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User Guide

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LAB-X5000
Benchtop XRF Analyzer

Hitachi High-Tech Analytical Science

- Before using the instrument, read the safety instructions and precautions carefully.
- Be sure to observe the safety instructions in this manual and the WARNING/CAUTION labels on the instrument.
- Keep this manual in a safe place nearby so it can be referred to whenever needed.

NOTICE:

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3. Hitachi High-Tech Analytical Science assumes no liability for any direct, indirect, or consequential damages arising from use not described in this manual.
   Utmost care must be exercised when using the instrument.
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Introduction

The LAB-X5000 is a benchtop elemental analysis instrument based on non-destructive energy dispersive X-ray fluorescence technology. It is designed to measure a range of materials including solids, powders, gels and liquids in seconds or minutes. The LAB-X5000 is available in several hardware configurations that are optimized for specific applications. In order to get the best performance it is necessary to create an empirical calibration curve derived from standards with known values.

Please note that this manual provides general instructions on typical use of the instrument. Operators should be properly trained in the use of the instrument. Please consult with your local Hitachi High-Tech Analytical Science representative to arrange for necessary training.

The contents of this manual are subject to change without notice. Hitachi High-Tech Analytical Science is not responsible for damage that may result from the use of misue of this document.

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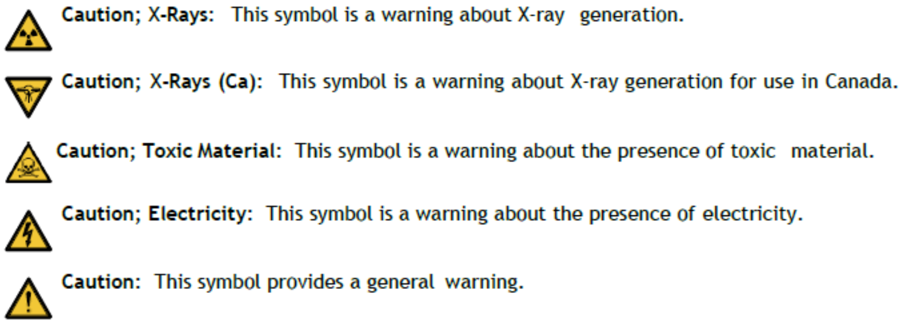
1. Safety Information

The Hitachi LAB-X5000 is a non-destructive bulk composition measurement instrument based on the principles of X-Ray fluorescence. The equipment provides safe, non-contact measurement of a wide variety of elements in multiple material types, including solids, liquids, powders, pastes, granules, films, filter papers, etc. During normal operation there are no accessible radiation dose levels above 0.1 µSv/hr (0.01 mrem per hour). The unit can be operated like any other piece of laboratory equipment, and users do not need to wear personal radiation dosimeters.

If the Lab-X5000 is used in a manner not specfified by Hitachi High-Tech Analytical Science, the safety features can be impaired.

1.1. Warning Symbols on Labels

These symbols appear in LAB-X5000 device and in the documentation.



Caution; X-Rays: This symbol is a warning about X-ray generation.

Caution; X-Rays (Ca): This symbol is a warning about X-ray generation for use in Canada.

Caution; Toxic Material: This symbol is a warning about the presence of toxic material.

Caution; Electricity: This symbol is a warning about the presence of electricity.

Caution: This symbol provides a general warning.

1.2. Radiation Safety

1.2.1. **The LabX-5000 contains a 30kV, 3W X-ray tube housed in a fully-shielded and interlocked radiation enclosure. In normal operation there is no radiation detectable above background level on any accessible surface.**

1.2.2. **On the LAB-X5000 at 100mm from any surface the radiation dosage is at background level (EU requirement: <1mS/h@100mm).**

1.2.3. **It is the owner's responsibility to ensure that local radiation guidelines and precautions are followed. Radiation monitoring, warning signs and other precautions may be necessary.**

1.2.3.1. The user is safely shielded from any radiation exposure from the X-ray tube and the test chamber.

1.2.3.2. Eight warning lights on the side corners of the instrument, as well as the software interface, indicate when radiation is being generated inside the test chamber.

1.2.3.3. If any of the interlocks fires, the power to the X-ray tube is immediately switched off.

1.2.3.4. Every instrument is tested for leakage at 17 points around the instrument prior to shipment.

NOTIFICATION: Use of ionizing radiation equipment is regulated and licensed in most countries, and many states. Check with your local regulators or Hitachi High-Tech Analytical Science representative to ensure that all applicable rules and regulations are met.

1.3. Safety Systems

1.3.1. Instrument is designed such that no access to the test chamber is possible while X-rays are being generated.

1.3.2. Each corner of the instrument has two orange indicators that light up when the X-rays are switched on. They are located on the corners and are labeled in both English and French: “X-RAYS ON” and “RAYONS X”

1.3.3. X-rays are automatically switched on and off by the instrument. X-rays will be switched on at the beginning of an analysis and switched off at the end of an analysis.

1.3.3.1. The upper button ring will glow red when X-rays are switched on.

1.3.3.2. The upper ring will be unlit when X-rays are switched off.

1.3.4. The operator must have the correct PIN to operate the instrument.

1.3.4.1. There are three levels of access for users. Access for each level has a unique PIN, and any PIN can be changed by the Manager.

1.3.4.2. It is simple to switch between access levels by accessing the dropdown menu and tapping Log Out. Enter the PIN for the desired appropriate access level.

1.3.4.3. Details of menu access at each login level is available in Appendix A

1.3.5. Annual Service should be performed by a Hitachi High-Tech Analytical Science Qualified Service engineers.

☞ **Information**

Routine radiation leakage surveys may be required by the state or country. See the governing body’s regulations or the agency.

1.4. Electrical Safety and Electromagnetic Compatibility

1.4.1. The LAB-X5000 unit contains high voltages. It is not necessary to remove the cover and the instrument should never be operated with its outer case removed. Service work should only be carried out by Hitachi High-Tech Analytical Science Qualified Service engineers.

1.4.2. The LAB-X5000 has been designed in accordance with IEC61010 (‘Safety requirements for electrical equipment for measurement, control and laboratory use, Part 1’) which encompasses the European Low Voltage Directive and IEC 61326-1 (‘EMC requirements for measurement, control and laboratory use, Part 1’) which encompasses the European EMC directive. The instrument must be installed correctly and used only for the purposes described in this manual. If you have any doubts about the installation or use of this instrument, contact either Hitachi Instruments or your local Hitachi High-Tech Analytical Science representative.

1.5. Beryllium precautions

The windows of detectors and X-ray tubes have thin beryllium window. Beryllium is a toxic compound; however, the beryllium window poses no health hazard when it is intact.

Caution; Toxic Material: Do not puncture, break or damage the beryllium window in any way. This can produce airborne particles. Prolonged inhalation of beryllium can cause cancer.

Do not allow the detector or X-Ray tube to come in contact with moisture, or condensation from high humidity. This can corrode the beryllium window, in particular if chlorine, sulphates, copper or iron is also present.

Contact the local Hitachi High-Tech Analytical Science representative for advice about beryllium, or if the beryllium window is pierced, broken, damaged or corroded.

1.6. Spillages

1.6.1. In the event of spillage of liquid from the sample cell, first check whether the liquid has been retained within the secondary (safety) window cell. If so, remove the liquid and replace the secondary window film. If there is any possibility that the liquid has gotten into the interior of the unit, immediately isolate the instrument from mains power supply and contact the service department at Hitachi High-Tech Analytical Science or your local Hitachi service representative.

1.6.2. Spillages on the outer surface can be wiped with a dry or damp cloth. Do not use organic solvents.

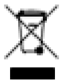
1.7. Safety with samples

1.7.1. Always handle the samples in accordance with the appropriate published Hitachi High-Tech Analytical Science method sheets and normal safety guidelines. If in doubt about the suitability of a sample for analysis, refer to the material safety data sheet. Exercise caution when handling and analyzing samples that give off flammable vapours.

1.7.2. This equipment is not designed to be used in explosive atmospheres.

1.8. Disposal

1.8.1. Contact the local Hitachi High-Tech Analytical Science representative for assistance.



WEEE: Within the EU, return the Lab-X5000 to the local Hitachi High-Tech Analytical Science representative for proper disposal in accordance with WEEE regulations.

The symbol means that used electrical and electronic products should not be moxed with general household waste. For proper treatmeant, recovery and recycling, return to the local Hitachi High-Tech Analytical Science representative. The correct disposal of product will help save valuable resources and prevent any potential negative effects on himan health and the environment, which would otherwise arise from inappropriate waste handling. Penalties may be applicable for incorrect disposal of waste, in accordance with national legislation.

2. Introduction to the LAB-X5000

The Hitachi High-Tech Analytical Science LAB-X5000 is a benchtop XRF analyzer that uses the principle of energy dispersive X-ray fluorescence (EDXRF) spectrometry to determine the concentrations of various elements in a range of materials.

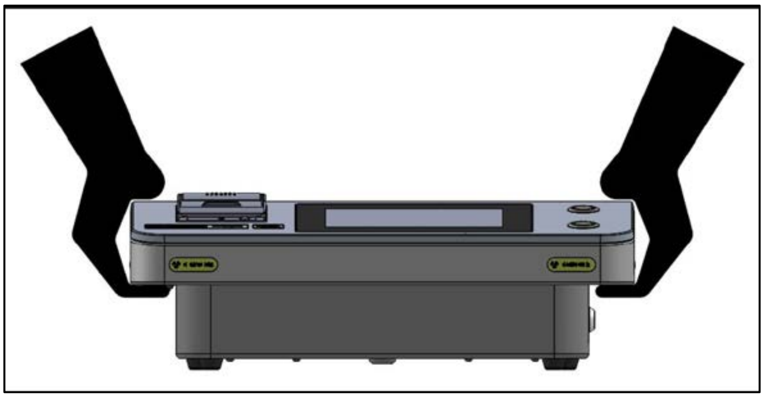
All operator inputs are accomplished via software control and are active using the integrated touch-screen computer. Software messages and results are shown on the touch-screen computer with analytical results, spectrum scans, etc., printed using the built-in thermal printer. Optional wireless (Wi-Fi) capabilities allow for transmission and receipt of data.

The LAB-X5000 is available in a range of models, differing mainly in the application packages and options supplied.

Lab-X5000 is intended for indoor use only. Please see chapter 7 Technical Specification for more information on environmental conditions.

⚠

WARNING! The Instrument weighs 20 kg / 44 lbs. Lift the instrument as shown in picture below.



2.1. Unpacking and Setting up

2.1.1. Open the packing case carefully, remove the LAB-X5000 and generally inspect for any

visible damage. Remove the small boxes/polyethylene bags containing accessories and spares.

2.1.2. Place the instrument on a flat-topped work surface, away from direct sunlight, where changes in ambient temperature are minimal.

2.1.3. Insert the power cord into the receptacle on the left underside of the instrument; insert the other end into an appropriately-grounded wall outlet. Mains cable must be applied in such a way which allows easy disconnection of the wall plug. If original mains cable needs replacement, it must be replaced only with cable which has same electric ratings. See Section 2.3.2 for details of power requirements.

2.2. Equipment Supplied

2.2.1. Each LAB-X5000 comes complete with a comprehensive accessories kit (PartNo.LX5K-PACK1) which comprises the following items:

Part No	Description	Quantity
54-CP1057	Roll of printer paper	5
SUGL50B	SUS for diagnostics	1
54-QX1008	SUS box	1
54-LX6879-2	Secondary safety windows	3
51-3963527	USB with user manual and factory database backup	1
54-LX320	Safety window assembly tool	1
54-QX1009	Silica gel desiccant 601-057	1
54-LB3181	SUS transit warning label	1
XRFMS005	XRF SUS instructions	1

2.3. Mains Power Connections

2.3.1. This instrument must be connected to a grounded mains supply. If you are in any doubt about your power supply, consult a qualified electrician before proceeding.

2.3.2. The LAB-X5000 is operated from a single-phase supply in the range of 100-240 V and 47 - 63 Hz.

2.3.3. The instrument can be switched on at the power switch, with is situated on the lower left side of the instrument.

2.4. Printer

2.4.1. Printer paper will be in the box containing the instrument’s accessories.

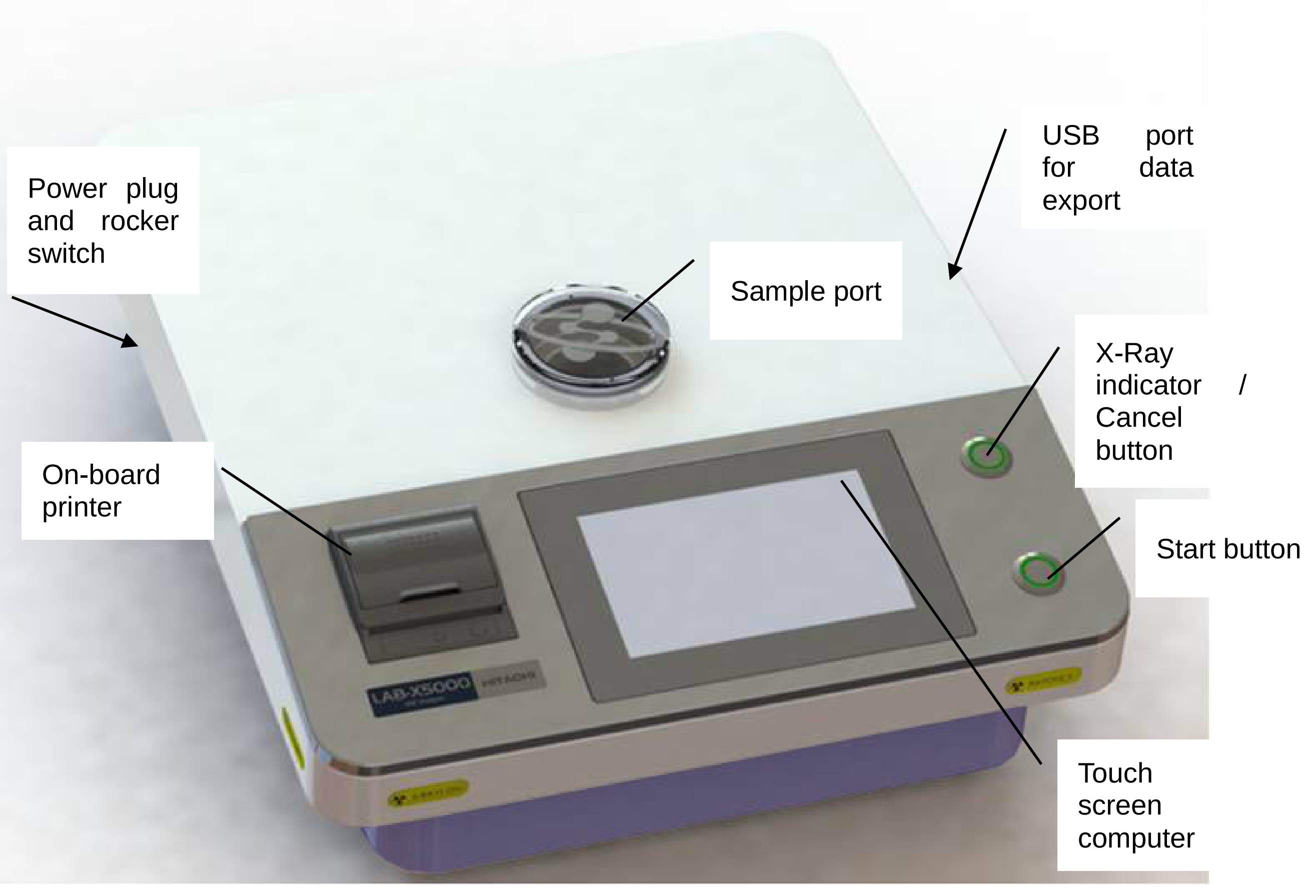
2.4.2. Lift the printer cover on the left front of the instrument using the tab on the top.

2.4.3. Place the roll of paper inside with the paper unrolling from underneath the roll, as you pull it.

2.4.4. Press firmly on the front corners of the printer cover until it snaps closed.

2.4.5. A small blue light flashes when the printer cover is not fully closed. It will remain solid blue when the cover is closed properly.

2.5. External Configuration



2.5.1. The instrument is identified by model and serial numbers located on plates at the rear of the instrument. Any inquiries and correspondence relating to the instrument must include these numbers.

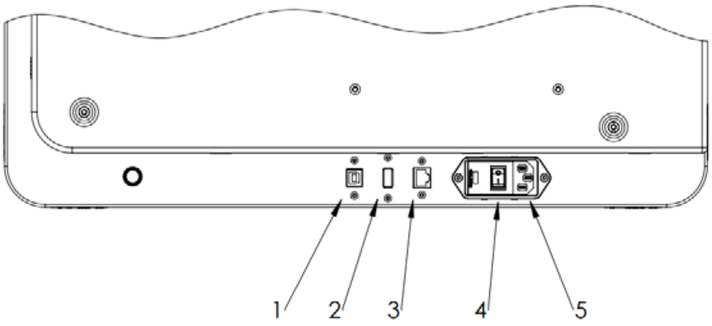
HITACHI
XRF Analyzer

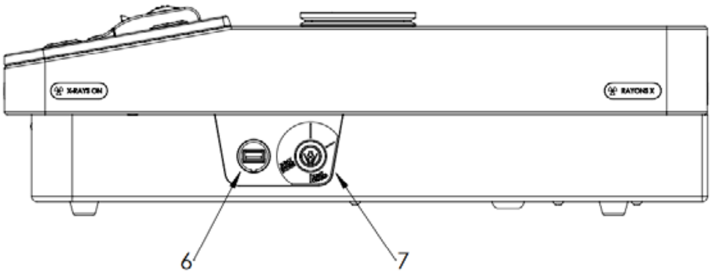
Type: LXDS2860
Rated voltage: 90 - 264 VAC
Rated current: 1.5 A
Rated frequency: 47 - 63 Hz
Fuse: T2A - 250 V
Weight: 20kg
SER No.: 100002
DATE: May 2017

Hitachi High-Tech Science Corporation
Designed by Hitachi High-Technologies in FINLAND
X-Ray tube made in USA
Detector made in FINLAND
Assembled in CHINA

X-Ray tube
Model No: 90026
Serial No: xxxxxx
Target material: Pd
High voltage: 4 - 30 kV
Max power: 3 W

2.6. Connectors





1. USB B connector
2. USB A connector
3. LAN connector
4. Power ON/OFF button
5. AC inlet
6. USB A connector
7. X-Ray ENABLE/DISABLE lock

2.7. Secondary Safety Windows

2.7.1. Secondary safety windows should be fitted into the sample port of the LAB-X5000 before any measurements are made. These prevent dust, accidental sample spillage, etc. from entering the analysis head and contaminating the system.

2.7.2. Assembly of secondary safety windows

2.7.2.1. Safety windows are assembled using a secondary window stage with clamping ring and a window film. This film will depend on the application.

> ☞ **Information**
>
> It is very important that the correct film is used in the safety windows. Incorrect film could invalidate the restandardization procedure.
>
> The safety window must be kept clean and dry. Do not touch the surface. Dirt, moisture, oils, or fingerprints may affect analysis results.

2.7.3. Secondary Window Assembly

2.7.3.1. Place the plastic outer ring on a flat surface. Note: If the ring has a stepped end, place the stepped end upwards.

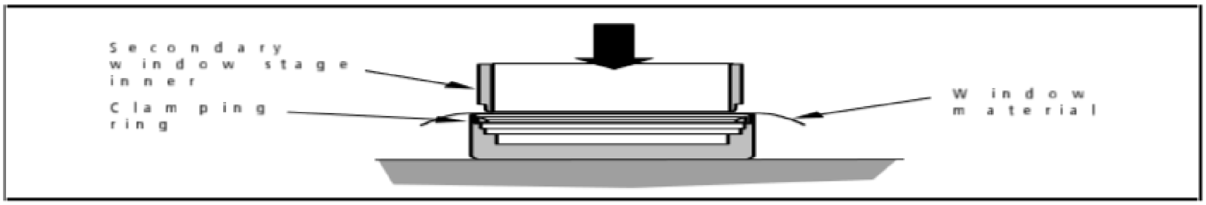
2.7.3.2. Place a piece of window material on top of the ring and press the plastic inner ring fully down onto the outer ring, trapping the window material.

2.7.3.3. Check that the assembled window is perfectly flat. If not, repeat the mounting

procedure with a new piece of window material. A certain amount of window tensioning can be achieved after the rings have been assembled, but take care not to overstretch or tear the window.

2.7.3.4. When the window is satisfactory, trim off the excess material.

2.7.3.5. Place the assembled secondary safety window into the sample port on the top of the analyzer.



2.8. Cleaning and maintaining the Instrument

2.8.1. Outer cover: Use a damp, not wet, cloth. Do not allow water to get inside instrument. Do not use solvents or pre-moistened wipes.
2.8.2. Computer touch screen: Use a dedicated screen wipe of microfiber or similar material.
2.8.3. Lab-X5000 has no periodic maintenance requirements

3. Using the LAB-X5000

All of the operations of the LAB-X5000 are controlled by the built-in touch-screen computer. All calibrations, methods, settings, and standards are available through this touchscreen interface. The computer is controlled through a series of taps and gestures that are intuitive and easily learned.

The X-ray fluorescence technique is a comparative, rather than an absolute, method of analysis. Therefore, a calibration curve must first be established before routine analysis can be carried out. Once a calibration has been created and stored, it will remain accessible for routine analysis from the main pull-down menu. It is a simple operation to restandardize an existing calibration.

3.1. Powering Up the LAB-X5000

A single rocker switch on the left side of the instrument powers the instrument and computer. The [0] position is the OFF position (no power) and the [l] position is the ON position (powered).

- **3.1.1. To power the instrument on, insert the key into the round lock on the right side of the instrument and turn it to “X-RAY ENABLE”**
- **3.1.2. Toggle the rocker switch under the left-hand side to the [l] ON position.**
  - **3.1.2.1. The computer will automatically load the software after a brief pause.**
  - **3.1.2.2. When the software is ready and the instrument is initialized, the front button ring will glow green to indicate READY.**
  - **3.1.2.3. The screen will show an X-Ray safety warning and a PIN is required to operate the instrument and software.**

WARNING

IONIZING RADIATION

This unit produces X-rays when energized.

Only trained people are authorized to operate this instrument.

- **3.1.3. To power the instrument off, choose logout ⇨ from the pull-down menu.**
  - **3.1.3.1. The software will progress to the log-in screen.**

3.1.3.2. When the software is in the log-in screen, toggle the rocker switch to the [0] OFF position.

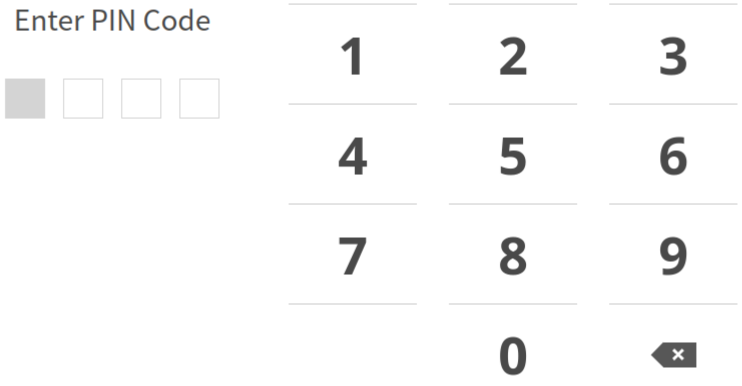
3.2. User Access and PINs

There are three customer-accessible levels of user access: Manager, Operator, and Advanced Operator. Engineer access is reserved for Hitachi authorized service personnel only. Each level has access to different menus and options, allowing managers to control which features and controls their analysts may access.

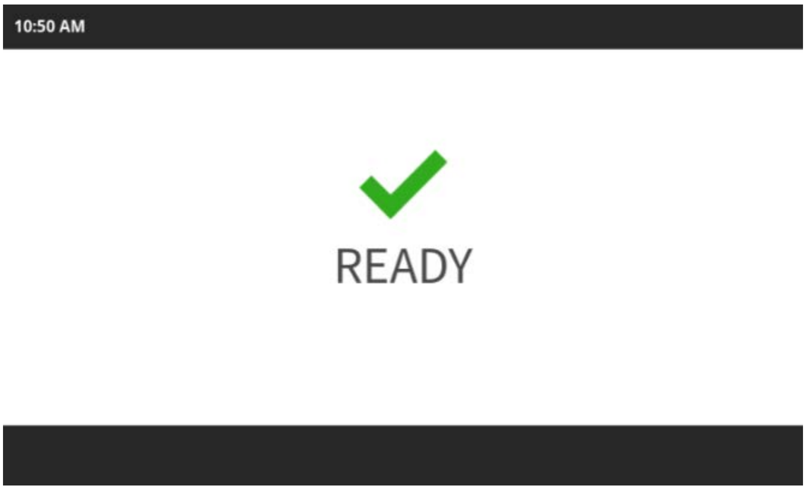
3.2.1. User level is chosen at startup and is determined by which PIN is input to access the software. The user can easily toggle between user levels using the dropdown menu described below.

3.2.2. When gaining access at startup, or when changing user levels from the pulldown menu, it is necessary to enter a PIN Code. The PIN Code is different for each access level, and may be changed by the Manager.

3.2.3. The standard Operator PIN is 0000. Advanced Operator and/or Manager PINs are provided at installation.



3.2.4. After the PIN Code has been entered, a Ready screen will appear. This indicates the instrument is ready to analyze.



3.3. How to Use the Software

The LAB-X5000 is controlled through a touch-screen on-board computer. Basic user training is provided at installation. All functions of the LAB-X5000 can be executed using the on-board touch-screen computer.

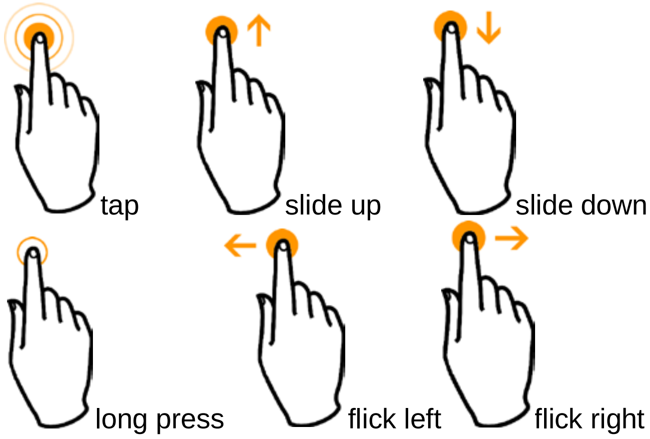
3.3.1. Gestures: There are six basic gestures used in navigating the software.

3.3.1.1. Tap is used to make selections.

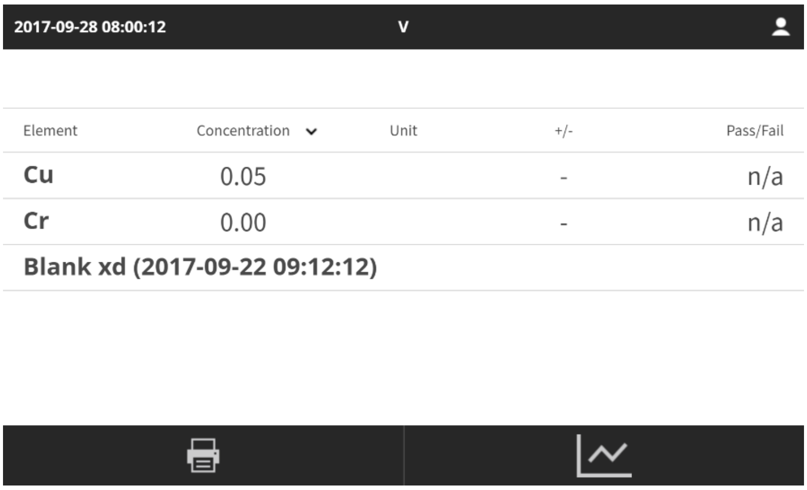
3.3.1.2. Slide or Drag is used to scroll views and menus.

3.3.1.3. Long press is used to “pin” and “unpin” elements in Measurements results.

3.3.1.4. Flick is when you quickly move your finger to jump ahead in a scrolling list.



3.3.2. Main screen: The main screen is divided into three information zones.



2017-09-28 08:00:12 V

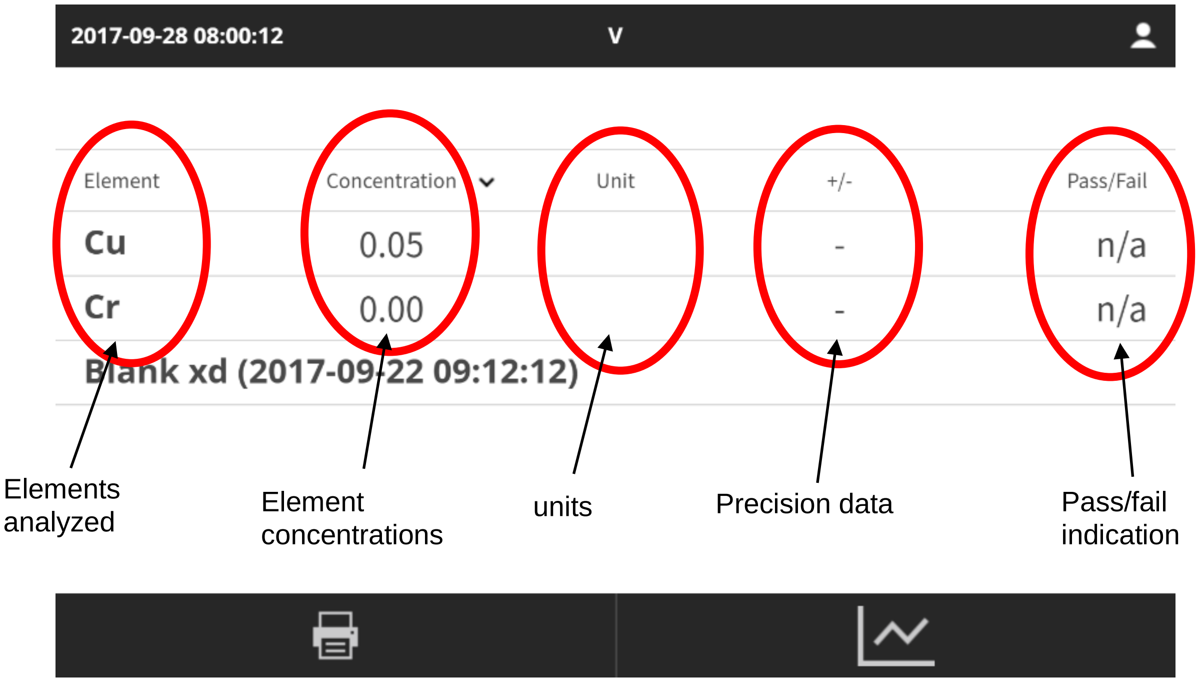
Element	Concentration	Unit	+/-	Pass/Fail
Cu	0.05		-	n/a
Cr	0.00		-	n/a

Blank xd (2017-09-22 09:12:12)

3.3.2.1. The black bar at the top of the screen contains static information relevant to the current sample analysis.

👤	Indicates current operator level, at top right corner.
V	Method in use is visible in the top center.
2017-09-28 08:00:12	Time and date of the last measurement are in the top left corner.
📶	If visible, Wi-Fi is activated and connected. In top right corner.

3.3.2.2. The center section contains the most recent analysis results. Flick right or left to view previous results.



2017-09-28 08:00:12 V

Element	Concentration	Unit	+/-	Pass/Fail
Cu	0.05		-	n/a
Cr	0.00		-	n/a

Blank xd (2017-09-22 09:12:12)

3.3.2.3. The black bar at the bottom of the screen contains buttons to toggle the center view.

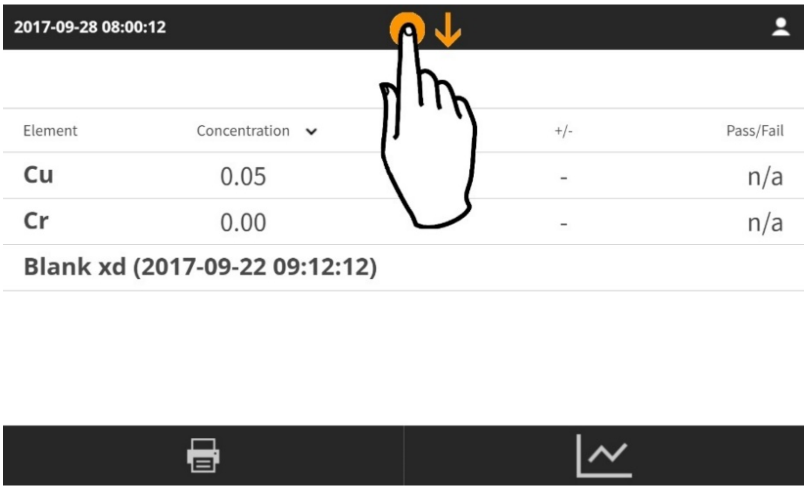
🖨	Print or reprint the result currently visible on the screen
🗑	Trash or delete the last measurement made. Only works on most recent result, will not delete older data.
📈 123	Use to toggle between viewing numerical results and spectral results.
⚙	Settings menu – not available in all screens. Tap this icon to access more options.

3.4. Analyze a Sample

☞ Information

This section assumes that a calibration has already been established. If not, refer to sections 5.4 and 5.5. for details on creating a calibration.

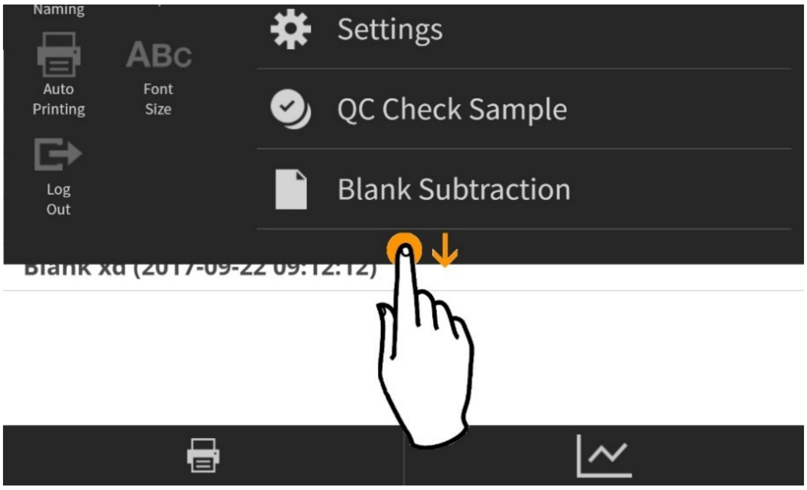
3.4.1. **Check that the Method is appropriate for the sample. In the pull-down menu, select Method.**



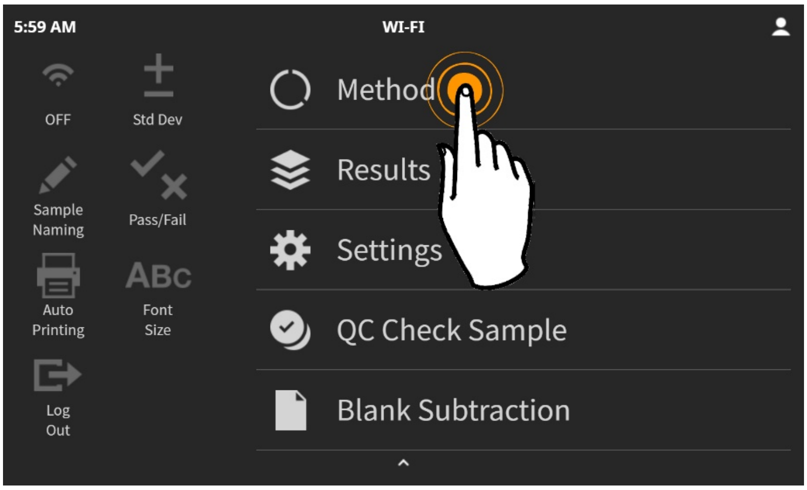
2017-09-28 08:00:12

Element	Concentration	+/-	Pass/Fail
Cu	0.05	-	n/a
Cr	0.00	-	n/a

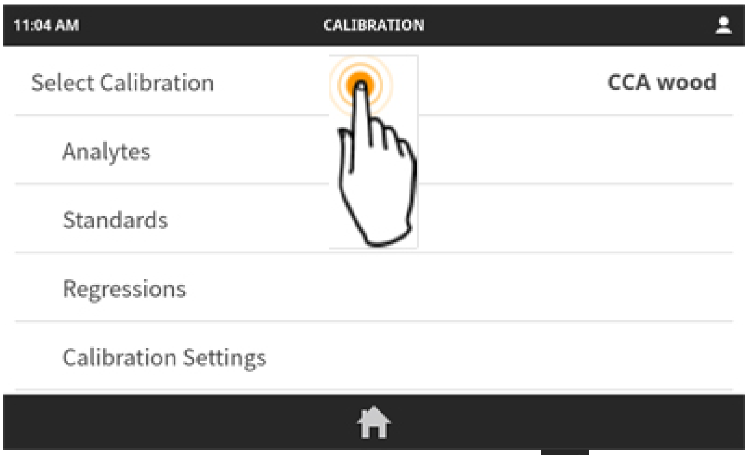
Blank xd (2017-09-22 09:12:12)



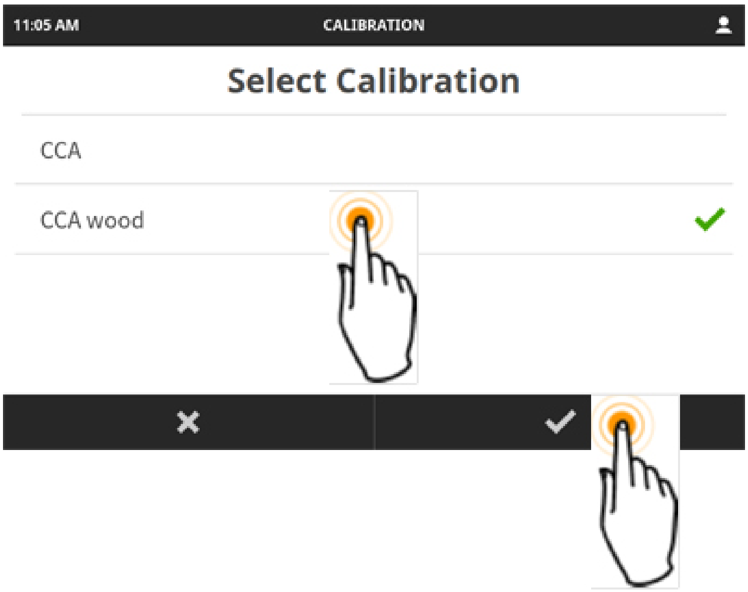
Blank xd (2017-09-22 09:12:12)



3.4.1.1. All methods stored in the instrument and available for use will be visible.

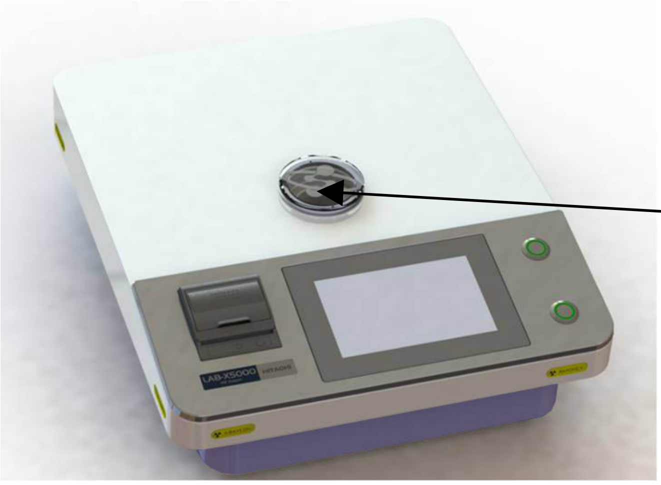


3.4.1.2. Select the correct method, then tap ✓ to accept the input and return to the Method menu.



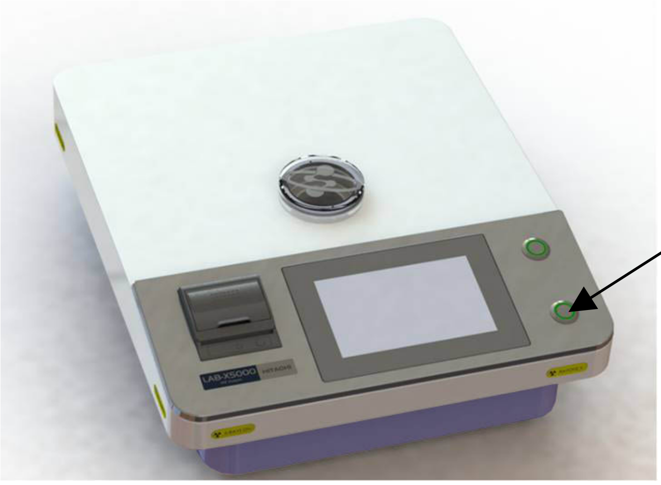
3.4.2. Name the sample (if Sample Naming is active in the pull-down menu).

3.4.3. Remove the glass cover from the sample port and insert a secondary safety window, if there is not one already installed. For instructions on secondary safety window assembly, see Section 2.6.3.5.


Remove glass cover and insert secondary safety window or sample cup into sample port here.

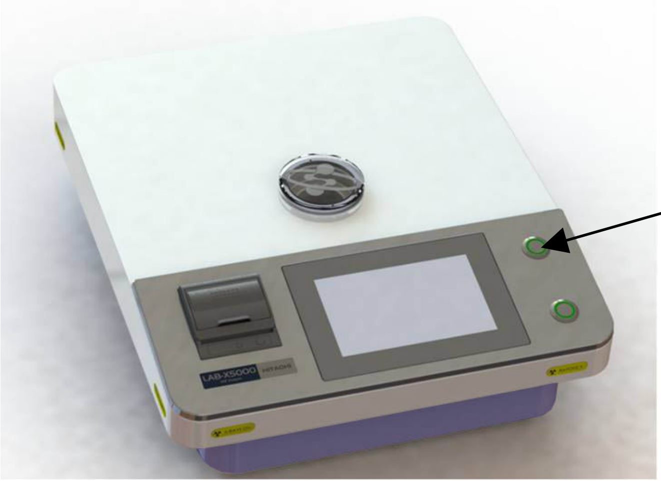
3.4.4. Leaving secondary safety window in place inside the sample port, insert a sample cup into the sample port on the top of the instrument as shown above.

3.4.5. Press the start button. It will have a lighted green ring around it when the instrument is ready.


When lighted ring is GREEN, instrument is ready to analyze. Press this button to START analysis.

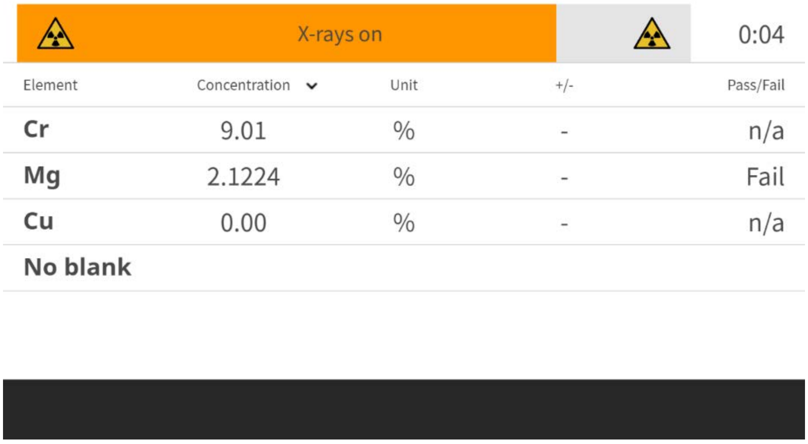
3.4.6. The sample will rotate out of the sample port and into the path of the X-rays. The lighted ring around the start button will extinguish and the cancel button ring will light red. This indicates that the X-Ray tube is powered and the sample is being analyzed.

3.4.6.1. Press the Cancel button to stop the analysis at any time, if necessary.


When lighted ring is RED, instrument is analyzing and X-rays are ON. Press this button to STOP analysis.

3.4.7. Wait for the analysis to finish and the results to appear.

3.4.7.1. Analysis progress will be displayed on the screen in a progress bar, with time remaining on the right.



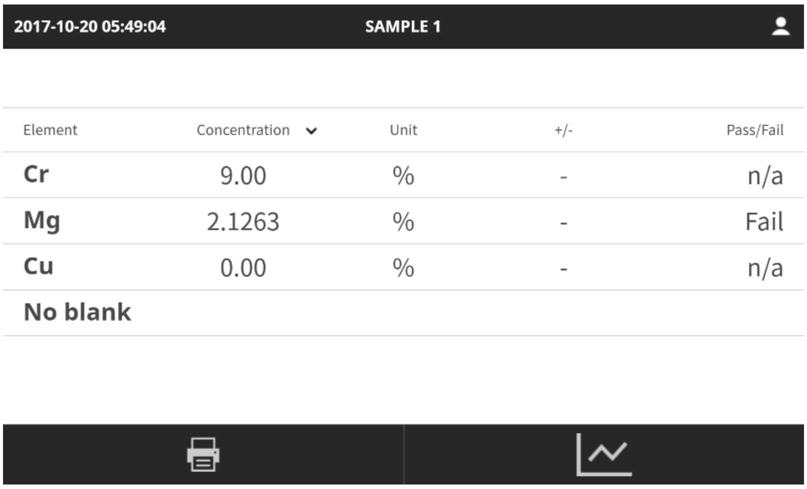
X-rays on 0:04

Element	Concentration	Unit	+/-	Pass/Fail
Cr	9.01	%	-	n/a
Mg	2.1224	%	-	Fail
Cu	0.00	%	-	n/a
No blank				

3.4.7.2. Concentrations visible during analysis are not final results until analysis is complete.

3.5. Analysis Results

3.5.1. After the measurement, the results screen will be displayed. The name of the sample is shown, along with the chemistry of the results, the measurement unit, the error, and the pass/fail determination, if one was programmed.

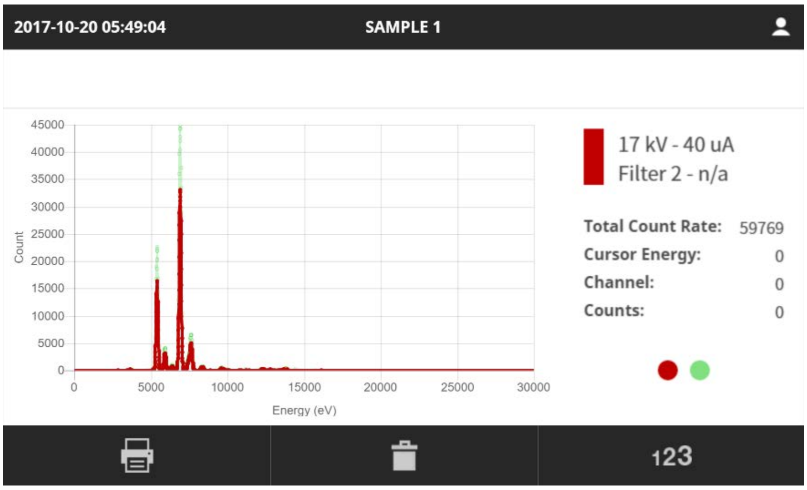


2017-10-20 05:49:04 SAMPLE 1

Element	Concentration	Unit	+/-	Pass/Fail
Cr	9.00	%	-	n/a
Mg	2.1263	%	-	Fail
Cu	0.00	%	-	n/a
No blank				

3.5.2. Swipe left or right to access previous results.

3.5.3. Tapping the 📈 button will toggle to the spectral view of the results.

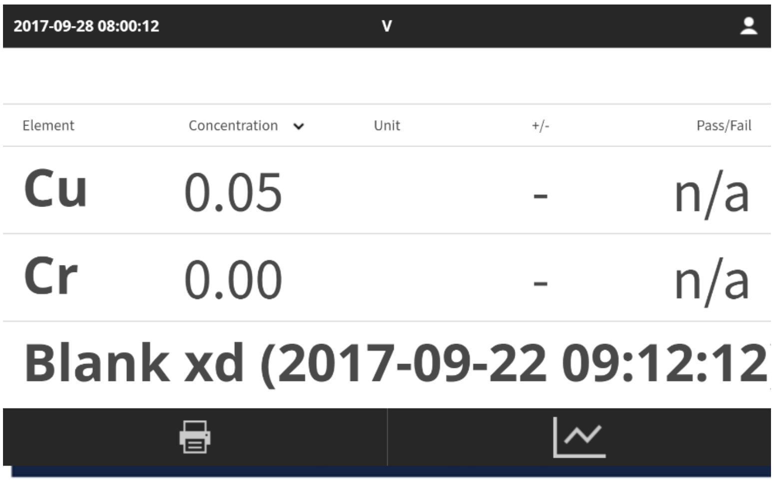


2017-10-20 05:49:04 SAMPLE 1

17 kV - 40 uA
Filter 2 - n/a

Total Count Rate: 59769
Cursor Energy: 0
Channel: 0
Counts: 0

3.5.4. Tap 123 to return to numerical results.



2017-09-28 08:00:12 V

Element	Concentration	Unit	+/-	Pass/Fail
Cu	0.05		-	n/a
Cr	0.00		-	n/a

Blank xd (2017-09-22 09:12:12

3.5.5. To delete a result, tap the trashcan 🗑 icon in the bottom black bar.

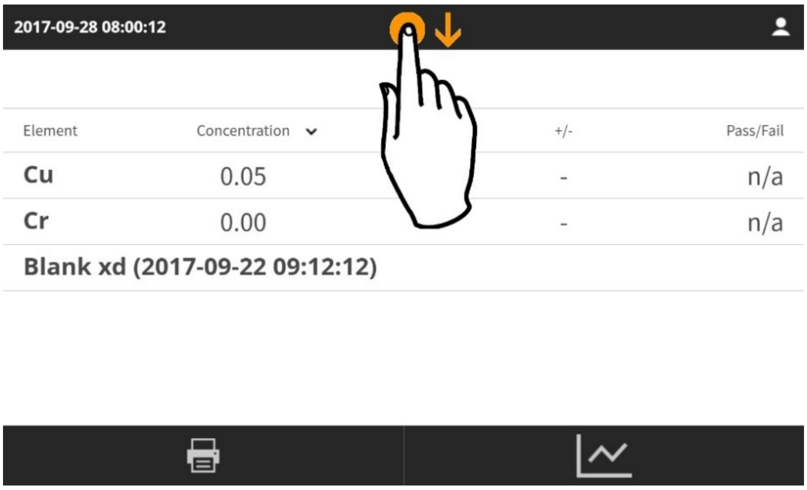
3.5.5.1. Note: only the current result can be deleted. Past results cannot be deleted in this manner.

4. Pull-Down Menu and Quick Options

The pull-down is the main screen for navigating within the software.

4.1. Accessing the Pull-Down Menu

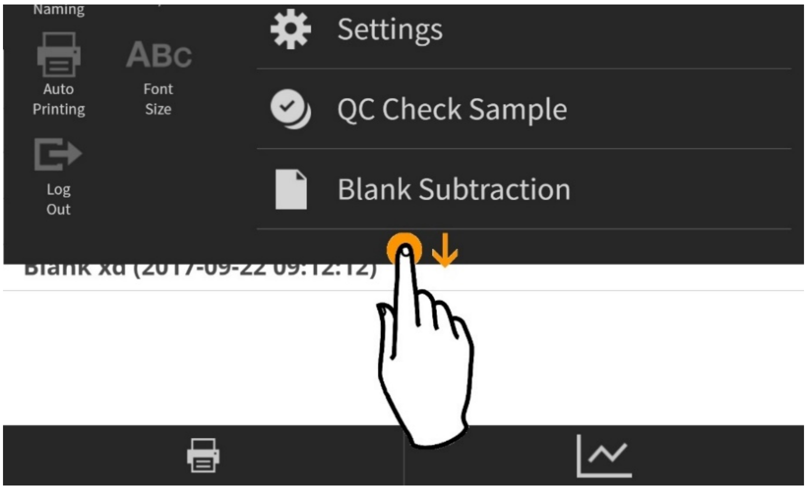
4.1.1. **To access the pull-down menu, drag the status bar down with finger.**



2017-09-28 08:00:12

Element	Concentration	+/-	Pass/Fail
Cu	0.05	-	n/a
Cr	0.00	-	n/a

Blank xd (2017-09-22 09:12:12)



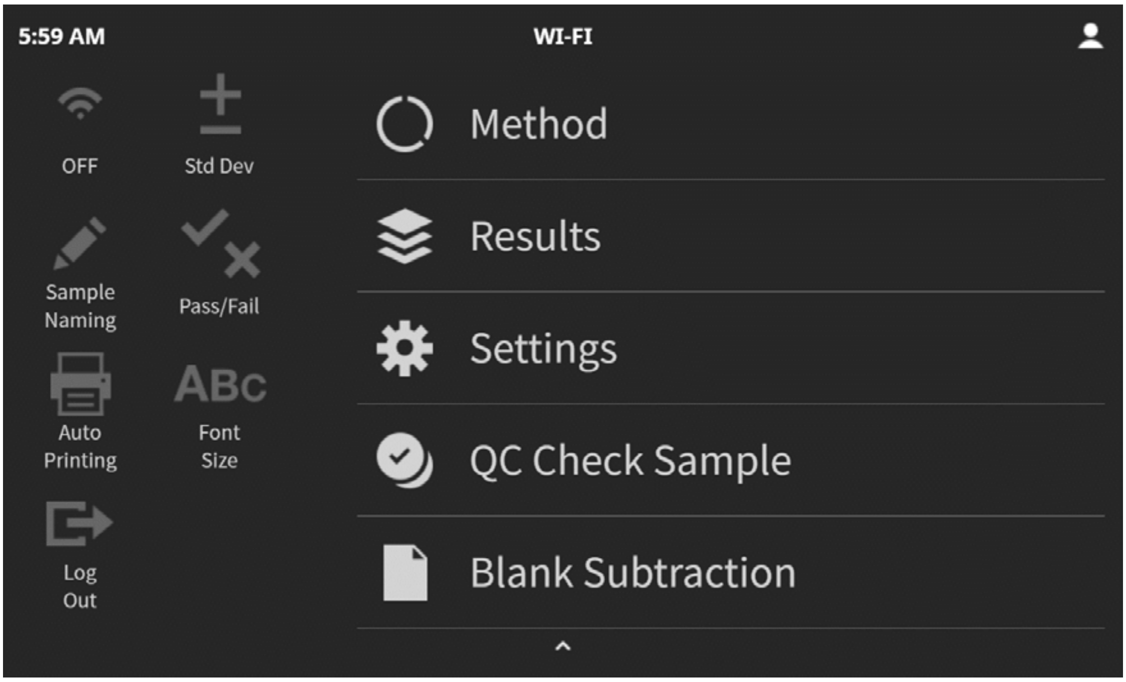
4.1.2. **From there you can quickly toggle wi-fi and other measurement settings on or off, access programs, sample naming, result history and settings.**

4.1.3. **Note that not all menus may be available, depending on the user level of access.**

Unavailable options may be visible, but greyed out, and tapping them will have no effect. Some options will not be visible when logged in as an Operator.

4.2. Quick Access Buttons

4.2.1. The Quick Access Buttons are on the left-hand side of the pulldown menu. They are shortcuts to frequently used options within the software.

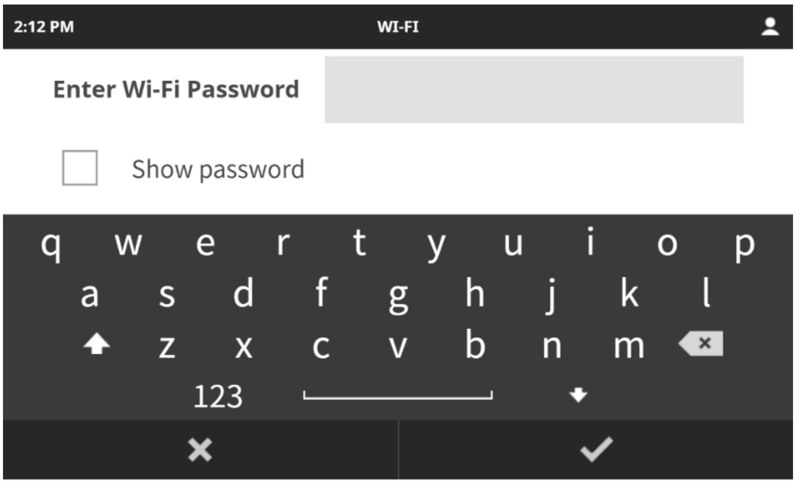


📶	**Wi-Fi button** is used to toggle Wi-Fi on/off. When turned on, it opens Wi-Fi settings, where a network can be selected.
±	**Std Dev** toggles whether the standard deviation is visible on the results screen.
✎	**Sample Naming** is used to name a sample prior to measurement.
✓✗	**Pass/Fail** is used to toggle between measurement pass/fail information on the results screen.
🖶	**Auto Printing** is used to select if sample measurement results will automatically print from the on-board printer. Note that it is possible to print results from any previous result.
ABC	**Font Size** is used to toggle between results displays. When in LARGE, only the first three rows of results will display on the screen. When in NORMAL, six rows of results including precision or pass/fail results will display.
⇨	**Logout** is used to exit the analysis software. Tap to begin the exit sequence. The instrument does not turn off after tapping this button.

4.3. Wi-Fi setup

4.3.1. Tap Wi-Fi button to open the Wi-Fi settings screen.
4.3.2. In Wi-Fi settings tap the Wi-Fi toggle to turn the Wi-Fi on/off.

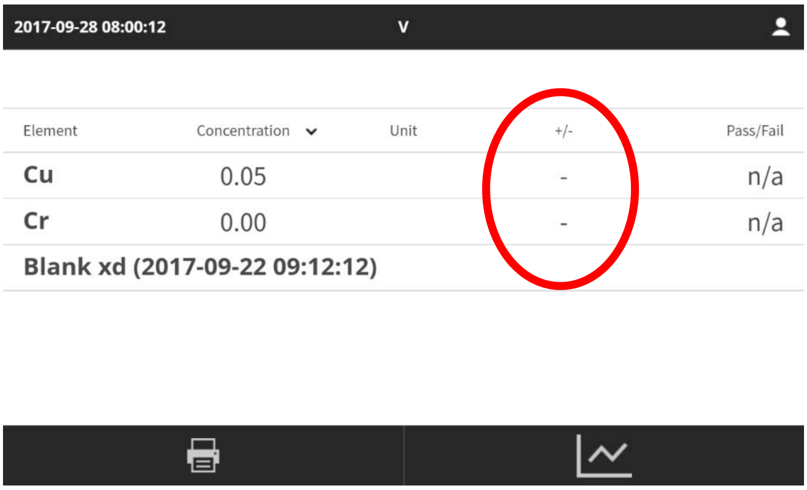
4.3.3. Tap a network to connect. Tapping it opens a dialog where you can select to connect. After connecting once, you'll connect automatically to the same network if it's available. If a password is required (a lock icon appears next to the network name), you will be asked to type it in.



4.3.4. When Wi-Fi is on and connected, you'll see the symbol in the status bar.
4.3.5. To prevent the instrument from automatically connecting to a network, tap the network's menu ⋮ and select Forget network.
4.3.6. To see details of a network you're currently connected to, tap its name. A status popup will open and display the Mac address, IP address, State, Signal Strength and Security

4.4. Standard Deviation

4.4.1. The Standard Deviation button ± toggles whether the error data displays on the analysis results screen after each measurement.


2017-09-28 08:00:12 V

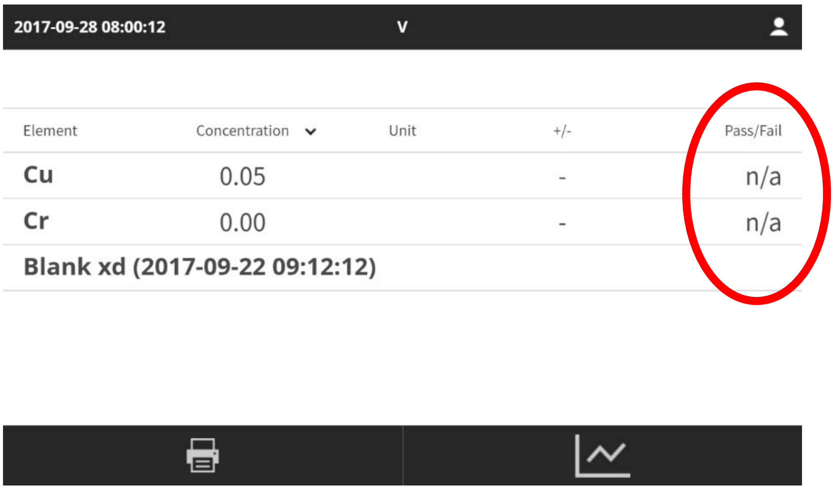
Element	Concentration	Unit	+/-	Pass/Fail
Cu	0.05		-	n/a
Cr	0.00		-	n/a

Blank xd (2017-09-22 09:12:12)

4.5. Pass/Fail

4.5.1. Use the Pass/Fail toggle ✓✕ to choose whether to display pass/fail data in the analytical results screen.

4.5.2. Limits for Pass and Fail are set in the Calibration Settings for each element.


2017-09-28 08:00:12 V

Element	Concentration	Unit	+/-	Pass/Fail
Cu	0.05		-	n/a
Cr	0.00		-	n/a

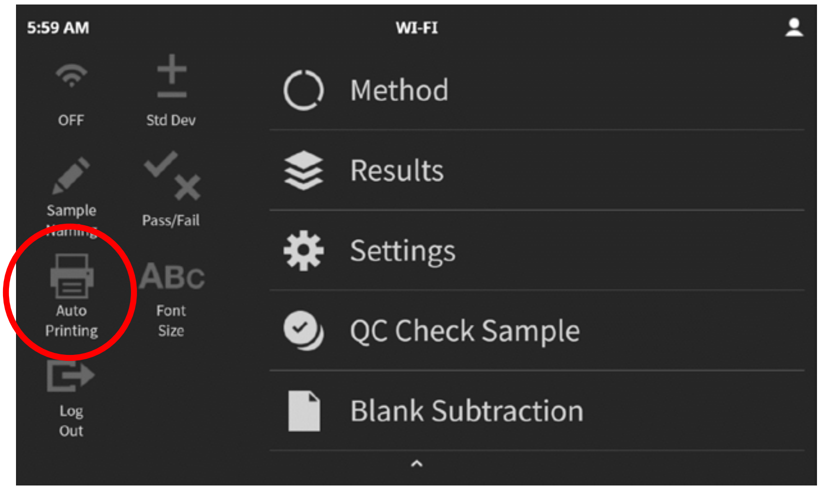
Blank xd (2017-09-22 09:12:12)

4.6. Auto Printing

4.6.1. Auto Printing

4.6.2. The Auto Printing setting is a persistent setting, meaning that once it is engaged, it remains engaged until turned off.

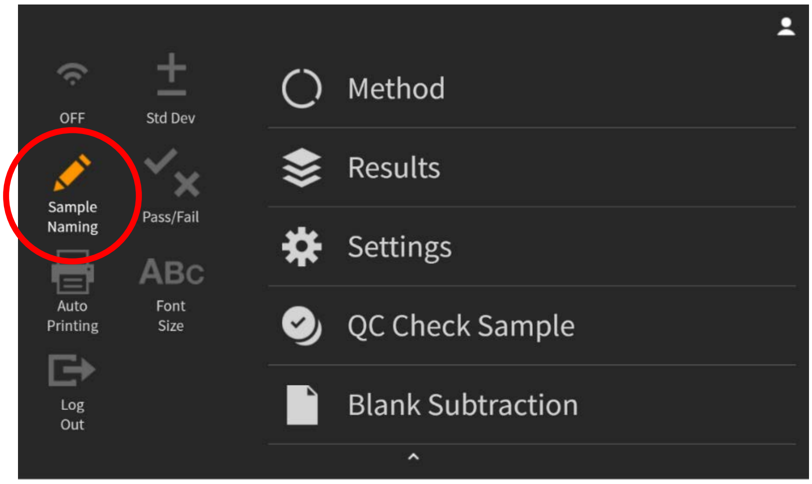
4.6.3. When the Auto Printing icon is selected, it will remain orange instead of grey.



4.7. Sample Naming

4.7.1. To find your measuring results easily later, you should name your samples. A default name is not given, and will save as a name-less sample along with time and date of the measurement.

4.7.1.1. ○ Open pull-down menu.



4.7.1.2. ○ Tap Sample Naming ✎.

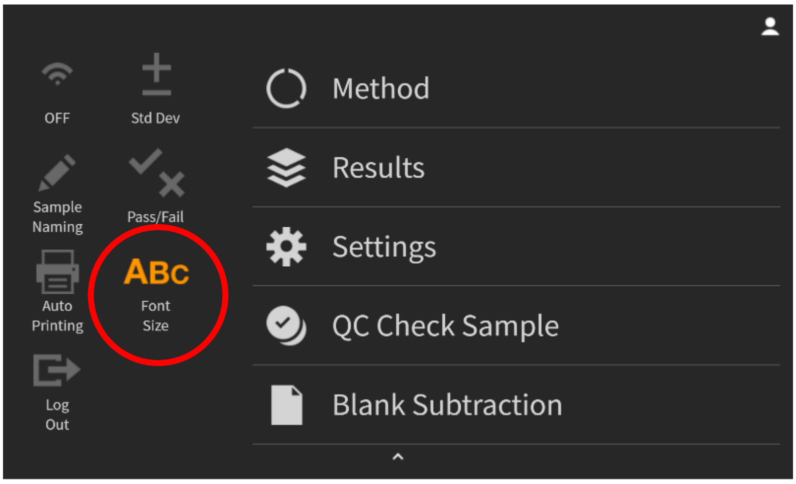
4.7.2. When the Sample Naming icon is selected, it will remain orange instead of grey.

4.7.2.1. When the Start button is pressed, the screen will change to allow you to name the sample prior to analysis.

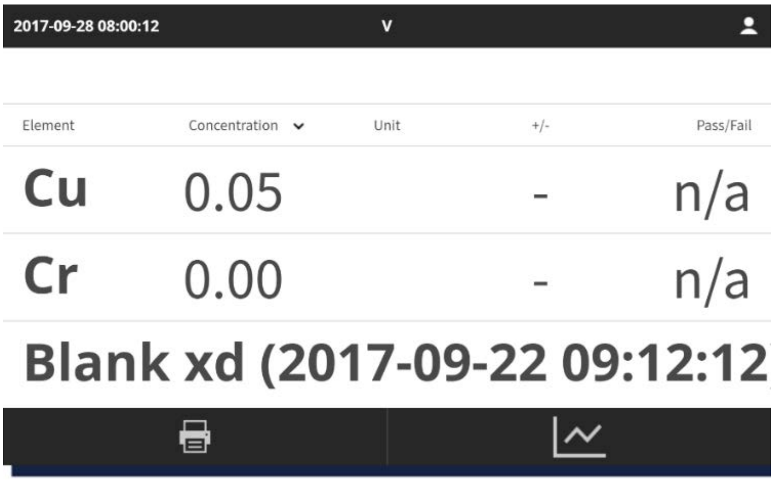
4.7.2.2. Once the done button is tapped, the X-rays will turn on and the analysis will begin.

4.8. Font Size

4.8.1. To view the results in large format, select Font Size ABC from the pull-down menu.



4.8.2. Only the first three rows of the results will display on the screen, in a larger font.



2017-09-28 08:00:12 V

Element	Concentration	Unit	+/-	Pass/Fail
Cu	0.05		-	n/a
Cr	0.00		-	n/a

Blank xd (2017-09-22 09:12:12

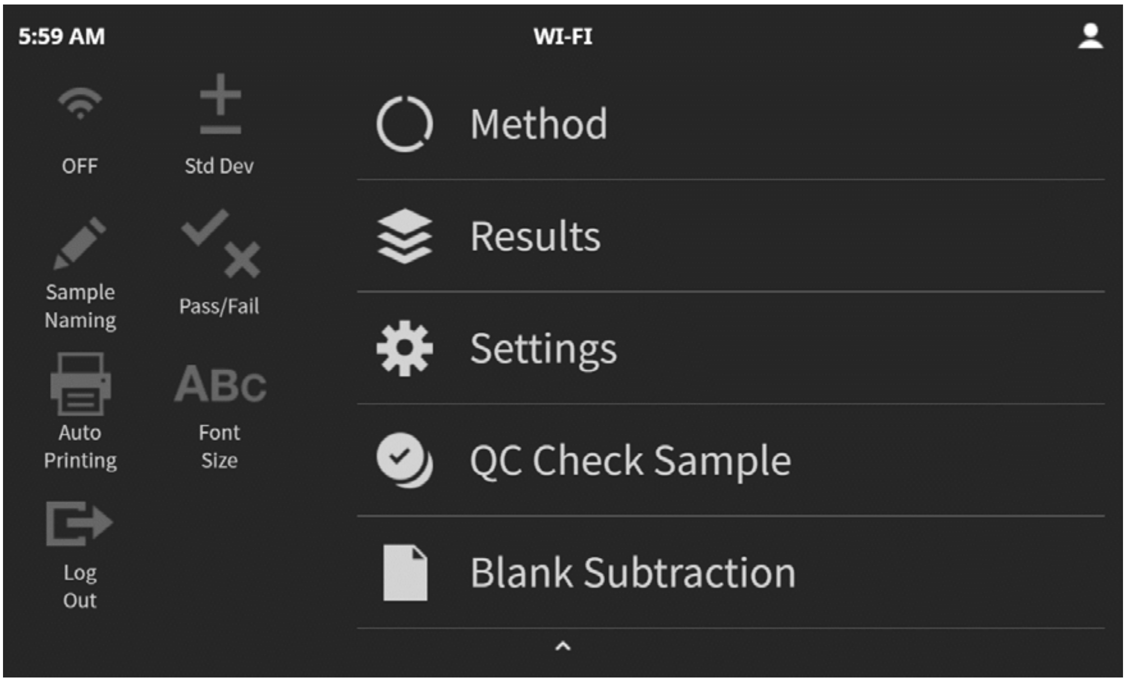
4.8.3. This is a persistent setting – once chosen, the icon will remain orange and the results will display as shown above until the icon is tapped again, turning off the large font display.

5. Options Menu

The right-hand side of the dropdown menu is the Options Menu. This is the main navigation panel for the software. Each choice in the menu leads to submenus where more settings and choices are available.

5.1. Accessing the Options Menu

5.1.1. **Slide a finger from top to bottom of the screen to access the pulldown menu. All Options are visible on the right-hand side of the pulldown menu.**



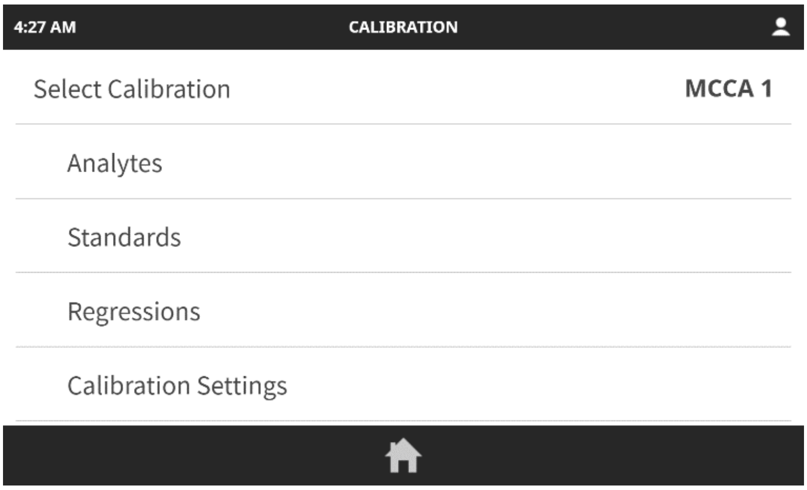
◯	**Method** is used to choose the parameters for the analysis

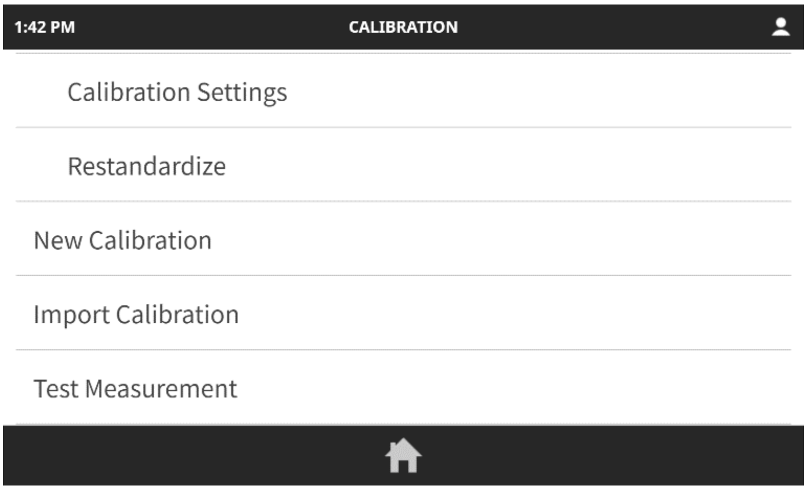
≡	**Results** are used to view the entire results history and choose results for exports.
⚙	**Settings** is used to access the menu of settings available to the operator
✓	**QC Check Sample** is used to measure a pre-defined check sample and compare results to set limits to validate calibration stability.
🗎	**Blank Subtraction** is used to measure a new blank sample to correct for base material changes. Only active when blank subtraction is applied to the calibration.

5.2. Method

5.2.1. **Selecting Method displays all Method options available at the current user level.   Note that not all options are available at all user levels.**

5.2.2. **Standards, calibration settings, and regressions are discussed in subsequent sections.**





Select Calibration	Choose from calibrations currently in the instrument, modify a calibration, or delete a calibration.
Analytes	Choose which analytes are in the method, display name, number of decimal places to display, and define lower and upper pass-fail limits for each element.

Standards	Define standards to be available for performing a regression. Each standard displays as an individual page where the user can set the standard name, composition and whether or not to include the standard in the regression. Standards are measured directly from these screens.	
Regressions	View the analytical data of the calibration standards, choose which type of calibration model is to be used, define the ratio model and power (for ratio modes). Choose modifications to absorption and enhancement corrections, additive corrections, and instrument corrections such as blank correction, overlap corrections, and restandardization.	
Calibration Settings	Choose calibration options	
	Edit Description	Type in a description of the calibration and/or matrix
	SmartCheck Settings	
	Post-Analysis Calculations	Define equations for calculated elements, such as oxides or isotopes. Define fixed values.
	QC Check Sample	Define name, elements, and concentrations
	Sample Spinner	On/off toggle
	Number of repeats	How many times to analyze the sample and average the results
	Number of Aliquots	How many replicate samples to average the results
	Export Calibration	Export the calibration to a USB stick for use on another LAB-X unit.
Restandardize	Re-analyze standards for a calibration to adjust for drift	
New Calibration	Build a new calibration	
Import Calibration	Import a calibration from a USB stick, that was built on another LAB-X unit	
Test Measurement		

5.3. Restandardize

5.3.1. Restandardization involves measuring the Setting Up Samples (SUSs) that were measured as part of the calibration. Once they have been measured they correct for any change in the position of the calibration line, both in terms of background or sensitivity change.

5.3.2. If the calibration involved a matrix correction, such as an overlap correction, then the SUSs used in the correction are also measured as part of the restandardization procedure.

5.3.3. Each SUS will have to be measured in turn to restandardize.

5.4. New Calibration

5.4.1. A calibration curve must be established before routine analysis can begin.

5.4.2. A new calibration can be created in one of three ways:

5.4.2.1. Defining the measurement conditions, typing in the concentration data for a set of standards, then analyzing those standards (this section)

5.4.2.2. Measuring a set of calibration standards using a Hitachi pre-programmed calibration (see section 5.5 Import Calibration)

5.4.2.3. Importing a calibration from another LAB-X instrument (see section 5.5 Import Calibration)

5.4.3. Before a calibration can be derived, a Test Measurement scan should be performed on standards similar to the samples expected to be routinely analyzed to ensure that the correct ‘window’ settings are used. (See Section 5.8 Test Measurement)

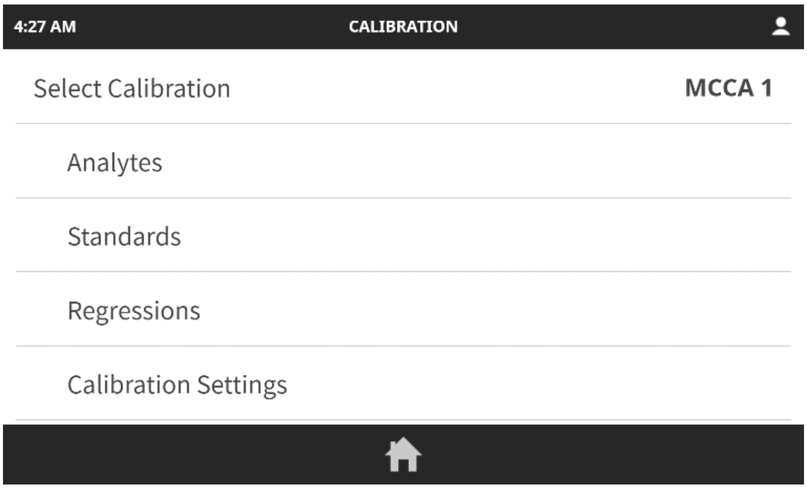
5.4.3.1. The series of standards needs to be similar to the unknown samples is composition, with a concentration range encompassing or exceeding the expected concentration range of the unknowns.

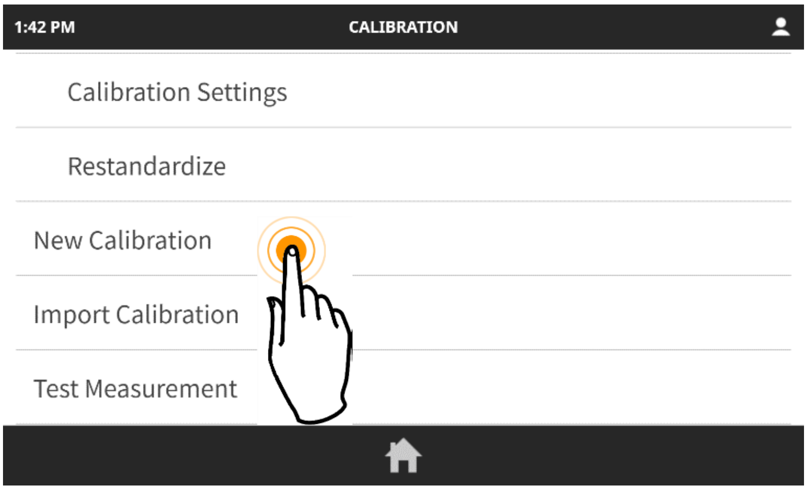
5.4.3.2. Standards can be certified reference materials, samples that have been well-analyzed using another analytical technique, or synthetics.

5.4.4. Using the chosen standards, a calibration can be derived by specifying the instrument measurement conditions, then measuring the standards and performing regression calculations, which may involve instrument or matrix corrections.

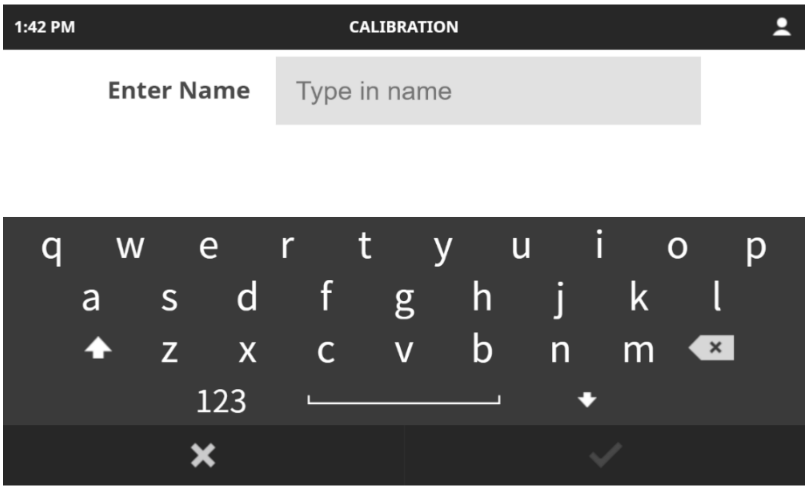
5.4.5. Once a calibration has been established, it can be saved for future recall and analysis, or can be exported to another LAB-X instrument.

5.4.6. To create a new calibration: from the pull-down menu, select Method, then select New Calibration.



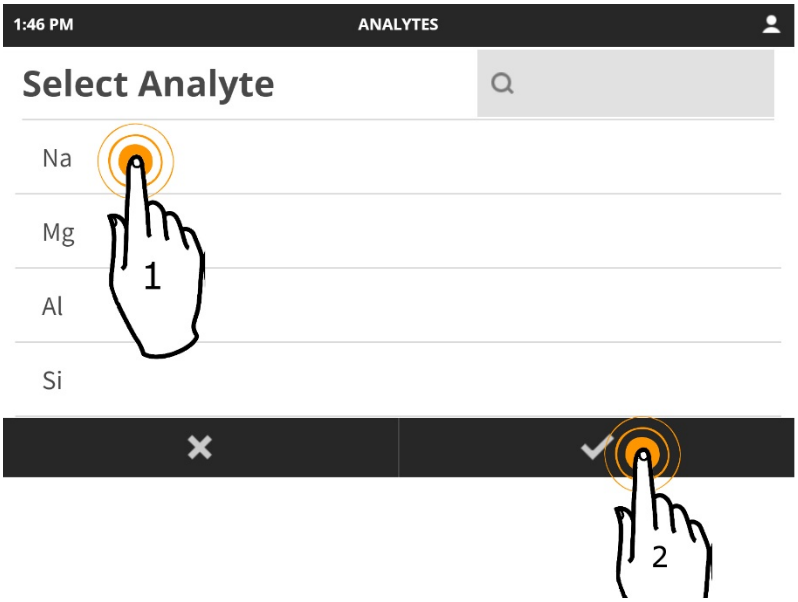


5.4.6.1. Type in a name for the new calibration, then tap ✓ to accept the data and return to the Calibration menu.

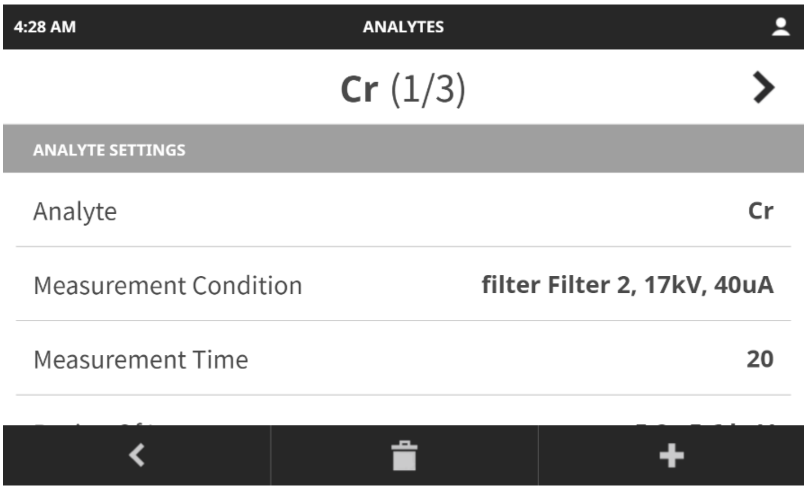


5.4.6.2. Select Analytes. Tap ✚ to add an analyte.

5.4.6.3. Tap one analyte to select, then tap ✓ to accept the data and return to the Calibration menu.



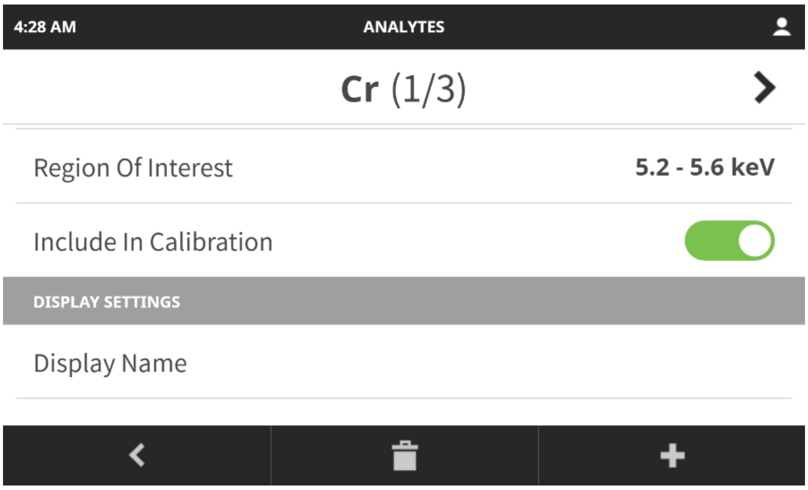
5.4.6.4. For each element, determine the settings for the following parameters:



Analyte Cr

Measurement Condition filter Filter 2, 17kV, 40uA

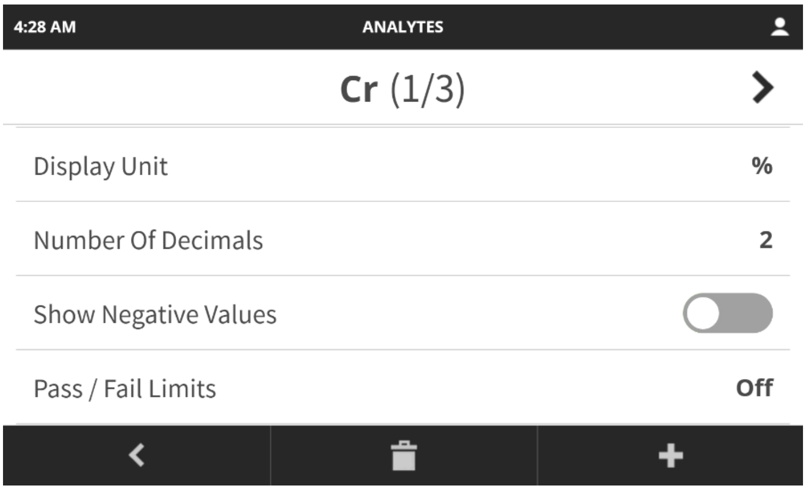
Measurement Time 20



Region Of Interest 5.2 - 5.6 keV

Include In Calibration ●

Display Name



Display Unit %

Number Of Decimals 2

Show Negative Values ○

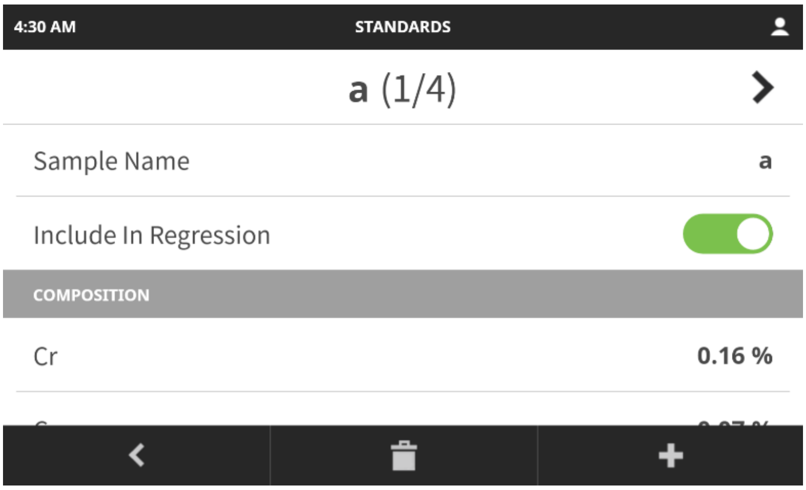
Pass / Fail Limits Off

Measurement conditions	Choose from saved conditions parameters
Measurement time	Type in a number of seconds for analysis (<999)
Region of interest (ROI)	Type in numbers for lower limit (keV) and upper limit (keV). Pre-defined values are offered by default. The user can choose to use different values. It is recommended to perform a Test Measurement to determine ROI limits.
Include in Calibration	Toggle on/off to show quantification of the analyte (on) or to collect data for corrections but not display calculated values (off)
Display Name	Default is 2-letter element abbreviation
Display Unit	Default is %
Number of Decimals	Select appropriate significant figures. The default setting is 2.
Show Negative Values	Toggle on/off to show (on) or hide (off) negative values. When set to off and calculated results are negative, the display will show <0. Default setting is off.
Pass/Fail Limits	Toggle to turn on. Once on, user can type in lower and upper limits for pass/fail criteria. Any sample in routine analysis where the measured concentration is outside the specified limits produces a red band in the Pass/Fail column of the analysis results.

5.4.6.5. Repeat for each element in the calibration.

5.4.6.6. Tap Standards. Tap + to add a standard.

5.4.6.7. Type in a name for the new standard, then tap ✓ to accept the data and return to the Standards menu.

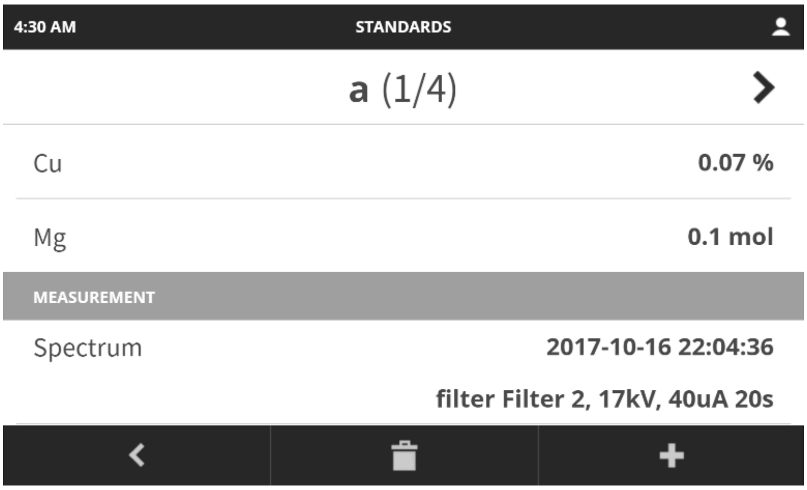


Sample Name: a

Include In Regression: on

COMPOSITION

Cr: 0.16 %



Cu: 0.07 %

Mg: 0.1 mol

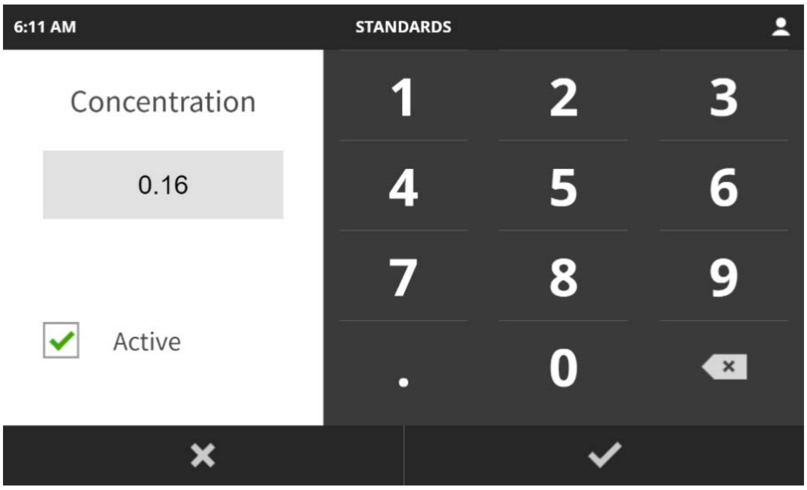
MEASUREMENT

Spectrum: 2017-10-16 22:04:36

filter Filter 2, 17kV, 40uA 20s

5.4.6.8. Include in Regression: toggle to turn on/off. Default is on. When turned off the standard can be used for configuring instrument corrections, but the standard will not be used to calculate the regression.

5.4.6.9. Tap an element. Type in the concentration of the element in the standard in the defined unit. An individual analyte from a standard can be omitted from the regression by tapping the box by “Active” to remove the checkmark.



Concentration: 0.16

☑ Active

5.4.6.10. Repeat for each element in the standard, until all elements included in the calibration have concentration values.

5.4.6.11. Press the Start button to analyze the standard.

5.4.6.12. When analysis is complete, tap ➕ to add another standard. Repeat for each standard in the set.

5.4.7. Regression Analysis: When all standards have been input and analyzed, from the pull-down menu select Method, then tap Regressions. The computer will calculate the regressions, then display “Calibration Complete”.

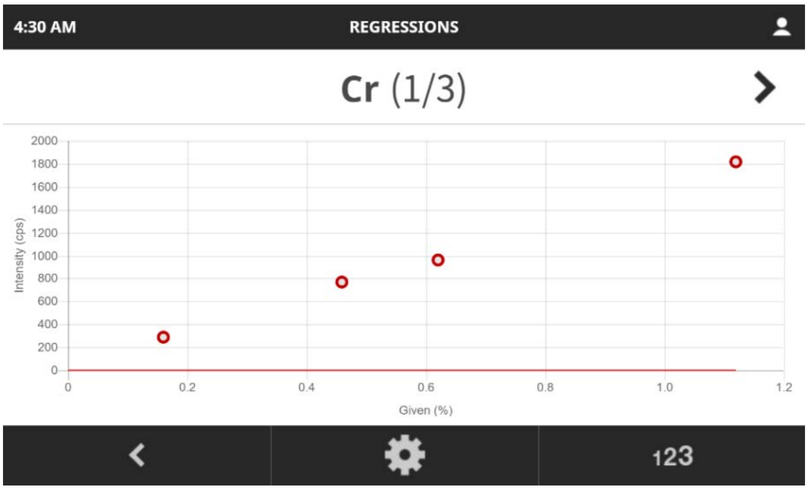
5.4.7.1. If there are not enough standards for the selected regression model, a warning will appear “More standards are needed for selected regression model”.

5.5. Regressions

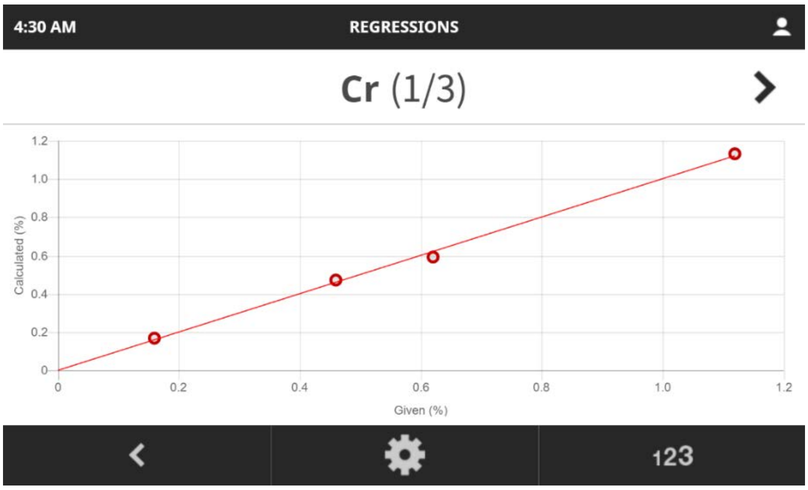
5.5.1. Select Method from the pull-down menu, then choose Regressions to perform a regression of the raw intensity data against the given concentration of the standards. The error analysis for all elements in the analyzed standards will be calculated. The minimum number of standards is 3. More are required when complex corrections are applied.

5.5.2. There are four options for viewing the regression data. They can be toggled by tapping the 📈 and 123 buttons to cycle through the displays.

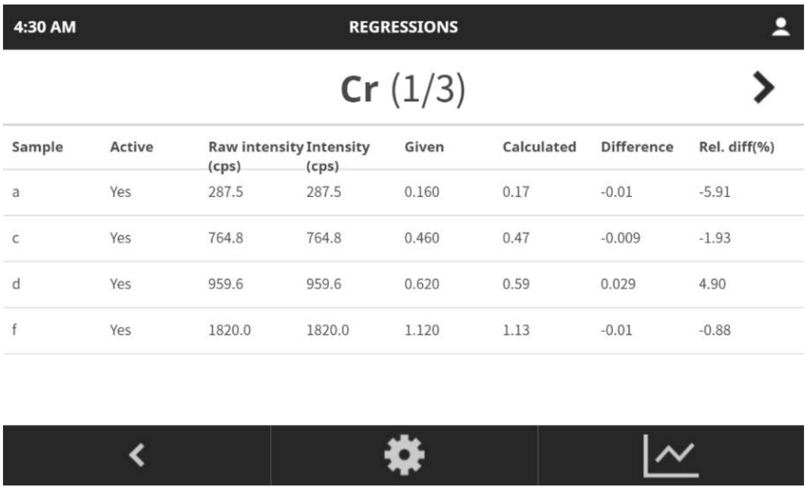
5.5.2.1. Graph 1: A graph of raw intensity (raw data) against concentration.



5.5.2.2. Graph 2: A graph of given concentration against calculated concentration.



5.5.2.1. Intensities: A display of the original (raw) data and the corrected intensity used to perform the regression. Corrected intensities are used when instrument corrections (Blank, Overlap) are applied.



Cr (1/3)

Sample	Active	Raw intensity (cps)	Intensity (cps)	Given	Calculated	Difference	Rel. diff(%)
a	Yes	287.5	287.5	0.160	0.17	-0.01	-5.91
c	Yes	764.8	764.8	0.460	0.47	-0.009	-1.93
d	Yes	959.6	959.6	0.620	0.59	0.029	4.90
f	Yes	1820.0	1820.0	1.120	1.13	-0.01	-0.88

5.6. Import Calibration

5.6.1. Measuring a set of calibration standards using a Hitachi pre-programmed calibration

5.6.1.1. Pre-programmed calibrations cannot be edited or deleted.

5.6.1.2. In order to use a pre-programmed calibration as the basis for a new one, it is necessary to copy the pre-programmed calibration and save it as a new calibration name, then measure an appropriate set of standards and SUSs.

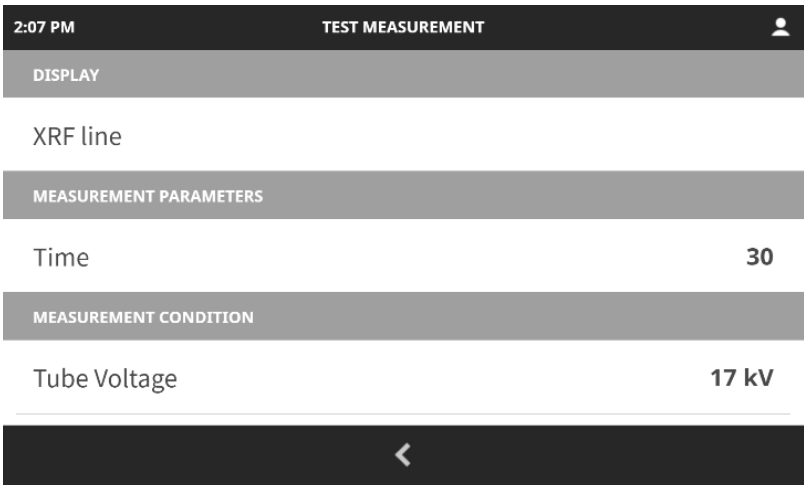
5.6.2. Importing a calibration from another LAB-X instrument.

5.7. Test Measurement

5.7.1. Test Measurement allows for a full spectrum scan of a sample, after which the resulting spectrum scan can be investigated (i.e. element peaks identified, view is zoomed in or out, saved to USB, or a second sample can be measured and the two spectra superimposed.)

5.7.2. Select Method from the pull-down menu, then tap Test Measurement.

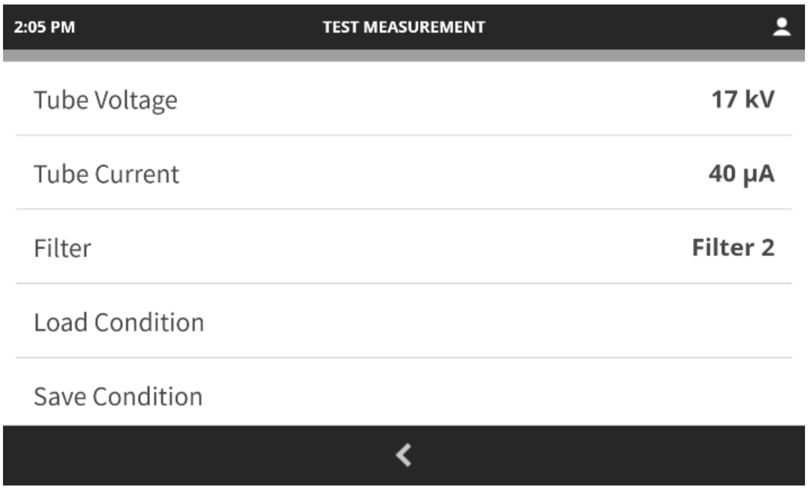
5.7.3. Sample conditions are configured prior to analysis, such as excitation conditions, filter, and analysis time.



XRF line

Time 30

Tube Voltage 17 kV

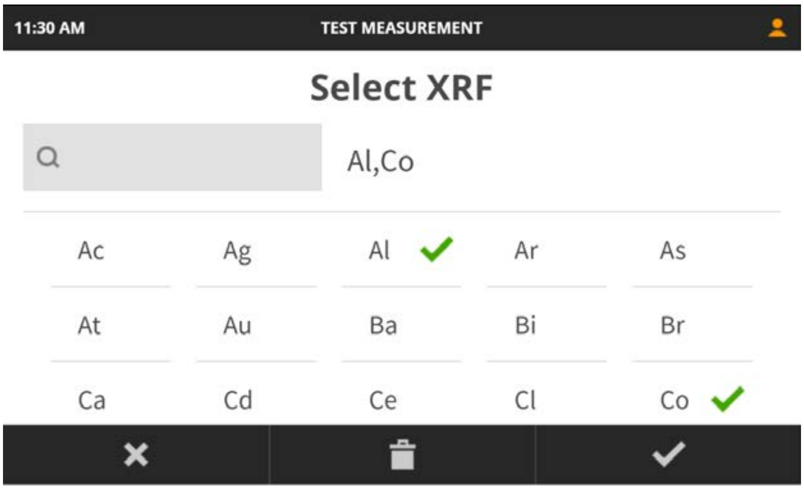


Tube Voltage 17 kV

Tube Current 40 μA

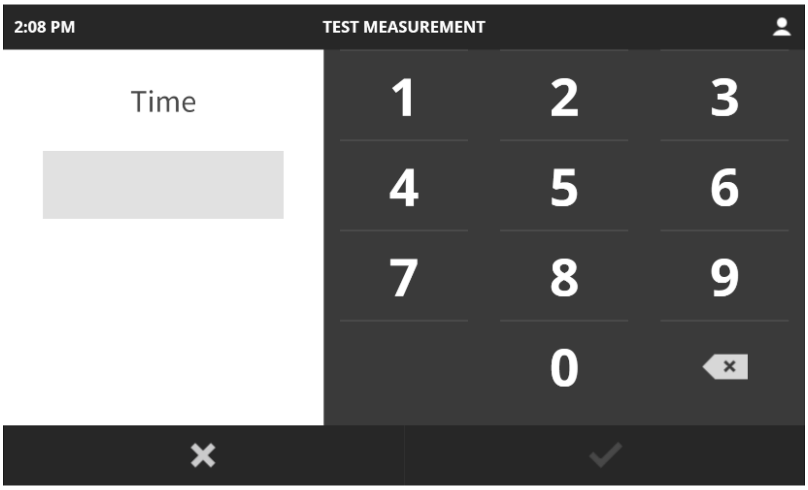
Filter Filter 2

5.7.3.1. Display: choose from XRF line. Elements from Na – U are available; multiple elements can be selected. Tap ✓ to accept input and return to Test Measurement menu.

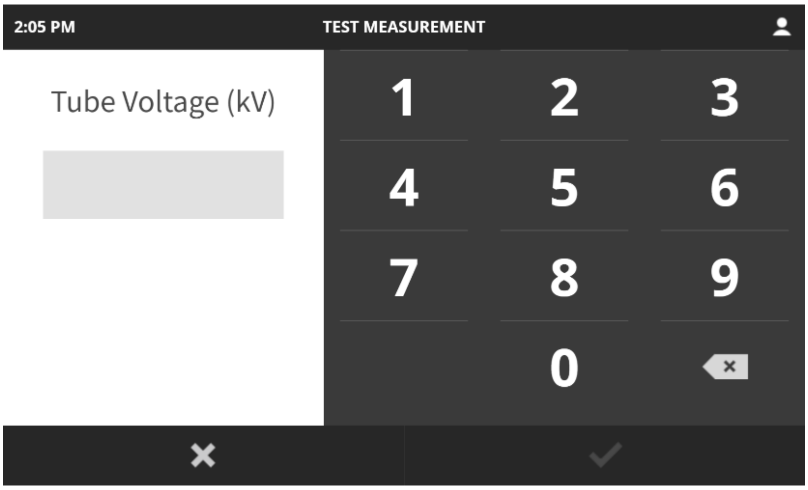


Al,Co

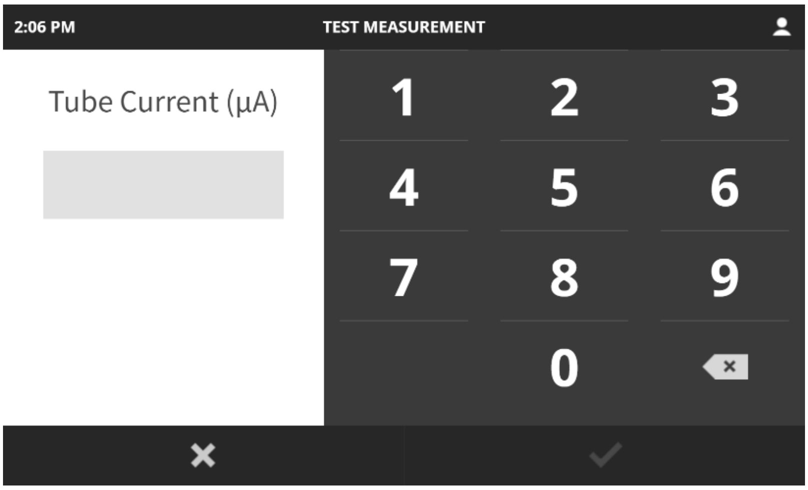
5.7.3.2. Analysis time: Any time can be specified up to a maximum of 999 seconds. Type in desired analysis time, then tap ✓ to accept the input and return to Test Measurement menu.



5.7.3.3. Tube Voltage: Tube voltage can be specified from 4kV to 30kV. Type in the desired voltage, then tap ✓ to accept the input and return to Test Measurement menu.



5.7.3.4. Tube current: Tube current can be specified in 1uA steps from 10uA to 750uA. The maximum value may be limited by the tube voltage previously specified. Type in the desired tube current and tap ✓ to accept the input and return the Test Measurement menu.

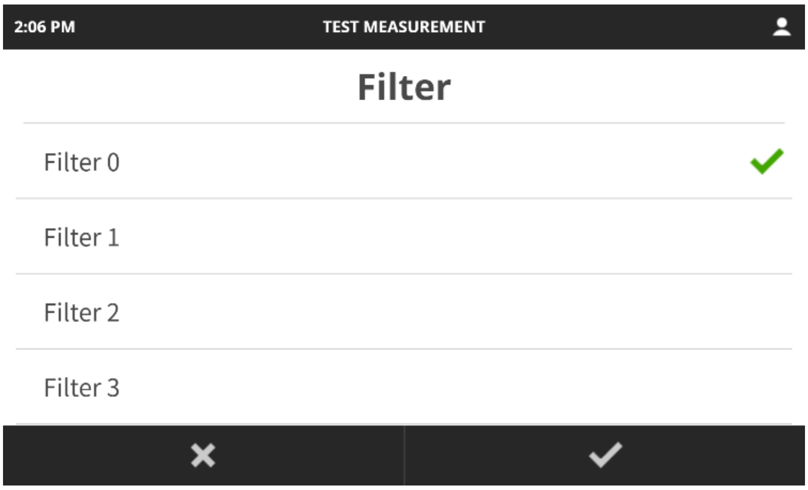


5.7.3.5. Filter: For those systems with filters, choose which, if any, filter should be used. Select a filter from the list of 5 possible filters, then tap ✓ to accept the input and return to the Test Measurement menu.

☞ Information

There are three filter options on the LAB-X5000, depending on options chosen and which X-ray tube is installed.

1. No filters.
2. Ti: (0) Open; (1) W1 Ti; (2) A6 thick Al; (3) Z1 Pd; (4) KF Kapton
3. Pd: (0) Open; (1) A4 Al ; (2) A6 thick Al; (3) Z1 Pd; (4) Z1 Pd KF Kapton


2:06 PM TEST MEASUREMENT

Filter

Filter 0 ✓

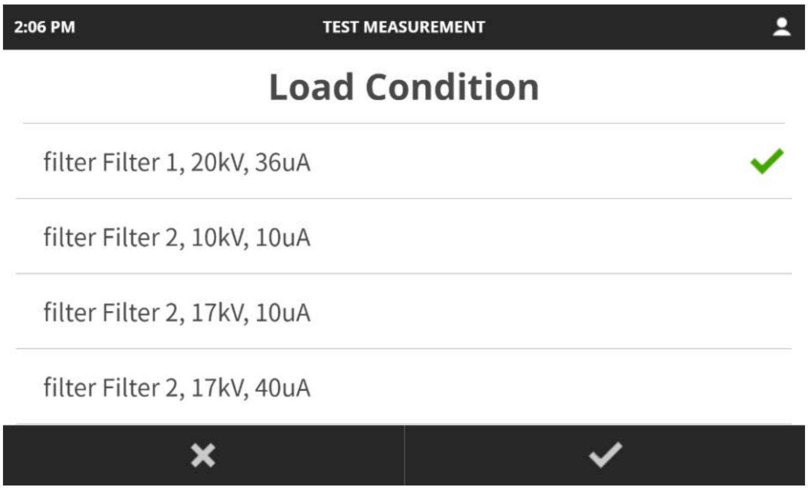
Filter 1

Filter 2

Filter 3

✕ ✓

5.7.3.6. Load condition: Use this option if you wish to choose a pre-loaded condition. Select the conditions you wish to use, then tap ✓ to accept the value and return to Test Measurement menu.


2:06 PM TEST MEASUREMENT

Load Condition

filter Filter 1, 20kV, 36uA ✓

filter Filter 2, 10kV, 10uA

filter Filter 2, 17kV, 10uA

filter Filter 2, 17kV, 40uA

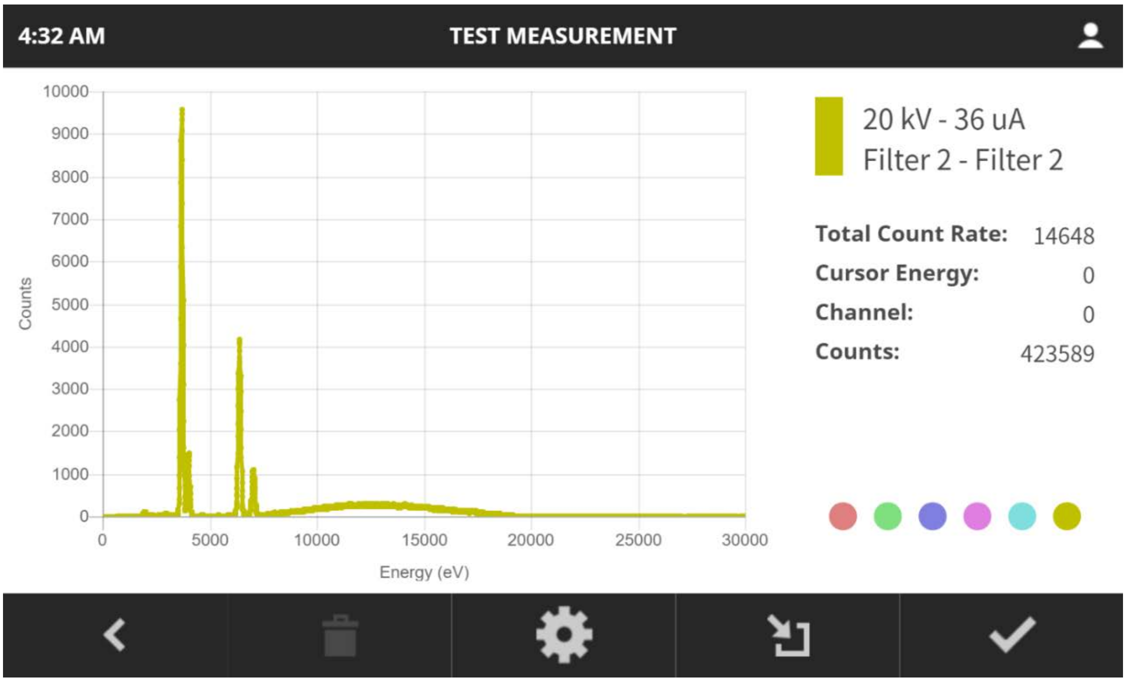
✕ ✓

5.7.3.7. Save condition: This is to save the conditions you have specified into the computer memory to be available later as a preconfigured set of conditions under Load Conditions.

5.7.4. Once all parameters have been set, press the Start button to begin the Test Measurement.

5.7.5. After the sample has been measured, the Test Measurement displays intensity in count on the vertical scale and energy level (in eV) on the horizontal scale.

5.7.6. In some cases, the L or M X-ray lines of high atomic number elements such as Pb could be the peak displayed on the spectrum. It is good practice to measure a blank sample prior to a Test Measurement sample using the same parameters. This will help identify any X-ray tube lines, diffraction peaks, etc. that may occur.



20 kV - 36 uA
Filter 2 - Filter 2

Total Count Rate: 14648
Cursor Energy: 0
Channel: 0
Counts: 423589

5.7.7. To choose a specific element to investigate, tap ⚙ to open the menu to label peaks.

5.7.8. To zoom in: use two fingers, starting together and spreading apart, to select an area to expand. To return to the previous scale, double-tap the spectrum.

5.7.9. Regions of Interest (ROI): to select a particular area of the spectrum, select a scale area that encompasses the energy level of interest and the entire peak of interest. Count rates and information on the right side of the screen will now display information for the selected region.

5.8. Results

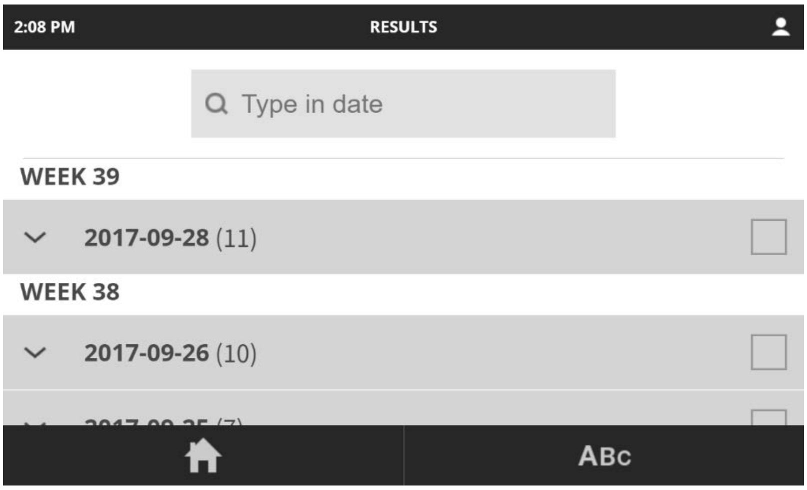
5.8.1. Results for all samples analyzed are stored in the on-board computer memory. Thousands of results are saved before the oldest results are overwritten by new results. It is good practice to backup and export analysis results on a regular basis.

5.8.2. Select Results from the pull-down menu.

5.8.3. All past sample results will be available as a list. Use the 📅 and ABc to toggle between sorting in a chronological list or in alphabetical order by sample name.

5.8.4. Select the ⌄ to show all results in a given week or named with a given name. Alternatively, you can search for a specific sample result by typing part of the sample name into the search box at the top of the screen.

5.8.5. To search, tap on 🔍 and type the date in the date box, or part of the sample name in the sample name box and press ✓ to begin the search.



WEEK 39

⌄ 2017-09-28 (11) ☐

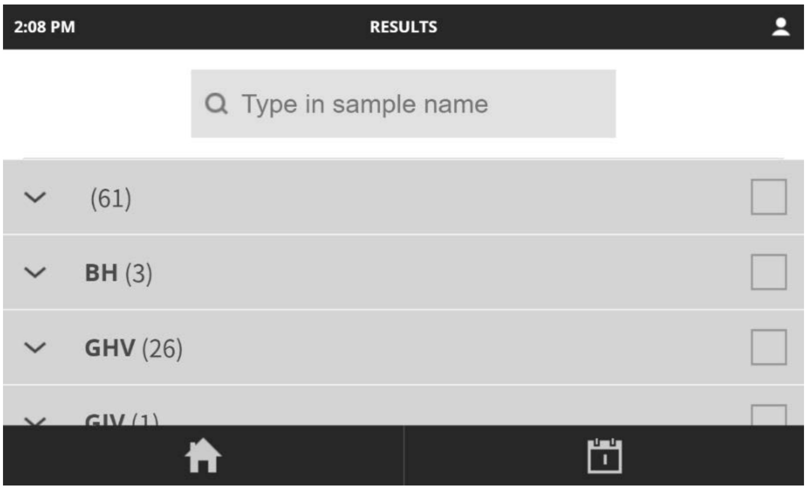
WEEK 38

⌄ 2017-09-26 (10) ☐

5.8.1. To select results, tap on the box at the right side of the sample name or date.

5.8.1.1. Selecting a week date will select all samples analyzed that week. Tap the ⌄ to view all samples analyzed that week. The number of samples in the batch is indicated in parentheses after the date.

5.8.1.2. Selecting a name batch will select all samples analyzed with that name. Tap the ⌄ to view all samples analyzed with that name. The number of samples in the batch is indicated in parentheses after the name.



⌄ (61) ☐

⌄ BH (3) ☐

⌄ GHV (26) ☐

5.8.2. To export selected results, insert a USB stick in the USB port on the right side of the instrument, then tap the Export ↗ button.

Date samples were analyzed

Number of samples in group

Tap box to un/select group

Tap to show individual results

Tap to export checked results

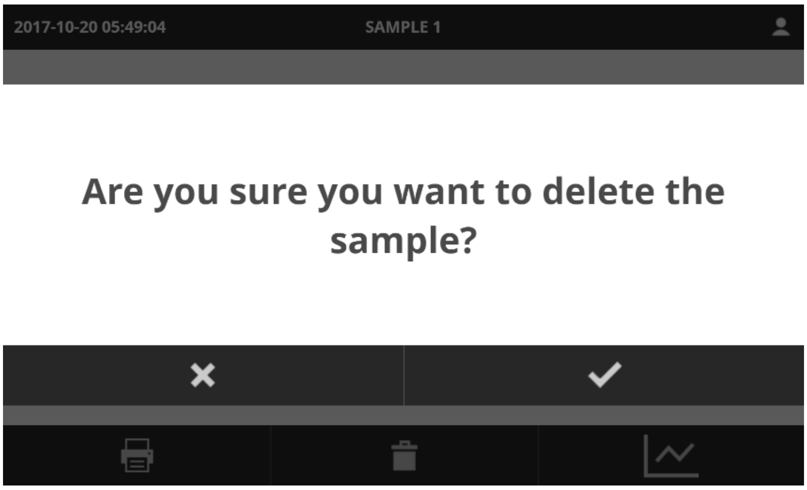
5.8.3. A progress bar will indicate successful transfer of data to the USB stick. Results will save to .csv format.

Exporting to USB
10 / 11

5.8.1. To delete samples, tap the box on the right to select. A green check mark will show for selected samples. Then tap 🗑 in the bottom black bar. A warning box will appear to confirm deletion.

5.8.1.1. Tap ✓ to confirm deletion.

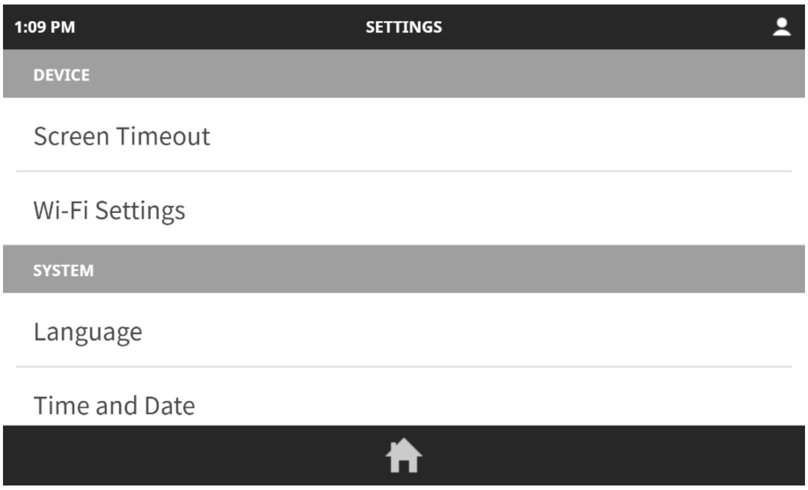
5.8.1.2. Deletion is not reversible.

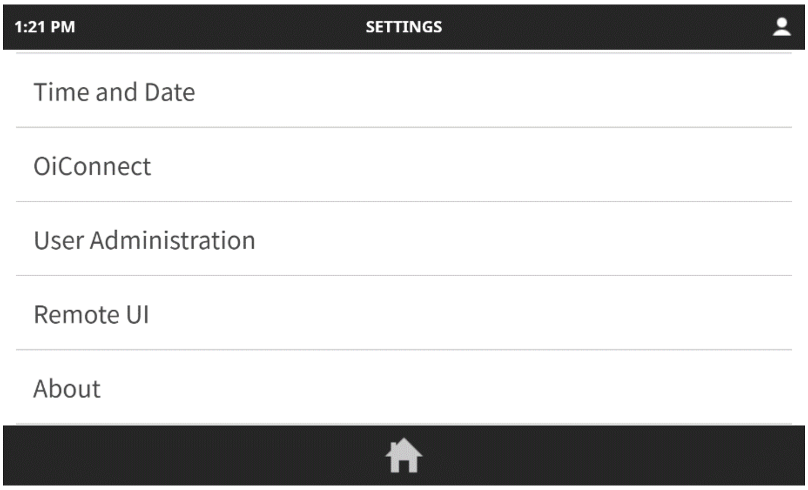


Are you sure you want to delete the sample?

5.9. Settings

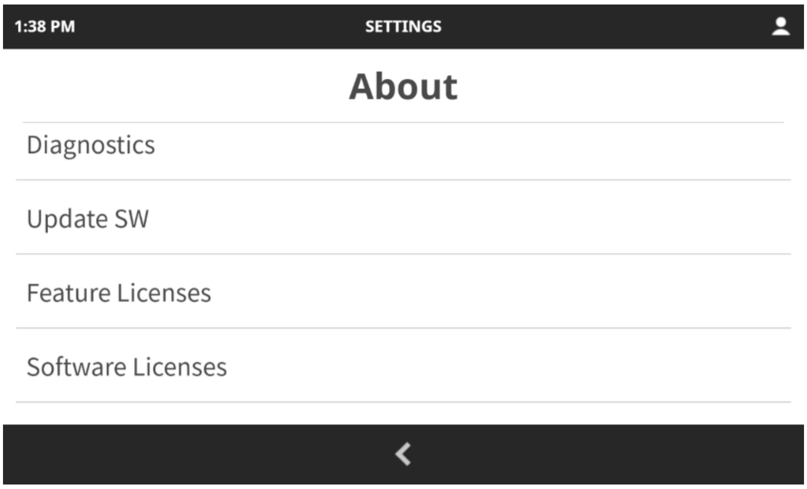
The settings menu contains the options for configuring, updating, and connecting your instrument. Not all options are available at all user levels.

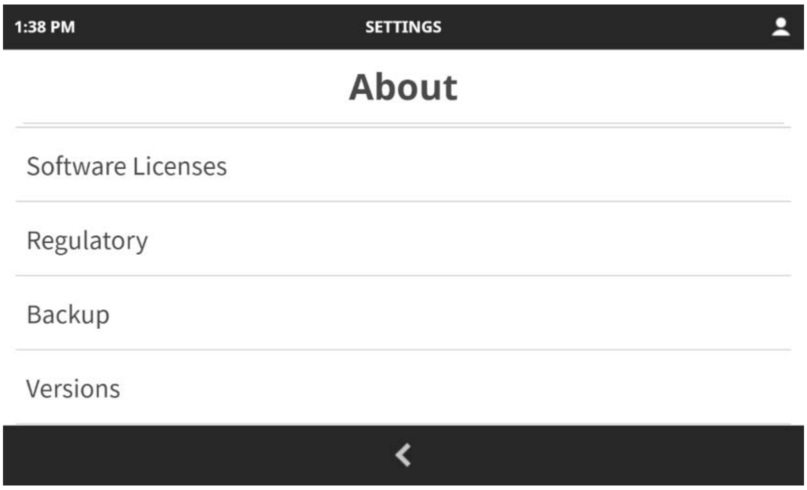




Screen Timeout	Determine the idle time before the screen goes dark.
Wi-Fi settings	Connect to a Wi-Fi network

Language	Choose the language for the instrument	
Time & Date	Set the time and date for the instrument timestamp	
LiveConnect	Set preference for LiveConnect	
	Enable LiveConnect	Toggle on/off
	Upload Old Measurements	Toggle on/off
User Administration	Set PINs for the various user levels	
About	Software, hardware, and service information	
	Diagnostics	Only available to Service
	Update Software	Only available to Manager and Service; requires USB stick with upgrade software.
	Feature Licenses	Only available to Service
	Software Licenses	Only available to Service
	Regulatory	Shows which international and national standards are met by the hardware and software. Does not reference state or local registration or licensing.
	Backup	Create, restore, backups to USB, or export instrument logs. Only available to Manager and Service.
	Versions	View data on current software build





6. Calibration Corrections

If a poor correlation of X-ray intensity against concentration is seen after Regressions are performed, then a correction may be necessary to improve the calibration fit.
Corrections should only be applied where appropriate, i.e. when a Test Measurement shows an overlap of one element on another.

> ☞ **Information**
>
> Some knowledge of X-ray fluorescence theory is required to check the validity of any corrections. If a calibration is over-corrected (i.e. many corrections applied to a single calibration) to make the correlation look good, analysis of unknown samples will produce inaccurate results.

6.1. Blank Subtraction

6.1.1. Blank subtraction 'forces' the regression to pass through for zero concentration at the measured intensity of the blank. A reference blank sample must be measured in the instrument correction section of the regression.

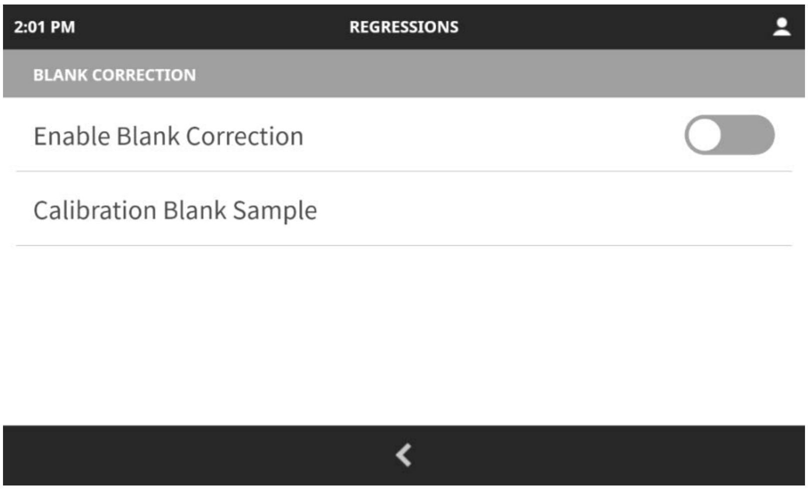
6.1.1.1. To apply a new blank correction to the regression: In the pull-down menu, select Method, then tap Regressions. Choose Blank Correction.

6.1.1.2. A screen to name the blank sample will display. Type in a name for the blank sample.

6.1.1.3. Insert the sample into the sample port and press the Start button. Allow the sample to analyze. Remove the blank sample when it rotates back into view.

6.1.1.4. To apply a new blank for measuring unknown samples, return the to routine analysis screen. Insert the sample to be adjusted in the sample port. Note that if Sample Naming is toggled OFF, you will not be given an opportunity to name the sample prior to analysis. If you wish to name the sample, access the pull-down menu and tap Sample Naming prior to analysis. Press the Start Button. Allow the sample to analyze. Remove the sample when it rotates back into view.

6.1.1.5. The analysis results will show the values of the blank that were subtracted, both on the printout, and on the saved results file.



6.1.2. Restrictions

6.1.2.1. When applying blank subtraction, the same blank must be used consistently. Do not subtract a blank from a 'backscatter' signal because the blank will give the highest intensity reading and correction will not be valid.

6.1.2.2. The blank sample can be a sample, standard or SUS that is representative of unknowns without the analyte(s) present.

6.2. Overlapping Segment

6.2.1. A Test Measurement will show if the element of interest is overlapped by an adjacent or nearby element. If overlap is observed, the intensity measured for this element cannot be correlated to the given concentration because of the interference of this overlapping element. An Overlap Correction will correct for this.

6.2.2. To calculate the amount of overlap onto the element of interest as setting up sample (SUS) containing the overlapping element, but not the element of interest, needs to be measured.

6.2.2.1. By measuring the SUS and performing a new Regression, the software will calculate the overlap factor and apply this during the Regression.

6.2.2.2. The overlap correction can be applied on more than one element.

6.2.3. To use Overlap Corrections, from the pull-down menu, select Method, then tap Regressions. Choose Overlap Corrections. Toggle to enable.

6.2.4. Tap ✚ to add a correction.

6.3. Ratio Correction

6.3.1. This option is used when two element segments need to be ratioed.

6.3.1.1. A primary use of this technique is in the analysis of oils, where oils of different matrix types can be analyzed using one calibration line by applying a ratio of the element being analyzed to a 'backscatter' or background signal.

6.3.2. Select the element to be ratioed.

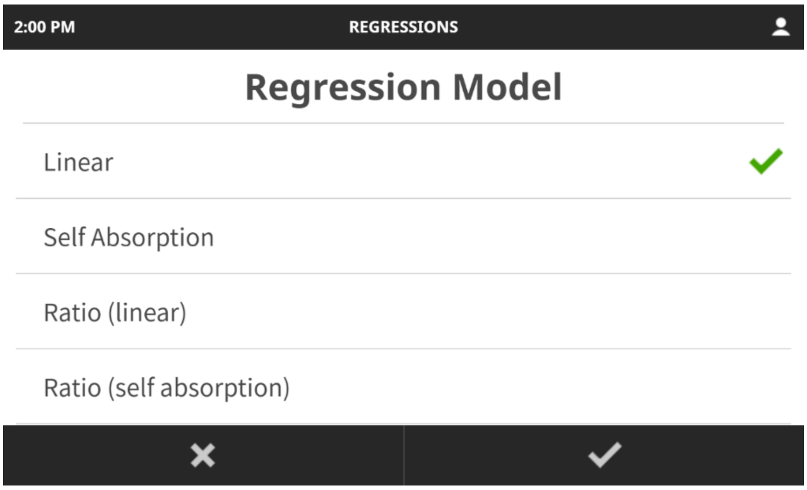
6.3.2.1. Use the pull-down menu to select Method, then tap Regressions.

6.3.2.2. In the Regressions menu, select Regression Model, and tap Ratio (linear) or Ratio (self-absorption).

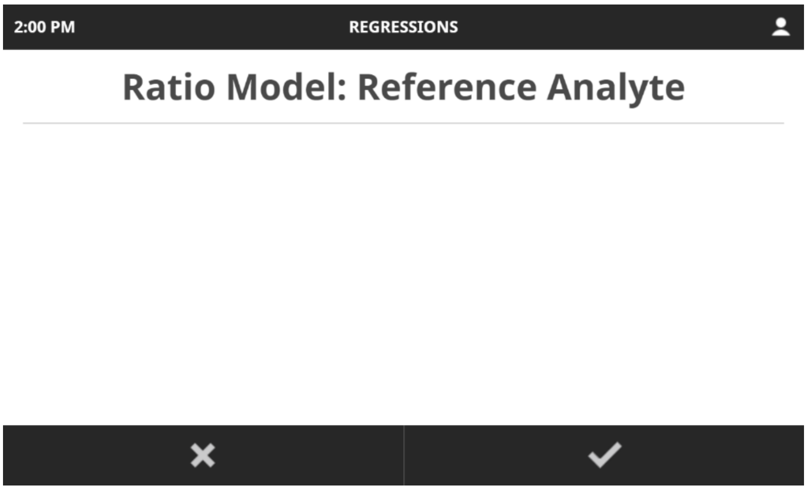
6.3.2.3. Tap ✓ to accept the choice and return to the Regressions menu.

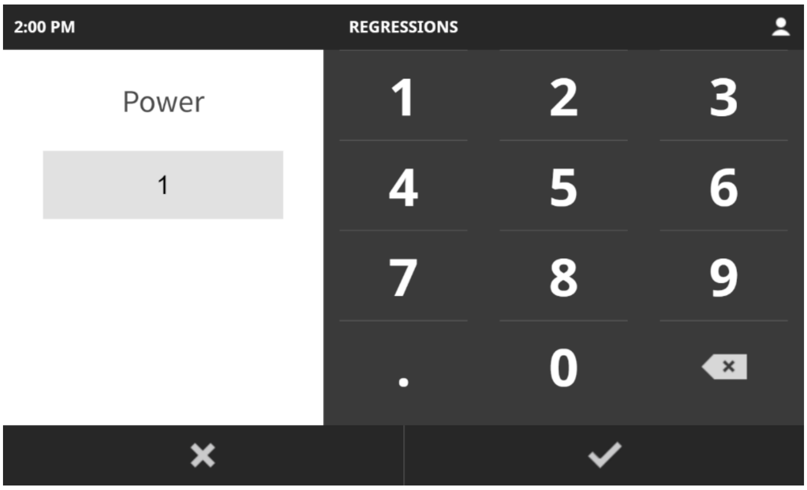
6.3.3. Select the Ratio Model. Tap Ratio Model: Reference Analyte, and select from the available options.

6.3.4. Define the Radio Model: power. This allows the denominator to be raised to the power required between 0.1 and 2. Normally if a simple ratio is required, the value would be entered as 1 (one).



Linear ✓





Power: 1

6.4. Mass Absorption Corrections

6.4.1. This correction should be applied when the intensity of radiation coming from a sample for one element has been modified by the presence of another element in the sample.

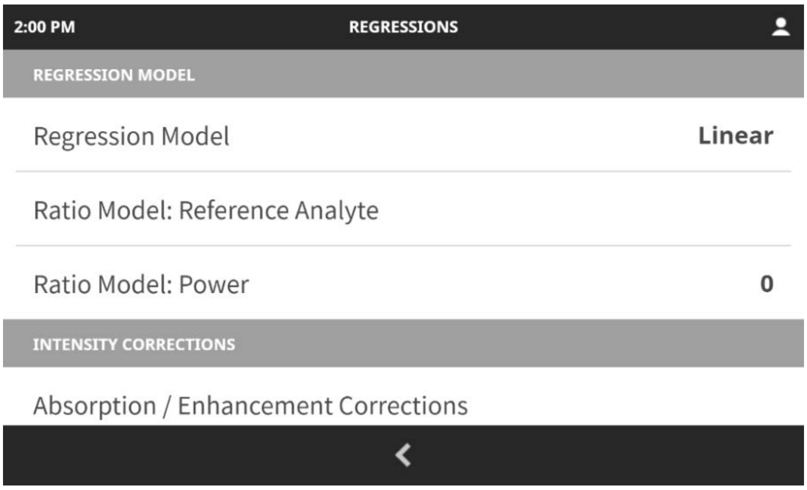
6.4.2. Example: when an element has been fluoresced by incident radiation from the X-ray tube, the element emits secondary or characteristic X-rays. Each element requires a certain minimum energy to excite it, and this is called the absorption edge

6.4.2.1. Ca value = 4.04keV. If a primary X-ray has an energy of 4keV then the calcium will not be excited because the incoming radiation is not energetic enough, but at energies greater than 4.04keV, the Ca will be excited.

6.4.2.2. In a sample containing S and Cl, the Cl radiation will be emitted t 2.622keV. The absorption edge for S is 2.47keV. The S in the sample will be excited by the Cl radiation and the intensity of Cl radiation emanating from the sample will be reduced by the presence of S.

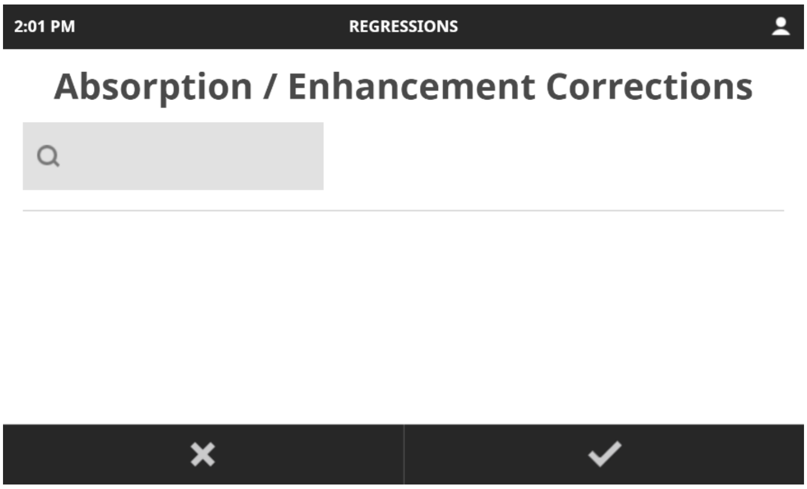
6.4.2.3. A mass absorption correction of S on Cl will correct for the effect.

6.4.3. Select Method from the pull-down menu, then tap Regressions. Choose Absorption/Enhancement Corrections.



Regression Model Linear

Ratio Model: Power 0



6.5. Setting Up Samples (SUSs) for Instrument Corrections

6.5.1. When an X-ray correction is to be applied, such as blank or overlap correction, then a sample or Setting Up Sample (SUS) must be measured before a Regression is performed.

6.5.2. If a Blank Subtraction has been specified, then a blank sample or SUS needs to be measured as an instrument correction.

7. Technical Specification

7.1. LAB-X5000 Series

Device type code	LXDS2860
Rated voltage	100 V AC to 240 V AC
Rated current	1.5A
Rated frequency	47 Hz – 63 Hz
Fuse	T 2A H 250 V
Average power consumption	60W
Operating temperature range	+10 ˚C to +35 ˚C / +50 F to + 95 F
Storage temperature range	-10 ˚C to +50 ˚C / +14 F to + 122 F
Recommended storage temperature	Room temperature
Operating humidity range	20 % RH to 80 % RH
Maximum operating altitude	2000 m / 6500 ft
Embedded storage	NAND 4 GB Flash Memory
Available storage space	2.3 GB
Maximum measurement count	100 000
Dimensions (W x L x H)	440 mm x 520 mm x 155 mm
Weight	20 kg
Pollution Degree	PD2
Ingress Protection degree	Not classified
Overvoltage category	II

7.2. X-Ray Tube

Target material	Palladium (Pd) or Titanium (Ti)
Window thickness	0.005 in
Anode voltage range	4 kV to 30 kV
Maximum anode current	0.75 mA
Maximum anode power	3.0 W
Nominal filament voltage	2.5 V
Maximum filament current	1.3 A
Minimum counts	24000 1/s
Ambient temperature range	0˚C to +40 ˚C / +32 F to + 104 F
Maximum package temperature	55 ˚C / +131 F

8. Appendices

Appendix A: Access for each PIN user level

	Operator	Advanced Operator	Manager
Feature access			
Calibration			x
Restandardization		x	x
Blank subtraction	x	x	x
Basic diagnostics	x	x	x
Advanced diagnostics (if relevant)			
OiConnect setup A11			x
Pull-down menu: Toggle Std display		x	x
Pull-down menu: Toggle Pass/fail display		x	x
Pull-down menu: Toggle Wi-fi		x	x
Pull-down menu: Toggle Std sample naming		x	x
Pull-down menu: Toggle auto printing		x	x
Pull-down menu: Toggle font size	x	x	x
QC check sample		x	x
Software update			x
Select method	x	x	x
View stored results	x	x	x
Delete last result	x	x	x
Test measurement	x	x	x
Backup			x
Restore			x

Appendix B: Sample Preparation

X-ray fluorescence spectrometry is a non-destructive method of analysis and it is possible to measure a wide range of sample types using this technique. The nature of the sample is a very important can adversely affect the accuracy of the analysis, however sample preparation is usually very simple. Basic requirements are the sample should have a flat surface, be homogenous, have an adequate thickness, and fit into the sample port of the instrument.

The LAB-X5000 will accept samples of diameter between 28 and 41 mm and height less than 39mm. Information in the preparation of several different sample types is given below. If information of a more specific nature is required, contact your local Hitachi High-Tech Analytical Science representative.

8.1. Solids

8.1.1. Normal machine shop techniques such as lathe turning, milling, and surface grinding can all be used to obtain a suitable smooth and flat surface to give consistent analytical results.

8.1.2. Lathe turning is the most widely applicable method (e.g. brasses, steels, nylons) although for materials with a tendency to smear, such as highly leaded brasses or antimonial lead alloys, an end milled surface is preferred. Surface grinding is generally highly reproducible.

8.1.3. Consistency of preparation is important and unknown samples should be prepared in the same way as the standards. Non-homogeneity should be eliminated or reduced as well as is possible.

8.2. Powders and Minerals

8.2.1. Most powders can be analyzed by simply pouring the powder into a sample cup.

8.2.2. The sample cup should be lightly tapped on a clean bench to settle the contents after pouring.

8.2.3. Non-homogenous samples, low-density ('fluffy') samples or powders with larger particles must be ground and compacted into a pellet. Pellets provide a flat, smooth sample of consistent density that is readily reproduced, minimizing error in analysis.

8.2.4. For information on pelletizing samples, contact your Hitachi High-Tech Analytical Science representative.

8.3. Liquids

8.3.1. Liquids are poured directly into LAB-X sample cups.

8.4. Organic Solids

8.4.1. Food materials such as chelates, organo-metallic additives, catalysts, and trace catalysts are very soft and cannot be prepared in standard mills.

8.4.2. Powdered samples can be milled in a shaker/mixer mill to homogenize.

8.4.3. Very soft materials, such as fats, can be homogenized in a food processor and then pressed into pellet form.

8.4.4. Calibration lines for elements in light organic matrices can be curved, leading to decrease in sensitivity at high levels. To dilute these samples to achieve linear calibrations, use starch or sugar.

8.5. Papers

8.5.1. A special device is available from Hitachi High-Tech Analytical Science for analyzing paper samples on the LAB-X5000. It was originally designed for the measurement of silicone coating thickness but can be used for other thin samples.

8.5.2. The sample holder includes a special support that reduces unwanted background signals.

8.6. Sample Cup Assembly

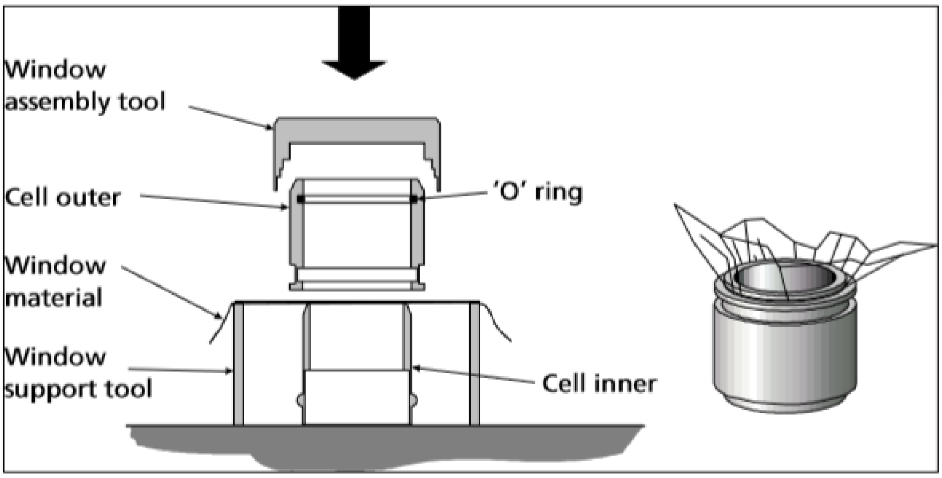
8.6.1. Sample cups are assembled as follows:

8.6.1.1. Place the white polypropylene inner ring, with leading edge toward the top on a clean, flat surface.

8.6.1.2. Position the window support tool around the inner ring.

8.6.1.3. Lay a piece of window material over the support tool so that the material is flat at the rim of the inner ring.

8.6.1.4. Push the polypropylene outer ring vertically down onto the inner ring. If necessary use the assembly tool to help push the outer ring down.



8.6.1. When assembling a sample cup or secondary window it is important to use a clean piece of window material.
8.6.2. Always visually inspect the assembled cell for leakages when filled with liquid.
8.6.3. Always replace the window material if it is creased or wrinkled.
8.6.4. Plastic sample cups are disposable after use.

(IMPORTANT) NEVER LEAVE A SAMPLE CUP FILLED WITH LIQUID IN THE INSTRUMENT SAMPLE PORT AFTER COMPLETION OF A MEASUREMENT.

8.6.5. Use a sample cup lid for volatile samples such as gasoline to reduce evaporation. Evaporation usually results in a change in composition of the sample, which may lead to incorrect results.

8.7. Filling Sample Cups

8.7.1. To measure elements below calcium in the periodic table a minimum sample depth of 2-3mm is sufficient
8.7.2. For heavier elements, fill the cups to the line in the interior of the sample cup to ensure a constant volume.

8.8. Testing for Sample Preparation Errors

8.8.1. To test for instrument precision, place a sample of material into the instrument and analyze it ten times in a row without removing it from the instrument. Calculate the standard deviation of these measurements for the instrument repeatability (precision).
8.8.2. To test for sample presentation error, place a sample of material into the instrument and analyze it ten times in a row, removing it from the instrument and replacing it back into the sample port for each analysis. Calculate the standard deviation of these measurements for the error introduced by sample presentation.
8.8.3. To test for sample preparation error, prepare ten identical samples of the same material using the intended sample preparation procedure. Analyze all ten samples in a row. Calculate the standard deviation of these measurements for the error introduced by sample preparation.
8.8.4. To test for sample thickness error, analyze a sample, then double the thickness of the sample by either stacking an identical solid sample on it, or increasing the depth of the

liquid or powder sample in the sample cup and analyze again. An increase in the signal implies that the original sample was not thick enough for the X-ray penetration depth, and samples must be made thicker, or of constant thickness.

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