray
Rb	rubidium (element 37), an alkali metal discovered 1861, from the Latin rubidus meaning deep red, which is the color it imparts to a flame

RBE	relative biological dose, a quality factor often denoted by Q used to assess the tissue damage caused by various types of radiation. Alpha particles have a $Q = 20$, while beta particles, x-rays and gamma rays are all the same with $Q = 1$
RCRA	Resource Conservation and Recovery Act, a federal law in the USA which governs the disposal of waste (solid and hazardous)
REE	rare earth element, a collection of 17 elements, Sc (21), Y (39) and the lanthanoids (57-71)
rem	Röntgen equivalent in man, an older term for measuring a unit of radiation dose based on the absorbed radiation and the biological efficiency of said radiation. Generally now replaced with Sv
Rh	rhodium (element 45), a transition metal, also a PGM, discovered in 1803, an expensive precious metal
RoHS	restriction of hazardous substances, a European directive on the use of hazardous substances in electrical and electronic equipment 2002/95/EC. It specifically restricts the use of Pb, Hg, Cd, Cr6+, PBB (polybrominated biphenyls) and PBDE (polybrominated diphenyl ether)
RMS	root mean square (sometimes called the quadratic mean)
RPD	relative percent difference - See Statistical Aspects of Spectrometry
RSD	relative standard deviation - See Statistical Aspects of Spectrometry
RSO	radiation safety officer
Ru	ruthenium (element 44), a transition metal, also a PGM, discovered in 1827, an expensive precious metal
S	sulphur (or sulfur) (element 16), a non-metal known since ancient times, sometimes called brimstone
Sample	the material to be analyzed
Sample cup	a device to contain a powdered (loose) or liquid sample for presentation to the instrument
SAP	sampling and analysis plan
Sb	antimony (element 51), a metalloid known since ancient times, a poison similar to arsenic

sd	standard deviation - See Statistical Aspects of Spectrometry
Se	selenium (element 34), a non-metal discovered in 1817
Secondary radiation	radiation that is scattered by a sample in multiple directions, typically at lower energy that the exciting primary radiation See Secondary Radiation
Shallow dose	sometimes referred to as "shallow dose equivalent", applies to the external exposure of the skin or an extremity, means dose equivalent at a tissue depth of 0.007cm averaged over an area of 1 cm ² (NRC) See Radiation and General Safety
Shutter	a device attached to the tube housing assembly which can totally intercept the useful beam (NRC)
Si	silicon (element 14), a metalloid first prepared in 1811, is the second most abundant element on earth
SI	Le Systèm International d'Unités (French) or International System of Units, the internationally recognized metric system and the world's most widely used system of measurement.
Sigma	the standard deviation of a population in statistics, commonly written as σ (lower case Greek alphabet)
Silica	Common name for silicon (IV) oxide, SiO ₂
Slope	a multiplication factor to adjust the slope (angle) of a calibration curve (line). Default is 1 (leaving the slope as it is), a number less than 1 reduces the reported value, a number larger than 1 increases the reported concentration
Sludge	a mixture of solids suspended in a liquid
Small spot	on instruments configured with small spot capability, the analysis area of the sample can be reduced to allow more accurate analysis of tiny parts
SOP	standard operating procedure
SpectraView	a way of viewing on the instrument screen the spectrum of a previously analysed sample
Spectrometer	a generic name for an analytical device which separates and reads any measurable properties in the electromagnetic spectrum
Specs	specifications (abbreviation)

Sr	strontium (element 38), an alkaline earth metal, discovered in 1798, 15 th most abundant element on earth
SRM	standard reference material, a certified reference material (CRM)
Standards (samples)	materials containing known concentrations of specific elements used to check the performance of an instrument
Standardizing	a way of calibrating an instrument, such as coating thickness
Stylus	an input tool for a graphic (touch) screen
Substrate	the base material of a sample
Sv	sievert, the SI derived unit of dose equivalent, describing the biological effects of radiation. This replaced rem, where $1 \text{ Sv} = 100 \text{ rem}$
SW version	Software version (abbreviation). As shown on the instrument specifications screen, this defines the version number
Ta	tantalum (element 73), transition metal discovered 1802
Tb	terbium (element 65), a lanthanide metal and REE, discovered in 1843
Тс	technetium (element 43), the lightest chemical element with no stable isotope, not naturally occurring on earth, a synthetic transition metal first reported manufactured in 1925, but discovery not confirmed until 1937
Te	tellurium (element 52), a rare metalloid discovered in 1782
Tetraethyl lead	an organometallic compound (CH ₃ CH ₂) ₄ Pb, the antiknock agent in leaded gasoline (petrol), now mostly banned for use in modern cars.
Threshold	a user defined minimum and / or maximum concentration value to determine pass or fail for a sample
Ti	titanium (element 22), a first row transition metal, discovered 1791
Trigger only method	a way to run the instrument whereby the x-rays are activated by pulling the trigger only - See Methods of Operation
Trigger and proximity sensor method	a way to run the instrument whereby the x-rays are activated only by pulling the trigger and engaging the proximity button simultaneously - See Methods of Operation

Trigger and Interlock method	a way to run the instrument whereby the x-rays are activated only by pulling the trigger and pressing the rear interlock button simultaneously - See Methods of Operation
Trigger-Interlock and Proximity sensor method	a way to run the instrument whereby the x-rays are activated only by pulling the trigger, engaging the proximity button and pressing the rear interlock button simultaneously - See Methods of Operation
TSP	total suspended particulate
U	uranium (element 92), heaviest of the naturally occurring elements, discovered 1789
V	vanadium (element 23), transition metal discovered 1801
V	volt
Vertical Slider	an on-screen tool allowing a cursor or other device to be moved up or down
Virtual keyboard	a software and hardware interface that allows users to enter alpha-numeric characters without the use of a physical keyboard. The XRF unit has a virtual keyboard employing a touch screen with sensing areas representing each key.
Volatile substance	a material that is easy to vaporize
W	tungsten (element 74), transition metal discovered 1783, heaviest metal with a known biological role. Also known as wolfram
W	watt, an SI unit of power, equal to 1 joule per second (energy conversion), it may also be defined in terms of mechanical energy (object moves at one meter per second against a force of one Newton. $1W = 1J s^{-1} = 1 kg m^2 s^{-3} = 1 N m s^{-1}$
WEEE	waste electrical and electronic equipment, an EU directive 2002/96/EC setting recycling and recovery rates for electrical goods to reduce toxic e-waste
x-ray	a form of electromagnetic radiation, with energy in the range 120eV to 120KeV (which is equal to a wavelength range of 10 to 0.01nm), also known as Röntgen radiation
XRF	X-ray fluorescence, an analytical chemistry technique
Z	atomic number, e.g. $Fe = 26$ and $Pb = 82$, also sometimes called the proton number as it is equal to the number of protons in the nucleus of an atom

Zn	zinc (element 30), transition metal known since ancient times
Zr	zirconium (element 40), transition metal discovered 1789

Technical Specifications

See "The Specs Screen" on page 427 for technical specifications.

XRF Theory

Brief Theory of XRF

When x-rays interact with matter, they cause other x-rays to be emitted that are characteristic of the elements present in the material being irradiated.

The reason this characteristic radiation is produced is caused by the very nature of atoms themselves. An atom can be considered as the smallest building block that can still be regarded as an element. It consists of a nucleus (the center part of an atom) made from positively charged protons and electrically neutral neutrons, and a surrounding electron cloud containing the same number of electrons as the nucleus contains protons, so an atom is a neutral particle. The number of protons in an atom defines the element itself and the number is referred to as the atomic number (sometimes also called the Z number). For example, a calcium atom has 20 protons and a lead atom has 82 protons. The electrons surrounding the nucleus are located in quantized orbitals of varying energy levels, referred to in XRF as "shells". A very simplified way of visualizing this is the "sun and planets" concept, where the nucleus (sun) is surrounded by orbiting electrons (planets). These "shells" are unique to each element and are identified by the letter series K, L, M, etc., where K is the closest to the nucleus, L is the second closest to the nucleus and so on. These letters may also be referred to as the principal quantum number n, where K is n=1, L is n=2 and M is n=3.

When x-rays enter an atom, there is a chance that they will interact with the electrons surrounding the nucleus and supply enough energy to cause the ejection (loss) of an inner electron. This process is called ionization as the atom becomes a positively charged ion, which is an unstable particle as it does not its' full complement of electrons. This vacant inner shell is filled by an electron from one of the outer shells in a cascade process. As the electrons move from one shell to another, they release their excess energy in the form of the characteristic x-rays detected by the instrument.

Figure 1-1 shows a diagrammatic representation of the hand-held XRF instrument. A miniature x-ray tube provides the source (primary radiation) which is used to excite the atoms in the sample. The characteristic fluorescent x-rays of the elements present in the sample enter the detector which registers the individual x-ray events and sends electronic impulses to the

preamp. The preamp amplifies and differentiates the signal and sends it to the digital signal processor (DSP) which shapes and produces the spectrum. The CPU is used to store the algorithms to calculate the concentration of the elements present, and the final results are stored and displayed.

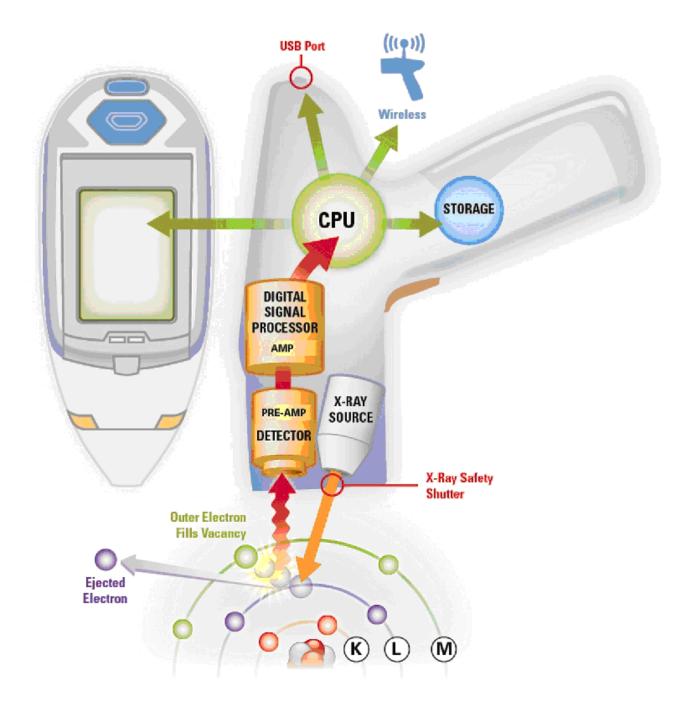


Figure 1. XRF Analysis Diagram

Statistical aspects of spectrometry

The mere mention of the word "statistics" can induce fear into the hearts of many people, however without a basic understanding of the science of statistics; the results from your instrument may be meaningless. Statistics is simply a mathematical way of collecting data, and then analyzing and interpreting it. This brief guide will take you through all the basics of statistics that will enable you to get the best from your instrument, with many worked examples and diagrams to explain the concepts.

Basic Terminology

In statistics, words have very particular meanings, which in the everyday world may seem to be very similar. However, describing an issue using the correct terminology may often result more rapidly in a solution to a problem. The following lists the most common terminology and describes the meaning of each one.

Accuracy can be defined as how close a reading is to the *true value*, and may be indicated using relative error, percent recovery, correlation coefficients or coefficient of determination.

Bell curve represents a normal or Gaussian distribution and describes that shape of the graph obtained, with a peak at the mean. These graphs vary in width indicating the size of sigma.

Bias may be random or systematic, and simply means that results are not as expected. A random error is caused by natural variations, and may be reduced by taking multiple readings and averaging. A systematic bias is generally when all readings are either higher or lower than expected, and average away from the expected (or true) value. This may be caused by a calibration issue, a matrix effect or incorrect assumption of the expected value.

Coefficient of determination (r^2) is the square of the correlation coefficient, with values between 0 and +1, and gives some indication of the goodness of fit.

Correlation coefficient (r) is a determination of the strength of a relationship between 2 variables, with a value of between -1 and +1 inclusive. +1 represents a perfect positive correlation between the variables, -1 represents a perfect negative correlation between variables, and zero (0) represents no correlation between variables.

Certified reference material (CRM) is a standard material that has been certified for certain elements with a given range of uncertainty. Typically analysis has been performed by multiple methods and multiple laboratories.

Empirical rule or *3-sigma rule* states that when considering a distribution that is normal (Gaussian or bell curve), 68% of the values are within 1 standard deviation, 95% are within 2 standard deviations and 99.7% are within 3 standard deviations.

IDL and *MDL* are instrument detection limit and method detection limit respectively. IDL is the best possible limit of detection, calculated from a "clean" sample, i.e. one with no interferences. A method detection limit is the limit of detection for a specific sample type (matrix) using particular instrument conditions. MDLs are typically worse than IDLs by a factor of 2 to 5 times.

Limit of detection (LOD) is the smallest concentration of an element that can be detected with reasonable certainty. It is generally regarded as indicating whether an element is present or not, and does not imply that a value obtained is accurate. LOD is usually calculated using 3 sigma.

Limit of Quantitation (LOQ) is the concentration at which a quantitative measurement can be made, i.e. there is a degree of certainty about the result. LOQ is usually calculated using 10 sigma.

Mean or average is the average of a series of readings from the same sample. It is calculated by taking the sum of all the individual values and dividing by the total number of the analyses performed.

Percent recovery is a measure of how close a read value is to the expected (true) value of the sample. It is calculated by dividing the obtained value by the true value and multiplying by 100. 100% would mean that the reading and the true value agree with one another. 85% would mean that the sample is reading 15% lower than expected, and 122% would mean that the sample is reading 22% higher than expected.

Precision can be defined as the *repeatability* of the results, in other words how close a series of analyses on the same sample are to one another. It may be measured in terms of *sigma* (*standard deviation*) or *relative standard deviation*.

Relative percent difference (RPD) is a numerical comparison of two experimental values. It is calculated by taking one value away from the other, dividing by the average of the 2 values and multiplying by 100.

Relative error is a measure of the difference between the read and the expected (true) value of a sample.

Repeatability is a measure of the variation in results obtained by running the same sample by the same person on the same instrument under identical conditions, generally over a short time period.

Reproducibility is a measure of the variation in results obtained by different people running different instruments. Reproducibility is generally worse that repeatability in absolute terms.

Standard Deviation or sigma is a measure of the variability within a sample set. The smaller the value, the closer all the results are to one another. It is calculated by taking a data set and first calculating the mean. Then the mean is subtracted from each individual reading and the calculated value is squared (this gets rid of any negative numbers). Then these calculated values are averaged and the square root is calculated. This square root is sigma, or one standard deviation. It may also be calculated easily in spreadsheets such as Excel, in Excel use "estdev(highlight cells of interest)".

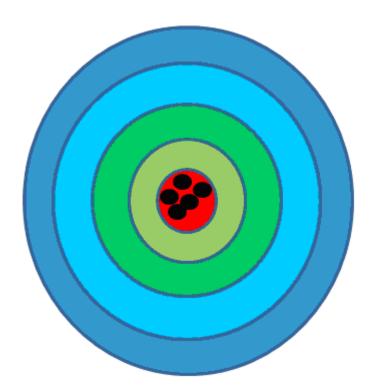
Relative Standard Deviation (RSD) is another way of looking at standard deviation, by relating it to the read value. It is calculated by taking the calculated standard deviation and dividing by the average (mean) value and multiplying by 100. It relates directly to the precision of the readings. For example, a RSD of 1% or less tells us that the data set is very similar to each other, and that we are well above LOD. A RSD of 33% or higher tells us that we are at or below the LOD.

True value is the actual or certified value of a sample.

Comparing accuracy and precision

This is often done visually using targets to demonstrate the differences between them. If we assume we have 5 arrows, and we are aiming at the red bull (center of target), then this diagram (*Target 1*) shows all 5 arrows in the center and so we have good accuracy and they are nicely grouped, so we also have good precision, this is generally what we aim for in terms of analysis, the correct value and all the readings are close to one another.

Figure 2. Target 1:



Target 2 shows that the arrows are all very close to one another, so we have good precision, but we are not close to the center of the target, so we do not have good accuracy, in fact we have a bias. This may be caused by a number of different reasons. For example, we may have a poor calibration, we may have the wrong value for the "true" value (due to a typographical error for example), or we may have a matrix effect for which we have not accounted.

Figure 3. Target 2:

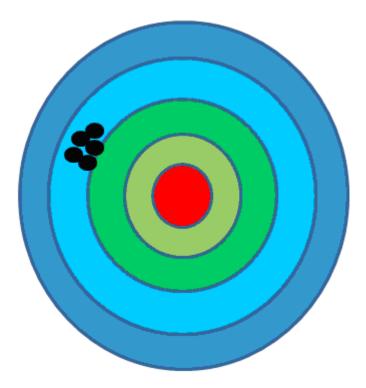


Figure 4. Target 3:

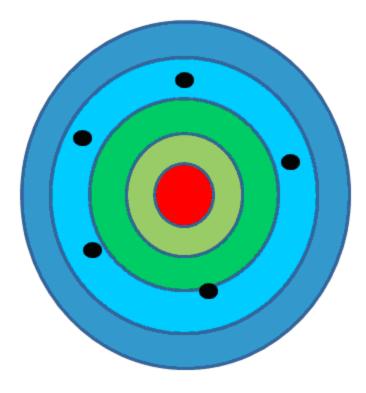
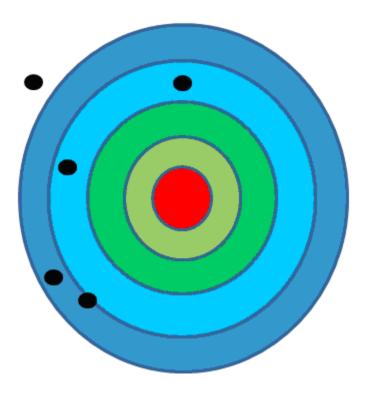


Figure 5. <u>Target 4:</u>



Target 3 shows good accuracy (if we average the numbers, we would end up in the middle), but poor precision. This is typical of when we are reading samples at or close to detection limit, if we average enough numbers we will get close to the correct value, but there is a large amount of scatter about that point.

Target 4 is neither accurate nor precise. This suggests we have a very big problem, there may be an issue with the instrument itself, or this could reflect bad (or no) sample preparation.

Example 1

An ore CRM is read 150 times. It contains 12.29% Ca, and gives us the readings in ppm as shown in *Table 1*. The average for these 150 readings is 128226.6ppm (i.e. 12.82%) and the standard deviation is 2191.5 (or 0.22%).

If we split them into groups, we see that we get 1 at 12.2%, 2 at 12.3%, 7 at 12.4% etc., and we see the typical bell curve forming in *Diagram 1*.

Based on this data, we would expect the following:

68% of all readings should be within 1-sigma. This is the average \pm 1 standard deviation, or in this example 12.82 \pm 0.22, i.e., 12.60 – 13.04%, and we see that we have 99 readings in this range, or 66%, pretty close to what we expect

95% of all readings should be between 12.82 ± 0.44 , or 12.38 - 13.26, and we have 146 readings or 97% in this range, again what we would expect.

The RSD is calculated as 0.22 / 12.82 *100 = 1.71%, which indicates that the readings are well above the LOD, and we can consider them quantitative.

The percent recovery is calculated as read/certified *100, or 12.82/12.29*100 = 104.3%, which is an excellent recovery. Anything between 95 and 105% is excellent ($\pm 5\%$), between 90-110% ($\pm 10\%$) is good, and a $\pm 20\%$ recovery is reasonable.

This data set demonstrates the information shown in *Target 1*, as we have good accuracy and precision.

Example 2

The same ore CRM also contains 28000ppm Cu, and gives us readings as shown in *Table 2*. The average for these readings is 33328ppm, and the standard deviation is 756.

The RSD is 756/28000 *100 = 2.7%, indicating that the readings are well above LOD, and therefore should be considered quantitative. However the percent recovery (33328/28000*100) is 119%, i.e. we are reading on average 19% higher than the certified value.

This data set demonstrates the information shown in *Target 2*, we have good precision and poor accuracy. Another way of considering this data is to plot the points and see where the certified value lies in relation to the readings, as can be seen in *Chart 2*.

Table 1. Table 1

| Ca read |
|----------|----------|----------|----------|----------|----------|----------|----------|
| 127701.7 | 126216.7 | 128323.6 | 126322.9 | 132187.6 | 126242.7 | 128259.1 | 128367.7 |
| 129661.0 | 128028.6 | 130710.2 | 123288.2 | 126605.9 | 130108.8 | 126356.0 | 129188.5 |
| 127413.7 | 126873.4 | 128468.8 | 124019.3 | 129879.8 | 130062.5 | 129645.6 | 129528.2 |
| 130101.6 | 127647.0 | 126533.3 | 127876.5 | 125222.5 | 128412.1 | 130426.1 | 130699.6 |
| 128207.4 | 129473.2 | 128761.3 | 125954.4 | 130795.1 | 127659.9 | 125046.3 | 128550.9 |
| 128943.4 | 122043.0 | 127487.5 | 130581.2 | 127958.8 | 131951.2 | 128050.4 | 132956.9 |
| 127874.1 | 129722.2 | 125640.7 | 126991.1 | 127327.5 | 128798.6 | 132187.3 | 131686.0 |
| 124963.7 | 128726.3 | 129718.2 | 128342.1 | 125764.9 | 128530.4 | 130459.2 | 126944.6 |
| 129684.8 | 131689.9 | 127698.4 | 127018.1 | 125477.3 | 127293.4 | 131384.8 | 125065.2 |
| 127799.3 | 130853.9 | 131691.9 | 124028.2 | 126517.7 | 127524.0 | 130158.7 | 128233.0 |
| 130256.1 | 127769.2 | 125481.0 | 127938.1 | 128243.2 | 128197.0 | 131457.4 | |
| 124723.1 | 128283.3 | 124260.0 | 129318.0 | 126778.4 | 126416.6 | 125649.6 | |
| 127022.5 | 129912.9 | 128582.6 | 127840.3 | 129127.9 | 125589.4 | 124797.8 | |
| 127302.0 | 130708.8 | 128027.9 | 129290.3 | 126987.1 | 128872.3 | 126570.1 | |
| 127383.1 | 125554.5 | 129167.6 | 130543.7 | 130735.8 | 131209.0 | 131537.4 | |
| 125546.1 | 126399.9 | 126199.1 | 129430.6 | 129365.6 | 126396.7 | 130975.4 | |
| 131038.3 | 123862.1 | 126380.1 | 126169.2 | 130026.1 | 131313.1 | 129401.4 | |
| 127713.0 | 125781.3 | 130631.4 | 127463.3 | 129005.6 | 129119.8 | 125967.9 | |
| 129868.9 | 127551.4 | 126474.0 | 130059.8 | 126297.1 | 129359.1 | 128008.1 | |
| 124477.3 | 127715.8 | 132939.1 | 127824.9 | 133425.3 | 127760.8 | 131910.9 | |

Figure 6. <u>Diagram 1</u>

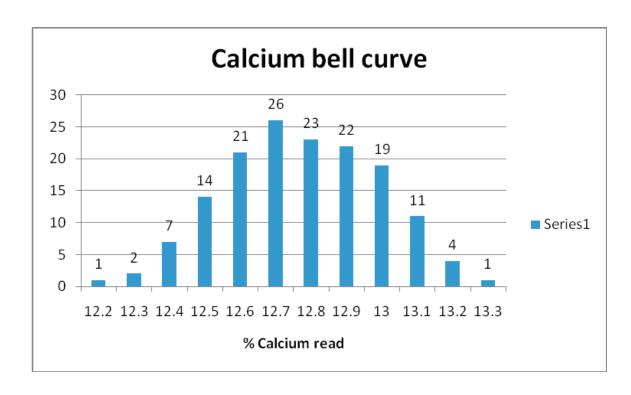
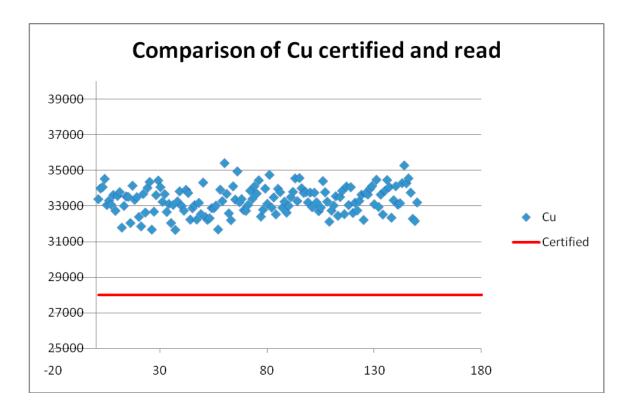


Table 2. Table 2

| Cu read |
|----------|----------|----------|----------|----------|----------|----------|----------|
| 33387.86 | 31856.34 | 32740.38 | 33687.23 | 34756.38 | 32933.31 | 33186.25 | 33076.11 |
| 33991.01 | 33680.85 | 33909.94 | 32589.40 | 32915.60 | 33749.85 | 32749.94 | 33159.21 |
| 34071.70 | 32634.30 | 33736.93 | 32204.39 | 33486.02 | 33210.62 | 33290.37 | 34292.28 |
| 34529.59 | 34025.62 | 32234.62 | 34111.94 | 32534.00 | 32713.45 | 33638.88 | 35284.71 |
| 33059.04 | 34357.05 | 32877.01 | 33365.14 | 33969.77 | 32913.59 | 32216.05 | 34283.20 |
| 33304.52 | 31673.18 | 33051.36 | 34948.63 | 33779.44 | 34403.56 | 33726.88 | 34560.44 |
| 33078.60 | 32682.12 | 32236.69 | 33222.50 | 32907.96 | 33779.25 | 33647.55 | 33753.34 |
| 33622.60 | 33615.13 | 33178.60 | 33392.14 | 33244.81 | 33246.47 | 33979.57 | 32279.92 |
| 32732.53 | 34437.75 | 32542.88 | 32769.55 | 32628.07 | 32118.03 | 34131.02 | 32156.34 |
| 33569.09 | 34063.87 | 34320.18 | 32735.69 | 33010.81 | 32740.08 | 33100.64 | 33202.32 |
| 33786.19 | 33237.50 | 32381.56 | 33066.61 | 33500.42 | 33030.59 | 34481.41 | |
| 31785.92 | 33672.32 | 32223.76 | 33863.22 | 33797.62 | 33550.79 | 32950.11 | |
| 33008.73 | 32669.46 | 32312.26 | 33399.00 | 34557.60 | 32448.79 | 33639.86 | |
| 33540.20 | 33118.25 | 32885.37 | 34117.85 | 33278.59 | 33508.32 | 32520.64 | |
| 33517.70 | 32043.74 | 32879.28 | 33703.34 | 34576.89 | 33859.27 | 33865.25 | |
| 32044.99 | 33063.97 | 33025.60 | 34448.46 | 34003.35 | 32543.92 | 34466.34 | |
| 34145.80 | 31654.85 | 31680.70 | 32404.05 | 33741.77 | 34100.24 | 34072.38 | |
| 33352.85 | 33261.42 | 33919.88 | 32790.72 | 33778.54 | 33053.59 | 32344.51 | |
| 33495.84 | 33827.10 | 33278.86 | 33982.80 | 33205.91 | 34062.01 | 33328.92 | |
| 32390.06 | 32998.97 | 35415.67 | 33122.56 | 33755.98 | 32609.61 | 34113.34 | |

Figure 7. Chart 2



Example 3

The same ore CRM also contains 24ppm Se, and gives us readings as shown in *Table 3*. The average for these readings is 33.8ppm, and the standard deviation is 6.7.

The RSD is 6.7/33.8*100 = 19.8%, indicating that the readings are only just above LOD, and therefore should be considered semi-quantitative at best.

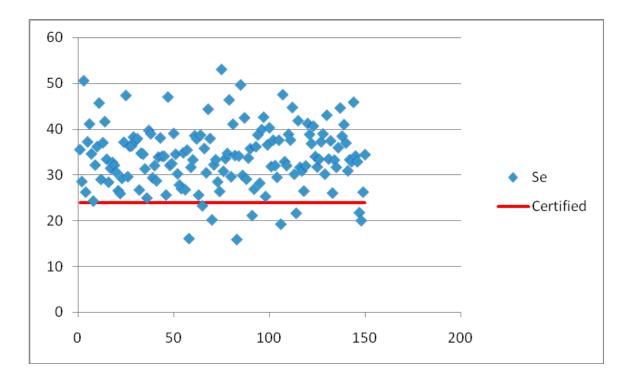
This data set demonstrates the information shown in *Target 3*, we have good accuracy and poor precision. Another way of considering this data is to plot the points and see where the certified value lies in relation to the readings, as can be seen in *Chart 3*. As can be seen, the points are scattered about the certified value (although there is a small positive bias). This demonstrates that in order to obtain the most accurate reading, a large number of readings must be taken. Any one single reading may be quite different from the true value.

It should also be pointed out that the standard error (2 sigma) for these readings is 13, which agrees well with our calculated sd of 6.7 (x 2 = 13.4), and the error readings should always be viewed by the user in order to determine how close you are to the LOD, and therefore give an indication of how variable the results may be.

Table 3. Table 3

| Se read |
|---------|---------|---------|---------|---------|---------|---------|---------|
| 35.51 | 26.52 | 28.69 | 38.54 | 41.08 | 31.82 | 38.82 | 30.88 |
| 28.53 | 25.92 | 33.89 | 37.86 | 34.23 | 37.59 | 36.82 | 33.32 |
| 50.58 | 29.23 | 38.07 | 25.57 | 15.85 | 32.02 | 40.66 | 32.88 |
| 26.21 | 37.12 | 34.07 | 38.69 | 34.11 | 29.45 | 34.00 | 45.88 |
| 37.20 | 47.35 | 34.06 | 23.24 | 49.67 | 37.56 | 31.72 | 34.10 |
| 41.11 | 29.60 | 25.59 | 35.76 | 29.88 | 19.22 | 33.39 | 32.87 |
| 34.60 | 36.25 | 47.01 | 30.45 | 42.47 | 47.54 | 37.21 | 21.73 |
| 24.26 | 36.32 | 32.06 | 44.35 | 29.03 | 32.87 | 38.95 | 19.98 |
| 32.14 | 38.26 | 32.45 | 37.94 | 33.68 | 32.02 | 30.16 | 26.23 |
| 36.17 | 37.45 | 39.05 | 20.15 | 35.72 | 38.86 | 43.04 | 34.42 |
| 45.70 | 37.91 | 34.51 | 32.19 | 21.10 | 37.61 | 33.46 | |
| 29.00 | 26.69 | 30.22 | 33.30 | 26.87 | 44.73 | 37.43 | |
| 37.02 | 34.74 | 27.81 | 28.47 | 36.15 | 30.13 | 26.01 | |
| 41.64 | 34.51 | 26.98 | 26.37 | 38.73 | 21.60 | 33.24 | |
| 33.41 | 31.31 | 34.81 | 53.06 | 28.19 | 41.85 | 31.65 | |
| 28.38 | 24.93 | 26.76 | 30.78 | 39.89 | 31.64 | 35.95 | |
| 31.44 | 39.70 | 35.42 | 33.56 | 42.60 | 30.85 | 44.62 | |
| 32.72 | 38.98 | 16.06 | 34.68 | 25.27 | 26.45 | 38.46 | |
| 32.10 | 29.33 | 31.65 | 46.39 | 36.55 | 31.98 | 40.99 | |
| 30.57 | 32.02 | 33.28 | 29.59 | 40.29 | 41.24 | 36.97 | |

Figure 8. Chart 3



Example 4

One question that is often asked is for how long to run a test, and the answer is for however long it takes to generate the results with enough certainty for an appropriate decision. The longer the integration time, the more precise the result.

Let's consider an example of Pb in soil. A soil sample containing approximately 5000ppm of Pb was analyzed 10 times each at different integration times (10s, 20s, 30s, 60s, 120s and 240s). The results are listed in *Table 4*. There are 2 main things to notice with this data, firstly the shorter the analysis time the greater the spread, so for the 10 second analysis time we go from 4876 – 5759ppm (883ppm spread) while for the 240s analysis time we go from 5271 – 5398ppm (127ppm spread). The spread that is seen is also reflected in the 2 sigma error that is reported for each of the individual tests as seen in the table. Secondly the average obtained from each of the 10 readings is very similar in all cases.

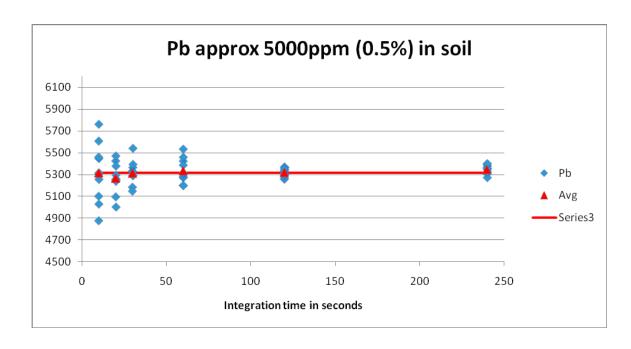
These two points can be seen diagramatically in *Chart 4*. So if we need the results for a 5000ppm Pb sample to have a 2 sigma error of less than 100ppm, we will need to read for 60s to achieve this criterion. But if I am more concerned with obtaining the best average value in the shortest possible time, then I would take 10 10s runs and average the value to obtain an average that will approximate a much longer run instead of taking a single 120s or 240s analysis.

Note that increasing the integration time only improves precision, it does not improve accuracy of the results.

Table 4. Table 4

Duration	Pb	Pb Error	Duration	Pb	Pb Error	Duration	Pb	Pb Error
10.2	5445.9	270.3	30.3	5364.1	126.0	120.4	5346.4	58.7
10.0	4876.5	248.9	30.0	5183.6	123.9	120.0	5316.0	58.6
10.0	5605.3	278.2	30.0	5329.3	127.9	120.0	5331.6	58.9
10.2	5029.0	251.8	30.0	5321.6	126.1	120.3	5364.8	59.0
10.2	5314.3	265.0	30.2	5290.8	126.1	120.1	5256.5	58.0
10.2	5254.6	260.3	30.0	5147.2	123.7	120.4	5302.0	58.5
10.1	5758.9	286.9	30.3	5539.1	130.1	120.1	5266.8	58.2
10.0	5100.9	257.9	30.3	5288.5	125.5	120.1	5368.8	59.3
10.0	5458.5	276.5	30.4	5392.2	128.7	120.1	5292.5	58.2
10.0	5304.9	270.7	30.3	5323.8	126.0	120.0	5350.5	59.0
20.1	5423.6	163.8	60.1	5270.2	81.9	240.1	5315.1	41.5
20.3	5002.0	152.4	60.1	5422.3	83.8	240.3	5355.2	41.9
20.2	5468.9	164.6	60.2	5385.1	83.0	240.3	5306.3	41.5
20.3	5258.9	160.0	60.2	5198.0	80.7	240.3	5377.6	42.1
20.2	5244.8	158.0	60.2	5278.1	81.7	240.3	5343.1	41.7
20.2	5251.0	159.5	60.2	5457.3	83.8	240.3	5271.4	41.3
20.3	5237.7	159.6	60.2	5300.4	81.8	240.2	5360.3	41.8
20.3	5376.5	161.2	60.2	5531.8	84.7	240.3	5322.5	41.6
20.3	5293.9	159.4	60.2	5200.0	80.8	240.2	5395.9	42.1
20.2	5095.2	156.1	60.2	5311.9	82.5	240.3	5398.5	42.2

Figure 9. Chart 4



Example 5

A question that is often asked is how to improve (i.e. reduce) the limit of detection. The LOD is related to error, and x-ray analysis depends on measuring the counts of photons hitting the detector. Therefore the most obvious way to improve the LOD is to get more counts. This is done mechanically by improving detectors (to "see" more), or by improving x-ray tubes (to give more excitation energy). But, most users merely increase the length of the integration time (longer time = more counts). Every time you double the analysis time, the LOD will improve by the $\sqrt{2}$ (or 1.414). If you triple the analysis time, the LOD will improve by $\sqrt{3}$ (or 1.732), if you quadruple the analysis time the LOD will improve by $\sqrt{4}$ (or 2.0), and so on. However, there is a law of diminishing returns (in terms of time) and you can never get to zero!

Let's consider a real example. A soil sample contains 10ppm Co, for how long do I have to integrate in order to be able to detect this concentration? *Table 5* shows the results of reading this sample at a few different integration times. Firstly we can see that our rule of using the square root of the time increase agrees very well with the instrument calculated LODs, for example going from 60s to 120s we would expect the 175ppm LOD to improve by $\sqrt{2}$ (175/1.414=123.8) and the instrument gives us 124pm. Similarly if we go from 60s to 240s we would expect an improvement of $\sqrt{4}$ (175/2 = 87.5) and the instrument reads 88. Essentially we need a ten times improvement of LOD from that obtained at 240s, so we need to run for 100 times longer (10 is the square root of 100) than the 240s, i.e. for 24,000 seconds (400 minutes or 6.66 hours). Clearly this is impractical, so we can never reach 10ppm Co.

Table 5. Table 5

Time	Со	Co err	Co LOD
30s	-62.7	176.8	265
60s	-9.6	116.4	175
120s	-35.2	82.6	124
240s	-55.5	58.8	88

This example demonstrates that we should understand the practical limits of the instrument and not try to exceed them.

Example 6

Accuracy can also be ascertained by looking at the coefficient of determination (r^2). This is done by running numerous standards or well-characterized samples and plotting the known or certified values (x-axis) against the read concentrations (y-axis). For example, a number of materials containing Cu at varying levels was analyzed using the mining mode. The results for these standards are tabulated in *Table 6* and the plot obtained can be seen in *Chart 6a*.

The coefficient of determination (r^2) is shown on the graph as being 0.9983. An r^2 of 1 would be a perfect fit, and this at 0.9983 is excellent. This tells us that the points all plot well on a line (in this case a linear plot), and so the relationship between them is good. This indicates that the calibration is valid for all sample types.

However the coefficient of determination is not the only thing we must observe on the chart, we must also consider the equation itself. The equation is y = 1.0038x + 289.61. This tells us we have a good agreement between the read and certfied values as we have y = 1.0038x. This first value should be very close to 1, if the value is much different then we have a bias. For example, *Chart 6b* shows a similar data series for nickel with an excellent r^2 of 0.9994 (i.e. all points plot well on the straight line), but the equation is y = 0.8647x + 25.428. As y = 0.8647x, this indicates that the reading is only 86% of the certified value, i.e. we have a negative bias on this instrument.

Table 6. Table 6

Sample	Cu ppm certified	Cu ppm read	Cu err	Sample	Cu ppm certified	Cu ppm read	Cu err
SiO2	0	1	32.2	NIST1645	109	226.93	66.92
BCS355	850	479.39	134.55	NIST1646	18	71.39	45.2
NIST113b	2953	2817.11	373.07	NIST1646a	10.01	34.7	37.28
CPB-1	2540	2854.62	416.69	NIST2780	215.5	253.7	58.42
MP-1	20900	21074.01	1055.52	NIST2781	627.4	1274.97	106.05
MRG-1	135	208.15	72.05	NCSDC28024	14	59.33	128.22
PTM1	302400	301525.4	19744.75	NCSDC73306	23	119.01	53.07
NCSDC190 05	65	102.18	62.96	NCSDC73308	22.6	74.47	39.39
NCSDC700 06	93.6	94.95	86.48	NCSDC73309	79	131.83	47.18
NCSDC700 10	140	80.53	68.3	NCSDC73323	144	164.01	55.97
NCSDC700 11	97.4	1	50.86	NCSDC77302	23.2	81.78	40.88
NCSDC735 09	28000	33023.99	1467.3	NCSDC77303	65.4	114.75	51.88
NCSDC735 10	960	1171.82	204.44	170-ES	228.7	380.35	63.79
NCSDC735 13	1380	1540.97	283.36	171-S	17.9	116.86	100.39
AGV1	60	110.98	47.95	172-PS	870.2	1185.28	120.44
CCU-1	247100	246932.9	15293.72	173-S	359.8	411.96	74.74
SU-1a	9670	10769.61	595.71	174-PS	3370.6	4722.92	323.92
RTS-1	595	782.03	116.85	175-S	1064	1629.33	161.2
RTS-2	670	790.29	164.23	176-CCD	1519	2148.84	159.18

RTS-3	2820	3200.3	247.08	177-S	4977.3	7250.36	314.08
RTS-4	280	371.04	175.6	179-S	978.1	1536.75	122.45
CZN-1	1440	1155.58	255.98	185-S	1517	2088.72	150.44
DTS-1	7.1	1	48.5	186-CCD	255	356.62	76.11
DWA1	4	38.98	39.09	189-S	1461.9	2361.88	159.84
PTC1	51600	52750.17	2930.05	191-ES	347.1	379.91	69.95
SO-1	61	150.45	51.93	192-ES	102.3	130.64	45.83
TILL-1	47	97.5	44.07	193-S	1173	1710.95	131.44
FER-2	45	39.16	69.73	195-S	1017.8	1514.52	129.88
G2	11	49.95	40.18	196-CCD	618	765.84	108.39
PACS-2	210	486.1	72.41	197-S	664	1166.79	121.17
SY-2	5	56.18	47.09	199S	154.7	183.69	54.76
TILL-2	150	278.58	57.69	200ES	91	158.06	45.68
CZN-3	6850	6590.49	565.4	201-S	673.5	1022.73	97.53
MA-3a	100	163.54	55.63	202-PS	919.3	1360.74	145.68
MESS-3	33.9	87.5	46.55	205-S	2522.2	4346.04	242.75
PI 3.12	14	92.78	130.56	207-S	1588	2303.46	167.76
IGS-21	7980	8556.18	531.84	209-S	2633.5	4505.38	243.95
NIST57a	40	92.47	52.11	212-PS	536.4	767.41	97.89
NIST330	8400	9194.91	380.03	T-215S	42.4	117.25	45.35
NIST331	910	1242.44	103.34	219-S	1543.5	2266.31	266.29
JSAC 0403	26.2	76.16	42.38	220-ES	36.6	77.38	38.98
BAM529-1	100	189.69	64.35	221-S	281.2	487.74	68.15
NIST691	320	290.04	223.49	222-PS	871.4	1162.97	116.15
NIST1633	128	257	63.85	225-S	44.5	101.86	43.57
NIST1633a	118	238.69	64.58	226-HD	315.6	608.84	72.56
228-ES	101.9	133.47	45.7	227-S	228.4	252.75	54.28
229-S	16.7	49.28	39.05				

Figure 10. Chart 6a

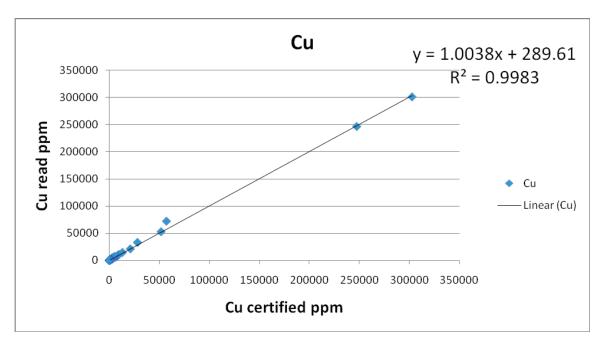
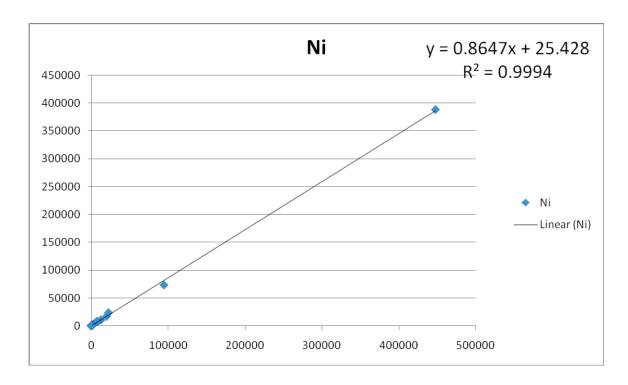


Figure 11. Chart 6b



Note

Your instrument was supplied with various reference materials (depending on the calibrations you have on your instrument) so that you can confirm its' analytical performance. It is recommended that you run these on a daily basis and keep a record of your results, that way you can easily confirm that your instrument is running at its optimal performance.

But if you are purchasing additional standards from other vendors beware, not all standards are created equally. Standards are manufactured for specific tasks, and may, or may not, be suitable for reading on your XRF. Always read the certificate carefully before analysis. For example many soil standards are certified for multiple elements as can be seen in the partial *Certificate* shown below, which shows part of a certificate of analysis for a natural matrix CRM.

Figure 12. Certificate

METALS ON SOIL

ANALYTE CONCENTRATIONS

<u>Element</u>	Reference Value	<u>S. D.</u>	Confidence Interval	Prediction Interval
Aluminum, Al	7,637	1,602	6,900 - 8,375	4,216 - 11,059
Antimony, Sb	(<3.2)			
Arsenic, As	339	51.1	319 - 360	232 - 447
Barium, Ba	1,839	1,108	1,411 - 2,266	0.00 - 4,177
Beryllium, Be	0.33	0.14	0.27 - 0.40	0.04 - 0.63
Boron, B	(17.2)			
Cadmium, Cd	369	46.3	350 - 388	271 - 466
Calcium, Ca	28,320	4,541	26,419 - 30,221	18,734 - 37,906
Chromium, Cr	441	50.1	419 - 463	335 - 547
Cobalt, Co	4.07	0.93	3.56 - 4.57	2.03 - 6.10
Copper, Cu	7.76	1.68	7.03 - 8.48	4.21 - 11.3
Iron, Fe	9,439	1,229	8,922 - 9,955	6,850 - 12,027
Lead, Pb	1,447	203	1,359 - 1,536	1,017 - 1,878
Magnesium, Mg	4,376	508	4,168 - 4,584	3,305 - 5,447
Manganese, Mn	173	15.0	167 - 180	141 - 205
Mercury, Hg	99.8	31.7	81.9 - 118	31.3 - 168
Molybdenum, Mo	(<0.8)			
Nickel, Ni	12.2	3.54	10.7 - 13.6	4.69 - 19.6
Potassium, K	1,992	442	1,793 - 2,191	1,050 - 2,934
Selenium, Se	518	74.0	487 - 549	362 - 674
Silicon, Si	171	65.0	106 - 236	0.00 - 366
Silver, Ag	132	82.6	95.5 - 167	0.00 - 306
Sodium, Na	313	50.0	286 - 339	205 - 420
Strontium, Sr	(408)			
Thallium, Tl	(<4.8)			
Vanadium, V	`19.3 [´]	4.48	17.4 - 21.3	9.83 - 28.9
Zinc, Zn	51.8	8.29	48.4 - 55.1	34.3 - 69.2

However, when using XRF to analyze this sample, the Cr reads consistently high, approximately 1700ppm. There is clearly a positive bias, but what is causing it? The answer lies in the fine print on the certificate of analysis, excerpts of which state:

"...The reference values were determined by USEPA SW846 (3rd edition) Methods 3050 and 6010,... The sample is suitable for other 3000-series metal digestion method procedures and 7000-series 9000-series spectroscopic methods."

Method 3050B is an acid digestion of sediments, sludges and soils for AAS, ICP, GFAAS and ICPMS . This method is not a total digestion method, it will only dissolve elements that could become environmentally available.

Any comparison of XRF values with a sample that has been analyzed after a partial digestion method is prone to have this type of bias, the XRF will typically always read higher.

The same is true when looking at extractable-type samples, for example, landfill samples run under TCLP (USA), or migration of certain elements in toys using EN 71 Part 3 (EU).

It should be noted that extraction rates of elements vary considerably from sample to sample, so it is not practical to apply a "correction factor" to adjust the XRF results to agree with another analytical technique using partial digestion or extraction methods.

Reference Documents

Service

See "Contact Us" on page 1 to find your nearest Service facility.

Warranty

Warranty statement

Seller warrants that the Products will operate or perform substantially in conformance with Seller's published specifications and be free from defects in material and workmanship, when subjected to normal, proper and intended usage by properly trained personnel, for the period of time set forth in the product documentation, published specifications or package inserts. If a period of time is not specified in Seller's product documentation, published specifications or package inserts, the warranty period shall be one (1) year (unless otherwise agreed upon at time of purchase) from the date of shipment to Buyer in the country of purchase. Any part replaced on an instrument, covered by the original factory warranty, will be warranted for the remainder of the instrument's factory warranty. Seller agrees during the Warranty Period, to repair or replace, at Seller's option, defective Products so as to cause the same to operate in substantial conformance with said published specifications; provided that Buyer shall (a) promptly notify Seller in writing upon the discovery of any defect, which notice shall include the product model and serial number (if applicable) and details of the warranty claim; and (b) after Seller's review, Seller will provide Buyer with service data and/or a Return Material Authorization ("RMA"), which may include biohazard or other Radiation safety decontamination procedures and other product-specific handling instructions, then, if applicable, Buyer may return the defective Products to Seller with all costs prepaid by Buyer. Replacement parts may be new or refurbished, at the election of Seller, the warranty of these parts expire with the instrument warranty. All replaced parts shall become the property of Seller. Shipment to Buyer of repaired or replacement Products shall be made in accordance with the Delivery provisions of the Seller's Terms and Conditions of Sale. Accessories and Consumables are expressly excluded from this warranty (see list A for details).

Notwithstanding the foregoing, Products supplied by Seller that are obtained by Seller from an original manufacturer or third party supplier are not warranted by Seller, but Seller agrees to assign to Buyer any warranty rights in such Product that Seller may have from the original manufacturer or third party supplier, to the extent such assignment is allowed by such original manufacturer or third party supplier.

In no event shall Seller have any obligation to make repairs, replacements or corrections required, in whole or in part, as the result of (i) normal wear and tear, (ii) accident, disaster or event of force majeure, (iii) misuse, fault or negligence of or by Buyer, (iv) use of the Products in a manner for which they were not designed, (v) causes external to the Products such as, but not limited to, power failure or electrical power surges, (vi) improper storage and handling of the Products, (vii) use of the Products in combination with equipment or software not supplied by Seller, (viii) Moderately heavy or excessive impact against any object, including but not limited to floors, walls, furniture, sample or other objects. A shock sensor is fitted inside of the instrumentation; warranty is void if this shock sensor is activated, (ix) Excessive water, moisture or condensing humidity that breaches the instrument seals, (X) Excessive or extreme ambient or direct temperature or (vi) Heavy vibrations directly to the instrument for extended periods of time. If Seller determines that Products for which Buyer has requested warranty services are not covered by the warranty hereunder, Buyer shall pay or reimburse Seller for all costs of investigating and responding to such request at Seller's then prevailing time and materials rates. If Seller provides repair services or replacement parts that are not covered by this warranty, Buyer shall pay Seller therefore at Seller's then prevailing time and materials rates.

ANY INSTALLATION, MAINTENANCE, REPAIR, SERVICE, RELOCATION OR ALTERATION TO OR OF, OR OTHER TAMPERING WITH, THE PRODUCTS PERFORMED BY ANY PERSON OR ENTITY OTHER THAN SELLER WITHOUT SELLER'S PRIOR WRITTEN APPROVAL, OR ANY USE OF REPLACEMENT PARTS NOT SUPPLIED BY SELLER, SHALL IMMEDIATELY VOID AND CANCEL ALL WARRANTIES WITH RESPECT TO THE AFFECTED PRODUCTS.

THE OBLIGATIONS CREATED BY THIS WARRANTY STATEMENT TO REPAIR OR REPLACE A DEFECTIVE PRODUCT SHALL BE THE SOLE REMEDY OF BUYER IN THE EVENT OF A DEFECTIVE PRODUCT. EXCEPT AS EXPRESSLY PROVIDED IN THIS WARRANTY STATEMENT, SELLER DISCLAIMS ALL OTHER WARRANTIES, WHETHER EXPRESS OR IMPLIED, ORAL OR WRITTEN, WITH RESPECT TO THE PRODUCTS AND INCLUDING WITHOUT LIMITATION ALL IMPLIED WARRANTIES OF MERCHANTABILITY OR FITNESS FOR ANY PARTICULAR PURPOSE. SELLER DOES NOT WARRANT THAT THE PRODUCTS ARE ERROR-FREE OR WILL ACCOMPLISH ANY PARTICULAR RESULT.

Accessories, Spares and Consumables - exclusions

(List A)

Specific warranties of some common accessories:

- Battery Charger and batteries 12 months
- Instrument accessories 12 months
- Consumable no warranty

- Soil Grinder no warranty
- Single-stage or two stage helium tank regulator 12 months
- Test stands, extend-a-poles and docking stations 12 months
- Parts or spares sold, installed or supplied outside of the product warranty period and not listed above – 12 months

19

Warranty