Koehler
INSTRUMENT COMPANY, INC.

K47900
Benchtop EDXRF Elemental Analyzer

Operation and Instruction Manual

REV A

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1 Introduction

The Koehler EDX1000 (K47900) is a Benchtop Energy Dispersive X-Ray Fluorescence (EDXRF) Spectrometer for the elemental analysis of sodium through uranium in solids, liquids, and powder samples as well as thin film coatings on solid substrates.

In EDXRF low energy "soft" X-rays (1-50keV) are emitted from an X-ray tube. These source X-rays enter the sample and cause the atoms in the sample to fluoresce their own characteristic low energy "soft" X-rays. These fluorescent X-rays are captured by the detector and counted by a multi-channel analyzer. The EDX1000 software then calculates the concentration of each element present in the sample.

This manual provides important information regarding safety, technical reference, installation requirements, operating condition specifications, user facility resource requirements, and operating instructions for the K47900 EDXRF Analyzer. This manual should also be used in conjunction with applicable published laboratory procedures. Information on these procedures is given in section 1.2.

1.1 Koehler's Commitment to Our Customers

Providing quality testing instrumentation and technical support services for research and testing laboratories has been our specialty for more than 50 years. At Koehler, the primary focus of our business is providing you with the full support of your laboratory testing needs. Our products are backed by our staff of technically knowledgeable, trained specialists who are experienced in both petroleum products testing and instrument service to better understand your requirements and provide you with the best solutions. You can depend on Koehler for a full range of accurate and reliable instrumentation as well as support for your laboratory testing programs. Please do not hesitate to contact us at any time with your inquiries about equipment, tests, or technical support.

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1.2 Recommended Resources and Publications

1. American Society for Testing and Materials (ASTM)
   100 Barr Harbor Drive
   West Conshohocken, Pennsylvania 19428-2959, USA
   Tel: +1 610 832 9500 • Fax: +1 610 832 9555
   http://www.astm.org • email: service@astm.org

   ASTM Publication:

   - ASTM D4294: Standard Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry
   - ASTM D5059: Standard Test Method for Lead in Gasoline by C-Ray Spectroscopy

2. International Organization for Standardization (ISO)
   1, rue de Varembé
   Case postale 56
   CH-1211 Geneva 20, Switzerland
   Tel: 41 22 749 01 11
   Fax: 41 22 733 34 30
   http://www.iso.org

   ISO Publication:

   - ISO 20847: Determination of Sulfur Content of Automotive Fuels – Energy Dispersive X-Ray Fluorescence Spectrometry
   - ISO 8754: Determination of Sulfur Content – Energy Dispersive X-Ray Fluorescence Spectrometry

3. Energy Institute (IP)
   61 New Cavendish Street
   London, WIM 8AR, United Kingdom
   Tel: 44 (0)20 7467 7100
   Fax: 44 (0)20 7255 1472
   http://www.energyinstpubs.org.uk/

   IP Publication:

   - IP 336: Determination of Sulfur Content – Energy Dispersive X-ray Fluorescence Method
   - IP 496: Determination of the Sulfur Content of Automotive Fuels – Energy Dispersive X-ray Fluorescence Spectrometry

1.3 General Safety

All users are instructed to read Section 8 of this manual pertaining to radiation safety prior to turning on the device!

External Warning Labels:

CAUTION: Risk of Danger
Consult information in manual.

CAUTION: Possible Hot Surface
Label is a warning of a possible hot surface. Consult information in manual.

CAUTION: Electrical Shock Hazard
Consult information in manual.

CAUTION: X-rays on Lamp
Consult information in manual.

CAUTION: X-rays Produced when energized. Operation by qualified personnel only.
-OR-
CAUTION: X-Radiation Attention: Rayonnements X
Or similar, consult information in manual

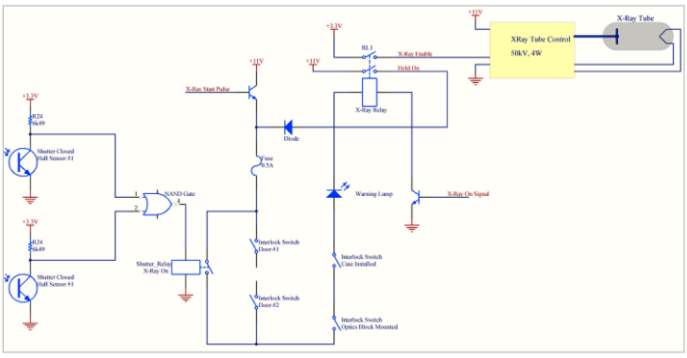
1.4 Component Descriptions

X-Ray Source (Tube and Generator): The EDX1000 incorporates a miniature 50kV (4 Watt) X-ray tube for analysis. The low power consumption X-ray tube source and its supporting generator are self-contained and self-shielded and fully interlocked by the EDX1000 to ensure safety of the operator.

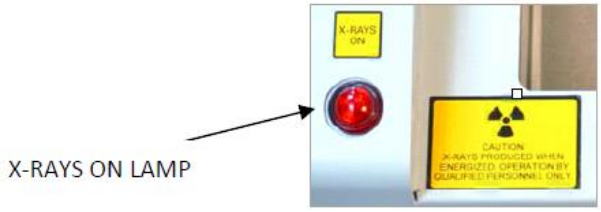
Interlocks: The system interlocks will prevent the X-ray generator from producing X-rays if and when the interlock circuit chain has been interrupted. The interlocks will shut down the tube voltage and filament current, thereby disabling the X-ray output. The interlock series circuit chain is also fuse protected.

1. Analysis has started but the sample chamber door is not closed. (CLOSE DOOR screen prompt).
2. The X-ray tube (generator) assembly is not mounted to the optics assembly.
3. The instrument cover has been removed.
4. The LED X-rays on warning lamp indicator is not functioning.

CAUTION: There are no internal user serviceable parts or fuses inside the instrument.



X-Rays On Lamp: The X-rays on warning indicator is located on the front left side of the instrument. The indicator lamp is a component of the interlock circuit.



Automatic Door Lock: The sliding sample chamber door is locked and unlocked during analysis by a software controlled motorized latch. The interlocks do not allow the analysis to start with the sample chamber door open. A “CLOSE DOOR” warning will also appear. Once the sample chamber door is closed, the door will automatically lock and will not unlock until the analysis is completed or canceled. *The latch is spring loaded and will automatically release if the instrument loses power.

Do NOT Force Door Open!

Power ON / Power OFF: The EDX1000 power switch is located on the right rear side of the instrument. Before turning the instrument switch off, we suggest using the "Restart" button once located on the Utilities screen. This is to make sure that any data storage or changes has been updated before powering down the instrument. There you will be prompted to either turn off the unit or restart. To help avoid software file corruption, do not shut off power during any save or backup operation.

Hazards: Beryllium (Be) windows can be found on both the X-ray tube and detector windows. Do not attempt to disassemble these devices.

Detector: The EDX1000 uses a compact Energy Dispersive X-ray Fluorescence (EDXRF) semi-conductor detector with a high performance two-stage thermoelectric cooler (Peltier) for great resolution and X-ray sensitivity.

CAUTION: Care must be taken NOT to touch the ultra-thin beryllium detector window under any circumstances. The Beryllium window may shatter with even the slightest form of physical contact, resulting in the need for a new detector replacement.

IMPORTANT: In the event of a spill or contamination; Do NOT attempt to clean the beryllium window surface with anything, including a cotton swab or soft brush, etc.

Tube Filters: The EDX1000 has up to 5 tube filters to optimize performance by selectively reducing the scattering from the X-ray tube thus creating "quiet spots" in the background signal.

On the Hardware Monitor screen, the filter wheel positions are labeled 1 – 7 starting with the shutter position as 1, open position as 2, etc.

Filter	Typical Voltage (kV)	Typical Elements to be Measured
Open	6.5	**Na – Cl K-lines** Zr, Mo L-lines, alternate for K, Ca K-lines in some applications
A	30	**K – Mo K-lines** **Sn – U L-lines**
B	50	**Ru – Pr K-lines** Alternate for K-Br K-lines in some applications
C	35	Alternate for K-Mo K-lines in some applications
D	15	Alternate for low levels of Ti – Ni K-lines in some applications
E	12	Alternate for low levels of K – Ti K-lines in some applications

External Power Supply: The EDX1000 is powered by an external 12 Volt output 120W AC input (100-240VAC) DC single output desktop power supply. The power supply is conveniently secured to the back on the instrument via a mounting bracket. The supply uses a standard universal 3 pole AC input power cord (IEC320-C14) with a wire gauge of 18 AWG or 16 AWG. The cord should also have a temperature rating of 105°C or higher and minimum 10 amp rating. Do NOT use any inadequately rated power cords.

The supply also features Short Circuit / Overload / Over Voltage and Over Temperature protections. There is also a LED power on indicator on the fully enclosed plastic case. Class I (with earth pin).

Fuses: There are no user replaceable fuses in the EDX1000.

Maintenance and Inspection: There are no internal user serviceable or replaceable parts inside the EDX1000 Benchtop EDXRF Analyzer. Printer Paper and sample window film are considered consumables. The sample window film may occasionally require replacement if it gets dirty. However, extreme care must be taken to avoid even the slightest physical contact with the ultra-thin beryllium detector window during film replacement.

The EDX1000 does not require any extraordinary inspections other than the periodic radiation survey as required by any local governing authority. If a failure is suspected, contact Koehler Instrument Company in the US or the distributor for support outside the US.

Cleaning: The EDX1000 is designed to work in most labs and light industrial environments. Should the need arise to clean any portion of the exterior such as the panel display windows or warning labels, we recommend gently wiping the unit only using mild soap and water. Also, it is important to take care and not allow any cleaning solution to spill or seep into any portion of the instrument.

Solution Spills: Liquid spills inside the sample chamber should always be avoided. A leaking sample cup should never be used in the EDX1000 under any circumstances. If a sample cup is found leaking, do not use it but rather prepare a new sample cup with film then transfer the contents to the new cup.

Sample cups containing liquids should never be allowed to remain inside the sample chamber beyond the necessary analysis time and MUST be

removed immediately after analysis. Some liquid samples may chemically attack the X-ray protective film and cause a leak into the sensitive X-ray optics located underneath the sample window resulting in significant damage to the instrument.

In the event a leakage spill onto the optics assemble has occurred DO NOT ATTEMPT TO CLEAN THE OPTICS ASSEMBLY. Contact Koehler Instrument Company or your local distributor.

Decommissioning: Do NOT dispose of instrument. To decommission the instrument please contact Koehler Instrument company for instructions.

Consumables, Accessories: The EDX1000 Benchtop Analyzer comes with the following standard accessories:

- Caution Sign (X-rays)
- Sample Cups, 3 Piece, Bag of 100
- XRF Slide Reference Chart
- Mylar Film Roll, 6µm, 3”x300’
- EDX1000 Sample Window Ring (32mm cup)
- EDX1000 User Manual
- Printer Paper (5 rolls), 3” Wide
- O-Rings (2 each), 1.275”ID x 0.70”
- EDX1000 Sample Changer Door Shipping Block
- Standards Storage Case
- EDX1000 Flat Sample Window Ring
- Teflon Background Standard for Application Support
- Gain Cal Sample for Service and Application Support
- Instrument AC Power Cable

Koehler Instrument Company offers consumables for the EDX1000 Benchtop Analyzer such as X-ray film, sample cups, accessories, printer paper and user replacement items such as window rings, etc. For a complete list of available consumables and accessories refer to Addendum section located at the back of this manual (Section 9).

2 Instrument Setup

The EDX1000 arrives fully assembled from the factory. In order to ensure proper setup: remove the secure door shipping block, carefully remove the protective sample window cover over the sample aperture, and install the sample window film and the window. Next, load the printer paper and make a few simple rear connections such as: attach the +12 volt power supply connector to the instrument first, then attach the AC power outlet power cord to the external power supply. Other optional connections can be made at this time such as attaching the network cable, helium purge, and camera connections.

It is important to keep and store the original shipping box, protective foam inserts, door shipping locks, and window cover in a safe area away from heat and moisture. Should you encounter any problems or questions relating to installation and setup please contact your local Koehler distributor or representative.

Equipment Modifications and Replacement Parts: Any modification or alteration of this equipment from that of factory specifications is not recommended and voids the manufacturer warranty, product safety, performance specifications, and/or certifications whether specified or implied, and may result in personal injury and/or property loss. Replacement parts must be O.E.M. exact replacement equipment.

Unit Design: This equipment is specifically designed for use in accordance with the applicable standard test methods listed in section 1.2 of this manual. The use of this equipment in accordance with any other test procedures, or for any other purpose, is not recommended and may be extremely hazardous.

2.1 Instrument Location Requirements

The EDX1000 is designed for safe operation under the following minimum conditions:

- Indoor use (avoid extreme direct sunlight)
- Altitude up to 2000 meters (6550 ft) above sea level
- Temperature Range 10°C – 40°C or (50°F - 95°F)
- Maximum relative humidity of 85%, non-condensing
- MAINS supply voltage fluctuations up to ±10% of nominal voltage
- Transient over voltages typically present on MAINS supply.

For optimum performance we recommend installation with the following conditions:

- Table top should be level, sturdy and sufficient to support at least 40 lbs (18.15kg)
- Requires no device mounting
- Un-interruptible power supply
- Position instrument to allow sufficient clearance of 4 or more inches in the back of instrument for easy access to power switch and other connections

Ventilation: Make sure there is sufficient spacing of at least 4 inches in the back and 2 inches on each side of the instrument to allow room air to flow freely around the instrument. No other air, flow mechanism, gasses or cooling liquid is required to cool and ventilate the instrument and its components. The helium purge is designed to purge the room air from the optical path in order to enhance the detection sensitivity of low energy x-rays. The purge does not function as a ventilation or cooling system.

Dust: Keep fibrous dust, sand, soil and metallic dust out of the room.

Atmosphere: The atmosphere must be free from corrosive gases such as SO_2, HCL, and H_2S.

Vibration: Vibration must not be sensed by a human body.

Electrical Noises: Excessive electrical noises may affect the instrument, take countermeasures against them. Typical countermeasures against noises are described below for your information:

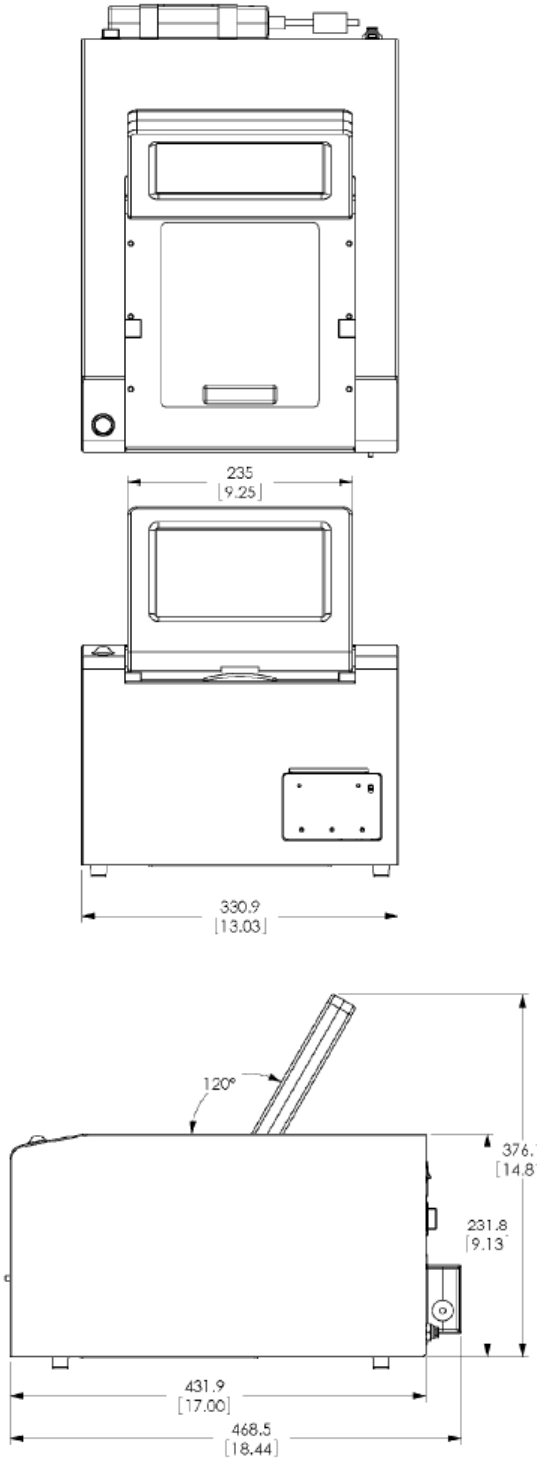
AC Power Circuit Considerations: When an electric furnace, high frequency furnace, SCR furnace, emission spectrophotometer, electric welder or the like is also connected to the same AC power circuit, electronic noises may affect the EDX1000. In this case, connect either the EDX1000 or the other devices to a different AC power circuit. If the devices cannot be separated the best solution is to install the EDX1000 to a UPS.

Ground System: Grounding is provided by the wall outlet using a three-prong plug. If grounding is not available by the plug and wall outlet, consult an electrician to determine proper ground connection, When another device that generates noises is connected to the same ground system or when the ground is independent but imperfect, noises may affect the instrument. Use a ground terminal with grounding resistance of 30 ohms or less.

RF Interference: The EDX1000 has been designed such that is complies with the standard concerning the electromagnetic compatibility (EMC) that has been established to minimize the emitted electromagnetic waves and maximize resistance to external electromagnetic waves. Do not install the EDX1000 within 30 feet of RF interferences, such as:

- High-output electric power instrument (electric welder, etc.)
- High-frequency instrument
- SCR instrument
- Emission spectrophotometer

2.2 Instrument Dimensions and Weight



2.3 Back Panel Connections

CAUTION – Always connect the External Power Supply connection to the instrument first before connecting the AC power cord to the AC outlet.

External Power Supply Connection 12 Volt 120W Desktop Power Supply

IEC320 Power Supply AC Power Cord Connector

RJ-45 Network Connection

External USB Port Connector

Helium Purge Connector (optional)

2.4 Printing and Paper Loading

The EDX1000 has an internal thermal printer for printing hard copy output results. The printout option is selected in the "Settings" of the Application Builder. The printer uses 80 mm wide thermal paper with a 72 mm print line width at a resolution of 576 dots per line. Both the thermal print head and the paper drive mechanism move during the printing process. The printer also has a built in paper optical sensor to detect the presence of the paper roll. There is also a platen detection switch used to detect if the printer door is secured.

Note: The chemically treated side of the thermal printer paper is the outward side of the paper. The printer does not detect if the paper is loaded incorrectly.

To tear off the printed results, gently pull the printer paper upwards right or left at a 45° angle.

Loading the Printer Paper

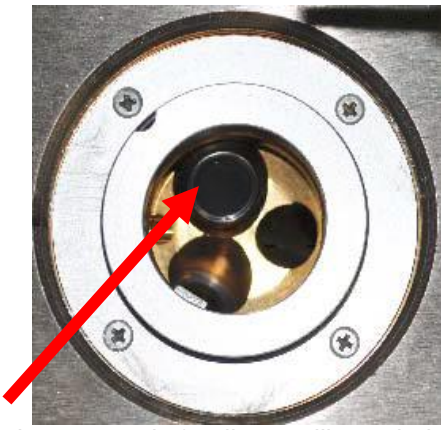
1. To open the printer paper compartment press the printer release button located on the front door panel then pull the door down.

2. Manually roll off about 4 inches of paper from the new roll then insert a fresh roll of paper as shown below. Note the chemically treated side (top pf roll) is held against the top cover of the analyzer during loafing and closing the door.

Printer Platen located on door is shown above. Note for best printing results clear any debris if found on platen before closing the door.

2.5 Sample Window Film

Periodically the sample X-ray film may get dirty and affect results and may require replacement. To replace the film the first step is to gently pry upward the sample window from the opposite side away from the detector.
Use caution not to touch the ultra-thin beryllium detector window.



This is the extremely fragile Beryllium window on the detector.

Next, lay the X-ray sample window film over the optics chamber smooth and flat. Position the sample window on the top of the optics assembly base then carefully push down evenly until the sample window snaps in place. Make sure there are no wrinkles in the film as shown below.

Use an x-acto knife to trim the excess window film around the outer edges of the sample window ring.

Above is the flat window ring with trimmed window film.

3 Software User Interface

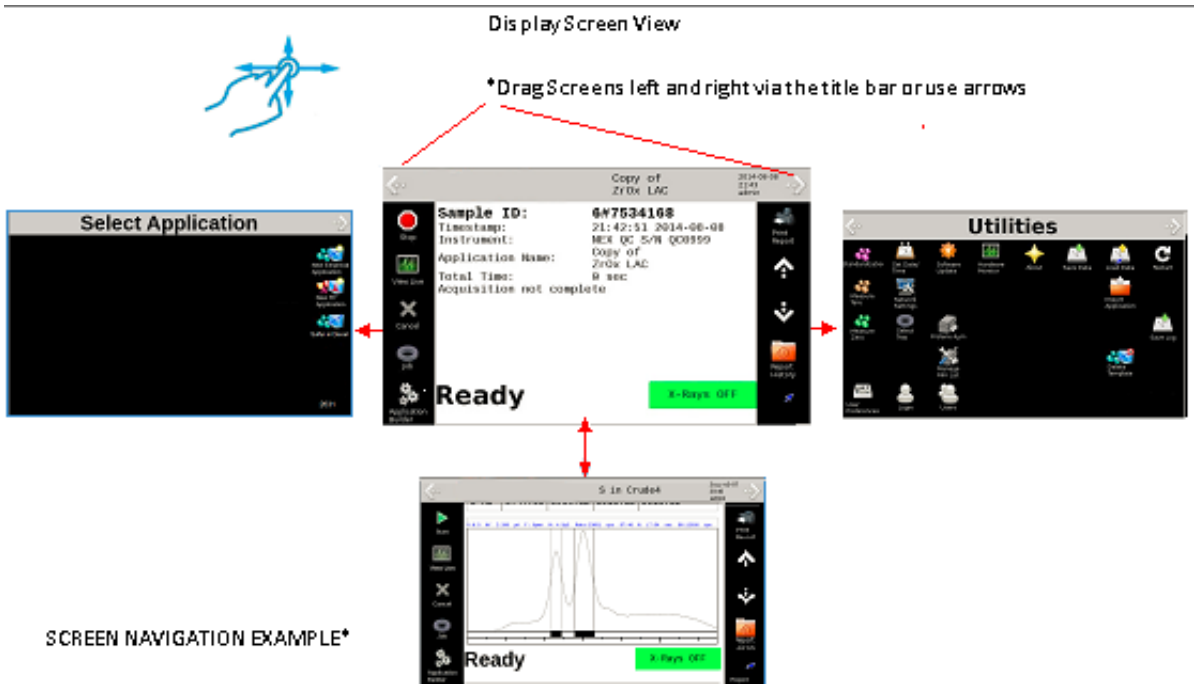
The EDX1000 uses an embedded computer running Linux with touch screen interface. The analyzer's software is a full screen application with no desktop windows, frames, minimize, maximize or [X] close buttons. The physical display panel is an 8inch WVGA 800 x 480 pixel touch screen interface. All functions are accessible via touch (tap). The user interface has available multi-language settings for all (UI) user interface items.

3.1 Screen Navigation

The three main software screens are the **Select Applications**, **Main** screen, and **Utilities**. Additional menu items are located on each of these screens. The screens may be changed via the screen drag technique (see example on next page) or by using the left, right, up, and down screen navigation arrows. After start-up and login, the screen may default to Main screen if selected.

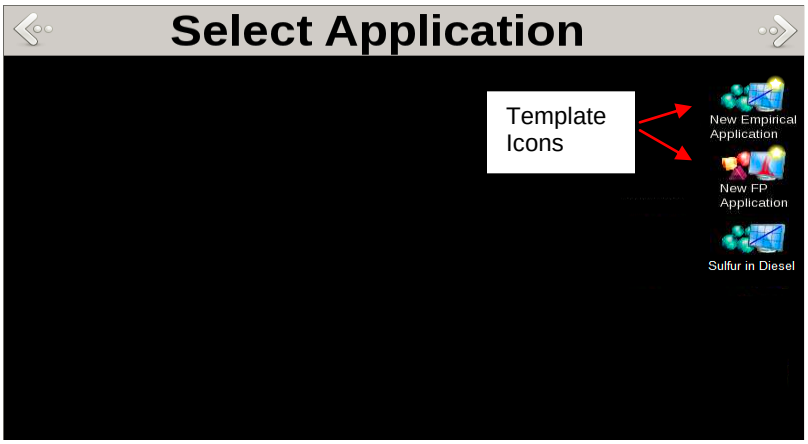
To switch to Select Application press and hold then drag the far left side of the screens title bar to the right or press the right arrow. Tapping the icon to select an application will open to the applications main screen. To return back to the Main screen, press and hold the right side of the bar and drag to the left or press the left arrow.

To select the Utilities screen, hold the right side of the title bar and drag to the left or use the right arrow. To return back to the main screen press and hold the left side of the title bar and drag to the right or use the left arrow.



3.2 Select Applications

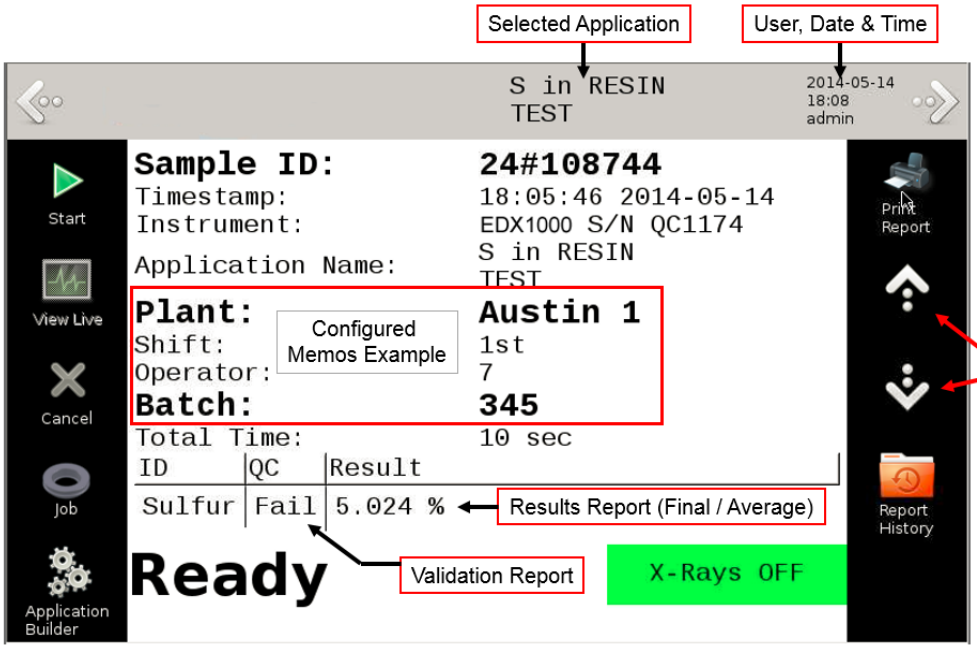
The Select Applications screen is where applications are selected and created by pressing the desired icon. A single screen has room for about 40 icons per page. New Empirical and new FP (optional) Applications may be created by selecting the “New” application template icon.



Empirical Application Icon

FP (fundamental Parameters) Application Icon

3.3 Main Screen



Hint* Drag this screen down or use Arrows to see Full Sample Data & Spectra

When using report memos with headers it may be necessary to scroll down to view the live results.

The Main Screen also displays the most recent measurement results.

The Main screen displays the [Sample ID], [Time Stamp], [Instrument ID], [Product ID] and other application specific parameters such as Report Memos and Live Results etc. This screen also displays the instrument status such as if the analyzer is in a "Ready" state, X-rays on or off, the remaining analysis count down time and if there are errors occurring during the sample measurement if a measurement is not currently in progress. Additional details such as net counts and spectra can be viewed by scrolling down on the screen.

Start – icon button starts the analysis process. The analysis should start immediately, if there are no specified input prompts such as Sample ID or other application specific input prompts.

Note: The purge, sample spinner and lock the door commands are sent at the beginning of the acquisition. The door latching must be confirmed before the hardware turns on the X-rays and starts a spectrum acquisition process. The shutter does not move until the door latching is confirmed. Once the acquisition is complete, the shutter is moved to the safe position and the X-ray tube is turned off, then the door unlocks.

Stop – Terminates the current sample measurement and reports acquired live result data.

Cancel – Terminates the sample measurement and discards all current sample measurement data results.

View Live – Display all real time spectrum, status messages, regions, conditions and count rates.

Job – Icon indicates whether a sample tray (5 or 6 position) is to be used or no tray.

Application Builder – This icon opens the application configuration and calibration screens which include Settings, Components, Conditions, De-convolutions, Standards, Measurement, and Calibrate.

Print Report – Sends the current results to the printer.

Page Up – Navigate towards the top of the screen

Page Down – Navigate towards the bottom of the screen.

Navigate to the Next Screen on right.

Navigate to the Next Screen on left.

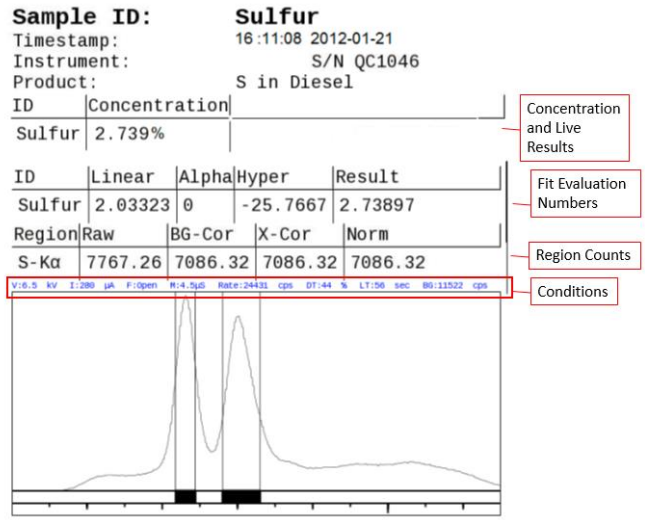
Report History – View a list of previous reports.

X-Rays On X-Rays OFF

Ready Status / Time Remaining – The Progress countdown timer indicates the remaining analysis time.

3.4 Data and Spectra

The top section or **Main** screen results represent the historical record of the previous measurement and is only updated once the next measurement is completed. Therefore any system parameter changes such as language, etc. will not appear on this screen until the next measurement is completed.



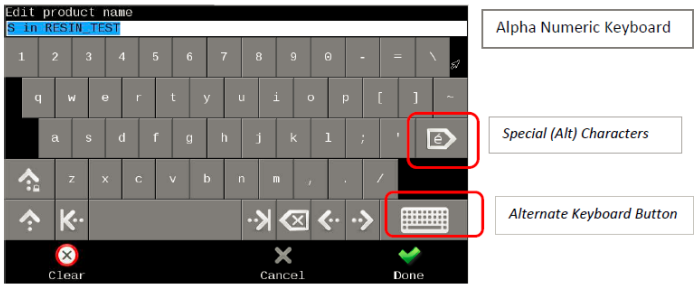
Fit Evaluation Numbers:

Linear: Concentration result based on a Linear fit.
Alpha: Shows percentage change in result from Linear with alpha corrections applied.
Hyper: Shows percentage change in result from Linear with Hyperbolic fit applied.
Region: Counts (in cps, counts per second)
Raw: Total cps in the region.
BG-Cor: The cps is the region after BG correction has been applied.
X-Cor: The cps in the region after BG correction and overlap "cross-corrections" have been applied.
Norm: Adjusted cps after the Standardize factor and offset have been applied.
BG: The cps in the BG region for the condition.

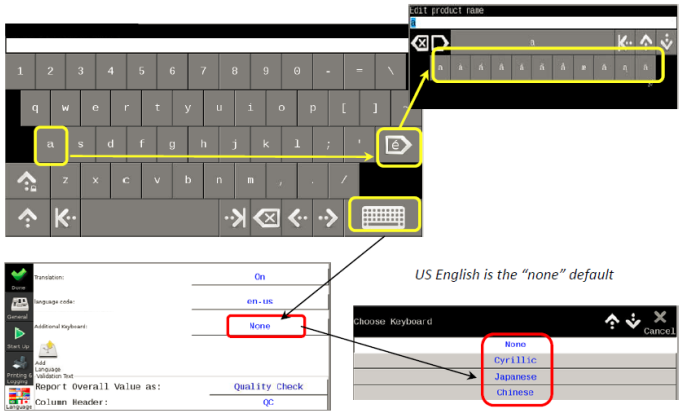
3.5 Software Keyboards

The EDX1000 series on-screen keyboard now has a special (Alt) character selection function key and an additional function key that provides the user with the option to switch back and forth between the default language keyboard and a second additional selectable language keyboard option to support the available multi-language options. The selectable keyboard is chosen on the [Utilities] / User Preferences / Language screen.

*Keyboard selections are NOT automatically linked to the selected language selections to provide for product ID input flexibility.



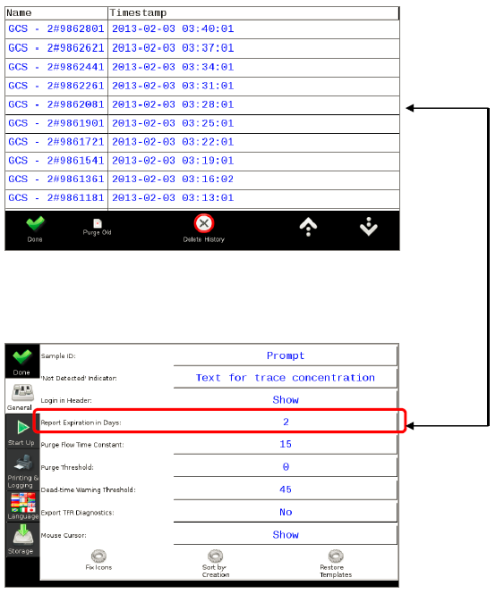
To select an alternate character, press the desired character on the keyboard then press the alternate character key then chose the character. The original keyboard screen will return with the alternate character entered.



*Change of language selections will require a software restart. Application names remain in original language created but can be changed in the Application Builder.

3.6 Report History

The instrument will hold the results history for a selectable number of days before the data expires and is deleted. The maximum number of history days is limited to 92. The history list will also be limited to a total of 1000 reports. The number of days to hold is set in the Utilities / User Preferences / General Screen ad the "Report Expiration in Days" value.



3.7 Application Builder

*Note this manual section explains the basic functionality of the screen features and buttons. However, for a detailed (step by step) practical approach to building and calibrating applications please refer to Section 5 – Empirical Application Development.

3.7.1 Settings

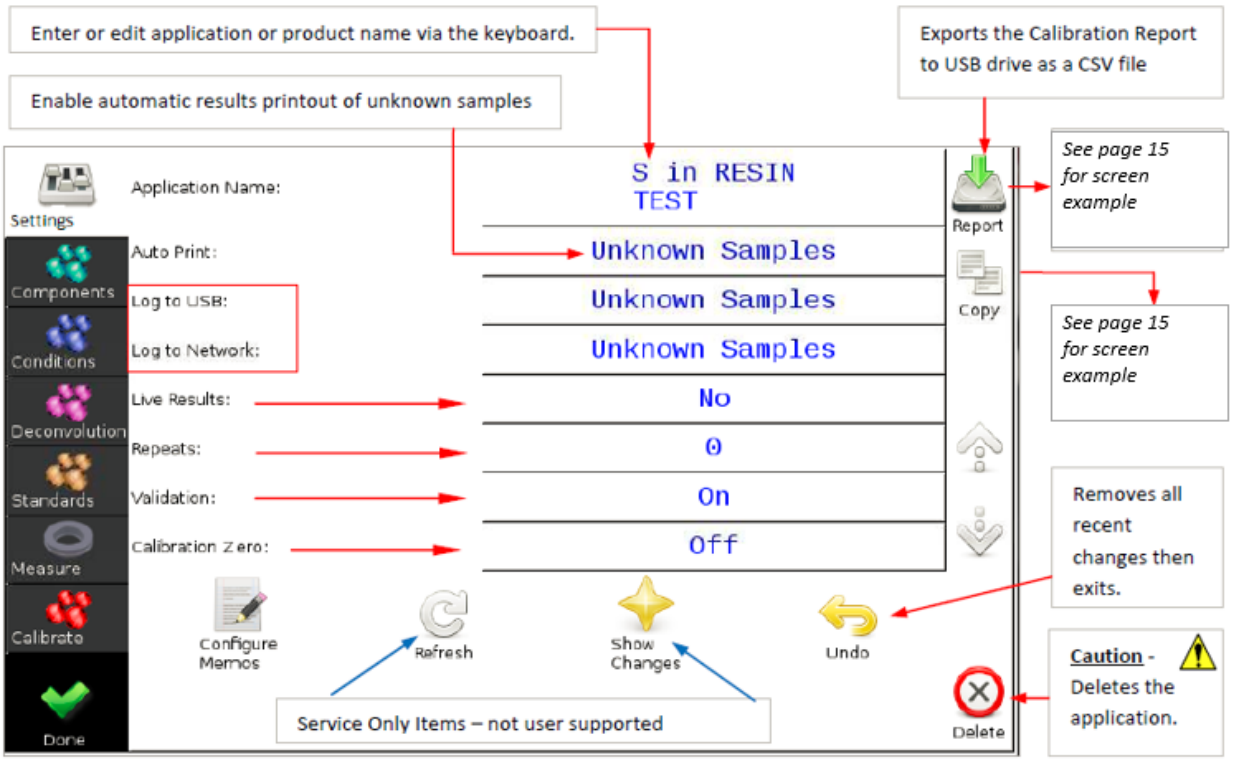
This screen is used to configure application specific parameters that are independent of the elements.

Note* Depending on the application and the template used, additional settings may appear.

Log to USB / Network – This enables the logging of Unknown Samples data CSV files to either the USB drive and / or network drive. These functions enable data logging of the intensities and spectra output and must also be selected in User Preferences / Printing & Logging screen.

Live Results – This function shows or hides the main screen real-time (preliminary) results computations during the acquisition. If analyzing samples with more than one condition, the live results values will not be displayed until after the first condition has completes. Live Results are not displayed in FP Applications.

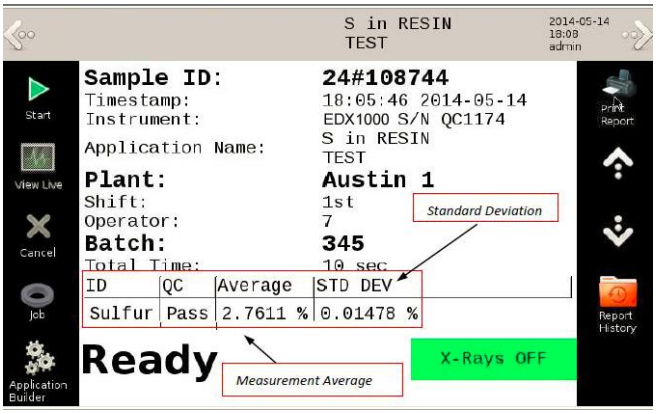
The Settings screen is shown below:



Repeats

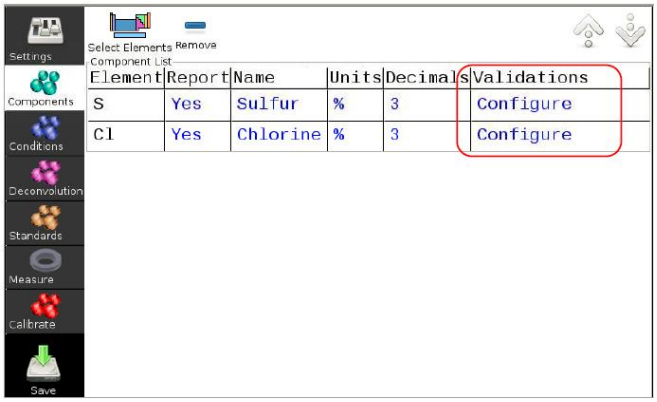
This option sets the number of repeat analyses for the unknown sample measurement. After all the repeat analyses are completed, the analyzer provides a main screen report showing the result average and the standard deviation.

See page XX for repeat function summary details. The option for choosing individual reports with summary or just summary only is set in the User Preference / Printing & Logging screen.



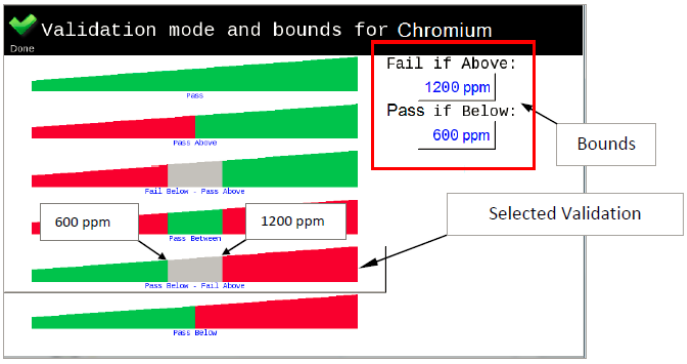
Validations

This feature is turned on or off in the application Settings screen and is configured on the Components screen. The validation function compares the sample to the selected mode and set bounds and also adds to the result report the pass, fail, or unsure quality check status. The validation Configure screen contains six (6) selectable “pass / fail” evaluation modes and the limit (bounds) values. All validation report texts can be changed in the Language screen of the User Preferences.



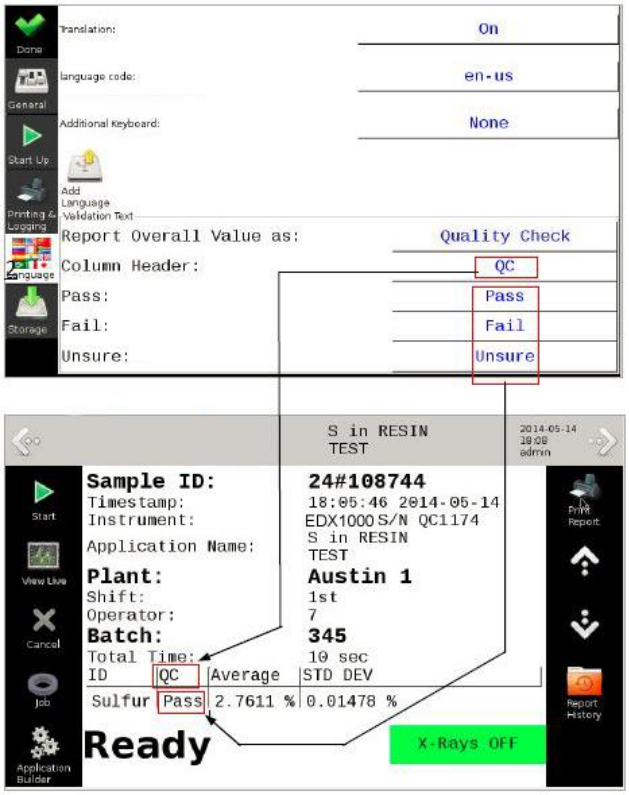
1. Starting with the validate option already enabled, then select the Components screen and select the Configure screen for the specific element to setup validation and the bounds.

2. Select the appropriate validation mode option, then enter the desired above and below bounds values (shown below).



3. To confirm or make changes to the validation report text and column header, precede the Language screen.

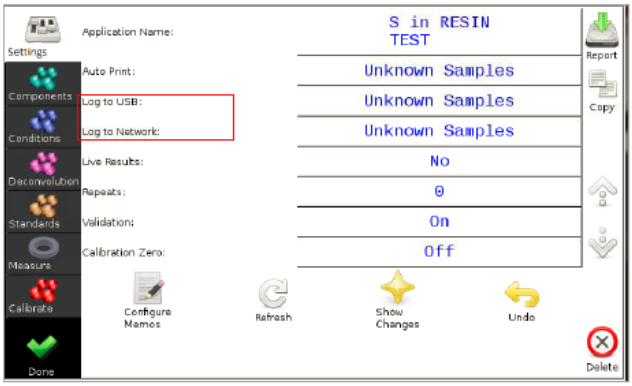
Note* it may be necessary to scroll down on the screen to see the validate status report depending on the number of elements configured in the sample or the size of the report memo.



Unknown Sample Data Logging

This feature provides for data logging of unknown sample results in both the CSV file format and the archiving of PDF file result reports. With this feature enabled, the data is exported to either or both the external storage locations such as a network folder and to a USB flash memory stick. The “Log to:” features must be enabled in the specific application builder Settings screen. Also the data type selections must be globally selected in the User Preferences / Printing & Logging screen.

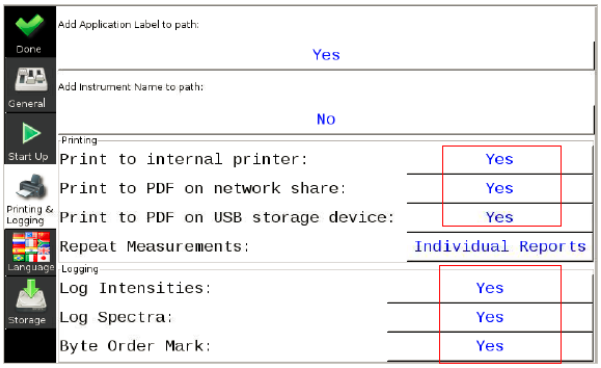
How to log Unknown Sample Data:
First choose the application for unknown sample result logging. Next, on the Settings screen enable Unknown Samples data to be logged to wither or both the USB storage device or Network.



Before logging sample data, make sure the logging settings are enabled on the Printing & Logging screen.

Utilities / User Preferences / Printing & Logging Screen

These are global settings that apply to all applications and are set in the Utilities / User Preferences / Logging & Printing screen. This must be set before the application will output unknown sample data result CSV files to the selected storage drive.

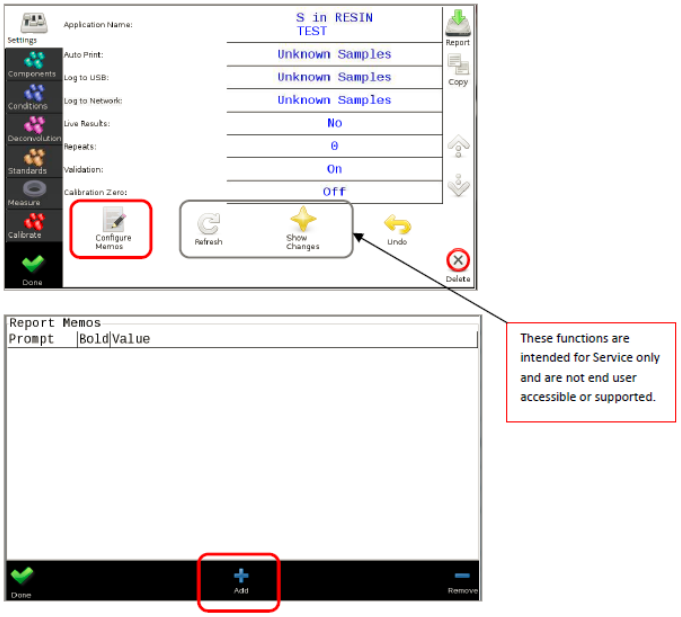


Configure Memos

This feature allows the user to setup customized prompts that will also appear on the reports. If using three or more prompts it may be necessary to scroll down to view Sample ID, Validation, Average, STD Deviation and Live Time Result Values on the display.

Designing Report Memos

In Application Builder, select Configure Memos, then press +Add.

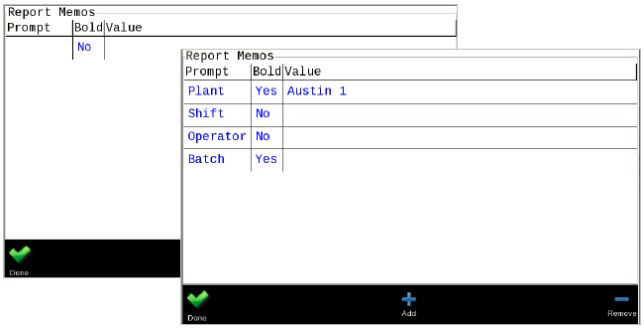


After pressing Add, design the report memo by pressing the desired field (even if blank).

Prompt: The name used for the report memo.
Bold: No means regular font, Yes means bold font
Value: The input field when starting a measurement of a sample.

- Leave blank and operator inputs new value for each sample.
- Enter a Value and that will show as the default value for each sample, and operator can still edit it when starting an analysis or leave it at default.

The next example shows four memos created: **Plant, Shift, Operator** and **Batch.**

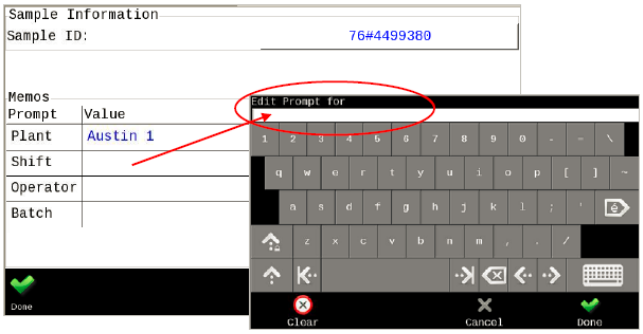


- The memo for Plant and Batch are set to print in bold font.
- The Plant memo has been set to default to a manufacturing site called Austin 1.

When design is complete, press Done and exit the Application Builder.

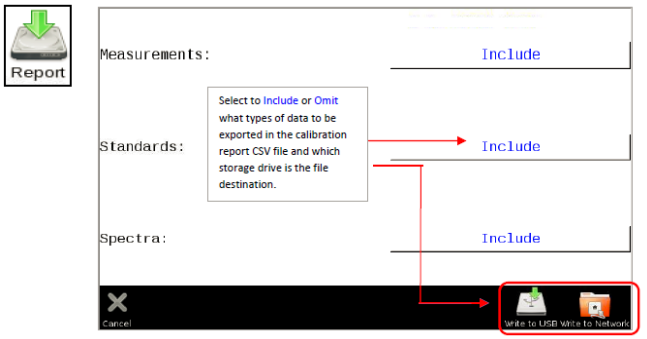
Measure a Sample

When measuring an unknown sample from the Main screen, the Sample ID prompt appears along with the prompts to use the memos. In this example, the operator can press on each Value field and enter a Sample ID, the Shift information, the Operator ID, and the Batch number. The default value for Plant can be left at default, as designed in the Application Builder, or edited if needed.

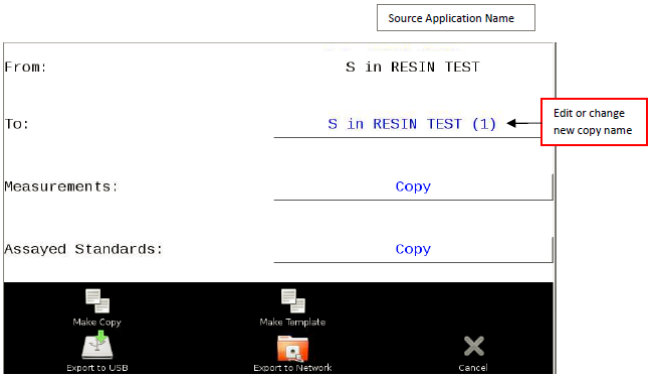


Report – How to Export a Calibration Report

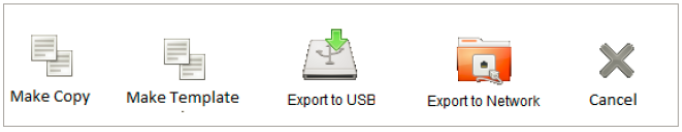
This screen generates a valuable calibration diagnostic report that may be extremely helpful with application support. The calibration report CSV file contains conditions, sample path settings, and regions. Also, measurements Standards, and Spectra may be included or omitted. The report is exported to either or both USB drive and the Network drive if selected.



Copy Applications – Export Applications – Making Templates



Command Buttons

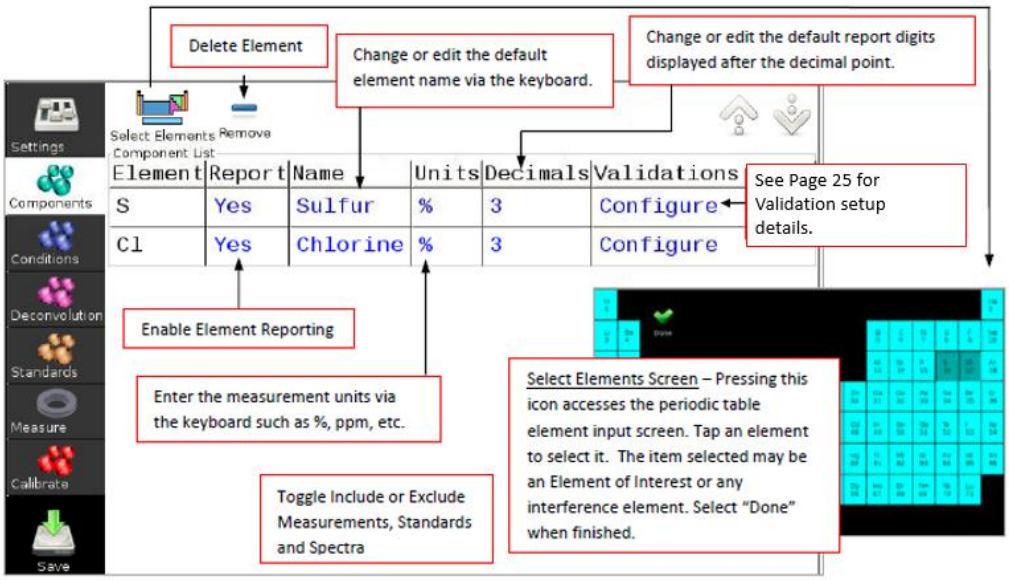


Make Copy – This button creates a copy of the selected application. The copy may include the existing Product Name, Measurements, or Assayed Standards if selected.
Make Template – Creates an application template from current application. The template does not include measurement data and cannot be altered after creation. Templates are only deleted from the Utilities screen with the appropriate user privilege.
Export too USB – Sends a copy of the selected application to the USB drive.
Export to Network – Sends a copy of the selected application to the network drive.

3.7.2 Components

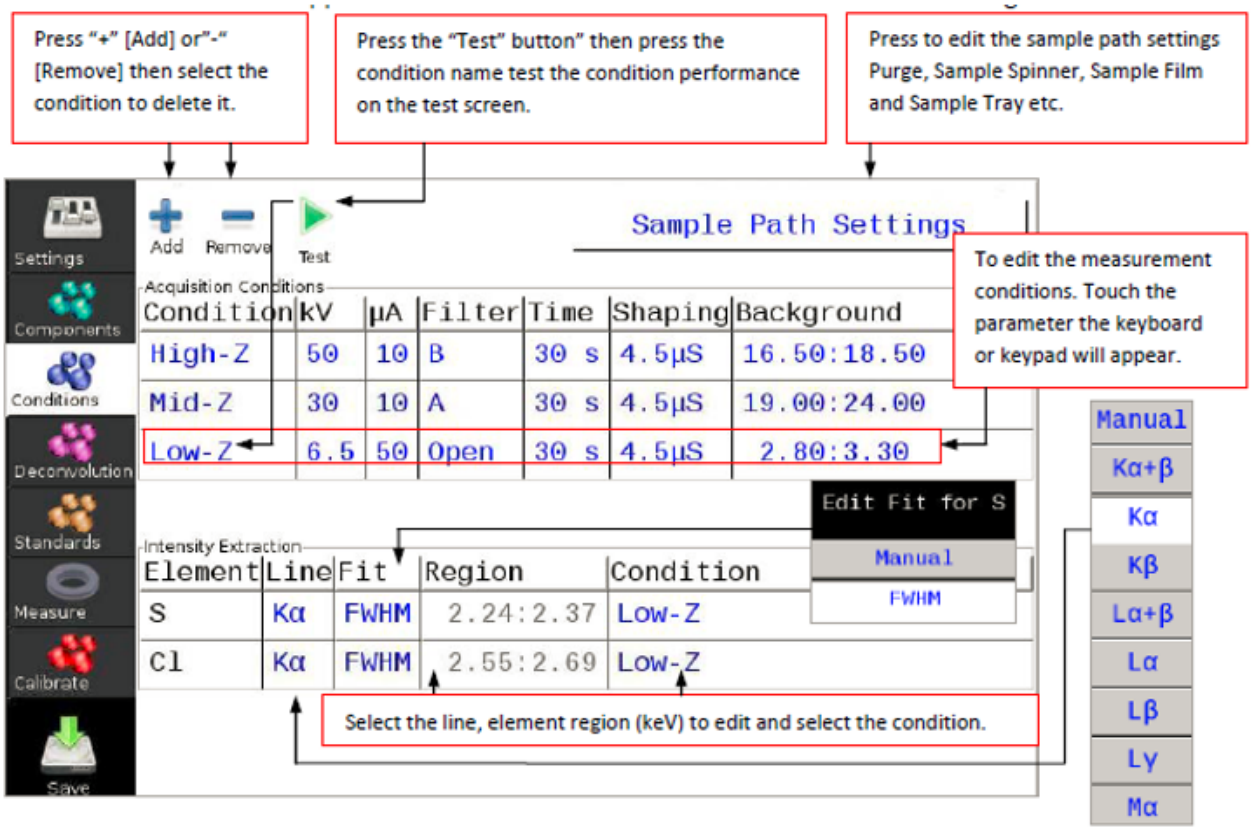
This screen is used to select the elements of interest, units of measure and the report precision.

*Note: “validations” will not appear on this screen if not enabled in the application settings screen.



3.7.3 Conditions

This screen is where the applications measurement conditions are selected and configured. The default regions are FWHM (Full width at half max) however they can be manually adjusted.



Condition Terms

Acquisition Configurations:

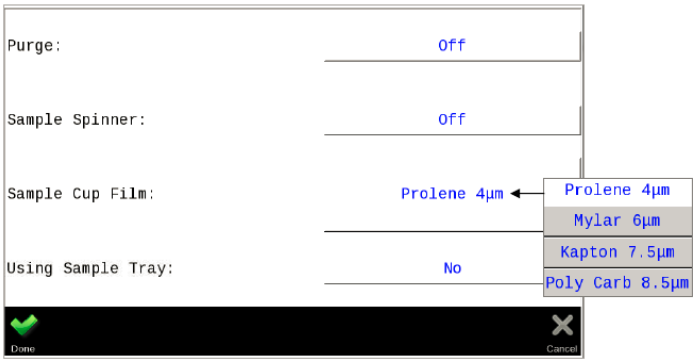
kV Tube voltage settings from 6.5keV to 50keV
µA Tube emission current
Filter Tube filter selections open or A – E
Time Analysis measurement time
Shaping Shaping time 3µS, 4.5µS, 9µS
Background Backscatter region for each condition

Intensity Extraction:

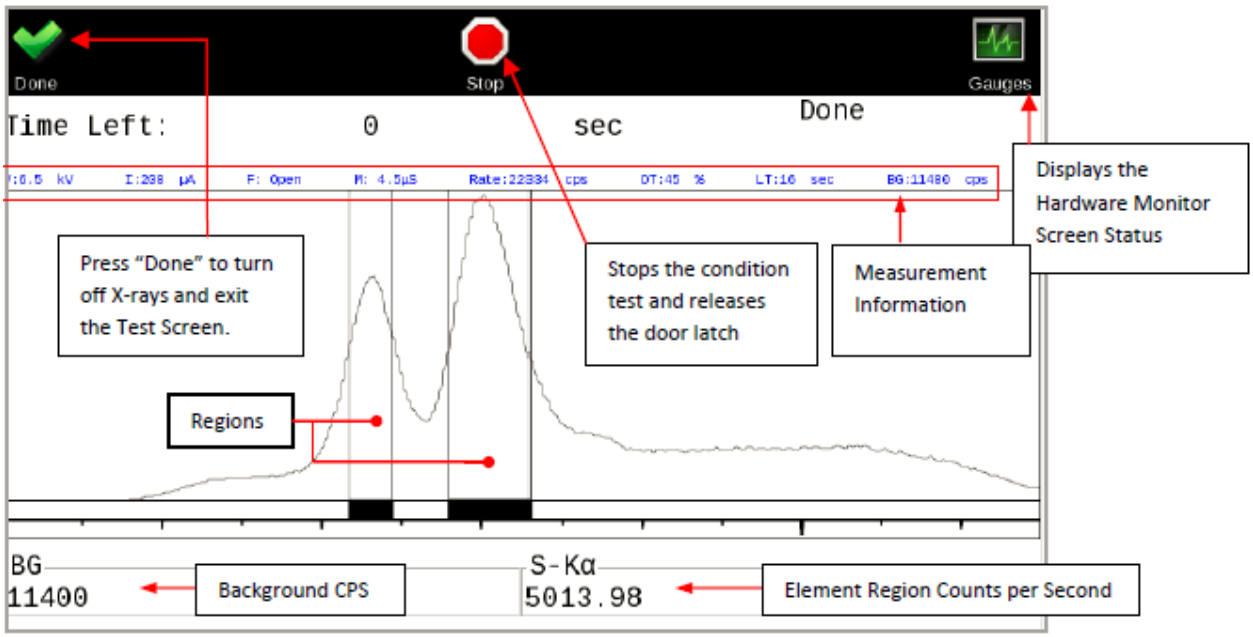
Element Element of interest
Line Manual, κα+β, κβ, κα, Lα+ β, Lβ, Ly, Mα
Region Energy region in keV for the element of Interest

Conditions – Sample Path Settings

This screen allows for specific application path configurations.



Conditions – Test Button Spectra

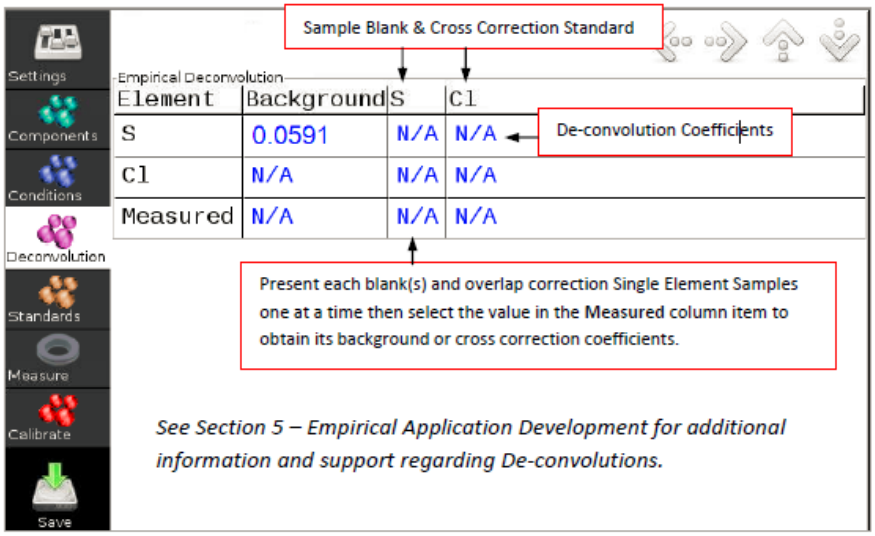


Measurement Information:

V: Voltage being used
I: Emission current being used
F: Filter being used
M: Shaping time being used
RATE: The throughout, total cps into the detector
DT: Dead Time
LT: Live Time
BG: cps in background region

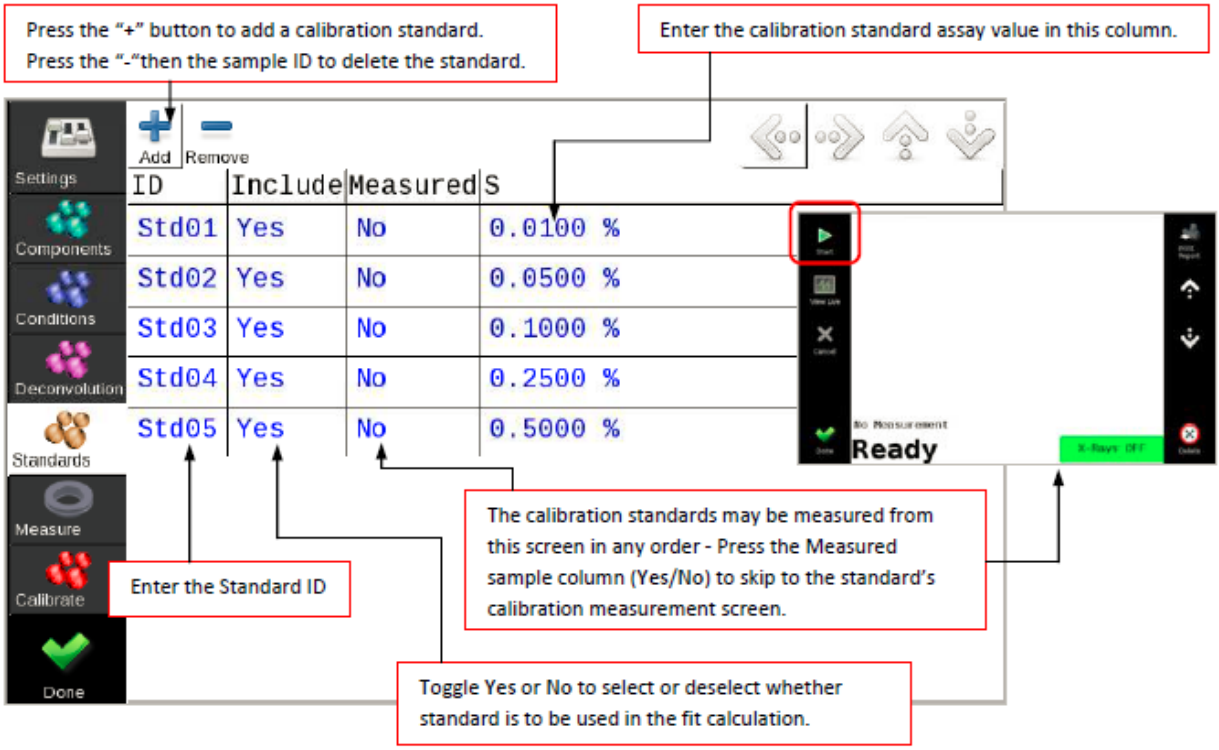
3.7.4 De-convolution

This screen displays the deconvolution coefficients and also is the means for acquiring new or updated background, cross corrections coefficients.



3.7.5 Standards

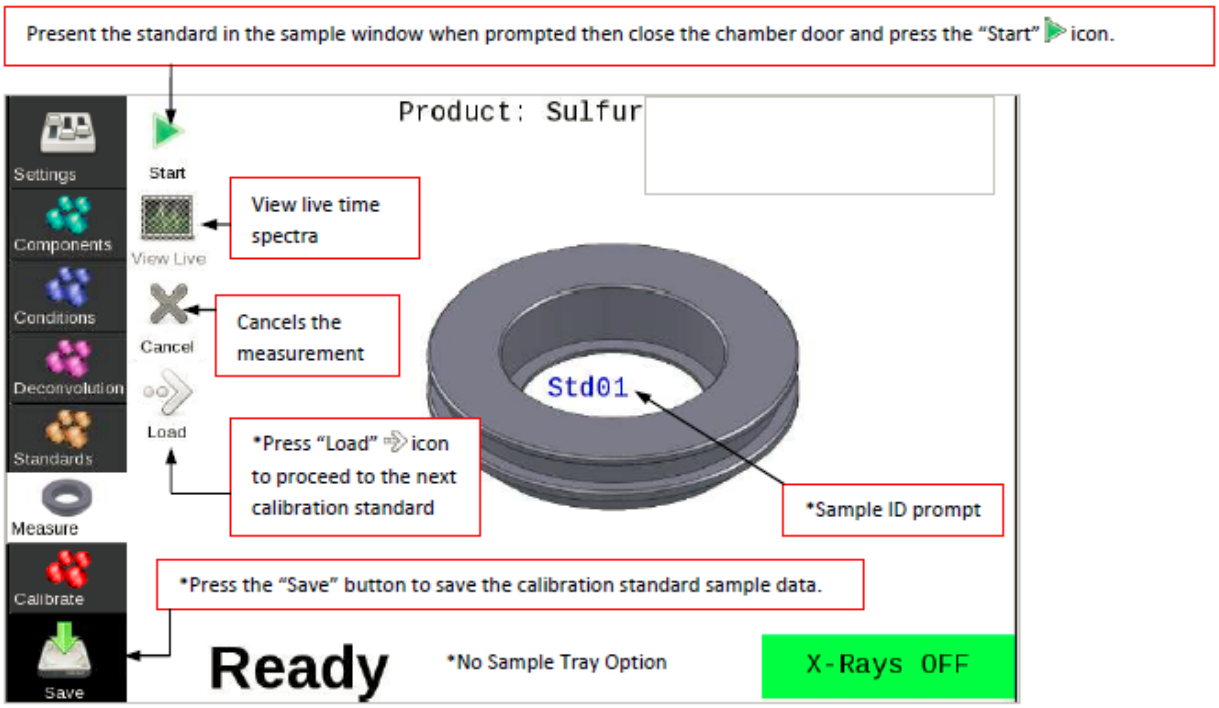
This screen is used to build the calibration standard table, enter the assay values, measure a standard, select or deselect which standards are used or not used in the calibration fit computation.



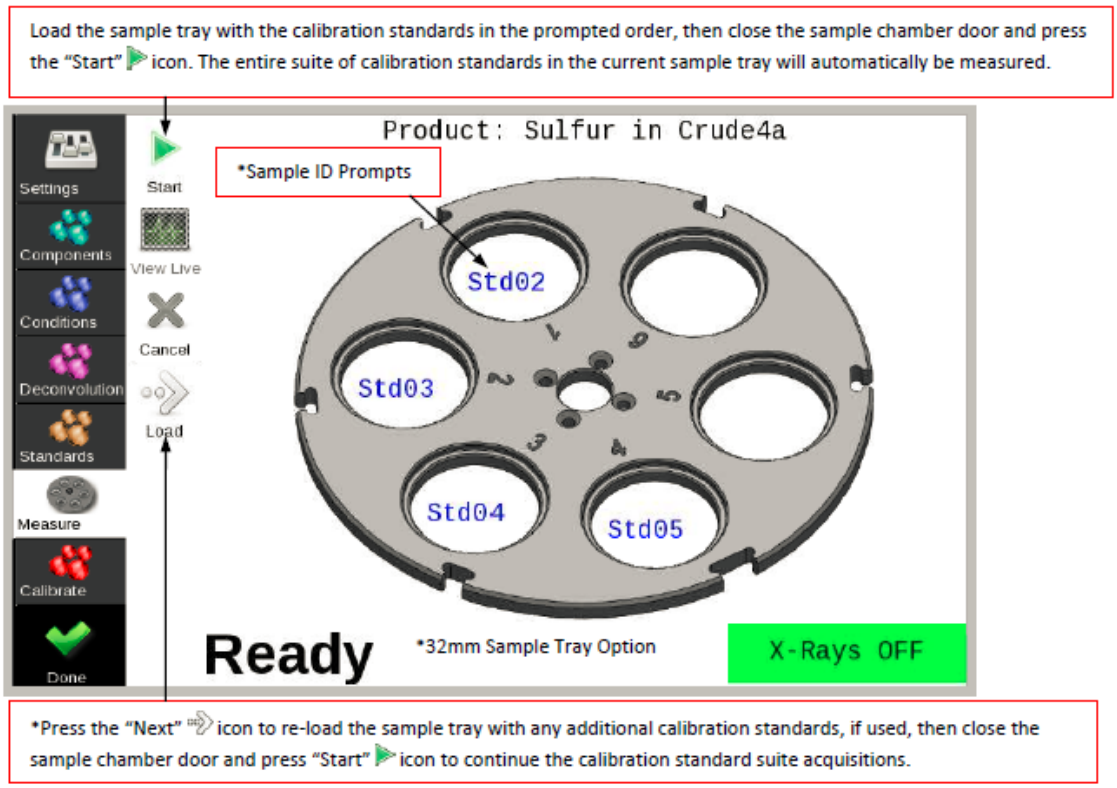
3.7.6 Measure Calibration Standards

No Sample Tray

The Measure screen provides a guided calibration flow sequence with sample or sample tray prompts.

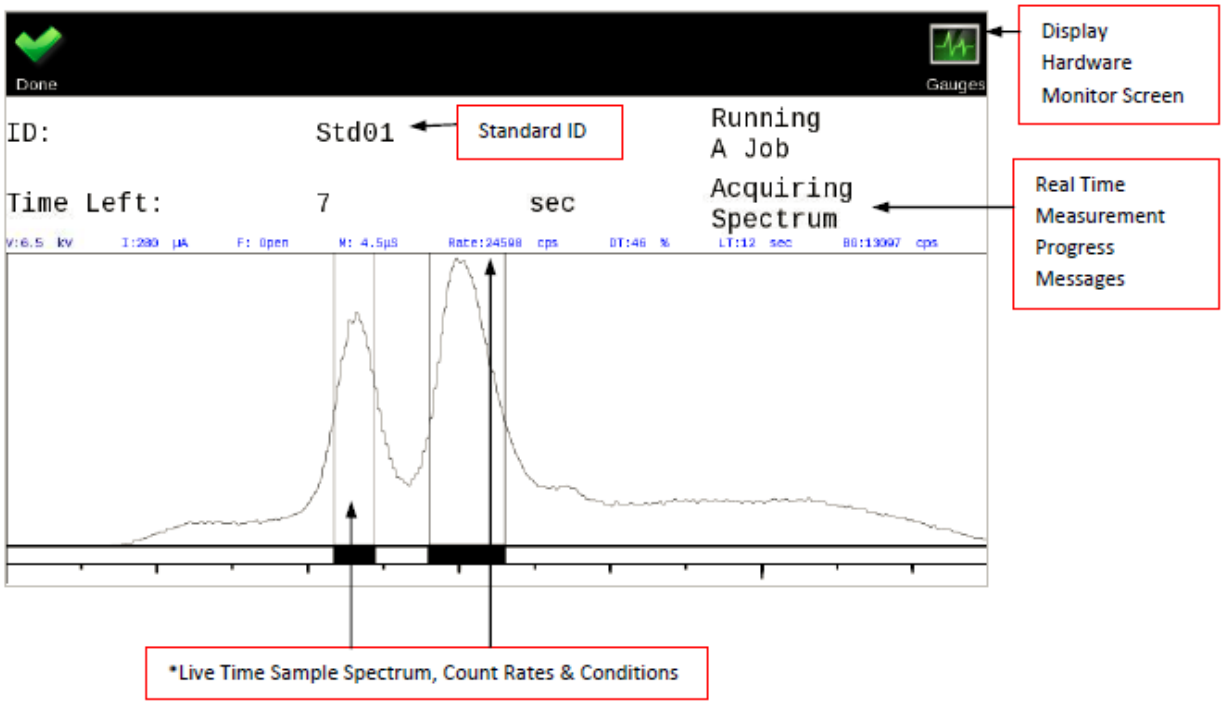


With Sample Tray



View Live Screen Example

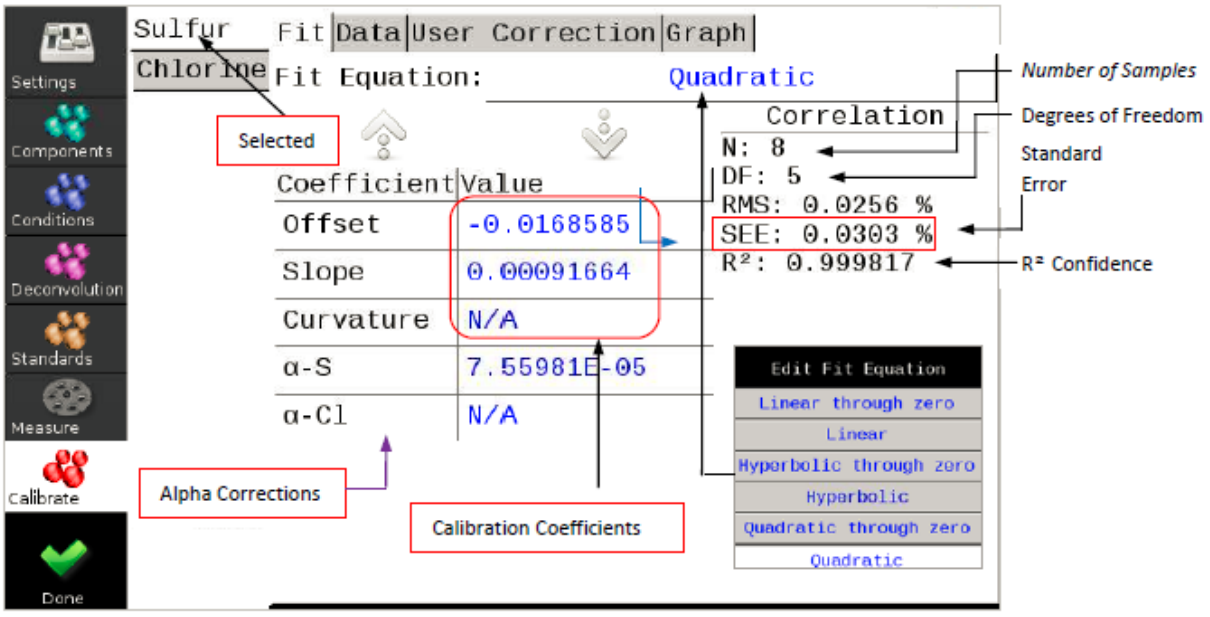
This screen is displayed when the View Live button is pressed during the calibration sample acquisition. The display is the real time spectra and count rate data.



3.7.7 Calibrate

Fit

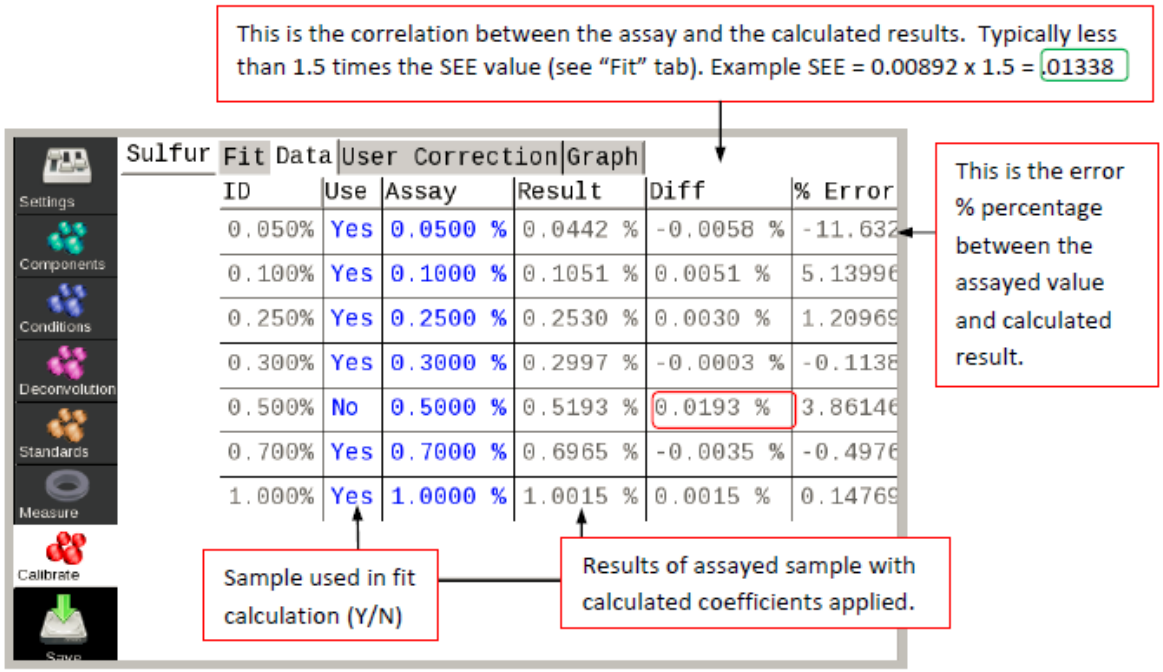
This screen displays the calibration coefficients (offset, shape, curvature, and intensity based Alphas), calculated from the included measured calibration standards with the selected fit equation applied. Also, the standard error estimate (SEE) and the R squared value is shown.



See Section 5 – Empirical Application Development for additional information with the calibration coefficients.

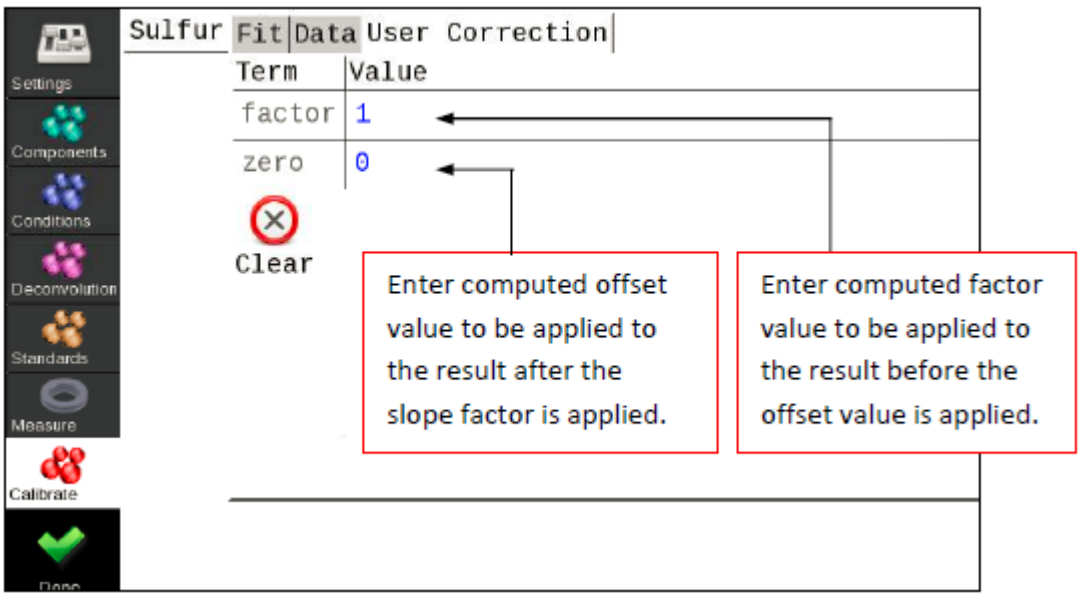
Data

This screen displays the standard assay values and the measured results computed with the current fit calibration coefficients applied. Also the deviation (Difference) and percentage error results are shown.



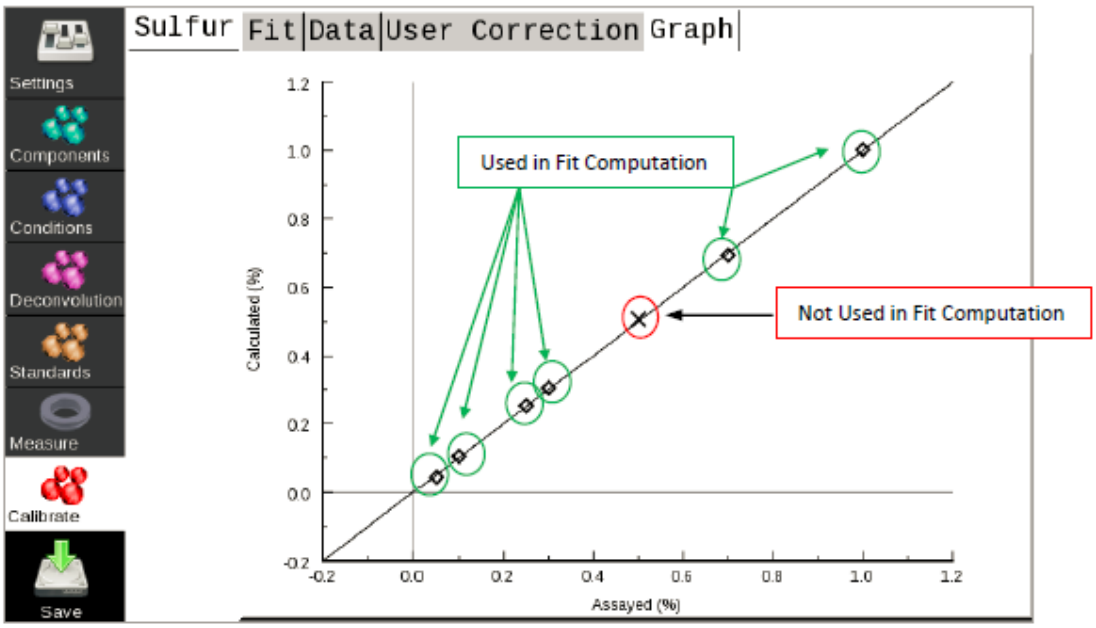
User Correction

User Corrections affords the user the option to "fine tune or tweak" the analyzer results with known assayed standards, but without full recalibration. This may be done if the analyzer has consistently been reading too high or low with multiple known reference standards. Before running the assayed standards to compute new user corrections, first verify the sample window film is not contaminated. If so, replace film taking care not to contact the beryllium detector window. If using a standardization correction factor re-run the standardization sample and confirm the new factor is between 0.90 – 1.1. If not within this range you may need to recalibrate.

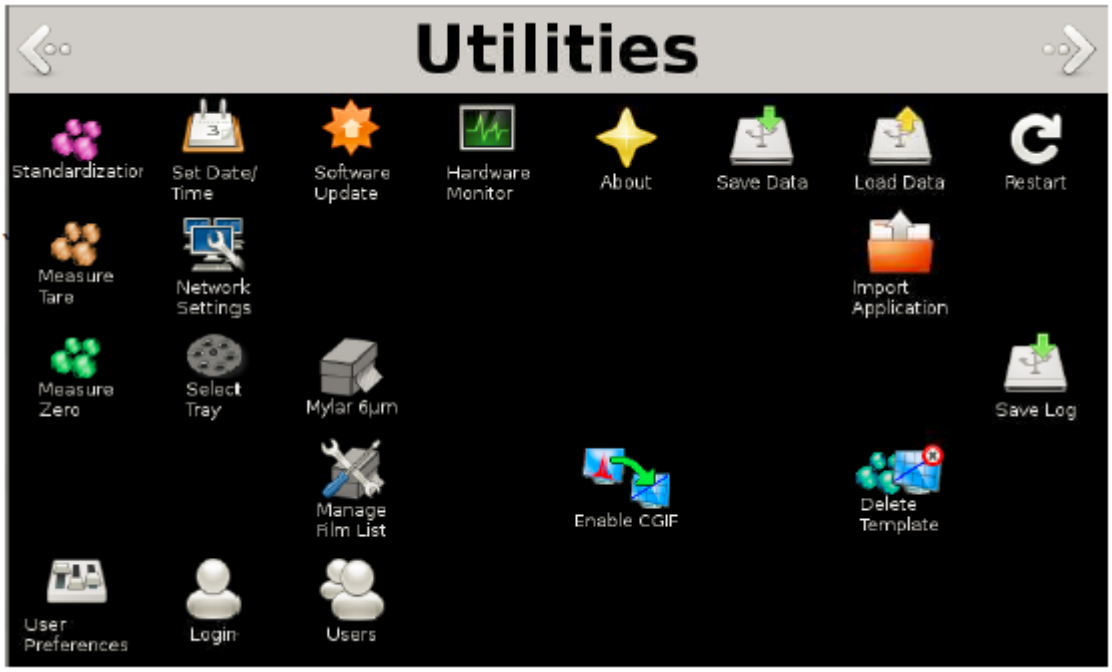


Graph

This scree provides a graphical representation showing the fit measured calculated values vs. the given assayed calibration standard value. This sample icons on this screen also indicates which standards are included or excluded for the fit computation (◊ = Yes, X = No)



3.8 Utilities



Icons

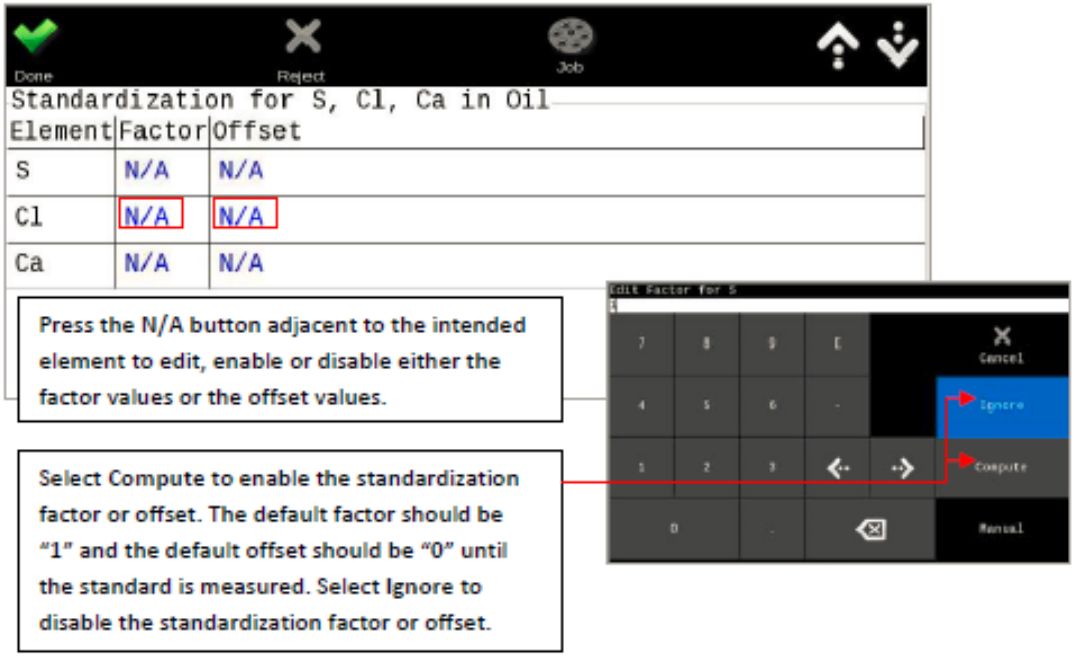
Standardization: Computes a ratio to be applied to the results as a drift correction. This effectively lengthens the calibration usefulness in some applications.
Measure Tare: Acquires the post analysis offset value to be subtracted from the results.
Measure Zero: Acquires the Zero value to be subtracted from the intensity.
User Preferences: Sets user customizations i.e. Startup Application, Language, Scroll- back size, Data logging, Printing, Reports, Login Header selection, Startup User, etc.
Set Date / Time: Sets the system clock and calendar (24 hour clock).
Network Settings: Displays the current DHCP dynamic IP Address of the instrument, if connected to an active network.
Select Tray: If equipped with the tray option allows user to select between 5 and 6-position trays or none.
Login: Screen dialog for entering the user name and password to login.
Software Update: This button for software updates via USB flash memory stick.
Safety Film: Sets the safety window film settings in the software (Prolene, Mylar & Kapton)
Manage Film List: User setup of different types of films and thickness (useful with FP).
Users: Setup and deletes users accounts and their various permission levels.

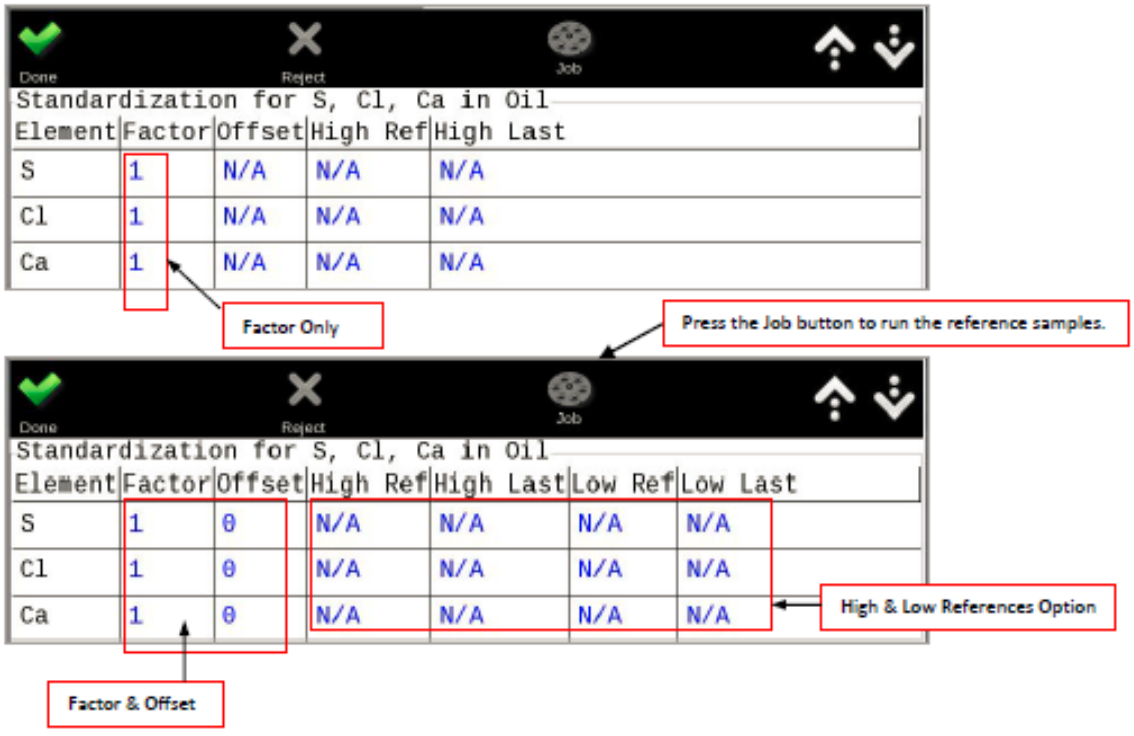
Hardware Monitor: Displays hardware status and configuration options intended for hardware related service diagnostics.
Service: *Service Technician use only (no user access). - Provides for detector gain and MCA resolution maintenance.
About: Reports current software and firmware build ID version and license.
Factory Settings: *Factory use only (no user access).
Factory Utilities: *Factory use only (no user access).
Save Data: Saves and exports existing application data, user logins and hardware configurations to the external USB or network storage drive.
Load Data: Loads all selected previously stored “Save Data” applications, app data, user logins, and hardware configurations from the USB or network storage into the instrument.
Import Application: Loads stored application data and calibration sample data stored from external USB or network storage into the instrument.
Delete Template: Delete template (use caution).
Restart: Saves all current changes and restarts the program.
Factory Reset: Factory use only.
Save Log: Saves the system debug file to the USB drive for factory support if necessary.

3.8.1 Standardization

Standardization – The optional standardization function may be used to tweak the reported results to compensate for result drift (in some applications) between calibration due dates.

How to Enable Standardize:

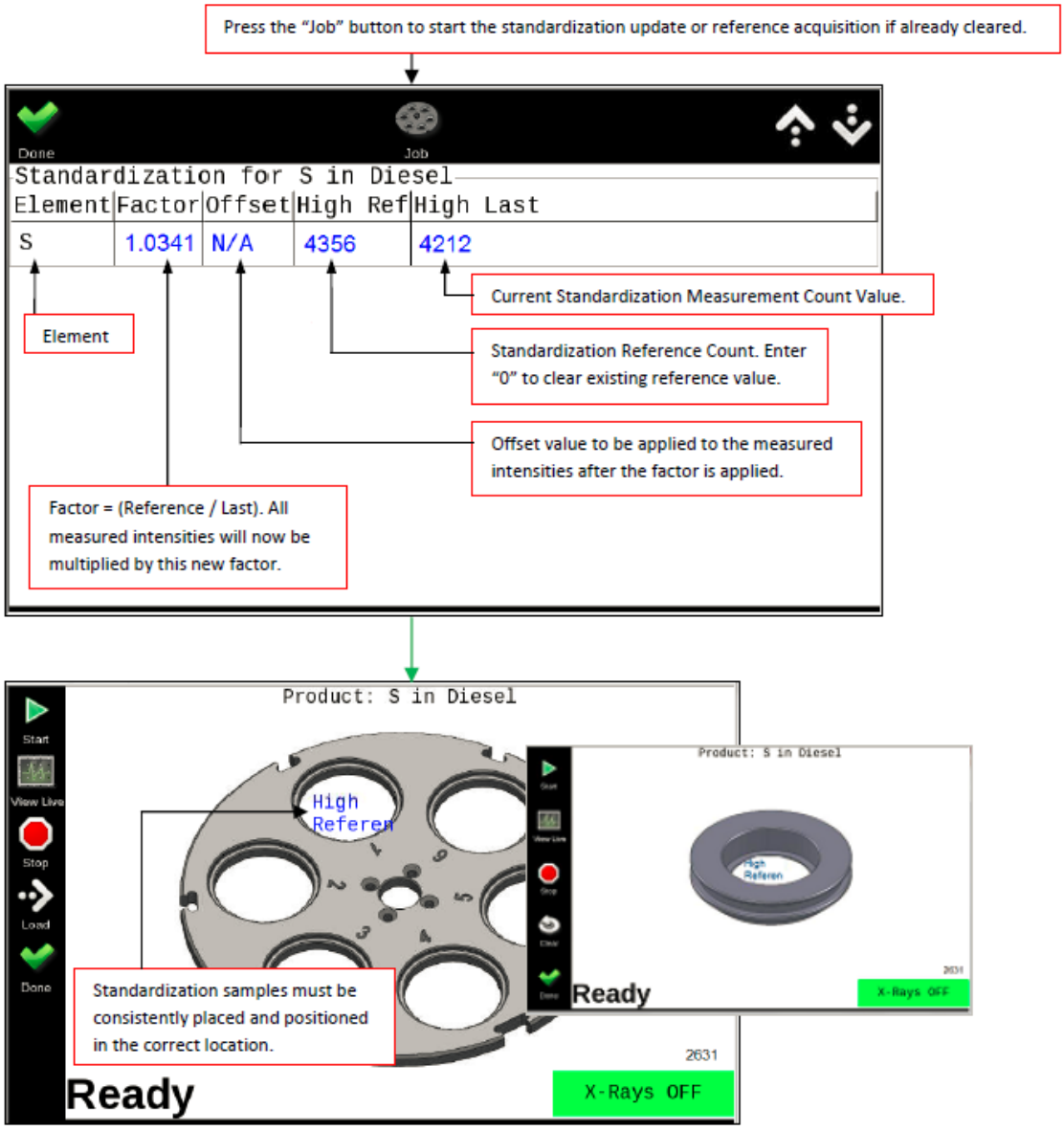




Standardizing

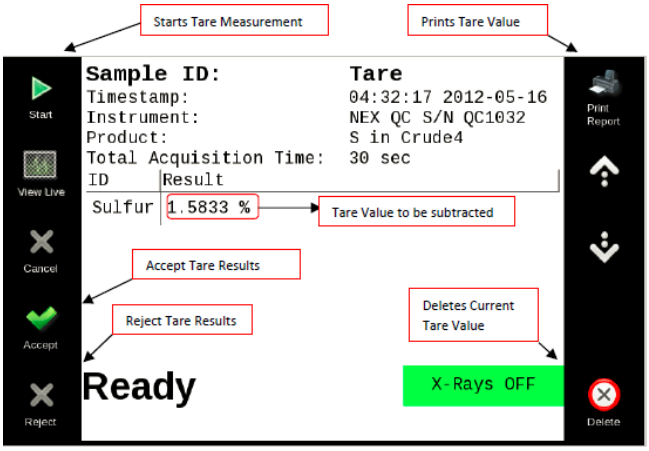
The frequency of standardization is determined by the user and is not intended to replace periodic calibrations; however it can be helpful by lengthening the usefulness of the existing calibration curve between calibration due dates. Standardization requires using the same exact single or multiple element standard; with the same physical alignment or positioning. It's important before acquiring calibration standard measurements to obtain a new calibration curve or fit, that the existing standardize references are manually reset to the default values (i.e. factor 1, offset =0). Once the fit curve has been successfully determined then new standardize references should be acquired.

How to Standardize



3.8.2 Measure Tare

The Tare function is used to acquire the background or baseline concentration value to be subtracted from the final reported concentration results.



The Measure Tare function button for the selected application is located on the Utilities screen.

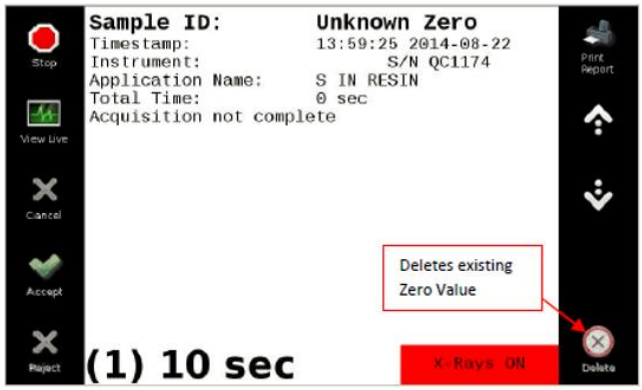
For example – When applying a silicon coating to a recycled paper stock which already contains a small amount of silicon, you will need to use the tare function to obtain a new tare value to effectively remove the paper stock background silicon value from the final applied coating results. Tare values are only applied after the sample's empirical fit computation.

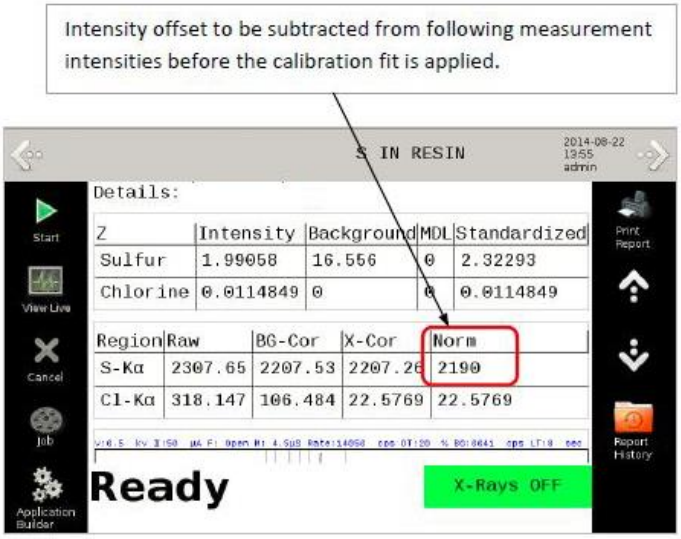
The tare function is not appropriate for all applications and must be used only for elements that are actually found within the sample. If the element that tarred element is not present then the tare function may introduce result repeatability issues.

To exit the Tare function press either Accept, Reject or Delete.

3.8.3 Measure Zero

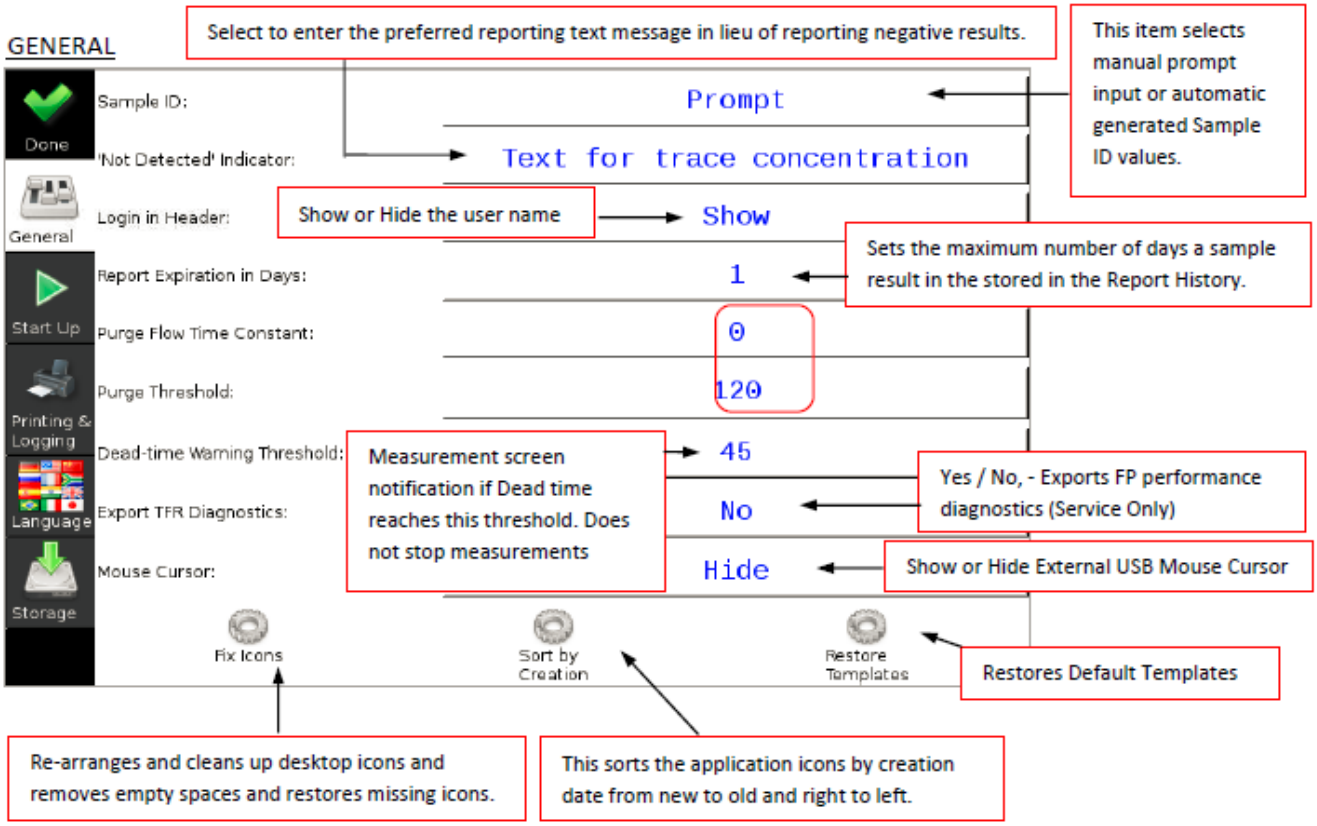
The Measure Zero function works like the tare function, except the Zero will be applied to the intensity values instead of the concentrations.





3.8.4 User Preferences

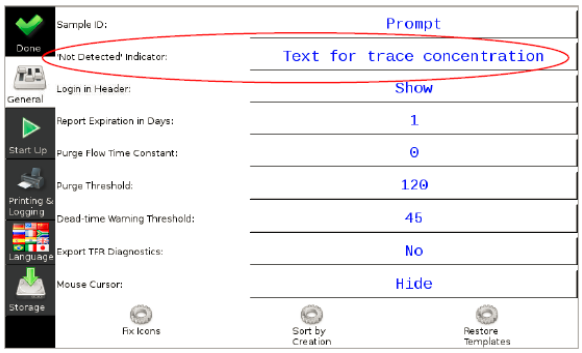
This screen allows customizing the Startup Application, Sample ID prompts, ND indicator text, Login Header, Language, Scroll back size, and the Startup User.



How to Setup the "Not detected" Indicator

On Utilities screen, select User Preferences.

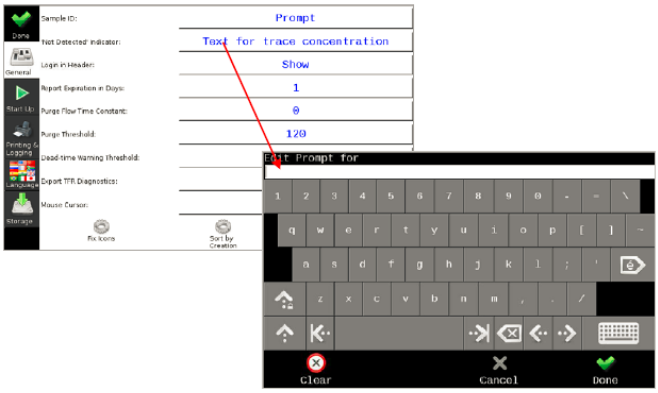
Concentration results for a sample that has a concentration below the detection limit may report negative values. This is valid in some cases due to the fit offset of the calibration, or may also report as a negative number due to other setup concerns in an application. The operator may not want negative results displayed, and this option gives the user a way to customize what the output will report when a sample with a negative concentration number is measured.

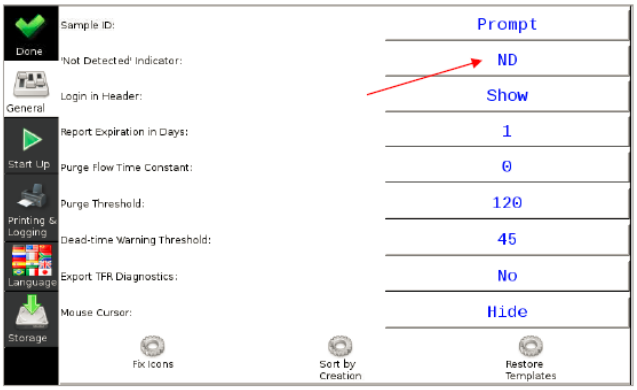


The default is set to display concentration as originally generated by the fit, whether the value is positive or negative.

In this example, the field is changed to show 'ND' instead of a negative result value. ND is a common output meaning "Not Detected".

Press the 'text for trace concentration' field and enter ND as the new field entry, or whatever notation is desired.

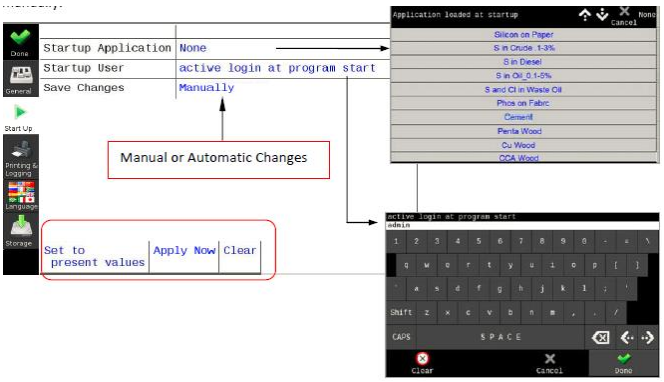




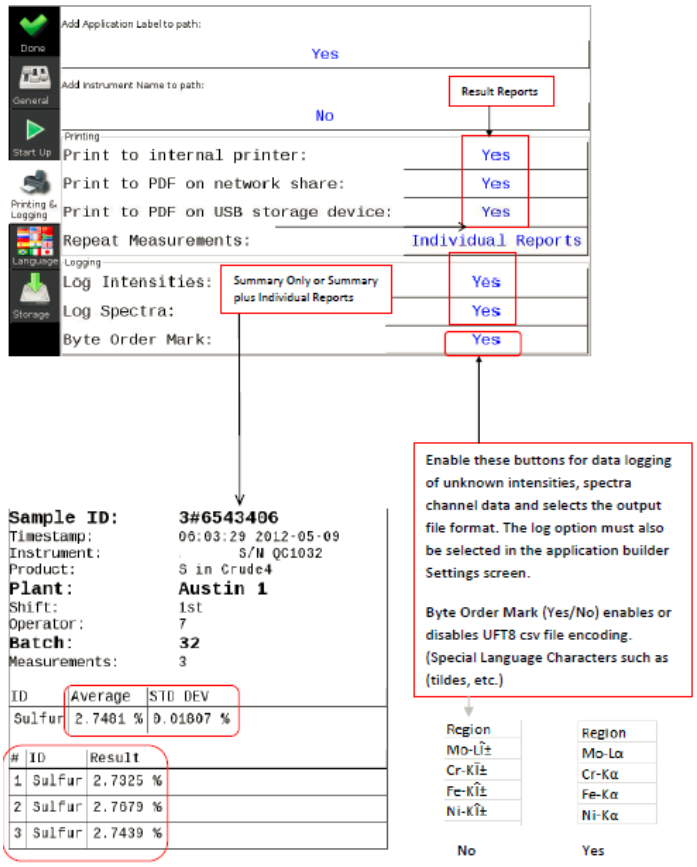
Press "Done" when complete. This is a global setting. When a sample is reporting a negative concentration, the value will be displayed as ND for all application methods.

Start-Up

Selects default start-up application and user at power up and whether changes are saved automatically or manually.

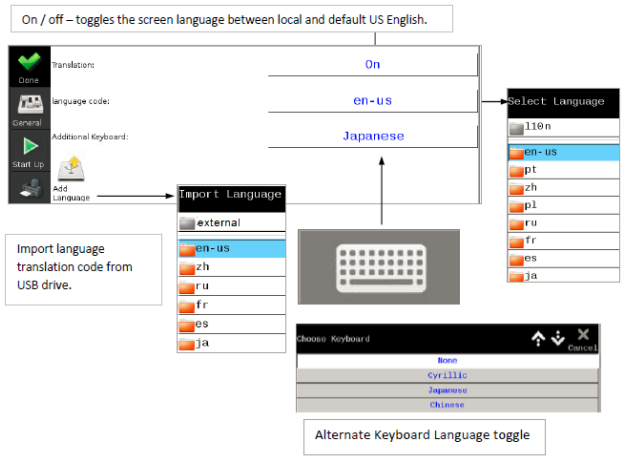


Printing and Logging

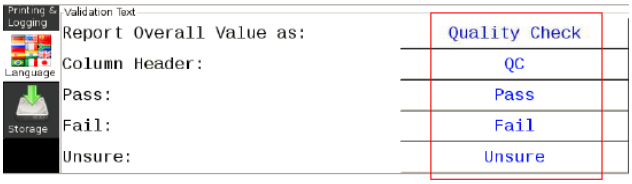


*The number of samples for repeat measurements is set in the application Settings screen.

Language and Validation Text



*See page 13 for details regarding Validation Setup and Function



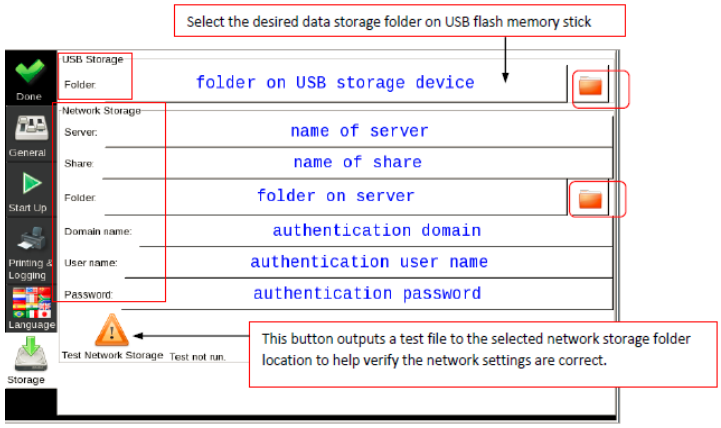
*Report default text messages may be edited here via the keyboard.

Report Overall Value as: Report prefix for net validity
Column Header: Column header for individual validity
Pass: Displayed when a compound or sample is good
Fail: Displayed when a compound or sample is bad
Unsure: Displayed when a compound or sample is neither good nor bad

Storage

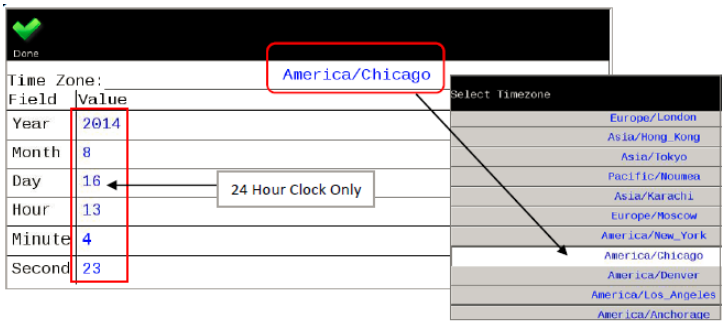
This screen is for selecting specific data storage folders for data logging, calibration reports, import and export of applications and data save backups, on both the network and USB flash memory stick. Also Server, Domain, User Name, Password, Folder location and share resource is configured here.

*May require company IT department support for your local network setup assistance.



3.8.5 Set Date / Time

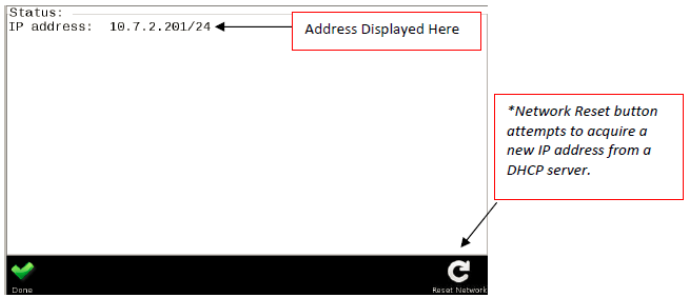
Press to select the time zone and to enter the date or time settings. Then press "Done"



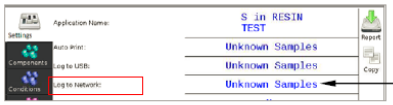
Important – The EDX1000 uses a rechargeable clock battery to maintain date and time values for about 3 weeks while the instrument is powered completely off. If the instrument has been off for a significant amount of time then check and reset (if necessary), the clock date and time. The clock battery will begin to automatically and continuously recharge while the instrument is on and will be fully recharged within about 1 hour of being powered on. The clock date and time values must also be current before attempting updating the instrument software.

3.8.6 Network Settings

Press the network settings icon then press the Reset Network button to display the DHCP IP address. Then press "Done" to exit the screen.



Network Data Storage



Select **Unknown Samples** to log the result data to the network drive (in the **Settings** section in the Application Builder screen).

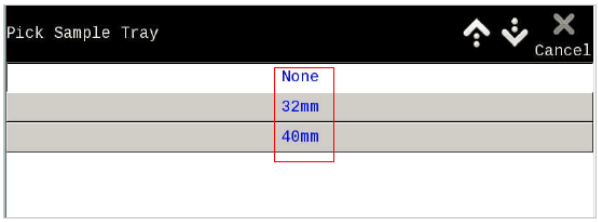
Exporting and Importing Applications to a Network Drive

Export to Network: This feature is located on the Application Builder / Setting / Copy screen. This provides for sending a copy of the selected application to the network drive.

Network Storage: This feature is located on the Utilities \ Import Application screen and provides a means to import a copy of the selected application from the network drive.

3.8.7 Select Tray

Tray options are “None”, “32mm” and “40mm”



Please note if a tray size is selected, but no tray is installed or is not functioning properly, the X-rays will come on at the start of analysis, however the count down time will not proceed. Press the “View Live” button to verify the progress message status is not stuck on “positioning sample”.

3.8.8 Software Update Procedure

The new software revision update installer is a single file located or copied to the USB drive.

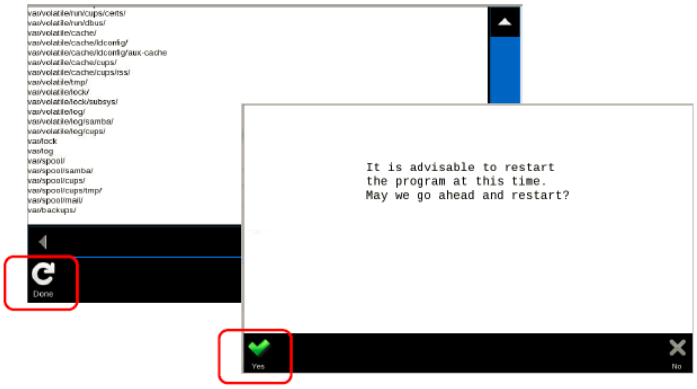
*Important before updating the software verify the instrument “Date and Time settings are current and always backup the system with a “Data Save”.

1. To update the software, first copy the update file to the existing USB drive. (If file already exist on USB drive overwrite it.)

2. The insert the USB drive in the analyzer’s USB port and proceed to the software Utilities screen.

3. Press the Software Update icon. Then select the update file from the screen.

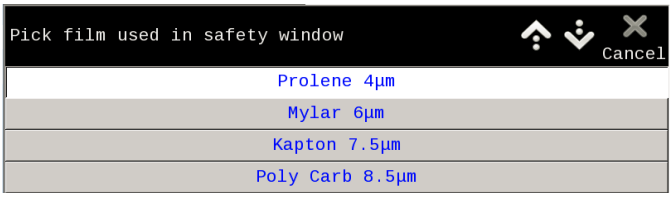
4. Press [Done] icon to start the software update process. Wait for the [Stop] button to change to a [Done] button. Then press [Done]. Press [Yes] to allow a restart.



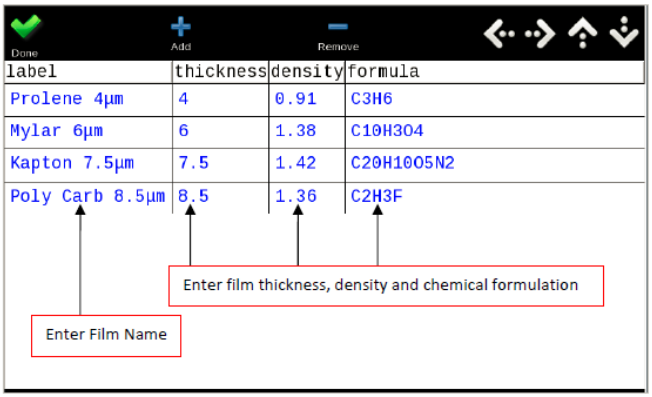
5. Go to the [Utilities] screen again, then press the [About] icon and confirm the Software Build ID, Qcu ID: and the Pcu ID: software firmware revision numbers are correct.

3.8.9 Safety Window Film

Select the recommended film from the safety window film list.

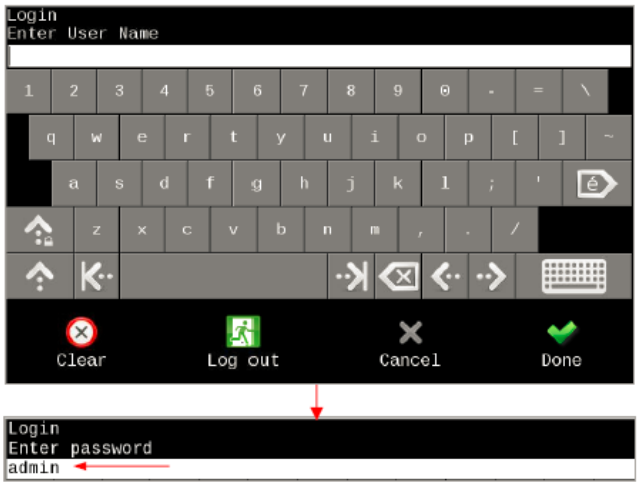


Manage Film List – This screen is used to manage and add additional safety films to the available film list.



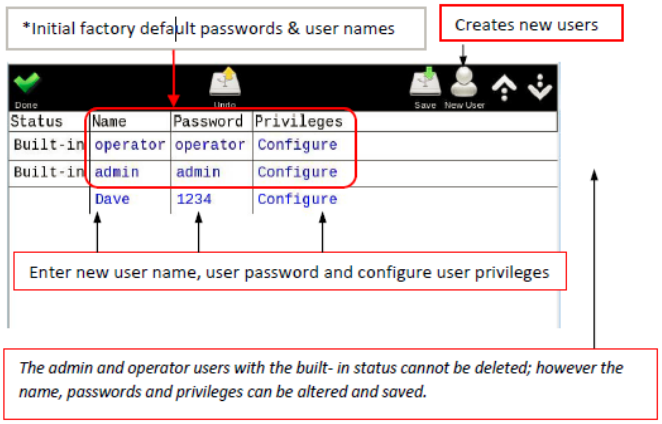
3.8.10 Login

The default admin password is “admin”. Use this screen to access the Users.



3.8.11 Users

Admin sets up and configures additional users and their various privileges.



“Undo” – rolls back the most recent change.
“Save” – Saves changes made.

User Privileges – Built-in

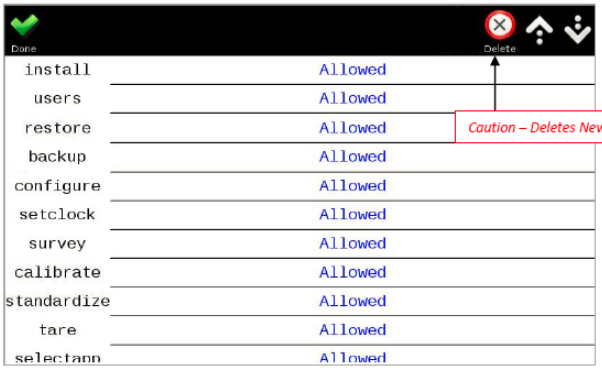
The selections are “Allowed” and “Not Allowed”

Built-In Admin Privileges– All Options

Built–In Operator Privileges - Limited Options

User Privileges – New Users

By default all new users are created as duplicates of admin with full admin privileges therefore it is suggested that specific user privilege limitations be assigned during new user creation. Duplicate or new users can only be deleted from the new user’s privilege screen.



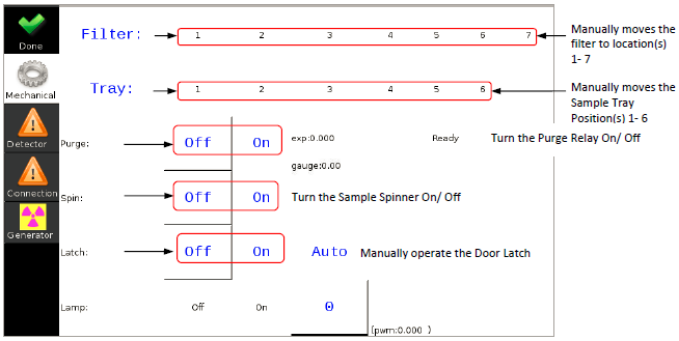
User Privileges – Functions

Install - Allows user to update software
Users - Permits changes to user settings and the “Your preferences” dialog. Also allows editing and creating of other users and their privileges.
Restore - Allows user to restore the user settings from data storage
Backup – Allows user to backup settings to the data storage
Configure – Allows user to make changes to application beyond the measuring standards
Setclock – Set permission to set the date/time
Survey – Enables the survey option button to appear in the hardware configuration,

Calibrate – Allows user to update standardization, import applications and save data
Standardize – Permits user to standardize application if enabled
Tare – Allows user to update the tare value if enabled
Selectapp – Allows user to change the application
Login – Selects if user is required to login

3.8.12 Hardware Monitor Screens

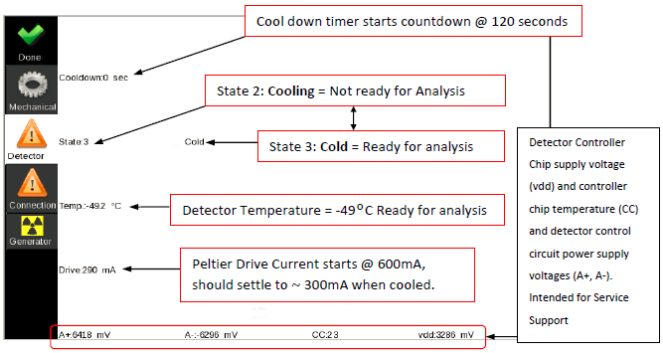
Mechanical Tests – This section is useful for troubleshooting electromechanical functions of the instrument.



*Note: Reboot the Software when leaving the Hardware Monitor if it does not reboot automatically.

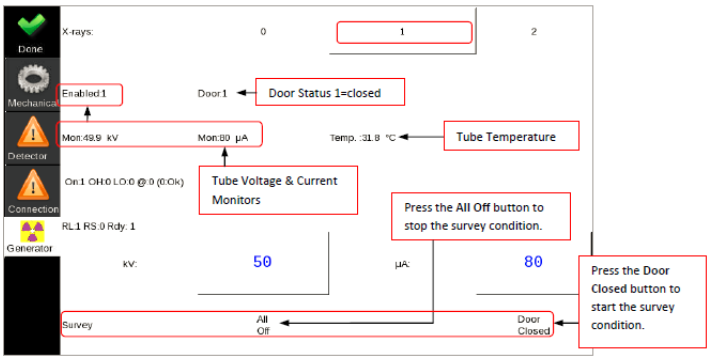
Detector Status

The screen displays the detector's cooling status. Upon power-up the detector starts to cool immediately and should be completely cooled (-49ºC) within 120 seconds. As the detector is cooling the **State 2 Cooling** message will be displayed also the drive current starts off at the maximum value of 600mA and eventually decreases to about 300mA. Once the detector temperature reaches -49ºC the detector is now ready for analysis and the screen and the State will change to **3** and a **Cold** status.



Generator Status / Radiation Survey

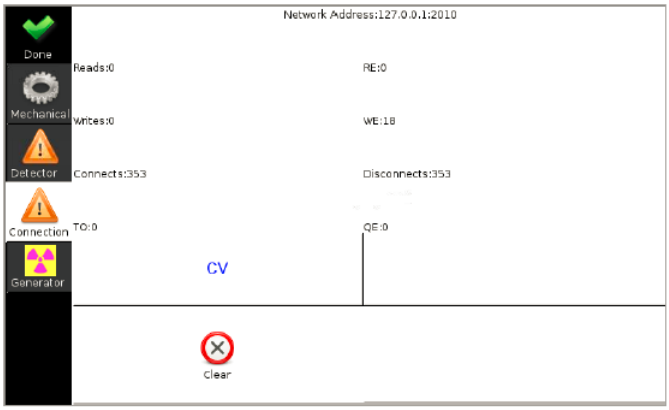
This screen is used to monitor the status of the X-ray tube voltage, current & temperature.



The conditions needed to obtain the radiation survey can be generated from this screen if the user has the privilege enabled. To perform the radiation survey close the door then press the **Door Closed** button to turn on the X-rays. Typically about two minutes should be sufficient to perform a through survey sweep. Once the sweep is completed press the **All Off** button to turn off the X-rays.

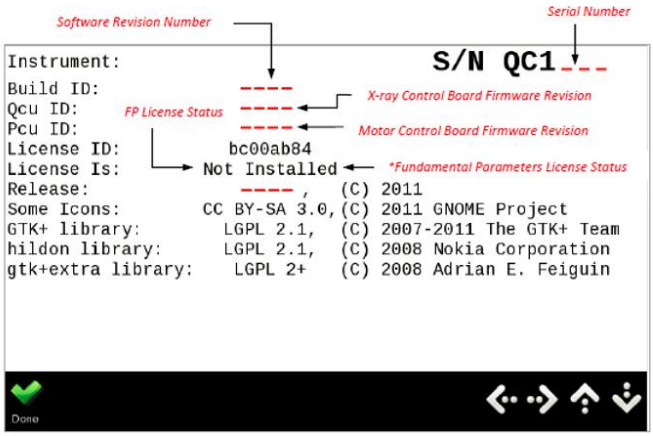
Connection Status

This screen is primarily used for service support and not user supported.



3.8.13 About Screen

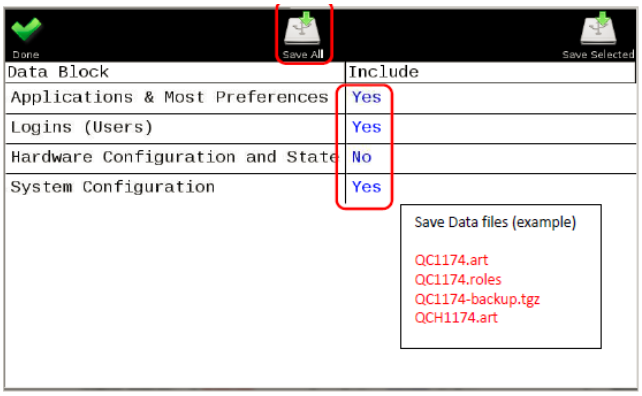
This screen displays the current software, firmware revisions and Fundamental Parameters Package license if purchase with the instrument.



3.8.14 Save Data

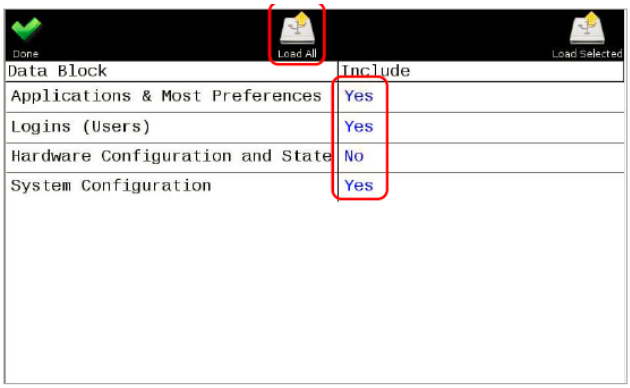
This function saves and exports (as selected) all existing applications and calibration data, user logins and hardware configurations to the selected external drive.

The Data Save function does not save or archive unknown sample data or results. To save and archive unknown sample data results, use the unknown sample result logging features found in the **Application Builder / Settings** screen. The amount of time required to complete the data save process will vary depending on the number of applications present and the complexity of those applications. If pressing the **Save All** button, do not remove the USB drive until the [Done] button appears at the bottom of the screen and the LED light on the USB flash memory stick has completely stopped flashing.



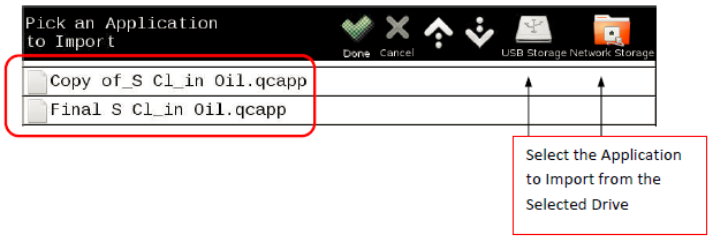
3.8.15 Load Data

This function loads selectable stored **Save Data** applications, calibration data, user logins, and hardware configurations from the selected drive into the instrument. **Data Load** does not restore any unknown sample data or results. Do not press the [Done] button until the LED on the flash drive has completely stopped flashing. Then remove the USB flash memory stick. Loading times may vary depending on the number of applications and the complexity of those applications.



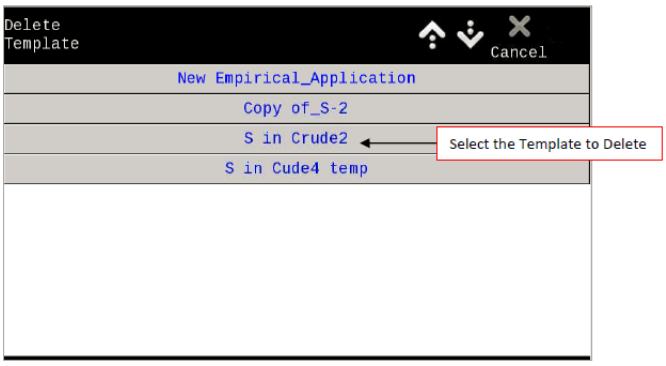
3.8.16 Import Application

Load previous exported application file = "{application name}.qcapp" from external storage (USB flash memory stick) back into the instrument, however does not recover or restore unknown sample data.



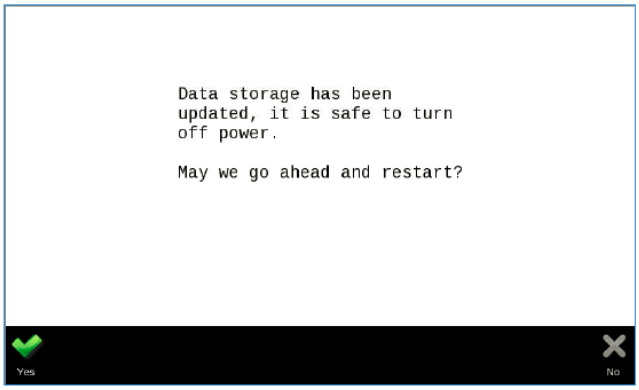
3.8.17 Delete Template

Permanently deletes the selected application template. Do not delete the original factory "New Empirical Application or New FP Application Templates". If inadvertently deleted return to the "User Preferences" and press the restore templates button.



3.8.18 Restart

Saves all current changes and restarts the program.



3.8.19 Save Log

This function saves an operating system debug file for factory support to the external USB drive.

4 Applications

4.1 XRF Theory Basics

*Reading this section on XRF Theory Basics is not an essential requirement to operate or calibrate the EDX1000.

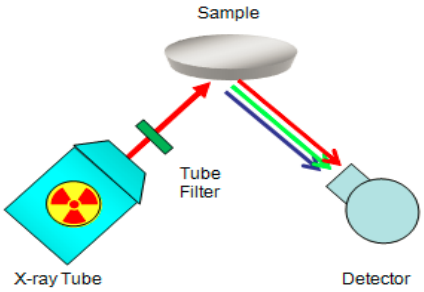
The EDX1000 Benchtop EDXRF uses (Energy Dispersive X-ray Fluorescence) for elemental analysis of sodium (Na) through Uranium (U) in solids, liquids, powders, alloys, etc. Analyzing samples on a calibrated instrument is a very simple task for non-technical personnel after minimal training. This manual section does not go into great detail about EDXRF theory. However, this is a very brief practical overview and introduction of some basic concepts and terms of EDXRF as it relates to the EDX1000.

Advance customer training sessions are available. If interested, please feel free to contact Koehler Instrument Company for scheduling information on the next customer training session. These sessions are designed to provide the user with additional theory, applications, and calibration techniques.

XRF Spectrometry

Using the EDX1000 qualitative and quantitative elemental analysis can quickly be performed via nondestructive X-ray fluorescence spectrometry. In theory, all elements found in the periodic table except (H & He) when properly excited, will emit a unique set of characteristic X-rays or X-ray photons. The energy level (keV) of the X-rays (i.e. photons) correlates to the periodic table element and the relative number of the photons that fluorescence correlates to the concentration of that element present in the sample.

Direct Excitation Concept:

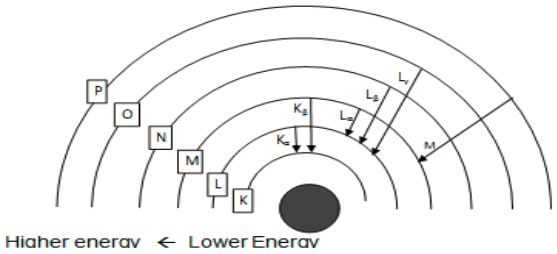


The sample measurement process starts with direct X-ray excitation through an optional selectable tube filter to excite the sample elements to fluoresce their own specific characteristic X-rays. The applied X-rays are at a specified voltage energy level (kV) and at a specified current (µA). Together these produce wattage or power. The formula for power is (Power=Voltage X Current). The EDX1000 X-ray tube is limited to 4 watts. The fluoresced sample X-rays (photons) are then collected by the Silicon PIN Diode Detector.

4.2 Units and Terms

X-rays - are the portion of the electromagnetic spectrum that is often described in terms of nanometer (nm) wavelengths and energy in kilo-electron volts (keV).
kV = kilovolts = tube potential (voltage, V)
Voltage – also called the tube voltage, sets the energy of the source X-rays
keV – kiloelectron-volts = X-ray energy
1V – of potential imparts 1eV of energy to 1 electron
µA – microamps (current, 1millionth of an Ampere)
Current – also called the emission current, sets the number of X-rays
Power = wattage (W = V x A)
W = kV x µA – (4 watts maximum)
cps – counts per second
% - is the wt/wt% when discussing concentration
ppm – parts per million by weight
ppb – parts per billion by weight
intensity – is counts per second (cps) or count rate. This refers to the quantity of X-ray photons striking the detector material over a one second time period.

4.3 Spectrum Lines



- Peaks are named by the shell the electron falls to
- Falls to K shell = K-line
- Falls from L to K = K-alpha line
- Falls from M to K = K-beta line
- M-line series is very weak

Within the structure of all atoms electrons are organized in concentric electron shells or energy bands K,L,M,N,O,P around the central nucleus which consists of proton and neutrons. Also each electron has two relevant to XRF energy values: the "absorption edge" and emission energy".

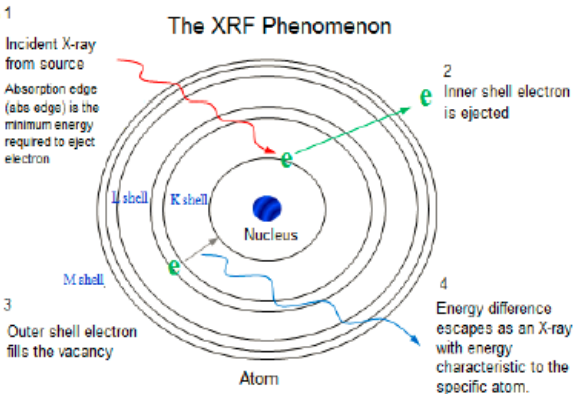
Absorption Edge

In order for a atom to emit a X-ray photon, external energy (incident X-rays) must be applied to the sample from an external source (X-ray tube). The applied energy must be adequate enough in magnitude to allow the inner shell electron to escape from the atom. When the electron escapes or is ejected, a "hole" is created. The energy level at which this occurs is called the "absorption edge".

Emission Energy

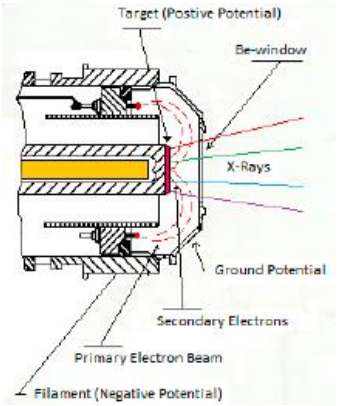
Once the "hole" is created in the inner shell the entire atom becomes unbalanced. In order to restore balance, an outer shell electron drops down to the lower shell position of the ejected electron . As the electron moves downward the energy difference between both shells is expelled as a X-ray photon. This energy is defined as the "emission energy" which is also the energy detected by the analyzer.

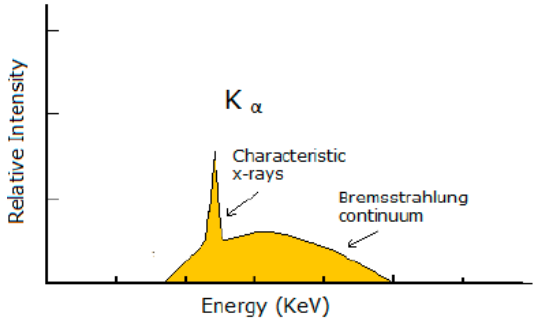
Since all elements have uniquely different electron configurations, multiple emission line energies may be detected. Because of this each element has its own unique characteristic X-ray energy spectrum. If an electron drops down only one shell it is energy level is described as "alpha"α. If it drops down two levels its energy is described as "beta" β.



4.4 X-ray Tube

There are two main types of X-rays produced by X-ray tubes relevant to EDXRF. The primary type and the most useful type is the characteristic X-ray from the target material itself (anode fluorescence). This occurs when the energy of the electron beam applied to the target material is greater than the "absorption edge" of the target material such as Ag, Rh, Pd, etc. The second type of X-ray radiation is called "Bremsstrahlung" radiation, which results from the electrons decelerating or braking as they enter the field surrounding the nuclei of the target material atom. This type of X-ray energy is rarely used in EDXRF due to increased spectrum background noise, however Bremsstrahlung is used in some applications as the source of excitation.





Tube Filters

Filters can be used between the tube and sample. A tube filter creates “quiet spots” in certain energy regions by reducing the background bremsstrahlung radiation. The following chart indicates typical use of filters when setting up a Condition.

FILTER	TYPICAL VOLTAGE (kV)	TYPICAL ELEMENTS TO BE MEASURED
Open	6.5	Na – Cl K-lines Zr, Mo L-lines, alternate for K, Ca K-lines in some applications
A	30	K – Mo K-lines Sn – U L-lines
B	50	Ru – Pr K-lines alternate for K – Br K-lines in some applications
C	35	alternate for K – Mo K-lines in some applications
D	15	alternate for low levels of Ti – Ni K-lines in some applications
E	12	alternate for low levels of K – Ti K-lines in some applications

4.5 Detection

The EDX1000 uses a two staged thermoelectrically cooled (Peltier) solid state semiconductor detector. As the X-ray photons enter the detector semiconductor surface, they are absorbed by the atomic structure. The semiconductor atoms will then eject electrons thereby creating an electrical charge pulse that is in proportion to the energy of absorbed X-ray photons. Typically, (excluding matrix absorption effects, to be discussed later) the greater the concentration of the element of interest present in the sample, the greater the number of EOI characteristic X-ray photons will be emitted from the sample to absorbed by the detector.

EDX1000 uses a Si PIN diode detector.
EDX2000 uses an SDD detector (silicon drift detector, also called silicon drift-chamber detector)

4.6 Shaping Time

The shaping time affects resolution and detector throughput. A long shaping time optimizes resolution, however detector throughput is lowered. Conversely, a short shaping time optimizes detector throughput, however resolution is broader.

The following chart gives guidelines for setting shaping time. Select short shaping time for applications when there are no overlapping peaks. Select medium shaping time when there are peaks that slightly overlap, and select long shaping time for applications measuring adjacent elements. Long shaping time is also typically used for measuring light elements even when no peak overlap occurs.

EDX1000 Si PIN Diode Detector

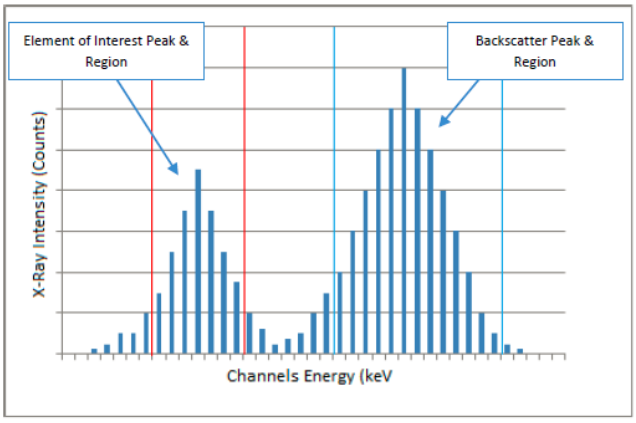
SHAPING TIME (us)	Approx. RESOLUTION (eV)
9	250
4.5	290
3	350

EDX2000 SDD Detector

SHAPING TIME (us)	Approx. RESOLUTION (eV)
4.5	160
2.25	190
1.5	200

4.7 Digital Signal Processor (DSP)

The detector pulses are channelized in to a spectrum by the internal Digital Signal Processor (DSP) unit. The processor generates the spectrum with its multi-channel analyzer algorithm that basically sorts out the pulses by their amplitudes into various counting channels that correlate to fluorescence energy values in keV. The number counts found in each channel can then be plotted to provide the viewable spectra display, however, it is the numerical sum of the channel counts within a specific range of channels during the sampling time period that actually corresponds to EOI fluorescence intensity (i.e. concentration) minus any matrix influence corrections. This specified range of EOI channels in the software is indicated in units of keV and is commonly referred to as “element regions”. Now that we have a numerical value (summarized channel count) that corresponds to the level EOI fluorescence, at a specific known element concentration and after applying corrections for the other influences and effects, a calibration curve can be determined with an appropriate set of assayed calibration standards.



*Simplified MCA Algorithm Concept Example

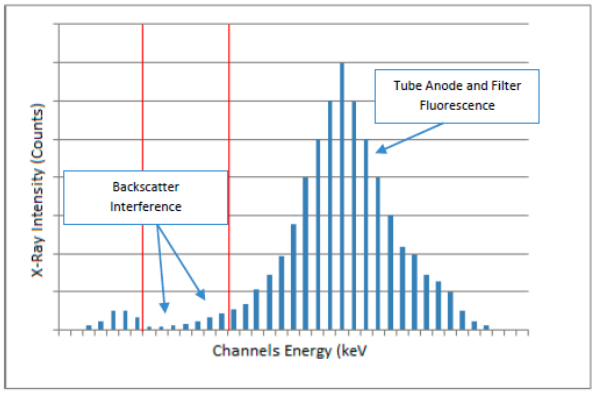
4.8 EDXRF Interferences

The three primary types of spectral interferences that may affect sample results are as follows.

4.8.1 Backscatter Interference

The background signal intensity level (or channel counts) found within the element of interest region when the element is not present. The most common source for this is the overlap spectra from the X-ray tube and filter fluorescence. Some minor additional sources may be environmental or atmospheric radiation and external dirty AC power line noise affecting the electronics.

The correction for backscatter overlap is accomplished by subtracting from sample net EOI region counts from the net counts of the same matrix EOI region but without the EOI being present. Refer to the Calibration Section for more details.

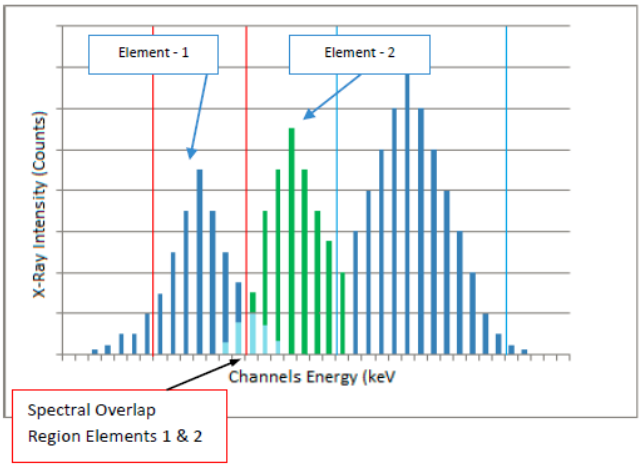


*Use a blank sample with no Na – U for general background correction

4.8.2 Spectral Overlap Interference

This occurs when the fluorescence energy peak of one periodic table element (Na – U) overlaps into the fluorescence energy peak of a nearby adjacent element. The amount of spectral overlapping is dependent on how close the energy peaks are to each other and the resolution of the detector. Spectral Interference is correct for by primarily cross corrections.

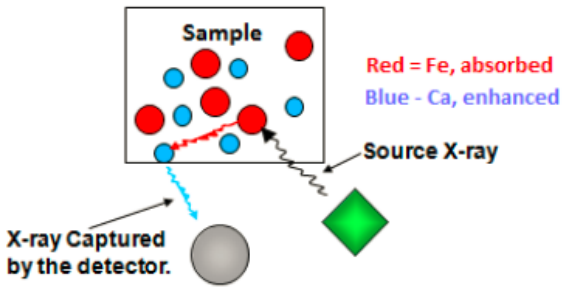
Cross Corrections adjustments are computed by first determining the net counts from an interfering element inside the region of interest of element of interest then subtracting it from the total EOI net counts.



4.8.3 Matrix Effects – Absorption & Enhancement

Absorption – Other elements found in the matrix may absorb or scatter a significant level of the source X-rays resulting in thereby reducing the fluorescence of the element of interest in the sample.

Enhancement – Characteristic X-rays of one element exciting another element of lower energy in the sample, enhancing its signal.



Absorption & Enhancement Example Fe, Ca

- Incoming source X-ray fluoresces Fe
- Fe fluorescence is sufficient in energy to fluoresce Ca
- Ca is detected, Fe is not, Fe is said to enhance Ca
- Ca can also absorb the Fe X-ray
- Incoming source X-ray fluoresces Ca
- Ca fluorescence interacts with Fe atoms
- Ca is not detected. Fe is said to absorb the Ca X-ray

5 Empirical Application Development

5.1 Overview

The operation of each screen is shown in **SECTION 3**. SECTION 5 shows the development of a generic application in order to illustrate the concepts of empirical calibration.

Note: Empirical calibration is often called "matrix-matched calibration." In other words, the matrix of the calibrations standards is the same as the matrix of the unknown samples to be measured.

In order to properly model background, overlap and matrix absorption properties of the application in an empirical calibration, the following samples are used:

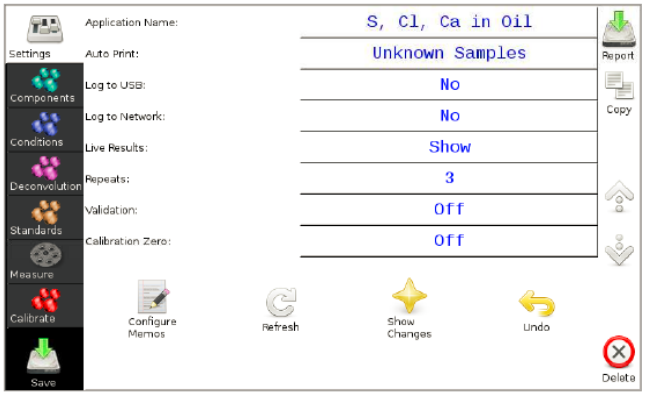
- A set of assayed calibration standards that characterizes the matrix of the unknown samples to be measured.
    - Each standard should contain all of the elements present in the application
    - There should be at least five distinct concentration levels for each element that evenly range from the expected low to the expected high concentration.
    - All the elements in the standards must vary independently of each other.

The basic steps to an empirical calibration are:

- Select elements.
- Develop the measurement Condition(s) to give optimum excitation and detection for the elements, set the Rate and evaluate and set regions.
- Measure blank sample to generate background correction factor.
- Measure Single Element Samples to generate peak overlap correction factors if there are overlapping peaks, also called cross-corrections.
- Define calibration standards and enter the assay values.
- Measure the calibration standards.
- Define the calibration fit model.
    - Linear, hyperbolic or quadratic
        - Linear for narrow concentrations ranges or low levels
        - Hyperbolic or quadratic for broad concentration ranges
        - Enable or disable matrix influence correction factors, also called alpha corrections
        - Add or drop samples from a fit

5.2 Example Calibration

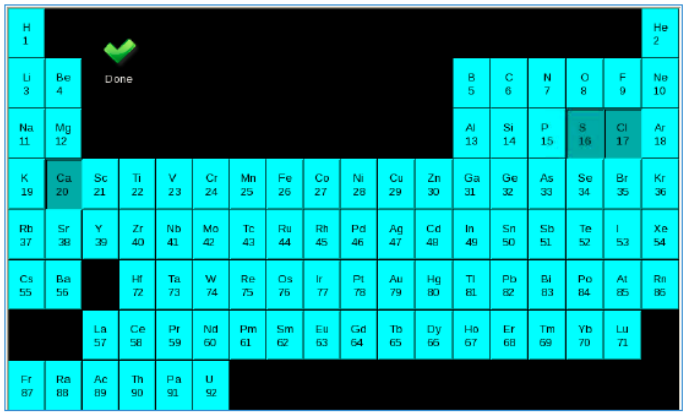
The following S, Cl, Ca in Oil application development is shown to illustrate the principles of empirical calibrations.



Select Elements

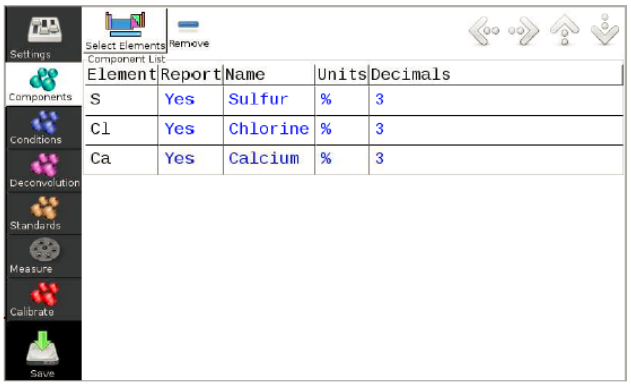
Tap Components on the flow bar then tap the periodic table icon the select elements.

Tap each element to select. Then tap Done.

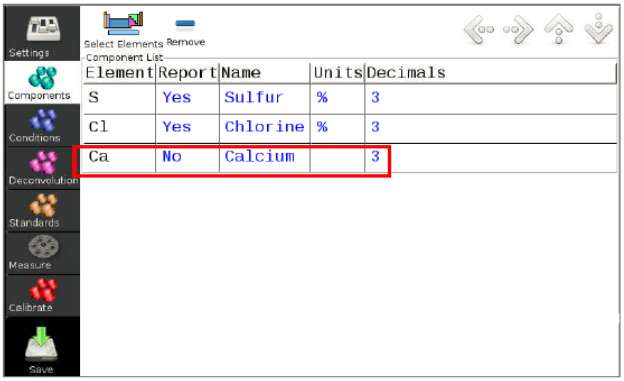


As shown in **SECTION 3,** each entry can be tapped to edit. In this example, there are no

assays for Ca, and so Ca is simply an interfering element, measured in order to enable matrix influence correction factors (alpha corrections) to compensate for Ca absorption and enhancement of S and Cl X-rays

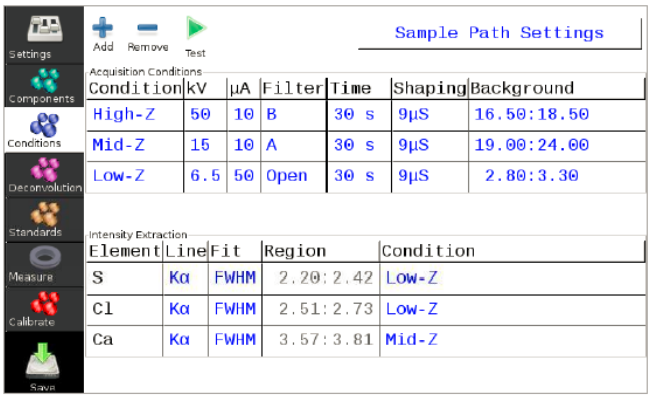


In this example, tap report “Yes” for Ca to toggle it to “No” so that Ca is not shown in the report, and tap the Units for Ca in order to clear that field.



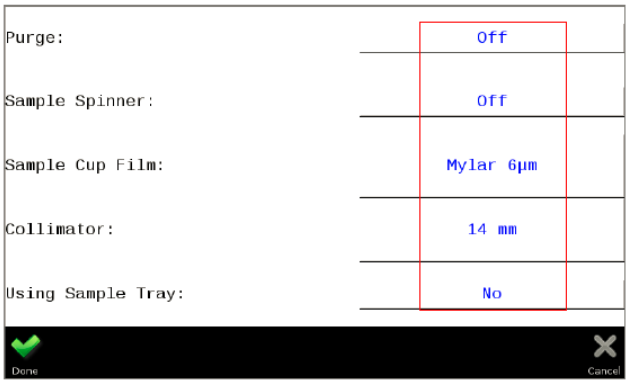
Develop the Measurement Conditions (Acquisitions Conditions)

Tap Conditions in the flow bar and note the default options.



The default settings are starting guidelines, and can be altered by the operator to optimize the particular application. As described in SECTION 3, the Acquisition Configuration can be edited by tapping any setting in blue. Add or delete Conditions using the + or – button. Edit any parameter in a Condition by tapping the entry and setting new value or selecting new entry from a list.

Turn the helium purge or spinner on or off for the application using the toggle buttons at the top of the screen.



Voltage, Filter and Shaping Time are set based on the application.

- **Voltage** is set so that it most efficiently excites the filter used
- The **Filter** is selected based on the group of elements to be measured with that Condition
- **Shaping Time** is selected based on elemental peak overlap
  - Use short shaping time when there are no overlapping peaks to be measured using that Condition
  - Use middle shaping time for medium resolution and throughput
  - Use long shaping time when there are adjacent elements present

Use these charts to help determine these settings:

FILTER	TYPICAL VOLTAGE (kV)	TYPICAL ELEMENTS TO BE MEASURED
Open	6.5	Na – Cl K-lines Zr, Mo L-lines, alternate for K, Ca K-lines in some applications
A	30	K – Mo K-lines Sn – U L-lines
B	50	Ru – Pr K-lines alternate for K – Br K-lines in some applications
C	35	alternate for K – Mo K-lines in some applications
D	15	alternate for low levels of Ti – Ni K-lines in some applications
E	12	alternate for low levels of K – Ti K-lines in some applications

EDX1000 Si PIN Diode Detector

SHAPING TIME (us)	Approx. RESOLUTION (eV)
9	250
4.5	290
3	350

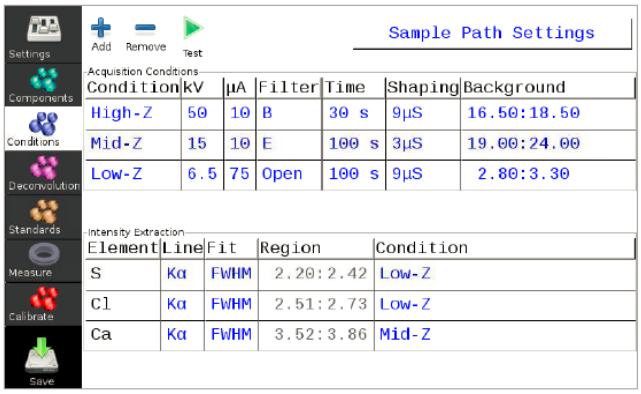
EDX2000 SDD Detector

SHAPING TIME (us)	Approx. RESOLUTION (eV)
4.5	160
2.25	190
1.5	200

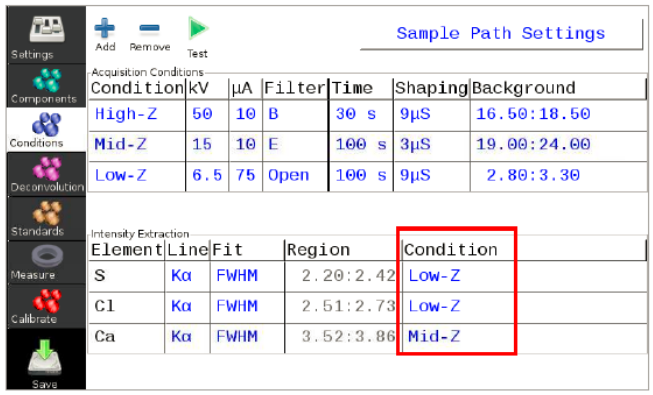
The general rule for setting the measurement Time is that the longer the measurement time, the less statistical error in the measurement. Typically, measurement Times between 60-300 sec are used. However, measurement times between 10-600 sec may also be used, depending on application and performance requirements.

The name of a Condition can be changed to any name desired by tapping the name of the Condition and editing it.

To continue the example of S, Cl and Ca in Oil, edit the Name, Voltage, Filter, and Shaping Time as per the above charts by tapping each field entry to edit. Also, select 100 seconds for each Condition's measurement Time.



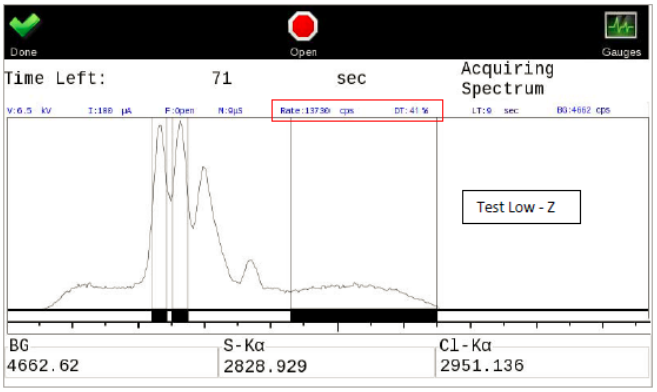
Next, associate each element with a Condition. For an element, tap the Condition field in the Intensity Extraction table and select the Condition to be used for that element.



Trial and Error Using the Test Button

The setup for each Condition is tested by tapping the Test button and then tapping a Condition. Trial-and-error is used to set the emission current (uA) and to adjust the background and elemental regions.

Setting the current:
Place the highest concentration calibration standard on the analyzer, tap the Test button and then tap the Condition, and view the results in the test measurement screen using the Conditions setup so far. When using Test to set the current, look at the Dead Time (DT).



- The Rate is the overall count rate in cps going into the detector. This is also called the throughput.

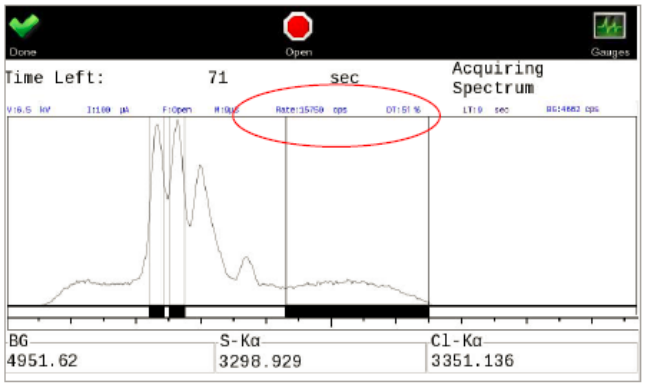
- The Dead Time is the amount of time spent processing detector pulses rather than gathering detector pulses.

Begin with a low current and take a Test measurement for a Condition, DT. Click Done to stop the test and return to the Conditions screen. Adjust the current up and take another Test measurement. Repeat this procedure until the highest DT is achieved without going over 40% for

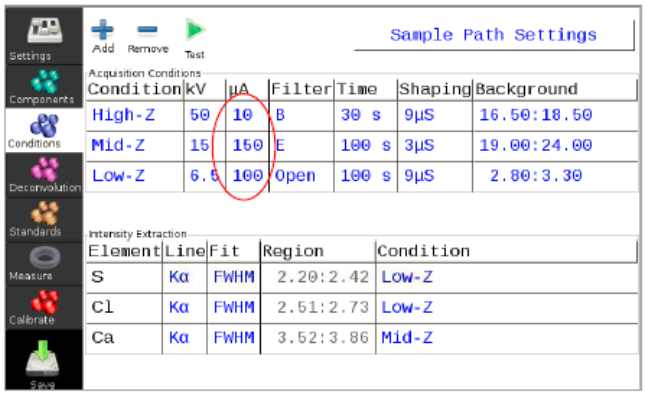
empiricals or 30% for FP applications. Increase the current until the DT target is met.

- By trial-and-error, begin with a low current and increment by 5-20uA steps until DT is 40% for empirical applications, 30% for FP applications, or max power is achieved.

After several trial-and-error iterations of slowly editing the current starting at a low value, the following currents were determined for the S, Cl and Ca in Oil example. In a general application with more than one Condition, repeat the trial-and-error process to set the current for each Condition used in the method.



At this point in the example, the current has been set:

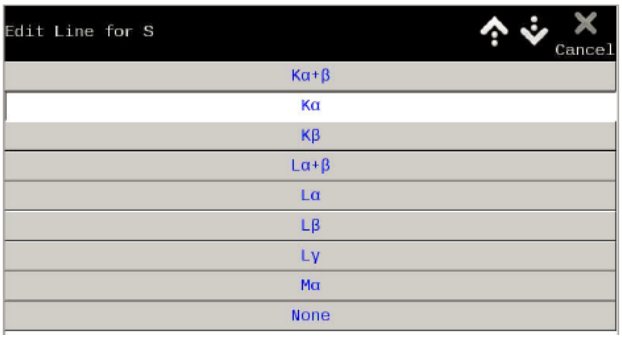


Next, use the same procedure of using the Test button and trial-and-error to evaluate and adjust the regions for each element and the background for each Condition.

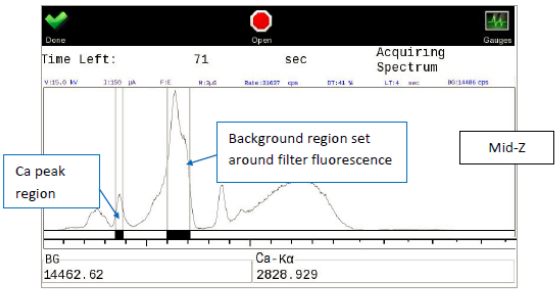
Adjust the background region for each Condition by tapping the Background area for the Condition in the Acquisition Configuration table. Use these rules to adjust a background region:

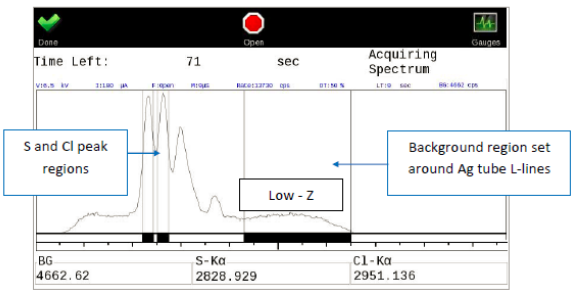
- The background region for a Condition must be higher energy than the elements measured by that Condition
- The background region must have sufficient cps to ensure good counting statistics
- There must be no element fluorescence in the background region
- The background region is typically set around:
  - The Ag tube lines (either Ag-Ka or Ag-La)
  - The filter fluorescence
  - A large area of bremsstrahlung radiation

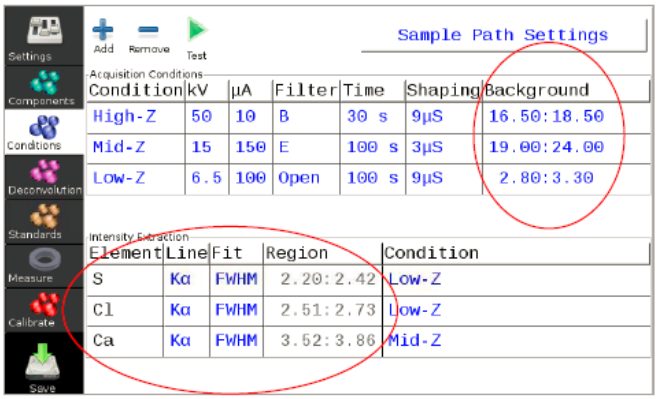
To adjust an element's region, select line for that element in the Intensity Extraction table. Select from a predefined line (Ka, La, etc.) or adjust a region manually by selecting Manual.



To continue the S, Cl and Ca in Oil example, several iterations are viewed using the Test button, and the regions are adjusted for optimum performance.



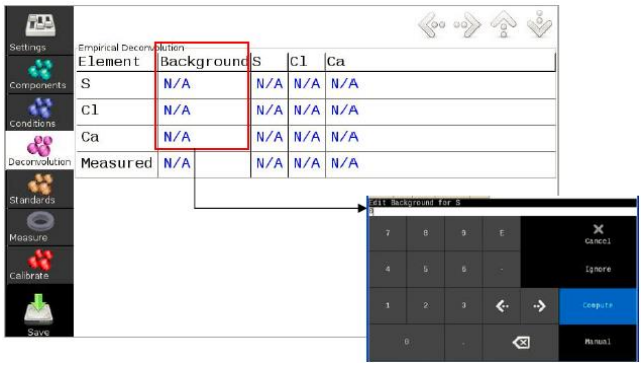


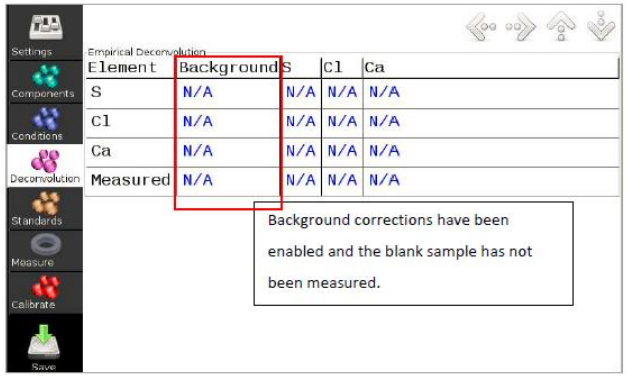


De-Convolutions

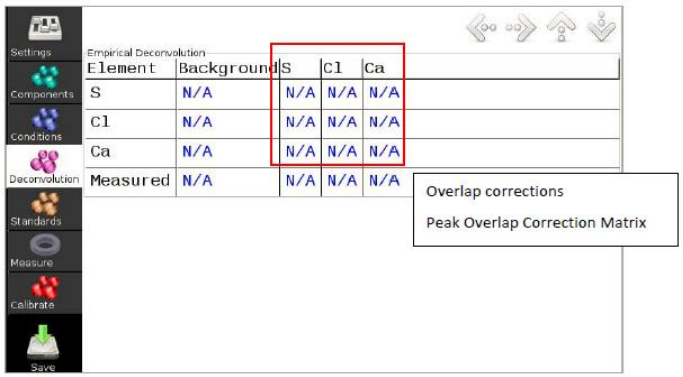
Typically when using Si PIN diode or SDD detector overlap corrections are not required. If desired, overlap corrections can be employed by measuring single element samples. Tap Deconvolutions in the flow bar. The deconvolutions are the background correction and peak overlap corrections. Note: The row labeled Measured indicates whether or not the required sample for that deconvolution has been measured.

To enable background correction, tap the N/A in the Background column for each element and set to Compute.



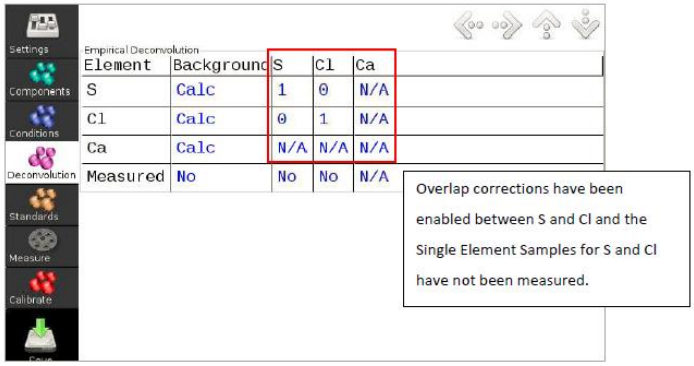


Overlap corrections, also called cross-corrections or X-cor, are calculated by linear algebra and matrix math. To read the matrix, elements in the column headers are interfering elements, while row labels indicate the elements as elements of interest. In order for proper overlap corrections to be calculated, a matrix of overlapping elements needs to be enabled in a symmetrical manner.



By similar process of tapping N/A, set appropriate N/A areas to Compute for elements that overlap. The element overlapping itself is also enabled, so that matrix math returns proper correction factors.

In the example S, Cl and Ca in Oil, Ca does not overlap any elements, but S and Cl overlap each other. The initial matrix is filled out by selecting Compute for S overlap S, Cl overlap S, S overlap Cl and Cl overlap Cl.



Notice that the Measured column indicates samples for deconvolutions have not yet been measured.

For background correction, a blank sample is measured. The blank can be the same matrix as the actual samples, yet containing no measureable elements, or a similar blank matrix. When an actual blank sample is not available, DI water or mineral oil can be used for liquid matrices, or the Teflon or Polyethylene supplied with the analyzer

can be used for solid matrices. As a blank powder, research grade boric acid is often used or other suitable inert blank powder.

To calculate overlap corrections, a Single Element Sample for each overlapping element is measured. The Single Element Sample for a particular element need not be the same matrix as the samples, but must follow these rules:

- Contains only that element.
- Gives a good peak for that element with as many cps as possible.
- Must give a Rate of <40% DT when measured with its condition.

Each deconvolution can be measured in the deconvolutions screen by pressing the No value in the measured row, or the deconvolution samples can be measured in the Measure screen just before the calibration standards.

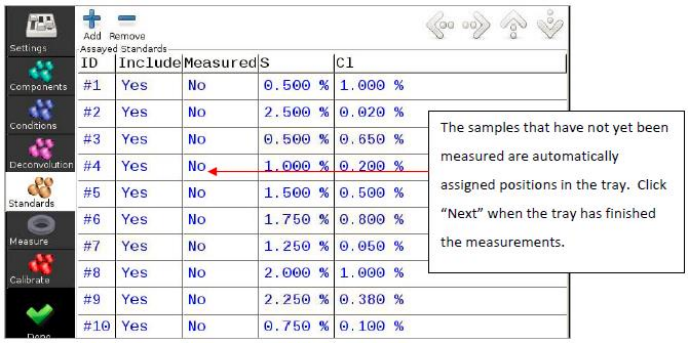
Standards

Tap Standards in the flow bar. Add a standard by tapping the + button. Enter an ID by tapping the blank space in the ID column and entering an ID. Enter assays by tapping the assay entry field and entering the assay value.

ID	Include	Measured	S	Cl
#1	Yes	No	0.500 %	1.000 %
#2	Yes	No	2.500 %	0.020 %
#3	Yes	No	0.500 %	0.650 %
#4	Yes	No	1.000 %	0.200 %
#5	Yes	No	1.500 %	0.500 %
#6	Yes	No	1.750 %	0.800 %
#7	Yes	No	1.250 %	0.050 %
#8	Yes	No	2.000 %	1.000 %
#9	Yes	No	2.250 %	0.380 %
#10	Yes	No	0.750 %	0.100 %

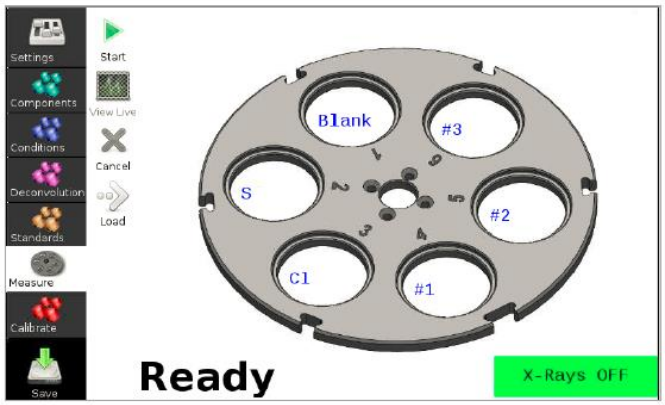
Measure

Tap Measure to measure the blank, Single Element Samples and calibration standards.



ID	Include	Measured	S	Cl
#1	Yes	No	0.500 %	1.000 %
#2	Yes	No	2.500 %	0.020 %
#3	Yes	No	0.500 %	0.650 %
#4	Yes	No	1.000 %	0.200 %
#5	Yes	No	1.500 %	0.500 %
#6	Yes	No	1.750 %	0.800 %
#7	Yes	No	1.250 %	0.050 %
#8	Yes	No	2.000 %	1.000 %
#9	Yes	No	2.250 %	0.380 %
#10	Yes	No	0.750 %	0.100 %

Press Load to indicate which samples need to be measured. Place samples in the tray in the positions shown on the screen. Press Start to begin the measurements. When complete press Load and the remaining samples to be measured will populate the tray.



After all calibration samples have been measured, continue to the Calibrate screen.

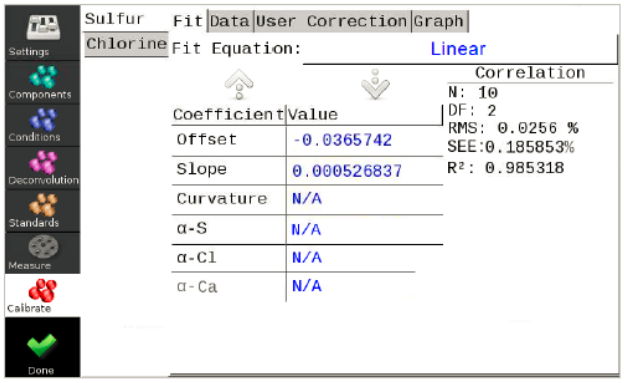
Calibrate

Tap Calibrate to view and edit the calibration fit model for each element. The following are some general guidelines:

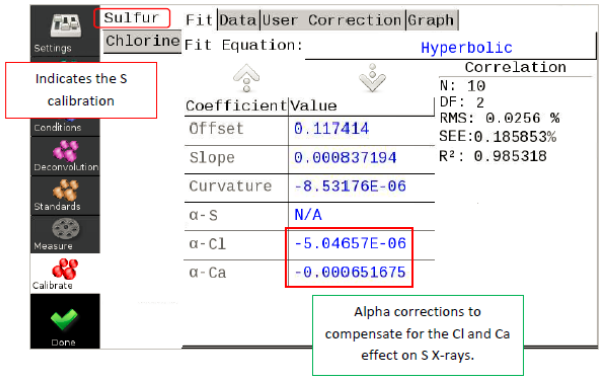
- All elements show a curved response across the range 0-100% due to self-absorption.
- Light elements respond linear for a larger part of their concentration ranges than heavier elements.
- Use a linear fit for low concentration ranges or narrow concentration ranges.
- Use a hyperbolic or quadratic fit for broad concentration ranges.

- Alpha correction compensate for variations in X-ray absorption and enhancement within the sample due to variations in elemental concentration among samples. Enable an alpha correction for each interfering element if the interfering element concentration varies in the application and is able to fluoresce or absorb the X-rays of the element being analyzed.

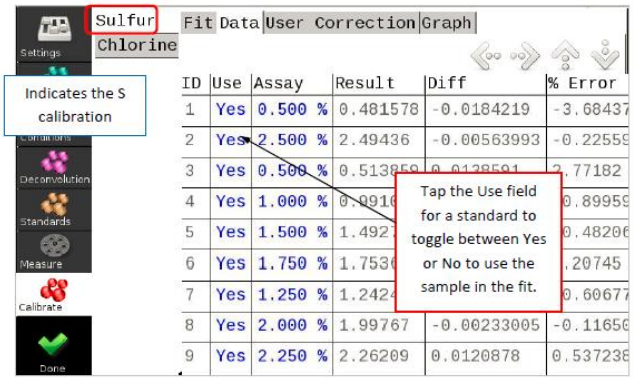
- Basic guidelines for selecting alpha corrections:
  - Elements at higher concentrations should be considered first, no matter the proximity to the element of interest's absorption edge
  - Consider major and minor elements near the element's absorption edge
  - Consider elements that vary greatly in concentration
  - Trace elements typically do not exhibit noticeable absorption / enhancement effects
  - The element must vary significantly in concertation to be considered for use as an alpha correction



To enable or change a selection, tap any field in blue font. Evaluate the fit by evaluating the SEE (Standard error of Estimate) and the R2 value (confidence).

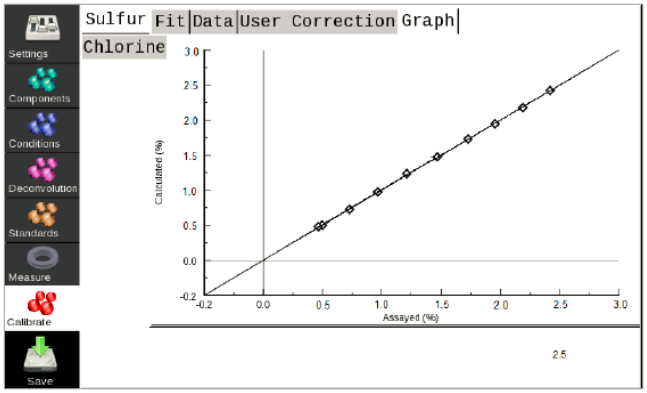


Select the Data and the Graph tabs to review the correlation information. Use the "Data" tab to add or drop samples from the fit.



Repeat this procedure for each element in the calibration. Note that in this example of S, Cl and Ca in Oil, there is no calibration for Ca because the Report toggle was set to No in the Component section at the beginning. Therefore, no assays column was available for Ca in the Standards screen and no fit in the Calibrate screen. However, Ca was measured in order to enable its alpha correction for its effect on the S and Cl fits.

Graph - Correlation



When calibration is complete, click Done in the flow bar.

6 EDX1000 Specifications

Excitation

- 50 kV X-ray tube
- 4W max power
- 6 standard tube filters with shutter

Detection

- High performance Semi-Conductor Detector
- Peltier electronic cooling
- Optimum balance of spectral resolution and max count rate

Sample Chamber

- Large 190 x 165 x 60 mm sample chamber
- Single position 32 mm sample aperture
- Single position 40 mm sample aperture
- Bulk sample aperture
- 6-position 32 mm automatic sample changer
- 5-position 40 mm automatic sample changer
- Single position 32 mm sample spinner
- Analysis in air or helium

Software & Application Packages

- Qualitative and quantitative analysis
- Normalization and validation feature
- Data export function
- LIMS compatible
- Simple flow bar wizard to create your own applications
- Icon driven software for control of spectrometer functions and data analysis
- Fundamental parameters (optional)

Environmental Conditions

- Ambient temperatures 10 - 35°C (50 - 95°F)
- Relative humidity <85% non condensing
- Vibration undetectable by human
- Free from corrosive gas, dust, and particles

User Interface

- 8” WVGA touch screen interface
- Embedded computer
- Internal thermal printer
- USB port
    - 1.0 or 2.0 Flash Memory Stick
    - Mouse connection
- Ethernet connection

Spectrometer Data

- Single phase AC 100/240V, 1.4A/0.7A (50/60Hz)
- Dimensions:331 (W) x 432 (D) x 376 (H) mm (13 x 17 x 14.8 in)
- Weight: 16kg (35lbs)

Options

- 6-position 32mm automatic sample changer
- 5-position 40 mm automatic sample changer
- Single position 32 mm sample spinner
- Helium purge (99.95% purity)
- Fundamental parameters

7 Warranty, Terms & Conditions

Warranty

We at Koehler would like to thank you for your equipment purchase, which is protected by the following warranty. If within one (1) year from the date of receipt, but no longer than fifteen (15) months from the date of shipment, Koehler equipment fails to perform properly because of defects in materials or workmanship, Koehler Instrument Company, Inc. will repair or, at its sole discretion, replace the equipment without charge F.O.B. its plant, provided the equipment has been properly installed, operated, and maintained. Koehler Instrument Company must be advised in writing of the malfunction and authorize the return of the product to the factory. The sole responsibility of Koehler Instrument Company and the purchaser's exclusive remedy for any claim arising out of the purchase of any product is the repair or replacement of the product. In no event shall the cost of the purchaser's remedy exceed the purchase price, nor shall Koehler Instrument Company be liable for any special, indirect, incidental, consequential, or exemplary damages. KOEHLER INSTRUMENT COMPANY, INC. DISCLAIMS ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING ANY IMPLIED WARRANTIES OF FITNESS FOR A PARTICULAR PURPOSE. Please save the shipping carton in the event the equipment needs to be returned to the factory for warranty repair. If the carton is discarded, it will be the purchaser's responsibility to provide an appropriate shipping carton.

Returned Goods Policy

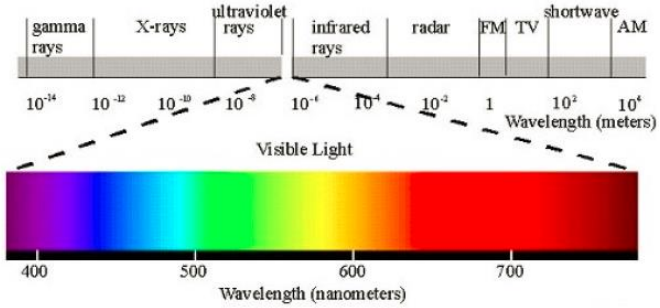
To return products for credit or replacement, please contact Koehler Customer Service with your purchase order number, our packing list/invoice number, the item(s) to be returned and the reason for the return. You will be issued a Returned Authorization (RA) number, which must be prominently displayed on the shipping container when you return the material to our plant. Shipping containers without an RA number prominently displayed with be returned to the sender. Goods must be returned freight prepaid. Returns will be subject to a restocking charge, the application of which will depend upon the circumstances necessitating the return. Some returns cannot be authorized, including certain products purchased from outside vendors for the convenience of the customer, products manufactured on special order, products shipped from the factory past ninety (90) days, and products which have been used or modified in such a way that they cannot be returned to stock for future sale.

8 Radiation Safety

Characteristics of Radiation

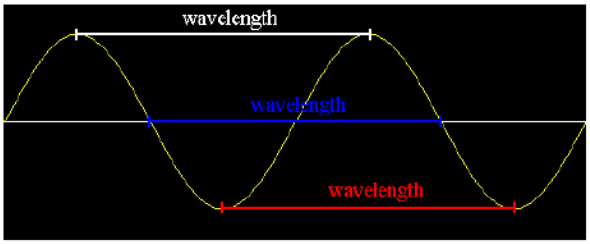
X- and gamma rays are part of what scientists refer to as the electromagnetic spectrum. They are waveforms that are part of a family in which some of the relatives are very familiar to us, such as light rays, infrared heat rays, and radio waves. However, X- and gamma rays cannot be seen, felt, or heard. Our normal senses cannot detect them. Since X- and gamma rays have no mass and no electrical charge, they are not influenced by electrical and magnetic fields and will travel in straight lines. Continued research over the years since Roentgen's discovery indicated that the radiation possesses a dual character. Acting somewhat like a particle at times and like a wave at other times. The name that has been given to the small "packets" of energy with these characteristics is "photon." It is said that the radiation photon is a wave that is both electric and magnetic in nature.

This diagram shows the electromagnetic spectrum. Notice the changes in wavelengths of the various waveforms.



Every point across the spectrum represents a waveform of differing wavelength. It should be noted that the lines between the groupings are not precise, and that each group phases into the next.

Waveforms may be graphically represented as following:



Sources of Radiation

X-Ray Tubes

A type of radiation commonly utilized is X-radiation. Whereas gamma radiation is one of the products of nuclear decay of radioactive elements, X-rays are produced in high voltage electron tubes. W.C. Roentgen discovered X-rays in the late 1800's while working with a cathode tube in his lab. X-rays can be produced in parcels of energy called photons, just like light.

To generate X-rays, three things are required. We need a source of electrons, a means of accelerating the electrons at high speeds, and a target material to receive the impact of the electrons and interact with them.

X-rays are generated when free electrons give up some of their energy when they interact with the orbital electrons or nucleus of an atom. The energy given up by the electron during this interaction appears as electromagnetic energy known as X-radiation. There are two different atomic processes that can produce X-ray photons. One is called Bremsstrahlung and the other is called K-shell emission. X-rays produced by Bremsstrahlung are the most useful for medical and industrial applications.

Units of Radiation Dose

There are four measures of radiation you will commonly encounter. These are: Activity, Exposure, Absorbed Dose, and Dose Equivalent. A short summary of these measures and their units will be followed by more in depth information.

Activity: The activity of a radioactive source is defined as the rate at which the isotope decays. Radioactivity may be thought of as the volume of radiation produced in a given amount of time. The International System (SI) unit for activity is the Becquerel (Bq) and the curie (Ci) is also commonly used.

Exposure: Exposure is a measure of the strength of a radiation field at some point in air. This is the measure made by a survey meter. The most commonly used unit of exposure is the roentgen (R).

Absorbed Dose: Absorbed dose is the amount of energy that ionizing radiation imparts to a given mass of matter. The SI unit for absorbed dose is the gray (Gy), but the “rad” (Radiation Absorbed Dose) is commonly used. 1 rad is equivalent to 0.01 Gy. Different materials that receive the same exposure may not absorb the same amount of energy. In human tissue, one Roentgen of gamma radiation exposure results in about one rad of absorbed dose.

Dose Equivalent: The dose equivalent relates the absorbed dose to the biological effect of that dose. The absorbed dose of specific types of radiation is multiplied by a "quality factor" to arrive at the dose equivalent. The SI unit is the sievert (SV), but the rem is commonly used. Rem is an acronym for "roentgen equivalent in man." One rem is equivalent to 0.01 SV. When exposed to X- or Gamma radiation, the quality factor is 1.

More Information on Radiation Units

Activity

The strength of a radioactive source is called its activity, which is defined as the rate at which the isotope decays. Radioactivity may be thought of as the volume of radiation produced in a given amount of time. It is similar to the current control on an X-ray tube. The International System (SI) unit for activity is the Becquerel (Bq), which is that quantity of radioactive material in which one atom transforms per second. The Becquerel is a small unit. In practical situations, radioactivity is often quantified in kilobecqerels (kBq) or megabecquerels (MBq). The curie (Ci) is also commonly used as the unit for activity of a particular source material. The curie is a quantity of radioactive material in which 3.7 x 1010 atoms disintegrate per second. This is approximately the amount of radioactivity emitted by one gram (1 g) of Radium 226. One curie equals approximately 37,037 MBq. The sources we will encounter will be 100 mCi or less activity.

The activity of a given amount of radioactive material does not depend upon the mass of material present. For example, two 100 milli-curie sources of Fe-55 might have very different masses depending upon the relative proportion of non-radioactive atoms present in each source. The concentration of radioactivity, or the relationship between the mass of radioactive material and the activity, is called the specific activity. Specific activity is expressed as the number of curies or becquerels per unit mass or volume. The higher the specific activity of a material, the smaller the physical size of the source is likely to be.

Exposure

Exposure is a measure of the strength of a radiation field at some point. It is a measure of the ionization of the molecules in a mass of air. It is

usually defined as the amount of charge (i.e. the sum of all ions of the same sign) produced in a unit mass of air when the interacting photons are completely absorbed in that mass. The most commonly used unit of exposure is the Roentgen (R). Specifically, a Roentgen is the amount of photon energy required to produce 1.610 x 1012 ion pairs in one cubic centimeter of dry air at 0°C. A radiation field of one Roentgen will deposit 2.58 x 10-4 coulombs of charge in one kilogram of dry air. The main advantage of this unit is that it is easy to directly measure with a survey meter. The main limitation is that it is only valid for deposition in air.

Absorbed Dose
Whereas exposure is defined for air, the absorbed dose is the amount of energy that ionizing radiation imparts to a given mass of matter. The most commonly used unit for absorbed dose is the "rad" (Radiation Absorbed Dose). A rad is defined as a dose of 100 ergs of energy per gram of the given material. The SI unit for absorbed dose is the gray (Gy), which is defined as a dose of one joule per kilogram. Since one joule equals 107 ergs, and since one kilogram equals 1000 grams, 1 Gray equals 100 rads.

The size of the absorbed dose is dependent upon the energy of the radiation, the strength (or activity) of the radiation source, the distance from the source to the irradiated material, and the time over which the material is irradiated. The activity of the source will determine the dose, rate which can be expressed in rad/hr, mr/hr, mGy/sec, etc.

Dose Equivalent
When considering radiation interacting with living tissue, it is important to also consider the type of radiation. Although the biological effects of radiation are dependent upon the absorbed dose, some types of radiation produce greater effects than others for the same amount of energy imparted. For example, for equal absorbed doses, alpha particles may be 20 times as damaging as beta particles. In order to account for these variations when describing human health risks from radiation exposure, the quantity called "dose equivalent" is used. This is the absorbed dose multiplied by certain "quality" or "adjustment" factors indicative of the relative biological-damage potential of the particular type of radiation.

The quality factor (Q) is a factor used in radiation protection to weigh the absorbed dose with regard to its presumed biological effectiveness. Radiation with higher Q factors will cause greater damage to tissue. The rem is a term used to describe a special unit of dose equivalent. Rem is an abbreviation for roentgen equivalent in man. The SI unit is the sievert (SV); one rem is equivalent to 0.01 SV. Doses of radiation received by workers are recorded in rems, however, sieverts are being required as the industry transitions to the SI unit system.

The table below presents the Q factors for several types of radiation.

Type of Radiation	Rad	Q Factor	Rem
X-ray	1	1	1
Gamma Ray	1	1	1
Beta Particles	1	1	1
Thermal Neutrons	1	5	5
Fast Neutrons	1	10	10
Alpha Particles	1	20	20

Significance of Radiation Dose

Radiation Protection Standards
The standards set forth in 10 CFR Part 20 for the protection against ionizing radiation will be followed.
http://www.nrc.gov/reading-rm/doc-collections/cfr/part020/

Biological Effects of Radiation
The occurrence of particular health effects from exposure to ionizing radiation is a complicated function of numerous factors including:

- **Type of radiation involved.** All kinds of ionizing radiation can produce health effects. The main difference in the ability of alpha and beta particles and Gamma and X-rays to cause health effects is the amount of energy they have. Their energy determines how far they can penetrate into tissue and how much energy they are able to transmit directly or indirectly to tissues.
- **Size of dose received**. The higher the dose of radiation received, the higher the likelihood of health effects.
- **Rate the dose is received.** Tissue can receive larger dosages over a period of time. If the dosage occurs over a number of days or weeks, the results are often not as serious if a similar dose was received in a matter of minutes.
- **Part of the body exposed**. Extremities such as the hands or feet are able to receive a greater amount of radiation with less resulting damage than blood forming organs housed in the torso.

- **The age of the individual.** As a person ages, cell division slows and the body is less sensitive to the effects of ionizing radiation. Once cell division has slowed, the effects of radiation are somewhat less damaging than when cells were rapidly dividing.
- **Biological differences.** Some individuals are more sensitive to the effects of radiation than others. Studies have not been able to conclusively determine the differences.

Exposure Symptoms
Listed below are some of the probable prompt and delayed effects of certain doses of radiation when the doses are received by an individual within a twenty-four hour period. Dosages are in Roentgen Equivalent Man (Rem).

0-25	No injury evident. First detectable blood change at 5 rem.
25-50	Definite blood change at 25 rem. No serious injury.
50-100	Some injury possible.
100-200	Injury and possible disability.
200-400	Injury and disability likely, death possible.
400-500	Median Lethal Dose (MLD) 50% of exposures are fatal.
500-1,000	Up to 100% of exposures are fatal.
1,000+	100% likely fatal.

The delayed effects of radiation may be due either to a single large overexposure or continuing low-level overexposure.

Example dosages and resulting symptoms when an individual receives an exposure to the whole body within a twenty-four hour period.

100 – 200 Rem:

First Day	No definite symptoms
First Week	No definite symptoms
Second Week	No definite symptoms
Third Week	Loss of appetite, malaise, sore throat and diarrhea
Fourth Week	Recovery is likely in a few months unless complications develop because of poor health.

400 – 500 Rem:

First Day	Nausea, vomiting and diarrhea, usually in the first few hours
First Week	Symptoms may continue
Second Week	Epilation, loss of appetite
Third Week	Hemorrhage, nosebleeds, inflammation of mouth and throat, diarrhea, emaciation

Levels of Radiation from Sources of Radiation

Radiation is part of our natural environment. We are exposed to radiation from materials in the earth, naturally occurring radon in the air, from outer space, and from inside our own bodies (as a result of the food and water we consume). This radiation is measured in units called millirems (mrems).

The average dose per person from all sources is about 360 mrems per year. However, it is not uncommon for any of us to receive far more than that in a given year due to due to medical procedures we may undergo. International Standards allow exposure to as much as 5,000 mrems a year for those who work with and around radioactive material.

Where you live		
	Gulf or Atlantic Coast State	28 mrem/yr
	Cosmic Radiation Denver, Colorado Area	63 mrem/yr
	Live within 50 mile of a nuclear plant	0.01 mrem/yr
	Live within 50 miles of a coal-fired plant	0.03 mrem/yr
Food & Water		
	From food (Carbon-14 and Potassium-40) and from Water (radon)	40 mrem/yr
	From air (radon)	200 mrem/yr
How You Live		
	Air travel	0.5 mrem/hr
	Watch TV (CRT)	1 mrem/yr
	Video Display Terminal (CRT)	1 mrem/yr
	Plutonium powered pacemaker	100 mrem//yr
Medical Test		
	X-ray – Extremity	1 mrem
	X-ray – Dental	1 mrem
	X-ray – Chest	6 mrem
	X-ray – Barium Enema	405 mrem
	Nuclear Medicine (e.g. thyroid scan)	14 mrem

Radiation Dose Limits for Occupational Workers and Individual Members of the Public

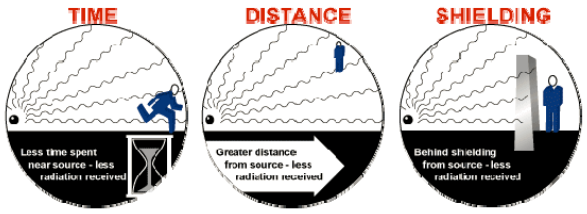
US Guidelines

Organ	NRC Limit (mrem/year)	Comments
Whole Body	5,000	Includes dose from both external and internal sources
Lens of the Eye	15,000	
Single Organ	50,000	Other than eye.
Extremities	50,000	The extremities include the arm or leg below the elbow or knee
Skin	50,000	
Embryo/Fetus	500 (for the entire pregnancy)	Applies only when a *Declaration of Pregnancy* has been submitted. Must be evenly distributed.
Occupational exposure of a minor	10% of the limits above	Applies to anyone under 18 years of age
Member of the General Public	100	

Canada Guidelines

	ICRP Effective Dose Limits (mSv)	Comments
Radiation Worker	20 (Average in a year) 50 (special circumstances)	
Pregnant Radiation Worker	2 mSv for the remainder of pregnancy	In the case of a female radiation worker who is pregnant, the fetus must be protected from radiation exposure for the remainder of the pregnancy once pregnancy has been diagnosed.
Member of the General Public	1	
Organ	**ICRP Equivalent Dose Limits (mSv/year)**	**Comments**
Lens of the Eye	150	Worker
Lens of the Eye	15	Public
Extremities	500	Worker - The extremities include the arm or leg below the elbow or knee
Extremities	50	Public - The extremities include the arm or leg below the elbow or knee
Skin	500	Worker
Skin	50	Public

Methods of Controlling Radiation Dose



Time

The radiation dose is directly proportional to the time spent in the radiation. Therefore, a person should not stay near a source of radiation any longer than necessary. If a survey meter reads 4 mR/h at a particular location, a total dose of 4mr will be received if a person remains at that location for one hour. In a two hour span of time, a dose of 8 mR would be received. The following equation can be used to make a simple calculation to determine the dose that will be or has been received in a radiation area.

Dose = Dose Rate x Time.

Distance

Increasing distance from the source of radiation will reduce the amount of radiation received. As radiation travels from the source, it spreads out becoming less intense. This is analogous to standing near a fire. The closer a person stands to the fire, the more intense the heat feels from the fire. This phenomenon can be expressed by an equation known as the inverse square law, which states that as the radiation travels out from the source, the dosage decreases inversely with the square of the distance.

Inverse Square Law: $I_1 / I_2 = D2^2 / D1^2$

Notice that increasing the distance is more effective at reducing the dose than reducing the time. If you double your distance to the source, a person can stay four times as long before receiving the same dose.

Shielding

The third way to reduce exposure to radiation is to place something between the individual and the source of radiation. In general, the denser the material the more shielding it will provide. Some of the most effective shielding are heavy metals such as depleted uranium and tungsten. These are very effective in shielding radiation because their tightly packed atoms make it hard for radiation to move through the material without interacting with the atoms. Lead and concrete are the most commonly used radiation shielding materials primarily because they are easy to work with and are readily available materials. Concrete is commonly used in the construction of radiation vaults. Some vaults will also be lined with lead sheeting to help reduce the radiation to acceptable levels on the outside.

Radiation Safety Practices

Area Postings

The following sign or local equivalent shall be posted at the entrance or area where X-ray producing equipment will be used

.

Instrument Labeling

X-ray Tube Devices

"CAUTION: X-RAYS PRODUCED WHEN ENERGIZED. OPERATION BY QUALIFIED PERSONNEL ONLY" or similar wording such as **"CAUTION: X-RADIATION ATTENTION: RAYONNEMENTS X"** is posted next to the X-ray tube lamp and any device which energizes the X-ray tube.

In addition, an easily visible, fail-safe warning light labeled with the words “X-RAYS ON” or similar wording is located on the X-ray head. This warning light will be illuminated only when the tube is energized.

Prevention of Accidental Exposure

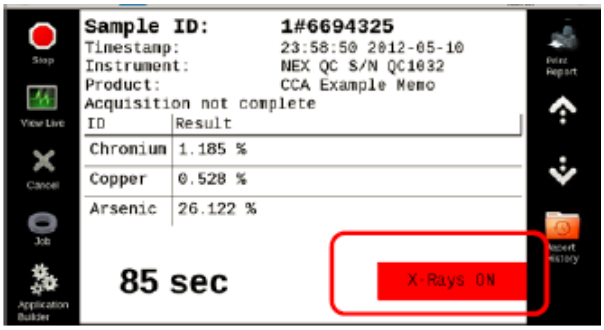
To minimize the risk of accidental exposure, all individuals working with X-ray generating devices must be trained to work with such devices and follow the basic guidelines below:

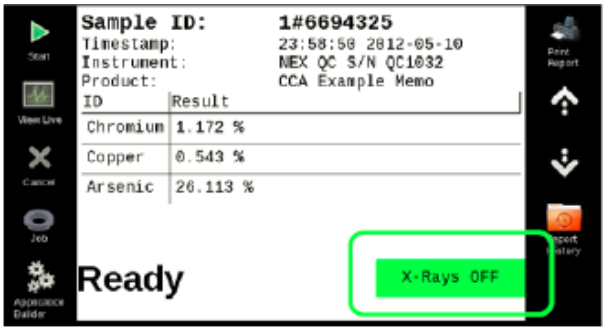
X-ray devices – Survey all devices upon power up when work is performed on the device which may impact the shielding or interlock circuits. Prior to returning the device to the customer or putting back in service, a survey at full power and stand by settings (if applicable) must be performed and recorded, and all safety interlocks must be tested.

EDX1000

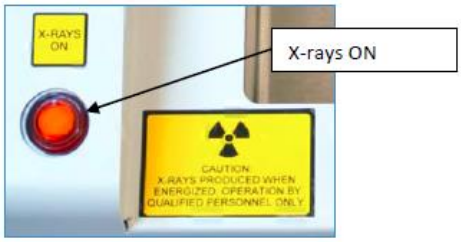
The power indicator is the WVGA Display mounted on the top of the instrument. The display will be active shortly after power is applied.

The X-rays enabled/disabled indicator is located on the lower right corner of the main display. When X-rays are enabled, the words “X-rays ON” are displayed with a red back drop. When X-rays are disabled, the words “X-rays OFF” are displayed with a green back drop.





The X-ray indicator lamp is located on the front of the instrument



DO NOT DISASSEMBLE THE UNIT
*Service is to be performed by authorized personnel only.

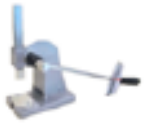
DO NOT DEFEAT SAFETY INTERLOK CIRCUITS
Defeating safety interlock circuits can lead to accidental exposure.

DAMAGED DEVICES
In the system is damaged, remove power from the device and contact your local distributor or representative immediately. The system poses no radiation risk when power is removed. System can be powered down by pressing the main power button located on the back of the analyzer or by removing the power cord to the system.

EXPOSURE RATES
Exposure rates at 5 cm from the surface and at the surface of all accessible areas are less than 1 micro sieverts per hour. Survey performed with front panel open.

9 Consumables, Accessories and Options

9.1 Consumables and Accessories

DESCRIPTION	LIST PRICE	IMAGE	NOTES
CUP, SAMPLE, 3 PIECE, BAG OF 100	Request Quote		32 mm sample cups
PAPER ROLL, PRINTER 3" WIDE	Request Quote		NEX QC Printer Paper
FILM, MYLAR, 6UM, 3"X300' ROLL	Request Quote		X-Ray Film
FILM, POLYPROPYLENE, 6UM, 3"X300'	Request Quote		X-Ray Film
FILM, PROLENE, 4 UM, 3"X300' ROLL	Request Quote		X-Ray Film
FILM, PROLENE, 4UM, 2.5" DIA, 500PC	Request Quote		X-Ray Film
FILM, ETNOM, 3UM 3" DIA, 100 PC	Request Quote		X-Ray Film
ASSY, SAMPLE PRESS	Request Quote		Sample Press w/ torque wrench
HOLDER, SAMPLE CUP, 32.5MM	Request Quote		32 mm sample tray cup holder
HOLDER, SAMPLE CUP, 41MM	Request Quote		Replacement 40 mm sample tray cup holder.

SPACER, CALIBRATION STANDARD, 40mm	Request Quote		Replacement Calibration Standard Spacer
RING, SAMPLE, FLAT, NEX-QC	Request Quote		ReplacementFlat Sample Window
RING, SAMPLE, 32MM, NEX-QC	Request Quote		Replacement 32 mm single sample cup holder
RING, SAMPLE 40 MM, NEX-QC	Request Quote		Replacement 40 mm single sample cup holder
O-RING, 1.375 ID x .70 W.2-028	Request Quote		Window Film O-Ring
TRAY ASSEMBLY,SAMPLE 6, POSITION	Request Quote		Replacement 6 postion sample tray
TRAY ASSEMBLY,SAMPLE 5, POSITION	Request Quote		Replacement 5 position Sample Tray
REGULATOR, HE, 2-STAGE W/FITTING	Request Quote		He Purge Option
FITTING, 1/4"M/6MM ONE TOUCH	Request Quote		He Purge Option
TUBING, WHITE, 6MM OD, 4MM ID	Request Quote		He Purge Option
TRANSORT CASE WITH FOAM FOR NEX-QC	Request Quote		Instrument Case
OPTION, SAMPLE SPINNER ASSY	Request Quote		Sample Spinner Accessory

9.2 Sample Spinner Accessory Installation

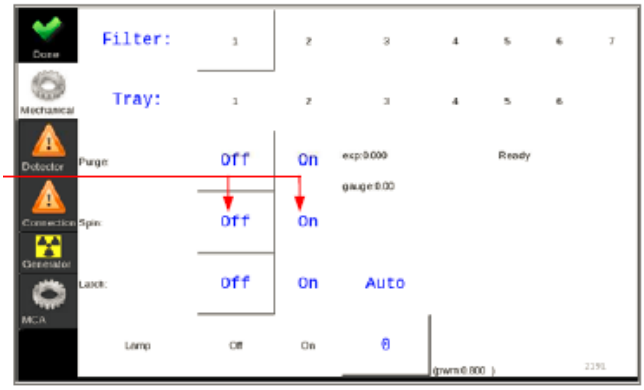
If attaching the sample spinner accessory to an EDX1000 with the optional sample tray, first remove the sample tray and cups holders. In the software "Utilities" screen select the "No Tray option icon". To return to the sample tray remove the sample spinner, re-install the window film, window and re-attach the sample tray, cups and finally, reselect the "sample tray option icon" on the Utilities screen.

1. While attaching the sample spinner accessory, use extreme caution to avoid damaging the beryllium detector window. Remove the existing sample window and film. Add new film and position the sample spinner (see Figure B) then carefully press down on the sides to snap the spinner to the aperture ring underneath.

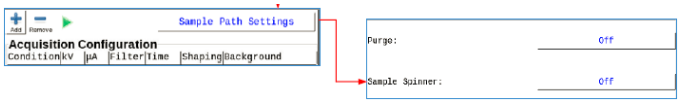
Figure B.

2. Plug in the sample spinner motor cable into the female stereo connector jack found on the right side of the sample chamber.

3. Test the sample spinner; go to the "Utilities", "Hardware Monitor" screen. Select the "Mechanical" tab then turn the spinner Off / On.



4. To use the sample spinner for a specific application. Select the application builder screen and under "Sample Path Settings" turn "Spinner" option on or off.



9.3 Sample Tray Option

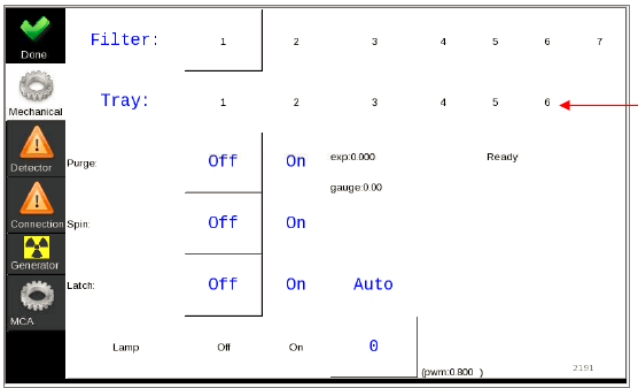
There are two types of sample trays available for the NEX QC's factory equipped with the sample tray option. These include a 6 position sample tray for use with 32mm Sample Cups and a 5 position sample tray for use with 40mm sample cups.

Installation the sample tray requires correctly aligning the drive index pin with the index pin slot found on the bottom of the sample tray. Once in place tighten the sample tray mounting knob to secure the tray, CAUTION - Do not over-tighten knob.

Bottom view of Sample Tray with Index Pin Slot

Sample Tray Drive & Index Pin

Test the sample changer function; go to the "Utilities", "Hardware Monitor" software screen. Select the "Mechanical" tab then visually confirm the select sample tray positioning over the sample aperture.



9.4 Helium Purge Option

X-rays that are emitted from light elements have long wavelengths which are easily absorbed in regular room air. The NEX QC helium flush option helps to minimize this absorption thereby significantly improving detection sensitivity of light elements at lower concentration levels. The Helium flush mechanism option flushes the area of the optics assembly underneath the sample window film with helium gas which does not absorb X-rays as much as room air.

Helium purity: 99.95%

The EDX1000 typically takes about 1 minute to purge the optics assembly with helium and about 8 minutes to recover back to room air. However the EDX1000 will also track and adjust the actual flush time based on the time of the most recent analysis.

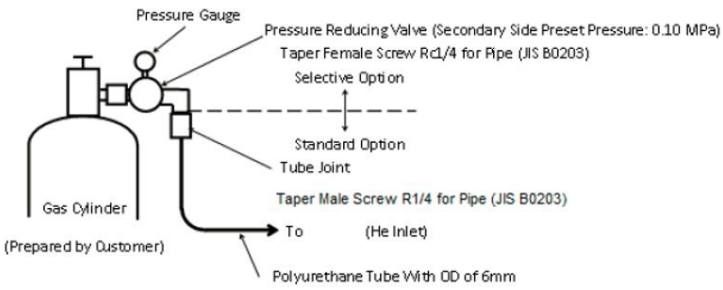
Helium Purge Setup

To setup the Helium Purge option the following articles are required.

- A user provided helium gas cylinder prepared by the user.
- A user provided pressure reducing valve with pressure gauge to be connected to the gas cylinder. (Sold Seperately)

If the EDX1000 was purchased with the helium purge option, it will includes 3 meters of OD: 6mm, ID: 4mm polyurethane tubing for the helium piping and a one-touch 6mm piping connection joint with connection screw: Thread R 1/4

Helium Gas System Diagram



1. Attach the pressure reducing regulator with pressure gauge to the gas cylinder, and then connect the supplied helium piping joint (R 1/4) to the pressure reducing valve connection screw.

2. Connect the supplied polyurethane tube with an outside diameter of 6mm to the one-touch joint. (Insert the end of the tube into the joint. When the tube is not disconnected even when it is pulled slightly, the connection can be judged to be normal. The tube can be disconnected easily by

pulling it with the release bush at the end of the joint pushed.)

3. Connect the tube from the helium regulator to the helium inlet in the lower left corner on the back of the EDX1000.

*Rear Panel Optional Helium Purge Inlet Connector with attached Polyurethane tube

4. Use the helium tank pressure regulator valve to set the tank pressure to approximately 10 psi (0.10MPa) or to achieve a sample chamber purge rate of 200 ml/min (0.2 liters/min). *Do not exceed 15 psi at the tank regulator output or at the helium inlet to the instrument.

The flow rate may change depending on the residual pressure in the helium gas cylinder, and measurement precision may be affected. When the residual pressure is low (0.5 MPa (~72 psi) or less, replace the helium gas cylinder soon.

The sample window must be inspected daily to insure it is not torn, slack or dirty. If the sample window film is torn or slack, the X-ray path may not be filled with helium gas correctly. This will result in lowered X-ray intensities affecting the analysis results.

Replacing the Helium Tank

When the helium gas cylinder pressure gauge reading is down to 0.5 MPa (~72 psi), it's time to replace the cylinder with a new one.

Procedure for Replacing the Helium Gas Cylinder

WARNING - A jet of gas from the cylinder is dangerous. Never direct the outlet of the cylinder toward a human body, especially a face. Also do not put your face near the pressure gauge.

1. Close fully the main valve of the cylinder.

2. Disconnect the polyurethane tube connected to the pressure reducing valve.

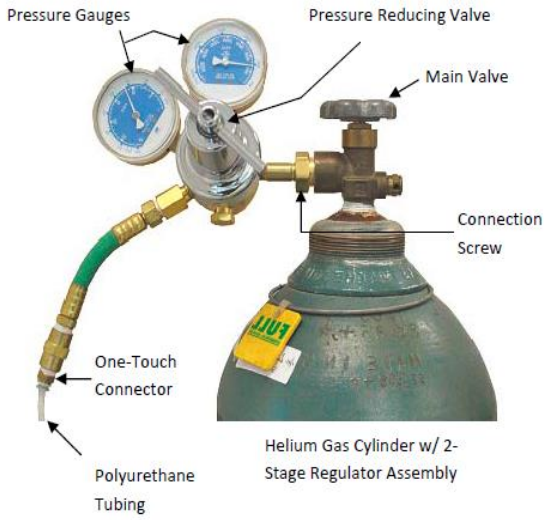
3. Turn the screw connecting the cylinder and the pressure gauge, and remove the pressure reducing valve with the pressure gauge from the cylinder.

4. Remove the cap of a new cylinder, and connect securely the pressure gauge that has been removed.

5. A cap may have been attached to the connection screw to prevent foreign matter such as dust from entering. In this case, remove the cap.

6. Attach the polyurethane tube.

Helium Gas Cylinder with 2-Stage Regulator



Helium Gas Cylinder w/ 2-Stage Regulator Assembly

Helium Tank w/ cap

Notes