PANalytical

EPSILON 3

Quick Start Guide

The Analytical X-ray Company

PANalytical

Epsilon 3

Quick Start Guide

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This is the original publication of Edition 2 of this Quick Start Guide, to be used with the Epsilon 3 software version 1 or higher.

ACKNOWLEDGMENTS

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Chapter 1

Introduction

1.1 GENERAL INFORMATION

This Quick Start Guide is intended to be used by all people that operate the Epsilon 3 spectrometer together with Epsilon 3 software version 1 (or higher). It provides full details of how to operate the spectrometer system.

This Quick Start Guide is not intended to provide you with details of maintenance procedures that should be carried out by trained and qualified engineers. Nor is it intended to act as a User's Guide for MS Windows. You should refer to the documentation and on-line tutorials provided with these products.

1.2 INTENDED USE

The Epsilon 3 spectrometer and the Epsilon 3 application software are designed to perform routine X-ray measurements over a long period of time. For example, you can use it to calibrate the instrument to measure the amount (or ratio to another element) of Ni in oil and then use the system to carry out that task over a number of months.

It is expected that the system will mostly be used by laboratory or factory personnel. One person will set up the system (for example calibrate the system for a particular application using standards) and then password protect the various system parameters etc. This would enable other laboratory personnel to use the system to measure samples but not to change (either by accident or on purpose) the setup or system information.

1.2.1 System

The system facilities (selected via the menu bar or toolbar) are used to create 'common' items that may be used in several applications or by several users (for example system condition sets, system moduli and so on). You can then use one or more of these system items (for example a condition set) when setting up an application in order to prevent having to enter the information over and over again. For example: if you know that the system is going to be used regularly for measuring oil samples, you can create a system condition set called "oil". Then, when you create an application for measuring oil you could just enter "oil" as the condition set name without having to remake the entire condition set.

1.3 CONVENTIONS USED

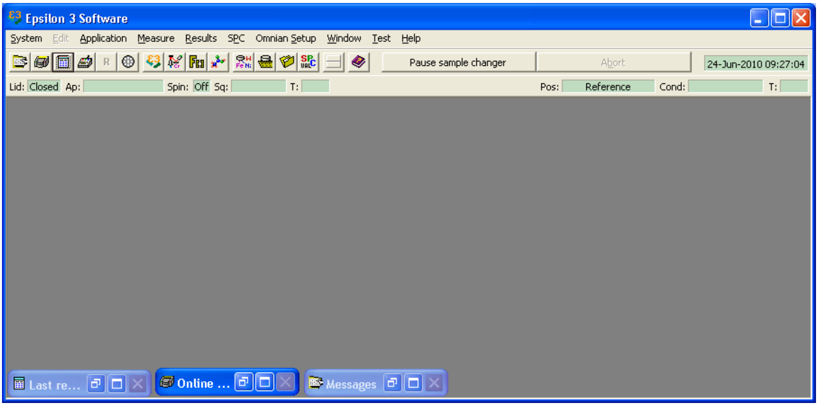
- An instruction is preceded by a bullet "●".
- Menu items, keys and pushbuttons on a window are shown in bold text, for example: **File** > **Open, Enter, Ctrl, Apply**, **Cancel**.
- Grids are table-like areas on windows used to input and display large amounts of information.
- There may be differences between the example screens given in this Quick Start Guide and what you see on your screen. In all cases, where there is a difference, follow what you see on your screen.

Chapter 2

Software User Interface

2.1 EPSILON 3 SOFTWARE MAIN WINDOW

When the Epsilon 3 software is started (see Chapter 3), the **Epsilon 3 Software** main window is opened.



24-Jun-2010 09:27:04

Lid: Closed Ap: Spin: Off Sq: T: Pos: Reference Cond: T:

Figure 2.1 Epsilon 3 Software Main Window

The following elements are displayed in the main window:

At the top of the screen:

- Menu bar
- Toolbar
- Spectrometer status bar

  NOTE: The spectrometer status bar is only displayed if it is enabled and the spectrometer is on-line.

At the bottom of the screen:

- **Last result** window
- **Online status** or **Offline status** window
- **Messages** window
- **Progress** window (only when system is on-line)

2.1.1 Menu Bar

The menu bar is displayed directly under the title bar in the **Epsilon 3 Software** main window. The menu bar displays a list of the menus available in the Epsilon 3 software. Most of the commands and functions of the Epsilon 3 software can be selected from the menus.

Menus or menu items that are not currently available are shown in light grey and cannot be selected.

NOTE: When the hardware is not on-line, the test menu is unavailable (light grey).

Table 2-1 Menus

Menu	Description
System	This menu gives access to the system management windows, **Epsilon 3 Software** main window options (for example: Toolbar displayed?), communications setup and logging, printing, and exiting.
Edit	This menu allows you to copy, paste or print selected text, spectra or graphs to and from the clipboard. With the **Select All** menu item, you can select the whole text or graphics in the active form.
Application	This menu allows you to open, close, display or delete existing applications and to create new applications.
Measure	This menu allows you to open, close or display the **measure** window for an application.
Results	This menu allows you to open, close or display the results window for an application.
SPC	This menu allows you to open, close or display the SPC values windows for an application.

Table 2-1 Menus (Continued)

Menu	Description
Omnian Setup	This menu allows you to set up Omnian sample parameters, calibration, standards and compound lists for measuring unknown samples using the Omnian optional software.
Window	This menu offers the usual Windows options for arranging the windows within the **Epsilon 3 Software** main window. It also offers a list of all open windows allowing you to select and activate the required window.
Test	This menu allows you to manually control and test the spectrometer.
Help	This menu allows you to access the Epsilon 3 On-line Help facility.

2.1.2 Toolbar

The toolbar provides a fast user-friendly way to access some of the commonly used commands. The toolbar is displayed by default when the software is first started, but can be turned off from the **System** menu.

The current PC system date and time are displayed on the right of the toolbar.

NOTE: The role of some of the buttons changes depending on whether function windows for that button are already open in the **application** window.

Table 2-2 Toolbar Buttons

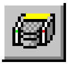
Button	Description
	Displays the **Messages** window.
	Displays the spectrometer **Offline status** window.

Table 2-2 Toolbar Buttons (Continued)

Button	Description
	Displays the **Last result** window.
	Displays the **Manual control** window.
®	Moves the sample changer to the reference position.
	Displays the **Sample changer** window.
ε3	Displays the **System parameters** window. See NOTE.
Cr	Displays the **System condition sets** window. See NOTE.
F()	Displays the **System moduli functions** window. See NOTE.
	Displays the **System compound definitions** window. See NOTE.
W Fe Ni	Opens an application or displays the **Open application** window.
	Opens a measurement or displays the **Measure application** window.
	Opens results or displays the **Open results** window.
SPC VAL	Opens SPC values or displays the **Open SPC values** window.

Table 2-2 Toolbar Buttons (Continued)

Button	**Description**
🟩	Starts an Easy measure, for applications that have an already opened **Measure application** window.
📖	Opens the Epsilon 3 On-line Help.

NOTE: If this window is already open, it is displayed at the front and made active.

2.1.3 Status Bar

Lid: Closed Ap: <Gain> Spin: Off Sq: 1/1 T: 46 23% Pos: Reference position Cond: <Gain> T: 46

Figure 2.2 Status Bar

If the spectrometer is on-line, the status bar is displayed by default when the software is first started. It can be turned off from the **System** menu.

This bar contains a number of information fields describing the status of the spectrometer. These fields cannot be edited.

Table 2-3 Spectrometer Status Bar Fields

Status Bar Field	**Description**
Lid:	Status of the spectrometer lid: “Open” or “Closed”.
Ap: (Application)	Active application during a measurement.
Spin: (Spinner)	Status of the spinner: “On” of “Off”. Only shown when spinner is present.
Sq: (Sequence)	Current sequence number.
T: (Time)	This menu allows you to open, close or display the **results** window for an application.
Pos: (Position)	Position of the sample in the sample changer.

Table 2-3 Spectrometer Status Bar Fields (Continued)

Status Bar Field	Description
Cond: (Condition)	The name of the current condition set.
T: (Time)	Time remaining for the current condition.

2.2 APPLICATION WINDOW

Each **application** window contains a form used for configuring, entering data, carrying out measurements or processing results.

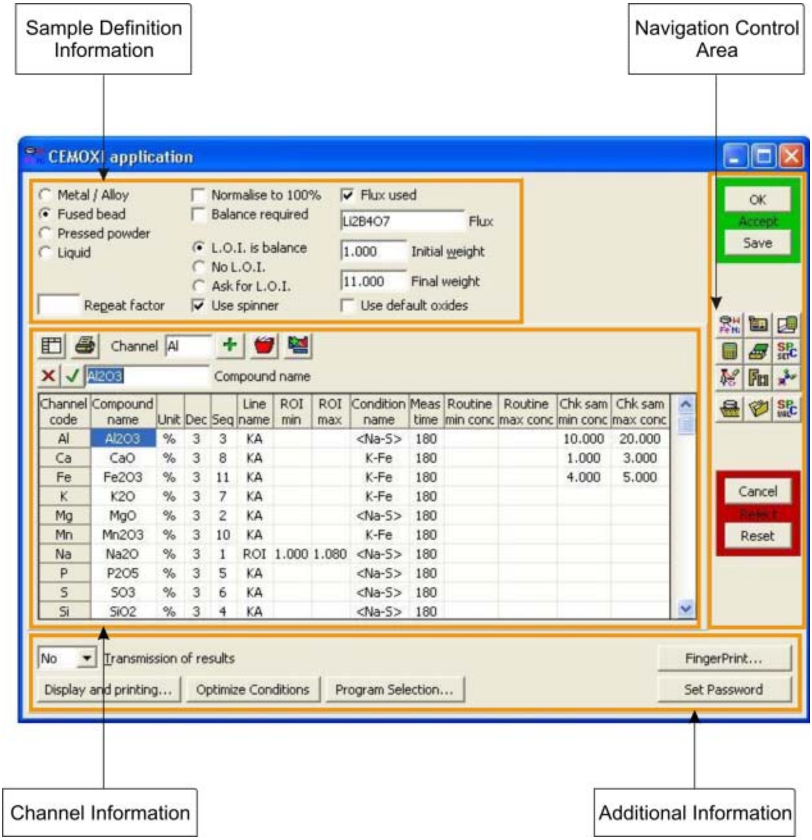
Up to 8 application windows can be opened or minimized, at any time within the **Epsilon 3 Software** main window. A further limit is imposed by the system resources: if resources drop below 10 %, the software will prevent you to open any further windows until you have closed some others.

The windows use a wide variety of controls and fields to display information, control the operation of the software and navigate around the other windows.

The **application** window has four areas:

- Sample definition information
- Channel information
- Navigation/control
- Additional information

The next sections describe the control and navigation buttons of the navigation/ control area and of the channel information area. For information regarding the other items and areas, see Chapter 4.



○ Metal / Alloy
● Fused bead
○ Pressed powder
○ Liquid

☐ Normalise to 100%
☐ Balance required

● L.O.I. is balance
○ No L.O.I.
○ Ask for L.O.I.

Repeat factor
☑ Use spinner

☑ Flux used
Li2B4O7 Flux
1.000 Initial weight
11.000 Final weight
☐ Use default oxides

Channel Al

Al2O3 Compound name

Channel code	Compound name	Unit	Dec	Seq	Line name	ROI min	ROI max	Condition name	Meas time	Routine min conc	Routine max conc	Chk sam min conc	Chk sam max conc
Al	Al2O3	%	3	3	KA			<Na-S>	180			10.000	20.000
Ca	CaO	%	3	8	KA			K-Fe	180			1.000	3.000
Fe	Fe2O3	%	3	11	KA			K-Fe	180			4.000	5.000
K	K2O	%	3	7	KA			K-Fe	180				
Mg	MgO	%	3	2	KA			<Na-S>	180				
Mn	Mn2O3	%	3	10	KA			K-Fe	180				
Na	Na2O	%	3	1	ROI	1.000	1.080	<Na-S>	180				
P	P2O5	%	3	5	KA			<Na-S>	180				
S	SO3	%	3	6	KA			<Na-S>	180				
Si	SiO2	%	3	4	KA			<Na-S>	180				

No Transmission of results

Figure 2.3 Typical Application Window

2.2.1 General Control Buttons

The the navigation/control area contains general control buttons, used to save or cancel any changes made in the window and to close the window.

Table 2-4 General Control Buttons

Button	Description
OK	Saves changed values and closes the window.
Save	Saves changed values but does not close the window.
Cancel	Closes the window without saving.
Reset	Resets changes values to the original value.

2.2.2 Grid Control Buttons

The channel information area contains a number of grid control buttons, normally displayed with the grids.

In addition, control buttons that only provide functions for a particular grid may be displayed. These additional control buttons are described when occurring in the following chapters.

Table 2-5 Grid Control Buttons

Button	Description
▦	Resizes the cells in the grid so that each column displays the longest entry.
🖨	Prints the active window.
✚	Adds a new item. Inserts the new item you have entered in the input field into the grid.

Table 2-5 Grid Control Buttons (Continued)

Button	Description
🗑	Deletes the selected item(s) from the grid.
🔑	Changes the password for this application.
✗	Restores the edited value to the value that was last saved or transferred to the grid.
✓	Saves the edited value to the selected grid position.

2.2.3 Navigation Buttons

The center of the navigation/control area contains of a number of navigation buttons, which are used to open or move to other windows for that application.

The buttons displayed in this area are the same on all the **application** windows.

Table 2-6 Navigation Buttons

Button	Description
W Fe Ni	Opens the **application** window.
🪪	Opens the **ident system** window.
📈	Opens the **recalibration program** window.
🖩	Opens the **calculation program** window.

Table 2-6 Navigation Buttons (Continued)

Button	**Description**
	Opens the **standards** window.
SPC SET	Opens the **SPC setup** window.
Cr	Opens the **condition sets** window.
F()	Opens the **moduli functions** window.
	Opens the **compound definitions** window.
The functions provided by the lower three buttons can still be used (via the toolbar) even if the application is protected by a password.	
	Opens the **measure sample** window.
	Opens the **results** window.
SPC VAL	Opens the **SPC values** window.

2.3 GENERAL USER ACTIONS

2.3.1 Resizing Grid Cells

When information is displayed in a grid, the size of the grid cells can be adjusted either manually or automatically.

To adjust the size of the cells manually:

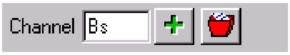
- Use the mouse in the column or row title to drag the selected columns or rows.

To adjust the size of the cells automatically:

- Click on the ▦ button. This automatically resizes the column widths and cell heights to fit the data in the grid.

2.3.2 Adding and Deleting Rows

If a selected row field and add/delete buttons are displayed above the grid, you can add and delete rows from the grid.



Channel Bs

Figure 2.4 Typical Add/Delete Grid Area

The selected row field normally shows the row containing the selected cell. If multiple rows are selected (for example: a complete column) the field is filled with “xxxx”.

To add a new row:

- Click in the relevant text field (in this case the **Channel** field) to insert the text cursor.
- Enter the name of the row to be added.
- Click on the + button or press the **Enter** key to insert the new row.

To delete a row:

- Select the row to be deleted by clicking on the row title. The title is shown in the selected row field.
- Click on the 🗑 button.

Chapter 3

Configuring the Epsilon 3 Software

3.1 INTRODUCTION

Viewing and changing the system parameters and default definitions is carried out in System Mode, via the following windows:

- **System parameters** window
- **System conditions sets** window
- **System moduli functions** window
- **System compound definitions** window

Each of these windows can be accessed by clicking on their respective toolbar buttons or by selecting the item from the **System** menu. If a system manager password has been set and the system is in User Mode, the password must be entered in the window before any of these windows will open.

3.2 CONFIGURING THE SYSTEM PARAMETERS

The **System parameters** window is used to view and allow changing some system parameters. It displays the following system parameters:

- X-ray tube parameters
- Filter parameters
- Conversion factors
- He system
- Transmission of results

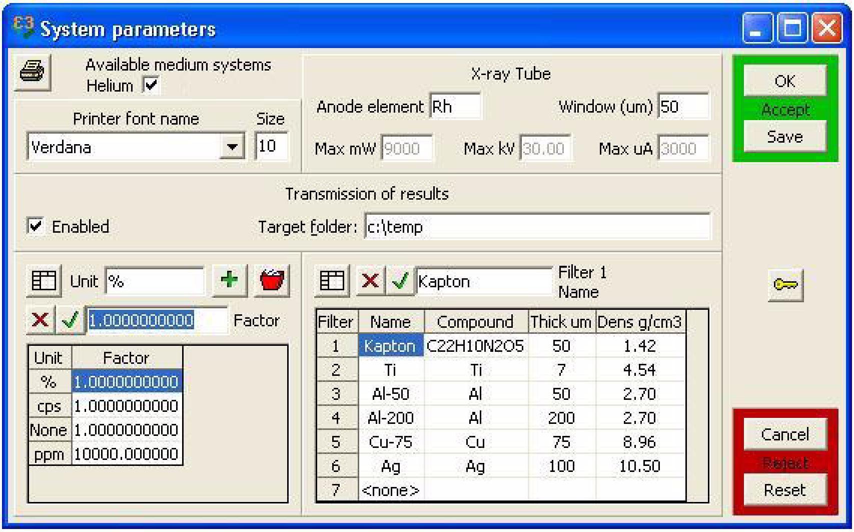
The **System parameters** window also allows you to set and change the system manager password and set the printer font and size.

To open the System parameters window:

- Click on the button or select **System** > **System parameters**.

  If a system manager's password has been set, a window is displayed requesting the system manager's password.

- Type the password and click on the **OK** button or press **Enter** to open the **System parameters** window.



Available medium systems
Helium ☑

Printer font name: Verdana
Size: 10

X-ray Tube
Anode element: Rh
Window (um): 50
Max mW: 9000
Max kV: 30.00
Max uA: 3000

Transmission of results
☑ Enabled
Target folder: c:\temp

Unit: %
Factor: 1.0000000000

Unit	Factor
%	1.0000000000
cps	1.0000000000
None	1.0000000000
ppm	10000.000000

Filter 1 Name: Kapton

Filter	Name	Compound	Thick um	Dens g/cm3
1	Kapton	C22H10N2O5	50	1.42
2	Ti	Ti	7	4.54
3	Al-50	Al	50	2.70
4	Al-200	Al	200	2.70
5	Cu-75	Cu	75	8.96
6	Ag	Ag	100	10.50
7	<none>			

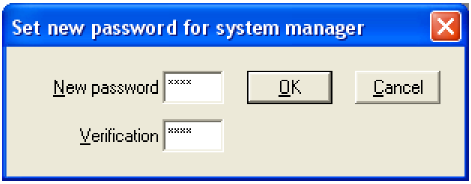
Figure 3.1 System Parameters Window

3.2.1 Setting or Changing the System Manager Password

If no system password is set, any password set in the application is disabled. Also, if the system password is deleted (no new password is defined), the application passwords are disabled. In both of these instances, any application password set is stored by the software and is automatically enabled when a new system password is set.

To set the system managers password

- In the **System parameters** window, click on the 🔑 button. The **Set new password for system manager** window is displayed.


Set new password for system manager

New password: ****

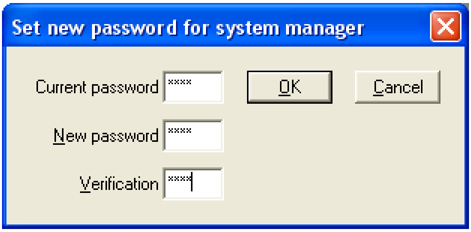
Verification: ****

Figure 3.2 Set New Password for System Manager Window

- In the **New password** field, enter the password. A “*” is displayed for each character as it is typed.
  NOTE: The passwords are NOT case sensitive.
- Enter the password again in the **Verification** field.
- Click on the **OK** button or press **Enter**.

To change the system managers password

- In the **System parameters** window, click on the 🔑 button. The **Set new password for system manager** window is displayed.


Set new password for system manager

Current password: ****

New password: ****

Verification: ****

Figure 3.3 Set New Password for System Manager Window

- In the **Current password** field, enter the existing password.
- In the **New password** field, enter the new password.
- Enter the new password again in the **Verification** field.
- Click on the **OK** button or press **Enter**.

3.2.2 Viewing the X-ray Tube Parameters

In on-line mode, the X-ray tube parameters are displayed in the **System parameters** window as light grey fields and cannot be changed. Some of the X-ray tube parameters are read from the spectrometer firmware.

NOTE: In off-line mode, the X-ray tube parameters can be changed. However, the correct settings are restored when you go on-line to the spectrometer.

Table 3-1 X-ray Tube Parameters

Parameter	Description
Anode element	X-ray tube anode material.
Window (um)	The thickness of the beryllium window of the X-ray tube.
Max mW	The maximum allowed power that can be applied to the X-ray tube (9000 W/15000 W).
Max kV	The maximum allowed voltage that can be applied to the X-ray tube (30.00 kV/50.00 kV).
Max uA	The maximum allowed current that can be applied to the X-ray tube (1000 µA/3000 µA).
Window (um)	The thickness of the X-ray tube’s beryllium window.

3.2.3 Enabling the Helium System

- Check the **Helium** check box to enable helium to be used in the applications.

3.2.4 Setting the Printer Parameters

The fields **Printer font name** and **Size** in the **System parameters** window allow you to set the printer font type and size (in points).

We do not recommend using font sizes below 8 points, as many people find this difficult to read.

The page setup for the default printer (paper size, landscape or portrait, etc.) is defined in the printer setup, which can be accessed from the **System** menu.

Graphical windows are always printed in landscape and fitted to the maximum printable size on the page.

3.2.5 Viewing the Filter Parameters

Table 3-2 shows the filters fitted to the system. For more information about the use of filters and their effect, see Chapter 13

Table 3-2 Filters

Filter number	**All X-ray Tubes**	**Thickness (µm)**
1	Cu-500	500
2	Ti	7
3	Al-50	50
4	Al-200	200
5	Cu-300	300
6	Ag	100
7	none	-

3.2.6 Enabling Transmission of Results

It is possible to enable the transmission of the measurement results to a folder on your hard drive. The folder must be an existing folder on your hard drive. The measurement results can be used by other applications for further processing.

The transmitted files have the following file name format:

XYYYYMMDDHHMMSS.QAN

where: X is fixed
YYYYMMDDHHMMSS is the year (YYYY), month (MM), day (DD), hour (HH), minutes (MM) and seconds (SS).

Transmission can be carried out both on-line or off-line.

- In the **System parameters** window, check the **Enabled** check box to enable transmission for an application.
- The measurement results of the specified application will be transmitted on-line depending on the application setting (**Yes**, **No** or **Ask**).
- The **Transmit** check box on the **Measure sample** window is set according the **System parameters** window setting.
- On the **results** window, you can transmit the highlighted samples off-line using the button.

Only the data from unknown samples, concentration and/or intensities are transmitted. Spectra and standard data are not transmitted.

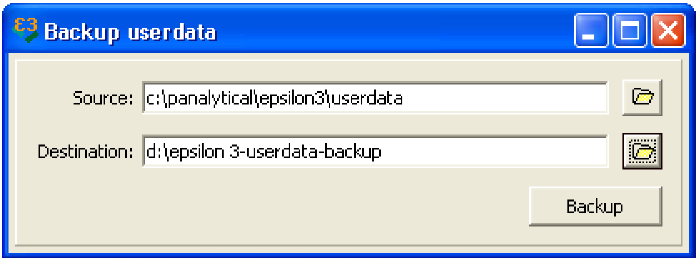
3.2.7 Setting the General Parameters

This section describes the following two general parameters which are useful for operating the Epsilon 3 software:

- Backup user data
- Select language

To backup user data:
We advise you to make several copies of your data to protect it from accidental loss or hardware and media failures. Keep at least one copy off-site in a secure location at all times.

- Select **System** > **Backup userdata** to open the **Backup userdata** window.



Source: c:\panalytical\epsilon3\userdata

Destination: d:\epsilon 3-userdata-backup

Figure 3.4 Backup Userdata Window

- Backup all user data that is new or updated in the source folder **\PANalytical\Epsilon 3\Userdata**. Select an existing destination folder and click on the **Backup** button to start the backup.

With each backup a new sub-folder is created in the destination folder with the following format:

YYYYMMDDHHMMSS

where: the format of the year (YYYY), month (MM), day (DD), hour (HH), minutes (MM) and seconds (SS) is used.

All files and sub-folders of the source folder are copied into the new destination sub-folder with the actual format.

The source folder and destination folder only have to be specified initially and are shown each time when the **Backup userdata** window is opened.

To select a language:

- The Epsilon 3 software is default delivered in the English language.
- To select a local language, select **System** > **Select language**. The **Select language** window is displayed.

Select language: Chinese

Figure 3.5 Epsilon 3 - Select Language Window

The following local languages are available:

- Chinese
- English
- French
- German
- Italian
- Japanese
- Polish
- Portuguese
- Russian
- Spanish

- Select the required language and click on **OK**. The selected language will be changed after restarting the Epsilon 3 software.

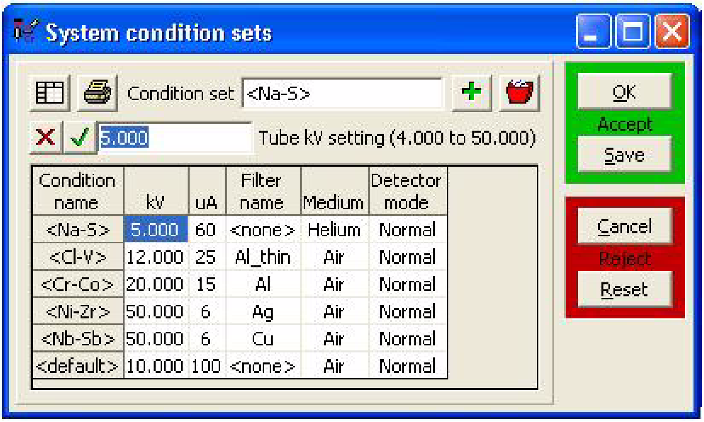
3.3 SETTING THE SYSTEM CONDITIONS

The **System condition sets** window is used to create condition sets that can be used in all applications.

The software automatically selects predefined conditions when selecting elements to be measured.

To open the System condition sets window:

- Click on the toolbar button or select **System > System Condition sets**. If a system password has been set, a window requesting the system manager's password is displayed (unless another system window is already open, in which case you will not be asked for the password again).
- Type the password and click on the **OK** button or press **Enter** to display the **System condition sets** window.



Condition set <Na-S>

5.000 Tube kV setting (4.000 to 50.000)

Condition name	kV	uA	Filter name	Medium	Detector mode
<Na-S>	5.000	60	<none>	Helium	Normal
<Cl-V>	12.000	25	Al_thin	Air	Normal
<Cr-Co>	20.000	15	Al	Air	Normal
<Ni-Zr>	50.000	6	Ag	Air	Normal
<Nb-Sb>	50.000	6	Cu	Air	Normal
<default>	10.000	100	<none>	Air	Normal

Figure 3.6 System Condition Sets Window

To add other measurement conditions for specific applications:

- In the **Condition set** field, give the condition set a name.
- Enter the required **kV**, **uA** and **Filter name**.
- If the helium system has been enabled, you must also choose between **Air** and **Helium** as the measuring medium.

3.4 DEFINING SYSTEM MODULI

System moduli are “standard” factors (calculation functions) that are of relevance to the type of measurement that your system will be used for. They are included into an application as a channel (with an asterisk as a last character). You can set the moduli for ratios, sums or a combination of both. The system then checks the measurement results and reports on these channels.

For example:

If you wanted to know what the ratio of Al to Si is in your result, you press F(x), enter a channel name and then the moduli **Al/Si**.

As previously stated, the moduli are used to define calculation functions in the results of applications.

The moduli defined at system level can be used in all applications.

NOTE: Additional moduli can be defined in an application, but they are only available to that application.

For example:

To find the total concentration of halogens in a sample, you would define a moduli in which the values of all the halogen channels (Cl, Br, etc.) were added together.

Moduli:	Ha*
Expression:	Cl + Br

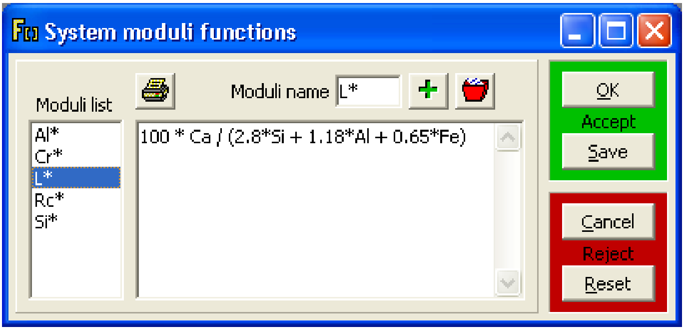
This moduli could then be entered as a channel in an application.

NOTE: Moduli names are one or two characters long with an asterisk (*) as the last character.

If the system is in User Mode and a system manager's password has been set, a dialog box requesting the system manager's password is displayed.

To open the System moduli functions window:

- Click on the F() toolbar button, or select the **System** > **System moduli**.



System moduli functions

Moduli list

Al*
Cr*
L*
Rc*
Si*

Moduli name: L*

100 * Ca / (2.8*Si + 1.18*Al + 0.65*Fe)

Figure 3.7 System Moduli Functions Window

To define a moduli:

- In the **Moduli name** field, enter the name for the moduli.
- In the definition field, enter the mathematical definition (valid expressions are: +, -, *, / and ^ (^ = “to the power of”)).
- Click on the ➕ button. The name of the moduli appears in the **Moduli list** field, with the moduli definition directly opposite in the definition field.

To edit a moduli definition:

- Enter the name of the required moduli in the **Moduli name** field or click on the required moduli in the **Moduli list** field.
- In the definition field, edit the moduli definition as required. The new value is automatically inserted into the grid.

To delete a moduli definition:

- Enter the name of the required moduli in the **Moduli name** field or click on the required moduli in the **Moduli list** field.
- Click on the 🗑 button or press the **Delete** key.

3.5 DEFINING SYSTEM COMPOUNDS

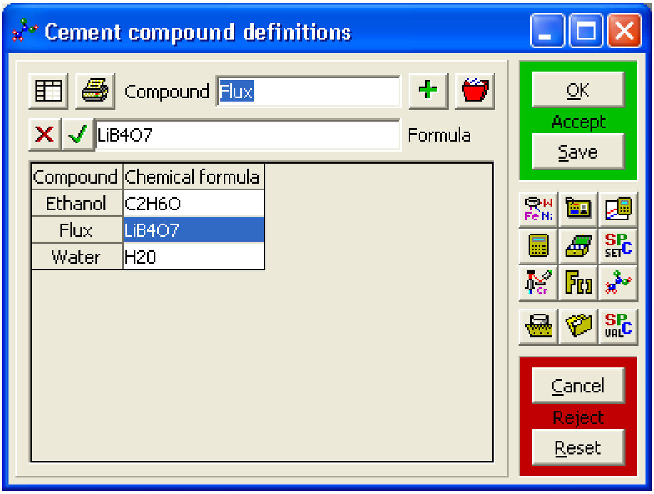
This function allows you to prepare a list of compound definitions that are available for all applications and for manual spectrometer operation. You must enter a name and the chemical formula for each compound. The defined compound name and formulas are displayed in the grid in alphabetical order.

The compound name defined can be used as a parameter in applications and can also be set as the name reported after completion of an analysis.

If a system password has been set, a window requesting the system manager’s password is displayed.

To open the System compound definitions window:

- Click on the toolbar button, or select the **System > System compound definitions**.



Cement compound definitions

Compound: Flux

Formula: LiB4O7

Compound	Chemical formula
Ethanol	C2H6O
Flux	LiB4O7
Water	H20

Figure 3.8 Compound Definitions Window

To define a new compound:

- In the **Compound** field, enter the name of the compound.

  NOTE: The input is case sensitive. Use the correct abbreviations for the chemical elements (for example aluminum = Al).

- In the **Chemical formula** field, enter the chemical formula of the compound.

- Click on the + button. The name of the compound and the chemical formula are automatically inserted into the grid.

To edit a compound definition:

- Enter the name of the required compound in the **Compound** field or click anywhere in the definition field. The compound name and formula are displayed in the corresponding fields.
- Edit the compound definition as required. The new value is automatically inserted into the grid.

To delete a moduli definition:

- Enter the name of the required compound in the **Compound** field or click anywhere in the definition field. The compound name and formula are displayed in the corresponding fields.
- Click on the 🗑 button or press the **Delete** key.

Chapter 4

Applications

4.1 INTRODUCTION

An application contains all the information necessary to perform a measurement. In normal use applications are set up by a specialist and the measurements are done by the operator. The application concept meets the requirements for pre-configured analysis procedures, allowing you to define a unique set of specifications for each application.

Every application can make use of the system configuration sets, moduli and compound definitions. Additional configuration sets, moduli and compounds can be defined for each application. These dedicated definitions can only be used within that application and take priority over any System level definitions with the same name.

Each application can be password protected. This prevents unauthorized users from opening or deleting an application or from closing an application measurement window.

A password protected application can still be copied with a new name. The copied application can then be opened, changed, etc. without the password.

4.2 HANDLING APPLICATIONS

Applications that are already defined can be opened, viewed, closed and deleted from the **Application** menu. An application can also be opened or viewed via a toolbar button. The role of this toolbar button varies, depending on whether other applications are already open.

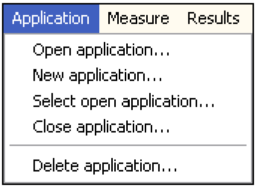


Figure 4.1 Application Menu

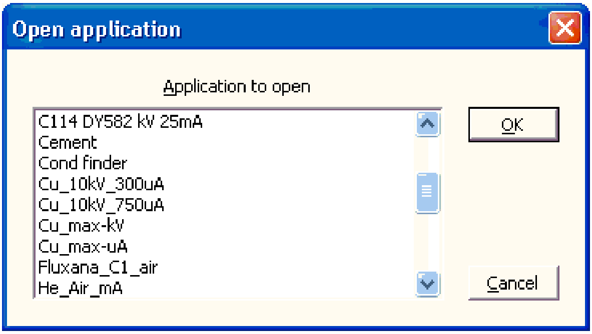
If no applications are currently open, the items **Select open application** and **Close application** are not available (greyed-out).

Applications can be password protected. If a password has been set (and the system password is set), the application password must be entered before the application window can be opened. You can copy the application, but cannot alter the data in the original without the password.

4.2.1 Opening an Application

To open an application from the menu bar:

- Select **Application** > **Open application**. The **Open application** window is displayed.



C114 DY582 kV 25mA
Cement
Cond finder
Cu_10kV_300uA
Cu_10kV_750uA
Cu_max-kV
Cu_max-uA
Fluxana_C1_air
He_Air_mA

Figure 4.2 Open Application Window

- Select the required application and click on the **OK** button or press **Enter**. If no password was set, the selected **application** window is displayed.
- If a password was set, the **Enter password for application** window is displayed.
  - Enter the password. A “*” is displayed for each character typed.
  - Click on the **OK** button or press **Enter**.
  - The selected **application** window is displayed.

To open an application from the toolbar:

- Click on the button. If no applications are currently open, the **Open application** window is displayed. Otherwise, select which application to open, see Section 4.2.2.

4.2.2 Selecting an Open Application

- Select **Application** > **Select open application**.
- If one application is already open, that **application** window is made the active window and displayed on top.
- If more than one applications are already open, the **Select open application** window is displayed.

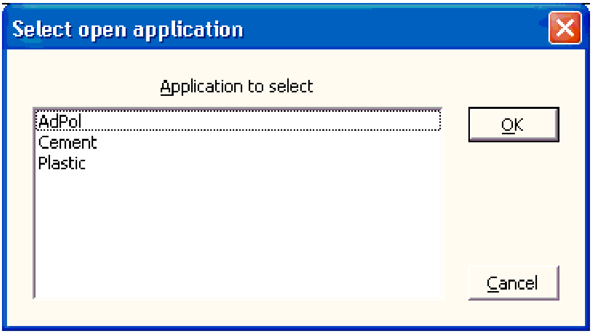


Figure 4.3 Select Open Application Window

- Select the required application and click on the **OK** button or press **Enter**. If no password was set, the selected **application** window is displayed.
- If a password was set, the **Enter password for application** window is displayed.
    - Type the password into the text input field. A “*” is displayed for each character typed.
    - Click on the **OK** button or press **Enter**.
    - The selected **application** window is displayed.

4.2.3 Closing an Application

When the **application** window is closed, any other open definition windows for that application are closed automatically, including the following windows:

- **ident system** window
- **moduli functions** window
- **compound definitions** window
- **standards** window
- **calculation program** window
- **SPC setup** window

A prompt is displayed for each window that will be closed with unsaved changes.

To close an application from the application window:

- In the navigation/control area of the **application** window, click on the **OK** button to save all current settings and data and close the window or click on the **Cancel** button to close the window without saving any changes.

To close an application from the menu bar:

- Select **Application > Close application**. The **Close application** window is displayed.

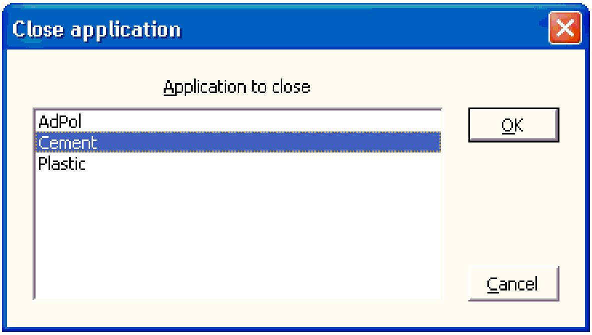


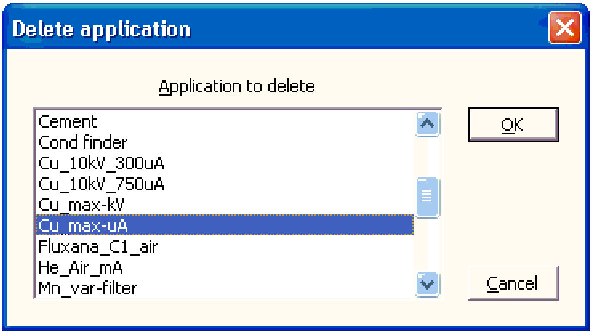
Figure 4.4 Close Application Window

- Select the application to close and click on the **OK** button or press **Enter**.

4.2.4 Deleting an Application

Deleting an application deletes all the data related to that application.

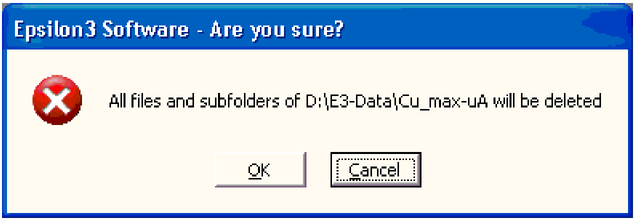
- Select **Application > Delete application**. The **Delete application** window is displayed.



Cement
Cond finder
Cu_10kV_300uA
Cu_10kV_750uA
Cu_max-kV
Cu_max-uA
Fluxana_C1_air
He_Air_mA
Mn_var-filter

Figure 4.5 Delete Application Window

- If a password was set, the **Enter password for application** window is displayed.
    - Enter the password. A "*" is displayed for each character typed.
    - Click on the **OK** button or press **Enter**.
- A warning is displayed for confirmation.



All files and subfolders of D:\E3-Data\Cu_max-uA will be deleted

Figure 4.6 Delete Warning Window

- Click on the **OK** button to delete the application.

4.3 DEFINING A NEW APPLICATION

To assemble a new application, you must define certain parameters and carry out a calibration. Additionally, a number of other parameters and actions can be defined.

This section describes the minimum steps you must perform to define an application.

The following steps MUST be performed in the following order to define an application:

1. Assemble the analytical program by:
   a. Creating a new application (Section 4.3.1).
   b. Defining the sample type (Section 4.3.2).
   c. Selecting channels (elements to be measured) (Section 4.3.3).
   d. Setting the channel parameters (Section 4.3.4).
2. Calibrate the application by:
   a. Entering the composition data of the standard samples (Section 4.4.1).
   b. Finding and calculating the line grouping (Section 4.4.2).
   c. Measuring the standard samples (Section 4.4.3).
   d. Performing deconvolution (Section 4.4.4).
   e. Calculating the regression lines (Section 4.4.5).
   f. Displaying the calibration results (Section 4.4.6).
3. Additionally, other parameters and actions may be needed, such as:
   a. Transmitting results (Section 4.3.5)
   b. Display and printing results (Section 4.3.6)
   c. Optimizing conditions (Section 4.3.7)
   d. Setting an application password (Section 4.3.8)
   e. Identifying material by FingerPrinting (Section 4.3.9)
   f. Automatic sample identification fields (Section 4.5)
   g. Application moduli definitions (Section 4.5)
   h. Application compound definitions (Section 4.5)
   i. Statistical Process Control (SPC)

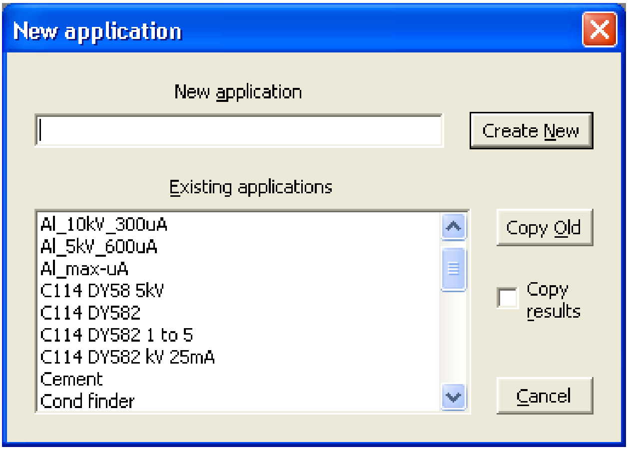
4.3.1 Creating a New Application

You can create an application either entirely from new or by copying and modifying an existing application.

NOTE: No password is required to copy an application; as this does not affect the original application.

To create a new application:

- Select **Application** > **New application**. The **New application** window is displayed.



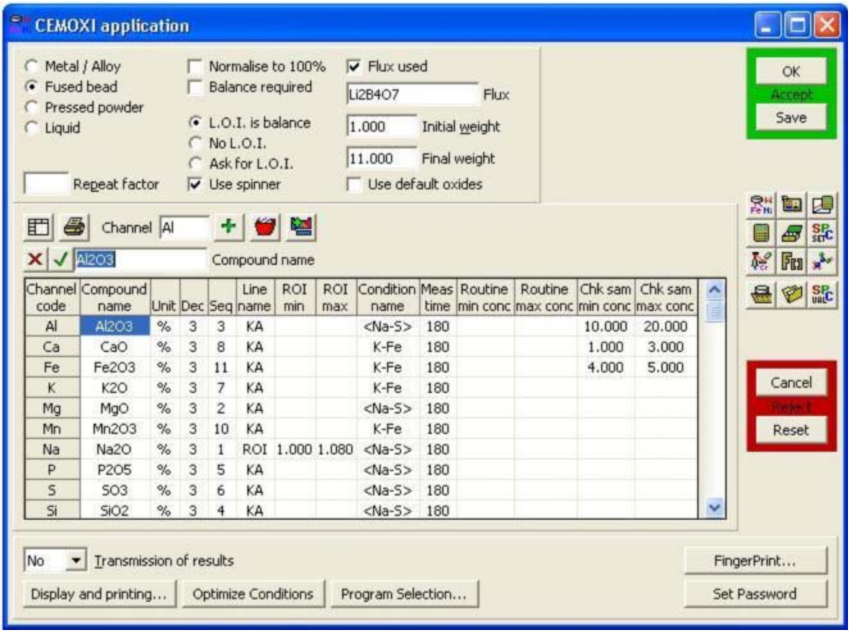
Al_10kV_300uA
Al_5kV_600uA
Al_max-uA
C114 DY58 5kV
C114 DY582
C114 DY582 1 to 5
C114 DY582 kV 25mA
Cement
Cond finder

Figure 4.7 New Application Window

- In the **New application** field, enter a name for the new application.
- Click on the **Create New** button. An **application** window is displayed with the new name.

To create a new application from an existing application:

- In the **New application** field, enter a name for the new application.
- Select the name of the application to be copied from the **Existing applications** list.
- When the results of the existing application are also required for the new application, check the **Copy results** check box.
- Click on the **Copy Old** button.
- An **application** window is displayed with the new name. The window will contain all the settings from the copied application, except the password and the results.



○ Metal / Alloy
● Fused bead
○ Pressed powder
○ Liquid
Repeat factor

☐ Normalise to 100%
☐ Balance required
● L.O.I. is balance
○ No L.O.I.
○ Ask for L.O.I.
☑ Use spinner

☑ Flux used
Li2B4O7 Flux
1.000 Initial weight
11.000 Final weight
☐ Use default oxides

Channel Al
Al2O3 Compound name

Channel code	Compound name	Unit	Dec	Seq	Line name	ROI min	ROI max	Condition name	Meas time	Routine min conc	Routine max conc	Chk sam min conc	Chk sam max conc
Al	Al2O3	%	3	3	KA			<Na-S>	180			10.000	20.000
Ca	CaO	%	3	8	KA			K-Fe	180			1.000	3.000
Fe	Fe2O3	%	3	11	KA			K-Fe	180			4.000	5.000
K	K2O	%	3	7	KA			K-Fe	180				
Mg	MgO	%	3	2	KA			<Na-S>	180				
Mn	Mn2O3	%	3	10	KA			K-Fe	180				
Na	Na2O	%	3	1	ROI	1.000	1.080	<Na-S>	180				
P	P2O5	%	3	5	KA			<Na-S>	180				
S	SO3	%	3	6	KA			<Na-S>	180				
Si	SiO2	%	3	4	KA			<Na-S>	180				

No Transmission of results

Figure 4.8 Application Window

4.3.2 Defining the Sample Type

You must define the sample type and give the required preparation information to enable the software to process the results correctly.

Parameters and information that must be entered depend on the selected sample type. For example, flux information is only needed for a fused bead sample.

After you have defined your sample type and sample preparation data, you must save it before proceeding any further.

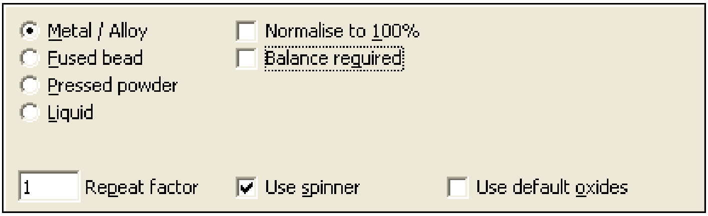
4.3.2.1 Normalise to 100% and Balance

The **Normalise to 100%** and **Balance** check boxes are offered for all sample types.

Normalise to 100%:	When a sample is analyzed, the sum of the calculated concentrations should be 100 %. In practice, the sum is likely to be slightly different than 100 %. Normalization is the recalculation of results in such a way that the sum of the concentrations is equal to 100 %. In general, the accuracy of the results is improved using normalization when the main component comprises over 60 % of the total sample.
Balance:	If **Balance** is checked, the concentration of the balance compound will be calculated as 100 % minus the sum of all the other compounds measured in the application. Using a balance compound is mandatory when the calibration model used is the fundamental parameters model or the Alpha model. Normally, the selected balance channel should be attributed to a compound that constitutes a very large proportion of the sample (e.g. 80 - 90 %).

4.3.2.2 Metal/Alloy Samples

This type of sample covers solid samples prepared without any additives, for example: solid metal or plastic samples.



● Metal / Alloy
○ Fused bead
○ Pressed powder
○ Liquid

☐ Normalise to 100%
☐ Balance required

1 Repeat factor
☑ Use spinner
☐ Use default oxides

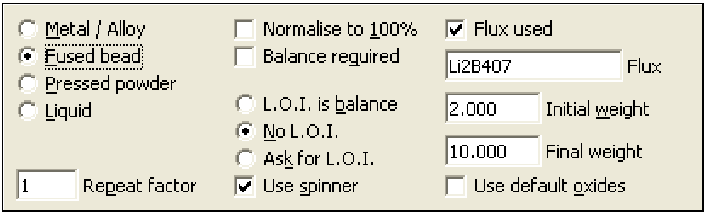
Figure 4.9 Metal/Alloy Sample Type Definition

The only choices are if you want to use normalization or balance selections. If **Balance required** is checked, you must enter a **Balance channel**.

If you do not define a **Balance channel**, the software will request one when you attempt to save the data.

4.3.2.3 Fused Bead Samples

A fused bead is a solid sample made by fusing the sample material with a flux into a ‘glass’ bead.



○ Metal / Alloy
● Fused bead
○ Pressed powder
○ Liquid

☐ Normalise to 100%
☐ Balance required

○ L.O.I. is balance
● No L.O.I.
○ Ask for L.O.I.

☑ Flux used
Li2B407 Flux
2.000 Initial weight
10.000 Final weight

1 Repeat factor
☑ Use spinner
☐ Use default oxides

Figure 4.10 Fused Bead Sample Type Definition

If you select **Fused bead** as the sample type, you must specify what you want the system to do with regard to Loss On Ignition (L.O.I.). If you check the **Flux used** check box, you must also supply the requested information.

Flux

Details of the flux used to make the bead must be defined in this window to enable the software to interpret the results correctly. Typical fluxes used include: lithium tetraborate, lithium metaborate, carbonates and nitrates.

- In the **Flux** field, enter the type of flux.

  NOTE: The name entered for the flux must be defined as a compound in the system or application setup.

- In the **Initial weight** field, enter the weight of sample material used to make the bead.
- In the **Final weight** field, enter the total weight of material (sample + flux) used to make the bead.

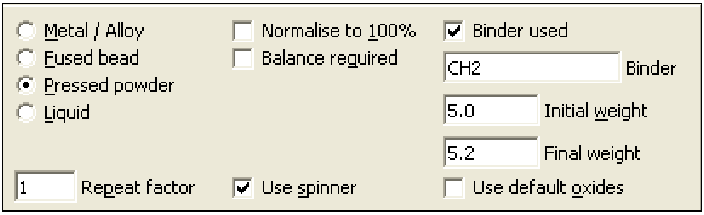
Loss On Ignition (L.O.I.)

The L.O.I. parameter accounts for the part of the sample that is lost when a fused bead is made. For example: crystalline water evaporates and carbonates are converted to oxides. The L.O.I. parameter is used in the calculation of concentrations or it can be determined by the analysis.

L.O.I. is balance:	Any difference between the sum of the calculated concentrations and 100 % is assumed to be the loss from making the bead. This option is not offered if either the check box **Normalise to 100 %** or **Balance** is checked.
No L.O.I.:	The L.O.I. value is ignored.
Ask for L.O.I.:	You are requested to input the L.O.I. value before starting a measurement.

4.3.2.4 Pressed Powder Samples

Powdered samples are normally compressed into pellets for measurement. The pellet usually requires the addition of a binding agent to hold it together.



○ Metal / Alloy
○ Fused bead
● Pressed powder
○ Liquid
1 Repeat factor
☐ Normalise to 100%
☐ Balance required
☑ Use spinner
☑ Binder used
CH2 Binder
5.0 Initial weight
5.2 Final weight
☐ Use default oxides

Figure 4.11 Pressed Powder Sample Type Definition

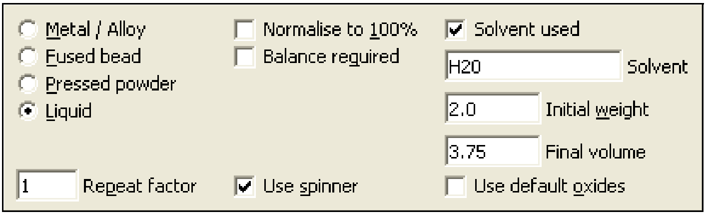
Details of any binding agent used to make the pressed powder pellet must be defined in this window to enable the software to interpret the results correctly. Typical binding agents that are used include: wax, cellulose, boric acid and citric acid.

- In the **Binder** field, enter the type of binding agent.

- In the **Initial weight** field, enter the weight of sample material used to make the pellet.
- In the **Final weight** field, enter the total weight of the material (sample + binder) used to make the pellet.

4.3.2.5 Liquid Samples

Liquid samples require special sample cups. Solvents may be used to dissolve the sample material, forming the liquid. They may also be used to dilute a liquid sample.



○ Metal / Alloy
○ Fused bead
○ Pressed powder
● Liquid
☐ Normalise to 100%
☐ Balance required
☑ Solvent used
H20 Solvent
2.0 Initial weight
3.75 Final volume
1 Repeat factor
☑ Use spinner
☐ Use default oxides

Figure 4.12 Liquid Sample Type Definition

Details of any solvent used must be defined here to enable the software to interpret the results correctly.

- In the **Solvent** field, enter the type of solvent.

  NOTE: If you enter a name (not a formula) for the solvent here, it must be defined as a compound in the system or application setup, where you can enter the formula.

- In the **Initial weight** field, enter the weight of sample material.
- In the **Final volume** field, enter the total volume of the sample (sample + solvent).

4.3.2.6 Use Spinner

The sample spinner will rotate the sample while it is being measured. The sample spinner is designed to reduce effects caused by non-homogeneous samples. The inhomogeneity may be an inherent property of that type of material or sample, or it may even be caused by a specific sample preparation.

An example of specific sample preparation would be the surface cleaning of metals, which is often done using abrasive paper. This leaves surface scratch marks, which tend to be all orientated in the same direction. X-ray fluorescence is very sensitive to surface effects. This means that the results may vary according to the sample orientation. The sample spinner reduces most such effects.

The sample spinner can be switched on or off on the **application** window using the check box **Use spinner**.

4.3.2.7 Use Default Oxides

When the check box **Use default oxides** is checked, the common oxide chemical formula of the element of interest is given in the compound name cell.

4.3.2.8 Repeat Factor

If you require the software to measure the same unknown sample more than once, you must set a repeat factor in the **Repeat factor** field of the **application** window. The effect of the repeat factor on the measuring procedure is described in Chapter 5.

NOTE: The **Repeat factor** field is also available in the **Measure sample** window. If you set it in the **application** window, you cannot change it in the **Measure sample** window. If you do not set it in the **application** window, you can then set the required **Repeat factor** in the **Measure sample** window.

NOTE: If the **Repeat factor** field is left blank or set to “0”, the measurement will continue endlessly until aborted by the user.

4.3.3 Selecting Channels

The channel information area of the **application** window allows you to select which elements to use in the application.

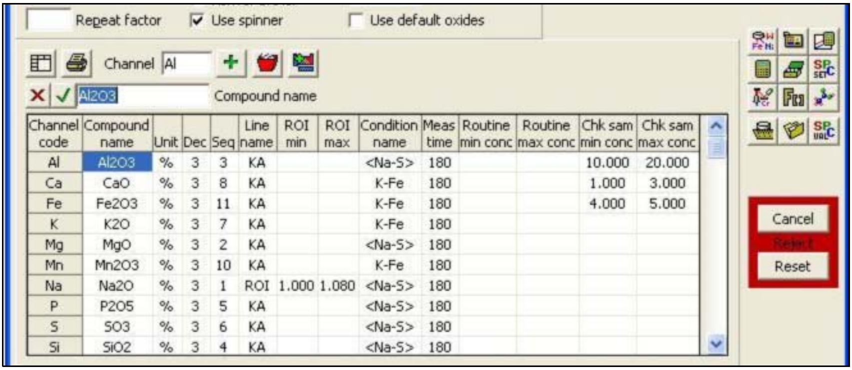
In addition, you can select moduli to be calculated as part of the application. The moduli can be defined at system level, see Section 3.5, or specific moduli can be defined for each application, see Section 4.5.

The moduli channels must have the correct special characters entered at the end of the name. The last character for a moduli channel must always be the character “*”.

In case your elements are present in your sample as an oxide, check the **Use default oxides** check box.

After you have defined your sample type and sample preparation data, you must save it before proceeding any further.

The channel grid of the **application** window below shows the application setup with ten measuring channels.



Repeat factor
☑ Use spinner
☐ Use default oxides

Channel: Al

Al2O3 Compound name

Channel code	Compound name	Unit	Dec	Seq	Line name	ROI min	ROI max	Condition name	Meas time	Routine min conc	Routine max conc	Chk sam min conc	Chk sam max conc
Al	Al2O3	%	3	3	KA			<Na-S>	180			10.000	20.000
Ca	CaO	%	3	8	KA			K-Fe	180			1.000	3.000
Fe	Fe2O3	%	3	11	KA			K-Fe	180			4.000	5.000
K	K2O	%	3	7	KA			K-Fe	180				
Mg	MgO	%	3	2	KA			<Na-S>	180				
Mn	Mn2O3	%	3	10	KA			K-Fe	180				
Na	Na2O	%	3	1	ROI	1.000	1.080	<Na-S>	180				
P	P2O5	%	3	5	KA			<Na-S>	180				
S	SO3	%	3	6	KA			<Na-S>	180				
Si	SiO2	%	3	4	KA			<Na-S>	180				

Figure 4.13 Application Window - Channel Grid

To select an element manually for the application:

- In the **Channel** field, enter the element symbol.
- Click on the + button, or press **Enter**.

To select all elements for the application:

- Click on the periodic table button: .

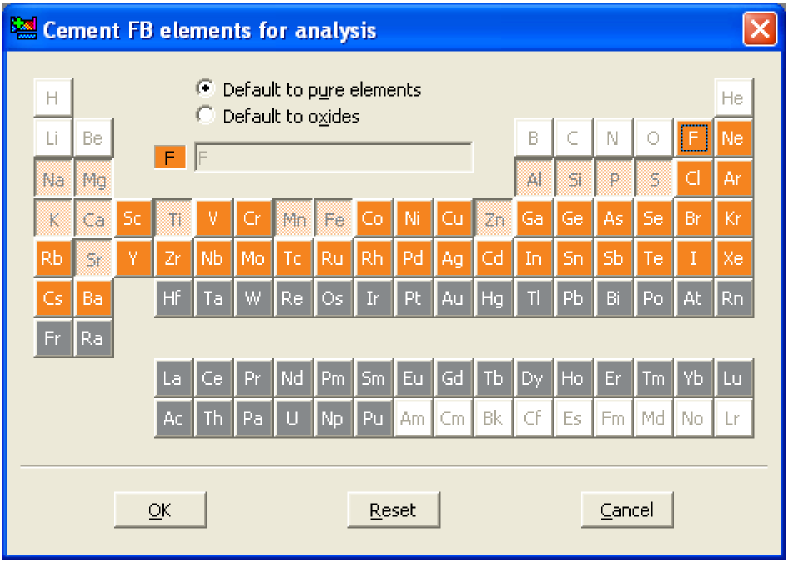


Figure 4.14 Periodic Table Window

- Select the elements you want to measure.
- Click on **OK**.

The new channel name is inserted into the channel grid. The measuring channels are listed in alphabetical order, followed by the manual input and moduli channels respectively. At first, the software proposes the default conditions defined at System level for the selected elements.

IMPORTANT: The moduli channels MUST be defined in the System moduli or Application moduli windows.

For light matrices (like oil or polymers), in order to correct for scatter from the specimen, you should create and use the Compton of the tube lines (Rh-KαC, Mo-KαC, or use a channel with the name Bs (back scatter) or Bsn (where n = 1 to 7).

To delete a channel from the application:

- Enter the channel name into the **Channel** field or click on the required channel in the channel grid.

- Click on the 🗑 button or press the **Delete**. key The channel is removed from the channel grid.

4.3.4 Setting the Channel Parameters

This section describes the parameters you can set for the measuring channels in the channel grid.

Table 4-1 Channel Parameters

Parameter	Description
Compound name	For the main channels, this field can be edited to include chemical formulae or compound definitions for the compounds of interest. The formulae and compound definitions must be correct, as the software uses this data in the calculations. **NOTE:** The input is case sensitive. You must use the correct upper and lower case letters in the chemical abbreviations (for example: Co = cobalt).
Unit	Specifies the units in which the concentrations should be given.
Dec	Specifies the number of decimals to use in the concentrations of the standards and results.
Seq	Specifies the sequence in which the channels will be reported (if you enter 0 they will not be listed in the **Results** window).
Line name	Specifies the line used to calculate the concentrations (KA, KB, LA, LB1, LB2, ROI).
ROI min	Specifies the Region of Interest lower level. If you specified the line type as ROI, here you specify the lower level of that region in keV.
ROI max	Specifies the Region of Interest upper level in keV.
Condition Name	Specifies the name of the condition set to be used for the measurement.
Meas time	Sets the measuring time. The value must be between 1 and 32767 seconds (9 hours 10 minutes).

Table 4-1 Channel Parameters (Continued)

Parameter	Description
Routine min conc/max	The routine minimum and maximum concentrations fields allow you to set concentration limits. If the measured concentration of a sample falls outside these limits, the result is displayed in red with either a “<” or “>” symbol to indicate which limit has been exceeded.
Chk sam min conc/max conc	The check sample minimum and maximum concentrations fields allow you to set concentration limits. If the measured concentration of a sample falls outside these limits, the result is displayed in red with either a “<” or “>” symbol to indicate which limit has been exceeded.

To set a channel parameter:

- Click on the parameter in the channel grid that you want to change.
- Enter in the new value and press **Enter**.

4.3.5 Transmitting Results

In the application, you can set the details of the protocol used for results transmission. When the check box **Transmission of results** is checked in the **System parameters** window, a drop-down list is enabled with the following items:

Table 4-2 Measuring Channel Parameters

Parameter	Description
Yes	Transmits the results automatically. The measured results are transmitted to the folder that was specified in the **System parameters** window.
No	Does not transmit the results.
Ask	Prompts you to perform a transmission for each measurement. The measured results are transmitted to the folder that was specified in the **System parameters** window.

Transmission of results can be done on-line or off-line.

The transmitted files have the following file name format:

XYYYYMMDDHHMMSS.QAN

where: X is fixed
YYYYMMDDHHMMSS relate to the year (YYYY), month (MM), day (DD), hour (HH), minutes (MM) and seconds (SS) the file was saved.

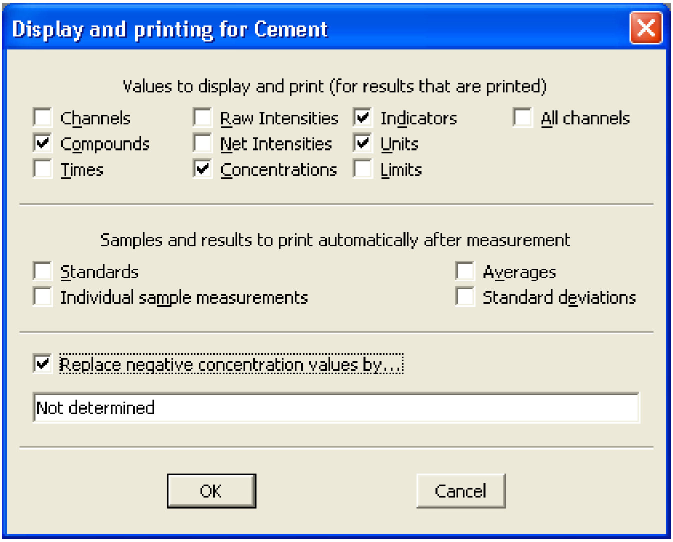
The transmitted files can be imported in other applications, e.g. Excel, for further data processing.

4.3.6 Displaying and Printing Results

You can select what parameters are displayed in the results tables and what sample results are printed.

To set the display and printing options:

- In the channel information area of the **application** window, click on the **Display and printing** button.
- In the **Display and printing** window, select the required options by checking the check boxes.
- Click on the **OK** button, or press **Enter**. Do not forget to save the data.



Display and printing for Cement

Values to display and print (for results that are printed)

☐ Channels
☑ Compounds
☐ Times
☐ Raw Intensities
☐ Net Intensities
☑ Concentrations
☑ Indicators
☑ Units
☐ Limits
☐ All channels

Samples and results to print automatically after measurement

☐ Standards
☐ Individual sample measurements
☐ Averages
☐ Standard deviations

☑ Replace negative concentration values by...

Not determined

Figure 4.15 Display and Printing Window

The **Values to display and print** frame defines which values to display in the results tables.

Table 4-3 Display Items

Parameter	**Description**
Channels	Measuring channel (element) name.
Compounds	Name of compound associated with that channel.
Times	Channel measuring time.
Raw Intensities	Directly measured count rate.
Net Intensities	Intensities corrected for background.
Concentrations	Calculated concentrations.
Indicators	Display warning indicators.
Units	Type of unit selected to display concentration results.

Table 4-3 Display Items (Continued)

Parameter	Description
Limits	Minimum and maximum concentration limits for a channel. If the result is outside these limits it is clearly marked in the Results window.
All channels	Display all channels.

The **Samples and results to print automatically after measurement** frame allows you to select the types of sample results to be printed automatically after each measurement is performed.

Table 4-4 Print Items

Parameter	Description
Standards	Results of standard sample measurements, used for calibration.
Individual sample measurements	Results of routine (unknown) samples. Check this item if you want to obtain a print of the individual measurements resulting from 'repeated'.
Averages	Average results of unknown samples measured more than once, using a repeat factor, or the result of a sample measured once (repeat factor 1).
Standard deviations	Standard deviations of unknown samples measured more than once, using a repeat factor.

4.3.7 Optimizing Conditions

The initial proposal made by the software for the measuring conditions can be further optimized using few measurements on a representative sample (i.e. standard or routine) for that type of material. When using this procedure, only the measuring conditions for a specific application will change and not the original system conditions.

To optimize the conditions:

- Click on the **Optimize Conditions** button and follow the instructions.

4.3.8 Setting an Application Password

You can set passwords for every application to prevent unauthorized personnel from opening and changing the application or from closing the **Application measure sample** window (if it is open).

NOTE: The application passwords are only enabled when the system password is enabled.

To set a password for an application:

- In the **application** window, click on the **Set Password** button.
- In the **New password** field, enter the password. A “*” is displayed for each character typed.
- Enter the password again in the **Verification** field.
- Click on the **OK** button or press **Enter**.
- In the navigation/control area of the **application** window, click on the **Save** button.

If the verification is correct, the **Set new password** window is cleared. If the verification is incorrect, an error message is displayed.

- Click on **OK** to clear this message and display a blank **Set new password** window, in which you can re-enter the password.

The password is only active if a system password has been set. If no system password has been set, the password is stored but stays inactive until the system password is set.

To change an application password:

- In the **application** window, click on the **Set Password** button. The **Set new password** window is displayed.
- In the **Current password** field, enter the current password. A “*” is displayed for each character typed.
- In the **New password** field, enter the new password.
- Enter the new password again in the **Verification** field.

- Click on the **OK** button or press **Enter**. Do not forget to save the application.

If the verification is correct, the **Set new password** window is cleared. If the verification is incorrect, an error message is displayed and you must repeat the procedure.

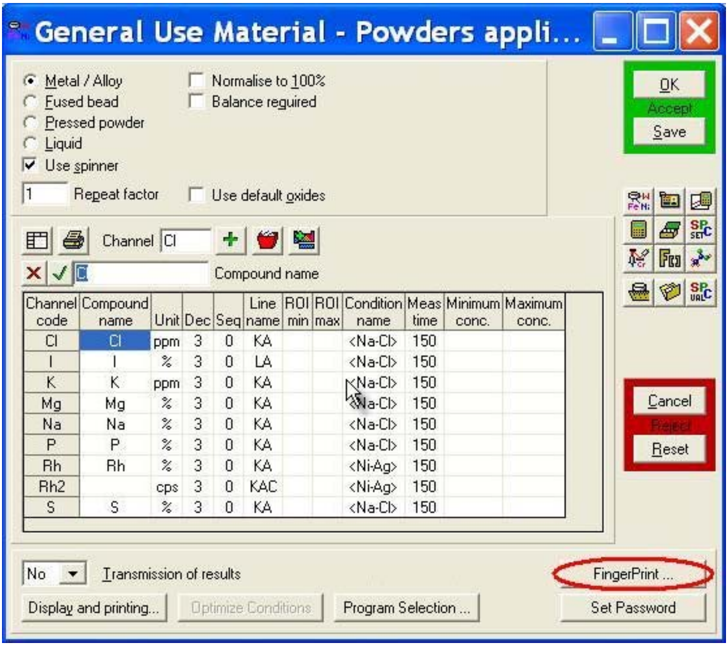
To remove an application password:

- In the **application** window, click on the **Set Password** button. The **Set new password** window is displayed.
- In the **Current password** field, enter the current password. A "*" is displayed for each character typed.
- In the **New password** field, do NOT enter a password.
- In the **Verification** field, again do NOT enter a password.
- Click on the **OK** button or press **Enter**. Do not forget to save the application.

4.3.9 Identifying Material by FingerPrinting

The Epsilon 3 can be used for rapid fingerprint identification of all samples types. The first step in the fingerprint process is the physical identification of types. Solids, liquids and powders should be kept separate. Furthermore, it is also advised to separate on the basis of grain size (for example, pellets and powders) should be measured using different applications.

When developing a FingerPrint application it is not necessary to have elemental concentrations. Simply identify the elements that would distinguish sample types/ identifications and the appropriate measuring conditions. The addition of reference samples/standards in the application becomes a library database of scans to which each measured sample is compared.



● Metal / Alloy
○ Fused bead
○ Pressed powder
○ Liquid
☑ Use spinner
1 Repeat factor

☐ Normalise to 100%
☐ Balance required
☐ Use default oxides

Channel Cl

Cl Compound name

Channel code	Compound name	Unit	Dec	Seq	Line name	ROI min	ROI max	Condition name	Meas time	Minimum conc.	Maximum conc.
Cl	Cl	ppm	3	0	KA			<Na-Cl>	150		
I	I	%	3	0	LA			<Na-Cl>	150		
K	K	ppm	3	0	KA			<Na-Cl>	150		
Mg	Mg	%	3	0	KA			<Na-Cl>	150		
Na	Na	%	3	0	KA			<Na-Cl>	150		
P	P	%	3	0	KA			<Na-Cl>	150		
Rh	Rh	%	3	0	KA			<Ni-Ag>	150		
Rh2		cps	3	0	KAC			<Ni-Ag>	150		
S	S	%	3	0	KA			<Na-Cl>	150		

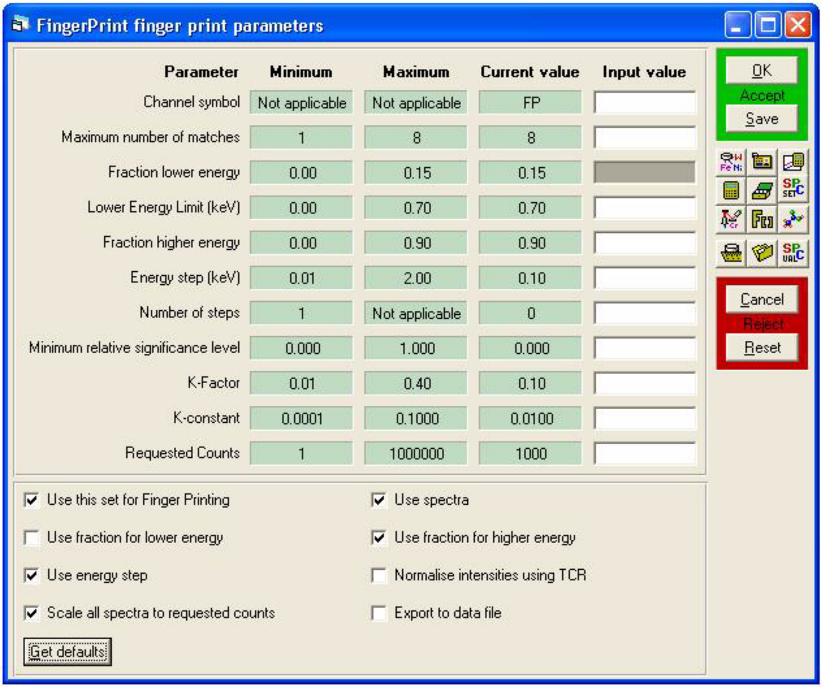
No Transmission of results

Figure 4.16 Application Example for FingerPrint

For advanced use, it is possible to combine a calibrated method with the fingerprinting method. It is important to note that the combination fingerprinting and quantification will follow the same requirements for optimization as a routine quantification application. This can result in extended measurement times.

4.3.9.1 FingerPrinting Example

An example of an application for identifying General Use Materials (GUMs) is displayed in the figure below. The elements (defining the GUMs) were chosen and the appropriate corresponding condition set was employed using the default conditions. The time was set at 150 seconds which matched the total measurement time. Next the FingerPrint option was chosen by selecting the **FingerPrint** button.



Parameter	Minimum	Maximum	Current value	Input value
Channel symbol	Not applicable	Not applicable	FP	
Maximum number of matches	1	8	8	
Fraction lower energy	0.00	0.15	0.15	
Lower Energy Limit (keV)	0.00	0.70	0.70	
Fraction higher energy	0.00	0.90	0.90	
Energy step (keV)	0.01	2.00	0.10	
Number of steps	1	Not applicable	0	
Minimum relative significance level	0.000	1.000	0.000	
K-Factor	0.01	0.40	0.10	
K-constant	0.0001	0.1000	0.0100	
Requested Counts	1	1000000	1000	

☑ Use this set for Finger Printing

☐ Use fraction for lower energy

☑ Use energy step

☑ Scale all spectra to requested counts

☑ Use spectra

☑ Use fraction for higher energy

☐ Normalise intensities using TCR

☐ Export to data file

Figure 4.17 Finger Print Parameters Window

This one-time setup includes choosing from a variety of distinguishing factors controlling the Chi² regression algorithm. For first time use we recommend the following settings in the **FingerPrint parameters** window:

- Create a library - measure standards

  Once the FingerPrint parameters have been set, simply add and measure standards to the database (add concentrations for methods where FingerPrint and quantification is desired) and measure each standard as per a routine application.

- Instant identification - results

  Automatic sample identification will result when measuring samples that match the library database type.

- XRF intelligence - capturing subtle differences

  The FingerPrint software employs a sophisticated statistical algorithm that examines the entire scan. This includes features which are not normally interpreted by XRF, such as peak shape and background profiles. A key advantage is that fingerprinting is a fully automated method without the need for interpretation.

4.3.9.2 FingerPrint Parameters

Table 4-5 FingerPrint Parameter Items

Parameter	Description
Use this set for Finger Printing	Must be checked in order to carry out finger printing.
Use spectra	If this box is checked, the fingerprinting of the sample is done on the spectral intensities. Because some of the reference spectra could have been determined a long time ago, the energy scale is automatically adjusted.
Use fraction for lower energy	The low boundary of the energy range (keV) is either the ‘lower energy limit’ or a fraction of the max energy of the condition used in the application. This fraction is defined by ‘fraction lower energy’.
Use fraction for higher energy	The high boundary of the energy range (keV) is either the max energy of the condition used in the application or a fraction of this energy. This fraction is defined by ‘fraction higher energy’.
Use energy step	It is necessary to choose a method of energy comparison. This parameter specifies step size in keV of the adjusted scan. You can also specify the number of steps between the lower and the higher energy boundary. The step size may also be varied when using this parameter. Alternatively it is possible to define the **Number of steps**. This is necessary when **Use energy step** parameter is not checked.
Normalise intensities using TCR	The preference setting enables users to normalize the spectra by the Total Count Rate (TCR).

Table 4-5 FingerPrint Parameter Items (Continued)

Parameter	Description
Scale all spectra to requested counts	As the name implies, this parameter simply re-scales all the comparative spectra to a specified total count level. If this parameter is selected, a value must exist in the proceeding matrix.
Export to data file	Allows you to export all data to a file.

4.4 CALIBRATING THE SYSTEM FOR AN APPLICATION

Analysis is achieved by comparing the measured intensity of the unknown (routine) samples with the regression lines established during the calibration, using standard samples of known composition. The regression lines are calculated for each element defined in the application.

The calibration consists of the following steps:

- Entering the composition data of the standard samples (see Section 4.4.1).
- Finding and calculating the line grouping (see Section 4.4.2).
- Measuring the standard samples (see Section 4.4.3).
- Performing deconvolution (see Section 4.4.4).
- Calculating the regression lines (see Section 4.4.5).
- Displaying the calibration results (see Section 4.4.6).

4.4.1 Entering Standard Sample Data

The first step in the calibration process is to define the composition of the standard samples. The composition is defined by entering the concentrations for the elements or compounds of interest. The quality of this data must also be indicated, whether it is known to be accurate (High) or is suspect (Low).

If the quality of the composition data is Low, it is used for matrix correction but it is not directly used in the calibration of the compound.

To enter the composition of the standard samples:

- Click on the button. The **standards** window for that application is displayed.

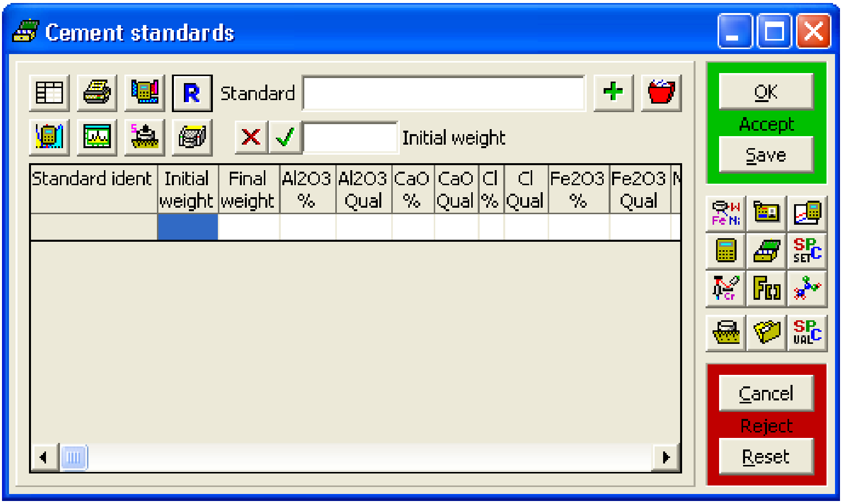


Figure 4.18 Standards Window

This window is used to enter the concentrations of the standard samples and to view the count rates obtained when measuring these samples.

To define a standard:

- In the **Standard** field, enter the name of the standard.
- Click on the + button. This inserts the standard into the grid.

Depending on the defined sample type, other columns for **Initial weight**, **Final weight**, **L.O.I.** and **Balance** automatically appear after the **Standard ident** field. The values entered in the **application** window are automatically inserted.

To enter the concentration data:

- Click on the required grid cell.
- This selects the cell and places the cursor in the **Edit** field.
- Enter the concentration of the element.
- The value is automatically inserted and the data quality is set to High.
- Press **Enter**.
- Enter the concentrations for the other elements in the same way.

To change the data quality level:

- Click on the required **Qual** field.
- This selects that cell and copies the current setting into the **Edit** field.
- Type the first letter of the required level (H = High, L = Low).
- The setting is automatically inserted into the **Qual** field.
- Click on the ✓ button.
- Select any other quality cells you want to change and edit the setting in the same way. See also Section 4.4.6.3.

To delete a standard:

- Click anywhere on the required standard in the table or enter the name in the **Standard** field.
- Click on the 🗑 button to delete the standard from the table.

To save the calibration standards input data:

- In the navigation/control area of the **application** window, click on the **Save** button.

4.4.2 Finding the Line Groups

The software uses deconvolution to calculate the count rates (expressed in units of cps = counts per second). Deconvolution is a mathematical procedure by which the relative intensities of the characteristic fluorescent lines are determined by fitting the detector response function to the measured spectrum. In order to do that, the characteristic lines to be considered must be made known to the software along with some detector specific information.

Some elements have many lines, some of which are weak or very close together. The software evaluates these lines and creates the appropriate line groups.

To automatically calculate the appropriate line groups:

- In the **standards** window, enter all concentrations for the compounds and click on the button.

The spectrum evaluation is done by non-linear least squares fitting, based on the AXIL algorithm developed at the University of Antwerp.

4.4.3 Measuring Standard Samples

When you have entered the standard samples concentration data, you must then measure the samples.

To measure the standard samples:

- Load the selected standard into the spectrometer.
- In the **standards** window, click on the standard(s) to measure in the grid.

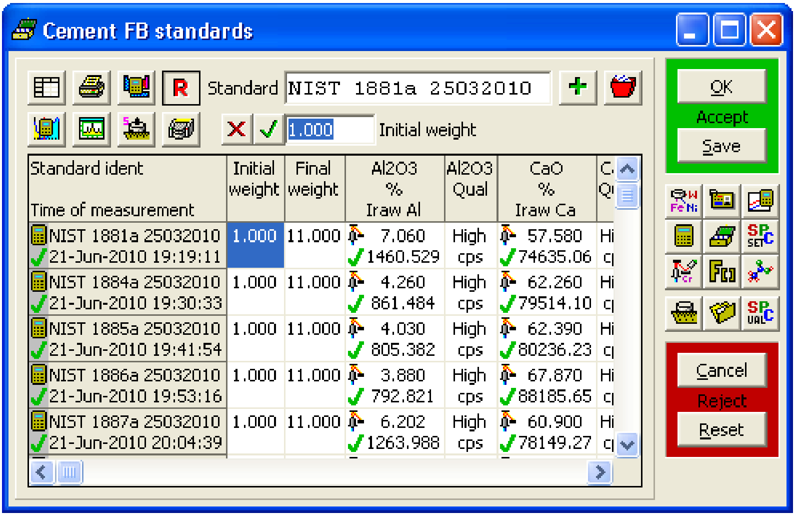
  NOTE: You can select multiple samples by pressing the **Ctrl** key or **Shift** key. Clicking on the button at the top of the **standards** window inserts all samples into the measuring queue at once.
- Click on the button at the top of the **standards** window.
- Select the proper position(s) on the sample changer.
- The selected sample is measured.

4.4.3.1 Viewing Standard Samples Measurement Results

After the standard samples have been measured, the count rates can be displayed (but not edited) in the **standards** window.

To display the raw (measured) count rates:

- Click on the R button.



Standard ident Time of measurement	Initial weight	Final weight	Al2O3 % Iraw Al	Al2O3 Qual	CaO % Iraw Ca	C Q
NIST 1881a 25032010 ✓ 21-Jun-2010 19:19:11	1.000	11.000	7.060 ✓ 1460.529	High cps	57.580 ✓ 74635.06	Hi c
NIST 1884a 25032010 ✓ 21-Jun-2010 19:30:33	1.000	11.000	4.260 ✓ 861.484	High cps	62.260 ✓ 79514.10	Hi c
NIST 1885a 25032010 ✓ 21-Jun-2010 19:41:54	1.000	11.000	4.030 ✓ 805.382	High cps	62.390 ✓ 80236.23	Hi c
NIST 1886a 25032010 ✓ 21-Jun-2010 19:53:16	1.000	11.000	3.880 ✓ 792.821	High cps	67.870 ✓ 88185.65	Hi c
NIST 1887a 25032010 ✓ 21-Jun-2010 20:04:39	1.000	11.000	6.202 ✓ 1263.988	High cps	60.900 ✓ 78149.27	Hi c

Figure 4.19 Standards Window

The symbols that can appear in the **standards** window have the following meaning:

▦ & ✗	The net intensities for this standard were calculated using a line overlap matrix that was out of date. The standard cannot be used for calibration.
▦ & ✓	The standard was deconvolved with the correct elements and line groups according to the application.
🧪 & ✗	The intensity was obtained using conditions that do not match the current conditions in the application. The intensity cannot be used for calibration.

K & ✗	The intensity was obtained using a line which does not match the current line in the application (for example KA was changed to KB in the application), or using an R.O.I. that does not match the current application R.O.I. The intensity cannot be used for calibration.
⚗ & ✓	The intensity was obtained using the correct line (or R.O.I.) and correct conditions according to the application.
If both ⚗ & ✗ and K & ✗ apply, then K & ✗ is shown (takes priority).	

4.4.3.2 Calculating Net Intensities for the Standards

If there are any red crosses in the table you have to measure that the standard(s) again or allow the software to recalculate the existing data.

To recalculate the existing data:

- Select all the standards by clicking on the top left cell of the grid.
- Click on the 🖩 button.

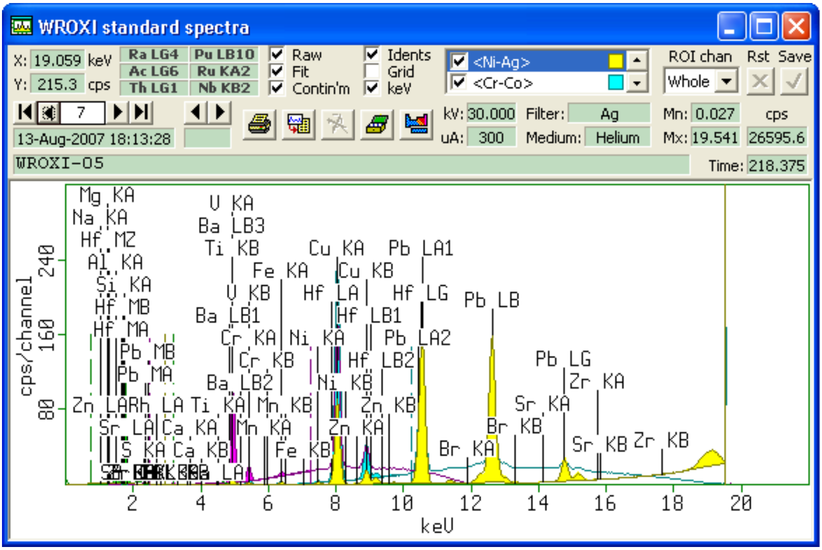
TIP: If you click on the R button before clicking on the 🖩 button, you will see the intensities appear in the table as they are deconvoluted.

4.4.4 Performing Deconvolution

After calculating the theoretical line groups and measuring the standards, it is advised to check and optimize the deconvolution of the obtained spectra.

To check and optimize the deconvolution:

- Select a certain standard in the **standards** window.
- Click on the 📈 button. The **standard spectra** window is displayed showing the measured sample spectrum of the standard.



X: 19.059 keV
Y: 215.3 cps

☑ Raw
☑ Fit
☑ Contin'm
☑ Idents
☐ Grid
☑ keV

☑ <Ni-Ag>
☑ <Cr-Co>

Whole

7

13-Aug-2007 18:13:28

kV: 30.000
uA: 300
Filter: Ag
Medium: Helium
Mn: 0.027
Mx: 19.541
cps 26595.6

WROXI-05

Time: 218.375

Figure 4.20 Standard Spectra Window

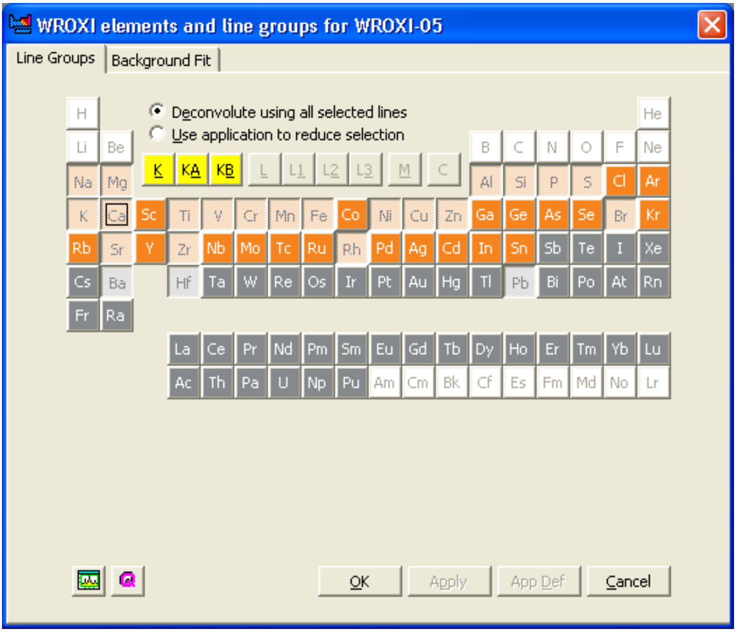
- In the **sample spectrum** window, click on the 🟦 button. The **elements and line groups** window (also called deconvolution window) is displayed, which consists of two tabs:
  - ◆ **Line Groups** (see Section 4.4.4.1)
  - ◆ **Background Fit** (see Section 4.4.4.2)
  - ◆ When the optional Omnian software is installed, an additional tab **Omnian** is also present. For more information, see Chapter 6.

4.4.4.1 Line Groups Tab

The **Line Groups** tab in the deconvolution window enables the user to include/exclude elements in order to improve the deconvolution of the standards and routine samples. After selection (see Figure 4.21), the software will automatically include those elements to the deconvolution.

In most cases, more elements are present in the standards or routine samples than selected by the user. Therefore it is better to include all elements in the deconvolution that are present in the spectrum (spectra). In that way, calculating possible line overlaps and matrix effects will be more accurate.

In some cases, the deconvolution can be improved even more when the K-lines (or L-lines) are de-coupled from each other, like KA and KB separately (or L1, L2 and L3), particularly for the tube lines where the KA and KB ratio depends on the type of sample material.
The K-lines (or L-lines) are de-coupled by clicking on the element of interest and selecting/deselecting K, KA or KB line (or L, L1, L2 or L3).



WROXI elements and line groups for WROXI-05

● Deconvolute using all selected lines
○ Use application to reduce selection

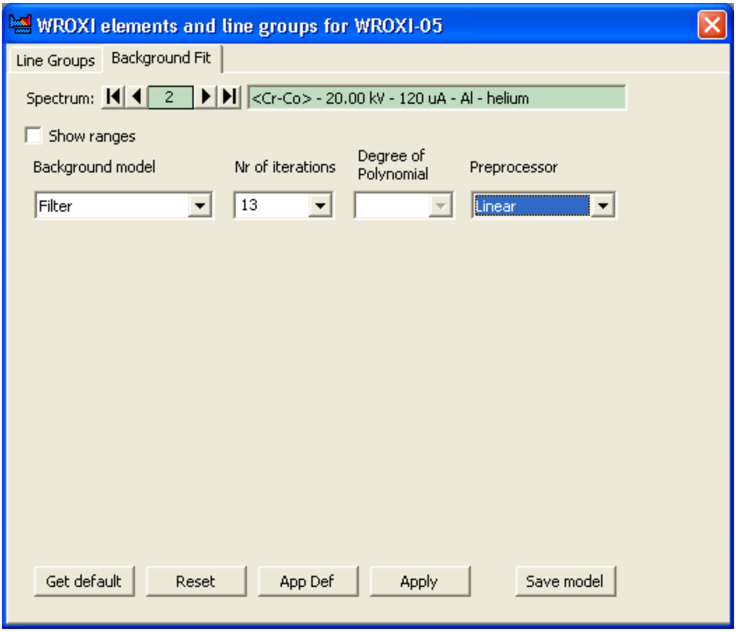
Figure 4.21 Elements and Line Groups Window - Line Groups Tab

4.4.4.2 Background Fit Tab

The **Background Fit** tab in the deconvolution window enables the user to select the best background fitting model. For each different measurement condition, it is possible to change and optimize the background fitting by configuring:

- Background model
- Number of iterations (only applicable to background models **Filter**, **Classic filter** and **Axil filter**)

- Degree of polynomial (only applicable to background models **Exponential** and **Linear Polynomial**)
- Preprocessor (only applicable to background models **Filter**, **Exponential** and **Linear Polynomial**)



Spectrum: 2 <Cr-Co> - 20.00 kV - 120 uA - Al - helium

☐ Show ranges

Background model: Filter

Nr of iterations: 13

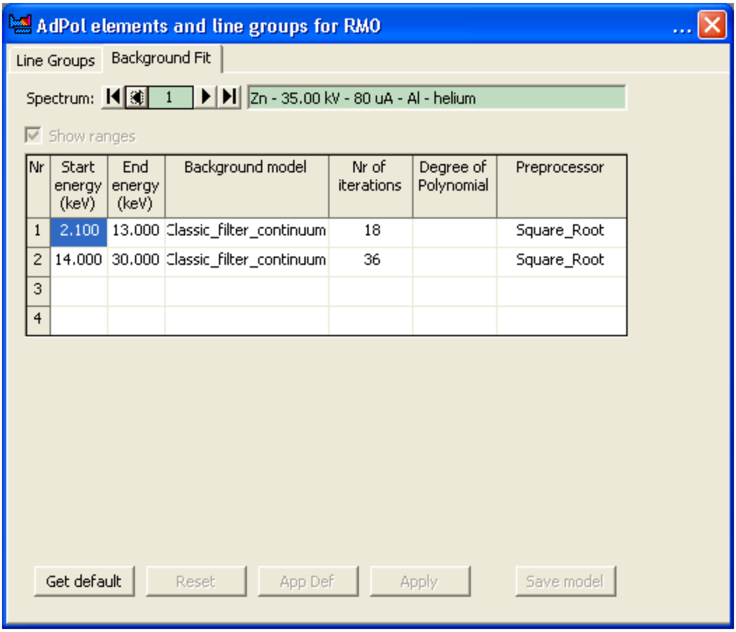
Degree of Polynomial:

Preprocessor: Linear

Figure 4.22 Elements and Line Groups Window - Background Fit Tab

Up to 4 different ranges can be combined to deconvolute the same spectrum. Each energy range can have its own background model.

After any change in the deconvolution window, save the changes by clicking on the **Save model** button and recalculate the intensities for all standards in the standards window using the updated background model.



Spectrum: 1 Zn - 35.00 kV - 80 uA - Al - helium

☑ Show ranges

Nr	Start energy (keV)	End energy (keV)	Background model	Nr of iterations	Degree of Polynomial	Preprocessor
1	2.100	13.000	Classic_filter_continuum	18		Square_Root
2	14.000	30.000	Classic_filter_continuum	36		Square_Root
3						
4						

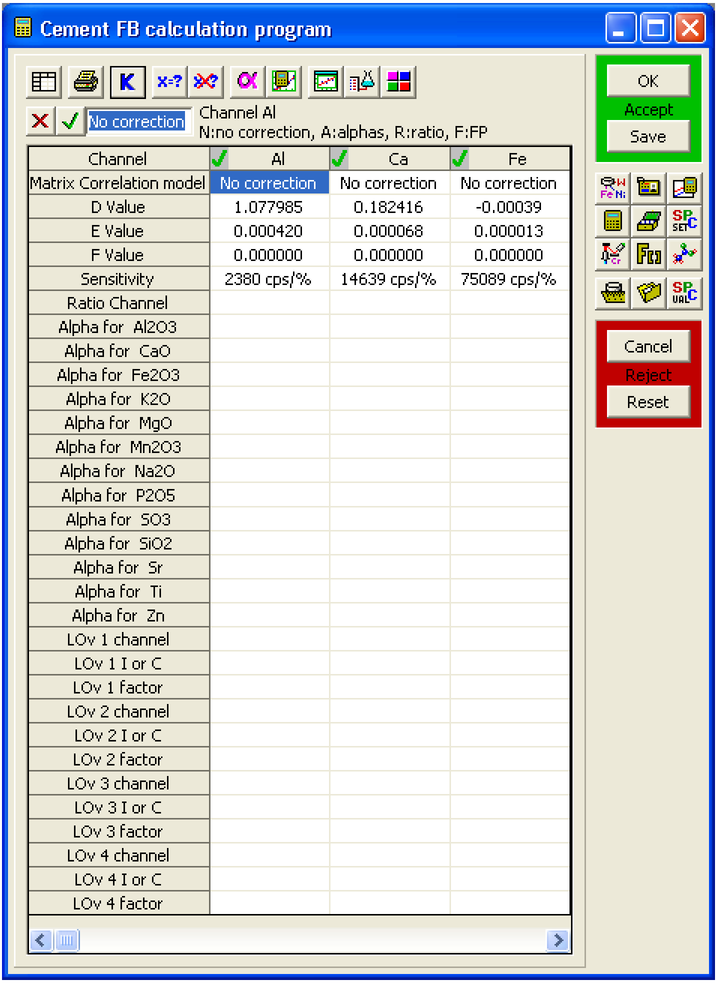
Figure 4.23 Background Model

4.4.5 Calculating the Regression Lines

When the standard samples have been measured, the regression lines for all the elements listed must be calculated. The calculations take a number of correction factors into account, i.e. Alphas, fundamental parameters (FP), Compton ration correction and Line overlaps.

To calculate the regression lines:

- Click on the 🖩 button to display an empty **calculation program** window.



No correction

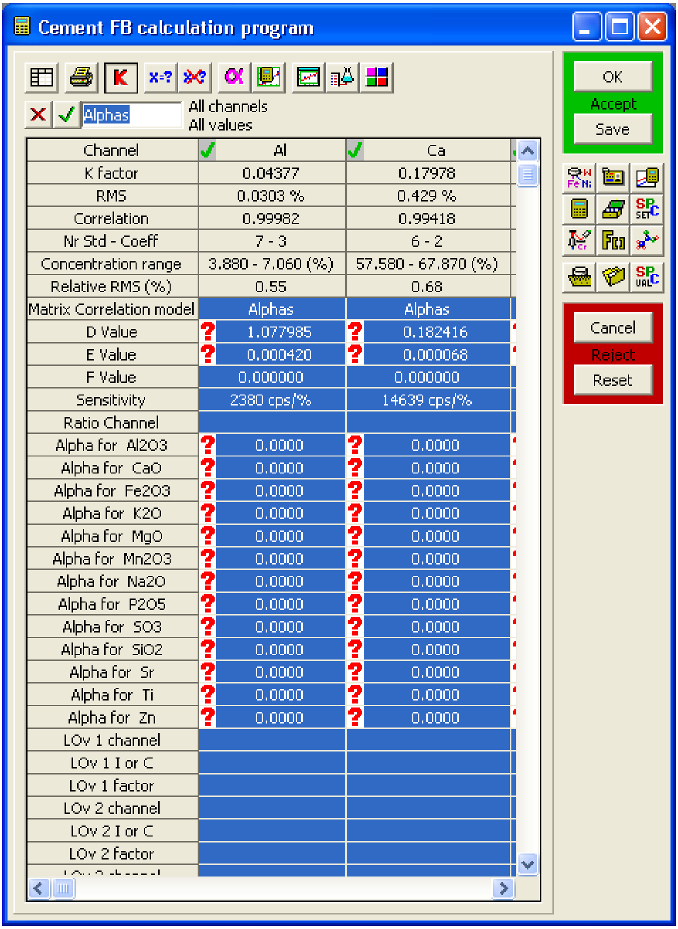
Channel Al
N:no correction, A:alphas, R:ratio, F:FP

Channel	✓ Al	✓ Ca	✓ Fe
Matrix Correlation model	No correction	No correction	No correction
D Value	1.077985	0.182416	-0.00039
E Value	0.000420	0.000068	0.000013
F Value	0.000000	0.000000	0.000000
Sensitivity	2380 cps/%	14639 cps/%	75089 cps/%
Ratio Channel			
Alpha for Al2O3			
Alpha for CaO			
Alpha for Fe2O3			
Alpha for K2O			
Alpha for MgO			
Alpha for Mn2O3			
Alpha for Na2O			
Alpha for P2O5			
Alpha for SO3			
Alpha for SiO2			
Alpha for Sr			
Alpha for Ti			
Alpha for Zn			
LOv 1 channel			
LOv 1 I or C			
LOv 1 factor			
LOv 2 channel			
LOv 2 I or C			
LOv 2 factor			
LOv 3 channel			
LOv 3 I or C			
LOv 3 factor			
LOv 4 channel			
LOv 4 I or C			
LOv 4 factor			

Figure 4.24 Calculation Program Window

- Click on the K button to display the K-value, RMS and correlation coefficient for all the channels at the top of the grid.
  NOTE: The F-value is not automatically set to be calculated.

- In the **calculation program** window, select all parameters by clicking in the top left cell of the grid (**Channel**). All cells that will be calculated are now selected (blue).
- Click on the x=? button. All values to be calculated are indicated by a red “?” in front of the value.



Alphas

All channels
All values

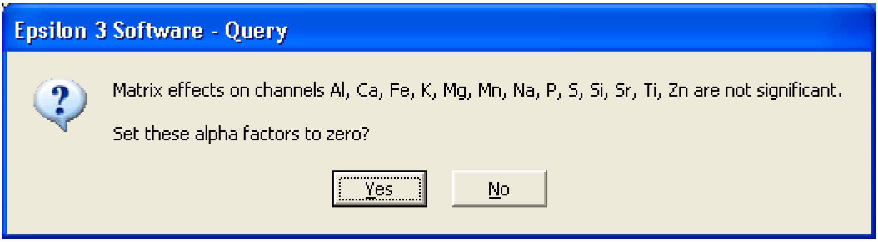
Channel	✓ Al	✓ Ca
K factor	0.04377	0.17978
RMS	0.0303 %	0.429 %
Correlation	0.99982	0.99418
Nr Std - Coeff	7 - 3	6 - 2
Concentration range	3.880 - 7.060 (%)	57.580 - 67.870 (%)
Relative RMS (%)	0.55	0.68
Matrix Correlation model	Alphas	Alphas
D Value	? 1.077985	? 0.182416
E Value	? 0.000420	? 0.000068
F Value	0.000000	0.000000
Sensitivity	2380 cps/%	14639 cps/%
Ratio Channel		
Alpha for Al2O3	? 0.0000	? 0.0000
Alpha for CaO	? 0.0000	? 0.0000
Alpha for Fe2O3	? 0.0000	? 0.0000
Alpha for K2O	? 0.0000	? 0.0000
Alpha for MgO	? 0.0000	? 0.0000
Alpha for Mn2O3	? 0.0000	? 0.0000
Alpha for Na2O	? 0.0000	? 0.0000
Alpha for P2O5	? 0.0000	? 0.0000
Alpha for SO3	? 0.0000	? 0.0000
Alpha for SiO2	? 0.0000	? 0.0000
Alpha for Sr	? 0.0000	? 0.0000
Alpha for Ti	? 0.0000	? 0.0000
Alpha for Zn	? 0.0000	? 0.0000
LOv 1 channel		
LOv 1 I or C		
LOv 1 factor		
LOv 2 channel		
LOv 2 I or C		
LOv 2 factor		

Figure 4.25 Calculation Program Window - Before Calculation

- Click on the α button to calculate the theoretical alpha factors.

NOTE: If you do not click on the α button before clicking on the 🖩 button, you will obtain empirical alpha factors (not theoretical alpha factors).

- A window may be displayed informing you if any of the correction factors are not at a significant level. We recommend that you accept the default, by clicking on **Yes**.



Epsilon 3 Software - Query

Matrix effects on channels Al, Ca, Fe, K, Mg, Mn, Na, P, S, Si, Sr, Ti, Zn are not significant.

Set these alpha factors to zero?

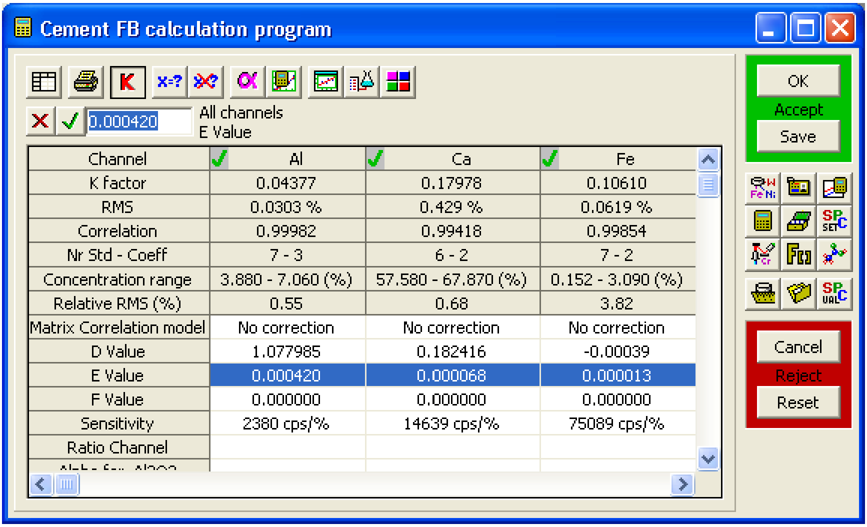
Figure 4.26 Correction Factors Message

- In the **calculation program** window, click on the 🖩 button to calculate the regression values.

  All the parameters have now been calculated, either by theory or regression analysis.

  NOTE: You can fix the values of individual or groups of parameters you do not want to calculate by selecting the required cells and clicking on the ✗? button.

- In some cases, calibration results can be improved using FPs (fundamental parameters) or Compton ratio correction. These corrections can be implemented in the calculation program when entering “F” (FP) or “R” (Ratio channel) in the **Matrix Correction model** field. For the Compton correction, also the **Ratio Channel** field must be filled with “Rh-KAC” or “Bs” (back scatter). These ratio channels can only be selected when included in the **application** window.



0.000420 All channels
E Value

Channel	✓ Al	✓ Ca	✓ Fe
K factor	0.04377	0.17978	0.10610
RMS	0.0303 %	0.429 %	0.0619 %
Correlation	0.99982	0.99418	0.99854
Nr Std - Coeff	7 - 3	6 - 2	7 - 2
Concentration range	3.880 - 7.060 (%)	57.580 - 67.870 (%)	0.152 - 3.090 (%)
Relative RMS (%)	0.55	0.68	3.82
Matrix Correlation model	No correction	No correction	No correction
D Value	1.077985	0.182416	-0.00039
E Value	0.000420	0.000068	0.000013
F Value	0.000000	0.000000	0.000000
Sensitivity	2380 cps/%	14639 cps/%	75089 cps/%
Ratio Channel			

Figure 4.27 Calculation Program Window - After Calculation

- In the navigation/control area of the **application** window, save the calibration by clicking on the **Save** button.

4.4.6 Displaying the Calibration Results

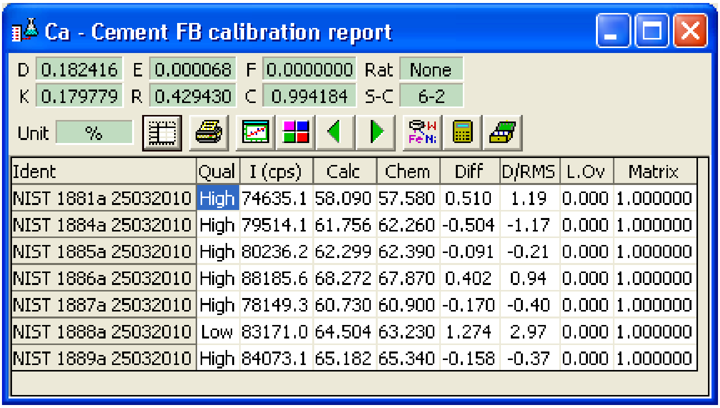
The calibration results for each channel can be viewed, either as a table or as a graph showing the regression line. These options are available from the **calculation program** window.

4.4.6.1 Calibration Results Table

The **calibration report** window displays the results for a channel in the form of a report table. It shows the relevant data of that channel for each of the measured calibration standards.

To display the calibration results in a table:

- In the **calculation program** window, select the required channel by clicking anywhere in that column of the grid.
- Click on the button at the top of the **calculation program** window. The **calibration report** window is displayed.



D 0.182416 E 0.000068 F 0.0000000 Rat None
K 0.179779 R 0.429430 C 0.994184 S-C 6-2
Unit %

Ident	Qual	I (cps)	Calc	Chem	Diff	D/RMS	L.Ov	Matrix
NIST 1881a 25032010	High	74635.1	58.090	57.580	0.510	1.19	0.000	1.000000
NIST 1884a 25032010	High	79514.1	61.756	62.260	-0.504	-1.17	0.000	1.000000
NIST 1885a 25032010	High	80236.2	62.299	62.390	-0.091	-0.21	0.000	1.000000
NIST 1886a 25032010	High	88185.6	68.272	67.870	0.402	0.94	0.000	1.000000
NIST 1887a 25032010	High	78149.3	60.730	60.900	-0.170	-0.40	0.000	1.000000
NIST 1888a 25032010	Low	83171.0	64.504	63.230	1.274	2.97	0.000	1.000000
NIST 1889a 25032010	High	84073.1	65.182	65.340	-0.158	-0.37	0.000	1.000000

Figure 4.28 Calibration Report Window

4.4.6.2 Calibration Report Window

The **calibration report** window is split in two parts: the upper part shows the calculated regression values and the lower part shows a grid containing the measurement results for each standard sample.

- Double-clicking in any cell of the grid selects that standard and opens the **calibration standards** window for that application. The concentration value of that channel for the selected standard is highlighted.

The **calibration report** window has a number of buttons for easily viewing the other calibration results.

Table 4-6 Calibration Report Window Buttons

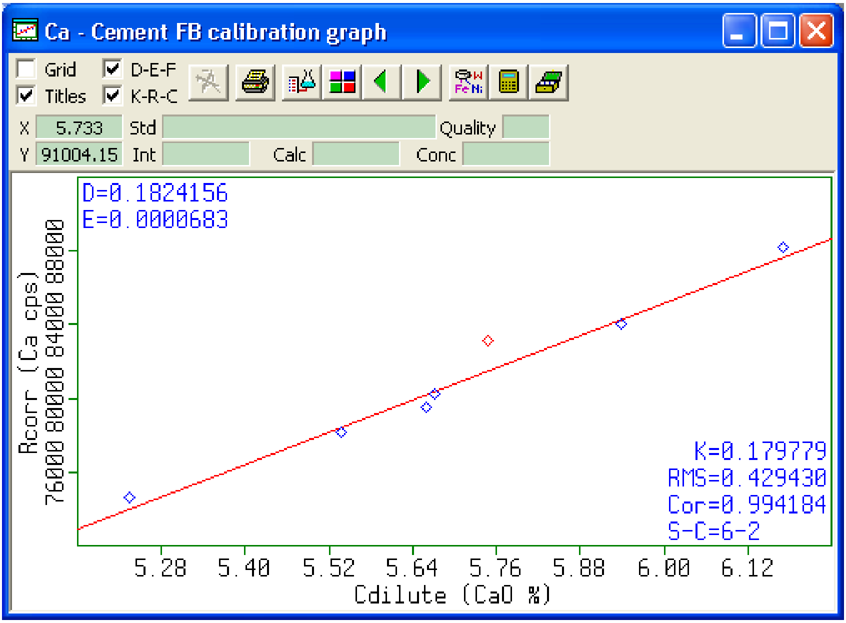
Button	Description
📈	Displays a graph of the results table.
◀	Displays the results of the previous channel in the alphabetical list on the **calculation program** window in a separate window.
▶	Displays the results of the next channel in the alphabetical list on the **calculation program** window in a separate window.
⊞	Tiles all open **calibration report** windows and minimizes all other open windows.
In addition, three application navigation buttons are available to open the **application** window, **calculation program** window and **standards** windows for that application.	

4.4.6.3 Calibration Results Graph

The calibration graph shows the calculated regression line, together with the calculated count rates for every standard.

To display the calibration results in a graph:

- In the **calculation program** window, select the required channel by clicking anywhere in that column of the grid.
- Click on the 📈 button at the top of the **calculation program** window. The **calibration graph** window is displayed.



Ca - Cementi FB calibration graph

☐ Grid ☑ D-E-F
☑ Titles ☑ K-R-C

X 5.733
Y 91004.15

Figure 4.29 Calibration Graph Window

4.4.6.4 Calibration Graph Window

The **calibration graph** window plots the count rate (cps) against the concentration (%) and shows the calculated regression line for that channel. Each point marked on the graph is the result from the measurement and calculations applied to a particular standard.

The position of the mouse cursor over the graphical display is shown in the **X** and **Y** fields, directly above the left side of the graph. The positional units are given in relation to the scales.

Clicking anywhere over the graph causes the nearest data point to be selected (color changes from blue to green). Details of the result represented by the selected data point are displayed above the graph. This includes: standard name, defined concentration, calculated concentration, etc.

The check boxes in the upper left corner of the **calibration graph** window allow you to display additional information on the graph.

- Double-clicking in the graph selects the nearest result point (measurement result from one standard) and displays the **calibration standards** window for that application. The concentration value of that channel for the selected standard is highlighted.
- Double-clicking on the graph scales or titles displays **Calibration graph scales** window.
- Right-clicking in the graph opens a menu that allows you additional functionality like **Select all, Copy to clipboard, Print, Zoom/Unzoom**.

If any of the standard result points are ‘outliers’ (not close enough to the calibration line) that should not be used for the calibration, you can mark them as low quality (not to be used for the regression).

To select outliers:

- Right-click in the graph and select **Toggle quality** or double-click close to the outlying point. The **standards** window is opened.
- In the **standards** window, mark the entry and change the quality to **Low**.
- Save the change and recalculate the regression. In the graph, you will see that the result point has changed from blue to red.
- This process is also completely automated by selecting menu item **Toggle quality**, provided that the ‘?’ is still available in the corresponding cell in the grid of the **calibration report** window.

Besides the same buttons as on the **calibration report** window, the **calibration graph** window has the following additional buttons for easily viewing the other calibration results.

Table 4-7 Additional Calibration Graph Window Buttons

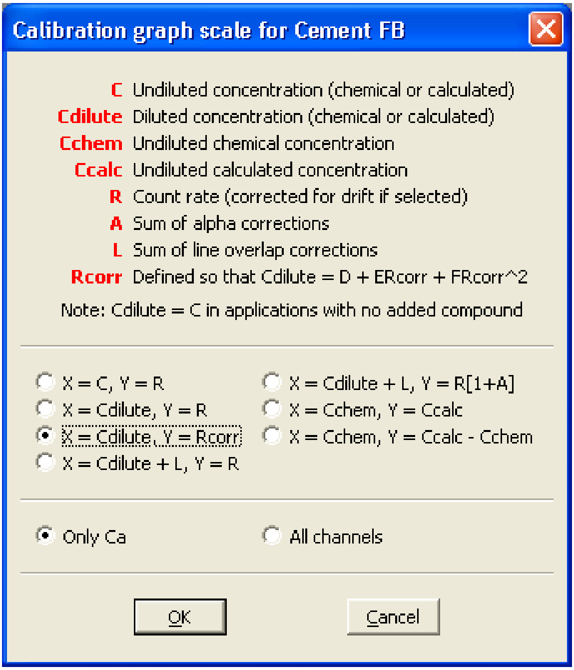
Button	Description
	Displays the results table of the graph.
	Zooms out to display the complete graph.
In addition, three application navigation buttons are available to open the **application** window, **calculation program** window and **standards** windows for that application.	

4.4.6.5 Graph Scales

The parameters of the X and Y scales in the results graph can be changed to enable you to compare different aspects of the calibration results.

To select other graph scales:

- In the **calibration graph** window, double-click on the graph scales. The **Calibration graph scales** window is displayed.
- Select the required option.
- Click on the **OK** button or press **Enter**. The **Calibration graph scales** window closes and the **calibration graph** window shows the graph for the selected scales.



Calibration graph scale for Cement FB

C	Undiluted concentration (chemical or calculated)
Cdilute	Diluted concentration (chemical or calculated)
Cchem	Undiluted chemical concentration
Ccalc	Undiluted calculated concentration
R	Count rate (corrected for drift if selected)
A	Sum of alpha corrections
L	Sum of line overlap corrections
Rcorr	Defined so that Cdilute = D + ERcorr + FRcorr^2

Note: Cdilute = C in applications with no added compound

○ X = C, Y = R
○ X = Cdilute, Y = R
● X = Cdilute, Y = Rcorr
○ X = Cdilute + L, Y = R
○ X = Cdilute + L, Y = R[1+A]
○ X = Cchem, Y = Ccalc
○ X = Cchem, Y = Ccalc - Cchem

● Only Ca
○ All channels

Figure 4.30 Calibration Graph Scale Window

4.5 OPTIONAL APPLICATION FUNCTIONS

A number of the functions in the **application** window and some of the other windows can be optionally defined. They are not required for the basic application definition, but provide additional options. See also the Epsilon 3 On-line Help.

NOTE: The Statistical Process Control (SPC) option is described in Chapter 7.

.

Table 4-8 Optional Application Functions

Function	**Associated Epsilon 3 Software Window**
Ident system function	**Ident system** window
Application moduli definitions	**Application moduli functions** window
Application compounds definitions	**Application compounds definitions** window

Chapter 5

Measuring Samples

5.1 INTRODUCTION

In this chapter we describe how to perform measurements of routine (unknown) samples.

The measurement of routine samples is normally carried out within a defined application, but samples can also be measured using the manual control function.

NOTE: If you are performing quantitative analysis, the measurement of calibration standards must be done prior to measuring unknown samples and is described in Chapter 4.

5.1.1 Measurement Overview

The steps for carrying out measurements are listed briefly below. These steps are described in detail in the following sections.

- Load the sample.
- Open the measure window for the required application.

NOTE: Up to 10 samples can be loaded in the spectrometer.

- Give an identity code to the sample.
- Start the measurement.

The progress of a measurement can be monitored on the status bar. More detailed status information can be viewed on the **Online status** window. The results are available as soon as the measurement is complete on the **Last result** window.

5.2 SAMPLE LOADING

Solid samples must be between 27 and 51.5 mm diameter with a maximum height of 4.9 cm on Epsilon 3 (with standard lid) and 10 cm on Epsilon 3 XL (with high lid) respectively. Liquid samples (typically 10 ml) must be placed in the special “P1” or “P2” plastic cup sample holders. These sample holders are then loaded in the same way as solid samples. Steel rings must be between 40 and 51.5 mm diameter.

Caution **General hazard**

Extra care must be taken with liquid samples to avoid damaging the liquid sample holder in any way, as this may cause the liquid to leak into the spectrometer.

The samples are loaded into the sample changer manually.

To load a sample into the spectrometer:

- Lift the spectrometer lid.
- Place the sample in the required position on the sample changer.
- Close the lid.

When you measure the sample, the sample and its position in the changer have to be identified to the software. This is done using the **Select changer position** window.

5.3 MEASURING A SAMPLE

Measurements of routine samples are normally carried out from an application (see Section 5.3.1 and Section 5.3.2). Alternatively, individual samples can be measured from the **Manual control** window (see Section 5.3.3).

5.3.1 Measuring a Routine Sample

5.3.1.1 Opening the Measure Sample Window

To open the measure sample window from the menu bar:

- Select **Measure > Measure application**. The **Measure application** window is displayed.

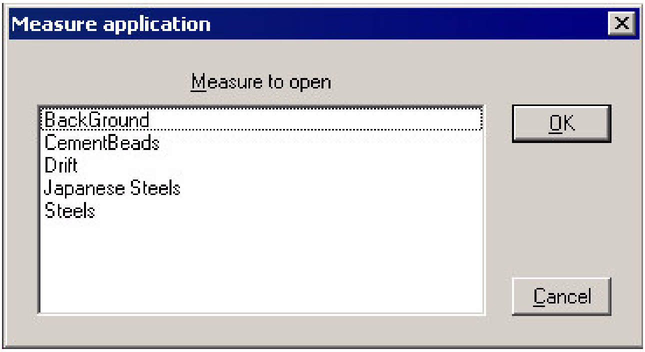
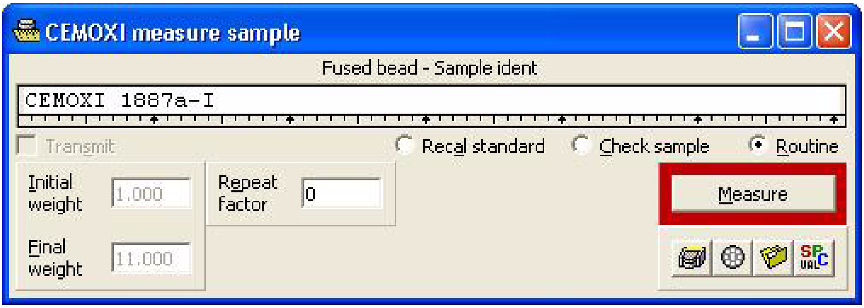


Figure 5.1 Measure Application Window

- Select the application to measure and click on **OK** or press **Enter**. The **measure sample** window of the selected application is opened.



Fused bead - Sample ident: CEMOXI 1887a-I

☐ Transmit ○ Recal standard ○ Check sample ● Routine

Initial weight: 1.000

Final weight: 11.000

Repeat factor: 0

Figure 5.2 Measure Sample Window

To open the measure sample window from the toolbar:

- Click on the button. If no **measure sample** windows are currently open, the **Measure application** window is displayed. Select the application to measure and click on **OK** or press **Enter**. The **measure sample** window of the selected application is opened.
- If one **measure sample** is already open, that **measure sample** window is made the active window and displayed on top.
- If more than one **measure sample** windows are already open, the software asks which **measure sample** window to open.

To open the measure sample window from an application window:

- In the navigation/control area of the **application** window, click on the button. The **measure sample** window of the current application is opened.

5.3.1.2 Measuring the Sample

- In the **Sample ident** field, enter the name of the sample.

  NOTE: This step is not necessary if the samples are automatically named by the application sample ident function.
- Load the sample into the spectrometer.
- Click on the **Measure** button to start the measurement. The **Select changer position** window is displayed.

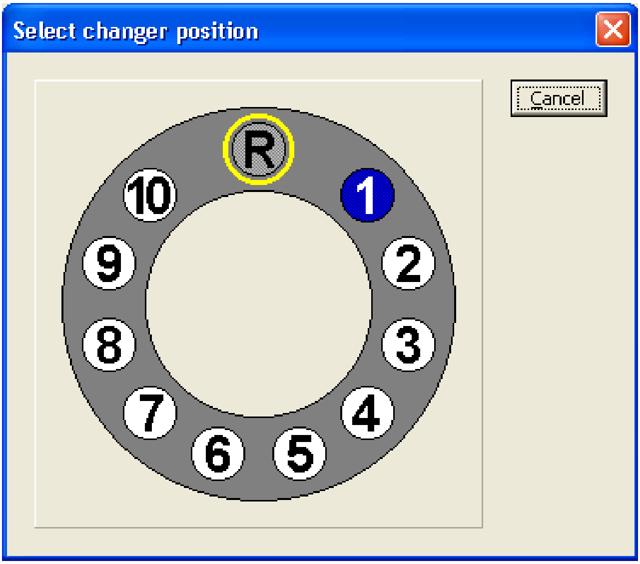


Figure 5.3 Select Changer Position Window

- In the **Select changer position** window, click on the position of the sample.
  The measurement will start automatically if the spectrometer is ready. If it is not ready (e.g. the lid is open or the gain is being measured) the measurement will not start.
- You can then set up further measurements by clicking on the **Measure** button and defining the position to measure. These settings are stored and the measurements are carried out when the current/previous measurements are complete.
- You can also change applications between measurements by selecting the **Measure application** window for a different application and then clicking on the **Measure** button in the new window.

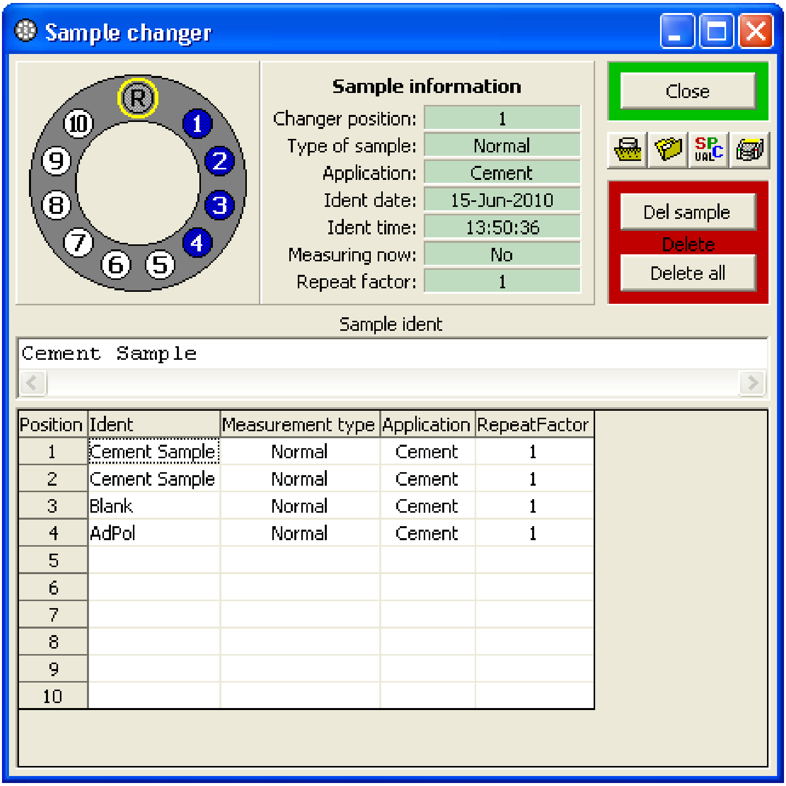
5.3.1.3 Sample Changer Window

The **Sample changer** window shows the status of the sample changer, including its current position and which positions have samples defined. It can also display details of the samples in the measuring queue.

Samples can be deleted individually or all at once.

To open the Sample changer window:

- Select **Measure** > **Sample changer** or click on the button in the **Online status** window.



Sample information

Changer position: 1
Type of sample: Normal
Application: Cement
Ident date: 15-Jun-2010
Ident time: 13:50:36
Measuring now: No
Repeat factor: 1

Sample ident: Cement Sample

Position	Ident	Measurement type	Application	RepeatFactor
1	Cement Sample	Normal	Cement	1
2	Cement Sample	Normal	Cement	1
3	Blank	Normal	Cement	1
4	AdPol	Normal	Cement	1
5				
6				
7				
8				
9				
10				

Figure 5.4 Sample Changer Window

The top left frame of the **Sample changer** window shows the status of the sample changer by a schematic picture, similar to the **Select changer position** window.

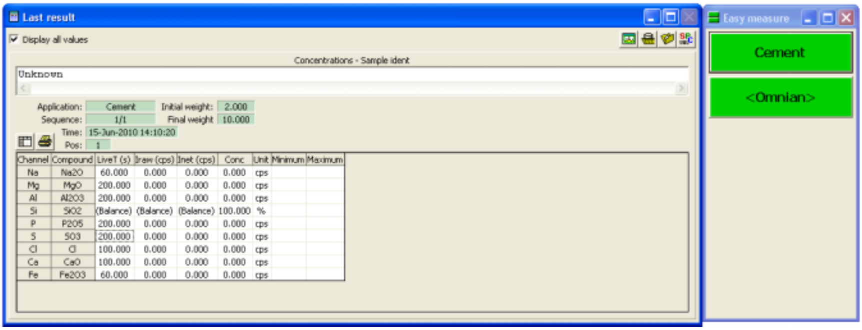
- Defined sample changer positions are shown with a white number on a blue background. Undefined positions are shown as a black number on a white background.
- The current position of the sample changer is shown by the yellow circle, indicating the measuring position.

The **Sample information** frame in the middle of the **Sample changer** window shows detailed information of the sample currently in the measuring position, including sample type (normal, calibration) and related application.

5.3.2 Easy Measuring

It is possible to set up the software to perform easy measurement of samples, for example if you have already set up a condition set and an open the **application** window.

- Open a **Measure application** window (see Section 5.3.1).
- Click on the 🟩 button on the toolbar. The **Last result** window and the **Easy measure** window are displayed. The **Easy measure** window contains a large green button with the application name.



☑ Display all values

Concentrations - Sample ident

Unknown

Application: Cement
Initial weight: 2.000
Sequence: 1/1
Final weight 10.000
Time: 15-Jun-2010 14:10:20
Pos: 1

Channel	Compound	LiveT (s)	Iraw (cps)	Inet (cps)	Conc	Unit	Minimum	Maximum
Na	Na2O	60.000	0.000	0.000	0.000	cps		
Mg	MgO	200.000	0.000	0.000	0.000	cps		
Al	Al2O3	200.000	0.000	0.000	0.000	cps		
Si	SiO2	(Balance)	(Balance)	(Balance)	100.000	%		
P	P2O5	200.000	0.000	0.000	0.000	cps		
S	SO3	200.000	0.000	0.000	0.000	cps		
Cl	Cl	100.000	0.000	0.000	0.000	cps		
Ca	CaO	100.000	0.000	0.000	0.000	cps		
Fe	Fe2O3	60.000	0.000	0.000	0.000	cps		

Figure 5.5 Last Result + Easy Measure Window

- In the **Easy measure** window, click on the green application button. An **Easy measure** window is displayed.

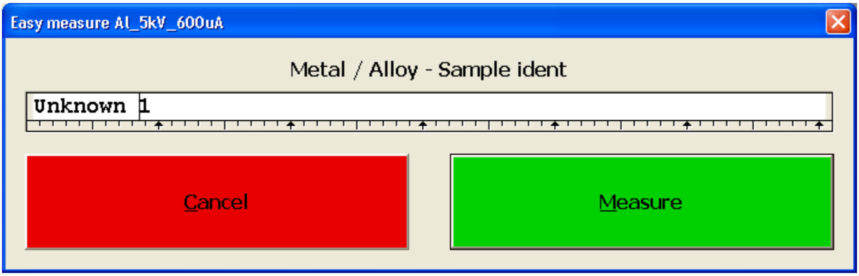


Figure 5.6 Easy Measure Window

- In the **Sample ident** field, enter the sample identity and click on the **Measure** button. The **Select changer position** window appears.
- In the **Select changer position** window, click on the position of the sample. The measurement will start automatically if the spectrometer is ready.

Every **Measure application** window that you now open created another green application button in the **Easy manage** window.

An application button remain active as long as the **Measure sample** window of that application is open or minimized. As soon as the **Measure sample** window is closed, the related application button disappears.

Up to 8 applications can be opened simultaneously in the **Easy measure** window.

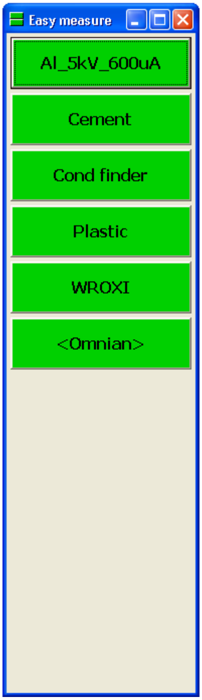
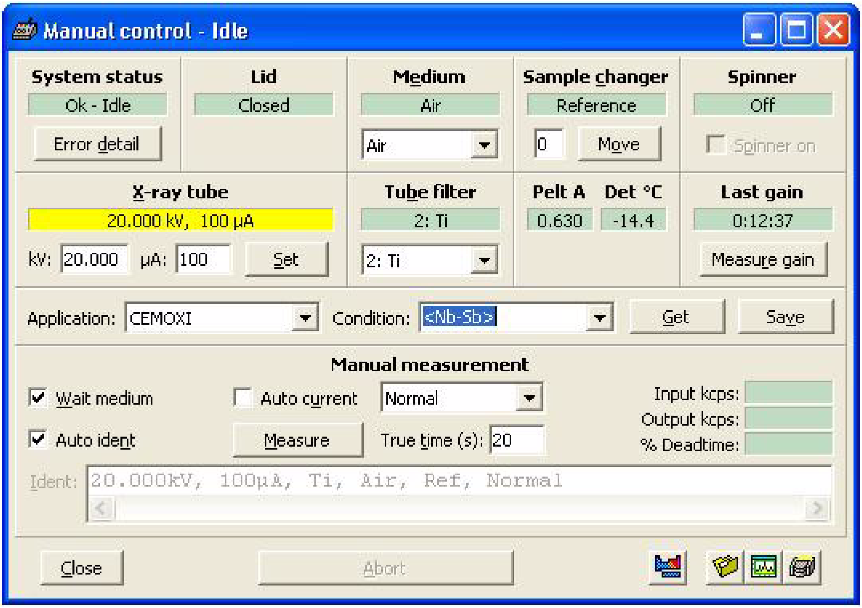


Figure 5.7 Easy Measure Window

5.3.3 Manual Measuring

The measurement of routine samples is normally carried out from a defined application, but samples can also be measured using the manual control function.

- Select **Measure** > **Manual control** or click on the button on the toolbar or in the **Online status** window, click on the button. The **Manual control** window is displayed.



System status: Ok - Idle

Lid: Closed

Medium: Air

Air

Sample changer: Reference

0

Spinner: Off

☐ Spinner on

X-ray tube: 20.000 kV, 100 µA

kV: 20.000 µA: 100

Tube filter: 2: Ti

2: Ti

Pelt A: 0.630

Det °C: -14.4

Last gain: 0:12:37

Application: CEMOXI

Condition: <Nb-Sb>

☑ Wait medium

☐ Auto current

Normal

☑ Auto ident

True time (s): 20

Input kcps:

Output kcps:

% Deadtime:

Ident: 20.000kV, 100µA, Ti, Air, Ref, Normal

Figure 5.8 Manual Control Window

- Place the sample in the sample changer.
- In the **Sample changer** frame of the **Manual control** window, enter the position of the sample and click on the **Move** button.
- Select or enter the measuring medium (**Medium**), measuring voltage (**kV**) and current (**µA**), filter (**Tube filter**), and measuring time (**True time**). You can also use the measurement conditions from an existing application by clicking on the **Get** button and selecting the name of the application.
- In the **Ident** field, enter a recognizable name for the measurement or check the **Auto ident** check box.
- Start the measurement by clicking on the **Measure** button in the **Manual measurement** frame.

To view the results of the manual measurement:

- Select **Results** > **Open results**. The **Open results** window is displayed.

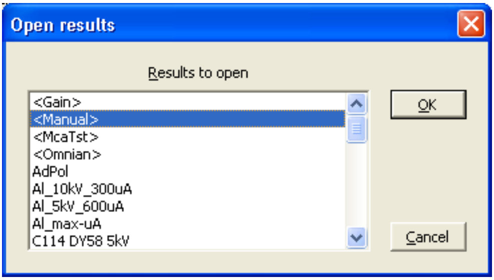
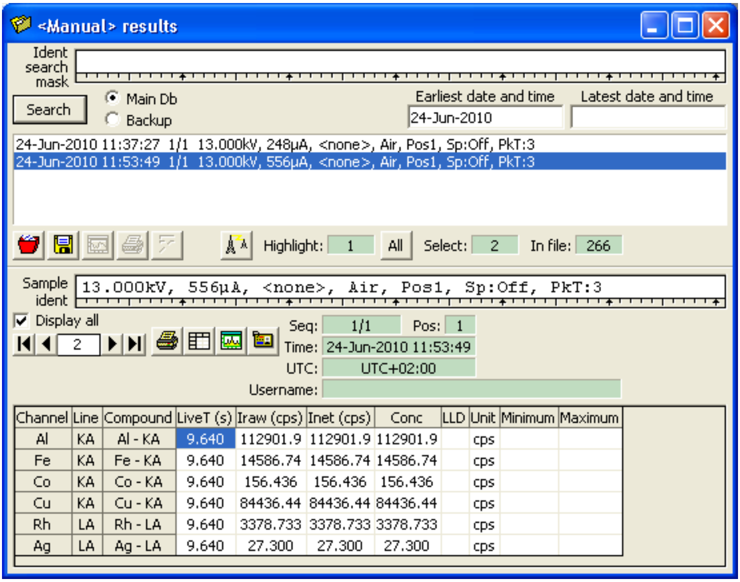


Figure 5.9 Open Results Window

- Select **<Manual>** and click on the **OK** button. The **<Manual> results** window is displayed.



Ident search mask:

● Main Db
○ Backup

Earliest date and time: 24-Jun-2010
Latest date and time:

```
24-Jun-2010 11:37:27  1/1  13.000kV, 248μA, <none>, Air, Pos1, Sp:Off, PkT:3
24-Jun-2010 11:53:49  1/1  13.000kV, 556μA, <none>, Air, Pos1, Sp:Off, PkT:3
```

Highlight: 1 Select: 2 In file: 266

Sample ident: 13.000kV, 556μA, <none>, Air, Pos1, Sp:Off, PkT:3

☑ Display all

2

Seq: 1/1 Pos: 1
Time: 24-Jun-2010 11:53:49
UTC: UTC+02:00
Username:

Channel	Line	Compound	LiveT (s)	Iraw (cps)	Inet (cps)	Conc	LLD	Unit	Minimum	Maximum
Al	KA	Al - KA	9.640	112901.9	112901.9	112901.9		cps		
Fe	KA	Fe - KA	9.640	14586.74	14586.74	14586.74		cps		
Co	KA	Co - KA	9.640	156.436	156.436	156.436		cps		
Cu	KA	Cu - KA	9.640	84436.44	84436.44	84436.44		cps		
Rh	LA	Rh - LA	9.640	3378.733	3378.733	3378.733		cps		
Ag	LA	Ag - LA	9.640	27.300	27.300	27.300		cps		

Figure 5.10 <Manual> Results Window

- Double-click on the sample of which you want to see the results.
- Click on the button.
- When you have a **Results spectra** window open, you can click on the button to show the elements and line groups.
- You can now proceed to handle the results as described in Chapter 7, or, if you want the software to check what elements are present (qualitative analysis), you can click on the button and the system will then highlight the elements present.

5.3.4 Measuring Large Samples

Measuring large samples is only possible for solid samples with a maximum height of 4.9 cm on an Epsilon 3 with a standard lid, and a maximum height of 10 cm on an Epsilon 3 XL with high lid.

To measure large samples:

- Turn off the HT.
- Open the lid.
- Remove the sample tray.
- Move the spinner arm back so that it is away from the measurement position.
- Position the large sample above the measurement position as shown in the figure below.
- Leave the lid open otherwise large sample mode cannot be selected.

NOTE: You cannot make automatic gain control measurements while in large sample mode.

Figure 5.11 Example of Large Sample Positioning

- Select **System > Enter "Large Sample" mode**. A message is displayed indicating to remove the sample tray and to move the spinner arm back so that it is away from the measurement position.
  NOTE: It is strongly advised to place a foil on top of the measuring station. Refer to the Epsilon 3 User's Guide.
- Click **OK** to continue.
- Close the lid.
- Switch on the HT.
- Measurements of large samples can now be carried out from an application (Section 5.3.1 and Section 5.3.2).
  Alternatively, individual samples can be measured from the **Manual control** window (Section 5.3.3).

To stop measuring large samples:

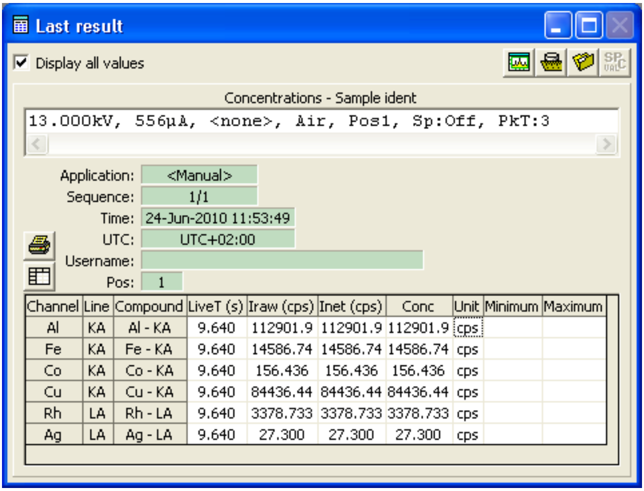
- Turn off the HT.
- Open the lid.

- Select **System > Exit “Large Sample” mode**. A message window is displayed indicating to remove any large sample, to replace the sample tray and to move the spinner arm forwards to engage the spinner cog wheel.
- Click **OK** to continue.
- Close the lid. The sample changer now moves to the reference position.
- Switch on the HT.

5.4 MEASUREMENT RESULTS

After a measurement has been completed, the results can be viewed in the **Last result** window and stored in the application database. The stored results can be recovered for further processing, see Chapter 7.

NOTE: The **Last result** window is one of the four windows that are displayed when the software is started. It cannot be closed, but it can be minimized.



☑ Display all values

Concentrations - Sample ident

13.000kV, 556µA, <none>, Air, Pos1, Sp:Off, PkT:3

Application: <Manual>
Sequence: 1/1
Time: 24-Jun-2010 11:53:49
UTC: UTC+02:00
Username:
Pos: 1

Channel	Line	Compound	LiveT (s)	Iraw (cps)	Inet (cps)	Conc	Unit	Minimum	Maximum
Al	KA	Al - KA	9.640	112901.9	112901.9	112901.9	cps		
Fe	KA	Fe - KA	9.640	14586.74	14586.74	14586.74	cps		
Co	KA	Co - KA	9.640	156.436	156.436	156.436	cps		
Cu	KA	Cu - KA	9.640	84436.44	84436.44	84436.44	cps		
Rh	LA	Rh - LA	9.640	3378.733	3378.733	3378.733	cps		
Ag	LA	Ag - LA	9.640	27.300	27.300	27.300	cps		

Figure 5.12 Last Result Window

The **Last result** window, as its name suggests, always contains the result of the last measurement carried out. It shows the measuring time, count rate (in cps) and concentration for each measured channel, as well as details of the sample and application.

Any concentration values that are outside the optional limits set in the **application** window, are displayed in red. This gives you an immediate visual indication of possible problems.

5.5 REPEAT MEASUREMENTS

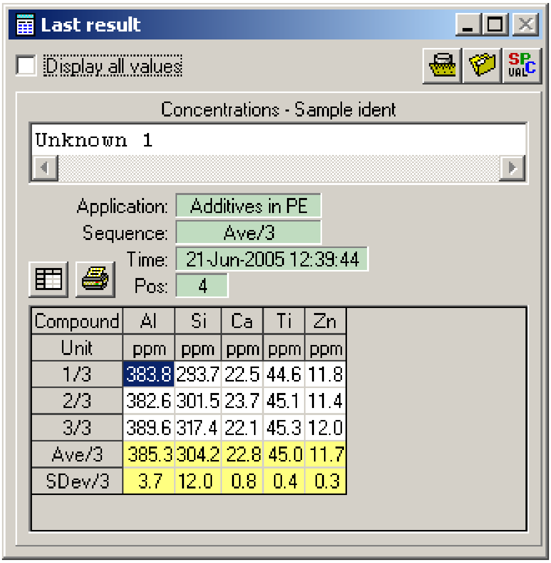
If the check box **Repeat Factor** is checked in the **application** window, the same sample is measured for the defined number of times. This is carried out automatically by the software, which shows the number of the current measurement in the measure window.

The field can be blank, “0” or a number between “1” and “9999”.

- If blank you must specify “0” or a non-zero number smaller than “9999” in the **Measure** window.
- If “0” the sample will be measured indefinitely (until aborted).
- If a number between “1” and “9999”, the sample will be measured the specified number of times.

The results of each measurement are stored separately. Additionally, for repeated measurements on one sample, the average and standard deviation results are calculated and stored.

When the measurement is complete, the **Last result** window shows the complete results for all repetitions and the average and standard deviation values.



Concentrations - Sample ident

Unknown 1

Application: Additives in PE
Sequence: Ave/3
Time: 21-Jun-2005 12:39:44
Pos: 4

Compound	Al	Si	Ca	Ti	Zn
Unit	ppm	ppm	ppm	ppm	ppm
1/3	383.8	293.7	22.5	44.6	11.8
2/3	382.6	301.5	23.7	45.1	11.4
3/3	389.6	317.4	22.1	45.3	12.0
Ave/3	385.3	304.2	22.8	45.0	11.7
SDev/3	3.7	12.0	0.8	0.4	0.3

Figure 5.13 Last Result Window

Chapter 6

Omnian Analysis

6.1 INTRODUCTION

The optional Omnian program can be used for characterization and analysis of unknown samples, or in situations where certified standards that match specific sample characteristics, are not available. Important applications include sample quantification, screening, failure analysis, as well as the comparison of different materials. Omnian can handle a wide variety of sample types such as solids, pressed powders, fused beads, loose powders and liquids.

Importantly, Omnian corrects for sample characteristics - thickness, volume and unmeasured ‘Dark-Matrix’ compounds, and adapts automatically to sample characteristics and associated matrix effects.

Omnian analysis uses fundamental parameters (FPs) to calculate the propagation of X-rays from the X-ray tube through the optical path to the detector. Observed intensities are compared with calculated intensities in order to give an estimate of the concentrations in an unknown sample.

This is done using an iterative method:

1. Assume a concentration
2. Predict the signal
3. Compare with measurement
4. Tune the concentrations
5. Return to predict the signal

The loop is repeated until the predicted signal matches the measured signal.

NOTE: It is essential for this method that it is known exactly what is happening in the optical path. All parameters must be known with at least the accuracy that is required for the concentrations. Parameters are tube spectrum, tube window thickness, propagation of X-rays in the measure chamber medium, thickness and composition of filters, etc.

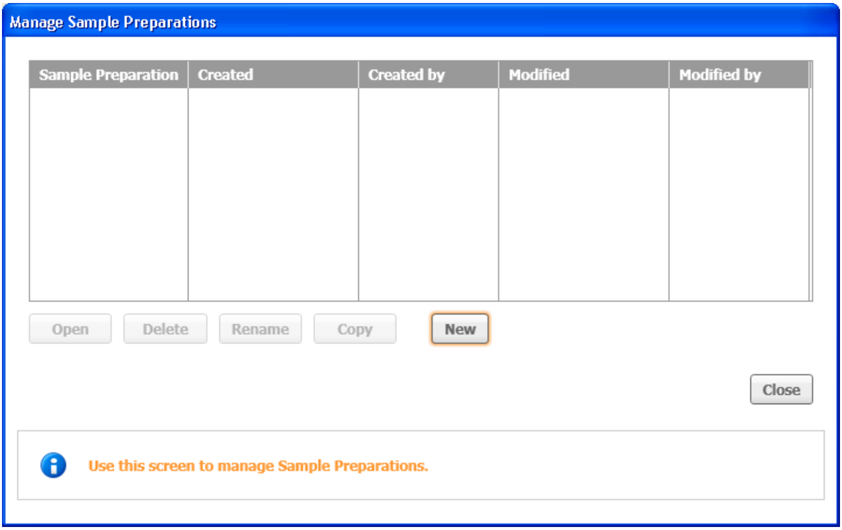
6.2 PREPARING OMNIAN MEASUREMENTS

Before the Omnian routine measurements can be performed, the following preparations are required:

1. Carrying out Omnian sample preparation (see Section 6.2.1)
2. Creating Omnian sample parameters (see Section 6.2.2)

6.2.1 Carrying Out Omnian Sample Preparation

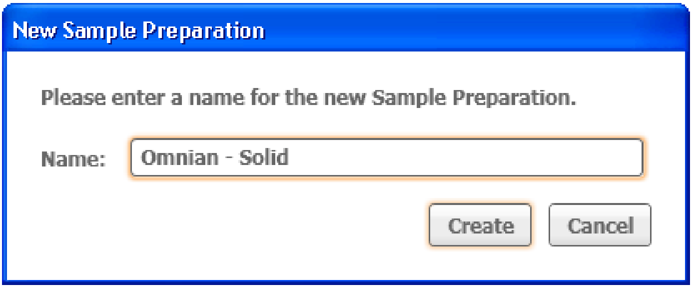
- Select **Omnian Setup** > **Manage sample preparations**. The **Measure Sample Preparations** window is displayed.



Use this screen to manage Sample Preparations.

Figure 6.1 Manage Sample Preparations Window

- Click on the **New** button. The **New Sample Preparations** window is displayed.



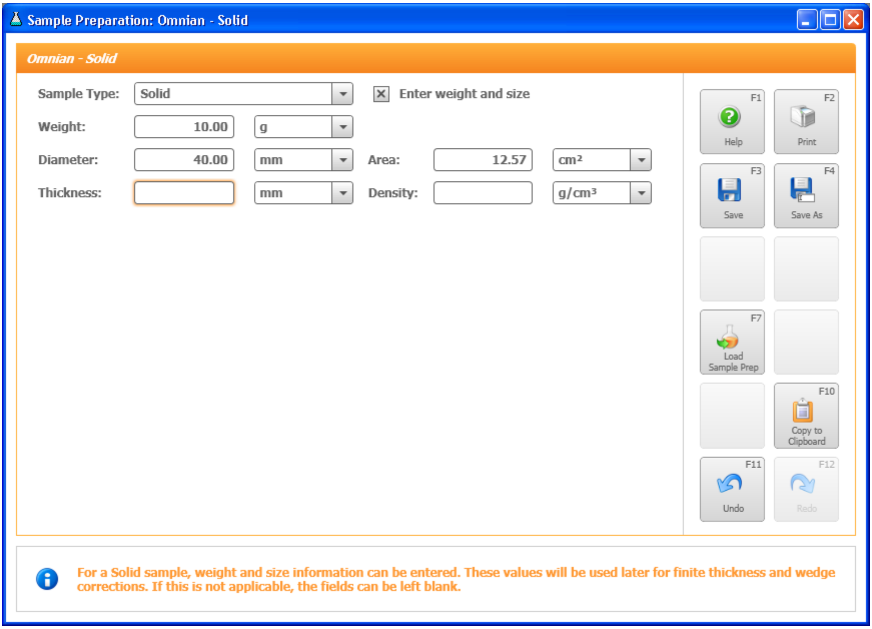
New Sample Preparation

Please enter a name for the new Sample Preparation.

Name: Omnian - Solid

Figure 6.2 New Sample Preparation Window

- In the **Name** field, enter a name (e.g. Omnian - Solid) for the new sample preparation and click on the **Create** button. The **Sample Preparation** window is displayed.



Sample Preparation: Omnian - Solid

Omnian - Solid

Sample Type: Solid

☒ Enter weight and size

Weight: 10.00 g

Diameter: 40.00 mm

Area: 12.57 cm²

Thickness: mm

Density: g/cm³

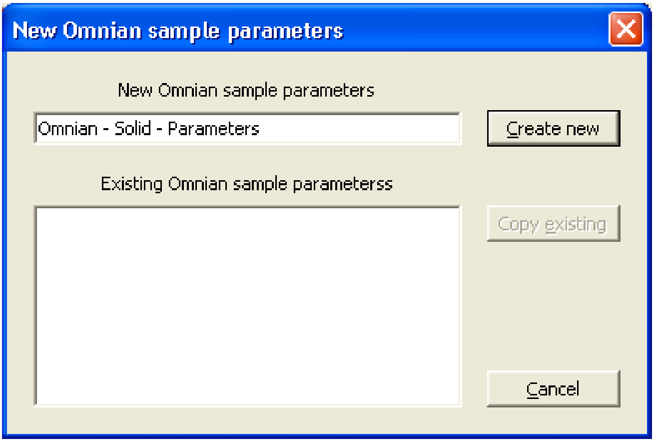
For a Solid sample, weight and size information can be entered. These values will be used later for finite thickness and wedge corrections. If this is not applicable, the fields can be left blank.

Figure 6.3 Sample Preparation Window

- In the **Sample Type** field, select **Solid**.
- Check the check box **Enter weight and size**.
- Enter the correct values in the fields **Weight** and **Diameter**. If finite thickness correction is needed, also enter the correct thickness of the sample in the **Thickness** field.
- Click on the **Save** button.
- Close the **Sample Preparation** window.

6.2.2 Creating Omnian Sample Parameters

- Select **Measure > New Omnian sample parameters**. The **New Omnian sample parameters** window is displayed.


New Omnian sample parameters

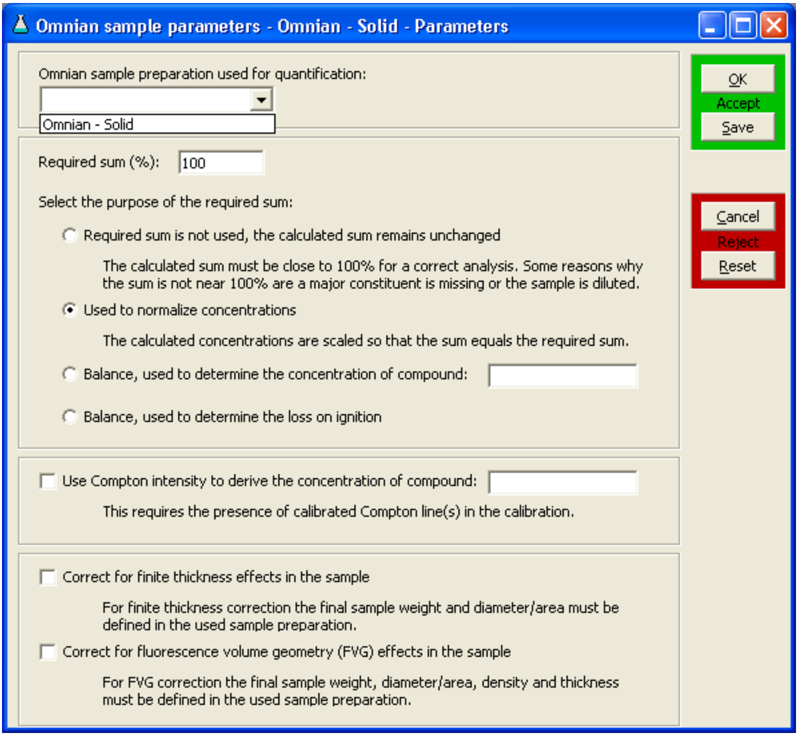
New Omnian sample parameters

Omnian - Solid - Parameters

Existing Omnian sample parameterss

Figure 6.4 New Omnian Sample Parameters Window

- In the **New Omnian sample parameters** field, enter a name (e.g. Omnian - Solid) for the sample parameters and click on the **Create new** button. The **Omnian sample parameters** window is displayed.



Omnian sample parameters - Omnian - Solid - Parameters

Omnian sample preparation used for quantification: Omnian - Solid

Required sum (%): 100

Select the purpose of the required sum:

○ Required sum is not used, the calculated sum remains unchanged

The calculated sum must be close to 100% for a correct analysis. Some reasons why the sum is not near 100% are a major constituent is missing or the sample is diluted.

● Used to normalize concentrations

The calculated concentrations are scaled so that the sum equals the required sum.

○ Balance, used to determine the concentration of compound:

○ Balance, used to determine the loss on ignition

☐ Use Compton intensity to derive the concentration of compound:

This requires the presence of calibrated Compton line(s) in the calibration.

☐ Correct for finite thickness effects in the sample

For finite thickness correction the final sample weight and diameter/area must be defined in the used sample preparation.

☐ Correct for fluorescence volume geometry (FVG) effects in the sample

For FVG correction the final sample weight, diameter/area, density and thickness must be defined in the used sample preparation.

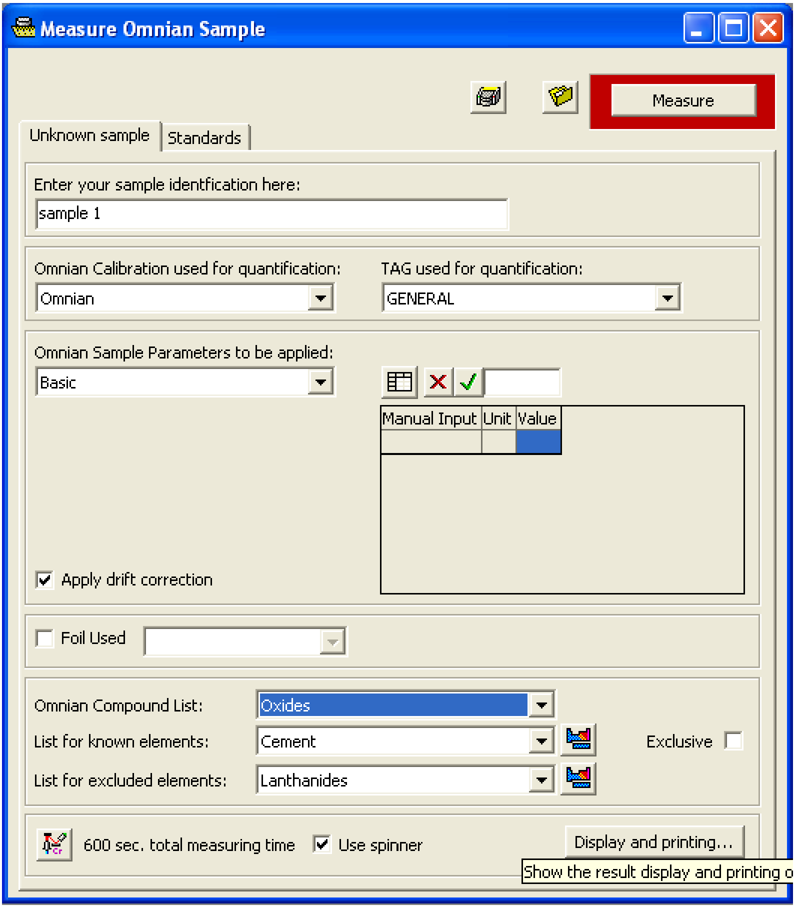
Figure 6.5 Omnian Sample Parameters Window

- In the **Omnian sample preparation used for quantification** field, select **Omnian - Solid**.
- Click on the **OK** button.

The Omnian measurement preparation is now completed and a routine Omnian measurement can be carried out (see Section 6.3).

6.3 PERFORMING A ROUTINE OMNIAN MEASUREMENT

- Select **Measure** > **Measure Omnian Sample**. The **Measure Omnian Sample** window is displayed.



Enter your sample identfication here: sample 1

Omnian Calibration used for quantification: Omnian

TAG used for quantification: GENERAL

Omnian Sample Parameters to be applied: Basic

☑ Apply drift correction

☐ Foil Used

Omnian Compound List: Oxides

List for known elements: Cement

Exclusive ☐

List for excluded elements: Lanthanides

600 sec. total measuring time

☑ Use spinner

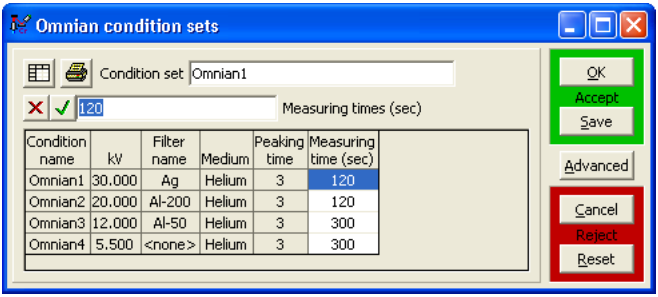
Figure 6.6 Measure Omnian Sample Window

If the sample you are going to measure has not been defined, enter the sample definition as follows:

- In the **Enter your sample identification here** field, enter the sample identification. Without this sample identification it is not possible to start the measurement.
- In the **Omnian Calibration used for quantification field**, select **Omnian**. The Omnian calibrations must be set up previously in **Omnian Setup > Manage Calibration**.
- In the **TAG used for quantification** field, select a TAG for the calibration. For Omnian the default TAG is **GENERAL**.
  Tags can be used as a keyword or term associated with or assigned to a piece of information used to steer the quantification of unknown routine samples. Tags can be set up in **Omnian Setup > Tags and additives**.
- In the **Omnian Sample Parameters to be applied** field, select an appropriate Omnian sample parameter.
- If drift correction is required, check the check box **Apply drift correction**.
- In the **Manual Input** grid, enter the correct values of the weights (e.g. from the sample and additives).
- When a foil is used (for example when measuring a liquid sample), check the **Foil Used** check box and select the foil that is used for the measurement. The thickness and density are filled in automatically.
- When applicable, select an **Omnian Compound List**. An Omnian compound lists must be previously created in **Omnian setup > Manage Compound Lists**.
- When applicable, select a **List for known elements** used for deconvolution. Click on the button to select the elements of interest.

To display the Omnian measuring conditions:

- Click on the button. The **Omnian condition sets** window is displayed.



Condition set Omnian1

120 Measuring times (sec)

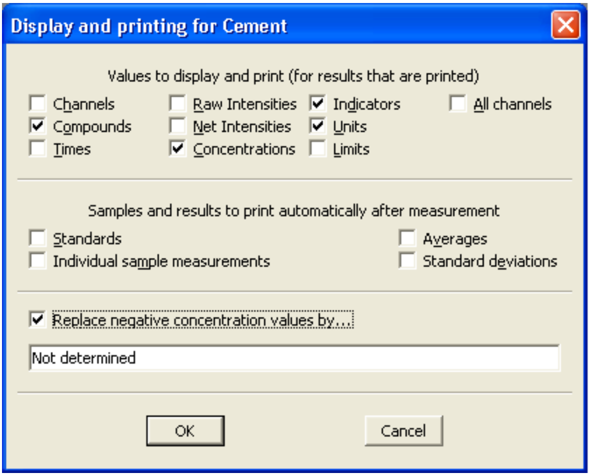
Condition name	kV	Filter name	Medium	Peaking time	Measuring time (sec)
Omnian1	30.000	Ag	Helium	3	120
Omnian2	20.000	Al-200	Helium	3	120
Omnian3	12.000	Al-50	Helium	3	300
Omnian4	5.500	<none>	Helium	3	300

Figure 6.7 Omnian Condition Sets Window

- If necessary, change the value of the **Measuring time** into the grid.
- If you agree with the conditions, click on the **OK** or **Save** button. Click on the **Cancel** or **Reset** button to cancel or abort the operation. The **Omnian condition sets** window is closed, returning to the **Measure Omnian sample** window.

To change the display and printing options:

- Click on the **Display and printing** button. The **Display and printing** window is displayed.



Display and printing for Cement

Values to display and print (for results that are printed)

☐ Channels
☑ Compounds
☐ Times
☐ Raw Intensities
☐ Net Intensities
☑ Concentrations
☑ Indicators
☑ Units
☐ Limits
☐ All channels

Samples and results to print automatically after measurement

☐ Standards
☐ Individual sample measurements
☐ Averages
☐ Standard deviations

☑ Replace negative concentration values by...

Not determined

Figure 6.8 Display and Printing Window

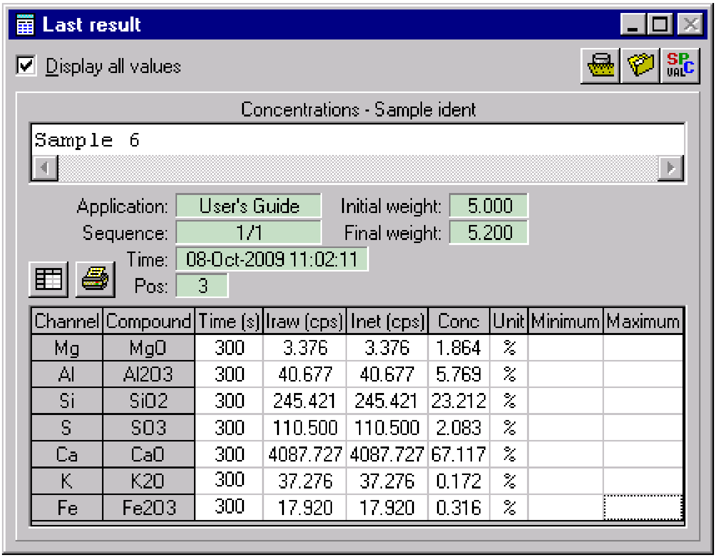
- Select the required options by clicking in the check boxes. See Section 4.7.2 for detailed information.

  NOTE: When using Omnian measurements, the field **Standards** is not applicable.
- Click on the **OK** button or press **Enter**.

To start the Omnian measurement:

- Ensure that the sample is available in the sample changer.
- Click on the **Measure** button.
- In the **Sample changer position** window, select the sample to be measured.
- If necessary, optimize deconvolution (see Section 4.8.4).

The measurement will be carried out and the results will be available in the **Last result** window.



☑ Display all values

Concentrations - Sample ident

Sample 6

Application: User's Guide
Initial weight: 5.000
Sequence: 1/1
Final weight: 5.200
Time: 08-Oct-2009 11:02:11
Pos: 3

Channel	Compound	Time (s)	Iraw (cps)	Inet (cps)	Conc	Unit	Minimum	Maximum
Mg	MgO	300	3.376	3.376	1.864	%		
Al	Al2O3	300	40.677	40.677	5.769	%		
Si	SiO2	300	245.421	245.421	23.212	%		
S	SO3	300	110.500	110.500	2.083	%		
Ca	CaO	300	4087.727	4087.727	67.117	%		
K	K2O	300	37.276	37.276	0.172	%		
Fe	Fe2O3	300	17.920	17.920	0.316	%		

Figure 6.9 Last Result Window

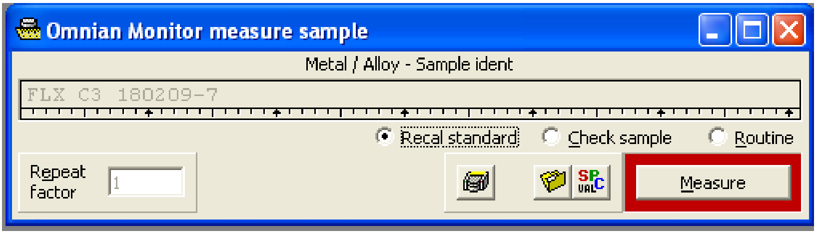
6.4 OMNIAN MONITOR

The application **Omnian Monitor** is included in the Omnian software package, together with a monitor sample FLX C3. If the pre-calibration is done in the factory, then the first measurement of the **Omnian Monitor** is done in the factory.

6.4.1 Measuring the Omnian Monitor Sample

- **Select Measure > Measure application.**
- In the **Measure application** window, select **Omnian Monitor**.

  In the **measure sample** window, select **Recal standard** as measurement type.



Metal / Alloy - Sample ident

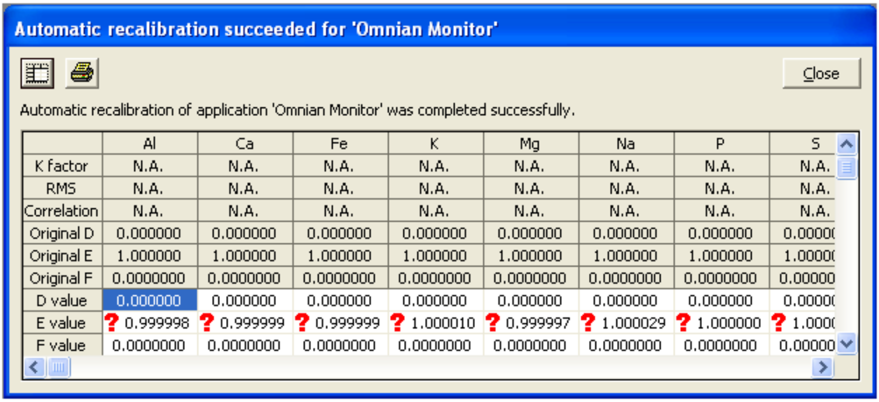
FLX C3 180209-7

● Recal standard ○ Check sample ○ Routine

Repeat factor: 1

Figure 6.10 Omnian Monitor Measure Sample Window

- Click on the **Measure** button to start the measurement.
- In the **Select changer position** window, click on the position of the Omnian monitor sample. The measurement will start automatically if the spectrometer is ready.
- After the measurement is finished the correction factors are automatically updated and the **Automatic recalibration succeeded** window is displayed.



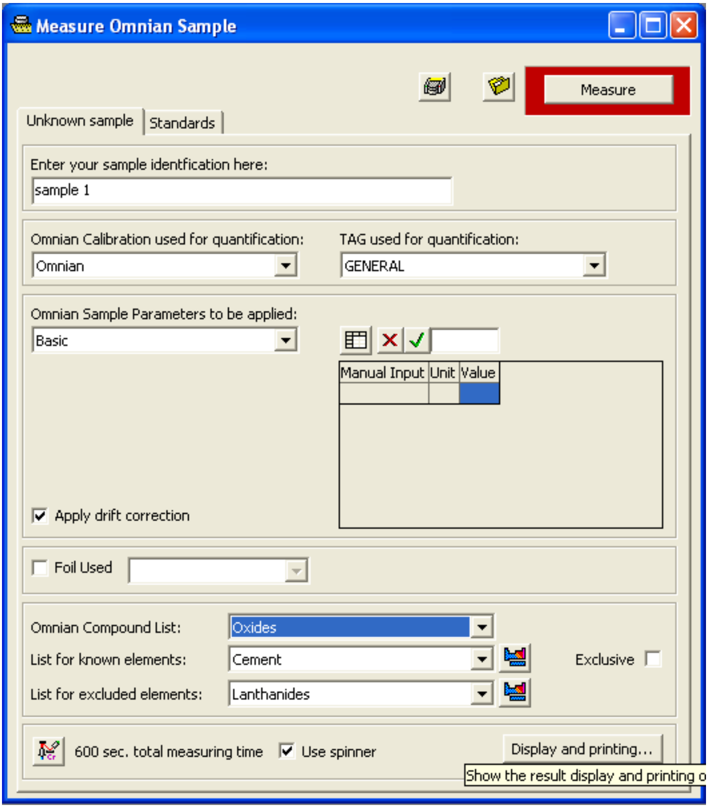
Automatic recalibration of application 'Omnian Monitor' was completed successfully.

	Al	Ca	Fe	K	Mg	Na	P	S
K factor	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
RMS	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
Correlation	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
Original D	0.000000	0.000000	0.000000	0.000000	0.000000	0.000000	0.000000	0.0000
Original E	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.000000	1.0000
Original F	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.00000
D value	0.000000	0.000000	0.000000	0.000000	0.000000	0.000000	0.000000	0.0000
E value	? 0.999998	? 0.999999	? 0.999999	? 1.000010	? 0.999997	? 1.000029	? 1.000000	? 1.000
F value	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.00000

- Click on the **Close** button.

6.4.2 Applying Drift Correction for the Omnian Sample

- In the **Measure Omnian Sample** window, check the check box **Apply drift correction** and click on the **Measure** button.



Enter your sample identfication here: sample 1

Omnian Calibration used for quantification: Omnian

TAG used for quantification: GENERAL

Omnian Sample Parameters to be applied: Basic

☑ Apply drift correction

☐ Foil Used

Omnian Compound List: Oxides

List for known elements: Cement

List for excluded elements: Lanthanides

Exclusive ☐

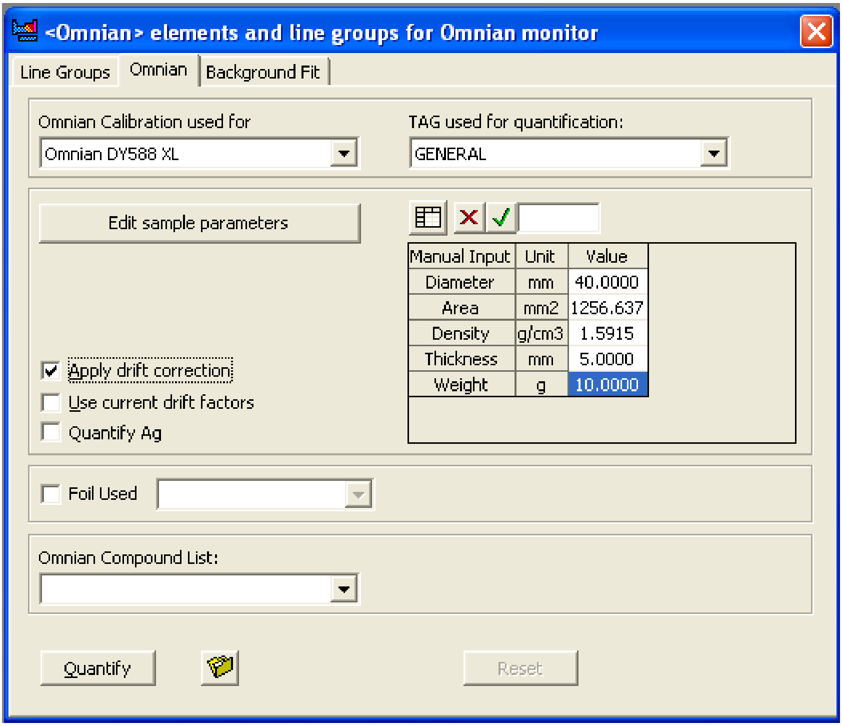
600 sec. total measuring time ☑ Use spinner

Show the result display and printing o

Figure 6.11 Measure Omnian Sample Window

- After the measurement, open the **results** window and select a measurement. Then click on the right mouse button to open the results pop-up and select **Display the spectra**.

- In the results spectra window, click on the button. The **elements and line groups** window is displayed.
- Select the tab **Omnian**.



Omnian Calibration used for: Omnian DY588 XL

TAG used for quantification: GENERAL

Manual Input	Unit	Value
Diameter	mm	40.0000
Area	mm2	1256.637
Density	g/cm3	1.5915
Thickness	mm	5.0000
Weight	g	10.0000

☑ Apply drift correction

☐ Use current drift factors

☐ Quantify Ag

☐ Foil Used

Omnian Compound List:

Figure 6.12 Elements and Line Groups Window - Omnian Tab

This window has two check boxes for drift correction:

Apply drift correction	Applies the drift correction factors stored with the result.
Use current drift factors	If checked, the software applies the current drift factors. This is useful in situations where the routine sample was measured without an up-to-date monitor. After the monitor is re-measured, the drift factors can be checked and the sample recalculated.

- Check the required drift correction check boxes and click on the **Quantify** button.

Chapter 7

Results Handling

7.1 INTRODUCTION

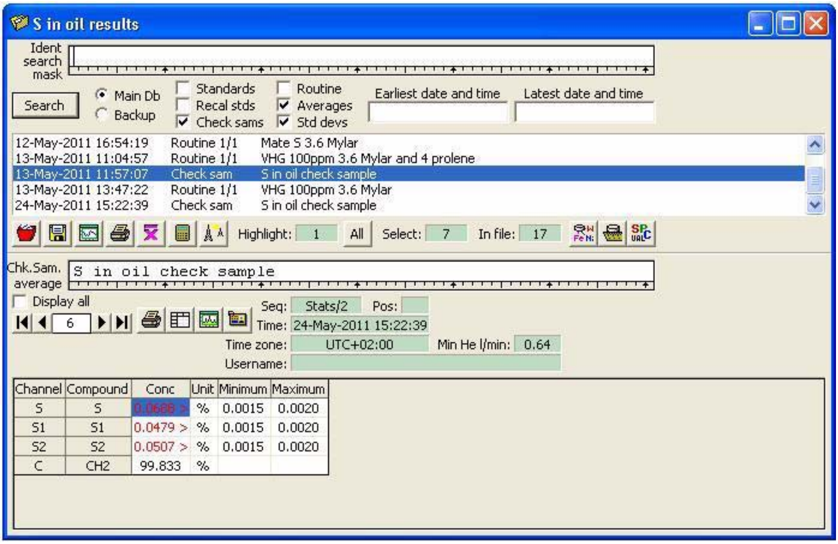
The results stored in the database can be accessed using the **results** window. The results for either the routine (‘Unknowns’) or ‘Standards’ samples can be displayed, backed up to an external disk (to make a backup database) or combined into reports. The results can also be shown graphically.

7.2 HANDLING APPLICATION RESULTS

7.2.1 Results Window

To open the results window:

- Select **Results** > **Open results** or click on the button in the navigation/control area of the **Application** window. The **Open results** window is displayed.
- Select the required application.



● Main Db ○ Backup

☐ Standards ☐ Recal stds ☑ Check sams

☐ Routine ☑ Averages ☑ Std devs

12-May-2011 16:54:19	Routine 1/1	Mate S 3.6 Mylar
13-May-2011 11:04:57	Routine 1/1	VHG 100ppm 3.6 Mylar and 4 prolene
13-May-2011 11:57:07	Check sam	S in oil check sample
13-May-2011 13:47:22	Routine 1/1	VHG 100ppm 3.6 Mylar
24-May-2011 15:22:39	Check sam	S in oil check sample

Highlight: 1 Select: 7 In file: 17

Chk.Sam. average: S in oil check sample

☐ Display all

6

Seq: Stats/2
Pos:
Time: 24-May-2011 15:22:39
Time zone: UTC+02:00
Min He l/min: 0.64
Username:

Channel	Compound	Conc	Unit	Minimum	Maximum
S	S	0.0688 >	%	0.0015	0.0020
S1	S1	0.0479 >	%	0.0015	0.0020
S2	S2	0.0507 >	%	0.0015	0.0020
C	CH2	99.833	%		

Figure 7.1 Results Window

- The upper half of the **results** window handles searching the results database, displaying the search results and what actions to carry out on the selected results. It also shows the number of results in the database selected and highlighted for processing.
- The lower half of the **results** window displays the results of the highlighted results, showing either the calculated average values for all the results or the standard deviations.

7.2.2 Searching Results

The fields in the upper half of the **results** window can be used to define a search of the database.

- Enter the search criteria:
  - Database: **Main Db** or **Backup**
  - Sample type: **Unknowns, Standards**, **Check sam** or **Routine**
  - Results type for repeat measurements: **Average** or **Std.Devs**
  - Sample ident as a text string: **Ident search mask**

    NOTE: The asterisk character “*” or question mark “?” can be used in the searches as a wild card to represent one or more characters.
  - Date and time limits: **Earliest date and time** and **Latest date and time**
- Click on the **Search** button.

7.2.3 Selecting and Handling Results

The search criteria that match the selected results in the application database are listed in the **results** window. Actions can be performed on the results using toolbar buttons or the right mouse button menu.

7.2.3.1 Using the toolbar

The toolbar on the upper half of the **results** window contains several fields and buttons for selecting and handling the results.

.

Table 7-1 Upper Half Results Window Fields and Buttons

Menu Item	Function
In file	Shows the number of entries in the database.
Select	Shows the number of results recovered with the defined search.
Highlight	Shows the number of recovered results that are highlighted.
All	Highlights all the results.
🖨	Prints the contents of the highlighted results.

7.2.3.2 Results Pop-up Menu

Clicking on the right mouse button in the **results** window opens the results pop-up menu, which contains the following items:.

Table 7-2 Results Pop-up Menu Items

Menu Item	Function
Export selected spectra	Exports the data of one spectrum into a text file, including the selected condition sets. Spectra are exported by default as MPS files. You can also export as SPE files. The MPS files have more information in the header. The **Export spectrum** window is displayed to specify the filename.
Transmit results	Transmits the highlighted results on-line or off-line. This menu item is only available when the check box **Transmission of results** in the **System parameters** window is checked. The sample results are written to the folder specified in the **System parameters** window. You can select to transmit results to a single file or to individual files. Selecting **Transmission of results** > **To a single file** will display the **Transmit Samples** window to enter the filename. Selecting **Transmission of results** > **Transmission to individual files** will create output files as specified in Section 3.2.6.

Table 7-2 Results Pop-up Menu Items (Continued)

Menu Item	Function
Display results	Displays all the elemental concentrations for each selected sample, including the average concentration and the standard deviation of the selected samples.
Export to file	Exports the results by default as TXT files. Exported results files can also be imported into office applications. The **Export spectrum** window is displayed to enter the filename.
Display selected spectra	Displays the **Results spectra** window of all highlighted samples.

7.2.4 Backing up the Database

Results from the database can be saved to another disk in order to backup important data or to transfer data from one PC to another.

- Select **System** > **Backup userdata**.
- The **Backup userdata** window is displayed showing the source folder.
- Select the destination folder and click on the **Backup** button.

7.2.5 Deleting Results

Any or all of the displayed results can be deleted in order to remove unwanted results from the storage device.

- In the **results** window, select the required results.
- Click on the 🗑 button. A message is displayed for confirmation.
- Click on the **Yes** button to delete the file.

7.2.6 Processing Results

The highlighted results can be processed and the results shown in the lower half of the **results** window. Two processing functions are available:

- Averaging
- Standard deviation

The averaging and standard deviation functions display a range of values as configured in the **Display and printing** window. This can include the average values or standard deviation values of measured count rates, corrected count rates and concentrations for all the measured channels, calculated over the highlighted results.

The calculated results shown in the grid can be printed by clicking on the 🖨 button, located immediately above the grid.

How the results are displayed varies slightly with the sample types:

- If needed, for unknown samples (**Unknowns**), check samples (**Check sam**) and routine samples (**Routine**), **Net count rate** can also be displayed.
- For standard samples (**Standards**), the known concentrations are shown in the **Conc** column.

7.2.6.1 Averaging

- Click on the $\bar{x}$ button to display the average values of the highlighted results.
- The calculated averages are displayed in the grid in the lower section of the **results** window.
  The **Seq** field indicates that the averaging process has been performed by the term **Ave/x**, where x is the number of results used in the calculation.

7.2.6.2 Standard Deviation

NOTE: More than one result must be highlighted before standard deviations can be calculated.

- Click on the $\bar{x}$ button to display the standard deviation values of the highlighted results.

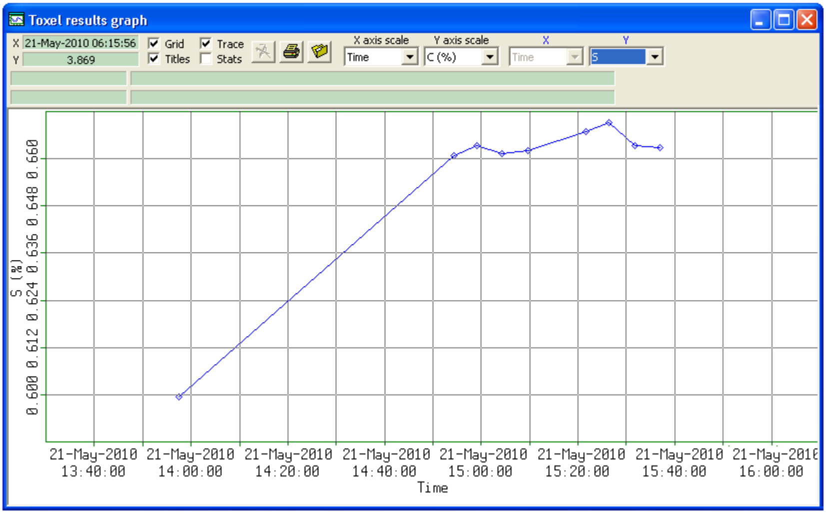
- The calculated standard deviations are displayed in the grid in the lower section of the **results** window.
  The **Seq** field indicates that the standard deviation process has been performed by the term **Sdev/x**, where x is the number of results used in the calculation.

7.2.7 Displaying Graphical Results

The selected results can be displayed graphically in a **results graph** window. The graph can also be printed out as part of a report.

- In the **results** window, select the required results.
- Click on the button. The **results graph** window is displayed.

7.2.7.1 Results Graph Window



Toxel results graph

X 21-May-2010 06:15:56
Y 3.869
☑ Grid ☑ Titles ☑ Trace ☐ Stats
X axis scale: Time
Y axis scale: C (%)
X: Time
Y: S

Figure 7.2 Results Graph Window

A number of fields and check boxes are available for customizing the display. In addition, a number of information fields are presented, shown with a green background..

Table 7-3 Results Graph Window Fields and Buttons

Menu Item	Function
X	Shows the Y axis position of the mouse cursor.The unit is the same as the unit of the scale.
Y	Shows the Y axis position of the mouse cursor.The unit is the same as the unit of the scale.
Sample ident field	Clicking with the mouse anywhere over the graph causes the nearest data point to be selected (color changes from blue to green). The sample ident of the results file name corresponding to this data point is also displayed in the information fields directly above the graph.
Grid	Superimposes a grid over the graph.
Titles	Displays the names of the axis in the graph area. This information is identical to that shown in the **X and Y axis scale fields**.
Trace	Draws a line joining all the data points in the graph, in the order they appear along the X axis.
Stats	Draws the statistical lines onto the graph.
X axis scale	Defines which units are used for the X axis: ◆ Time (default): date ant time the samples were measured ◆ C (%): concentration in % ◆ C (ppm): concentration in ppm ◆ I (cps): count rate in cps
Y axis scale	Defines which units are used for the Y axis: ◆ C (%): concentration in % ◆ C (ppm): concentration in ppm ◆ I (cps): count rate in cps

Table 7-3 Results Graph Window Fields and Buttons (Continued)

Menu Item	Function
X (drop-down)	Directly displays the X axis selection.
Y (drop-down)	Allows you to select which measured channel to display. Additionally, if **Y axis scale** is **I (cps)**, you can select to the following items or this axis: ◆ Raw ◆ Net ◆ Corrected

7.2.7.2 Handling Graph Results

To select a result:

- Click on or near a result in the graph. The selected result is shown in a different color than the other results and the measurement time and identification fields of the selected result will be filled.

To show a result:

- Double-click on or near a result in the graph. The selected result is displayed in the periodic table of the **elements and line groups** window, which will automatically be displayed.

To edit a result:

- Click on the right mouse button in the **results graph** window to open the edit pop-up menu, which contains the following items:

Table 7-4 Edit Menu Items

Menu Item	Function
Select all	This item is disabled for spectra and graphs.

Table 7-4 Edit Menu Items (Continued)

Menu Item	Function
Copy to clipboard	Copies the graph to the clipboard.
Print	Prints the current graph.
Export data points	Exports the data of one result graph into a text file. The **Export data points** window is displayed to specify the file name. Data points are exported by default as EDP files. These files can also be imported into spreadsheet applications.
Zoom	Zooms in on the graph.
Unzoom	Zooms out to the original size of the graph, with all the points fitted on the graph.

7.2.8 Transmitting Results

Results from the application database can be transmitted to a folder on your hard drive in order to import data or to transfer data into other applications.

Transmitting the highlighted results can only be done off-line. This function is only available when the check box **Transmission of results** in the **System parameters** window is checked.

The results are written to the folder specified in the **System parameters** window. See also Section 4.7.1. The folder must be an existing folder on your hard drive. The file name will be the same as the sample result date (XYYYYMMDDHHMMSS with the extension QAN). The measurement results can be used by other applications for further processing.

To transmit results:

- In the **results** window, select the required highlighted results.
- Click on the button.
  If you have selected more than one result, a file transfer confirmation message is displayed asking if you want to confirm each transfer separately.

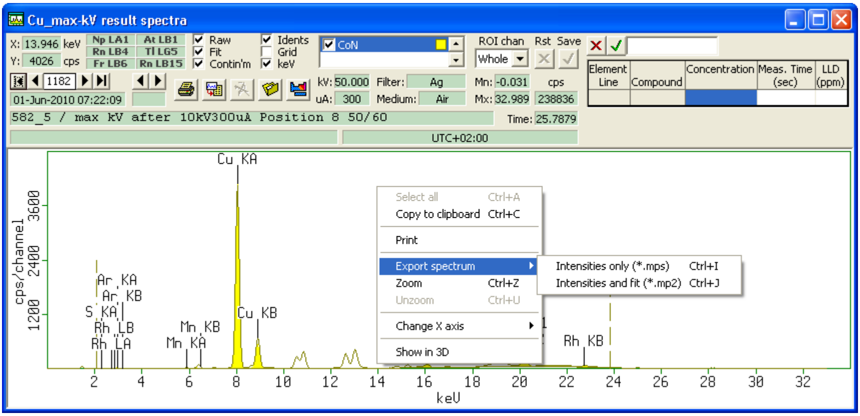
- Click on the **Yes** button if you wish to confirm each transmission, click on the **No** button to transmit all results automatically.
- When you clicked on the **Yes** button, a message window is displayed before each transmission. Click on the **Yes** button to transmit the results of the sample.

7.3 HANDLING SPECTRA

If you have a sample that you have measured, but you do not know what elements are present in the sample, you can use the quantitative analysis capabilities of the Epsilon 3 software to find out what is present in your sample.

To open the result spectra window:

- Select **Results > Open results** to open the **Result spectra** window
- Select the results you require and click on the 📈 button. The **result spectra** window opens showing the spectra of the selected measurement.



Cu_max-kV result spectra

X: 13.946 keV
Y: 4026 cps
1182
01-Jun-2010 07:22:09
kV: 50.000 Filter: Ag
uA: 300 Medium: Air
Mn: -0.031 cps
Mx: 32.989 238836
582_5 / max kV after 10kV300uA Position 8 50/60
Time: 25.7879
UTC+02:00
ROI chan: Whole

Element Line	Compound	Concentration	Meas. Time (sec)	LLD (ppm)

Figure 7.3 Result Spectra Window

To edit the result spectra window:

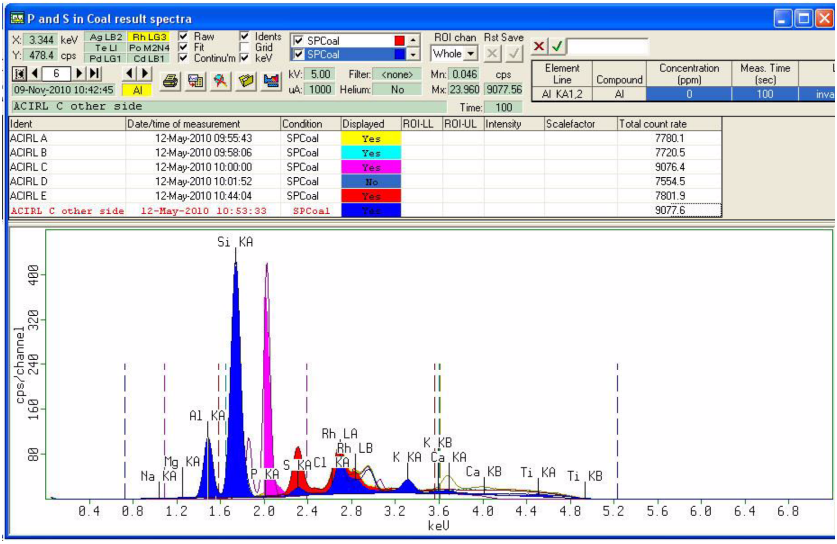
- Click on the right mouse button in the **result spectra** window to open the edit pop-up menu, which contains the following items:.

Table 7-5 Edit Menu Items

Menu Item	Function
Select all	This item is disabled for spectra and graphs.
Copy to clipboard	Copies the graph to the clipboard.
Print	Prints the current graph.
Export spectrum	Exports the data of one spectrum into a text file including the selected condition sets. The **Export spectrum** window is displayed to specify the file name. Spectra are exported by default as MPS files. You can also export as SPE files. The MPS files have more information in the header. The importing of result spectra can be used as offline measurements in the Epsilon 3 software. Exported spectra files can also be imported into spreadsheet applications.
Zoom	Zooms in on the graph.
Unzoom	Zooms out to the original size of the graph, with all the points fitted on the graph.
Change X axis	Defines the scale used for the X axis.
Show data for multiple spectra	Shows a grid with information of all selected spectra.
Remove selected sample	Removes the selected sample from the graph.
Intensity offset	Increases or decreases an offset for all spectra.You can also reset the offset to its original value.
Show in 3D	Shows the spectra in a three dimensional view.

To view multiple spectra in the results spectra window:

- In the **Edit** menu, click on **Show data for multiple spectra** to view all selected spectra together in the **results spectra** window.



X: 3.344 keV
Y: 478.4 cps
☑ SPCoal
☑ SPCoal
ROI chan: Whole
09-Nov-2010 10:42:45
Al
kV: 5.00
uA: 1000
Filter: <none>
Helium: No
Mn: 0.046
Mx: 23.960
cps 9077.56
Time: 100
ACIRL C other side

Element Line	Compound	Concentration (ppm)	Meas. Time (sec)
Al KA1,2	Al	0	100

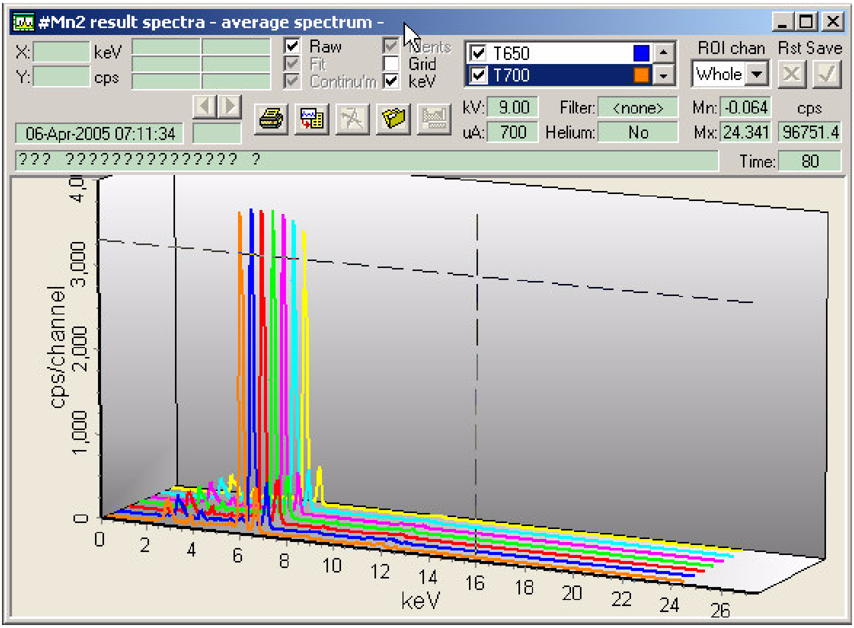
Ident	Date/time of measurement	Condition	Displayed	ROI-LL	ROI-UL	Intensity	Scalefactor	Total count rate
ACIRL A	12-May-2010 09:55:43	SPCoal	Yes					7780.1
ACIRL B	12-May-2010 09:58:06	SPCoal	Yes					7720.5
ACIRL C	12-May-2010 10:00:00	SPCoal	Yes					9076.4
ACIRL D	12-May-2010 10:01:52	SPCoal	No					7554.5
ACIRL E	12-May-2010 10:44:04	SPCoal	Yes					7801.9
ACIRL C other side	12-May-2010 10:53:33	SPCoal	Yes					9077.6

Figure 7.4 Results Spectra Window with Multi Spectra Data

NOTE: It is possible to adjust the ROI on the **results spectra** window by pressing the **Shift** key and the left mouse button at the same time.

To view the spectra in 3D:

- In the **Edit** menu, click on **Show in 3D** to view the 3D representation of the results spectra:



#Mn2 result spectra - average spectrum -

☑ Raw
☑ Fit
☑ Continu'm
☑ Elements
☐ Grid
☑ keV

☑ T650
☑ T700

ROI chan: Whole

06-Apr-2005 07:11:34

kV: 9.00
uA: 700
Filter: <none>
Helium: No
Mn: -0.064
Mx: 24.341
cps: 96751.4
Time: 80

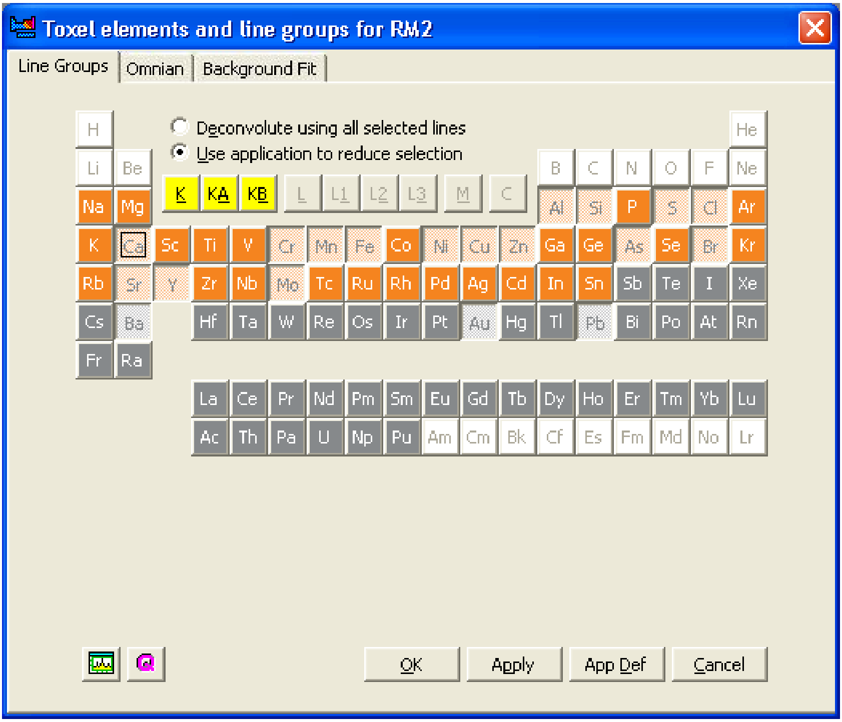
??? ??????????????? ?

Figure 7.5 3D Results Spectra Window

NOTE: The Graph ROI area is not shown in the **3D results spectra** window.

To view the elements present:

- In the **results spectra** form, click on the button to show the **elements and line groups** window.
- Click on the button to highlight the elements that are present in the **elements and line groups** window.



Toxel elements and line groups for RM2

○ Deconvolute using all selected lines

● Use application to reduce selection

Figure 7.6 : Elements and Line Groups Window showing Elements Present

Chapter 8

Statistical Process Control (SPC)

8.1 INTRODUCTION

Statistical Process Control (SPC) enables you to monitor the stability of a process. SPC continuously checks the measurement results and alerts you to possible problems. This continuous monitoring of the results not only spots individual results that are outside the limits, but also recognizes long term trends in the measuring results, that are unlikely to be spotted by the operator. This assists you in taking corrective action quickly and efficiently.

A process is monitored by selecting a number of values that can be measured and that should remain constant over a long time period. Each of these values is analyzed repeatedly and checked for certain trends and limits. Each particular value will have its own limits. The trends looked for will normally be similar for all the values used to monitor a particular process.

The spectrometer is designed to measure concentrations of elements or compounds in samples. You will therefore normally use concentration values as the input to the SPC calculations. But, you can use the measured intensity values instead of the concentrations.

SPC must be set up for each application, if required. When the SPC protocol is initiated for an application, each set of results is sent to the SPC module. Initially, the results are stored in the SPC data file for that application until there are enough results to establish the limits and constraints. Then all new results are checked against the SPC limits and constraints. If a limit is violated, a warning is displayed immediately.

8.2 TERMS AND DEFINITIONS FOR SPC

Table 8-1 : SPC Terms and Definitions

Term	Explanation
Individual X-values	Each measurement result is treated individually as an X (average) value. It is not possible to use R (range) values with individual data values.
Groups	The sample measurement results are split into data groups for analysis. These data groups may be between 2 and 24 samples long and may or may not overlap, as required. When a data group is completed, new X- and R-values are produced which can be analyzed.

Table 8-1 : SPC Terms and Definitions (Continued)

Term	Explanation
X-values	These are the averages of the samples in each data group. This can also be used for individual samples.
R-values	These are the ranges of the samples in each data group. The R-value for a group is the maximum data value minus the minimum data value in that group.
Control groups	These are the data groups of the initial measurement results stored to calculate the limits and constraints for SPC.
Control limits	These are calculated from the control groups and give the allowed ranges for the X- and R-values, that are produced as subsequent samples are measured. The control limits cannot be calculated until sufficient samples have been measured to generate the required number of control groups.
Sigma	This is the standard deviation values used for the control limits. The values are normally estimated by applying a simple formula to the average of the R-values in the control groups.
Real Sigma	Sigma values are calculated by finding the standard deviations for the X- and R-values in the control groups.
Limit checks	Test that each individual sample or group falls within the defined X-and R-values limits (UCL and LCL) for that check.
LCL	Lower Control Limit. This is the lowest permitted value for that limit check. It is set three sigma below the calculated mean value.
UCL	Upper Control Limit. This is the highest permitted value for that limit check. It is set three sigma above the calculated mean value.
Maximum sequence	These checks count the current sequences of X-values or R-values, which are continuously increasing/decreasing or above/below the mean value. The length of any such sequence is compared to the maximum permitted length, set on the **SPC setup** window.

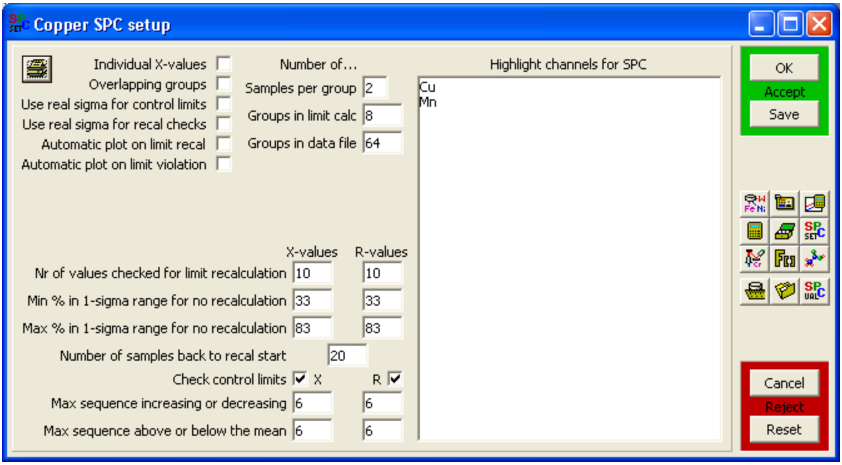
8.3 SETTING UP SPC

The **SPC setup** window can only be accessed from the **application** windows.

8.3.1 SPC Setup Window

To open the SPC setup window:

- In the navigation/control area of the **application** window, click on the SPC SET button. The **SPC setup** window is opened.



Copper SPC setup

☐ Individual X-values
☐ Overlapping groups
☐ Use real sigma for control limits
☐ Use real sigma for recal checks
☐ Automatic plot on limit recal
☐ Automatic plot on limit violation

Number of...
Samples per group 2
Groups in limit calc 8
Groups in data file 64

Highlight channels for SPC
Cu
Mn

	X-values	R-values
Nr of values checked for limit recalculation	10	10
Min % in 1-sigma range for no recalculation	33	33
Max % in 1-sigma range for no recalculation	83	83
Number of samples back to recal start	20	
Check control limits	☑ X	R ☑
Max sequence increasing or decreasing	6	6
Max sequence above or below the mean	6	6

Figure 8.1 SPC Setup Window

The fields in the **SPC setup** window allow you to define all the parameters for the SPC checks:

Table 8-2 : SPC Setup Window Fields

Field	Description
Individual X-values	If checked, each X-value is one sample result. If cleared, each X-value is the average of a group of sample results.
Overlapping groups	If checked, the groups of samples overlap (each group starts one sample after the previous group started). If cleared, the groups of samples do not overlap (a new group starts directly after the previous group).
Number of Samples per group	Specifies the group size between 2 and 24.
Number of Groups in limit calc	Specifies the number of groups over which the SPC limits are calculated.
Number of Groups in data file	Specifies the total number of groups (or individual samples) stored in the SPC data file.
Use real sigma for control limits	If checked, the real standard deviations of the X- and R-values to set the control limits are calculated. If cleared, estimated sigma values are used.
Use real sigma for recal checks	If checked, the real standard deviations of the X- and R-values are used when doing recalibration checks. If cleared, the estimated standard deviations of the X- and R-values are used when doing recalibration checks.
Automatic plot on limit recal	Plots the final X/R chart automatically before the recalculation.
Automatic plot on limit violation	Plots an X/R chart automatically when one of the checks fails for a channel.

Table 8-2 : SPC Setup Window Fields (Continued)

Field	Description
Nr of values checked for limit recalculation (X-values and R-values)	A record is kept of the number of X-values set in this field. If the percentage of these X-values within 1-sigma of the average is not within the defined range, the SPC limits are recalculated, providing no SPC limits have been violated. If this number is left blank, a record is kept of all X-values, but no limit recalculation is done.
Min % in 1-sigma range for no recalculation (X-values and R-values)	Specifies the lower limit for the allowed percentage of X-values within 1-sigma of the average.
Max % in 1-sigma range for no recalculation (X-values and R-values)	Specifies the upper limit for the allowable percentage of X-values within 1-sigma of the average.
Number of samples back to recal start	Sets the offset from the last sample to the first sample to be used in a limit recalculation. This offset must cover enough samples for SPC limit calculation and must not exceed the number of samples in the file.
Check control limits (X and R)	If checked, the system checks that the X and/or R are within the control limits.
Max sequence increasing or decreasing	Sets the number of sequentially increasing or decreasing X-values that generate a warning message. If this is set to zero, no check is made.
Max sequence above or below the mean	Sets the number of sequential X-values above or below the average that generate a warning message. If this is set to zero, no check is made.

NOTE: Recalculation can be disabled by leaving the field **Number of samples per group** blank.

8.3.2 Selecting Channels for SPC

The measuring channels in the application to which you wish to apply SPC can be individually selected. In the **SPC setup** window, all the channels for the application are listed in the field **Highlight channels for SPC**. If only the channel name is shown against a white background, the SPC check for that channel is disabled. This is the default situation.

To select a channel for SPC:

- In the **Highlight channels for SPC** field, click on the channel name. The name is highlighted and the value being checked is shown.
- Clicking further on the channel name takes you through the parameters. Figure 8.1 shows all the different parameters for which values can be checked.

8.3.3 Initializing the SPC Data File

After setting the parameters and selecting the required channels, you must save the changes and initialize the SPC data file. This enables the SPC module, which then collects all measurement results in the data file.

To initialize the SPC data file for that application:

- In the navigation/control area of the **application** window, click on the **Save** or **OK** buttons to save the new parameters and settings. A message is displayed informing you that the data file will be initialized.

  NOTE: If you have entered a parameter value outside the permitted range, or entered a value that causes another field to be outside its permitted range, a warning message is displayed to inform you. Clicking on the **OK** button will highlight the affected check box.
- Click on the **OK** button to start the initialization.

The SPC module now collects measurement results in the data file. When sufficient results have been collected the SPC limits are calculated for all selected channels.

8.4 CALCULATING SPC LIMITS

The SPC limits are calculated for each channel over a set number of values. The number of values used for limit calculation is set in the field **Nr of values checked for limit recalculation** in the **SPC setup** window.

The total number of samples used in limit calculation depends on the number of groups, group size and whether or not the groups overlap.

The SPC limits are calculated as follows:

- The averages of the X- and R-values are calculated. These values are Xav and Rav.
- The standard deviations of the X- and R-values are calculated. These standard deviations may be either the actual standard deviations or estimated standard deviations based on the average of the R-values, Rav. The standard deviations calculated here are used for the various checks involving sigma factors. The choice between real and estimated standard deviations for sigma checks is made in the **SPC setup** window.
- The control limits for the X- and R-values are calculated. These values are LCL(X), UCL(X), LCL(R) and UCL(R). These are set to be plus and minus 3-sigma from the averages. Again, actual or estimated standard deviations may be used to calculate these limits.

  NOTE: It is normal to use the estimated standard deviations to calculate the control limits. But, you should use the same type of sigma calculations (real or estimated) for both the recalculation checks and for the control limits.

8.4.1 Initial Limit Calculation

Samples are accumulated in the SPC data file by measuring samples. As soon as enough samples are present in the data file, the SPC limits are calculated for all selected channels. The first sample in the data file marks the start of the first calculation group.

IMPORTANT: Many of the SPC functions are not available until enough samples are present in the data file and the SPC limits have been calculated.

8.4.2 Automatic Limit Recalculation

Limit recalculation (Recal) will be automatically carried out for certain channels when the percentage of X- or R-values within 1-sigma of the average is not within the range specified in the **SPC setup** window.

When automatic recalculation occurs, the first sample in the control group is chosen by counting back through the stored results, by the number of samples set in the field **Number of samples back to recal start**, starting from the last sample.

NOTE: Limit recalculation of a channel is only possible if none of the SPC limits for that channel have been violated.

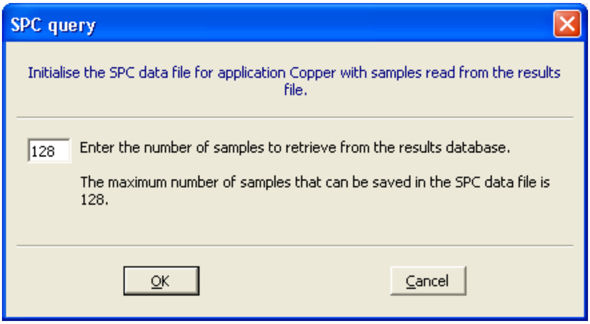
8.4.3 Manual Limit Recalculation

If the SPC limits are accurate and the results are normally distributed, then approximately two thirds of the X- and R-values should be within 1-sigma of their respective averages. If this is not the case, the limits should be recalculated.

You can manually recalculate the limits in the **SPC values** or **SPC counters** window.

To recalculate the limits:

- In the **SPC Values** or **SPC Counters** window, select the measuring channels you wish to recalculate in the grid.
- Click on the **Recal** button. An SPC message is displayed asking for the number of samples to use in the recalculation. The minimum number of samples required and the number of available files in the data file are shown in the window.



SPC query

Initialise the SPC data file for application Copper with samples read from the results file.

128 Enter the number of samples to retrieve from the results database.

The maximum number of samples that can be saved in the SPC data file is 128.

Figure 8.2 SPC Message Window

- Enter the number of samples to use.
- Click on the **OK** button. The SPC module recalculates the limits using the number of files you have specified.

8.5 CHECKING SPC LIMITS

The SPC limit checks can be carried out on either or both of the X- and R-values. As sample results are received, X- and R-values are produced. These values depend on the group size, whether the groups overlap and whether the X-values are to be taken individually.

If X-values are to be taken individually, then an X-value is produced by every sample and the X-value is simply the result for the channel in question. If X-values are not taken individually, then an X-value is produced as each group of samples is completed and the X-value is the average over the group of the results for the channel in question.

An R-value is produced as each group of samples is completed and the R-value is the range over the group of the results for the channel in question (highest result in group minus lowest result in group).

If the groups overlap, then each group starts one sample after the start of the previous group and ends one sample after the end of the previous group. This means that every sample marks the end of a group and so X- and R-values are produced for every sample. In effect, the X-values are a moving average and the R-values are a moving range.

If the groups do not overlap, then each group starts one sample after the end of the previous group. This means that if the group size is N, then every Nth sample marks the end of a group and so X- and R-values are produced after every Nth sample.

X-values limits are only checked when a new X-value is produced, and in the same way, R-values limits are only checked when a new R-value is produced. The next sections describe the checks on X- and R-values. If any limit is violated, a warning message is displayed.

8.5.1 Value Out of Limits

X-values are checked to see if they are within the control limits LCL(X) and UCL(X). R-values are checked to see if they are within the control limits LCL(R) and UCL(R).

NOTE: In many cases, LCL(R) is less than zero and is not checked because no R-value can be less than zero.

This check can be turned on and off individually for X- and R-values by checking or clearing the corresponding **Check control limits** check boxes.

8.5.2 Sequence of Increasing or Decreasing Values

The last few X- and R-values are checked to see if they have formed an increasing or decreasing series. The number of X- and R-values to be used for this check can be set in the field **Max sequence increasing or decreasing**. If the number to check is set to zero, the check is disabled.

8.5.3 Sequence of Values Above or Below the Average

The last few X- and R-values are checked to see if they are either all above the average or all below the average. The number of X- and R-values to be used for this check can be set in the field **Max sequence above or below the mean**. If the number to check is set to zero, the check is disabled.

8.6 SPC VALUES

The results values, counters and the warnings can all be viewed in the **SPC values** windows.

Three **SPC values** windows are available:

- **SPC Values**
- **SPC Counters**
- **SPC Warnings**

You can select which window to display by clicking on the related button at the top of the **SPC values** window.

The SPC module can be re-initialized from each of the three **SPC values** windows and channels can be recalculated from both the **SCP Values** and from the **SPC Counters** window.

The results for one or more channels can also be displayed graphically from these windows.

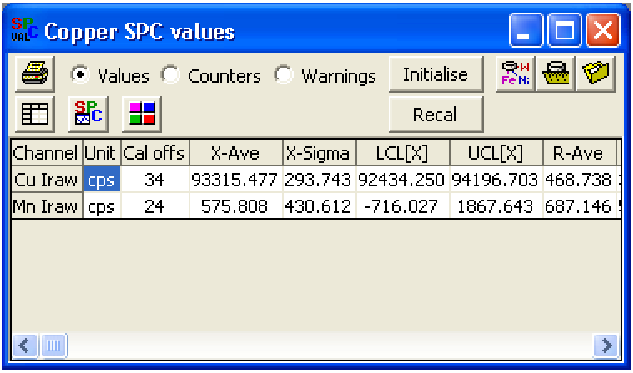
To open the SPC values windows:

- Select **SPC** > **Open SPC values** or click on the SPC VAL button on the toolbar or in the navigation/control area of the **application** window. If the SPC values have been initialized, the values are displayed in the **SPC Values window**. If the SPC has not been initialized, the **SPC Warnings** window is displayed.

8.6.1 SPC Values Window

The **SPC Values** window displays all the values and limits calculated by the SPC module. This includes: the average and sigma values, the upper and lower limits and the number of samples since each channel was recalculated (**Cal offs**).

NOTE: In many cases LCL(R) is less than zero and is not checked because no R-value can be less than zero.



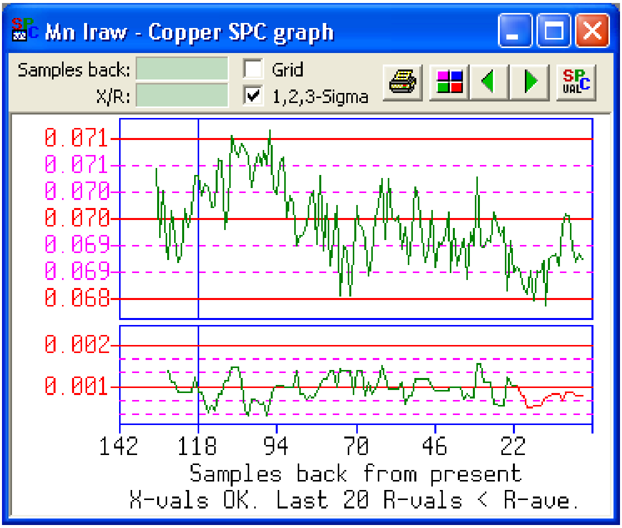
● Values ○ Counters ○ Warnings

Channel	Unit	Cal offs	X-Ave	X-Sigma	LCL[X]	UCL[X]	R-Ave
Cu Iraw	cps	34	93315.477	293.743	92434.250	94196.703	468.738
Mn Iraw	cps	24	575.808	430.612	-716.027	1867.643	687.146

Figure 8.3 SPC Values Window

8.6.2 SPC Graph Window

The calculated X- and R-values for both can be shown graphically in the **SPC graph** window. They are both shown on the same window with the X-values shown at the top.



Samples back:
X/R:
☐ Grid
☑ 1,2,3-Sigma

X-vals OK. Last 20 R-vals < R-ave.

Figure 8.4 SPC Graph Window

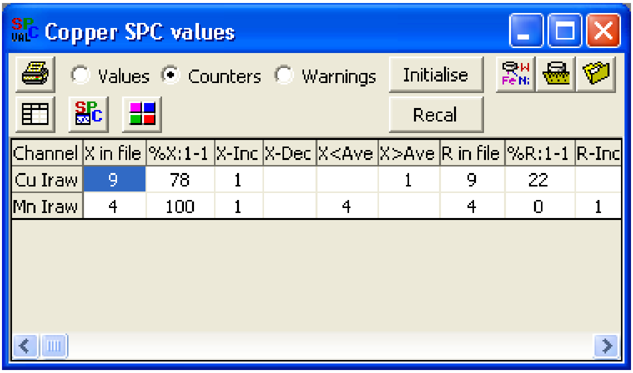
The **SPC graph** window contains several fields and buttons for selecting and handling the channels in the graph..

Table 8-3 SPC Graph Window Fields and Buttons

Menu Item	**Function**
Samples back	Shows the position of the cursor on the graph.
X/R	Shows the X- or R-value, depending on the cursor.
Grid	Displays a grid on the graphs.
1,2,3-Sigma	Displays the sigma values on the graphs.
🖨	Prints the graphs.
⊞	Tiles all open **SPC graph** windows.
◀	Displays the graph of the previous channel.
▶	Displays the graph of the next channel.
SPC VAL	Displays the **SPC Values** window.

8.6.3 SPC Counters Window

The **SPC Counters** window displays the counters used in the limits checking, for both X- and R-values. This includes % of values within the 1-sigma range, number of sequential increasing and decreasing values and the number of sequential values above and below the calculated averages.



○ Values ● Counters ○ Warnings

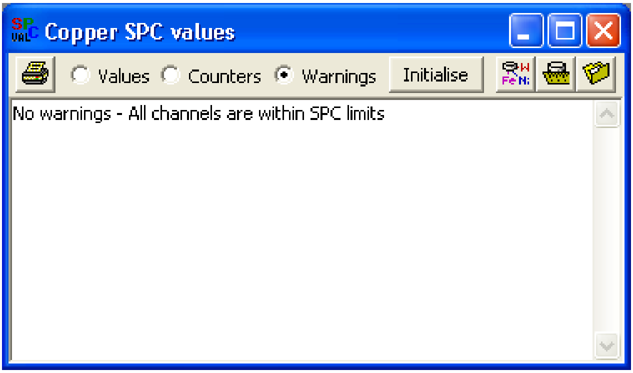
Channel	X in file	%X:1-1	X-Inc	X-Dec	X<Ave	X>Ave	R in file	%R:1-1	R-Inc
Cu Iraw	9	78	1			1	9	22	
Mn Iraw	4	100	1		4		4	0	1

Figure 8.5 SPC Counters Window

Any counter values that are outside the limits are shown in red. This gives a clear indication of any problems in addition to the information displayed on the **SPC Warnings** window.

8.6.4 SPC Warnings Window

The **SPC Warnings** window displays any warning message that is generated by the SPC module. A warning message is generated if the SPC module is not correctly initialized or any values are outside the limits.



○ Values ○ Counters ● Warnings

No warnings - All channels are within SPC limits

Figure 8.6 SPC Warnings Window

Chapter 9

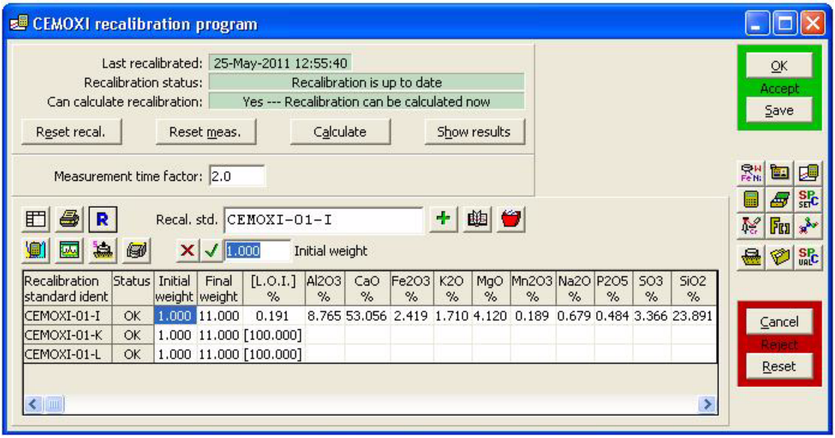
Recalibration

9.1 INTRODUCTION

Recalibration is used for applications that were measured on a different Epsilon 3 system and have to be used on the current Epsilon 3 system, or to refresh an existing calibration but using less standards.

9.2 CARRYING OUT A RECALIBRATION

- Copy the original application to the Epsilon 3 working directory.
- Open the application, in which you want to carry out a recalibration of standards or routine samples.
- In the **application** window, click on the button. The **recalibration program** window is displayed.



Last recalibrated: 25-May-2011 12:55:40
Recalibration status: Recalibration is up to date
Can calculate recalibration: Yes --- Recalibration can be calculated now

Measurement time factor: 2.0

Recal. std. CEMOXI-01-I

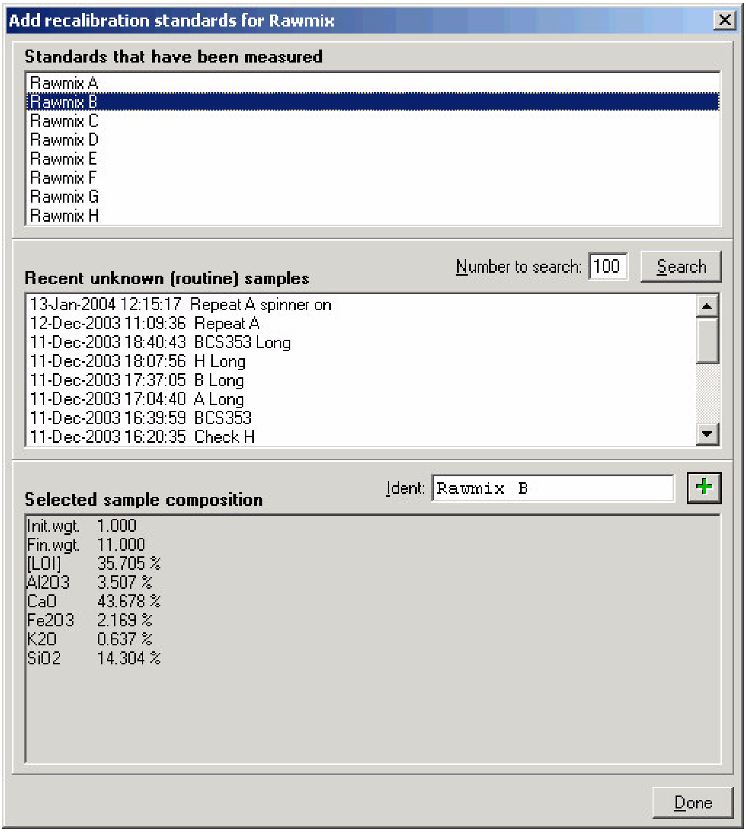
1.000 Initial weight

Recalibration standard ident	Status	Initial weight	Final weight	[L.O.I.] %	Al2O3 %	CaO %	Fe2O3 %	K2O %	MgO %	Mn2O3 %	Na2O %	P2O5 %	SO3 %	SiO2 %
CEMOXI-01-I	OK	1.000	11.000	0.191	8.765	53.056	2.419	1.710	4.120	0.189	0.679	0.484	3.366	23.891
CEMOXI-01-K	OK	1.000	11.000	[100.000]										
CEMOXI-01-L	OK	1.000	11.000	[100.000]										

Figure 9.1 Recalibration Program Window

- In the **Recal. std.** field, enter a new name for the recalibration standard.
- The field **Measurement time factor** is used to multiply the application measurement time with a factor to improve the counting statistics. The default value is “2”.

- Click on the button. The **Add recalibration standards** window is displayed.



Standards that have been measured

Rawmix A
Rawmix B
Rawmix C
Rawmix D
Rawmix E
Rawmix F
Rawmix G
Rawmix H

Recent unknown (routine) samples

Number to search: 100

13-Jan-2004 12:15:17 Repeat A spinner on
12-Dec-2003 11:09:36 Repeat A
11-Dec-2003 18:40:43 BCS353 Long
11-Dec-2003 18:07:56 H Long
11-Dec-2003 17:37:05 B Long
11-Dec-2003 17:04:40 A Long
11-Dec-2003 16:39:59 BCS353
11-Dec-2003 16:20:35 Check H

Selected sample composition

Ident: Rawmix B

Init.wgt.	1.000
Fin.wgt.	11.000
[LOI]	35.705 %
Al2O3	3.507 %
CaO	43.678 %
Fe2O3	2.169 %
K2O	0.637 %
SiO2	14.304 %

Figure 9.2 Add Recalibration Standards Window

The **Add recalibration standards** window has three main sections:

Standards that have been measured	Lists the standards that have been measured on the original Epsilon 3.
Recent unknown (routine) samples	Lists the unknown (routine) samples measured on the original Epsilon 3 system.
Selected sample composition	Displays the concentration of the highlighted sample or standard.

Both standards and unknown (routine) samples can be used as recalibration standards. When one of the samples in the upper or the middle part (**Standards that have been measured** or **Recent unknown (routine) samples**) is highlighted, the concentrations of this sample are shown in the lower part (**Selected sample composition**).

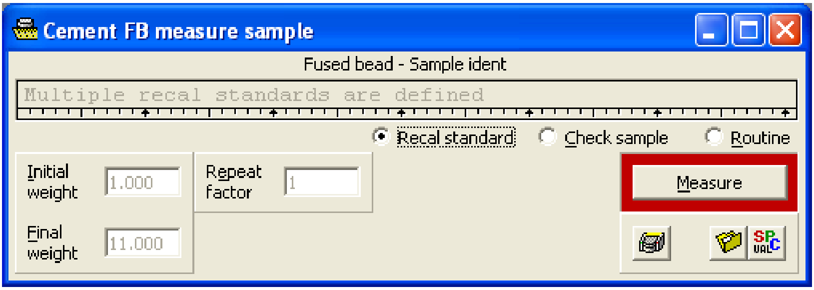
To add a selected sample to the recalibration program:

- Highlight the sample and click on the + button. The grid in the **recalibration program** window is automatically updated.

  For one-point recalibration, only one sample is needed as a recalibration standard. In this case, only the E value is recalculated. When using more than 1 recalibration standard, then D and E values can be recalculated, where D is the intercept and E is the slope of the calibration line.
- Click on the **Save** button to save the **recalibration program** window.

To measure the recalibration standards in the recalibration program:

- Highlight the standard and click on the button.
- The recalibration standards can also be measured by clicking on the button, which displays the **measure sample** window.



Cement FB measure sample

Fused bead - Sample ident

Multiple recal standards are defined

● Recal standard ○ Check sample ○ Routine

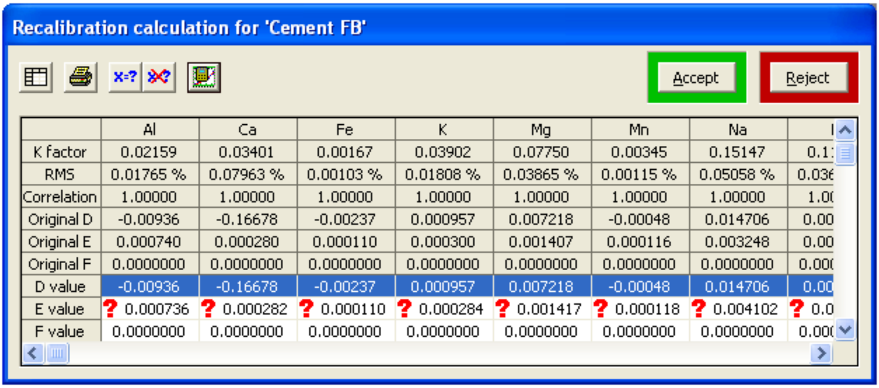
Initial weight: 1.000

Repeat factor: 1

Final weight: 11.000

Figure 9.3 Measure Sample Window

- Specify the sample type as **Recal standard** and click on the **Measure** button.
- By default, the E value is recalculated automatically. After the recalibration measurements are finished, the **Recalibration results** window is displayed. Click on the ☒ button to close the window.
- If both D and E values must be recalibrated, click on **Calculate** button to open the **Recalibration results** window.

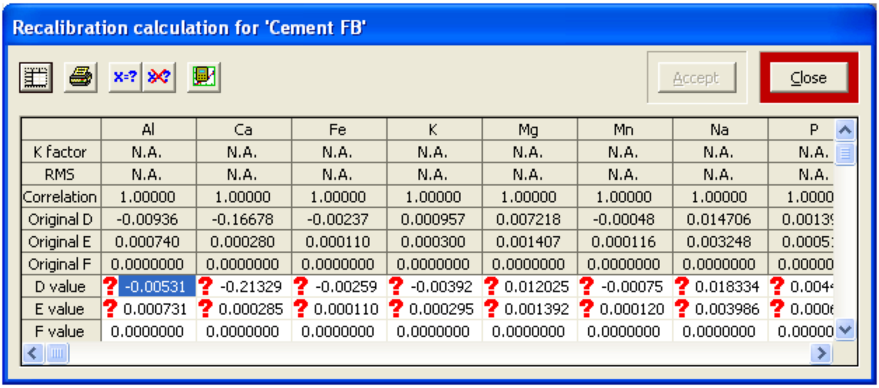


Recalibration calculation for 'Cement FB'

	Al	Ca	Fe	K	Mg	Mn	Na	
K factor	0.02159	0.03401	0.00167	0.03902	0.07750	0.00345	0.15147	0.1
RMS	0.01765 %	0.07963 %	0.00103 %	0.01808 %	0.03865 %	0.00115 %	0.05058 %	0.03
Correlation	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.0
Original D	-0.00936	-0.16678	-0.00237	0.000957	0.007218	-0.00048	0.014706	0.00
Original E	0.000740	0.000280	0.000110	0.000300	0.001407	0.000116	0.003248	0.00
Original F	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.00
D value	-0.00936	-0.16678	-0.00237	0.000957	0.007218	-0.00048	0.014706	0.00
E value	? 0.000736	? 0.000282	? 0.000110	? 0.000284	? 0.001417	? 0.000118	? 0.004102	? 0.0
F value	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.00

Figure 9.4 Recalibration Calculation Window - Highlighted D Value

- Highlight the row **D value** and click on x=? button. These parameters are now indicated by a "?"



	Al	Ca	Fe	K	Mg	Mn	Na	P
K factor	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
RMS	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
Correlation	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.0000
Original D	-0.00936	-0.16678	-0.00237	0.000957	0.007218	-0.00048	0.014706	0.0013
Original E	0.000740	0.000280	0.000110	0.000300	0.001407	0.000116	0.003248	0.0005
Original F	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.00000
D value	? -0.00531	? -0.21329	? -0.00259	? -0.00392	? 0.012025	? -0.00075	? 0.018334	? 0.004
E value	? 0.000731	? 0.000285	? 0.000110	? 0.000295	? 0.001392	? 0.000120	? 0.003986	? 0.000
F value	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.0000000	0.00000

Figure 9.5 Recalibration Calculation Window Before Recalculation

- Click on button to carry out the calculation. The original and the new D and E values are shown in the **Recalibration calculation** window.
- Click on the **Accept** button to save the results. From now on, every time after the recalibration standard measurements are finished, the D and E values will be recalculated automatically.

In case of transferring a calibration from another system to the current system, it is recommended to measure a few standards as unknown to validate the recalibration before analyzing routine samples.

In case of refreshing an existing calibration on the current system, it is recommended to measure a check sample to validate the recalibration before analyzing routine samples.

Chapter 10

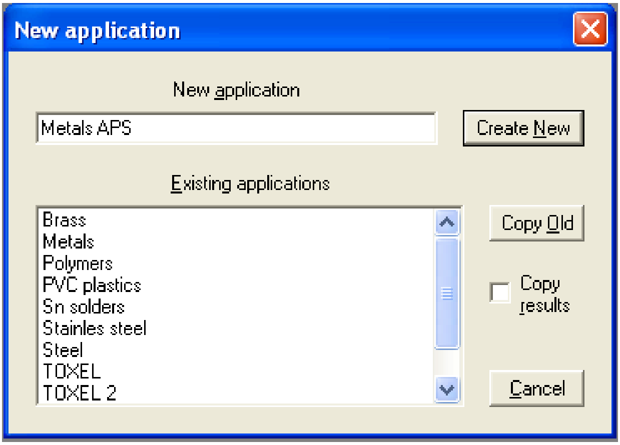
Automatic Program Selection (APS)

10.1 INTRODUCTION

Automatic Program Selection (APS) gives you the ability to use a short measurement program that measures a few key elements, after which the software automatically selects the appropriate application.

10.2 SETTING UP AUTOMATIC PROGRAM SELECTION

- Create a new application as a base application for APS.



New application: Metals APS

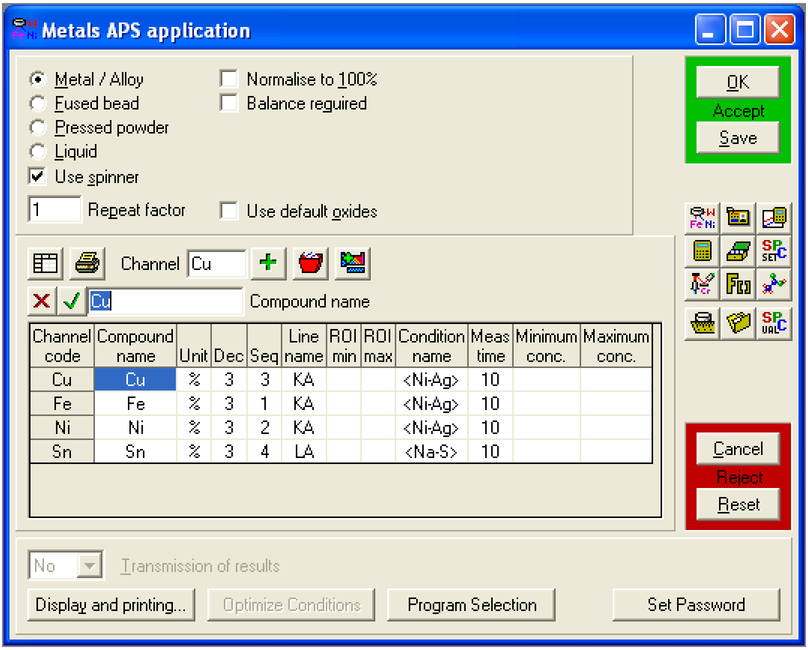
Existing applications:

Brass
Metals
Polymers
PVC plastics
Sn solders
Stainles steel
Steel
TOXEL
TOXEL 2

☐ Copy results

Figure 10.1 New Application Window

- Fill in the name of the base application and press the **Create New** button. The **APS application** window is displayed.



● Metal / Alloy
○ Fused bead
○ Pressed powder
○ Liquid
☑ Use spinner
1 Repeat factor

☐ Normalise to 100%
☐ Balance required
☐ Use default oxides

Channel Cu

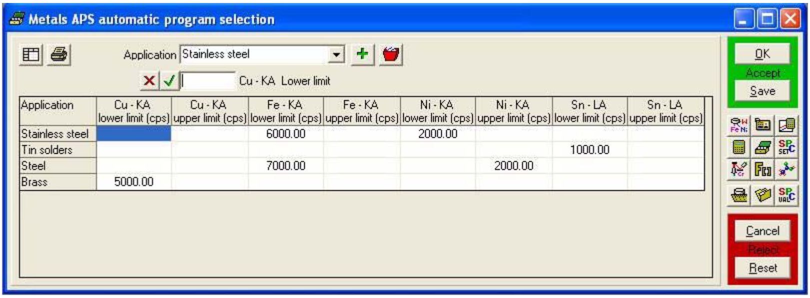
Cu Compound name

Channel code	Compound name	Unit	Dec	Seq	Line name	ROI min	ROI max	Condition name	Meas time	Minimum conc.	Maximum conc.
Cu	Cu	%	3	3	KA			<Ni-Ag>	10		
Fe	Fe	%	3	1	KA			<Ni-Ag>	10		
Ni	Ni	%	3	2	KA			<Ni-Ag>	10		
Sn	Sn	%	3	4	LA			<Na-S>	10		

No Transmission of results

Figure 10.2 APS Application Window

- Using intensity criteria, select the elements that APS has to check for.
- Include a measurement condition and short measuring time for each element.
- Click on the **OK** button to save the application.
- Click on the **Program Selection** button to open the **APS automatic program selection** window.



Metals APS automatic program selection

Application: Stainless steel

Cu - KA Lower limit

Application	Cu - KA lower limit (cps)	Cu - KA upper limit (cps)	Fe - KA lower limit (cps)	Fe - KA upper limit (cps)	Ni - KA lower limit (cps)	Ni - KA upper limit (cps)	Sn - LA lower limit (cps)	Sn - LA upper limit (cps)
Stainless steel			6000.00		2000.00			
Tin solders							1000.00	
Steel			7000.00			2000.00		
Brass	5000.00							

Figure 10.3 APS Automatic Program Selection Window with Intensity Criteria

10.3 ADDING AN APPLICATION IN THE APS WINDOW

- Select an application from the drop-down list and click on the + button.
- Enter the upper and lower limits of the count rate for each of the selected applications into the grid.
- Save the criteria by clicking on the **OK** button.

NOTES:

a. Lower and/or upper intensity criteria can be set for one or more elements.

b. APS will select a certain application when all criteria for that specific application are fulfilled.

c. When no criteria are set for a certain application, that application will be used.

d. The order of the applications listed in the **APS automatic program selection** window is important. The software will scan the list of applications from top to bottom and the first application that fulfills all criteria will be used for measuring the unknown sample.

e. The order of the applications in the list can easily be changed by drag and drop of the application name to the appropriate place in the grid.

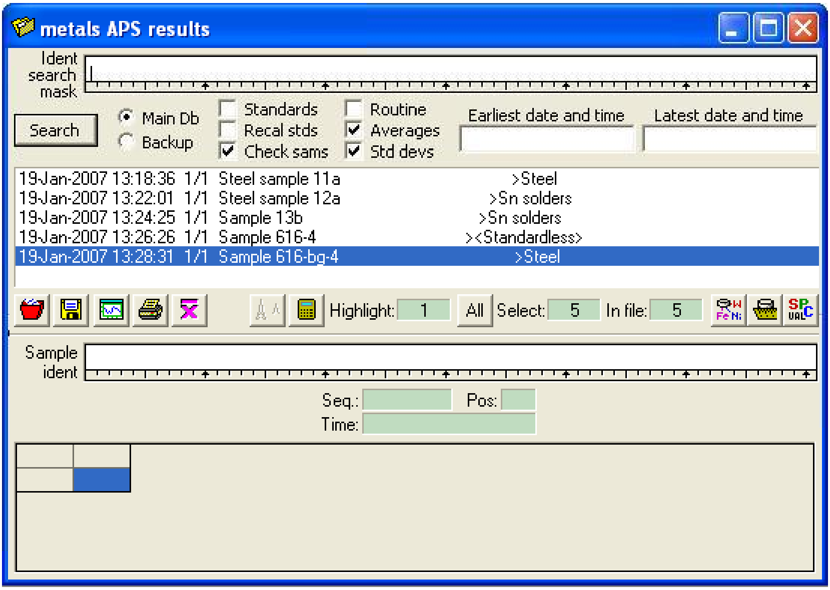
10.4 CARRYING OUT AN APS MEASUREMENT

- In **Measure application** window, select the base application that will be used for APS and click on the **OK** button. The **Measure sample** window is displayed.
- Enter the name of the routine sample and click on the **Measure** button.

10.5 VIEWING THE RESULTS

The result of the APS measurement (checking which application to use) is visible in the **APS results** window of the APS application.

In this window, the name of each sample is extended with the name of the application that is used to quantify this sample. The actual result of the unknown sample is visible in the **APS results** window of that specific application.



● Main Db
○ Backup
☐ Standards
☐ Recal stds
☑ Check sams
☐ Routine
☑ Averages
☑ Std devs

```
19-Jan-2007 13:18:36  1/1  Steel sample 11a              >Steel
19-Jan-2007 13:22:01  1/1  Steel sample 12a           >Sn solders
19-Jan-2007 13:24:25  1/1  Sample 13b                >Sn solders
19-Jan-2007 13:26:26  1/1  Sample 616-4             ><Standardless>
19-Jan-2007 13:28:31  1/1  Sample 616-bg-4               >Steel
```

Highlight: 1
Select: 5
In file: 5

Figure 10.4 APS Results Window

Chapter 11

Enhanced Data Security

11.1 INTRODUCTION

The Enhanced Data Security is an optional extension to the Epsilon 3 software.

Changing the Enhanced Data Security configuration is restricted to users with a sufficient privilege level (configurable via the **User administration** window).

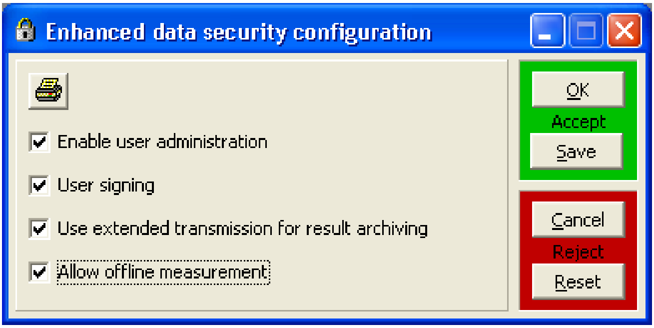
11.2 ENHANCED DATA SECURITY FEATURES

11.2.1 Enhanced Data Security Configuration Window

To open the Enhanced data security configuration window:

- Select **System** > **Enhanced data security**.

  NOTE: This menu item is not available when you have not installed the Enhanced data security software option.



☑ Enable user administration

☑ User signing

☑ Use extended transmission for result archiving

☑ Allow offline measurement

Figure 11.1 Enhanced Data Security Configuration Window

The 🖨 button will generate a report with the data present in this form.

11.2.1.1 Enable User Administration

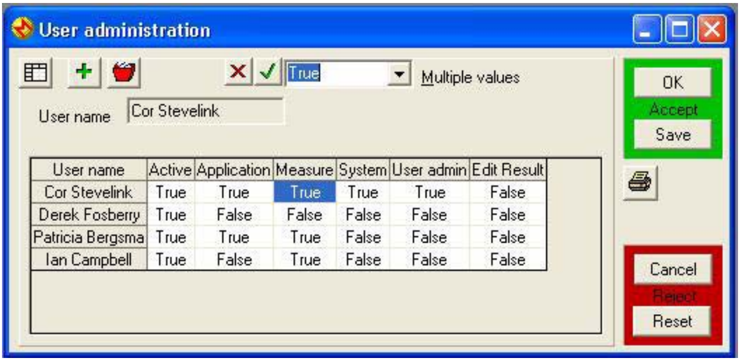
User administration is designed to work in a windows network environment.

The user that started the Epsilon 3 software and checked the **Enable user administration** check box in the **Enhanced data security configuration** window becomes automatically the active user and gets the full access rights in the **User administration** window. The (PC) user name is shown on top of the main window.

These access rights can be changed in the **User administration** window.

The **User administration** window can also be used to add, modify and delete users to comply with the Enhanced data security regulations.

NOTE: If the **Enable user administration** check box is checked in the **Enhanced data security configuration** window, at least one user must be added to the user administration with both **Active** set to **True** AND **User admin** set to **True**.
If not, logging off the Epsilon 3 software will no longer allow you to login again!



True

User name: Cor Stevelink

User name	Active	Application	Measure	System	User admin	Edit Result
Cor Stevelink	True	True	True	True	True	False
Derek Fosberry	True	False	False	False	False	False
Patricia Bergsma	True	True	True	False	False	False
Ian Campbell	True	False	True	False	False	False

Figure 11.2 User Administration Window

11.2.1.2 User Signing

When **User signing** is checked, the user has to sign each time via the **Set signature** window at the following trigger moments that generate or change result data:

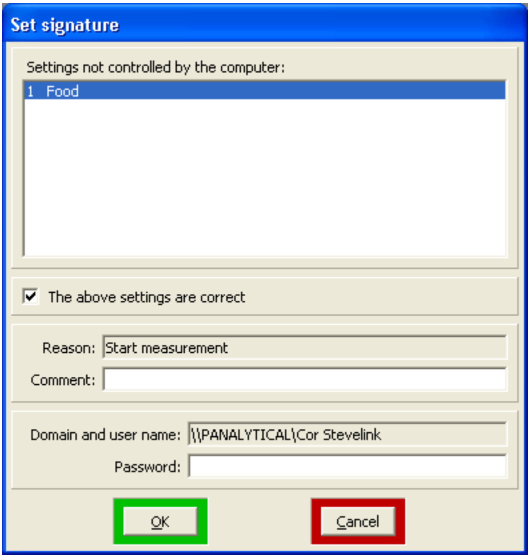
- Start measurement
  Start a manual measurement via the **Measure** window, the **Measure Omnian sample** window or the **easy measure** window.

- Abort measurement
  Abort a measurement via the **Manual control** window.
- Start batch measurement
  Start a sample changer list via the **Measure** window.

NOTE: The check box **User signing** can only be checked when User validation via operation system is switched on.

Note that it is not possible to change a running batch when the user signed for it.

All actions which lead to new/altered results are also stored in the **Message** window.



Settings not controlled by the computer:

1 Food

☑ The above settings are correct

Reason: Start measurement

Comment:

Domain and user name: \\PANALYTICAL\Cor Stevelink

Password:

Figure 11.3 Set Signature Window

11.2.1.3 Use Extended Transmission for Result Archiving

Although **Use of extended transmission for result archiving** is not actually a part of the Enhanced Data Security, it is related/linked to other Enhanced Data Security features and therefore can be modified only via the **Enhanced data security configuration** window when the Enhanced Data Security option is licensed

11.2.1.4 Allow Offline Measurement

When **Allow offline measurement** is checked, the **Offline input** window is displayed.

11.2.2 Editing of Measurement Results

When the check box **Enable user administration** is checked on the **Enhanced data security configuration** window, it is not possible for the active user to edit the measurement results. This feature can be switched on and off by checking or clearing the check box.

NOTE: Editing of measurement results is always possible but the 21 CFR Part 11 (see Section 11.2.4) requires that dates are accompanied with time zone information and signature.

When **Enable user administration** is checked, this involves the following actions on the **Results** window:

- Recalculating the selected results
  Clicking on the **Recalculate** button on the **Results** window will display a warning message and the action will not be executed.
- Editing a sample ident
  Clicking on the **Edit sample ident** button on the **Results** window will display a warning message and the action will not be executed.
- Deleting results
  Clicking on the **Delete** button on the **Results** window will display a warning message and the action will not be executed.

11.2.3 Time zones

21 CFR Part 11 (see Section 11.2.4) requires that dates are accompanied with time zone information.

21 CFR Part 11 supports the way in which Windows® handles time zones as an offset to Coordinated Universal Time (UTC), formerly called Greenwich Mean Time (GMT).

NOTE: The clock goes forward and backward for daylight saving time but UTC remains the same all year around.

An offset to UTC is therefore often presented as “UTC+/-<HH:MM>”, where <HH:MM> represents the current offset from UTC in 24-hours notation.

As an example, consider the time zone at PANalytical Almelo, The Netherlands. When standard time is active, the time zone representation will be “UTC+01:00”. During daylight saving time, the time zone representation will be “UTC+02:00”.

NOTE: Some countries in the world have times off by less than one hour, e.g. half an hour or even a quarter of an hour!

You should adjust the clock of your computer when daylight saving time changes.

When **Enhanced data security** is enabled, the time zone is added to the **Results** database. For existing results it is impossible to obtain this number afterwards.

The time zone (UTC) and User name will be shown as extra fields on the following windows:

- **Results** window
- **Results spectra** window
- **Results report** window (printout)
- **Result graph report** window (printout)

11.2.4 21 CFR Part 11 Support

The United States of America’s Food and Drug Administration (FDA) have issued regulations relating to the use of electronic records and electronic signatures. These requirements (21 CFR Part 11) have implications for the pharmaceutical and related industries and their suppliers. The processes in the pharmaceutical industry should be 21 CFR Part 11 compliant and PANalytical X-ray analysis products should support such processes.

- In order to comply with the 21 CFR Part 11 regulations with respect to the handling of results, ALL check boxes in the **Enhanced data security** window must be checked.

Chapter 12

Off-line Operation

12.1 INTRODUCTION

Sample measurements can be simulated using the **Offline status** window, see Figure 12.1. This allows the intensities normally produced by measuring a sample to be entered from file(s). The data entered is then processed exactly as if it is from a measured sample.

This method can be used to simulate measurements of: routine samples and calibration standards.

The **Offline status** window is only available when the spectrometer is off-line, i.e. not connected to the PC, not switched on or the software has released the communication connection between the PC and the spectrometer.

12.2 OFF-LINE MEASUREMENTS

The measurement of routine and calibration samples can be simulated in the **Offline status** window. This is done by attempting to carry out the measurement as normal. Then, when normally the spectrometer would be measuring the sample, the **Offline status** window is displayed, allowing you to enter the simulated results.

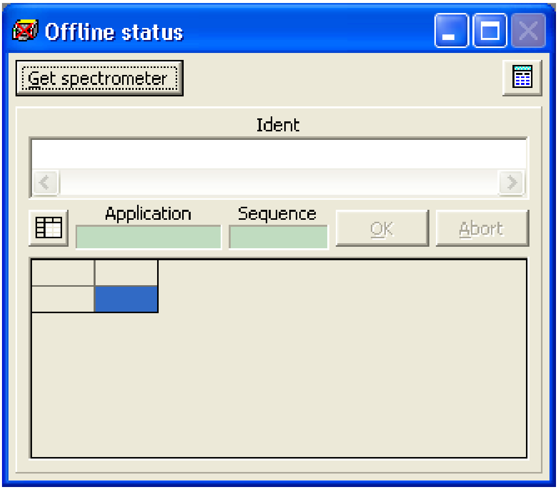


Figure 12.1 Offline Status Window

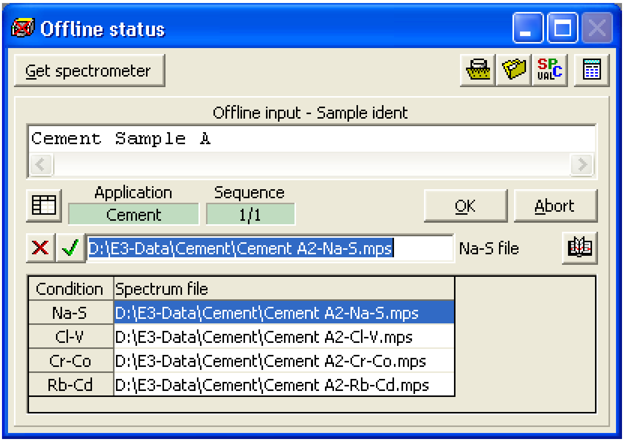
12.2.1 Starting an Off-line Measurement

NOTE: Measurements must have been exported before starting an off-line measurement.

The navigation buttons displayed at the top of the **Offline status window** will vary according to the type of measurement being carried out: routine or calibration.

To simulate a routine sample measurement:

- Click on the toolbar button or select **Application > Open application**.
- Select the required application from the list and click on the **OK** button.
- In the navigation/control area of the **application** window, click on the button. The **measure sample** window of the current application is opened.
- In the **Sample ident** field, enter the name of the sample.
- Click on the **Measure** button to start the measurement. The **Offline status** window is displayed, showing details of the application and sample.



Offline input - Sample ident

Cement Sample A

Application: Cement

Sequence: 1/1

Na-S file: D:\E3-Data\Cement\Cement A2-Na-S.mps

Condition	Spectrum file
Na-S	D:\E3-Data\Cement\Cement A2-Na-S.mps
Cl-V	D:\E3-Data\Cement\Cement A2-Cl-V.mps
Cr-Co	D:\E3-Data\Cement\Cement A2-Cr-Co.mps
Rb-Cd	D:\E3-Data\Cement\Cement A2-Rb-Cd.mps

Figure 12.2 Offline Status Window

12.2.2 Carrying Out an Off-line Measurement

To enter the simulated intensities:

- Click on the 📖 button and find the required MPS or SPI file.
- Click on the ✓ button to fix the value in the field.
- Repeat this operation for each condition.

NOTE: The conditions of the spectra in the files must match those of the application. Also, you must specify a file for all conditions in the application.

- Click on the **OK** button
- If the application is set for a sequence of measurements, the sequence number increments and you are required to enter the data for the next measurement. This is repeated until the sequence is completed.
- When clicking on the **OK** button after the final measurement in the sequence, the software calculates the results and displays the results in the **Offline measurement** window.

  NOTE: You have to enlarge the window to see the full results.
- For any channel you did not enter data for, the message **No intensity** is displayed.

The results of an off-line measurement are treated in exactly the same way as the results for a normal measurement. This means that the results can be viewed in the **Last result** window and the results are stored in the results database file for that application. These results can be recovered and displayed like any normal measurement results.

12.2.3 Cancelling an Off-Line Measurement

An off-line measurement can be cancelled at any moment by clicking on the **Abort** button.

Each measurement must be either completed or cancelled before any other measurement can take place. If you attempt to start another measurement before the current measurement is complete, a warning message is displayed informing you the current measurement is still in progress.

Chapter 13

Tips for Setting Up Measuring Conditions

13.1 INTRODUCTION

This chapter gives some hints about setting up conditions (measurement parameters: voltage, current, filter, medium and time). It is assumed that you are familiar with the theory of X-ray emission in general (absorption edges, line energies etc.).

We do not attempt to provide the ‘one and only’ way to find the best conditions for an application, nor to discuss the subject in detail. We are just giving hints and some background information about why you should try a certain way.

13.2 DEFINING THE PROBLEM

With regard to measuring conditions there are basically two types of problems:

1. Setting up an application to measure samples with known elements.
2. Setting up an application to measure unknown samples.

Up to a certain point, both problems can be handled in the same way.

However, in the first case you may decide to ignore certain features in the spectrum because it is of no interest to the problem (for example process control).

In the second case the fine tuning of the conditions may be a long and tedious job.

Furthermore, for a known sample, one of the elements may be present in a very low concentration, such that special care must be taken to detect it.

In general, the problem is that you need to ‘see’ the spectral lines with a good signal-to-noise ratio.

13.3 SIGNAL-TO-NOISE RATIO

If you have a line with a low signal-to-noise ratio you can improve it by:

1. Increasing the intensity of the primary X-rays.
2. Increasing the measuring time.
3. Averaging several measurements of the same sample.

13.3.1 Increasing the Intensity

You can increase the intensity of the primary X-rays by increasing the power applied to the X-ray tube (up to the tube limits). Normally this is done by increasing the current, as the voltage is usually set to a pre-determined level in order to observe certain features (for example to excite certain elements) in the measured spectra.

An important factor to be considered is the detector count rate. When you increase the current, the count rate increases. A potential negative effect is the peaks ‘shift’ towards the lower energies. Both these effects may degrade the results and should therefore be avoided.

13.3.2 Increasing the Measuring Time

You can decrease the effect of noise by increasing the measurement time. However, this cannot be done indefinitely as the noise is inversely proportional to the square root of the measurement time and the maximum useful measurement time is determined by the detector’s intrinsic noise.

13.3.3 Averaging Several Measurements of the Same Sample

You can measure the same sample several times and then average the results to reduce the effect of noise. This improves the signal-to-noise ratio by a factor of the square root of the number of times it is measured (for example, if you measure 10 times, the improvement factor = $\sqrt{10}$).

13.4 SPECTRAL LINES

Once again, the main goal is to see spectral lines, either a certain few that you are specifically interested in, or all of the lines present in the sample. The remainder of this chapter will concentrate on that subject.

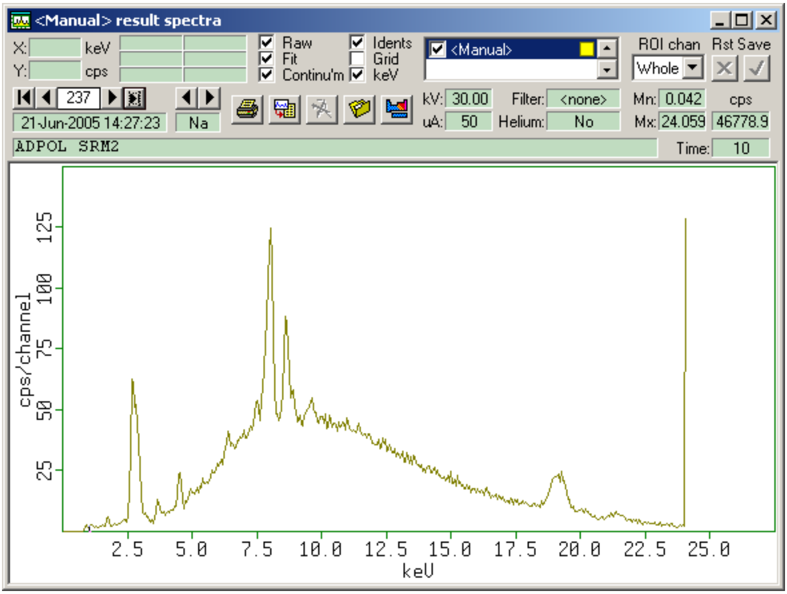
13.5 MANUAL MODE OR APPLICATION MODE

There are two ways that you can start a measurement in Epsilon 3 (see also Chapter 5 and Chapter 7):

1. Using manual mode (see Section 5.3 and Section 7.3).
2. Using application mode.

13.6 INSPECTING A SPECTRUM

If you do not have any prior knowledge about a certain condition to be used, put the sample in the sample changer and use manual mode. An example of a resulting spectrum is shown in Figure 13.1.



<Manual> result spectra

X: keV
Y: cps

☑ Raw
☑ Fit
☑ Continu'm
☑ Idents
☐ Grid
☑ keV

☑ <Manual>

ROI chan: Whole

237

21-Jun-2005 14:27:23

Na

ADPOL SRM2

kV: 30.00
uA: 50
Filter: <none>
Helium: No
Mn: 0.042
Mx: 24.059
cps: 46778.9
Time: 10

Figure 13.1 Sample Spectrum - First Try

You can see a strong line at 2.8 keV that can be attributed to RhLα (from the X-ray tube). Other peaks are located near 8 keV due to CuKα, 8.6 keV due to ZnKα and 19 keV due to RhKα.

Because the sample is a polymer standard, we know that it also contains Na, Mg, Al, Si, P, S, Ca and Ti. Deconvoluting the spectrum with all these lines shows where the lines of these elements are located in the spectrum. Please note that the signal-to-noise ratio of those lines is too low to provide a reliable intensity value. To increase the signal-to-base ratio of these lines, we can do the following:

1. Using a filter for high energy lines.
2. Decreasing the voltage for low energy lines.

13.7 USING FILTERS

The effect of a filter is two-fold:

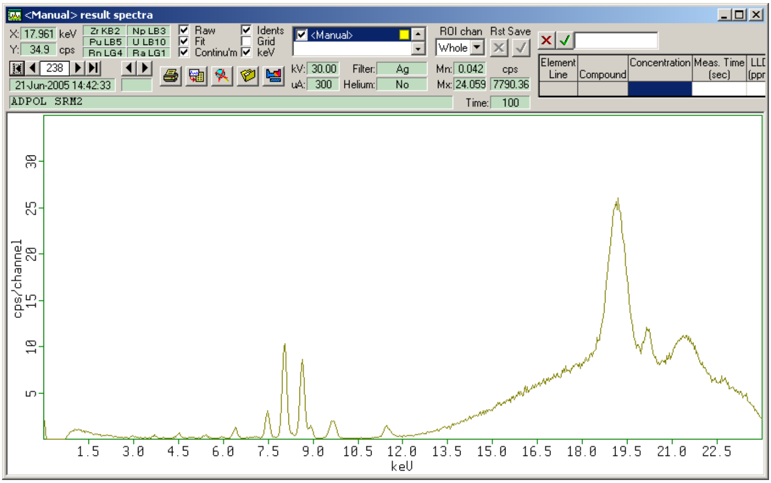
1. The overall intensity of the signal is decreased.
2. The ratio of high energy intensity to low energy intensity is increased.

The first effect is because X-ray photons are absorbed by the filter.

The second effect is because the absorption is not uniform over the whole spectral range. We want to find the optimum filter (the results show the lines that you want to see) and set the current high enough to get enough counts, but not too high.

One way to find out the best filter to use is to create an application with, for example, 6 conditions. For each of the conditions, the voltage applied to the X-ray tube must be the same and the filters must be different. The current must be chosen such that the count rate is optimal. If you check the **autocurrent** check box, this is done automatically.

Figure 13.2 shows a spectrum using the Ag filter. The same sample is shown in Figure 13.1, but without the filter. The measuring time was 100 s.



<Manual> result spectra

X: 17.961 keV
Y: 34.9 cps
238
21-Jun-2005 14:42:33
ADPOL SRM2
☑ Raw
☑ Fit
☑ Continu'm
☑ Idents
☐ Grid
☑ keV
☑ <Manual>
ROI chan: Whole
kV: 30.00
uA: 300
Filter: Ag
Helium: No
Mn: 0.042
Mx: 24.059
cps: 7790.36
Time: 100

Figure 13.2 Sample Spectrum with Optimum Filter

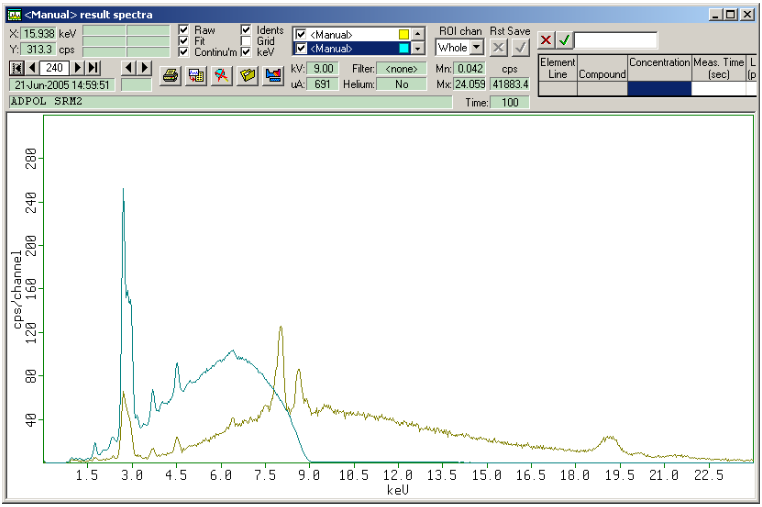
Several low intensity lines are now visible in the energy region around Cu.

To find out to which elements these lines belong:

- In the **results spectra** window, click on the button to open the **Elements and line groups** window and then click on “Cu” in the periodic system.
- Use the right arrow to go through the elements in the periodic table (towards higher energies) and highlight the elements that match the lines.
- Finally perform another deconvolution.

13.7.1 Backscatter Bulge

For light elements there can be a high backscatter bulge in the spectrum, hiding low intensity lines. Figure 13.3 shows two spectrums, they are both of the same sample, but measured with two different tube voltages: 9 and 30 keV.



X: 15.938 keV

Y: 313.3 cps

☑ Raw ☑ Fit ☑ Continu'm ☑ Idents ☐ Grid ☑ keV

☑ <Manual> ☑ <Manual>

ROI chan: Whole

21-Jun-2005 14:59:51

kV: 9.00 uA: 691

Filter: <none> Helium: No

Mn: 0.042 Mx: 24.059 cps 41883.4

ADPOL SRM2

Time: 100

Figure 13.3 Backscatter Bulges

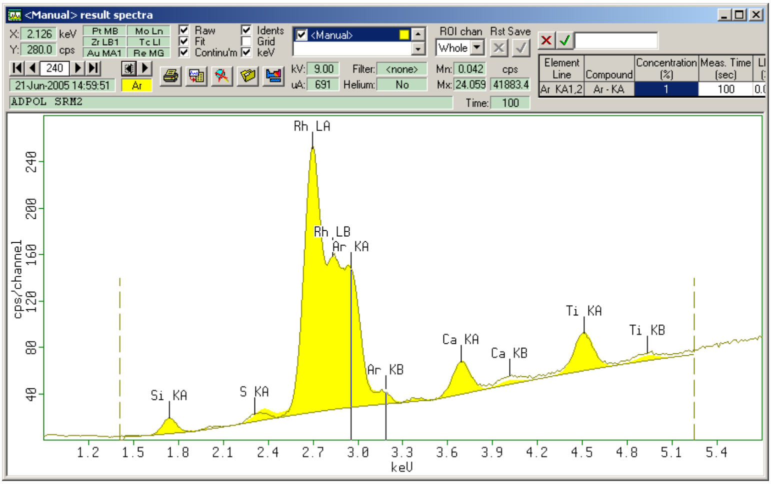
For both voltages, the backscatter bulge is visible. For 9 keV the complete backscatter bulge is visible, for the 30 keV most of it is visible. One of the elements to be measured from this sample is Cu. Theoretically, 9 keV is enough to show the Cu lines, but in this case it is better to change the voltage settings.

13.8 Rh, Ar, Ag AND Mo LINES

It may occur that a spectrum shows lines from Rh, Ar, Ag and Mo. Although these elements can of course be present in the measured sample, this is not necessarily the case. In the Epsilon 3, the primary X-rays are generated in a Rh, Mo or Ag X-ray tube, thus these lines may be present.

For the other elements, the contribution is intrinsic to the hardware. These line intensities are normally very small.

When using manual mode, these lines must be added manually. This is not necessary in the application mode. Figure 13.4 shows an example of a spectrum that contains Rh and Ar lines (from air) in the region from 2.8 to 3.2 keV.



<Manual> result spectra

X: 2.126 keV
Y: 280.0 cps
☑ Raw
☑ Fit
☑ Continu'm
☑ Idents
☐ Grid
☑ keV
☑ <Manual>
ROI chan: Whole
240
21-Jun-2005 14:59:51
Ar
kV: 9.00
uA: 691
Filter: <none>
Helium: No
Mn: 0.042
Mx: 24.059
cps: 41883.4
ADPOL SRM2
Time: 100

Element Line	Compound	Concentration (%)	Meas. Time (sec)
Ar KA1,2	Ar - KA	1	100

Figure 13.4 Spectrum Containing Rh and Ar Lines

13.9 FIXED LINE RATIOS

The Epsilon 3 software contains a database with all measurable K, L, and M lines, including line ratios between the lines (for example between Kα and Kβ). This ratio is used when fitting the spectrum in the deconvolution step.

For the K lines this is mostly correct. However, for the L lines, the actual ratio in the spectrum may differ from the theoretical ratio. This means that when you fit a spectrum, the L line ratio must thus be a free parameter. This can be done by deselecting (greying) the **L** button in the periodic table and selecting (yellowing) the **L1**, **L2**, and **L3** buttons.

13.10 LIGHT ELEMENT ANALYSIS CONDITIONS

Elements in the range of Al to K can be measured in air or helium. When analyzing these elements in air, the precision may be worse because of variations in air pressure and temperature. When air pressure correction is switched on in the application, the instability is corrected by using the Al intensity of the gain measurement.

When measuring light elements Na to S in an analysis it is better to use helium because this obtains higher intensities.