# UiO : Naturhistorisk museum

# Niton XL3t GOLDD<sup>+</sup> Portable XRF Analyzer

User instructions





# WARNING : risk of exposure to radiation

DO NOT use this equipment without training



Created by Eirini Zacharaki

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# Geo Lab

Department of technical and scientific conservation | Seksjon for konservering og forskningsteknikk

Portable X-ray fluorescence analyzer
Niton XL3t GOLDD<sup>+</sup> Portable XRF Analyzer

# Contact | Kontakt oss

Lab manager	Laboratorieleder	
Nélia Castro	tlf: 228 51641*	e-mail: nelia.castro@nhm.uio.no

#### Professor

Øyvind Hammer tlf: 228 51658\* e-mail: oyvind.hammer@nhm.uio.no

\* If you call from the laboratory telephone, digit the last 5 digits of the number only. Exp: Nélia Castro - 51641

# Contents

1.	Introduction	2
2.	Hygiene and safety in the laboratory	2
2.1.	Emergency contacts	2
2.2.	Safety summary for Niton XL3t GOLDD <sup>+</sup> Analyzer	3
2.3.	Radiation safety for Niton XL3t GOLDD <sup>+</sup> Analyzer	3
3.	Configuration	6
4.	Sample preparation	7
5.	Operating instructions for analyze of Geological Samples	7
5.1.	Startup	7
5.2.	Perform System Check	8
5.3.	Select Mining Cu/Zn Mode	9
5.4.	Verify and/or change filter position time	10
5.5.	Performing a Measurement on hand specimens	11
5.6.	Download data from the Niton XL3t GOLDD <sup>+</sup> Analyzer	11
5.6.1.	Installing the NDT software and USB driver	11
5.6.1.	Downloading data	12
5.7.	Shut down the Niton XL3t GOLDD <sup>+</sup> Analyzer	13
5.8.	Recharging the Battery Pack	13

### 1. Introduction

The Norwegian Centre for Paleontology at the Natural History Museum is equipped with a Niton XL3t GOLDD<sup>+</sup> Analyzer, manufactured by Thermo Fisher Scientific Inc. The Niton XL3t GOLDD<sup>+</sup> is a hand-held X-ray Fluorescence (XRF) elemental analyzer, optimized for analyze of geological samples, designed for portable use, both in the lab and in the field. It provides high count rates, lower detection limits, and the ability to measure light elements (Mg, Al, Si, P and S) without helium or vacuum assistance, which results in fast non-destructive analysis. We will sometimes refer to it as "the portable XRF analyzer" in this document.

Operation of this equipment is restricted to trained personnel familiar with the analyze system as well as the product documentation, general safety precautions, radiation safety precautions, and laboratory rules. New users need to undergo training before they are allowed to work with this equipment. Note that there is risk of exposure to radiation during the use of this equipment. **DO NOT use the equipment prior to receiving radiation safety training.** Contact the laboratory manager or deputy if you want to be trained to operate this equipment.

# 2. Hygiene and safety in the laboratory

#### 2.1. Emergency contacts

Fire: **110** Ambulance: **113** Police: **112** 

In case of **unexpected incidents** during the use of the equipment, contact immediately one of the persons listed below.

#### Lab manager, Nélia Castro

tlf: 228 51641 e-mail: nelia.castro@nhm.uio.no

#### Professor, Øyvind Hammer

tlf: 228 51658 e-mail: oyvind.hammer@nhm.uio.no

# 2.2. Safety summary for Niton XL3t GOLDD<sup>+</sup> Analyzer

Personal injuries can result from the inappropriate use of the equipment. The following warnings apply to the use of the portable XRF analyzer:

WARNING	Consequences	Safety measures
Beware of radiation!	Exposure to radiation, including direct tissue damage (e.g. skin burns).	Never aim the primary beam at yourself or others! Never handle samples during analysis!

The following cautions should be taken when using the Niton XL3t GOLDD+ Analyzer:				
Action	Consequences	Safety measures		
Holding the analyzer	Foot/leg injury	When holding the analyzer, a		

Foot/leg injury	When holding the analyzer, always use		
Equipment damage (e.g. dropping the instrument).	the wrist strap.		
Equipment damage	Always transport the analyzer in its padded carrying case, and store it in its case whenever it is not being used.		
Equipment damage	Always remove the battery pack when transporting or storing your analyzer.		
	Foot/leg injury Equipment damage (e.g. dropping the instrument). Equipment damage Equipment damage		

# 2.3. Radiation safety for Niton XL3t GOLDD<sup>+</sup> Analyzer

The portable XRF analyzer has an X-ray tube that emits radiation when the instrument is turned on, the password is given, and the mode of analysis is selected. In other words, radiation is emitted during a measurement.

The primary beam is a directed beam out of the front of the analyzer that has high dose rates of radiation (Figure 1, beam in red). The secondary beam, or scattered beam, has much lower dose rates of radiation (Figure 1, beam in yellow).



Figure 1: Primary (red) and Secondary (yellow) beam.

The shutter-open indicator lights are flashing when the primary beam is on, Figure 2.



Figure 2: The Shutter-Open indicator lights.

Exposure to radiation represents risks to the human health. In order to ensure your safety and of those around you please follow these precautions during the use of the portable XRF analyzer:

1) Follow the ALARA radiation safety principle. ALARA is an acronym for "As Low As Reasonably Achievable". The three major principles to assist with maintaining doses "As Low As Reasonably Achievable" are time, distance and shielding.



**Time:** Reducing the time of exposure can directly reduce radiation dose.

**Distance:** Increasing the distance between you and the radiation source you will reduce exposure by the square of the distance. Doubling the distance between your body and the radiation source will divide the radiation exposure by a factor of 4.

Shielding: Lead or lead equivalent shielding for X-rays and gamma rays is an effective way to reduce radiation exposure.

2) Never point the instrument at yourself or other.



- 3) Never look into the primary beam.
- 4) Avoid holding the front of the analyzer when the X-ray tube is energized and the shutter is on.

5) Do not hold the samples during measurement.

Appropriate positioning of the samples is required prior to the measurement.

There should always be a sample in contact with the measurement window when the X-ray tube is on.

6) When sitting or standing with your feet below the work surface, the shielding effectiveness of the work surface should be evaluated and increased as necessary. A typical approach is to affix a sheet of lead, tin, copper, steel, or other heavy metal to the top or underside of the work surface.



EASY SAFE SOLUTION

7) If you are frequently handling low density samples, you should consider the use of test stands, backscatter shields, or the equivalent.

Before start using the portable XRF analyzer, please take the online safety training course, titled Radiation Safety for X-ray Tube-based Instruments, available for free in the following link:

<u>https://www.thermofisher.com/no/en/home/industrial/spectroscopy-elemental-isotope-analysis/portable-analysis-material-id/xrf-radiation-safety-training.html</u>

CHALLENGE

# 3. Configuration

The Niton XL3t GOLDD<sup>+</sup> Analyzer is a single unit, hand-held, portable X-ray fluorescence (XRF) elemental analyzer, designed for use both in the lab and in the field. An overview of the portable XRF analyzer is given in Figure 3.



Figure 3: Analyzer overview.

The portable XRF analyzer may be operated on rechargeable Li-batteries (battery pack). It is delivered with two battery packs. When fully charged, the battery packs provide approximately 6–8 hours of use. Recharging of one battery pack takes 2–3 hours (see instructions in section 5.8).

Although the instrument is robust, designed for use in the field, the transparent film of the measurement window (Fig. 3) is very thin and sensitive to physical damage. If the membrane of the measurement window becomes frayed, ripped, or contaminated with particulate matter, it has to be replaced. DO NOT use the equipment in these conditions. Contact the lab manager or deputy for help.

# 4. Sample preparation

The Niton XL3t GOLDD<sup>+</sup> Analyzer can analyze samples in situ with no previous sample preparation. However, sample preparation can increase accuracy of data.

Contact the lab manager or deputy if you need more information about sample preparation.

#### 5. Operating instructions for analyze of Geological Samples

#### 5.1. Startup

Insert a charged battery into the analyzer and turn it on by pressing and holding the *On/off/escape button* on the control panel until the *LCD touch screen* powers on (Fig. 4). (Instructions to battery installation and charging in section 5.8)



Figure 4: The control panel.

2) Follow the screen instructions to log on. When the startup is completed, the *Start Screen* will be replaced by the *Logon Screen*. Tap anywhere on this screen to continue. The *Logon Screen* will be replaced by a *Warning Screen (Fig. 5)*, 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. Enter the *password (1234)*, followed by the *Enter button (Fig. 6)*.

Warning	Enter Password			
Caution Radiation	7	8	9	
THIS EQUIPMENT PRODUCES RADIATION WHEN LIGHTS DRE FLASHING	4	5	6	
AKE EBASHING.	1	2	3	
Press YES to proceed.	0	Bac	ckspace	
Press NO to log out.	Clea		Enter	
Yes No	1234			

Figure 5: Warning Screen

Figure 6: Logon Screen

- 3) Wait five (5) minutes before using the analyzer, allowing the instrument electronics to stabilize.
- 4) Check the date/time. The time should be set correctly for accurate and verifiable record keeping. From the *Main Menu*, select the *System icon* then the *Specs icon*.

**NOTE:** The portable XRF analyzer can be stored and operated safely in temperatures from -5° to +50°C. You will not be able to take a measurement if the analyzer overheats. If it is hot to the touch, allow it to cool before testing.

# 5.2. Perform System Check

1) Select the *System Check Icon* on the *Main Menu* to perform a system check (Fig. 7). Do this once every working day, as part of your normal startup routine.



Figure 7: How to perform a system check.

2) While performing the system check, your screen will show a progress bar indicating the progress of the calibration. When the system check is complete, *"System OK"* appears on the screen. Select *Close* to return to the main menu (Fig. 8).

System Check
System OK
Close

Figure 8: Completing the system check.

NOTE: If you see any result other than "System OK", perform another system check. If the result is still not "System OK", please notify the lab manager or deputy and DO NOT use the equipment.

# 5.3. Select Mining Cu/Zn Mode

1) From the *Main Menu*, select the *Sample Type icon*, then the *Soil & Minerals icon*, then the *Mining Cu/Zn icon* (Fig. 9).



Figure 9: How to select the operation mode.

NOTE: Mining Cu/Zn mode is the most used analyze mode at the Geo Lab, since it covers the range of elements and concentrations more often found in geological samples. This mode of operation is optimum for any sample whose elements of interest are present at 0.5% (5000 ppm) or greater.

Concentrations of the following elements can be determined: Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Rb, Sr, Zr, Nb, Mo, Pd, Ag, Cd, Sn, Sb, Ba, W, Au, Pb and Bi.

All concentrates are displayed in wt. % by default, but can be changed to ppm.

<u>Detection Limit</u>. For an element to be analyzed, the measured concentration of the sample must be at least three times the standard deviation of the measurement. The detection limit depends on the composition of the sample.

<u>Precision</u>. The precision of the measurement is two times the standard deviation (sigma), which corresponds to 95% confidence. An element is classified as 'detected' if the measurement concentration (in ppm) is at least 1.5 times the precision.

<u>Quantitative results</u>. A measurement result is considered 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. The error of a single measurement is highly dependent on sample density and measurement time.

<u>When not to use Mining Cu/Zn mode</u>: There are direct overlaps between the spectral peaks for Cu and Ta and Zn and 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.

For further information, consult the instrument manual or contact the lab manager or deputy for help.

# 5.4. Verify and/or change filter position and time

The Niton XL3t GOLDD<sup>+</sup> Analyzer is equipped with excitation filters that optimize the analyzer's sensitivity for various elements. The <u>High range</u> filter is used to optimize barium (Ba) through silver (Ag). The <u>Main range</u> filter provides optimum sensitivity from manganese (Mn) through bismuth (Bi). The <u>Low range</u> filter is used to optimize from titanium (Ti) through chromium (Cr). The <u>Light</u> range filter is typically used in light element analysis (down to magnesium (Mg)).

At the Geo Lab we typically select all filters and set values of 30 sec for each filter, which means that each measurement takes about 2 min to be completed.

If you want to deviate from these settings, please follow the instructions:

1. From the *Main Menu* select the *Advanced icon*, then the *Element Range icon*.

2. Select the box next to the filters you want to select and set the desired length of time (in sec) for each filter. Select the *Save button* and return to the *Main Menu* (Fig. 10).

Advanced Settings			Adjust Ele	ment Range	3 <b></b>	]	Element Ra	nge
							Mode	
				NST N	AL		Mining Mode	<b>T</b>
	Č			S SLBr K	Zr 🔶			Time
Sample			Data	Element	Benent		🤶 🔽 Main Range	30.0
Analyze Type	System		Return	Itampe	Sorting		? 🗸 Low Range	30.0
1 298 👔	Sel.		x <pb<y< td=""><td>%</td><td></td><td></td><td>2 Vich Pance</td><td>20.0</td></pb<y<>	%			2 Vich Pance	20.0
111588 Au		,		ppm			N N NIGH Kange	30.0
Data Idvanced	System Check		Element Thresholds	Display Units	Start/Stop Setup			
			1		Ĭ,			
Lagolf			Consumer Goods	Safety	Adjust Calibration		Save	2

Figure 10: How to verify and/or change filter position and time.

# 5.5. Performing a Measurement on hand specimens

- 1) Determine whether to average multiple readings or to take one measurement.
- 2) Hold the portable XRF analyzer by the handle, ensuring the wrist strap is properly placed on the wrist.
- 3) Provide a clean and flat surface of sample for analysis. Position the sample so that the analyzer may be safely operated (see Section 2.3). Place the front end of the analyzer on the desired analysis spot, ensuring that the nose of the analyzer is touching the sample. *NOTE: The sample must cover the analysis window.*
- 4) Start a reading by pressing and holding the trigger. Continue the reading for the predetermined measurement time. *NOTE: The four LED lights will flash during measurement (Fig. 2)*.
- 5) Stop the reading when measurement is complete by letting go of the trigger.
- 6) Repeat for averaging or choose a new sample and repeat.

# 5.6. Download data from the Niton XL3t GOLDD<sup>+</sup> Analyzer

Download of data from the portable XRF is done using the NDT (Niton Data Transfer) software.

#### 5.6.1. Installing the NDT software and USB driver

The installation file and driver can be found in the following link:

https://www.dropbox.com/s/mu8jclig5ljqts7/Thermo%20Fisher%20Scientific%208.4.zip?dl=0

#### Or in the folder:

nhm-z-fritt on lagrinsgshotell (Q:)\Økern-GeoLab\Software\pXRF

- 1) Click in *NDTSetup.exe* to start installation and follow the instructions on the screen to complete the installation.
- 2) After the installation you receive a request on screen to register the analyzer. This is not necessary. Click *Ignore* and then *Finish*.

The program is now installed but you need to install the driver manually.

- 1) Turn on and log in to the portable XRF analyzer.
- 2) Connect it to your computer via USB.
- 3) Open the *Control Panel* in your PC, right click in the *Device Manager* and open it as administrator.
- 4) Click in *Ports (COM & LPT)*, open the *USB Serial Device*, go to the tab *Driver* and click *Update Driver*.
- 5) You will be asked how you want to look for the program drive. Choose *Look in the Computer for the Program Drive*. Click in *Browse* to look for the folder where the driver is (see above).
- 6) Follow the screen instruction to install the *Thermo NITON USB Port*.

Program and drive are now installed.

#### 5.6.1. Downloading data

- 1) Open the NDT program
- 2) Click Download and choose the "USB Serial Device" port. Click OK and then Connect (Fig. 11).



Figure 11: Using the DNT program to download data from the portable XRF analyzer.

- 3) Click *Query Readings* and select the data you want to download.
- 4) Select the *Destination Folder* and *File Name*.
- 5) Click Download.

NOTE: Once you saved your data to your computer, please delete it from the portable XRF analyzer, so the analyzer has enough memory available for the next user.

### 5.7. Shut down the Niton XL3t GOLDD<sup>+</sup> Analyzer

- 1) Push and hold the On/off/escape button for about 10 seconds to shut off power to the analyzer.
- 2) Inspect the condition of the transparent film of the measurement window. Clean from particulate matter by blowing on it.
- 3) Remove the battery pack.
- 4) Charge the battery pack (Section 5.8), so the equipment is ready to use at any time.
- 5) Store the analyzer and battery packs in its padded carrying case.

#### 5.8. Recharging the Battery Pack

- 1) Remove the battery pack from the analyzer by sliding back the catch on the bottom of your analyzer's pistol grip and dropping the battery out into your hand.
- 2) Connect the charger to the wall outlet.
- 3) Place the battery pack upside down into the charger. Make sure that the key shoulder is in front and the thin side is down (Fig. 12).



Figure 12: Rear and side views of battery pack showing key.

4) Inspect the charger's indicator lights: *red*, indicates that the charger's power is on; *yellow*, indicates that the battery pack is charging; *green*, indicates that the battery pack is fully charged.

NOTE: Do not leave the battery pack connected to the charger for excessive periods of time after it is fully charged.