S4 Training Manual

M80-E00024 2/05

S4 Training Manual

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Address comments to:

Service Department
Bruker AXS Inc.
5465 East Cheryl Parkway
Madison, Wisconsin 53711-5373
USA

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Table of Contents

1 Introduction to SPECTRA*plus*

SPECTRA*plus* has provisions for doing qualitative, standardless, and quantitative measurements in one integrated package. It includes a sophisticated Fundamental Parameter program to correct for “matrix” effects, and a flexible reporting tool for distributing the analytical results.

This section provides a brief introduction to the SPECTRA*plus* software. It covers how to start the various programs that make up SPECTRA*plus* and gives a brief overview of the programs used for day-to-day “routine” operation, for standardless analysis, and for setting up quantitative methods.

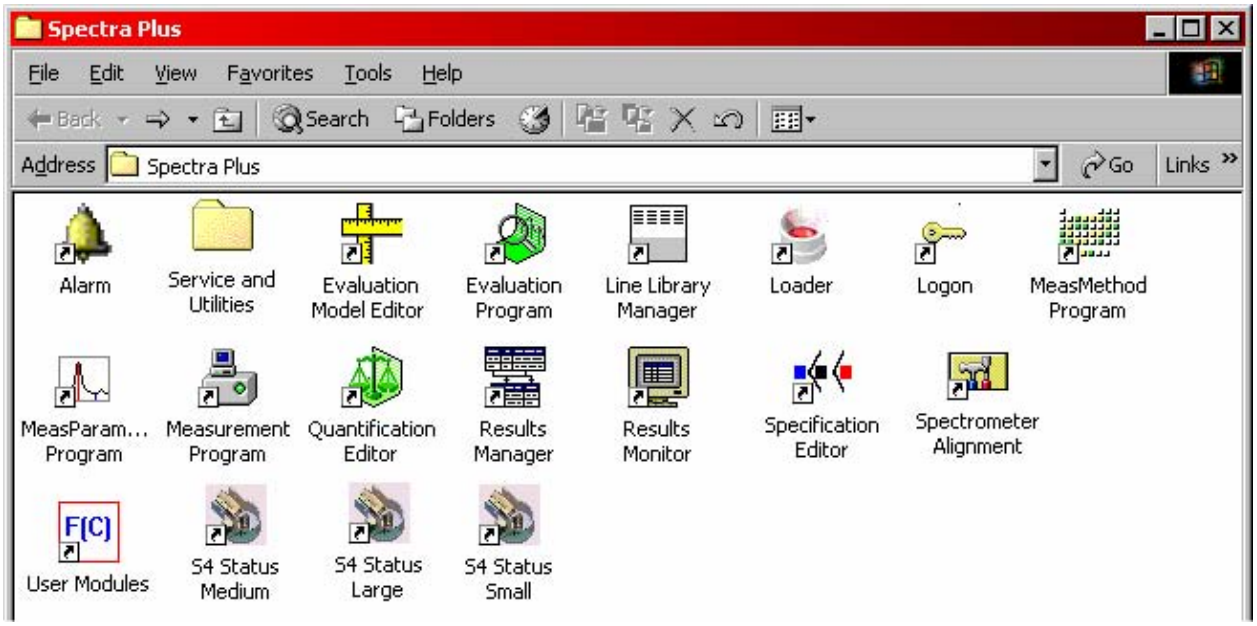
1.1 Starting the SPECTRA*plus* programs

Two methods can be used for starting the SPECTRA*plus* programs. In addition to the methods given here, some users prefer to add the most commonly used SPECTRA*plus* icons to the Windows Quick Launch toolbar, or to the Microsoft Office toolbar.

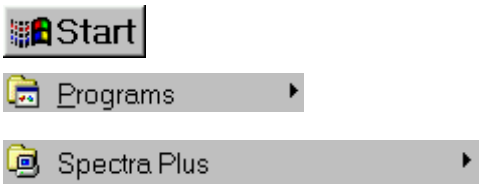
Method #1: Using the Spectra Plus icon on the Windows Desktop:

Double-click the **Spectra Plus** folder on the Windows Desktop to open the list of shortcuts for launching the SPECTRA*plus* programs.

Double-click the icon for the desired program.



Method #2: Using the Windows Start button:



Click the **Start** button on the task bar.

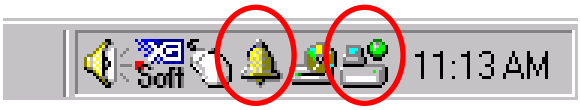
Click on **Programs**.

Click on **Spectra Plus**.

Click on the icon for the desired program.

1.2 Tasks That Must Always Be Running

Two tasks must always be running for the SPECTRA^{plus} software to function correctly. These are normally started automatically using the Windows Startup folder, but they can also be started manually using the shortcuts provided. Icons for these tasks should be visible on the right side of the Windows task bar as shown below.



The first task is the **Measurement** program. This program handles communications between the SPECTRA^{plus} software and the instrument. The icon for this program can be displayed in four different ways:

Circle is **Black**.	Displayed when the Measurement program is first started. Status of instrument is not known.
Circle is **Green**.	Displayed when the instrument is idle (not doing any measurements).
Circle is **Yellow**.	Displayed when the instrument is busy measuring samples.
Circle is **Red**.	Displayed when the instrument has a fault, or there was an error during a measurement.

The second task is the **Alarm Monitor** program. This program provides information on errors from the instrument.

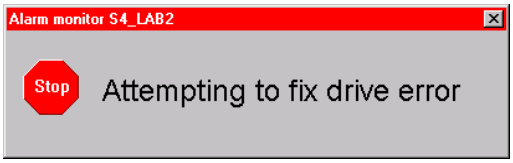
Displayed when there are no errors.

Displayed when the Measurement program is not communicating with the instrument.

Displayed when an error or warning has been detected from the instrument.

When an error has been detected from the instrument, the Alarm Monitor program displays a window giving information on the error and any corrective action that should be taken by the operator.

SPECTRA*plus* always attempts to correct most errors.



1.3 Programs Used for Routine Analysis

The following four programs are used by the operator for routine analysis of day-to-day samples. These are the only programs an operator typically needs to use.

The **Logon** program is used to log on and log off. An Operator must be logged on before measurements can be started. All measured results are maintained in an Access database. Part of the information recorded in this database is the Operator who measured the sample.

The **Results Monitor** program is used to display and print the results of measurements. This program is normally running all the time. Sample results are displayed, printed, and accepted or rejected using this program.

The **Loader** program is used to define the samples to be measured, to start and stop measurements, and to monitor the progress of measurements.

The **Status** program(s) are used to display a virtual view of the inside of your S4 system. It will be updated in real time.

1.4 Programs Used for “Standardless” Analysis

There are two programs that are used for “standardless” or semi-quantitative analysis. These are in addition to the programs already mentioned in Section 1.3, which are required to measure the samples.

The **MeasMethod Program** is used to define a *Measurement Method* that dictates the elements and lines to measure, the measurement strategy, the spectrometer mode for measurement, etc. Several pre-defined “standardless” Measurement Methods are delivered with the SPECTRA*plus* software.

The **Evaluation Program** is used to calculate sample composition using an interactive format. This allows the user to make decisions during the calculation phase that might improve the results.

1.5 Programs used for Quantitative Analysis

One single program is used to set up quantitative methods using a set of calibration standards. All other required programs can be launched from this single program (FQUANT).

The **Quantification Editor**, or **FQUANT**, is used to set up user-defined calibrations. All functions that need to be done as part of this process can be carried out from this one module. It uses a “Windows Explorer” -type interface that guides the user through the setup, calibration and validation process.

A *Material* is defined which specifies a list of elements or compounds present in the samples to be analyzed, and a list of *Standard Materials* with their concentrations for use in calibrating the instrument.

A *Preparation Method* is defined to indicate how the samples will be prepared.

A set of *Standard Samples* (or “Prepared Specimens”) is created by applying a Preparation Method to the set of Standard Samples.

From this point on, SPECTRA*plus* works with the element composition of the Prepared Specimens, since the Prepared Specimens (and not the original samples) are what is actually measured by the instrument.

The following programs are normally launched from FQUANT, rather than from clicking their icons.

The **MeasMethod** program uses the composition of the Standard Samples to create a Measurement Method, which can easily be modified by the user. This allows selection of the lines to measure based on the actual composition of the samples being measured, instead of the composition of the "original" samples.

The **MeasParameters** program can be used to optimize measuring parameters, to select peak and background measurement positions, and to visualize spectral overlaps for the lines in the Measurement Method.

The **Loader** program is used to measure intensities on the calibration standards, drift correction samples, and any "control" samples that will be used to validate the final calibration.

The **Model Setup** program can be used to define an *Evaluation Model* which links the files and other information needed to measure and evaluate samples in one place. The use of an Evaluation Model is optional. By default, SPECTRA*plus* links the needed files by their names:

Measurement Method file name =
Calibration file name =
Results Format file name, etc.

The **Results Manager** program is used to define the output format of results displayed and printed by the Results Monitor program. This includes options like output order, concentration units, number of decimals, etc.

FQUANT is then used to calculate the calibration coefficients using "Fundamental Parameter" or "Empirical" -based mathematical models to correct for "matrix" effects if needed.

The following program can be launched from **FQUANT**, rather than clicking its icon.

The **Evaluation** program can be used to validate the calibration by calculating the composition of “control” samples that were measured with, or after, the calibration samples.

1.6 Additional Programs and Utilities

The **Specification Editor** program is used to define limits and/or alloy specification (type standardization).

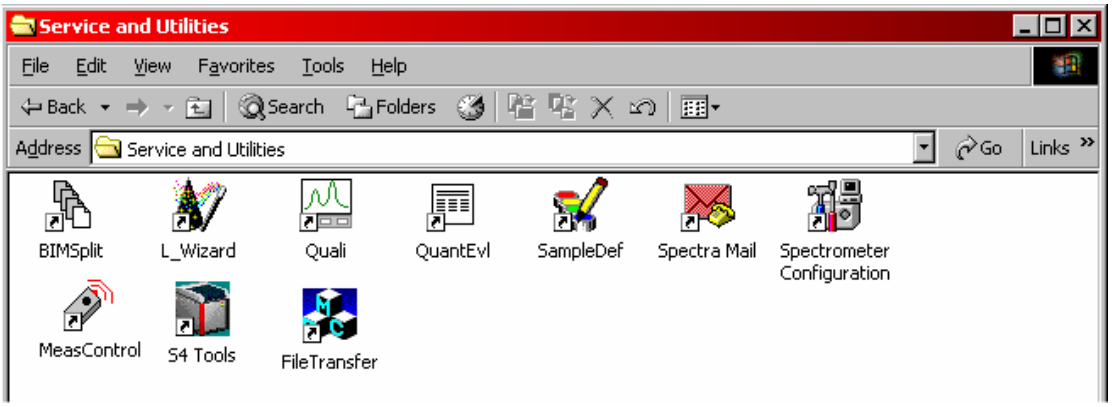
This utility can be used to define upper and lower warning and alarm limits for check samples or materials. It writes the definition into the Specification database.

The **User Modules** program is used to define modules which use the data obtained from the calibration to calculate user-defined values. This is to create calculations such as the Bogue clinker composition or other ratios.

The **Line Library Manager** program is used to inspect and manage the line library. It allows the user to check for drift correction data, import and export lines, and copy details to the clipboard.

The **Spectrometer Alignment** program is used to align the instrument. It realigns and sets the high voltage for the detectors, performs the goniometer alignment, and adjusts the peak positions. It is possible to start the drift correction for the 34mm mask size with this application.

The “Service and Utilities” folder on the desktop contains the following additional shortcuts. All applications mentioned above can be found by navigating to the \SPECPLUS\ directory on your hard disk when not stated otherwise.



The **S4Tools** program (located under \SPECPLUS\S4Tools\) is used to monitor the hardware of the system as well as to troubleshoot the unit.

The **MeasControl** program (formerly known as DIRECT COMMANDS) is used to communicate directly with the instrument using two-letter commands.

The **Spectrometer Configuration** application manages the system and password configuration of SPECTRA*plus* and the S4 system.

The **Spectra Mail** application is used to extract all relevant data for an application from your system and databases (a .spm file) so that you can email them to the support hotline.

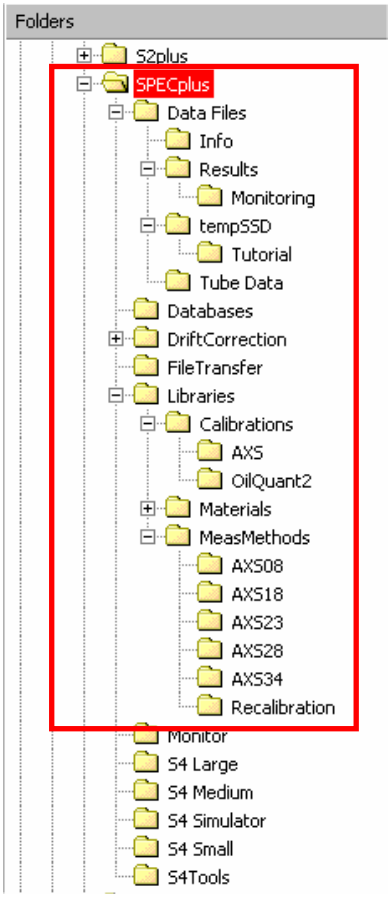
The **BIMSPLIT** utility can divide and reassemble a .zip or .spm file into “chunks” so that they can fit onto a floppy diskette or make up email-friendly attachments.

The **L_WIZARD** utility is used to manage the copy-protection license for SPECTRA*plus*. With this utility, you can recreate a license (with an emailed code) as well as check the types and number of licenses.

The **SampleDef** application enables you to modify and create .def files used by the Loader. These .def files control the functionality of the Sample Edit window.

The **LIBMANAGER** utility is used to manage and inspect the lines in your Line Library.

1.7 File Structure and File Locations of SPECTRA*plus*



Directories in the red box (including the subdirectories) should be backed up using the PC's internal CD burner on a regular schedule. These directories contain the USER DATA.

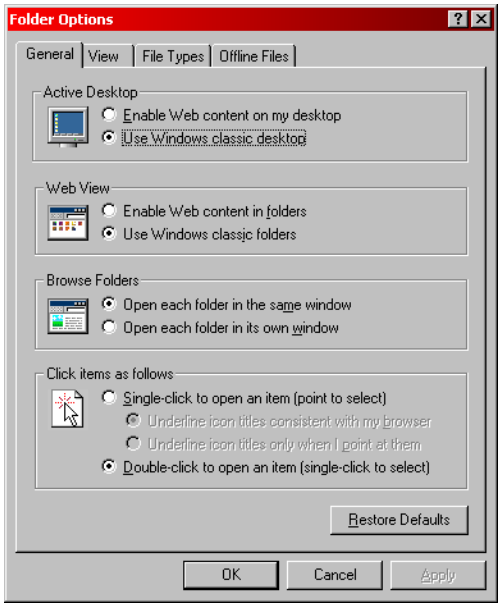
1.8 Table of File Types used in SPECTRA*plus*

File Extension	Description	Directory \SPECPLUS\
CNF	Configuration file	Libraries
MDB	Database file	Databases
FLL	Line Library	Libraries
XRD	Parameter files	Libraries
MM	Measurement Methods	Measmethods (also in AXS and Driftcorrection subdirectories) As a rule, user-created quantitative or qualitative Methods should be stored under \Measmethods, whereas all Methods pertaining to the Standardless Method should be stored in their respective AXS subdirectories according to the used mask size.
WZM	Query Measmethod Wizard file	Measmethods
ML	Query Mapping List file	Measmethods
DEF	Loader Definition File	Measmethods
RTF	Rich Text Format files for merging results into formatted word reports.	Measmethods
MLB	Module file	Measmethods
SSD	Raw data file from a measurement	TempSSD and Materials
FCL and FQN	Calibration files	Calibrations
EVM	Evaluation Model	Calibrations or Calibrations\Axs
ERR	Error log files	Info
TSV	Summary Alignment files	Info
LOG	Application Log files	Info
STS	Service Tools Status files	S4Tools
EXE	Executables	SPECPLUS
Others	Internal System files	SPECPLUS

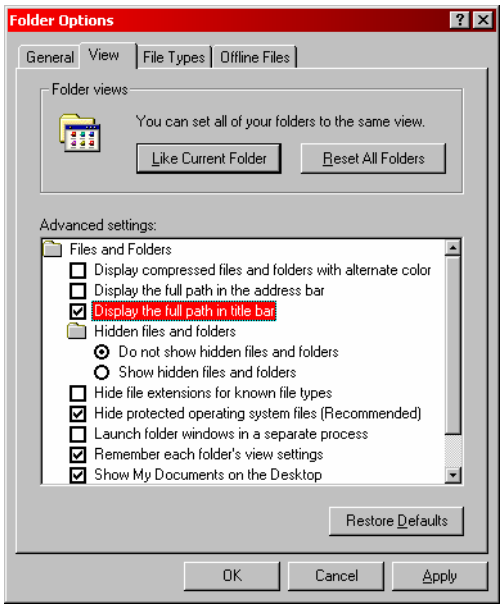
1.9 Recommended Settings for Windows

The following folder options are recommended when using the Explorer to navigate in Windows:

1. Within Windows Explorer, select the Tools menu.
2. Select “Folder Options”.
3. Select and inspect the General tab.



4. Select the View tab.



5. Make sure to select “View Details”.

2 Standardless Program

This section covers the Standardless Program that is part of the SPECTRA^{plus} software. The Standardless Program enables virtually any sample to be quickly analyzed, and an estimated composition calculated from the data. It is most useful for samples that are encountered infrequently in the laboratory, where calibration standards may not be available. Even if standards are available, the total number of samples to be measured may not warrant the effort to make a specific calibration. It is also referred to as a universal calibration.

2.1 Using the Standardless Program

Measuring “Unknown” Samples Using a Standardless Method

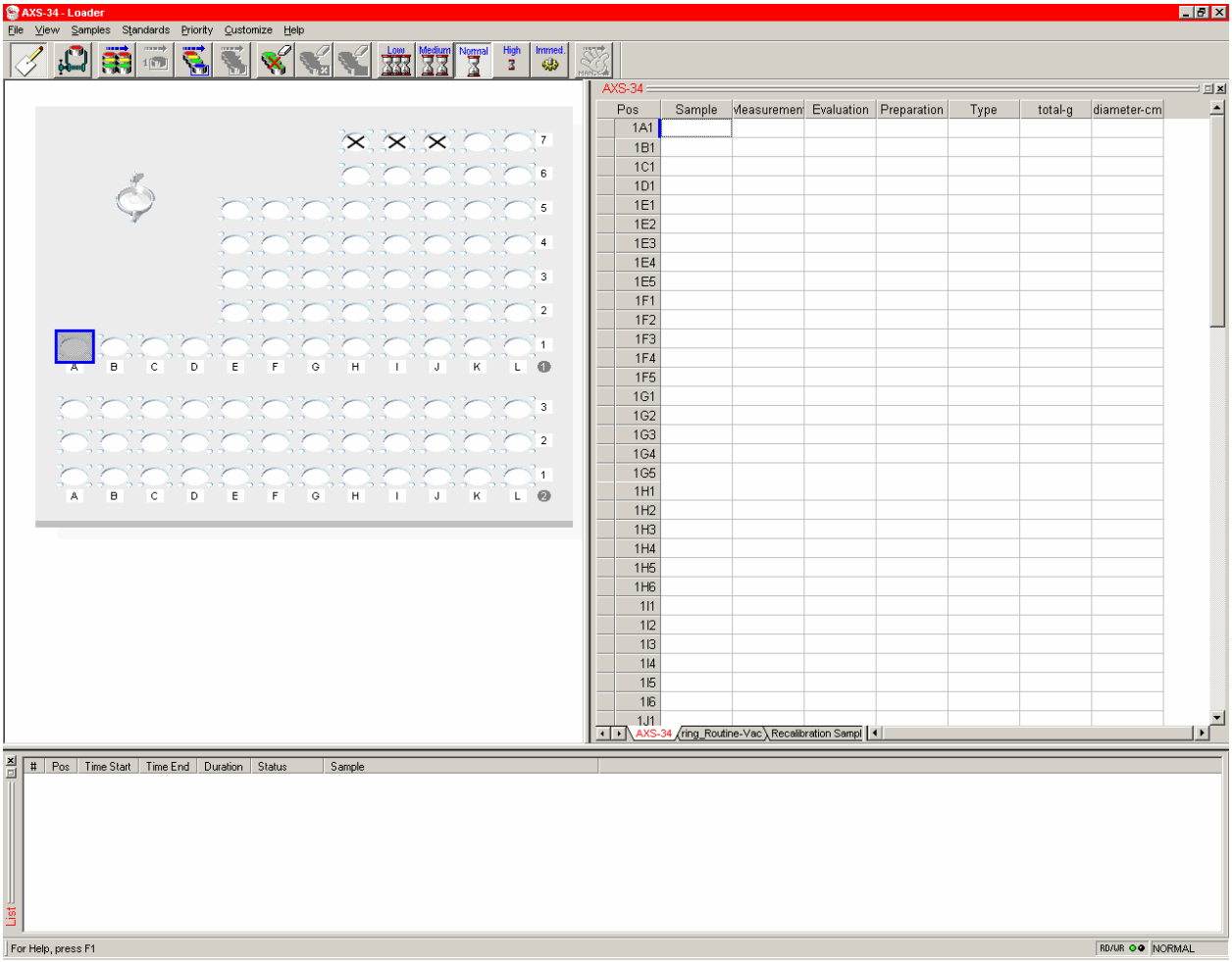
Making a “standardless” measurement on a sample is no different than making a “normal” measurement on a sample. The only requirement is that the Measurement Method selected to measure the sample contains a selection of “pre-calibrated” lines from the Line Library, since these are required to calculate the sample composition. In order to separate standardless Methods from quantitative Methods we use different Loader definition files, which will show us only the standardless Methods for a given mask size.

Run the **Loader** program.

Clear any samples, if necessary, using the icons “Clear New”, “Clear Error” and “Clear Done”.

Click **View** on the menu, and check the options as shown below (all options are checked).

Arrange the windows as shown, or to your own preferences.



To use the provided standardless Methods, please use the following definition files:

34mm mask AXS-34

28mm mask AXS-28

18mm mask AXS-18

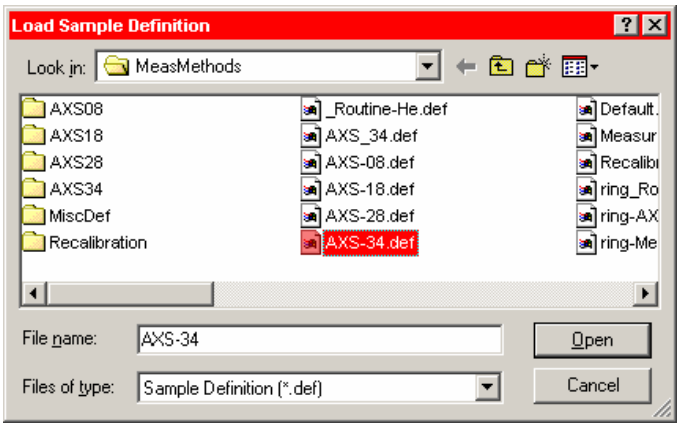
23mm mask AXS-23

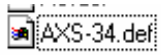
08mm mask AXS-08

If the “AXS-34” tab is not shown, select **Samples→Definition File** from the menu.

Click the **drop-down arrow** in the “Look in” box.

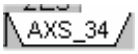
Make sure the “**\SPECplus\Libraries\MeasMethods**” folder is selected.



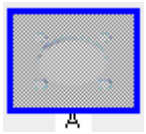


Select “**AXS-34**”.

Click the **Open** button.

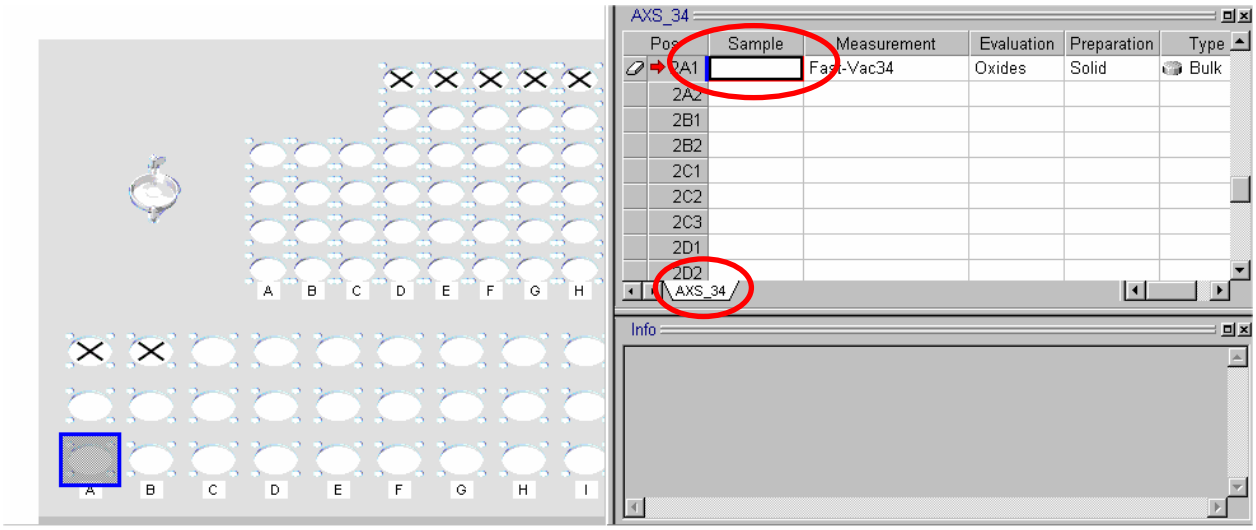


Click on the **“AXS-34”** tab to select it.



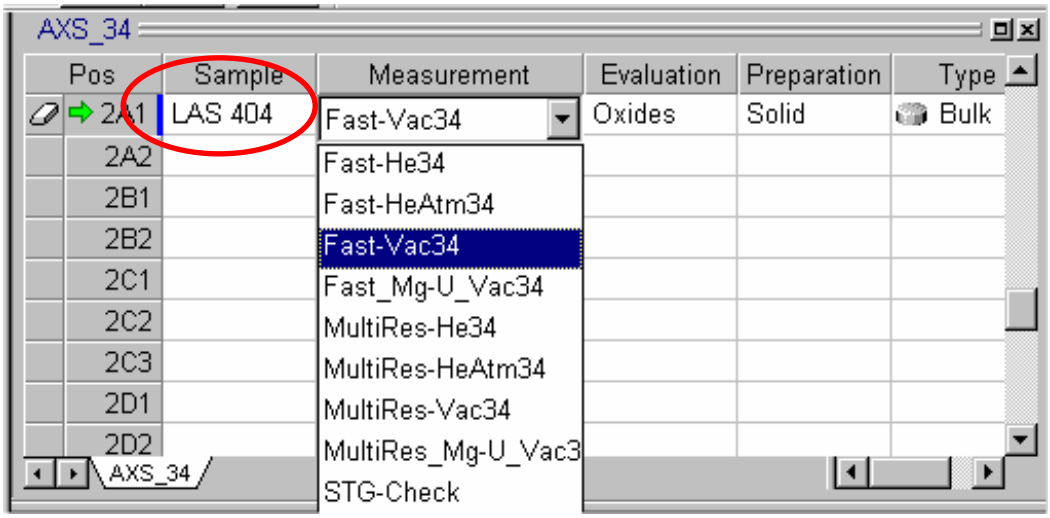
Right-click on the sample position where the first sample is loaded.

Click on the **Edit Sample** button.



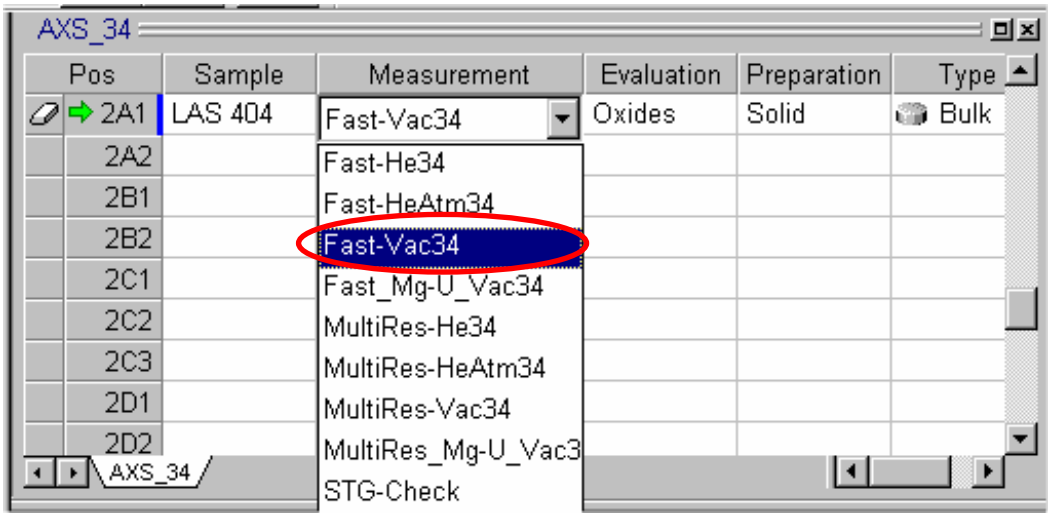
LAS 404

Type the name for this sample in the **Sample** column, then press the **<Tab>** key.



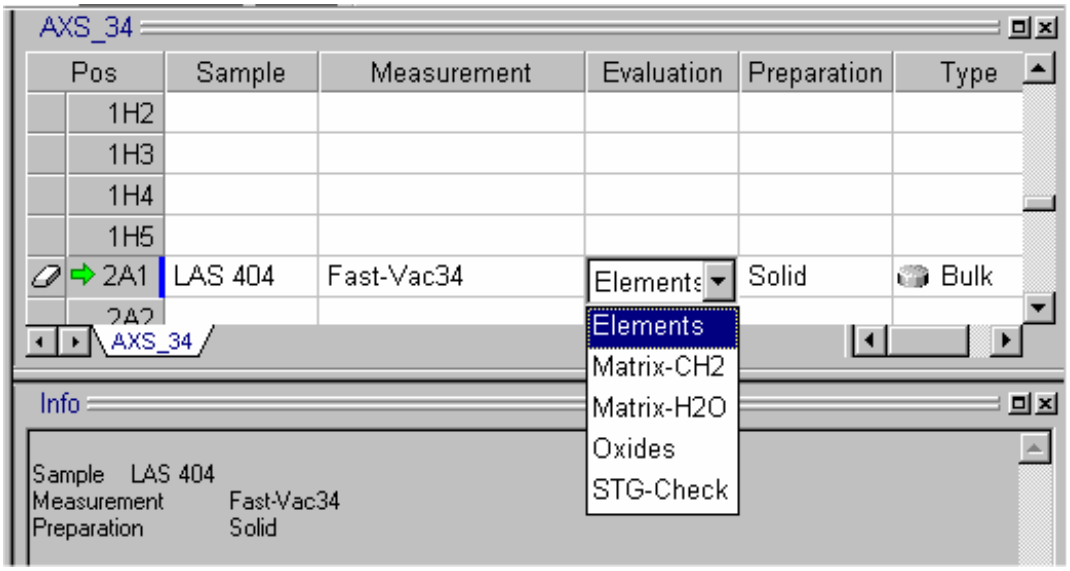
Fast-Vac34

Select one of the displayed Methods with which to measure this sample (Fast-VAC34).



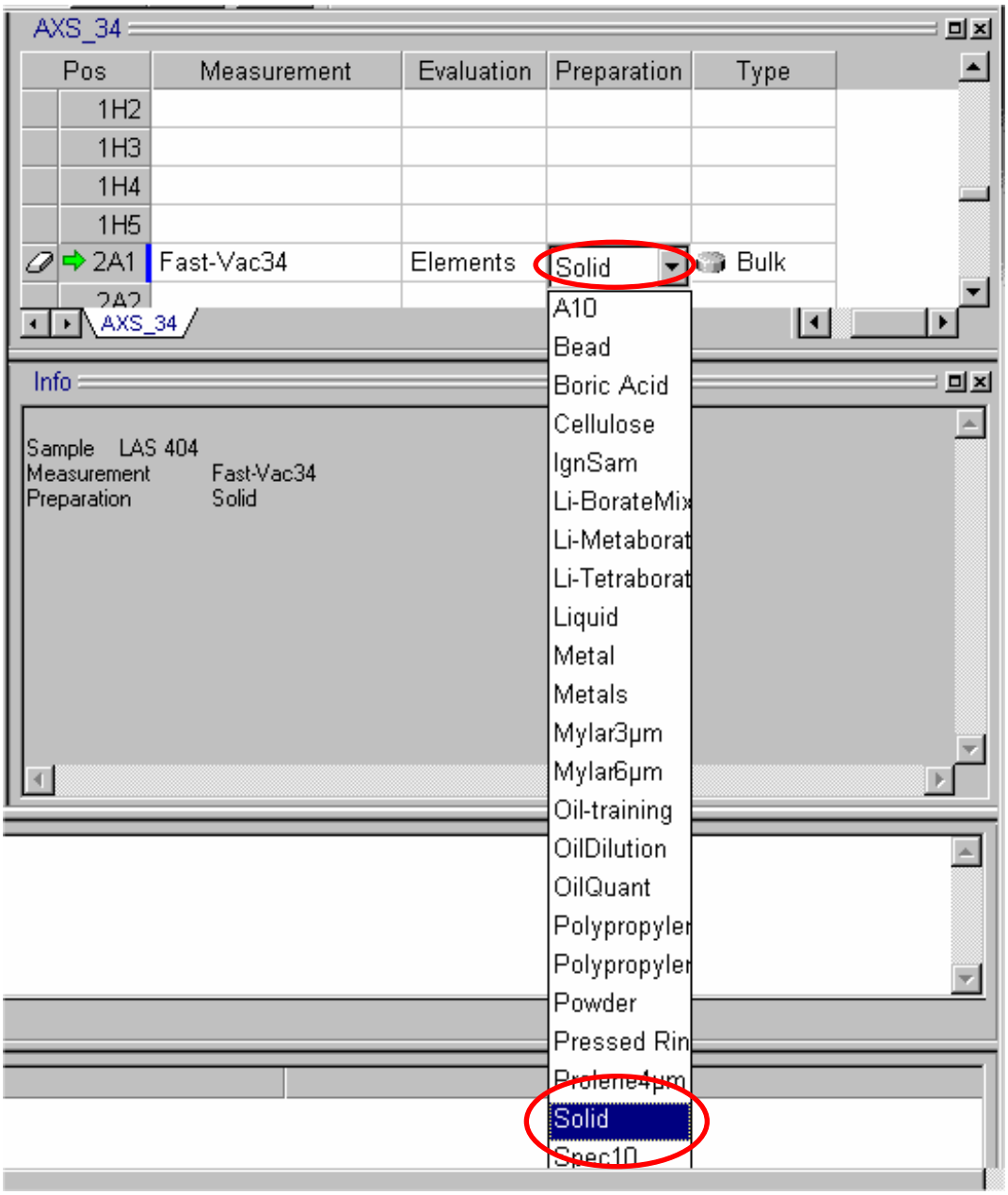
Click in the **Evaluation** box to open the drop-down list.

Select **“Elements”** as the type for this sample.



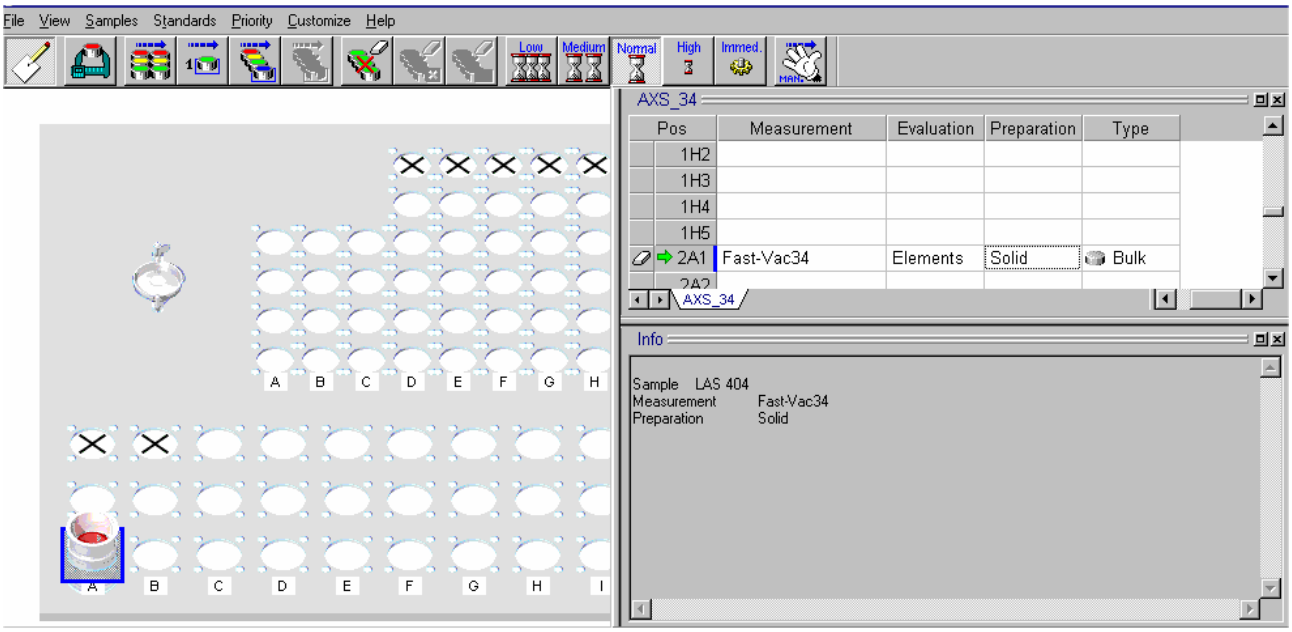
Preparation Click in the **Preparation** box to open the drop-down list.

Solid Select **"Solid"** as the preparation for this sample.



Type Click in the **Type** box to open the drop-down list.

Bulk Select **"Bulk"** as the type for this sample.



Additional fields for weight of sample (in g) and weight of additive (in g) are included in the newer Def files for AXS*. They will be pre-filled with the default value for the preparation.

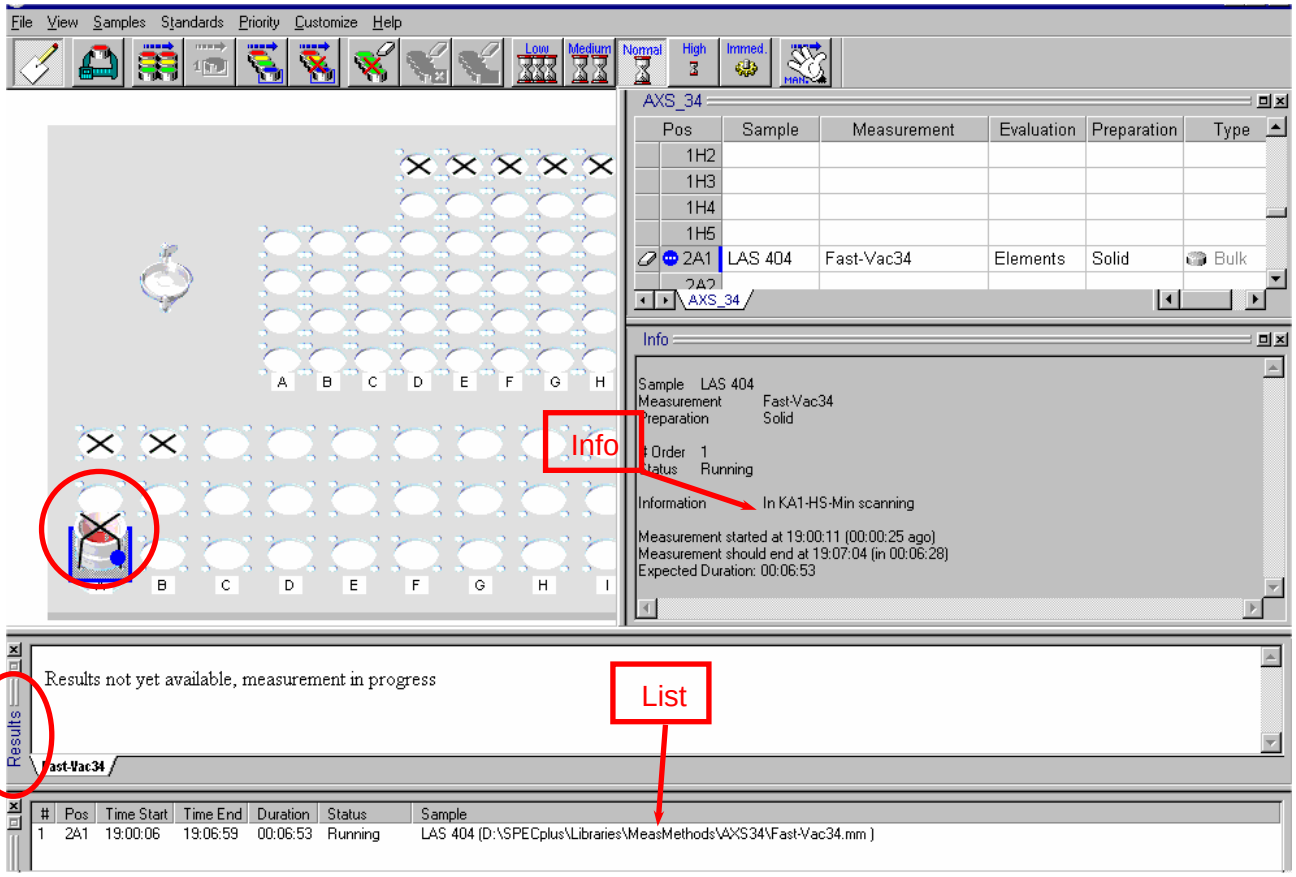
Click the **"Send All Samples"** icon to start the measurement.

Once the sample has been loaded, the measurement will start. This is indicated by the blue circle with the moving dots seen on the sample holder.

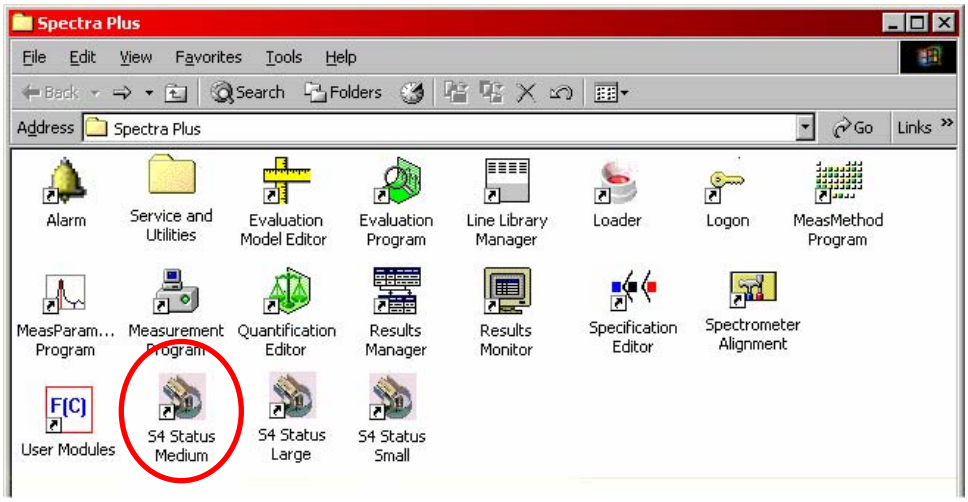
The Sample Info window will show the current status of the selected sample (line being measured, time left, etc.).

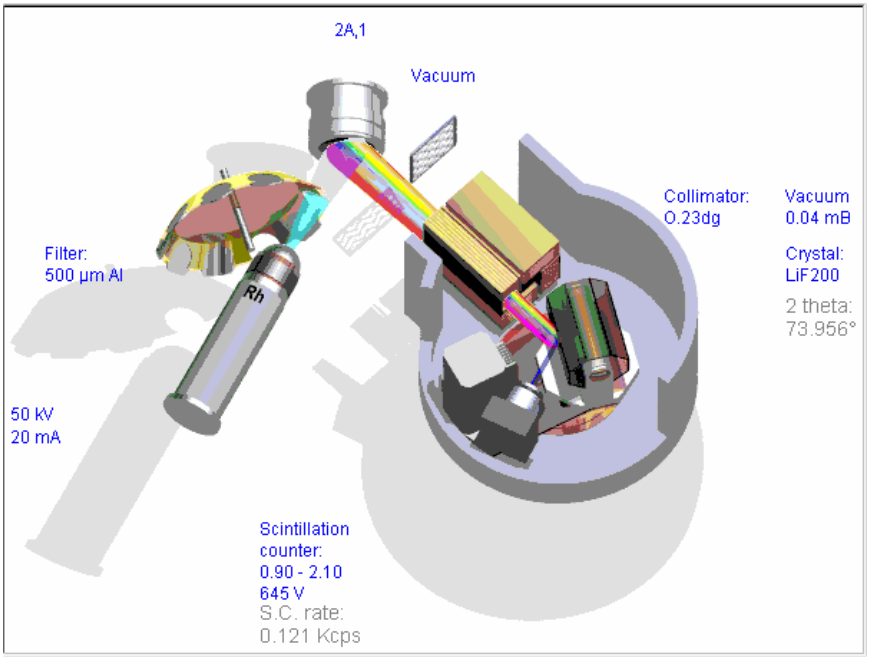
The Sample List window shows the current sample queue.

The Results window will show the results based on the automatic evaluation of the selected sample.



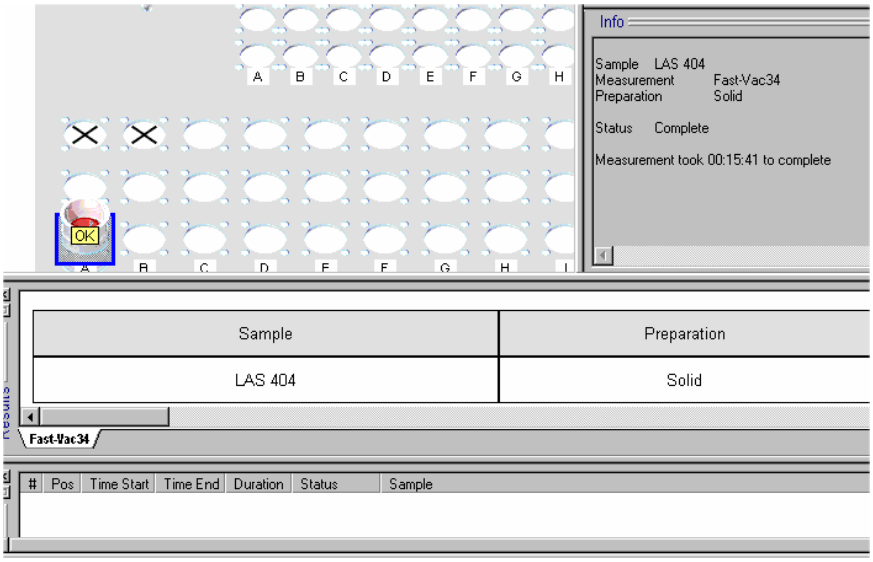
Select the **Status** display from the SPECTRA^{plus} folder on the desktop and follow the measurement “online”.





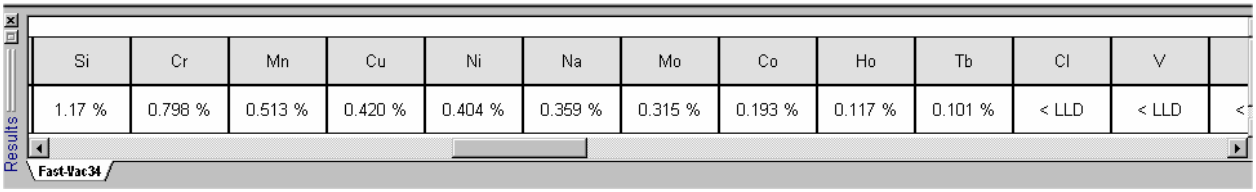
Close the Status display when done and switch back to the Loader.

When the measurement of the sample has completed, the sample holder should have an **"OK"** displayed on it.



Click on the sample and observe the Results View of your Loader screen.

Use the navigation bar to slide to the right to see all the automatically-found elements and their concentrations.



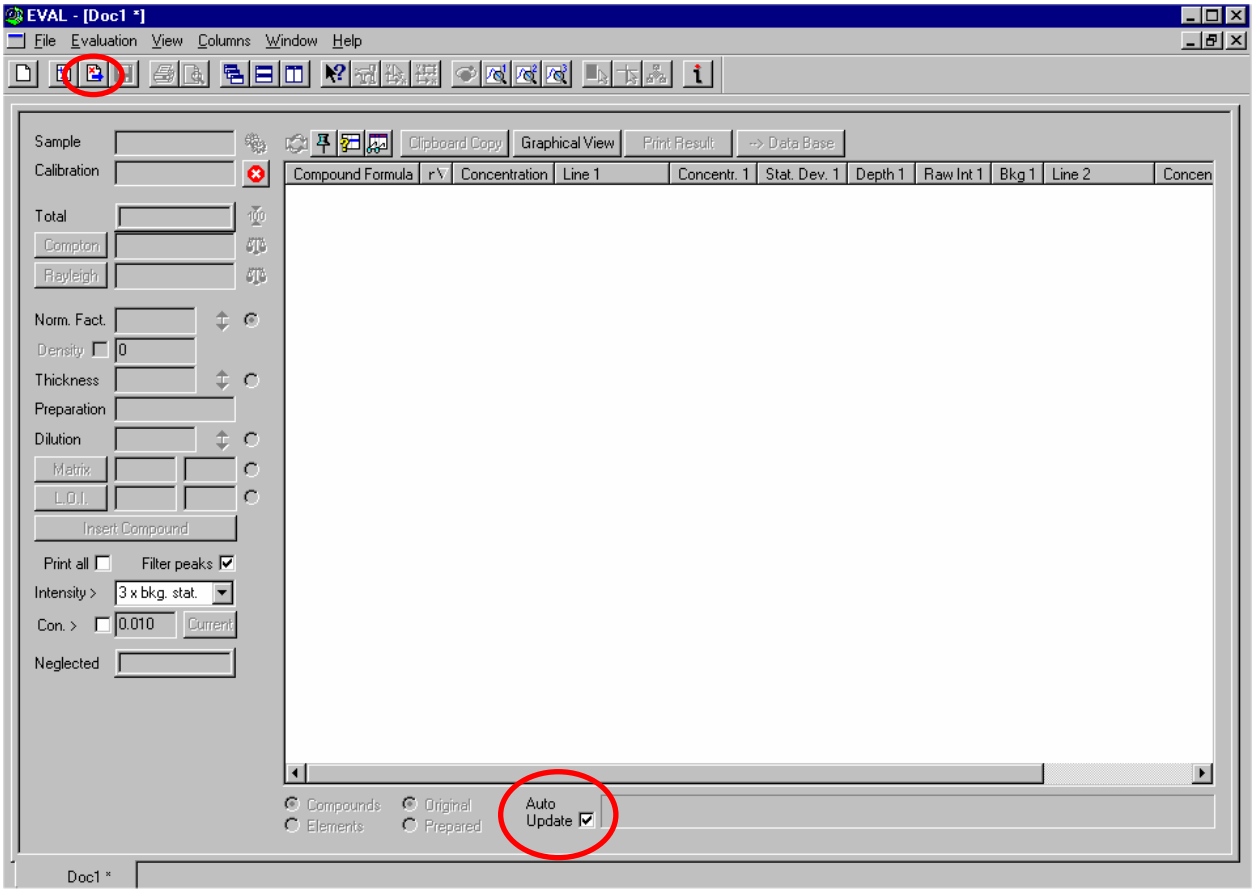
2.2 Calculating Standardless Results for an Unknown Sample

This section covers the interactive evaluation of a sample's composition using the SPECTRA^{plus} software. It uses the data from a "low-alloy steel" sample measured with the Standardless Measurement Method described in Section 2.3, and measured as outlined in Section 2.1. As you work through this example, your results will be different than those shown because your results are based on a different measurement of the sample, and a different drift correction of the Standardless Program. Nonetheless, the concepts presented can be used with any similar set of data.

Run the **Evaluation** program.

Make sure the **Filter Peaks** box is checked.

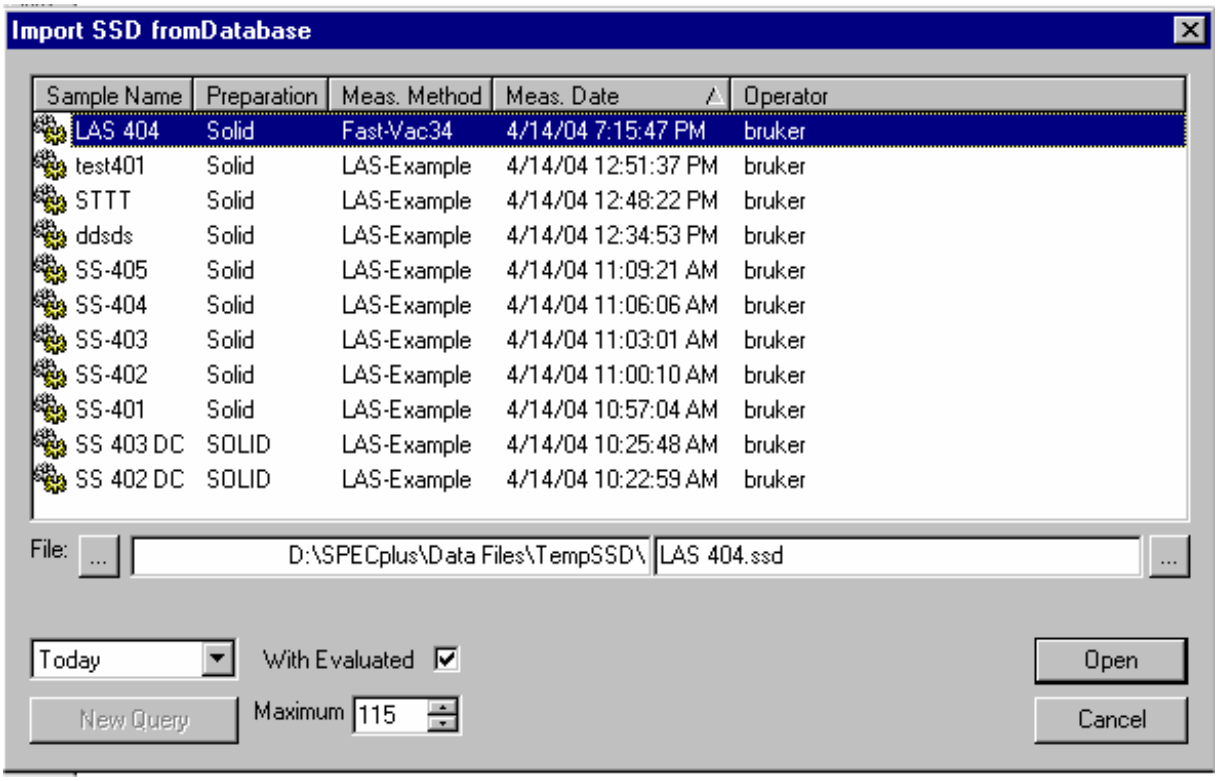
Click the "**Next SSD File**" icon.



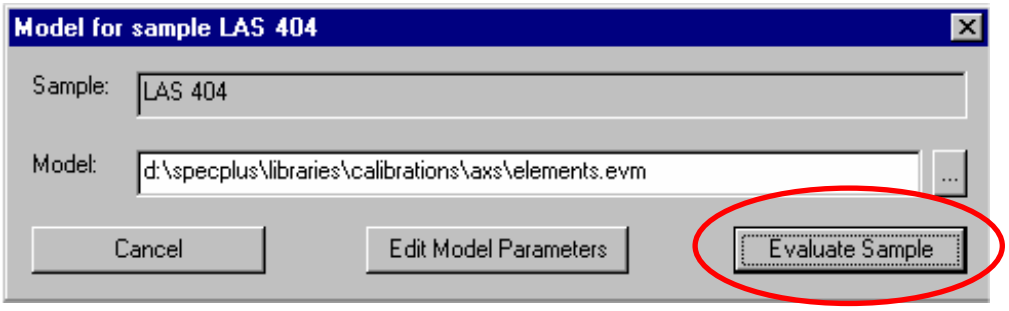
Set the **Search Control** to when the sample was measured (e.g., “Today”, “Yesterday”, etc.).

LAS 404 Highlight the sample name **“LAS 404”.**

Click the **Open** button.

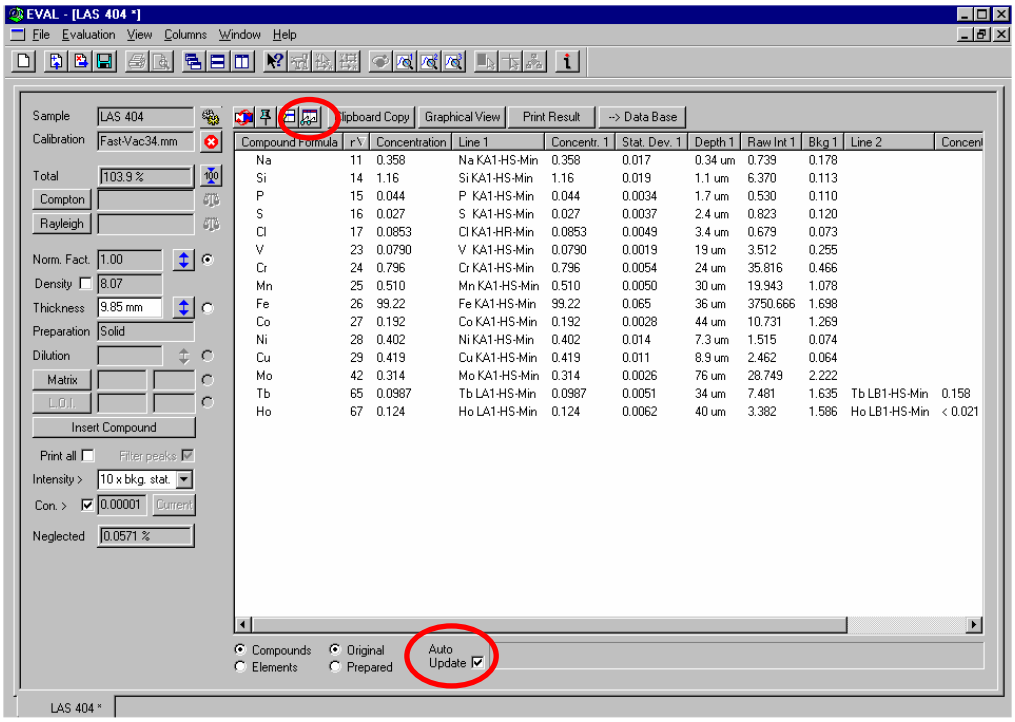


Click the **Evaluate Sample** button.



Make sure the **Auto Update** box is checked. This will tell SPECTRA^{plus} to recalculate the sample composition as changes are made to the assumptions about this sample.

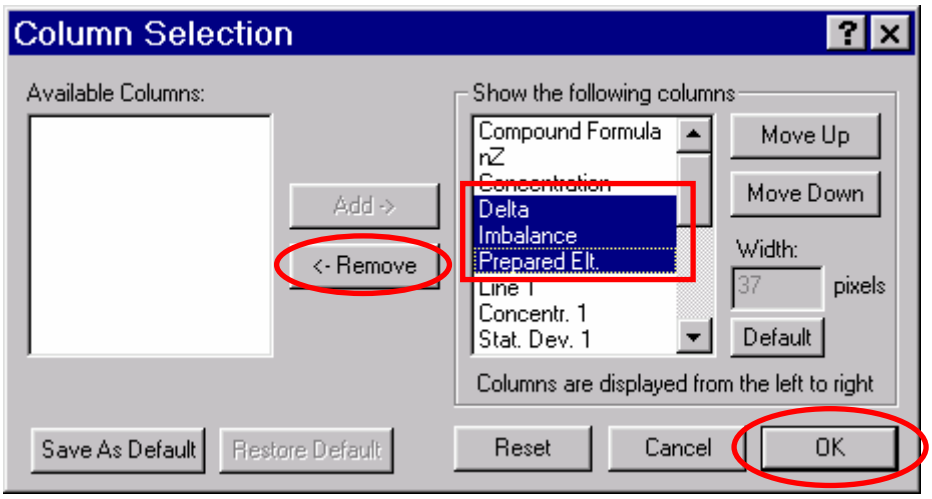
Click the "**Eyeglass**" icon.



Click the **Reset** button.

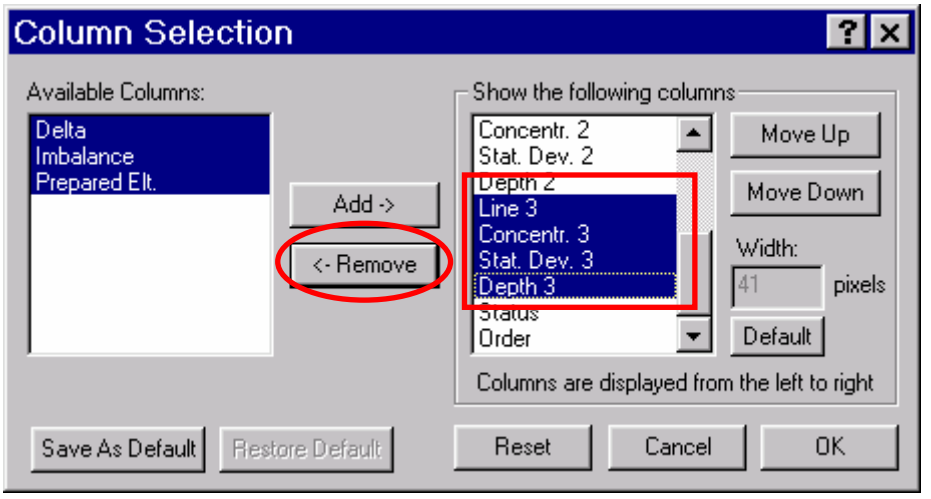
Highlight **“Delta”**, **“Imbalance”**, and **“Prepared Elt.”** by clicking on one, then holding the **<Ctrl>** key down while clicking on the others.

Click the Remove button.



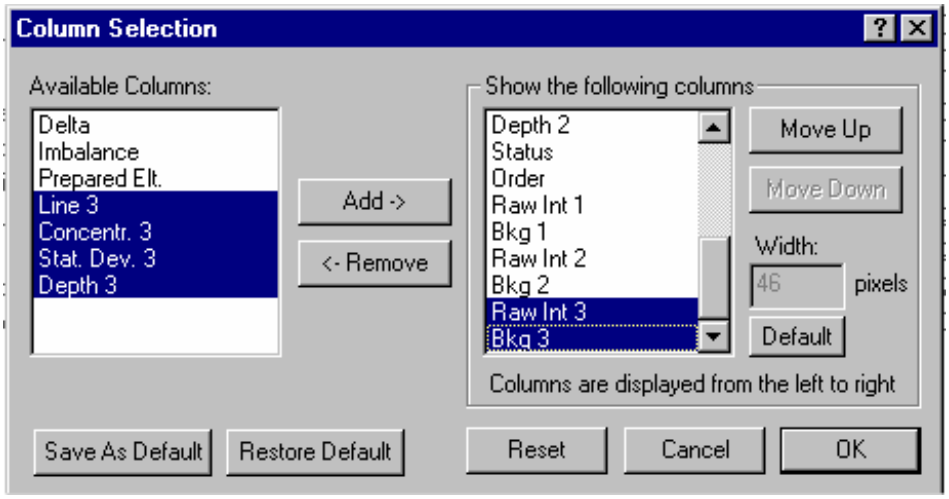
Highlight **“Line 3”**, **“Concentr. 3”**, **“Stat. Dev. 3”** and **“Depth 3”**. The Measurement Method used to measure the sample measured a maximum of two lines.

Click the Remove button.

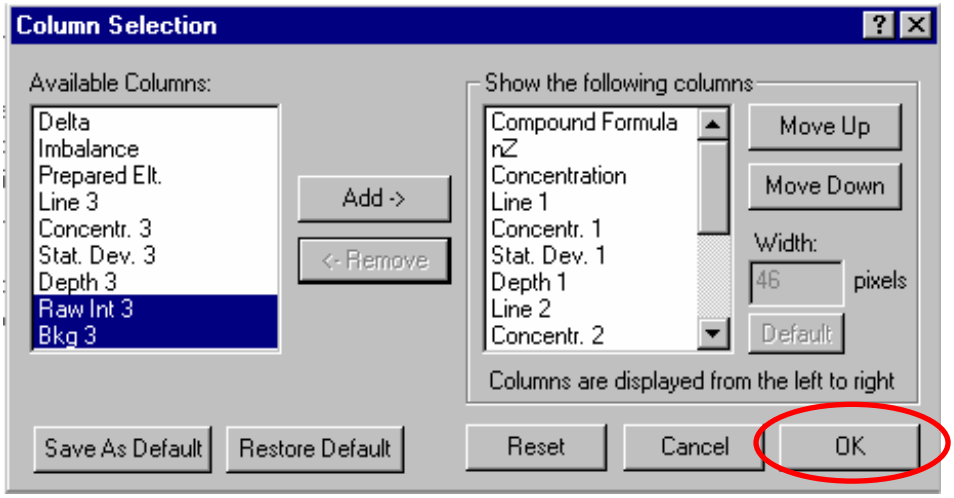


Highlight "**Raw Int 3**" and "**Bkg 3**".

Click the **Remove** button.



Click the **OK** button.

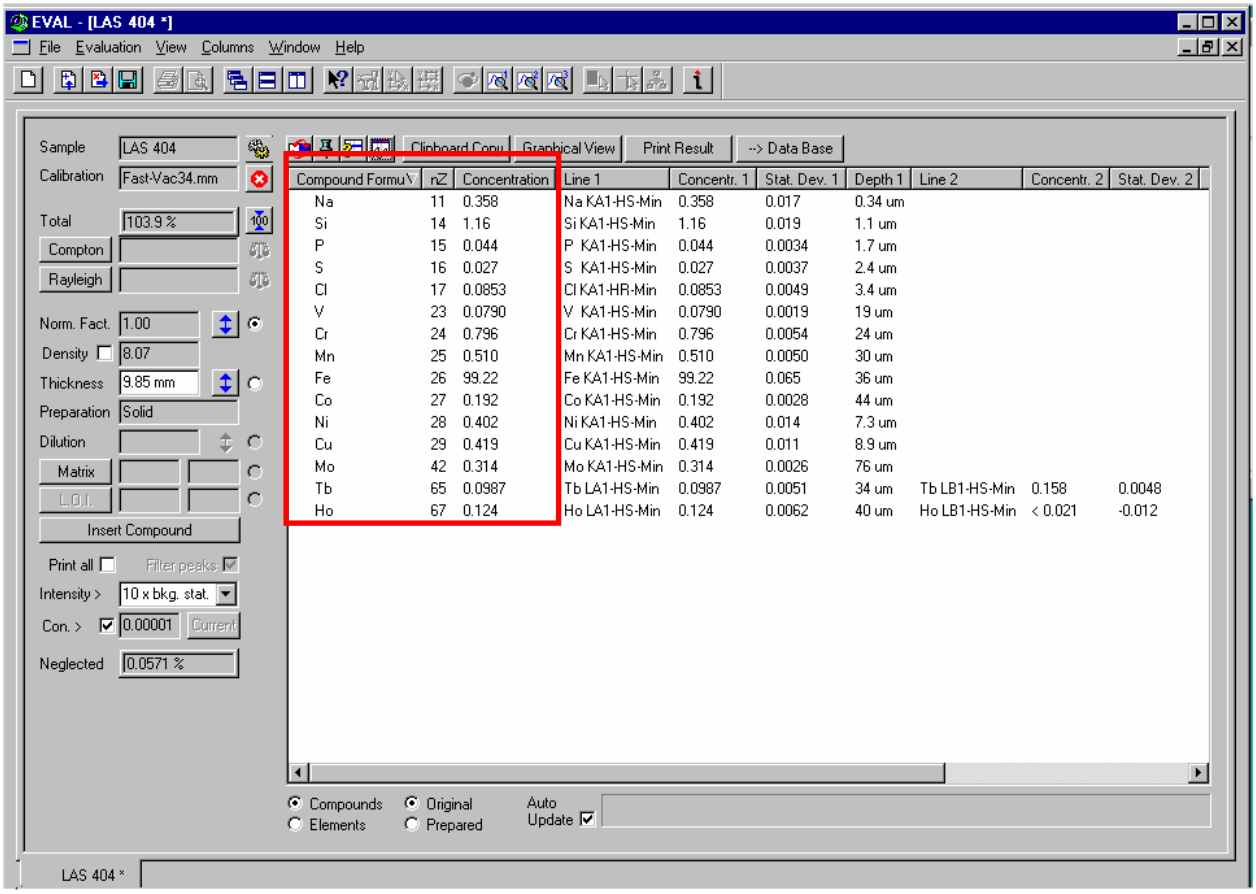


Click the "**Save as Default**" button to save these settings as the default.

The calculated composition for this sample should now be listed as shown below. The columns have the following meanings:

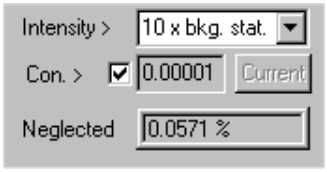
- Compound Formula — The reported element or compound name.
- nZ — The atomic number of the reported element, or **key** element from a compound.
- Concentration — The reported concentration for each element or compound listed.

Note that sorting can be done on any displayed column by clicking on the column header which toggles between a forward and reverse sort of the data.



For up to 3 measured lines, the following information is displayed for each line:

- Line n — The name of the line measured.
- Concentr. n — The concentration calculated from the intensity of the measured line.
- Stat. Dev. n — The error in the concentration from counting statistics for the measured line.
  This is an indication of the *reproducibility*, not the *accuracy*, of the results.
- Depth n — The estimated depth in the sample where 90% of the measured radiation is coming from (“effective layer depth”).



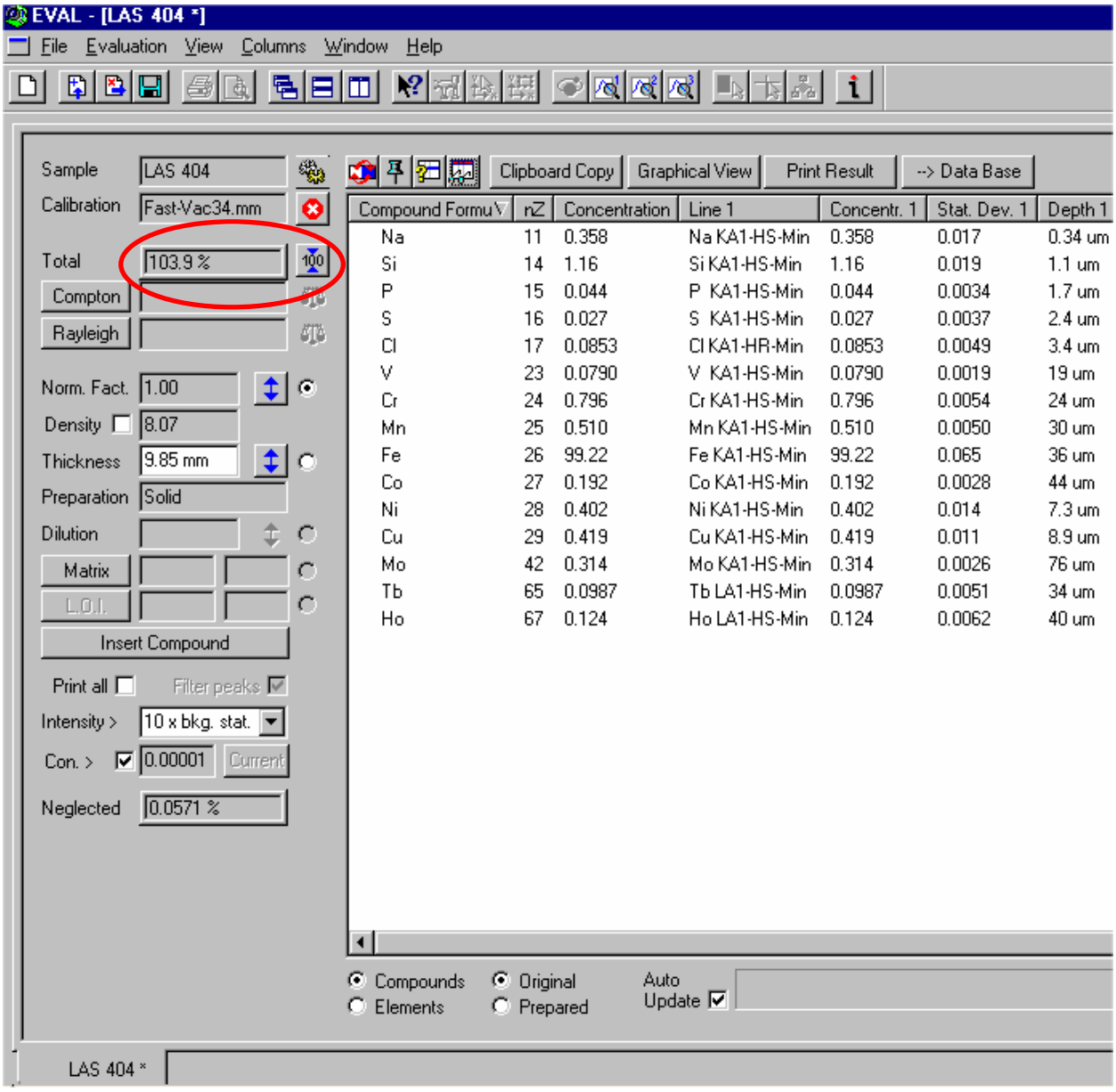
SPECTRA*plus* uses filters to display only significant concentrations calculated from a sample.
The following two filters are used:

- Intensity > Sets how many standard deviations a peak intensity must be above the background intensity in order to have its calculated concentration displayed. This prevents displaying concentrations for elements that have statistically insignificant peaks.

- Con. > Sets an absolute concentration threshold that must be exceeded before a calculated concentration is displayed. This can be used to limit the display of concentrations to only significant values.

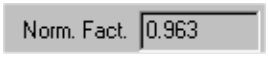
- Experiment with these controls to see what effect they have on the displayed concentrations.

- When you have finished, reset them as shown in the diagram below.



The sum of the concentrations is displayed in the **Total** box. The closer this sum is to 100%, the more confidence one can have in the calculated results.

Click the **Optimize** icon to normalize the concentrations to 100%.



The total should now be 100%, and a normalization factor will be displayed in the **Norm. Fact.** box.

Click the **Reset** icon to reset the “Normalization Factor” to one and recalculated the concentrations.

At this point, we will start making decisions about how the sample composition will be calculated. If these decisions match the “reality” of the sample we should obtain good results, and if these decisions do not match the “reality” of the sample the results obtained will be questionable.

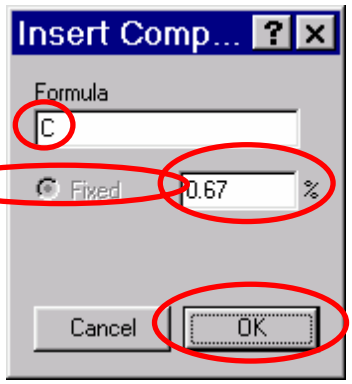
If any compositional information is known about this sample, it should be entered to improve the calculated composition. For example, this sample has 0.67% Carbon.

Click the **Insert Compound** button.

Type **“C”** in the Formula box.

Type the concentration for carbon (**“0.67”**) in the Fixed box.

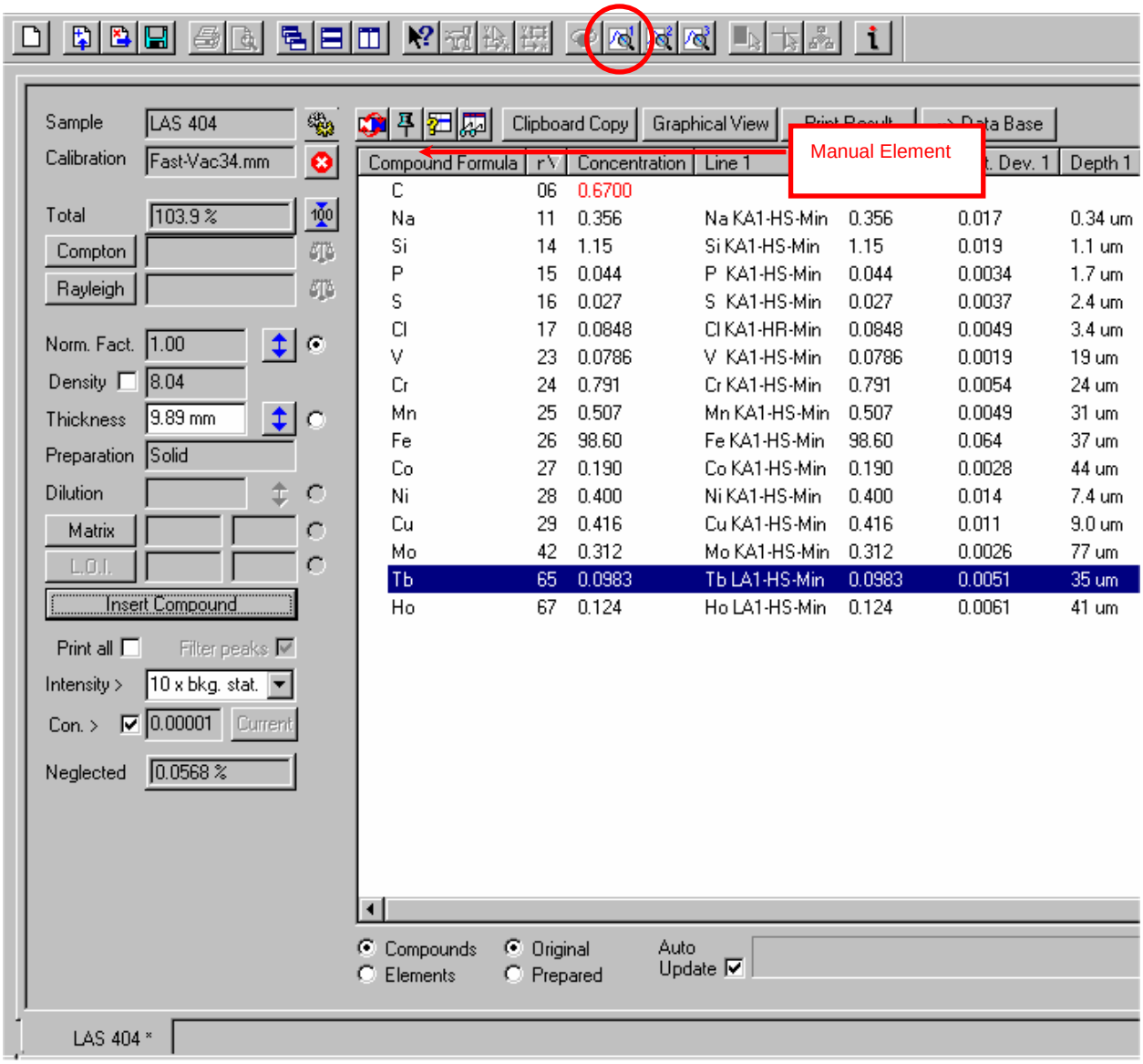
Click the **OK** button.



The concentration for Carbon has now been fixed to 0.67%, and it is displayed in red to indicate this.

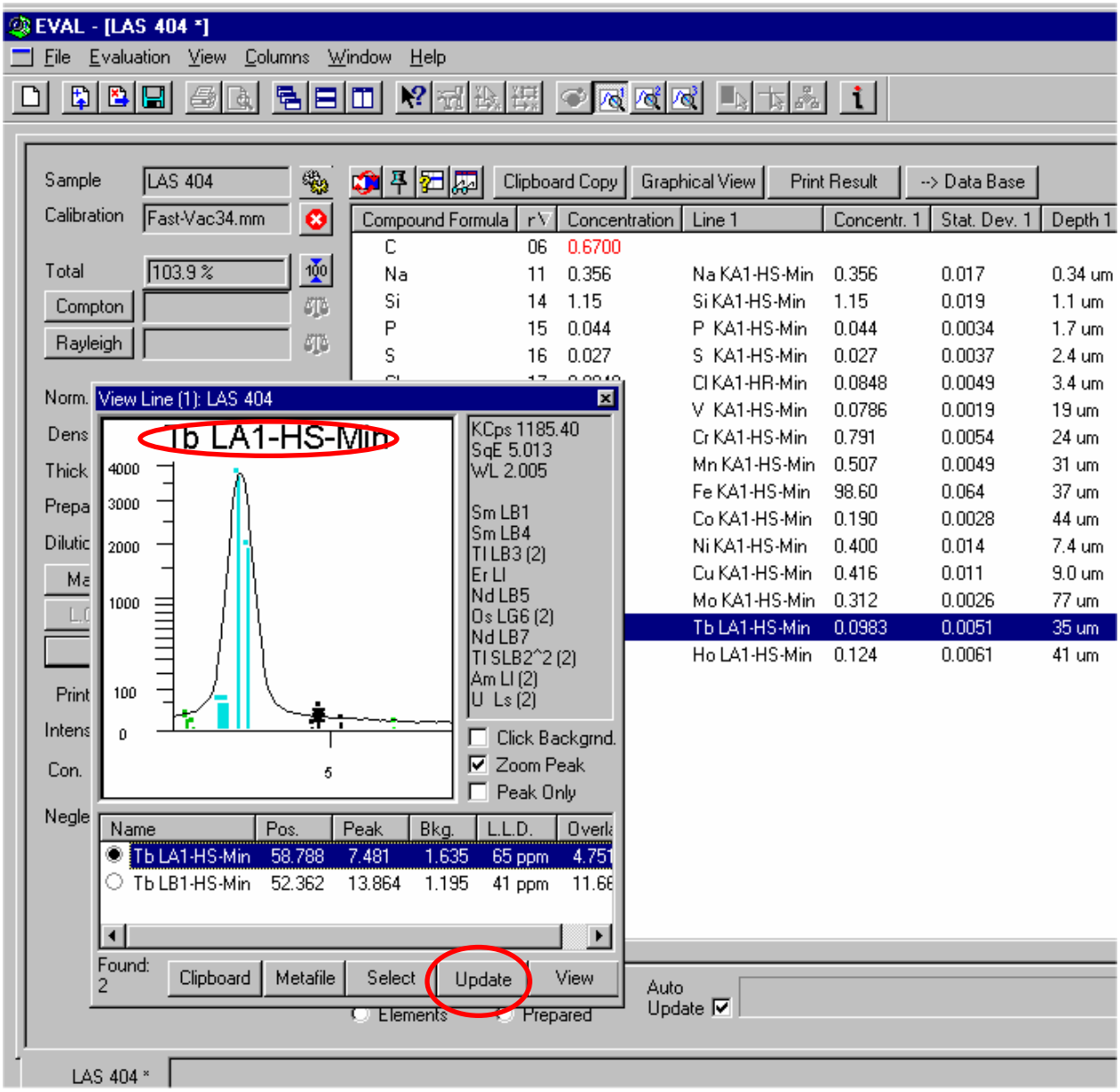
All concentrations that are calculated by the SPECTRA^{plus} software should be verified by the user to ensure that they should be reported. This is easiest when the data has been measured as scans, which is the case for this sample. The measured spectra can be viewed on the screen to ensure that a peak can be seen, and that the peak is from the element in question and not from some other element present in the sample.

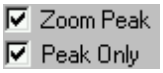
Click the "**Toggle Line 1**" icon to open a small window for viewing the spectra.



Tb — Click on the **"Tb"** line to highlight it (or click on one of the other lines with a low concentration).

Update — Click the **Update** button in the View window to display the intensity data for Terbium.





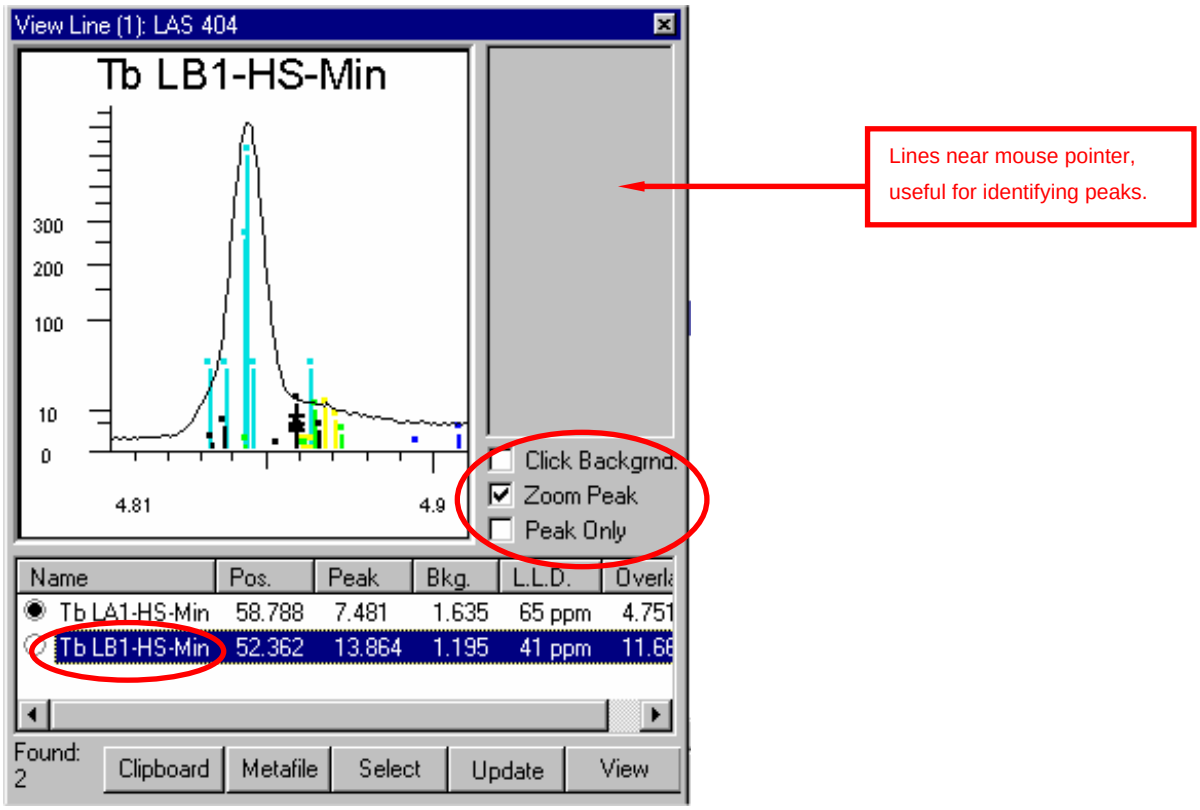
Make sure the **Zoom Peak** and **Peak Only** boxes are checked.

This is the spectra for the **"Tb-LA1-HS-Min"** line, it does not look like a peak is really present.

Note that when the mouse pointer is moved over the data, a list of lines near the mouse pointer is displayed in the upper right corner of the View window.

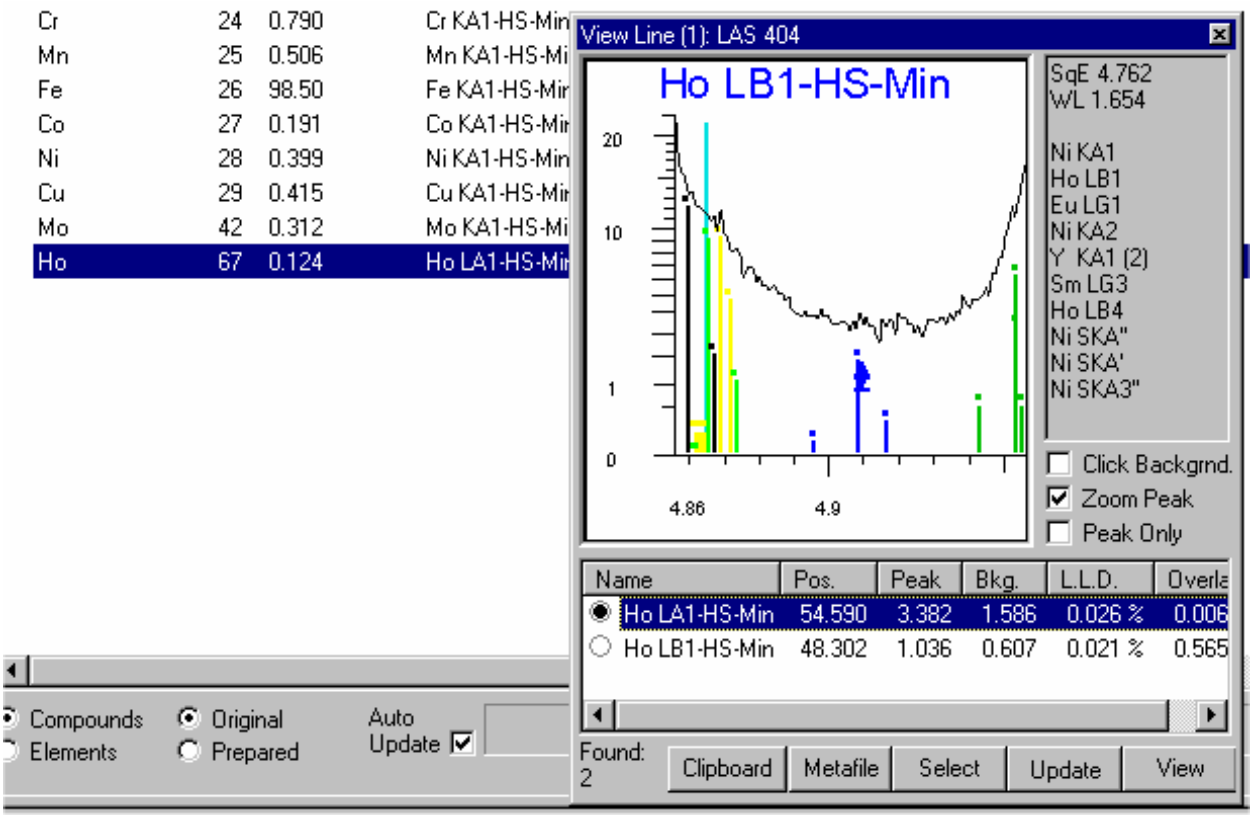
Tb LB1-HS-Min

Click on the **"Tb-LB1-HS-Min"** line to view its spectra.



This is the spectra for the **"Tb-LB1-HS-Min"** line; it does not look like a peak is really present either.

Click the **Push Pin** icon to set this concentration to zero.



Display the spectra for Holmium **("Ho")**:

Click on the **"Ho"** line to highlight it.

Click the **Update** button in the View window to display the intensity data for Holmium.

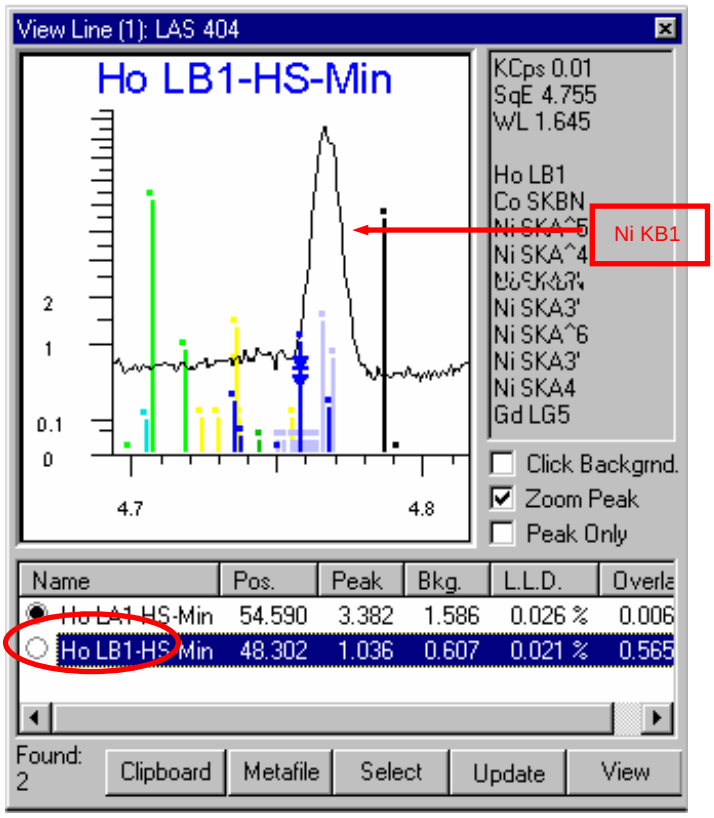
There is no apparent peak.

Ho LB1-HS-Min

Click on the **"Ho LB1-HS-Min"** line to see if it is more usable.

Click the **Push Pin** icon to set this concentration to zero.

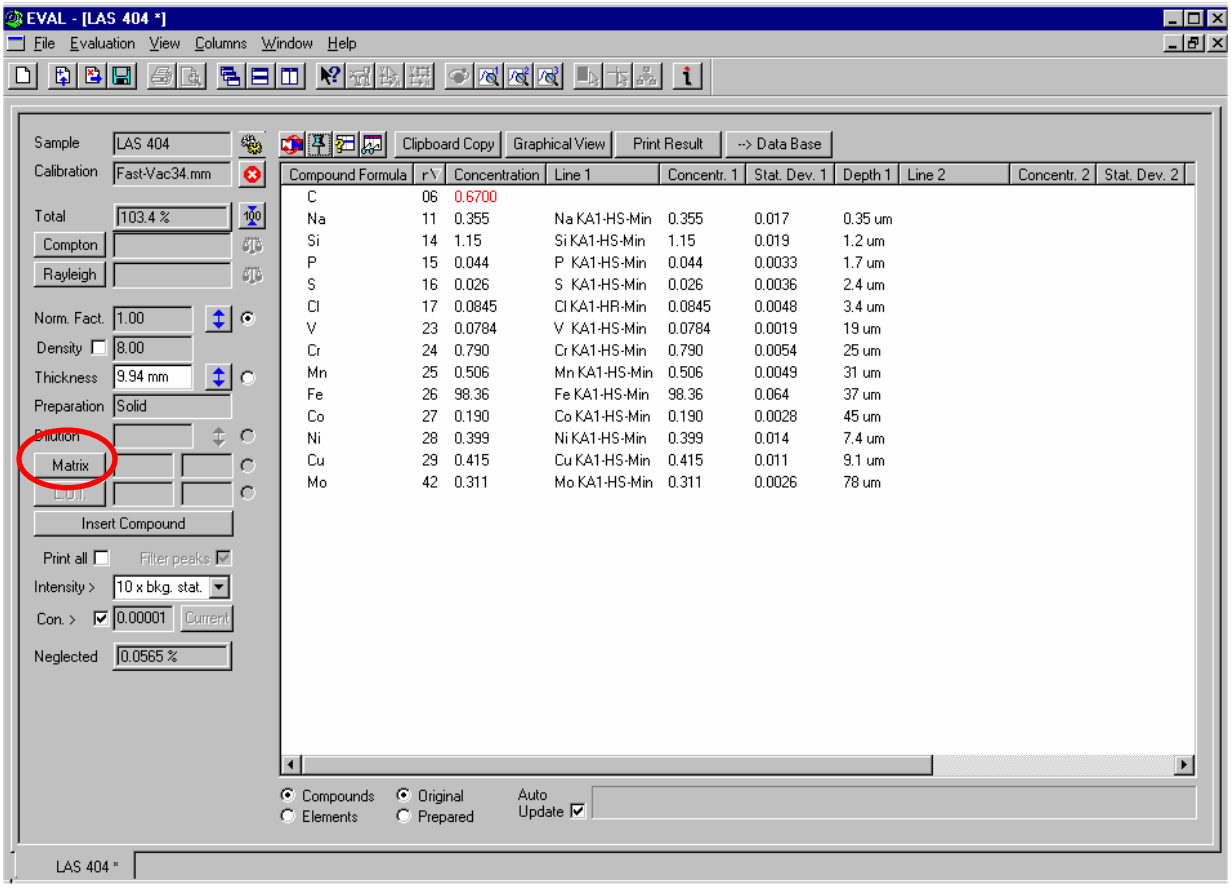
Close the View window by clicking the **X** in the upper right corner.


View Line (1): LAS 404
Ho LB1-HS-Min
KCps 0.01
SqE 4.755
WL 1.645
Ho LB1
Co SKBN
Ni SKA^5
Ni SKA^4
Ni SKA3'
Ni SKA^6
Ni SKA3'
Ni SKA4
Gd LG5
Ni KB1
2
1
0.1
0
4.7
4.8
Click Backgrnd.
Zoom Peak
Peak Only
Name
Pos.
Peak
Bkg.
L.L.D.
Overla
Ho LA1 HS-Min
54.590
3.382
1.586
0.026 %
0.006
Ho LB1-HS-Min
48.302
1.036
0.607
0.021 %
0.565
Found: 2
Clipboard
Metafile
Select
Update
View

A “low-alloy steel” sample contains mainly Iron (Fe), or in other words, the *matrix element* is Iron. Since SPECTRA^{plus} has calculated too high a concentration for Fe in this sample (105.6% in this example), it would be better to have Fe calculated as the matrix element.

Matrix

Click the Matrix button.

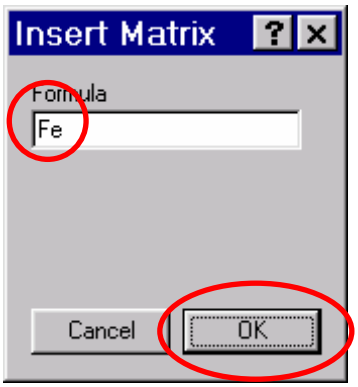


Fe

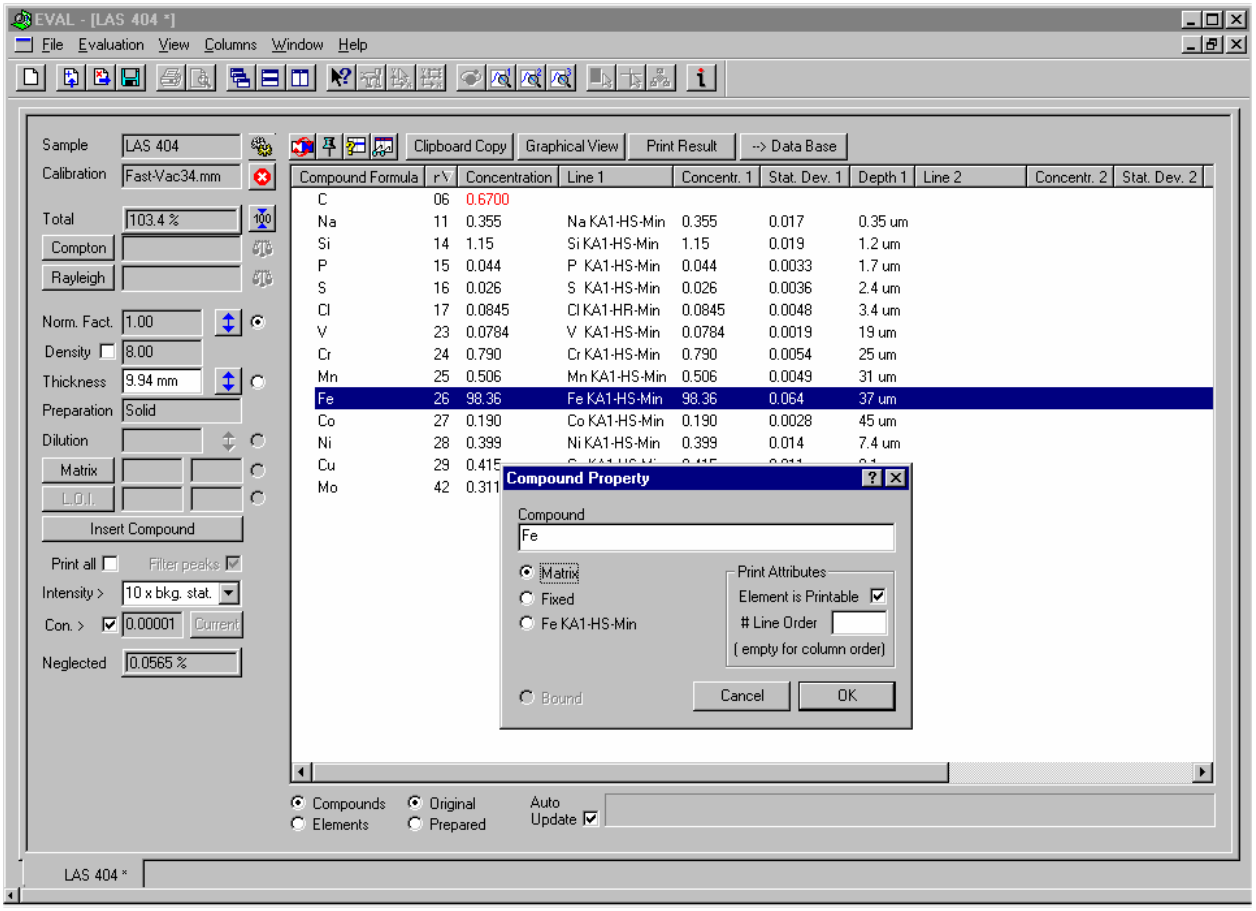
Enter **“Fe”** in the Formula box.

OK

Click the OK button.

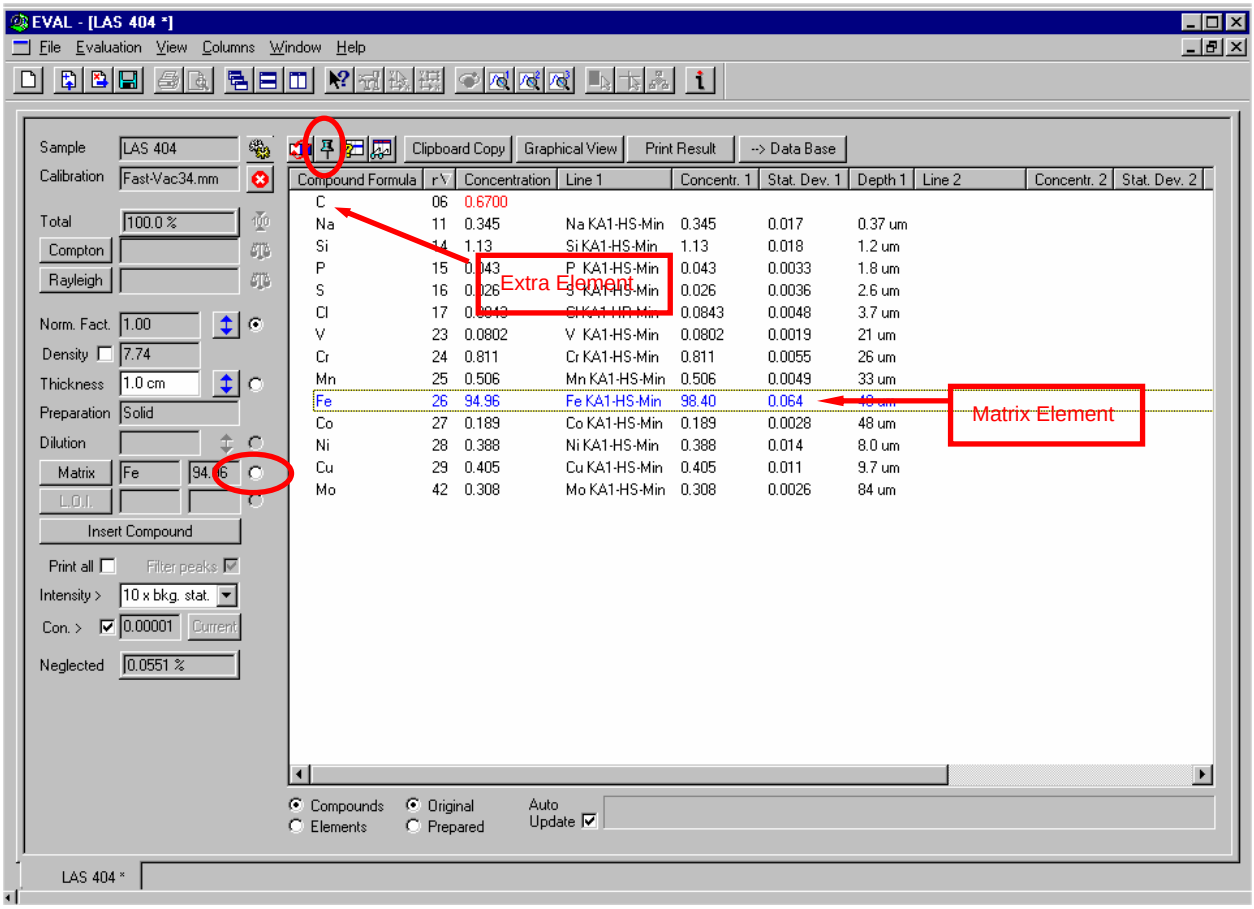


Or right-click on Fe and select Matrix.



Iron (Fe) is now calculated as the Matrix or “Balance” element, and its concentration is displayed in blue to indicate this.

Other elements may now appear, like Terbium (Tb) in this example. This is because changing the concentration for any element changes the whole sample composition. We can view the spectra for any new elements that have appeared, and if desired, eliminate them with the **Push Pin** icon as done before.



Once we are satisfied with the results, we can print and save them.

Click the **Print Result** button to print the sample composition.

Click the "**--> Data Base**" button to write the sample composition to the Measure database for later retrieval with the Results Monitor or Manager programs.

2.3 Creating and Modifying Standardless Measurement Methods

Several default Standardless Measurement Methods are delivered with the SPECTRA^{plus} software.

A user has the option of defining specialized Standardless Measurement Methods that are more suited to his needs, e.g., reducing the number of elements measured, selecting the lines to be measured, and setting the counting times for the lines measured. If nothing else, the user should check the Mask and Counting Times of the supplied programs to ensure that these are suitable for his samples.

Run the **Measurement Method** program.

Click the **File Open** icon.

Click the drop-down control in the "**Look In**" box.

Navigate to the "**\SPECplus\Libraries\MeasMethods\AXS34**" folder for methods based on 34mm.

Fast-Vac34

Select one of the existing Standardless Measurement Methods to use as a template, like "Fast Vac34".

Click the **Open** button.

NOTE: If you get the message "some lines were not found in the library", it only means that some of the lines in the Measurement Method cannot be measured on your instrument because of hardware limitations (crystal, collimator, etc.). This message can be ignored, and the OK button clicked to close the error window.

Click the **Parameters** tab to select it.

On this tab you can set:

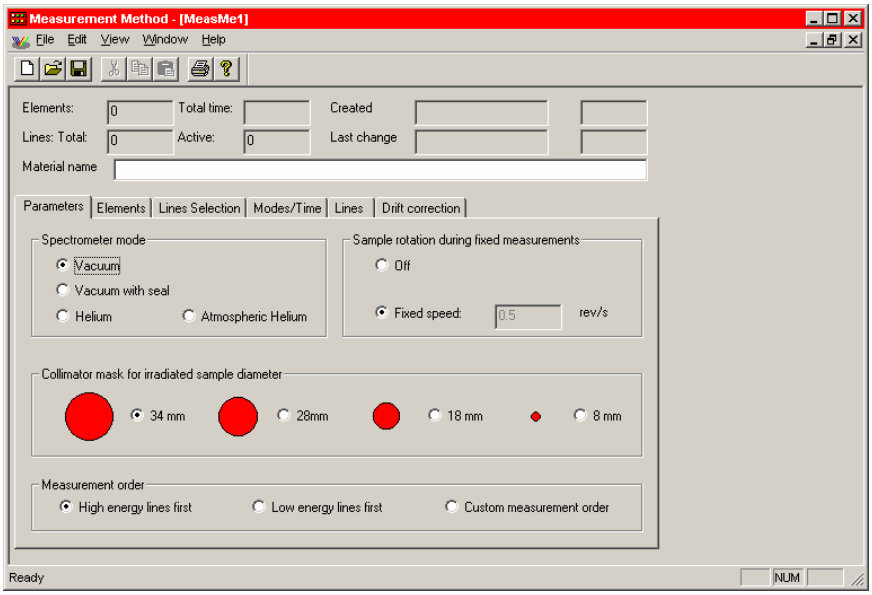
Spectrometer mode (vacuum, helium, etc.)

Sample rotation (off or on)

Collimator mask size

Default measurement sequence

If nothing else, ensure that the correct Mode and Mask are being used for the samples you intend to measure with this program.



Click the **Elements** tab to select it.

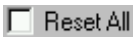
This tab shows which elements have been selected for the Measurement Method, regardless of whether or not the element is actually measured.

Elements can be removed from the Measurement Method by clicking on the appropriate element symbol to turn it gray.

Click the **Lines Selection** tab to select it.

This tab can be used to force SPECTRA^{plus} to re-select the lines to measure. Proceed as below ONLY TO RE-SELECT THE LINES BEING MEASURED.

Select the "**Use precalibrated lines**" button.



Check "**Reset All**" to force all lines to be re-selected, leave unchecked to only re-select the lines for any new elements that were added.

Click the **Apply** button to apply the changes you made.

Click on the **Modes/Time** tab.

On this tab the following can be set:
- Measurement mode: Scans or Fixed-Points (Scans is the default).
- Measurement time (10 seconds is a good time to use).

Both of these settings can be made on a "Global" basis, which applies to all lines, or on a "Local" basis, which applies only to select lines. See the SPECTRA^{plus} Reference Manual (M84-Exx025) for more detailed information.

Click on the **Lines** tab.

This tab can be used to add and remove specific lines from the Measurement Method. Lines on the left side will be measured, and lines on the right side are available but will not be measured.

Lines can be moved from the right (non-measured) to the left (measured) with the **Add** button.

Lines can be moved from the left (measured) to the right (non-measured) with the **Remove** button.

The "**(C)**" after a line name indicates a pre-calibrated line. Only pre-calibrated lines should be chosen for a Standardless Measurement Method, since these are the only lines from which concentrations can be calculated.

Click on the **Recalibration** tab.

This tab contains information on the drift correction of the Standardless Program. For the default library lines, this information can be viewed by the user, but not modified because it is protected from changes.

Use **File→Save As** to save any changes that were made under a different file name.

It is suggested that the name selected should include some form of coding with at least the type of lines, the measurement mode, the mask size, and the counting times for clarity, such as **"st-less-HS-Vac-34mm-10s-scan"**. It is clear that, when a sample is measured using this Measurement Method, the measurements will involve the High Sensitivity lines and will be done in a vacuum path. In addition, the samples should be loaded into 34 mm sample holders, and that each line will be measured for 10 seconds.

As an exercise, create a method based on Fast Vac34 selecting only the elements which are present in the LAS sample (see wooden box inside the lid) using 34mm, scan, 10 sec time.

Save this method as **LAS check** (You will need this next).

2.4 Using the “STG-2” Sample to Check the Standardless Drift Correction

The “STG-2” glass sample can be measured as a control sample that can check the effectiveness of the Standardless drift correction just performed.

Select the **Loader**.

Load the definition file **STG2.def** for the STG check.

Click on the “**STG2**” tab on the bottom of the Sample Edit window.

Click the position on the Loader where you placed the STG2 sample in a 34mm cup.

Type in “**STG Test**” as the sample name; the rest will be automatically filled in when using the STG2 def file.

Otherwise, measure it by using the AXS-34 def file (older versions of SPECTRA^{plus} or units shipped before Jan 2004). Choose the **STG-check.mm** and the **STG-check evm (model)** with **solid** as a preparation.

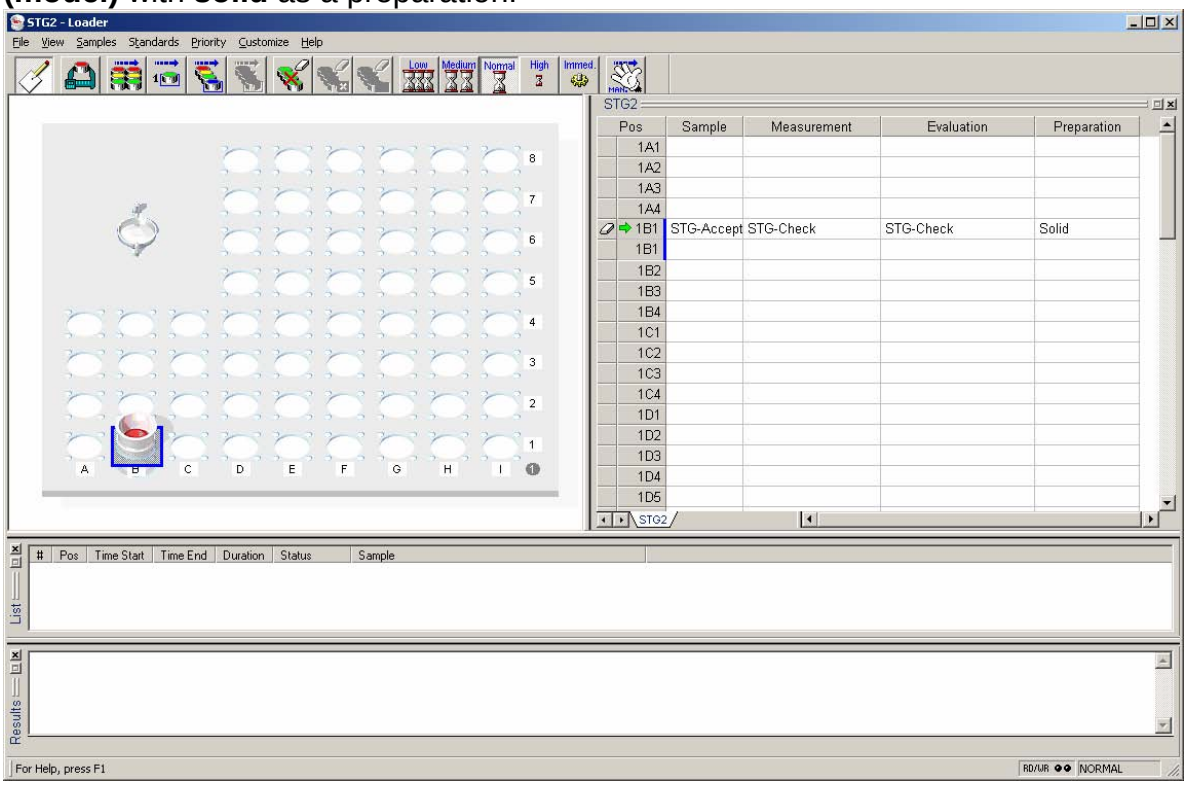


Figure 1 - Loader Screen with STG Check Def and Sample

Start the sample by selecting “**Send Selected Sample**” or by pressing the **F10** key.

The sample locks down and will start measuring.

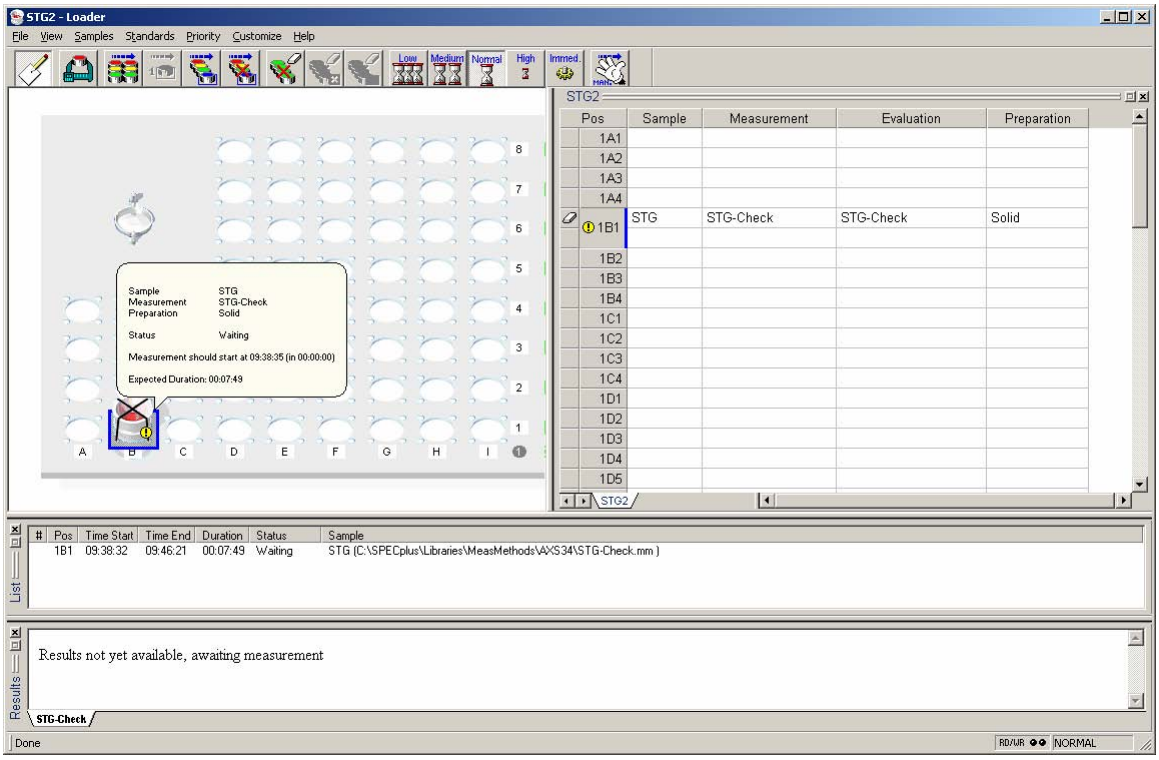


Figure 2 - STG check running

When the sample displays with a yellow **OK** tag, the measurement is finished. In SPECTRA*plus* 1.6.6, it is possible to view the results of the measurement in a separate window pane (See Figure 3).

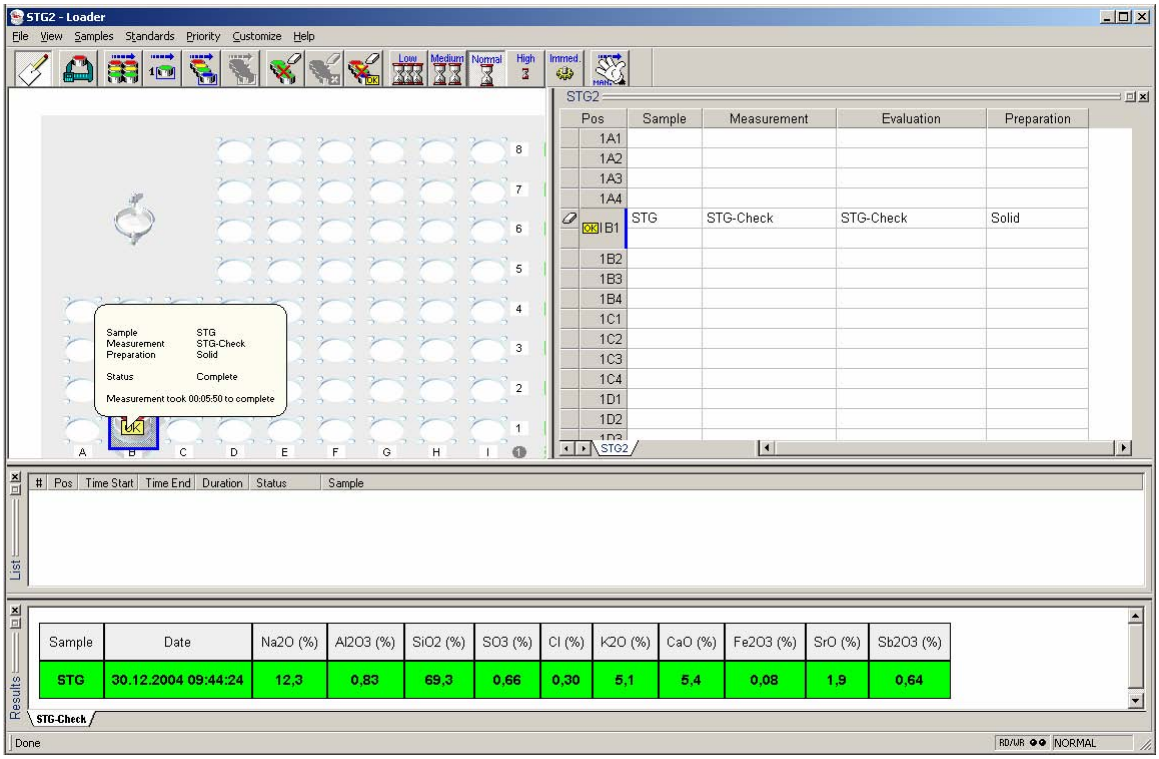


Figure 3 - STG check done with results displayed in LOADER

Select the **Results Monitor**.

Select **File→New Monitoring View**.

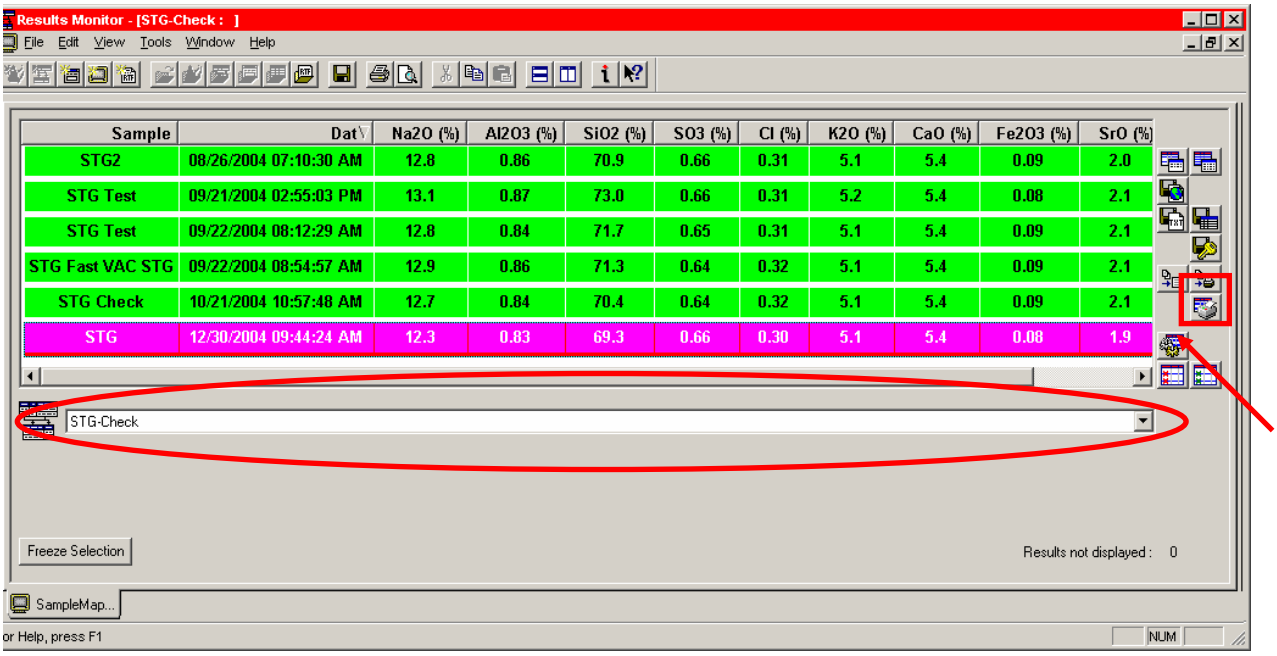


Figure 4 - Results Monitor with STG check results

Select **STG Check** from the drop-down window as a report format (red oval in Figure 4). Press Update (red arrow) and highlight the entry with the current measurement.

Select **Print Results** (red square in Figure 4). This will print the results on the default printer.

Check the concentrations in the printout and compare them with the concentration on the measurement obtained at the installation on your site.

If any of the concentrations are NOT within the prescribed limits, it is necessary to drift correct the Standardless Method, and if this fails, to perform the alignment of the unit. After the drift correction is performed, the STG measurement has to be repeated to validate the positive outcome.

To interactively evaluate the STG measurement:

Run the **Evaluation Program**.

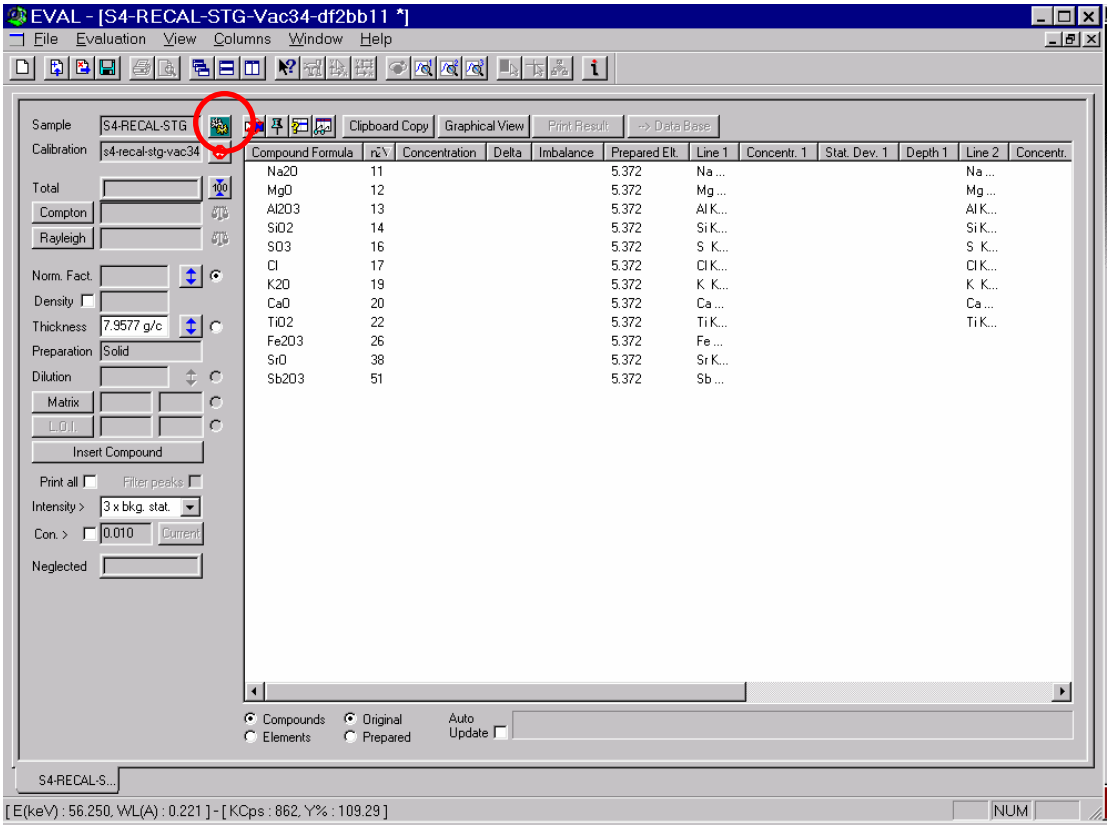
Click the **Next SSD File** icon.

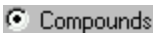
Set the **Search Control** to when the sample was measured (e.g., “Today”, “Yesterday”, etc.)

Highlight the sample name.

Click the **Open** button.

Click the **Gears** icon to ensure that the sample results are calculated.





Make sure that “**Compounds**” is selected.

Make sure that “**Original**” is selected.

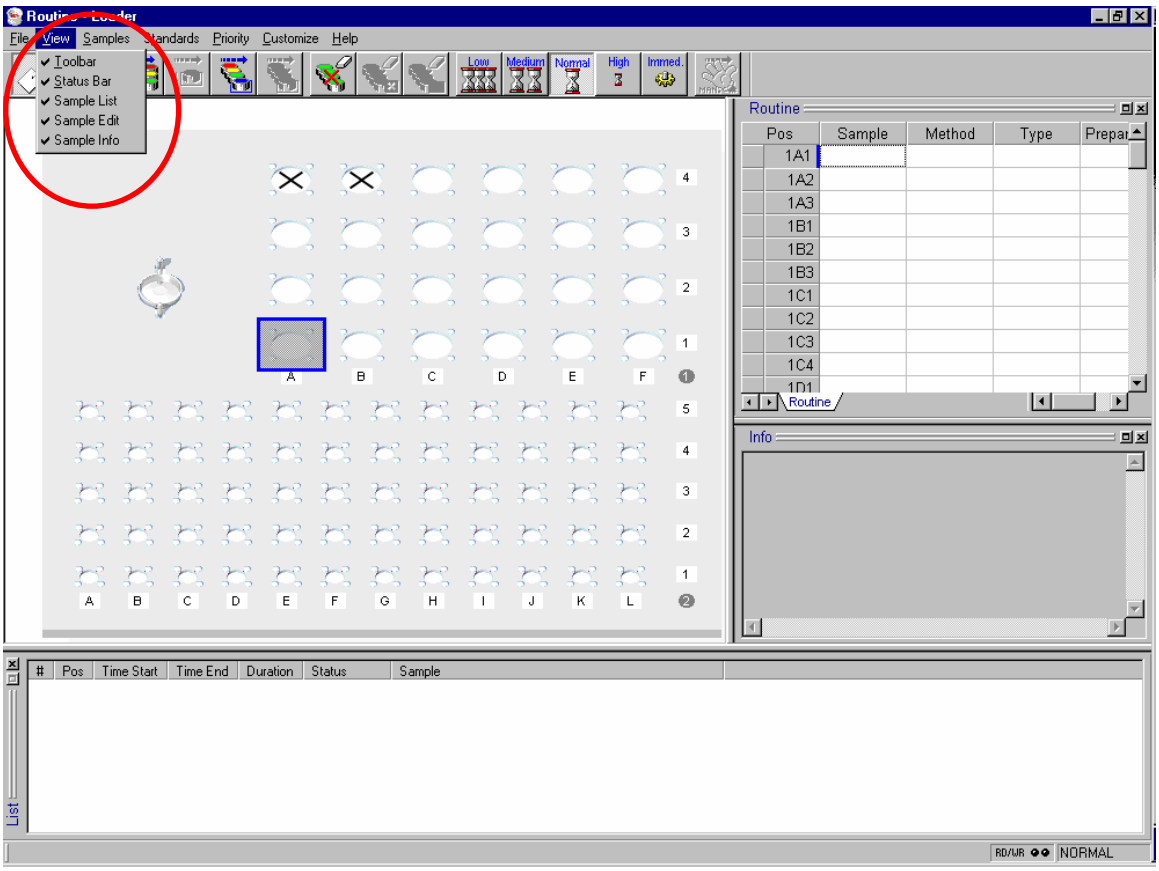
2.5 Drift Correction of the Standardless Program

Prior to measuring any samples with the Standardless Program, the user must measure some **drift correction samples** on his instrument to adjust for the sensitivity of his instrument. These drift corrections must be performed in **every Mode and Mask combination** that will be used to measure unknown samples. This guide details making the drift corrections for samples measured in a vacuum path with a 34mm sample holder, but other Mode/Mask combinations are measured in a similar fashion.

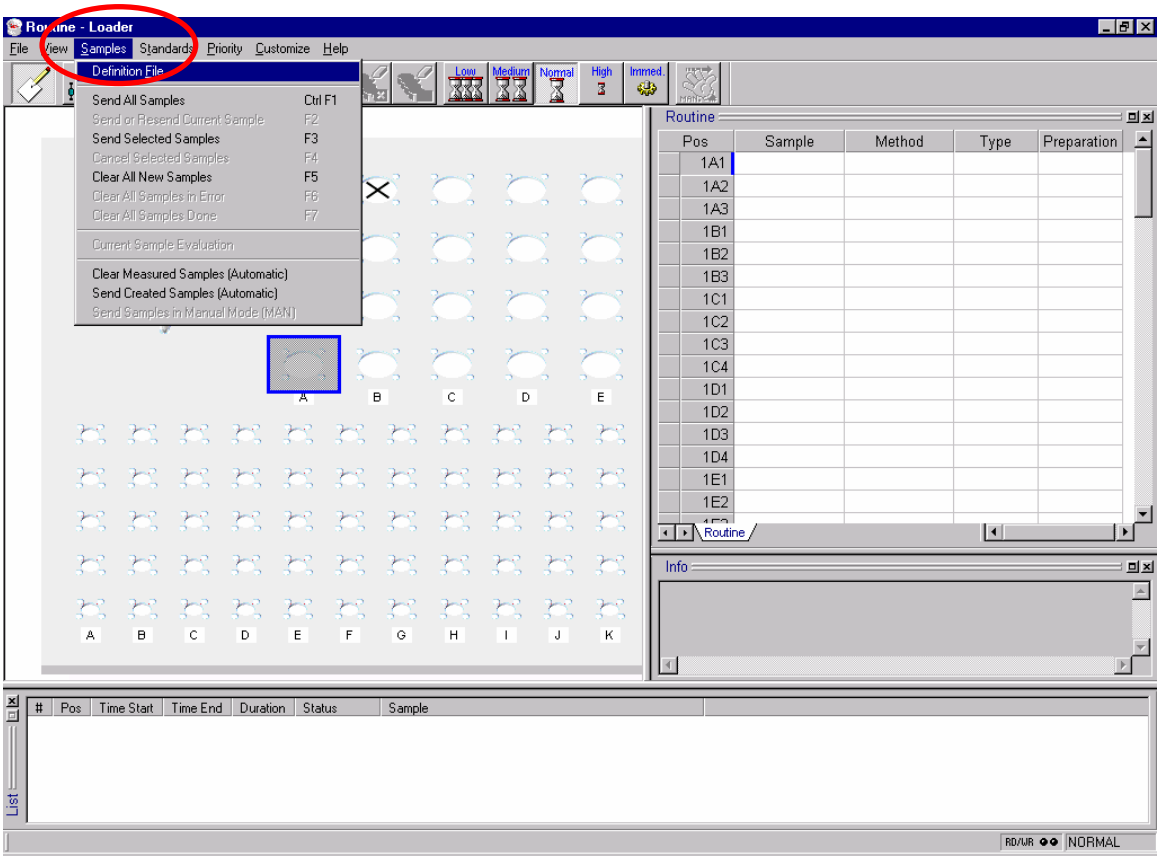
Start the **Loader**.

Click **View** in the menu, and make sure all options are checked.

Arrange the Edit, Info, and View windows as shown, or to suit your tastes (see Section 2.1).

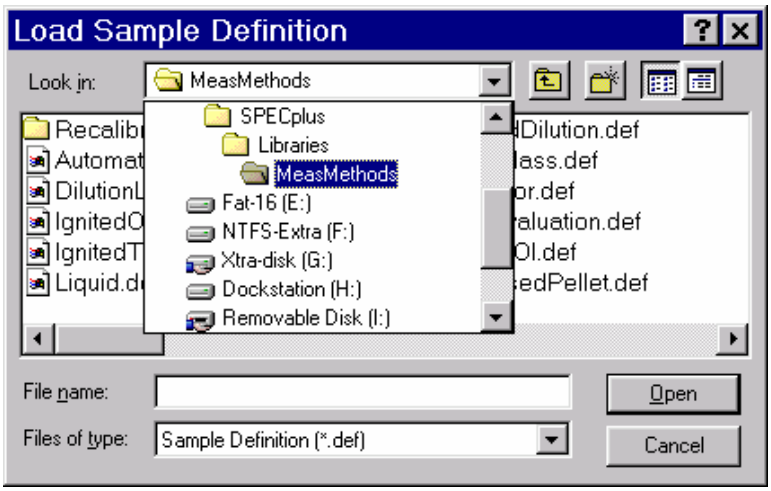


Select **Samples→Definition File…** from the menu.



Click the drop-down arrow in the “**Look in**” box.

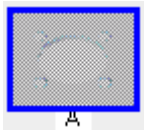
Make sure the “**\SPECplus\Libraries\MeasMethods**” folder is selected.



Select “**S4-RECALxx.def**”, where xx = Mask size.

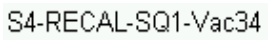
Click the **Open** button.

Click on the **S4-RECAL34** tab to select it.

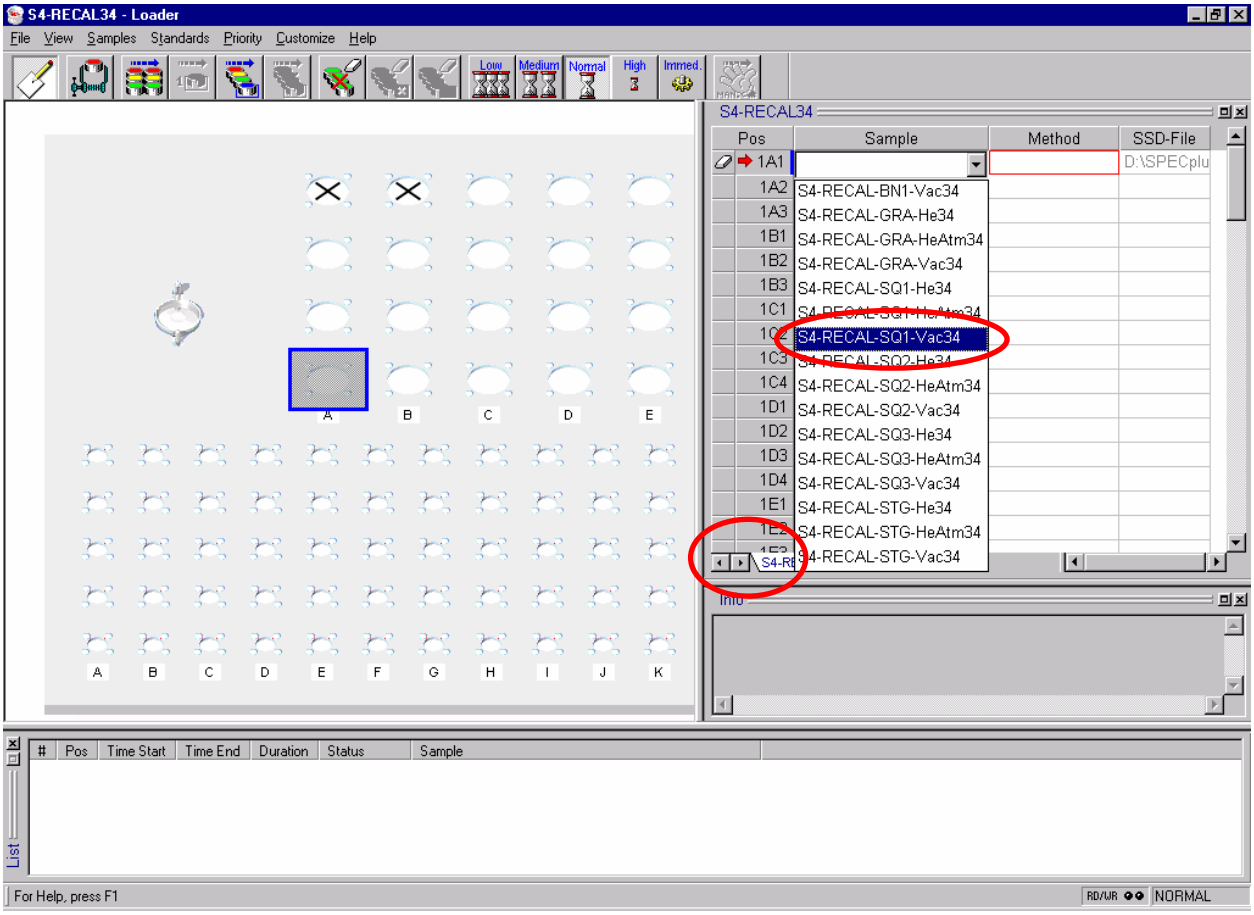


Right-click on the sample position where the first sample is loaded.

Click on **Edit Sample**.



Select “S4-RECAL-SQ1-Vac34” from the list of samples.

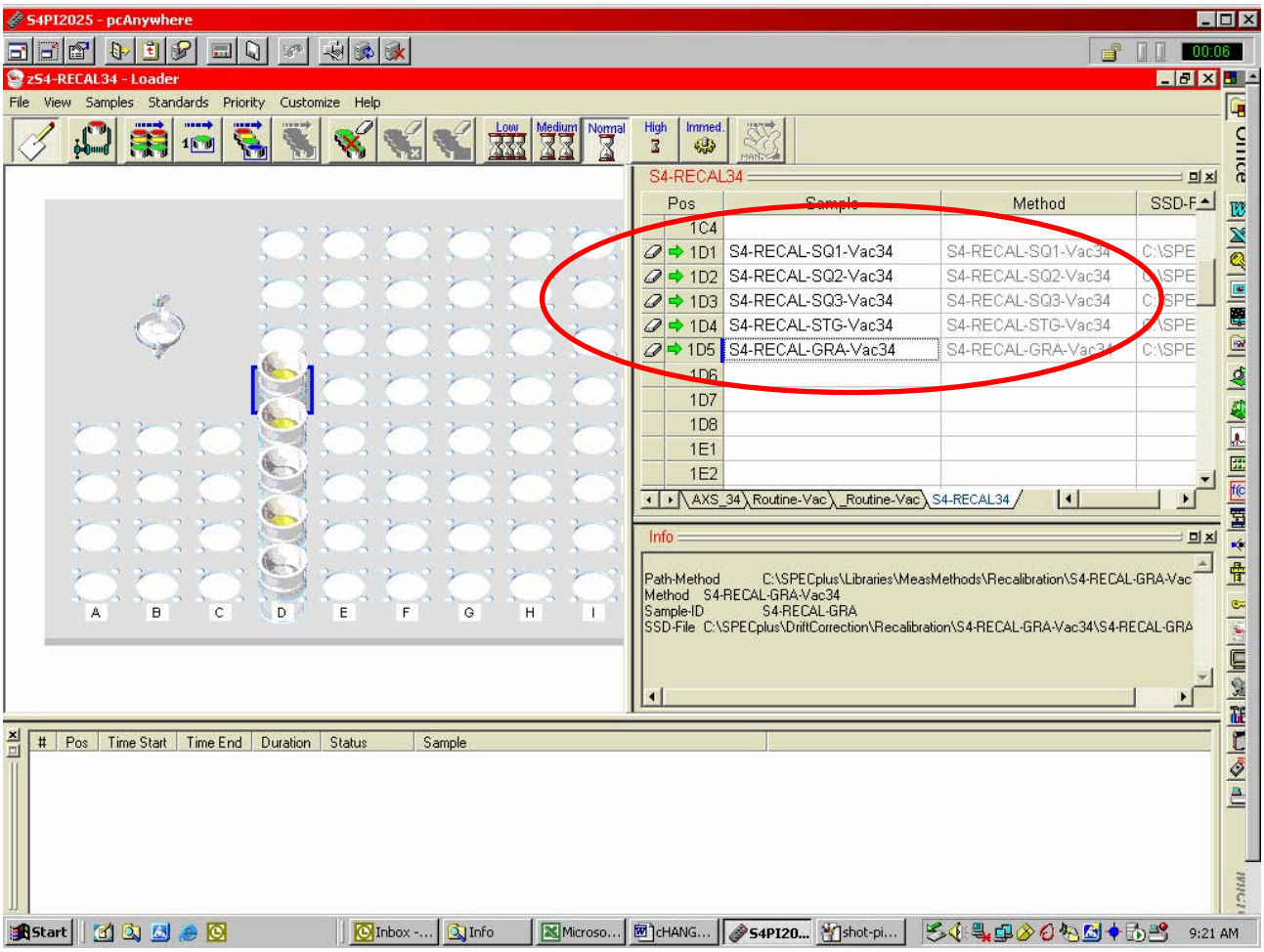


Continue selecting samples until all 5 or 6 have been selected:

Pos	Sample
1A1	S4-RECAL-SQ1-Vac34
1A2	S4-RECAL-SQ2-Vac34
1A3	S4-RECAL-SQ3-Vac34
1B1	S4-RECAL-GRA-Vac34
1B2	S4-RECAL-BN1-Vac34
1B3	S4-RECAL-STG-Vac34

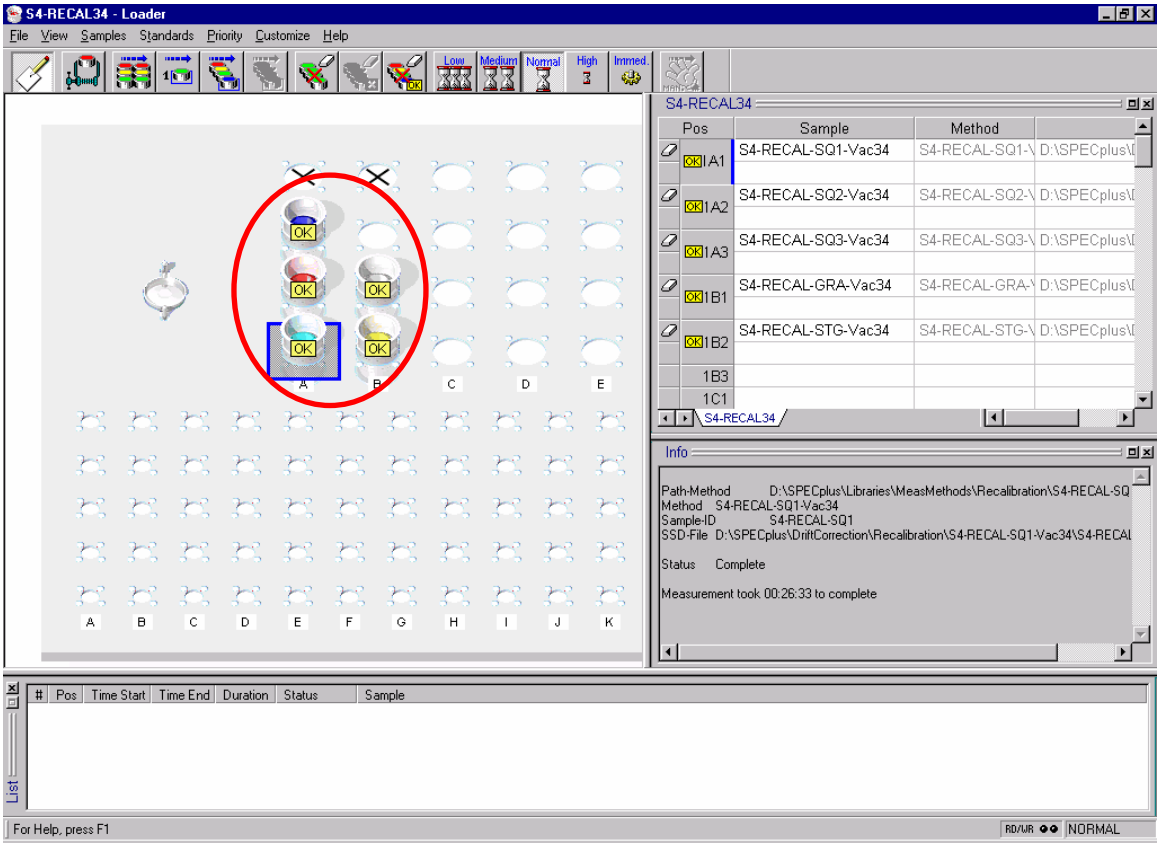
S4-RECAL-SQ1-Vac34
S4-RECAL-SQ2-Vac34
S4-RECAL-SQ3-Vac34
S4-RECAL-GRA-Vac34
S4-RECAL-STG-Vac34

Optionally “S4-RECAL-BN-Vac34” (only for OVO-B or OVO-N crystal)



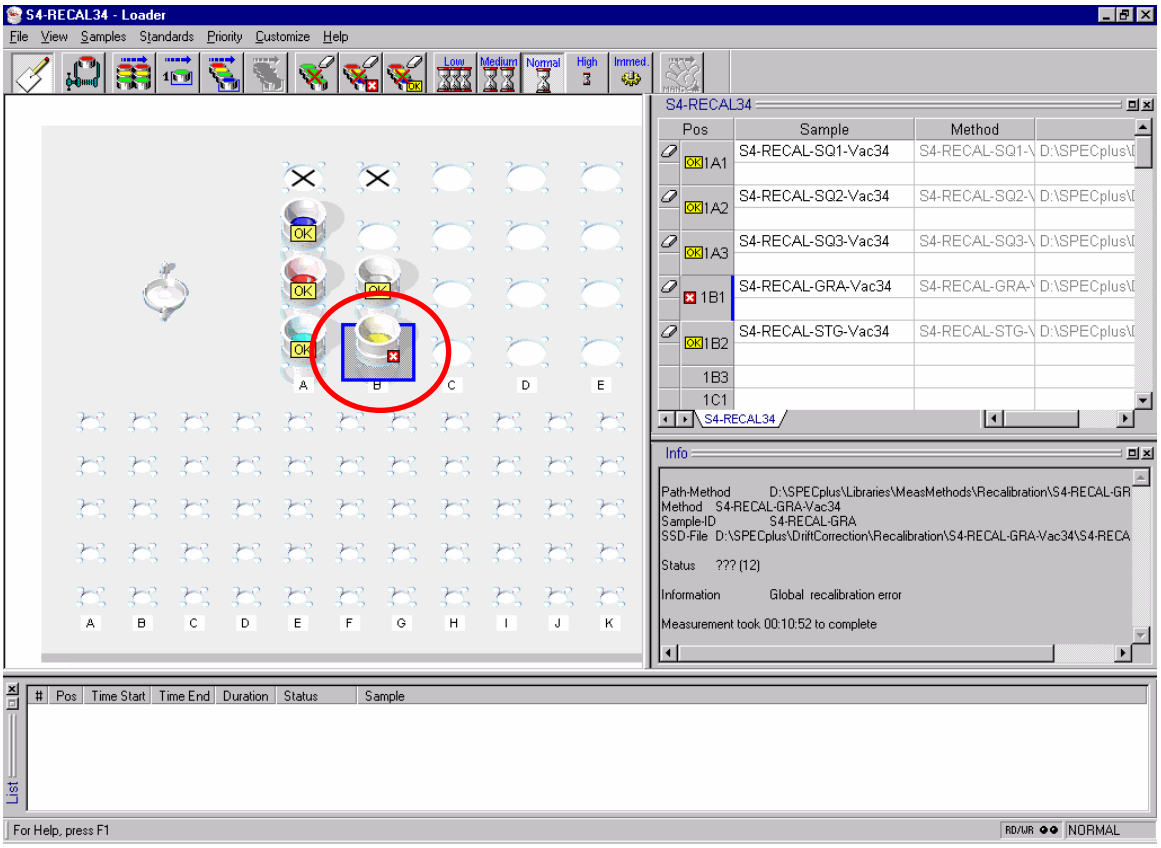
Click the **Send All Samples** icon to start the measurements.

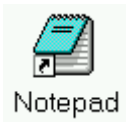
As each sample finishes, it should display “**OK**” on its cup. This indicates that the drift correction was successful.



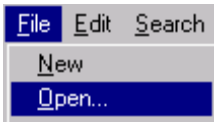
If any of the sample holders have a **Red X** on them, this indicates that the drift correction failed.

There will also should be an error window open which lists the cause of failure:

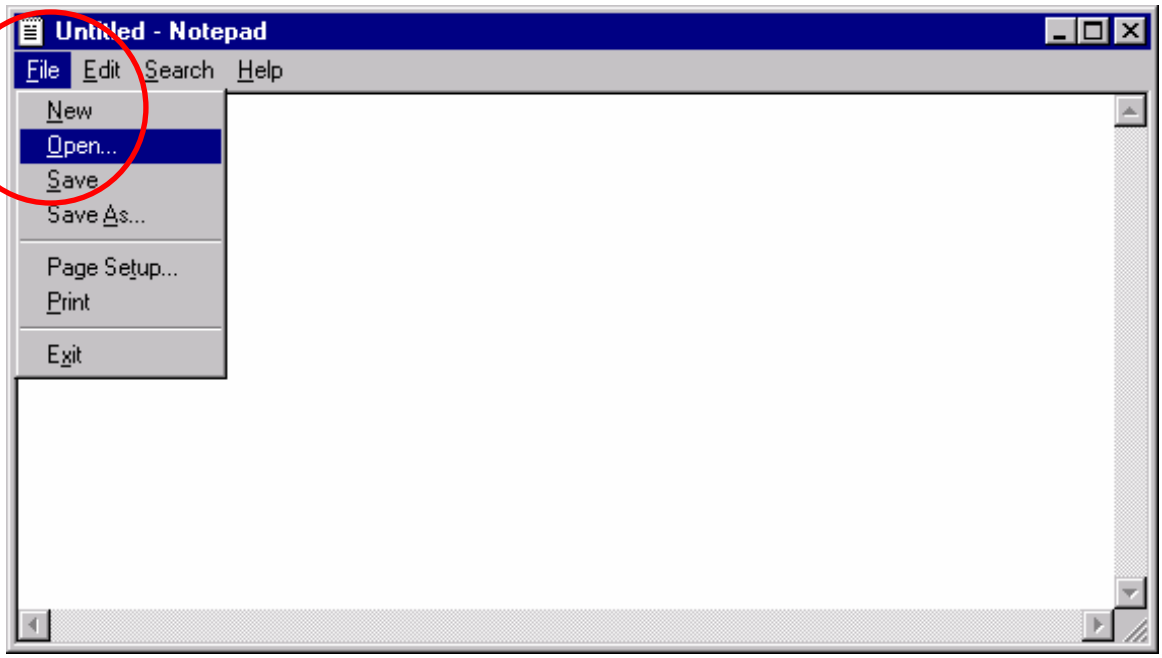




To obtain more specific information on the error, run the Windows **Notepad** program.

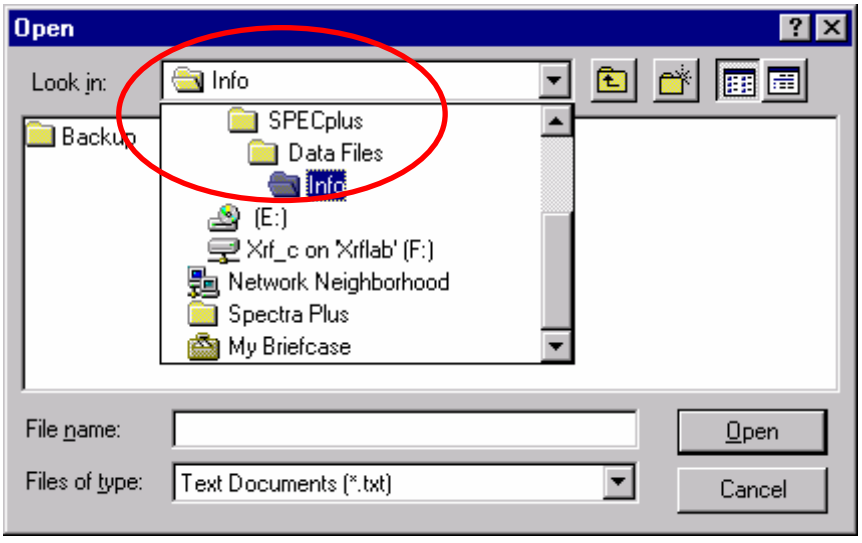


Click on **File→Open**.



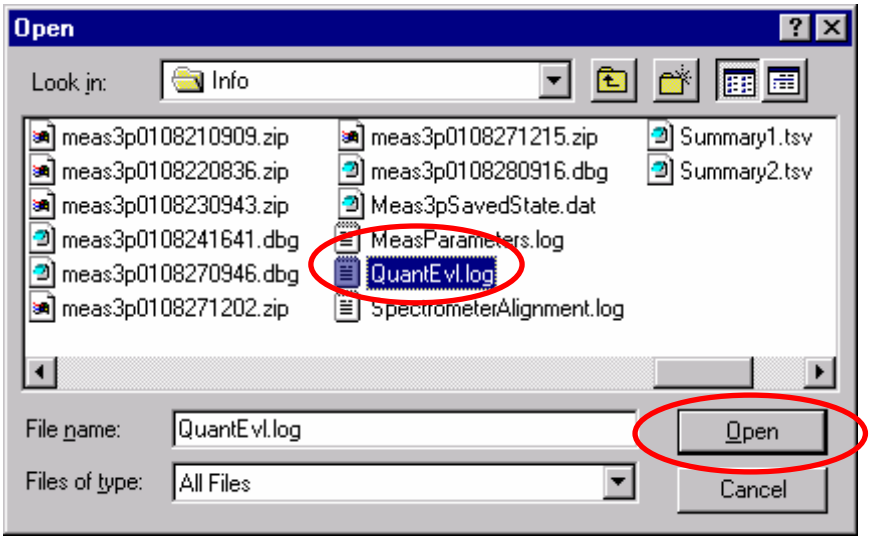
Click the drop-down control in the "**Look in**" box.

Navigate to the "**\SPECplus\Data Files\Info**" folder.

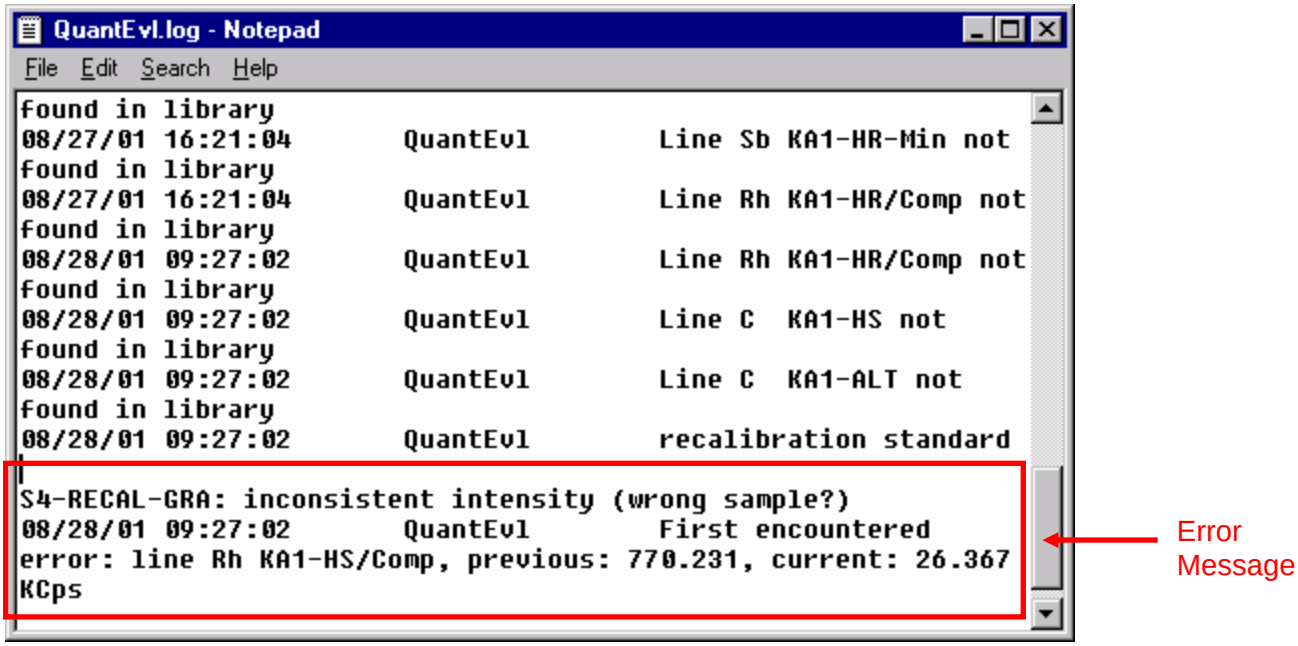


Click on the "**QuantEvl.log**" file.

Click the **Open** button.



Press the **<Ctrl>** + **<End>** keys to move to the bottom of the file (or use the scroll bar on right side of window).



3 Quantification Program

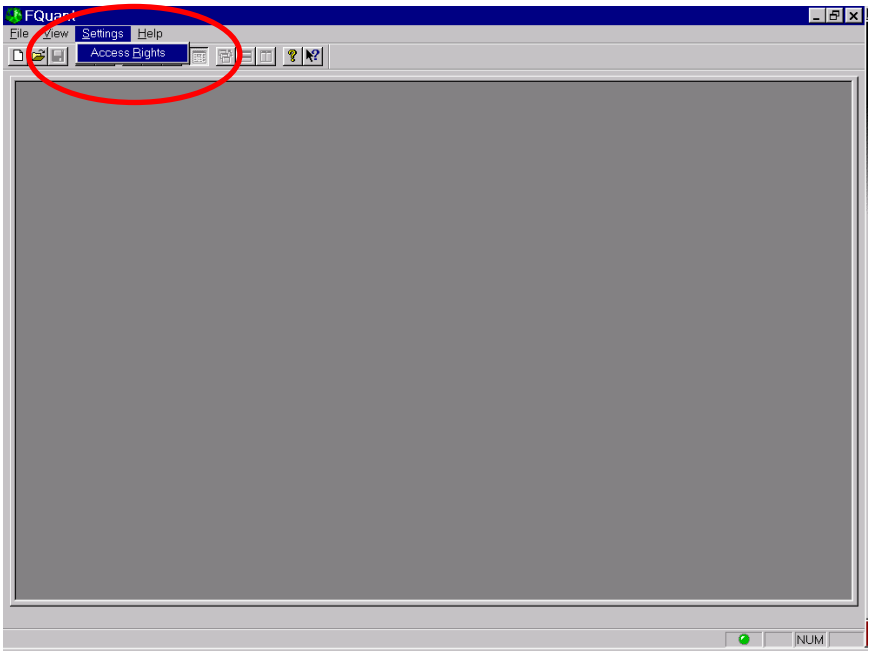
This section covers the Quantification Program that is part of the SPECTRA^{plus} software. It is based on an example using a set of five “Low-Alloy Steel” samples to cover the entire calibration and analysis of unknown samples using the SPECTRA^{plus} software.

3.1 Introduction to the Quantification Program (FQuant)

Click the **Quantification Editor** icon to start the FQuant program.

If you set an “Access Rights” password in FQuant, to gain access:

Click **Settings→Access Rights** on the menu.

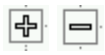


The document window is split into two parts:

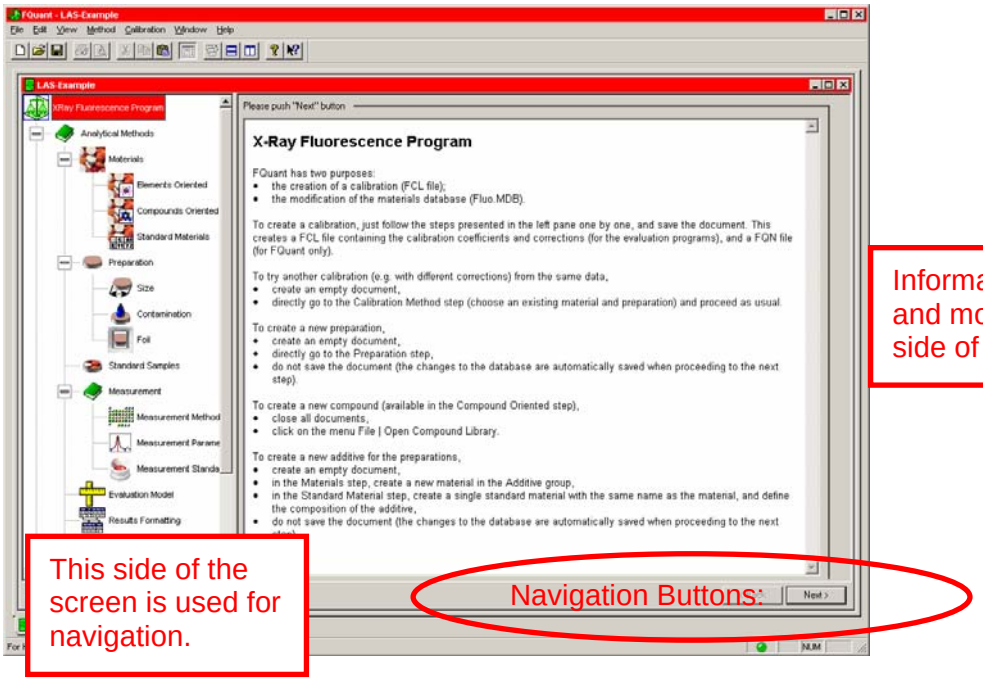
The left side of the window has an “Explorer-type” interface that can be used to navigate by clicking on one of the listed functions using the mouse. The **Next** and **Back** buttons in the lower right corner can also be used to move between functions in a sequential manner. This is the most foolproof means of using the software since it ensures that all functions are carried out in the correct sequence and that nothing is missed.

The right side of the window is used to display previously defined information and provides the opportunity to define new information, or to change existing information.

Some of the functions listed in the “Explorer” window are placeholders, and require no input. When one of these is selected, the right side of the window displays:
Please push “Next” button



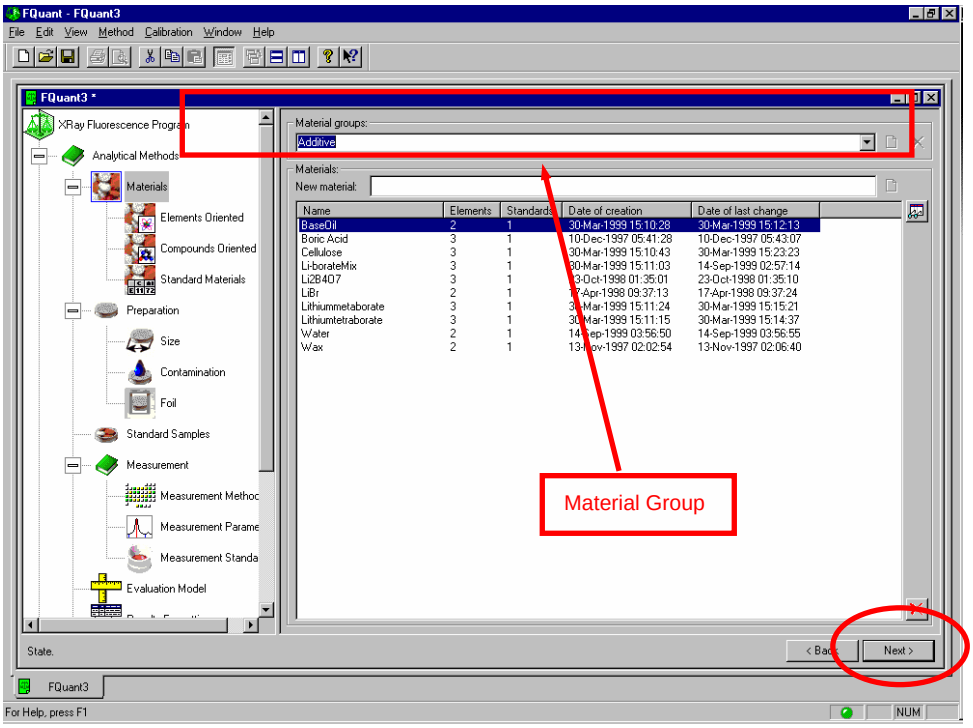
The main options in the “Explorer” window can be expanded and collapsed using the “[+]” or “[-]” symbols for the desired function.



The general concept of the SPECTRA^{plus} software is to break down the development of a new analytical method into logical steps, with the X-ray physics being modeled as closely as possible during each step. Briefly, these steps are as follows:

Materials:	Is used to define what we are trying to analyze. It is also used to define any other materials that might be used in the preparation of the XRF specimens such as binders, fluxes and cup-closing foils.
Preparation:	Is used to define exactly how the XRF specimens were prepared.
Standard Samples:	Is used to define “prepared” XRF specimens by applying a Preparation to a Material. In this way, the true composition of the specimen being measured by the XRF instrument is known.
Measurement:	Is used to define a Measurement Method that can be used to measure intensities from the samples. It is also used to optimize the measurement conditions, and to measure the set of calibration standards.
Evaluation Model:	Is optional. By default, SPECTRA^{plus} expects all parts of an Analytical Method to have the same name. This includes the Measurement Method used to measure the intensities, the Fluorescence Calculation List used to calculate the concentrations, and the Results Formatting file used to control the display and output of the results. One use of an Evaluation Model is to specify the names for each part of the Analytical Method so that the individual parts do not have to have the same names.
Results Formatting:	Is used to define how to report the results to the screen and the printer. This includes order of output, concentration units, number of decimals, etc.
Calibration:	Is used to calculate the calibration coefficients and then save these into a Fluorescence Calculation List. SPECTRA^{plus} has an integrated Fundamental Parameters program for handling matrix effects.
Interactive Evaluation:	Can be used to recalculate the composition of a control sample or calibration standard to allow fine-tuning of the calibration curve.

Click the **Next** button twice to move to Materials.



In SPECTRA^{plus}, a Material is any physical thing used in the laboratory for measurements by the XRF instrument. These materials include the samples to be analyzed, additives used to prepare the XRF specimens for measurement, contaminates picked up by samples during their preparation, and cup-closing foils placed on plastic cells when measuring liquid or loose powder samples. The advantage to this approach is that all of these things are defined the same way. This means that to define a binder, foil, or an actual material to be analyzed, one would use the same steps and the same “tools”.

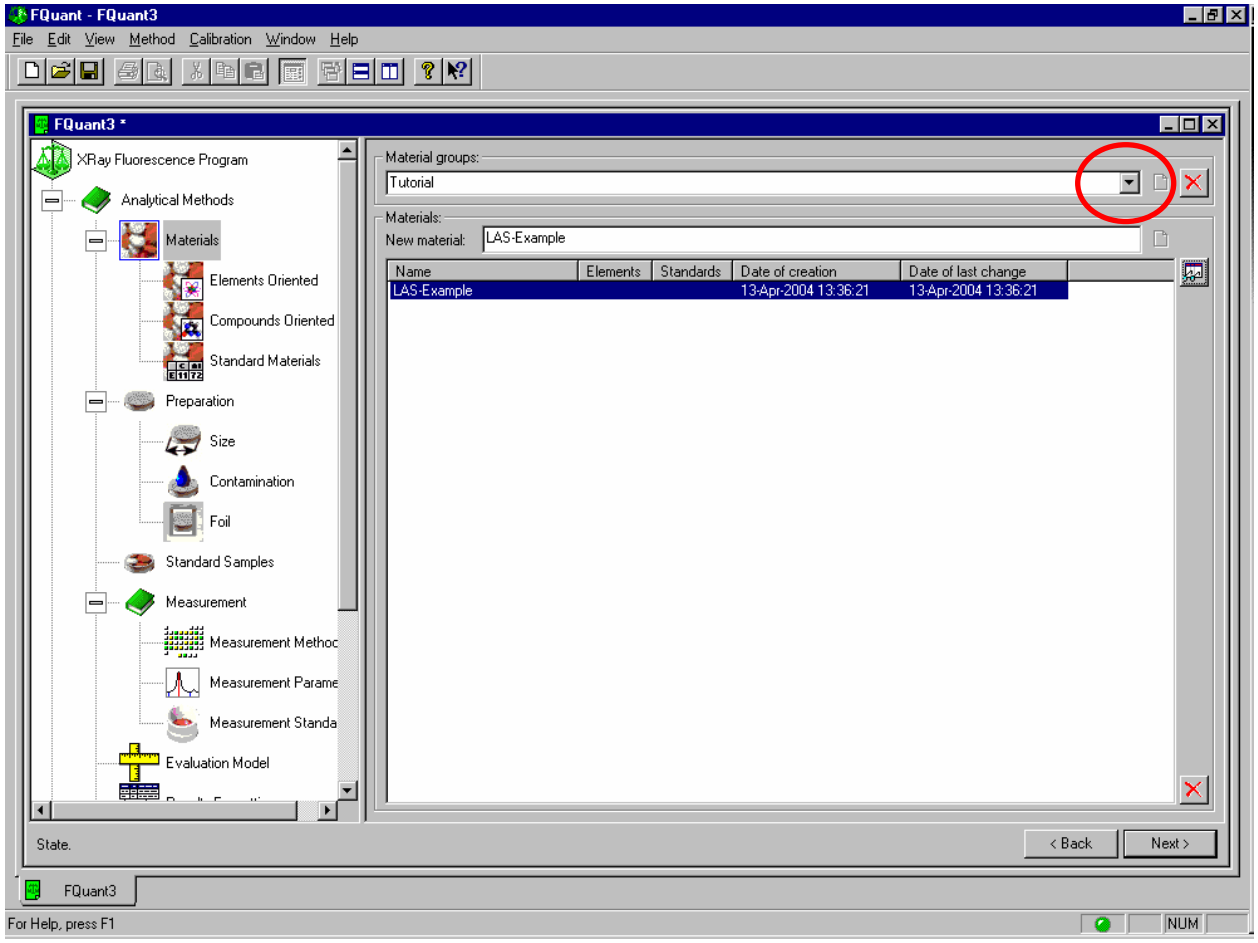
SPECTRA^{plus} uses Material Groups to keep the list of materials logically arranged. The user can define new Material Groups to meet his specific needs. There are four special Material Groups that are used for the following purposes:

Additive:	Anything added to samples during their preparation must be defined in this Material Group. These include binders, fluxes and diluents.
Contamination:	All contaminates picked up by samples during their preparation must be defined in this Material Group. For example, powdered samples can pick up tungsten carbide when they are ground in a tungsten carbide grinding vessel.
Foils:	All cup-closing foils used to prepare liquid and loose powder samples must be defined in this Material Group.
Tutorial:	This Material Group contains example materials used in the SPECTRA^{plus} Tutorial Manual (M84-Exx030). This allows one to learn how to use the software without making any actual measurements on their instrument.

Select the **Additive Material Group** by clicking the drop-down control in the Material Group box.

Select the **Tutorial Material Group** by clicking the drop-down control in the Material Group box.

This shows the list of pre-defined materials that are used in the SPECTRA*plus* Tutorial Manual (M84-Exx030).



3.2 Defining a New Material

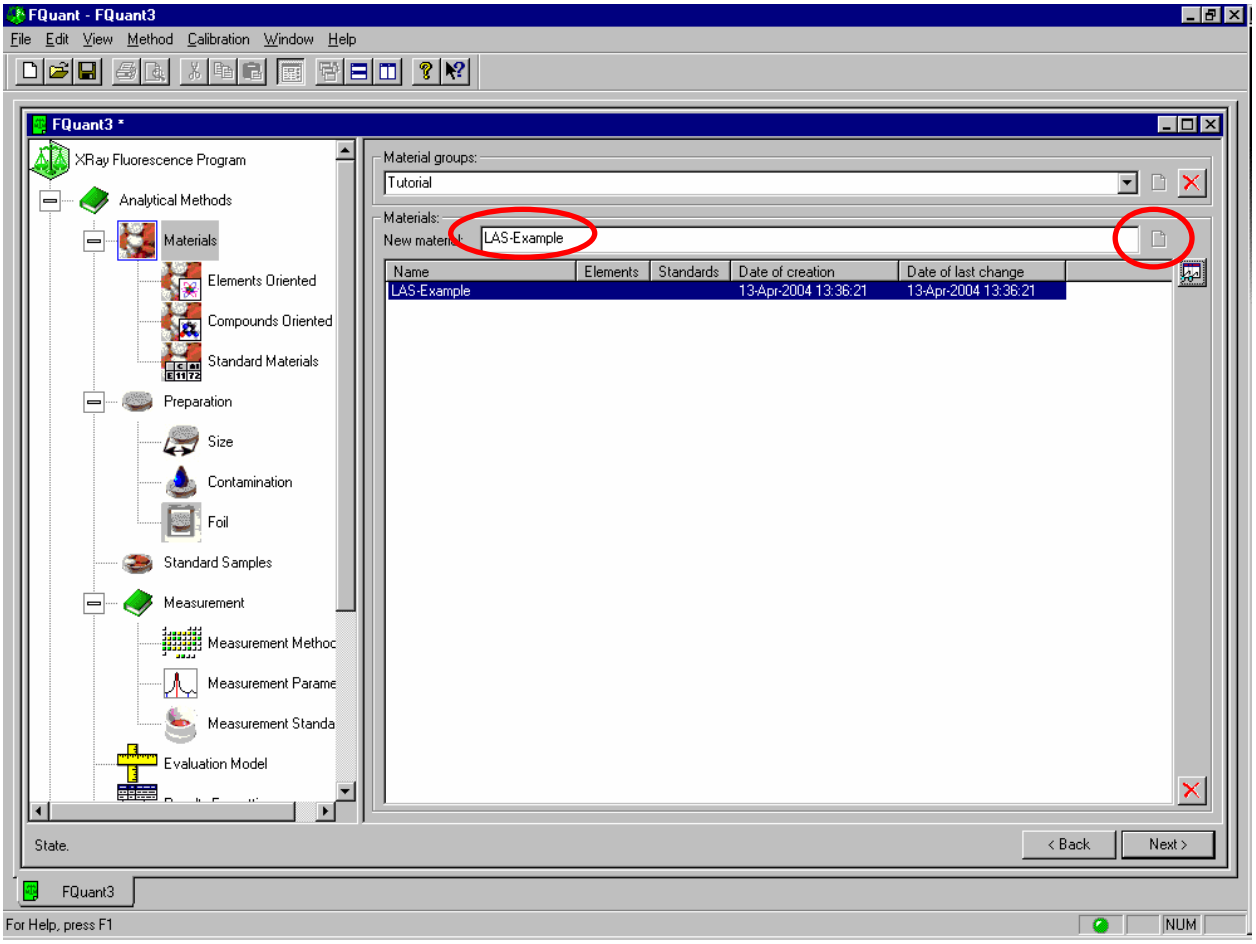
In this section, a new Material for Low-Alloy Steels will be added to the “Training” Material Group.

Make sure that **Materials** is highlighted in the “Explorer” window.

Make sure the **Tutorial Material Group** is selected.

Type “**LAS-Example”** as the new Material name.

Click the “**New Material**” icon, or press the **<Enter>** key.



Click the **Next** button to move to the Elements Oriented screen.

The Elements Oriented screen can be used to define the elements or oxides present in a Material by simply clicking the appropriate element symbol on the periodic table.

Before each element symbol is selected, its Type can be chosen using the **Element** or **Oxide** buttons at the bottom of the window.

Since only elements and some pre-selected oxides can be defined using the Elements Oriented screen, SPECTRA^{plus} also provides a Compounds Oriented screen for entering more exotic compounds.

When specifying what is in our Material we must at least define:

All elements that are to be determined by XRF (measured).

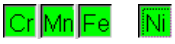
All elements or compounds that will have manual input of their concentrations (concentrations given when the sample is measured).

Any single element that is to be determined as the balance to 100%.

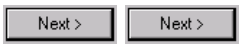
If the Fundamental Parameters portion of the SPECTRA^{plus} software will be used to correct for matrix effects, then enough elements and/or compounds must be defined to enable definition of 98 to 100 percent of the sample composition.

In this example Cr, Mn and Ni will be determined by XRF, and Fe will be determined as the “balance” element: Fe = 100% - (%Cr + %Mn + %Ni)

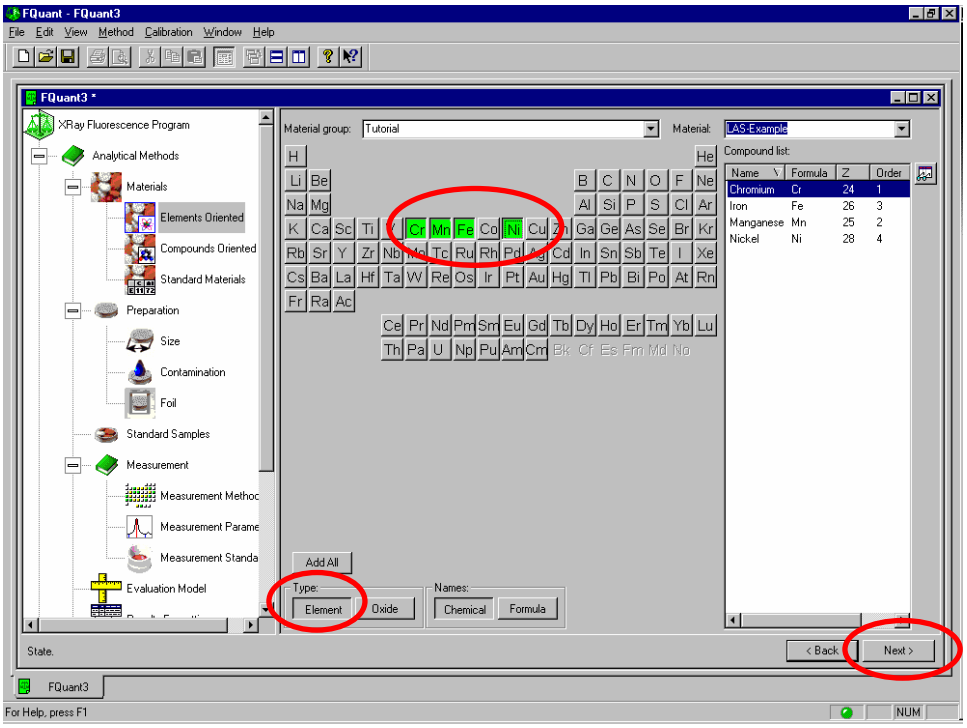
Make sure the **Element** button has been selected.



Click **Cr**, **Mn**, **Fe**, and **Ni** on the periodic table to add these elements to the “LAS-Example” material.



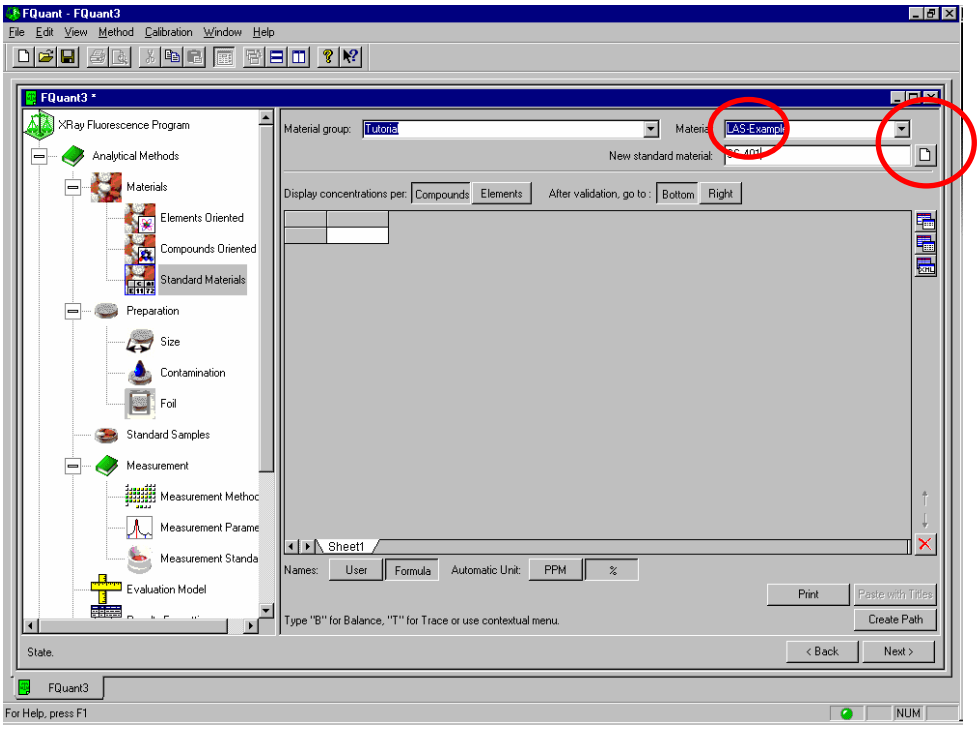
Click the **Next** button twice to go to the Standard Materials screen.



In the next steps, a list of Standard Materials or “calibration standards” will be defined along with their compositions. This list will ultimately be used to calibrate the XRF instrument for analyzing these types of Materials.

Type the name of the first calibration standard (**“SS 401”**) in the New Standard Material box.

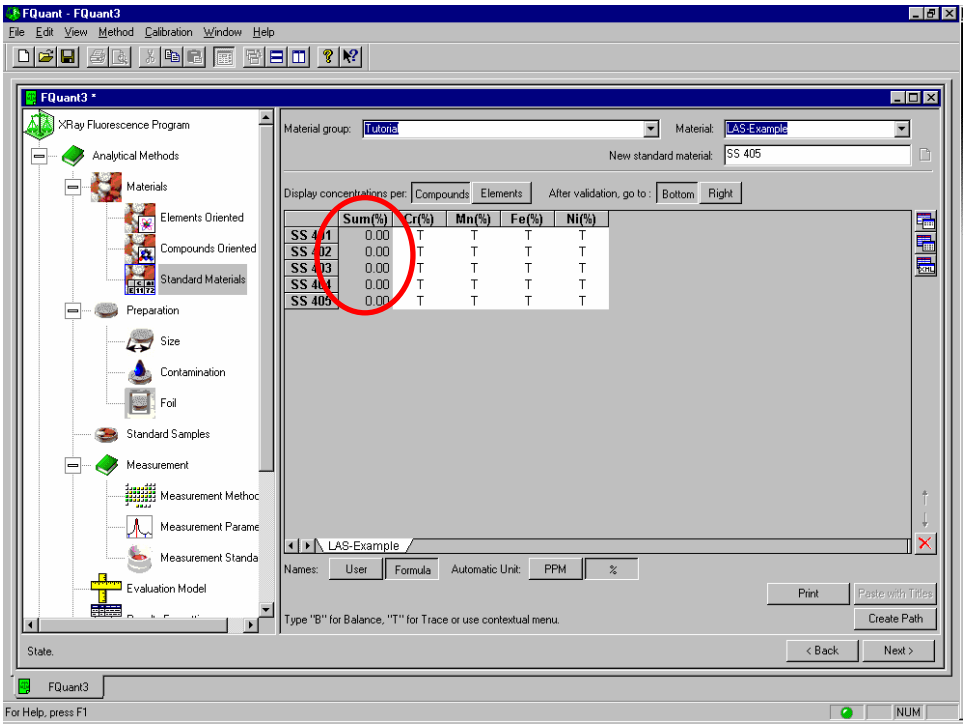
Click the **New** icon or the enter key to add this name to the list of Standard Materials. DO NOT USE symbols such as /?$&:, or leading and trailing blanks.



“SS 401” has been added to the list of “Standard Materials”.

Continue to enter the names for each of the five calibration standards: **“SS 401”, “SS 402”, “SS 403”, “SS 404” and “SS 405”**.

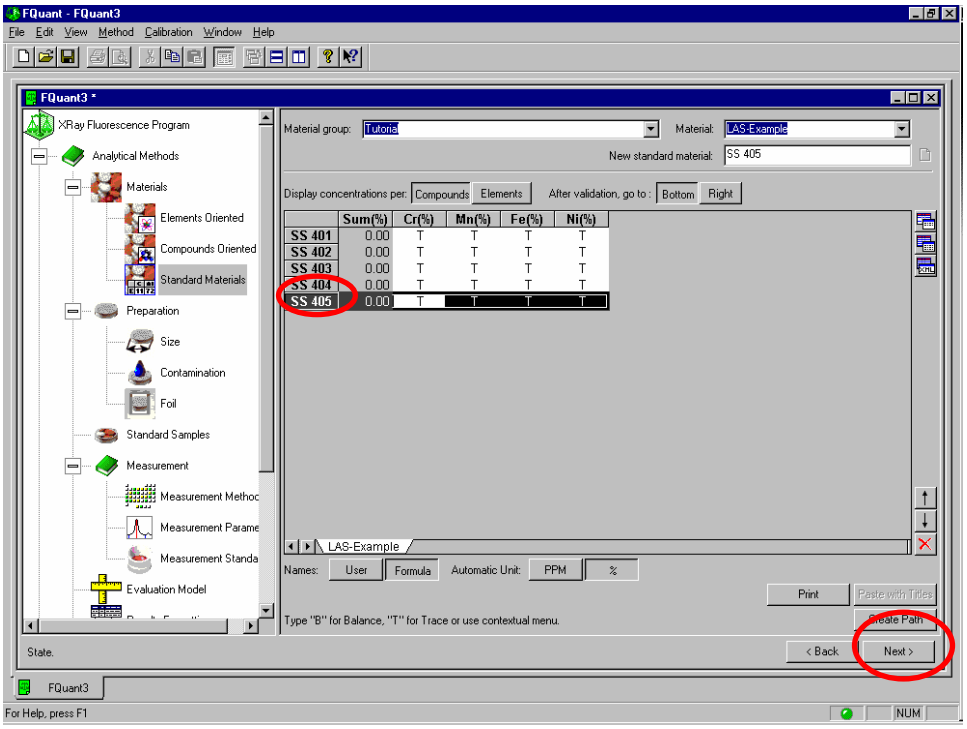
Click the **New** icon after each name has been entered.

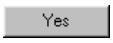


If a mistake is made when entering the names, this can be corrected by:

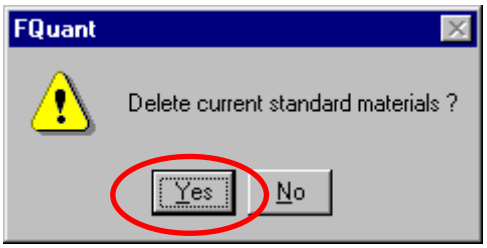
Select std to delete — **Click** on the name of the standard entered in error until its row is highlighted.

 Click the **Delete** icon.



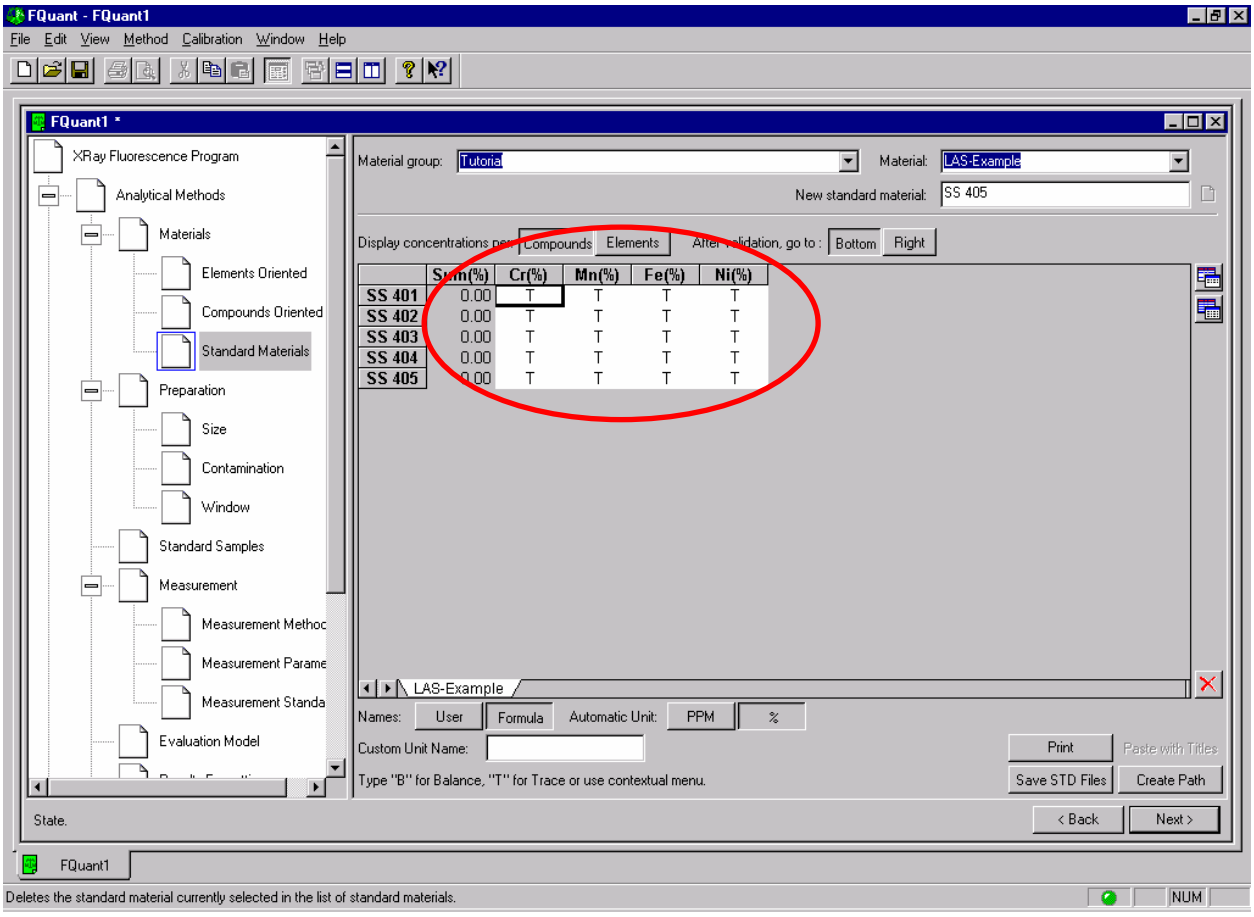


Click the **Yes** in the warning window to delete this standard.

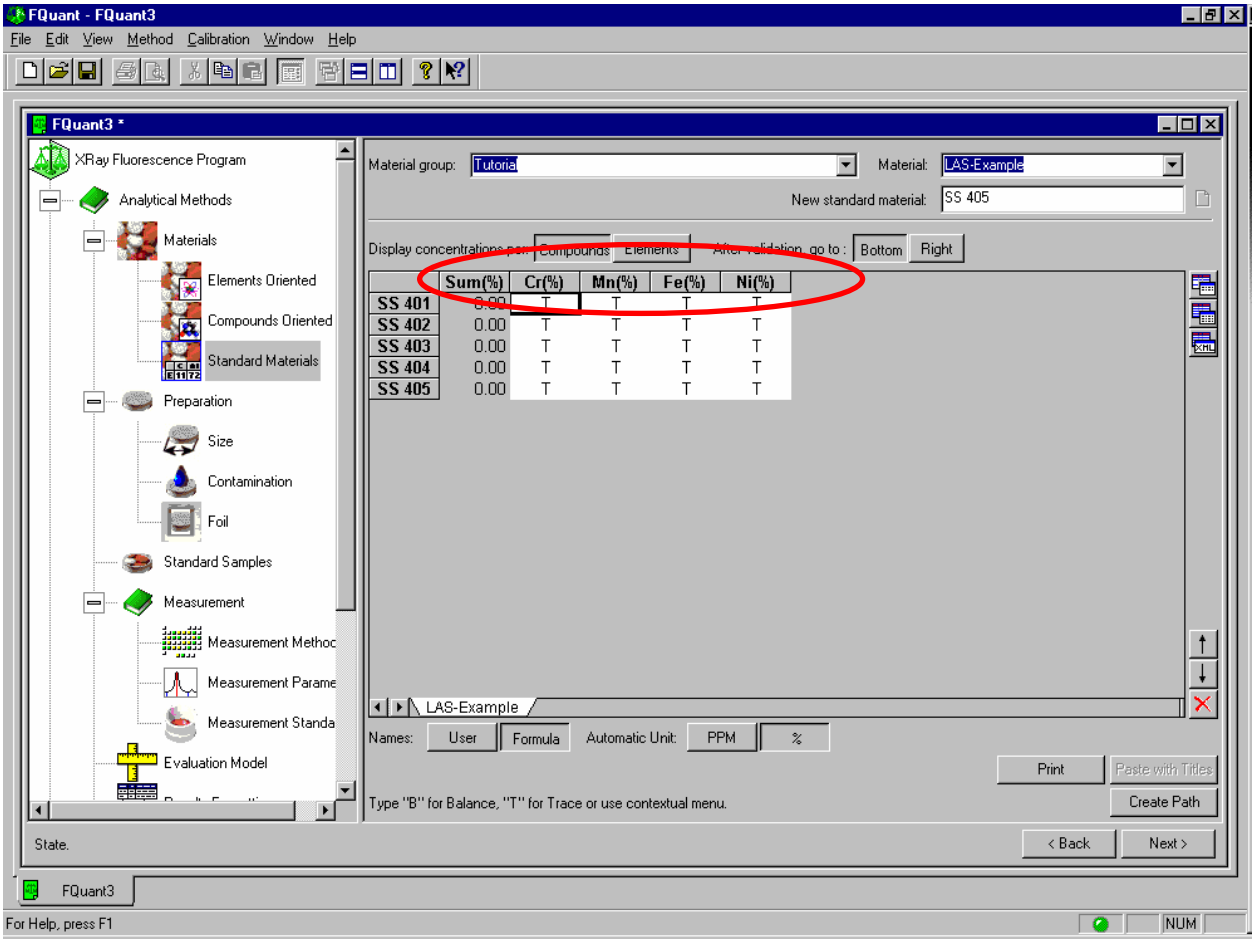


Once the names for all the calibration standards have been entered, the next step will be to set the display format for each element. In this example all element concentrations are in weight-% and will use 3 fixed decimal places, so the format for all elements can be set at once instead of setting them individually.

Left-click anywhere in the white cell area to select it.



Left-click the first cell, hold down the left mouse button and drag the mouse to highlight one row across all the columns. It doesn't matter which row is selected.

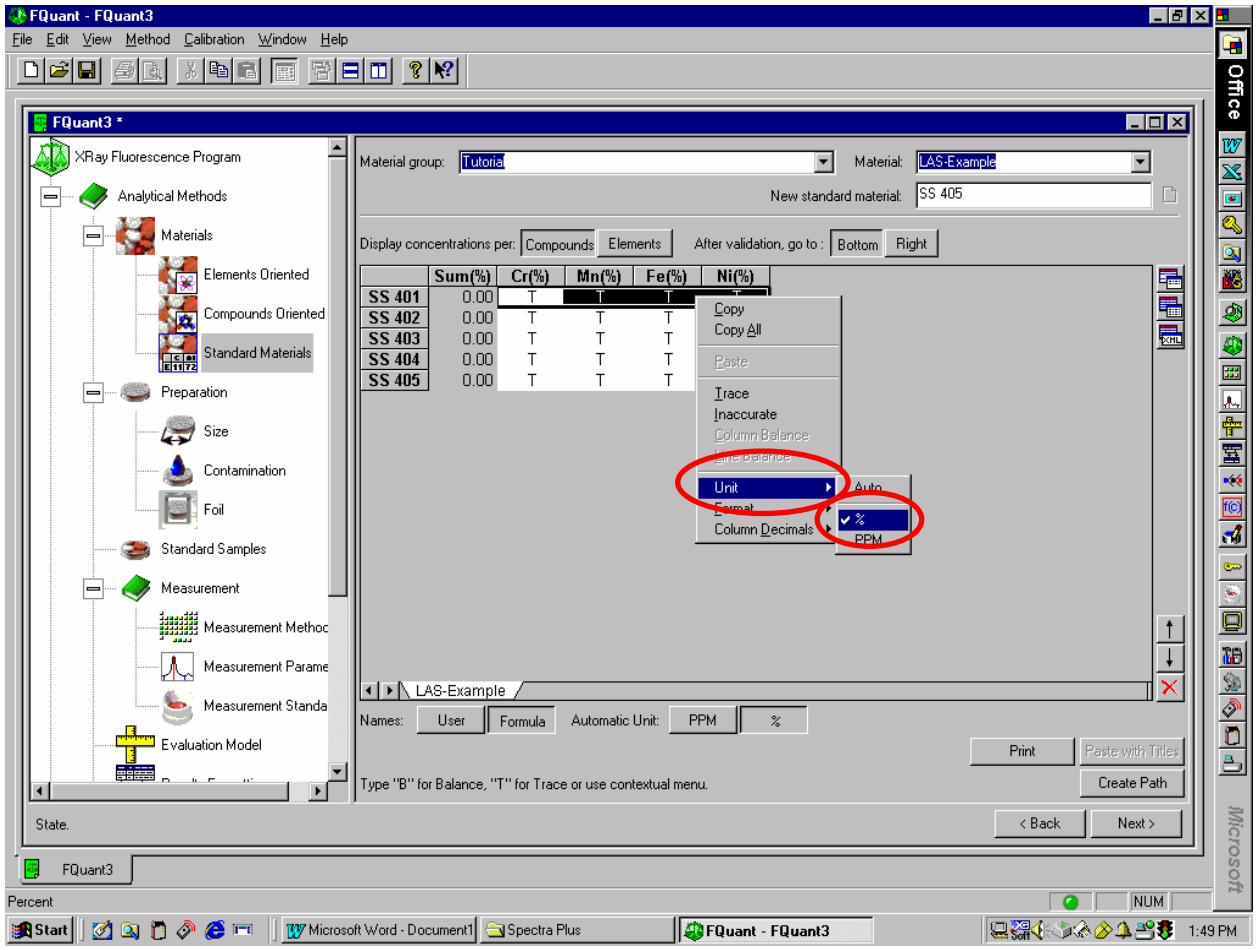


T

Unit

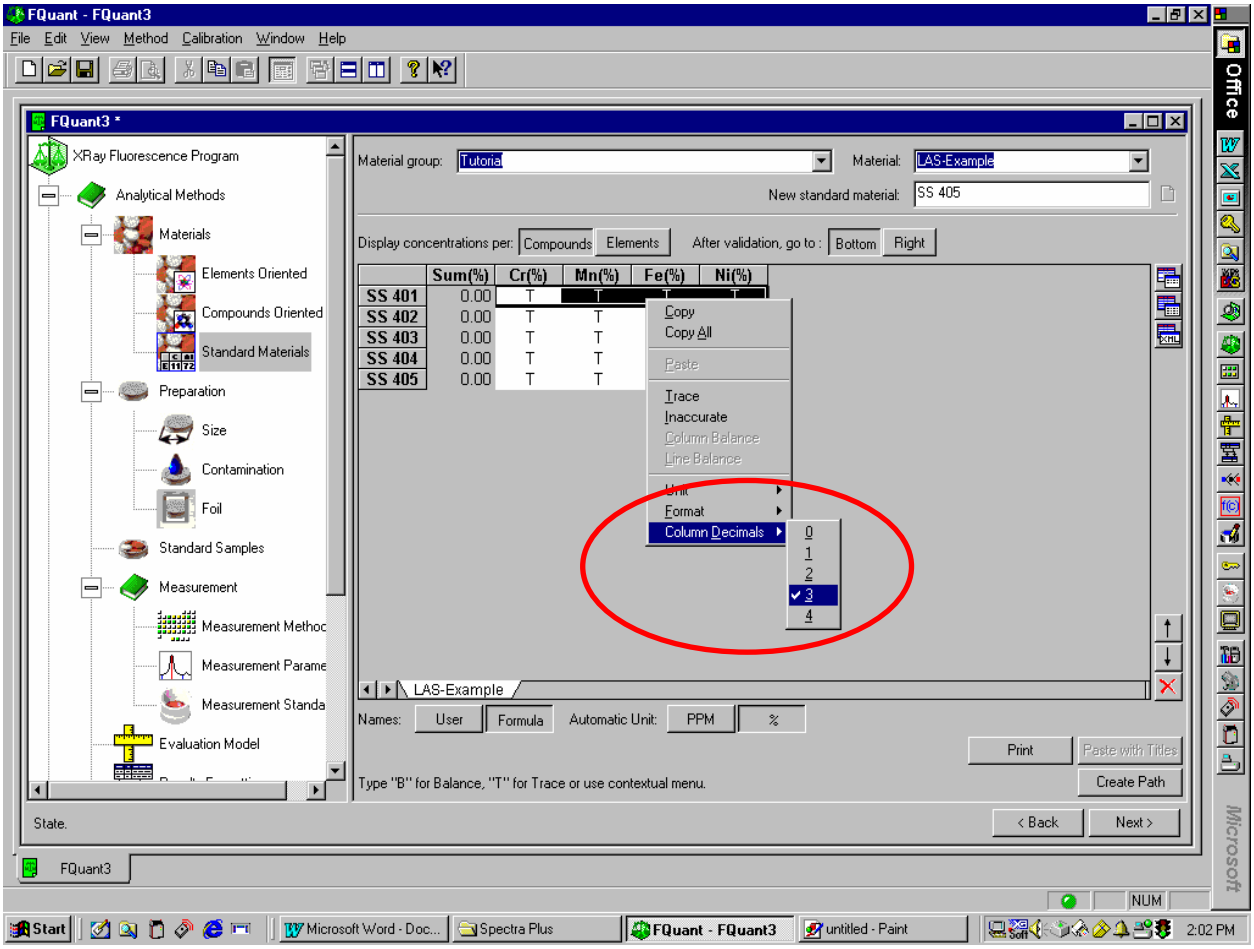
Right-click any of the highlighted cells.
Select **Unit**, then **%** from the pop-up menus.

%



Make sure that one row is still highlighted; if not, re-select one row.

Right-click on any of the highlighted cells.
Select **Column Decimals**, then **3** from the pop-up menus.



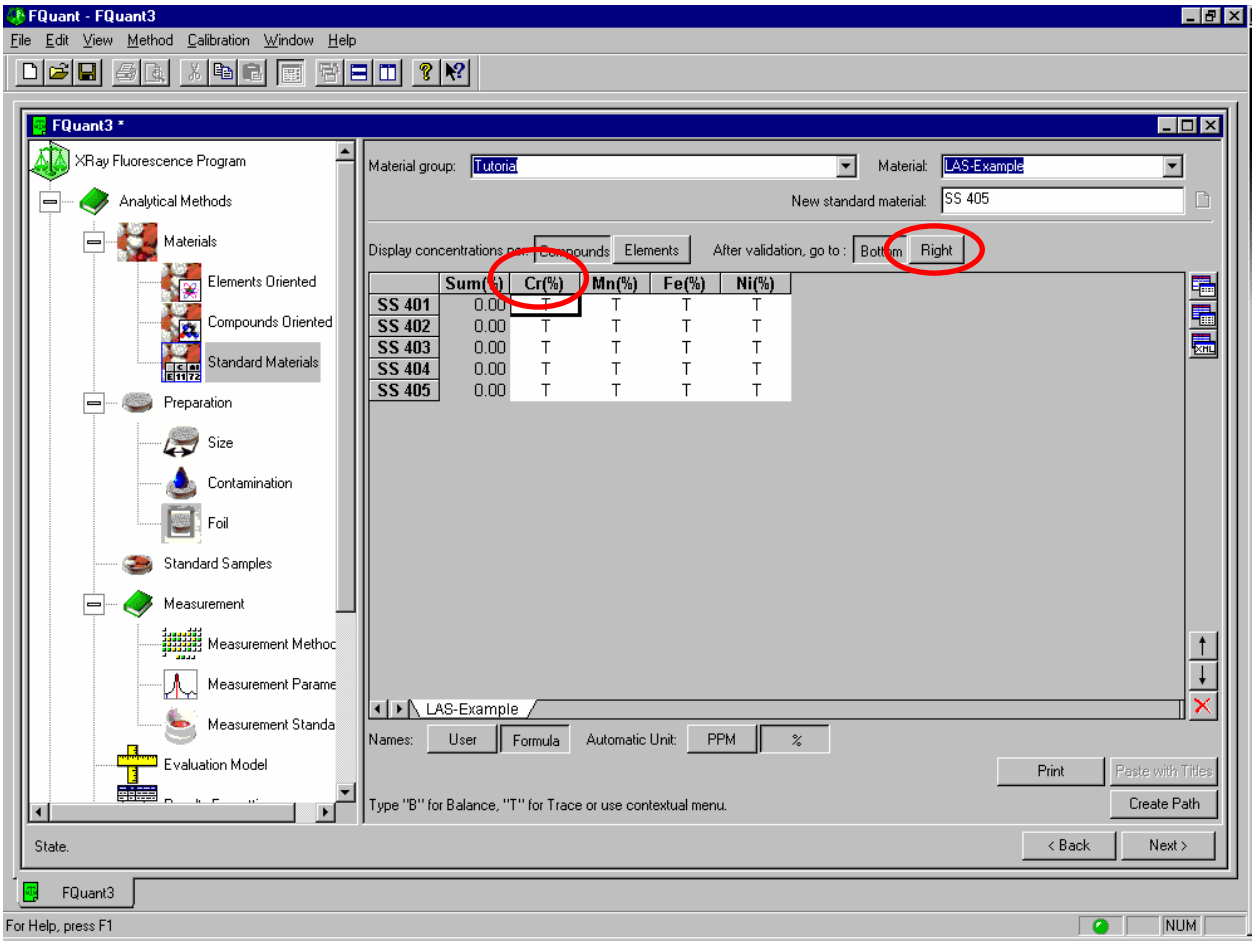
T

Left-click on the first cell to select it for input.

Note that initially all concentrations are set to “T” for “Trace”. This means that the concentration is not known, but is assumed to be close to zero.

Bottom Right

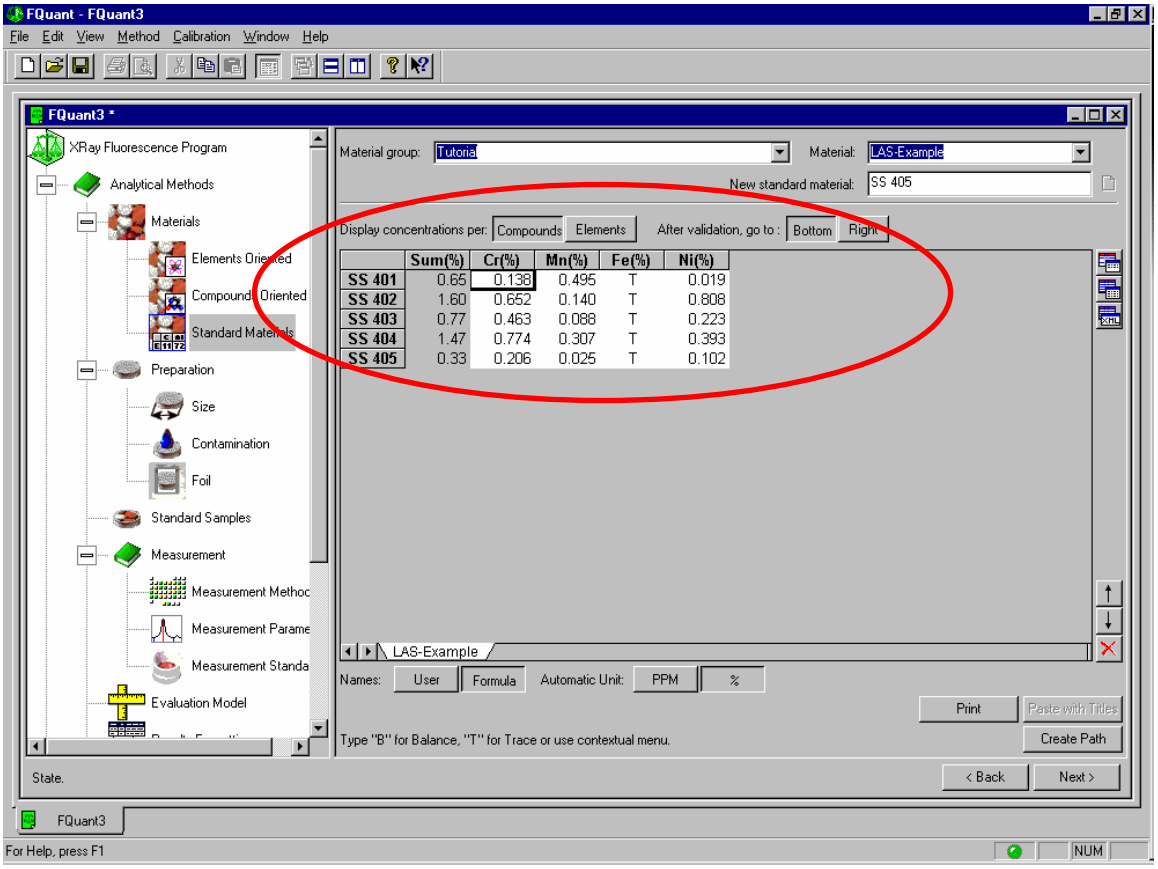
Use the **Bottom** and **Right** buttons to control in which direction the next cell will be selected after pressing the **<Enter>** key.



Cr(%)	Mn(%)	Fe(%)	Ni(%)
0.08	1.00	T	0.02
0.55	0.19	T	0.73
0.42	1.69	T	0.24
0.68	0.52	T	0.46
0.21	1.28	T	0.12

Enter the concentration values given in Table 1 into this form or use the values from the box of your installation standards, e.g., SS XXX/3.

Do not enter any values for Fe at this time.

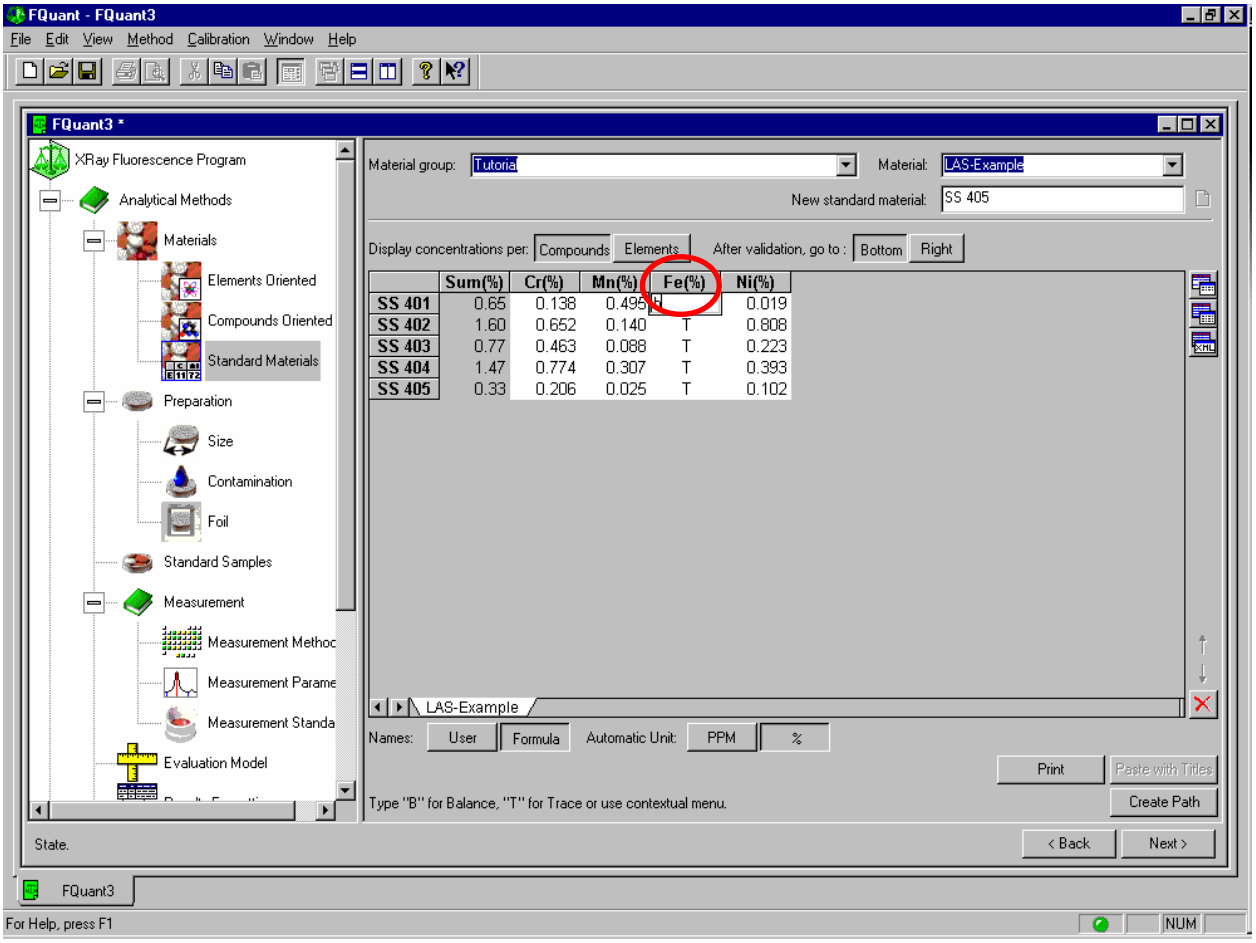


Standard	Cr(%)	Mn(%)	Fe(%)	Ni(%)
SS 401	0.08	1.00	Bal	0.02
SS 402	0.55	0.19	Bal	0.73
SS 403	0.42	1.69	Bal	0.24
SS 404	0.68	0.52	Bal	0.46
SS 405	0.21	1.28	Bal	0.12

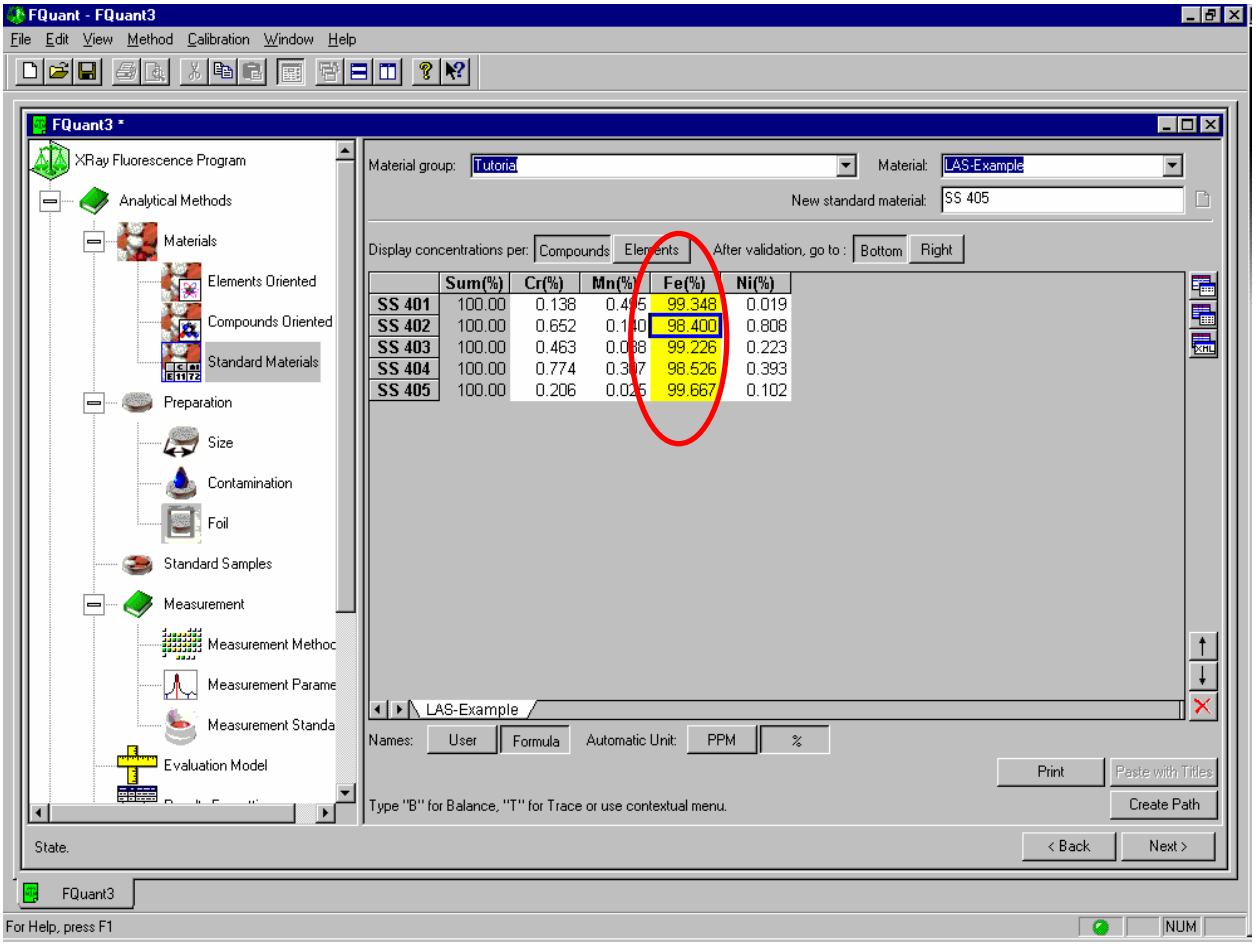
Table 1 - Concentration values for the LAS calibration standards (these are for LOT 0! Use the ones from your BOX!)

Type a **"b"** for one of the concentration values for Fe, then press the **<Enter>** key.

This indicates to SPECTRA^{plus} that Fe will be calculated as the "balance" element instead of being measured by XRF like the other elements.



The column for Fe should now be displayed in yellow, which indicates that Fe is the “balance” or “matrix” element.

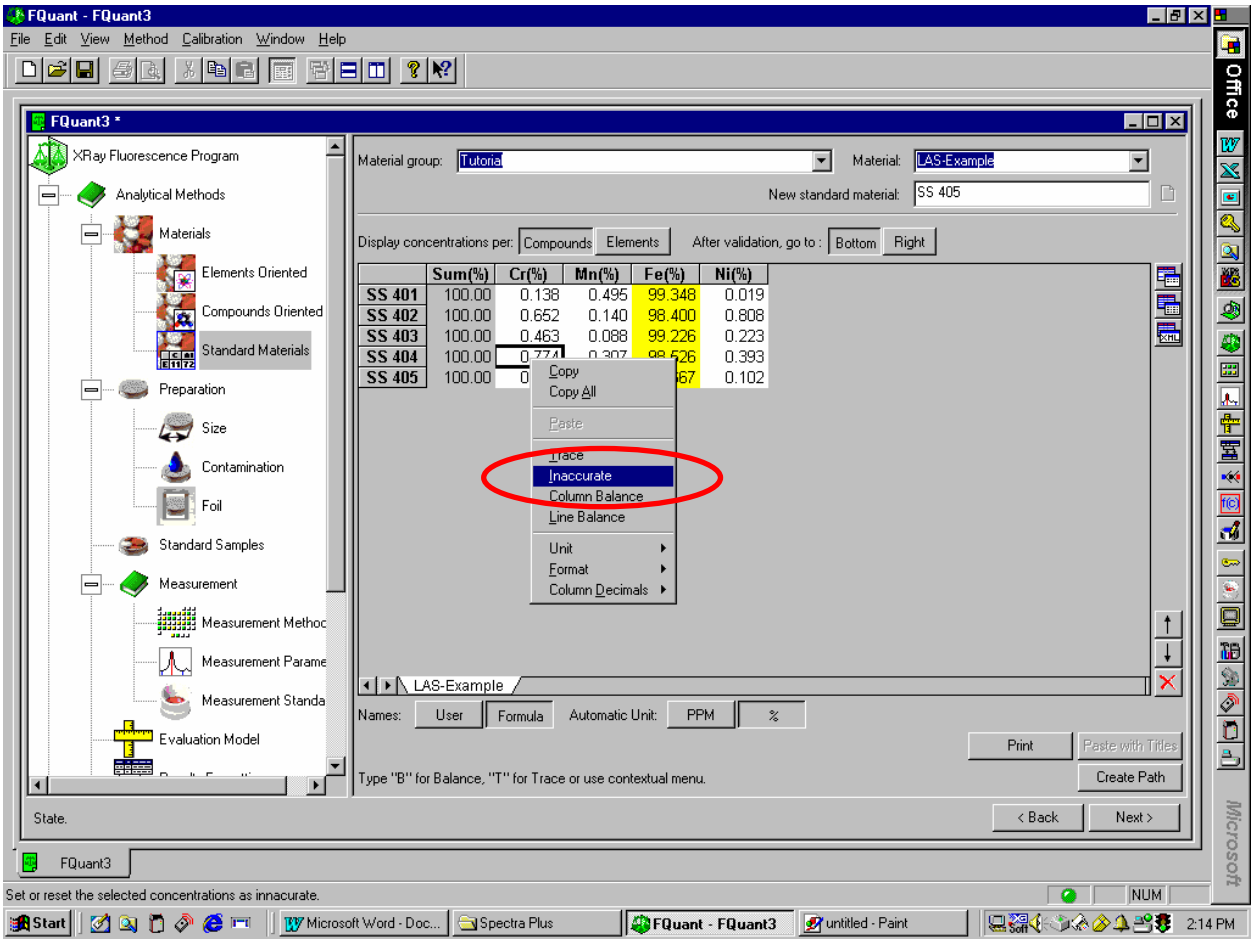


Sometimes approximate concentrations are known for some elements, but these values are not certified. SPECTRA*plus* allows flagging these uncertified concentration values as “inaccurate”.

To flag a concentration value as inaccurate:

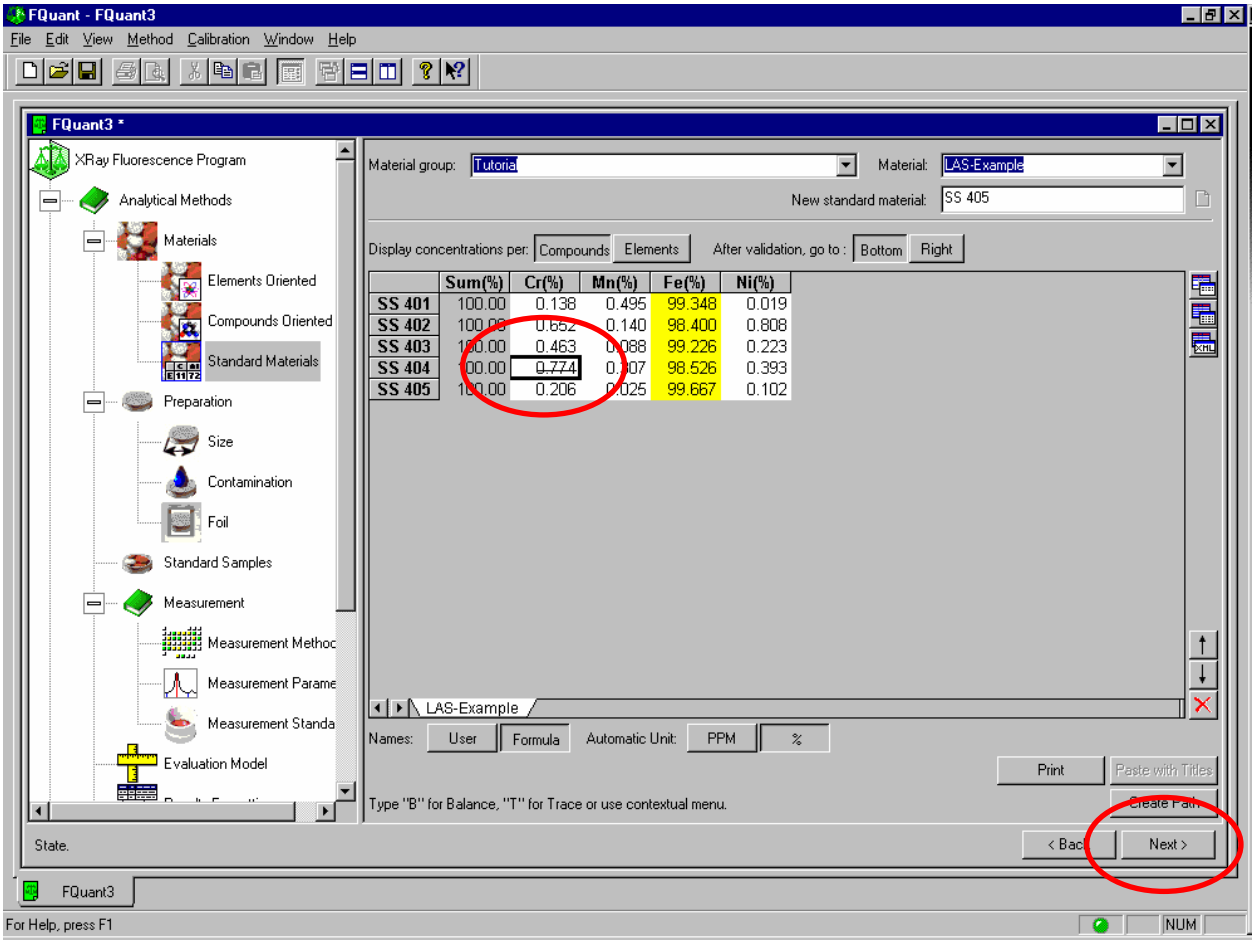
Inaccurate

Right-click on the value and select **Inaccurate** from the pop-up menu.



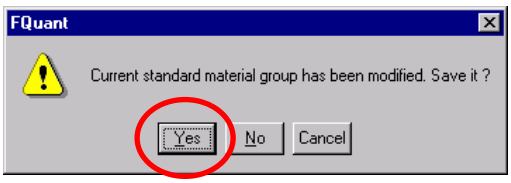
Concentration values declared as inaccurate are displayed with a line drawn through them.

NOTE: If you have set any of the concentrations to “inaccurate”, reset them before proceeding.



Click the **Next** button to go to **Preparation**.

Click the **Yes** button to save the changes that were made in the calibration database (\SPECplus\Databases\Fluo.mdb).



3.3 Defining Specimen Preparation

Preparation is used to define how specimens were prepared for measurement by the XRF Instrument. This will allow SPECTRA*plus* to know the “true” composition of a prepared specimen. This is important since the measured sample (specimen) is often not the same as the original sample.

On this screen we can select one of the SPECTRA*plus* pre-defined preparation methods, or define our own preparation methods.

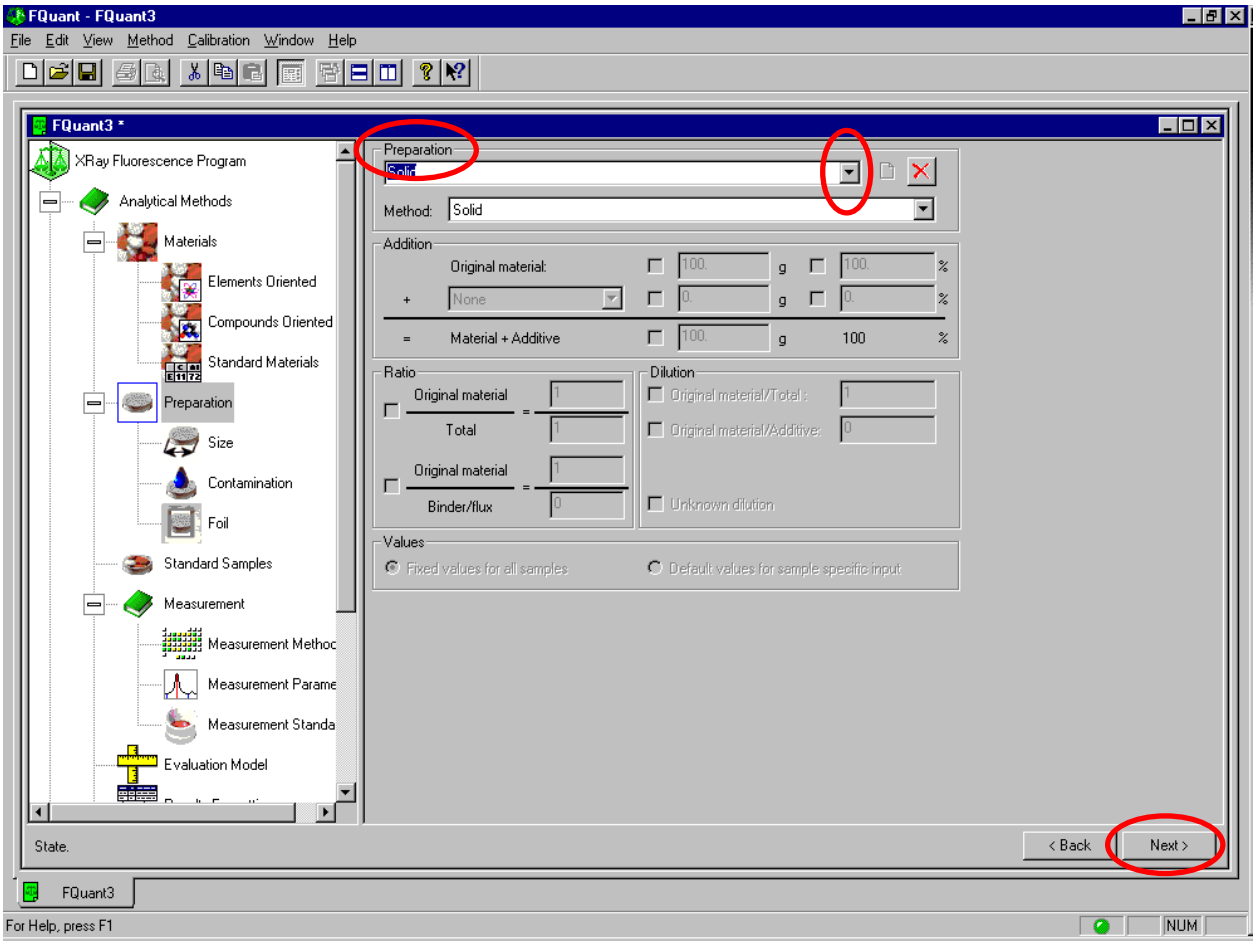
Defining a new preparation method is covered in the SPECTRA*plus* Tutorial Manual, but briefly it involves the following steps:

1. Type a name in the Preparation field, and click the **New** icon.
2. Select the method of preparation (Fused Bead, Pressed Pellet, etc.)
3. If anything was added to the sample (binder, flux, etc.) select this in the “Addition” field. Note: Only Materials from the special Material Group named “Additives” can be selected here.
4. Define the weights or ratios of the original Material and any added compound.

In this example, the specimens were prepared as “Solid” samples with nothing added.

Click the drop-down control for **Preparation** and select **Solid** as the Preparation.

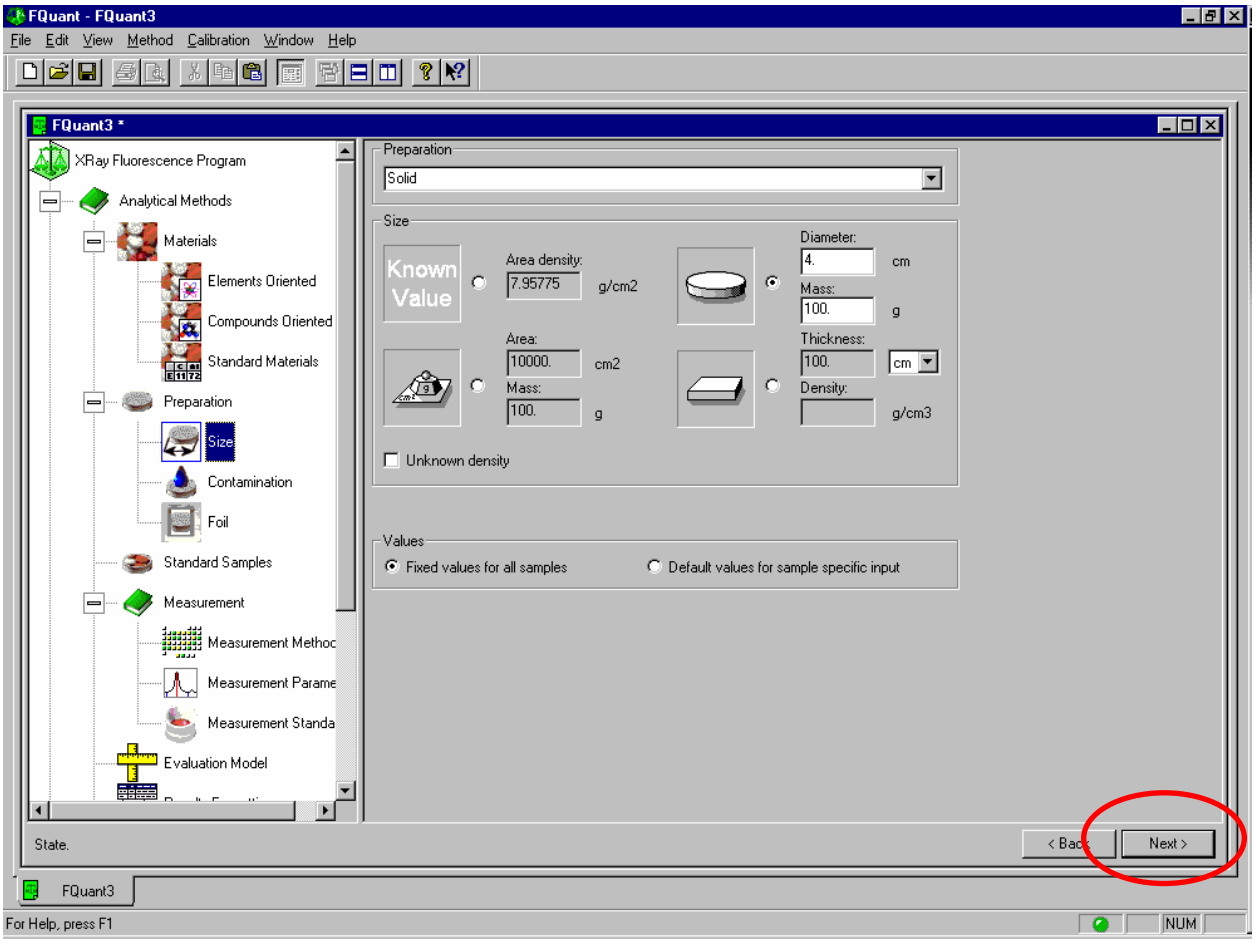
Click the **Next** button to move to Size.



Size allows entering an Area Density parameter for the prepared specimens. This will allow SPECTRA^{plus} to correct the intensities for samples that may not be infinitely thick to the X-rays being measured.

In this example, the samples are infinitely thick.

Click the **Next** button to move to “Contamination”.



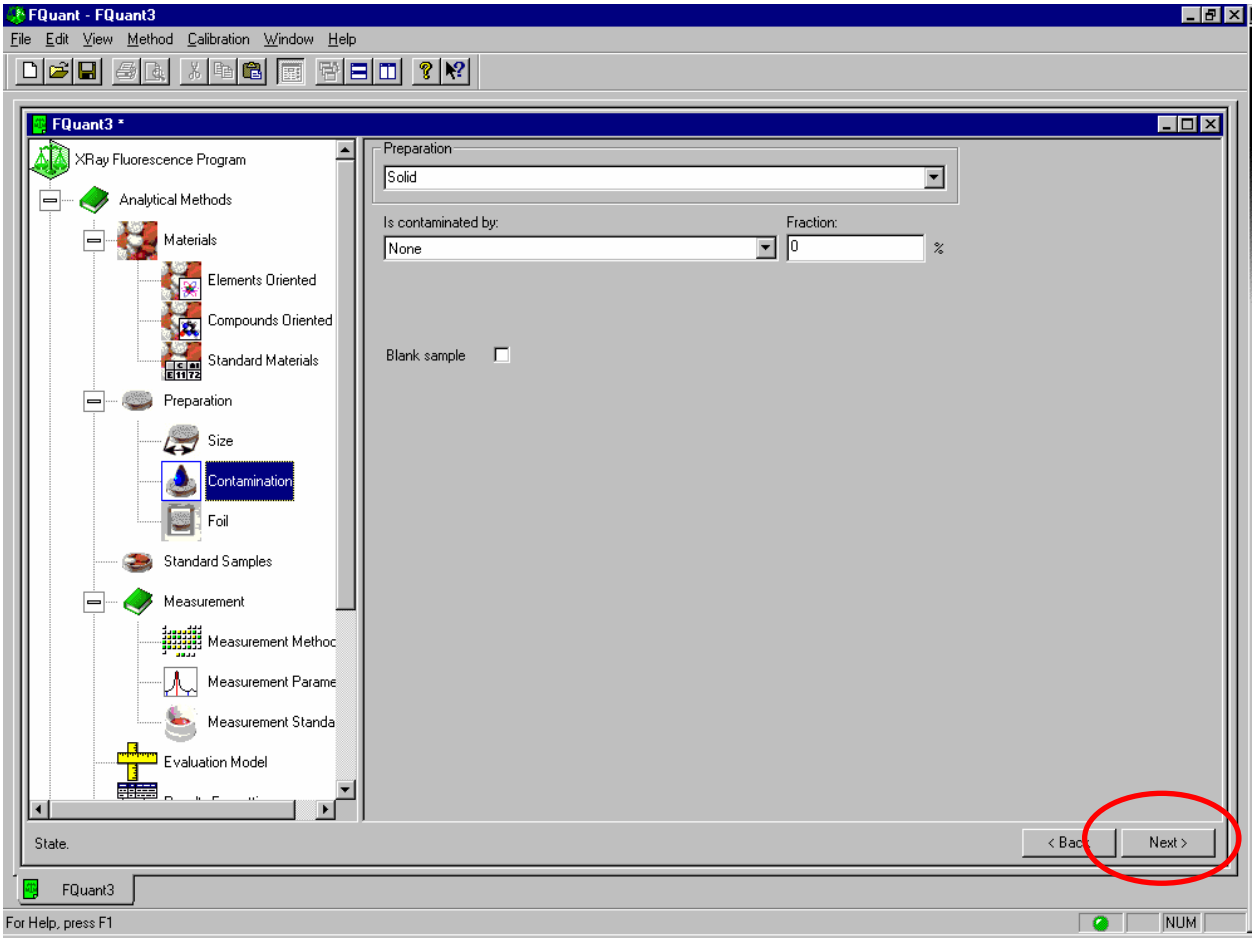
Contamination involves selecting a Material from the “Contamination” Material Group, and specifying the percent of this contaminate being picked up by a sample during its preparation.

This is also where a “blank” sample can be declared.

See the SPECTRA*plus* Reference Manual (M84-Exx025) for more information on this topic.

In this example, the samples are not contaminated during the preparation phase.

Click the **Next** button to move to **Foil**.

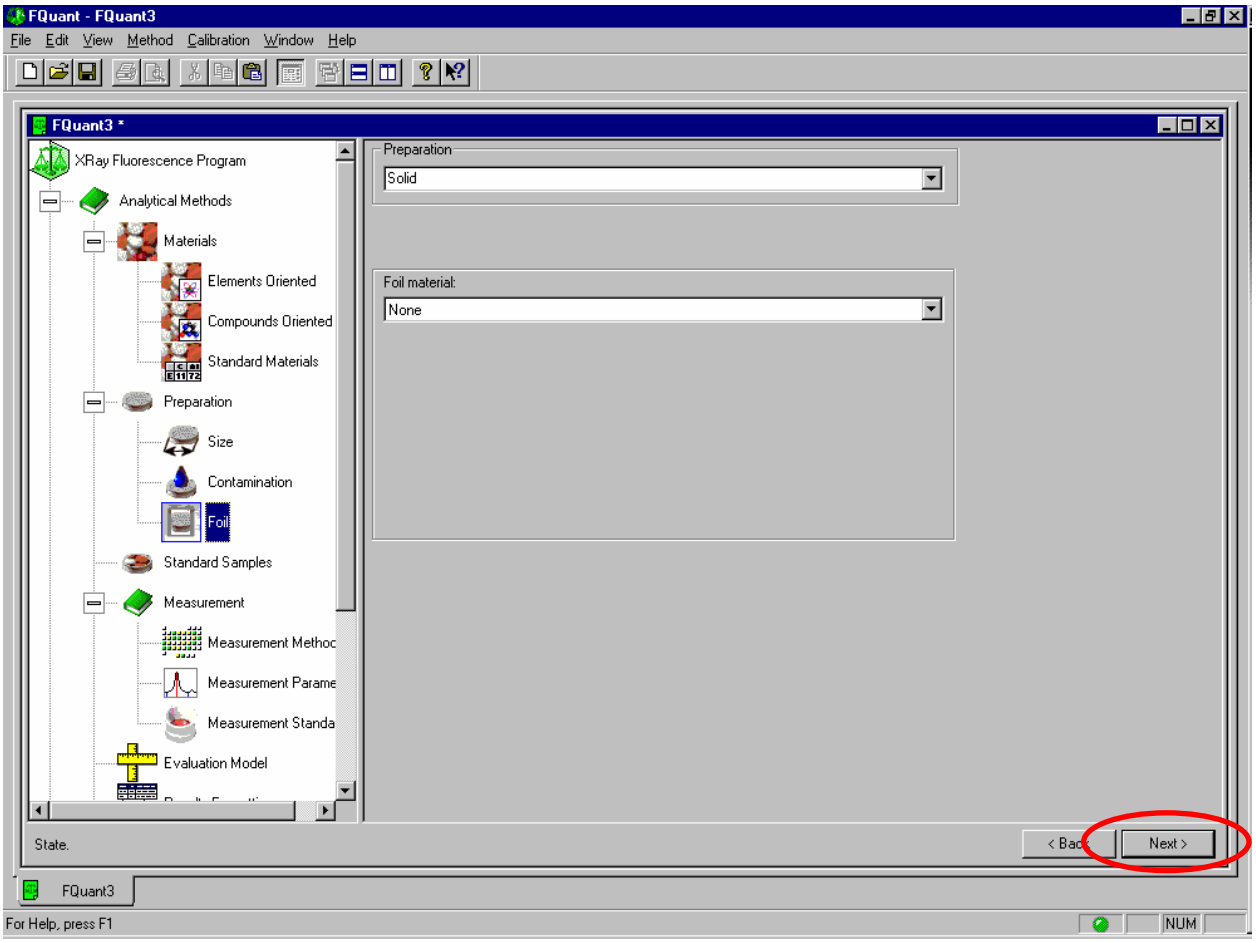


Foil allows selecting a Material from the “Foils” Material Group, and specifying the Area Density of this foil. This allows SPECTRA*plus* to adjust the measured intensities for their absorption by this foil.

See the SPECTRA*plus* Reference Manual (M84-Exx025) for more information on this topic.

In this example, the samples are not covered with a foil.

Click the **Next** button to move to **Standard Samples**.



3.4 Creating Standard Samples

The primary purpose of this step is to create a set of Standard Samples (or “Prepared Specimens”) by applying a Preparation Method to the Standard Materials (or “Calibration Standards”). This allows a conversion of the concentrations in the Standard Materials to concentrations in the Standard Samples (or “Prepared Specimens”). The concentrations in the Standard Samples (or “Prepared Specimens”) will include any Additives and Contaminates that have been declared under “Preparation”, and will be minus any volatiles if the Preparation Method indicates a “Fused” sample.

From this point on, SPECTRA^{plus} will use the “elemental concentrations” on a “prepared specimen” basis.

This step is important for many reasons, including the following:

The XRF instrument is measuring intensities from the elements in the prepared specimen, not from compounds in the original material, although in some cases these may in fact be the same.

SPECTRA^{plus} can make intelligent choices when selecting X-ray lines to measure in later steps, because it can base its selection on the concentration of each element in the prepared specimen instead of in the original material.

In order for the Fundamental Parameter-based matrix corrections to work properly, SPECTRA^{plus} needs to know the composition of the specimen that is actually being measured by the XRF instrument.

☑ Short names

Make sure “Short names” is checked. This keeps the Standard Sample name the same as the Standard Material name.

☐ All preparations

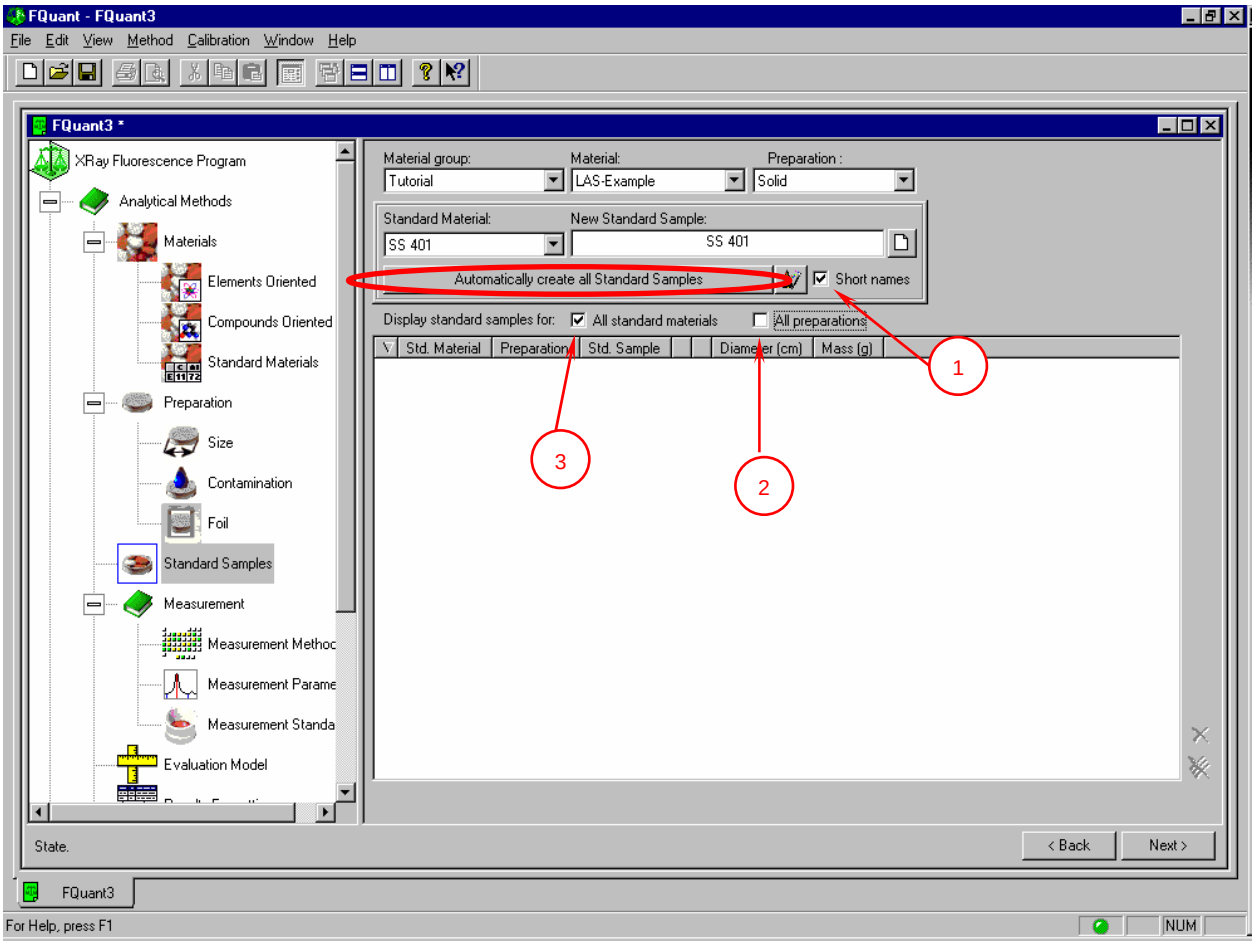
Make sure “All preparations” is un-checked. This will only show Standard Samples prepared as “Solid”.

☑ All standard materials

Make sure “All standard materials” is checked. This shows Standard Samples for all Standard Material names, not just the Standard Material currently selected.

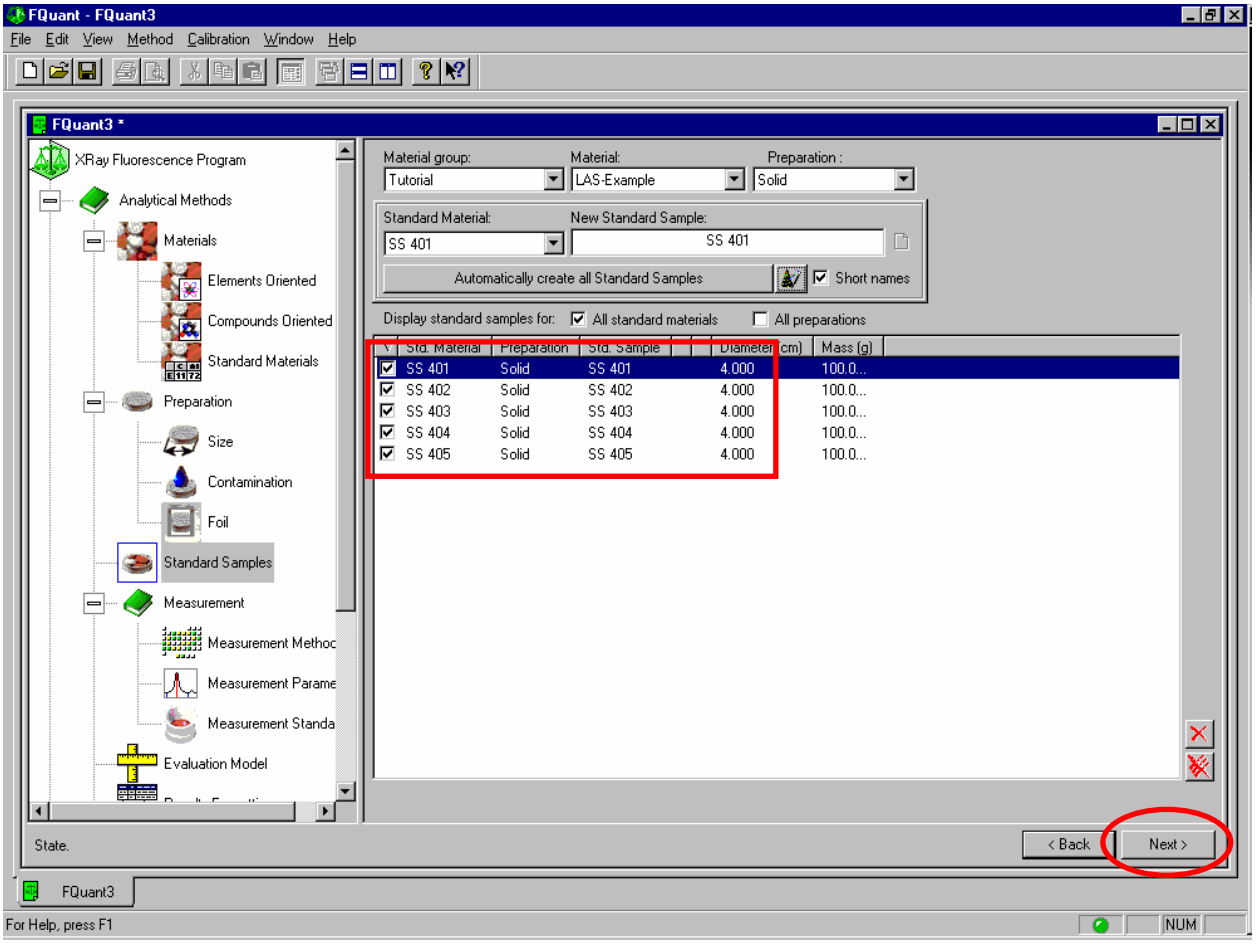
Automatically create all Standard Samples

Click the **Automatically create all Standard Samples** button.



The list of Standard Samples has now been created.

Next > Next > Click **Next** twice to go to **Measurement Method**.

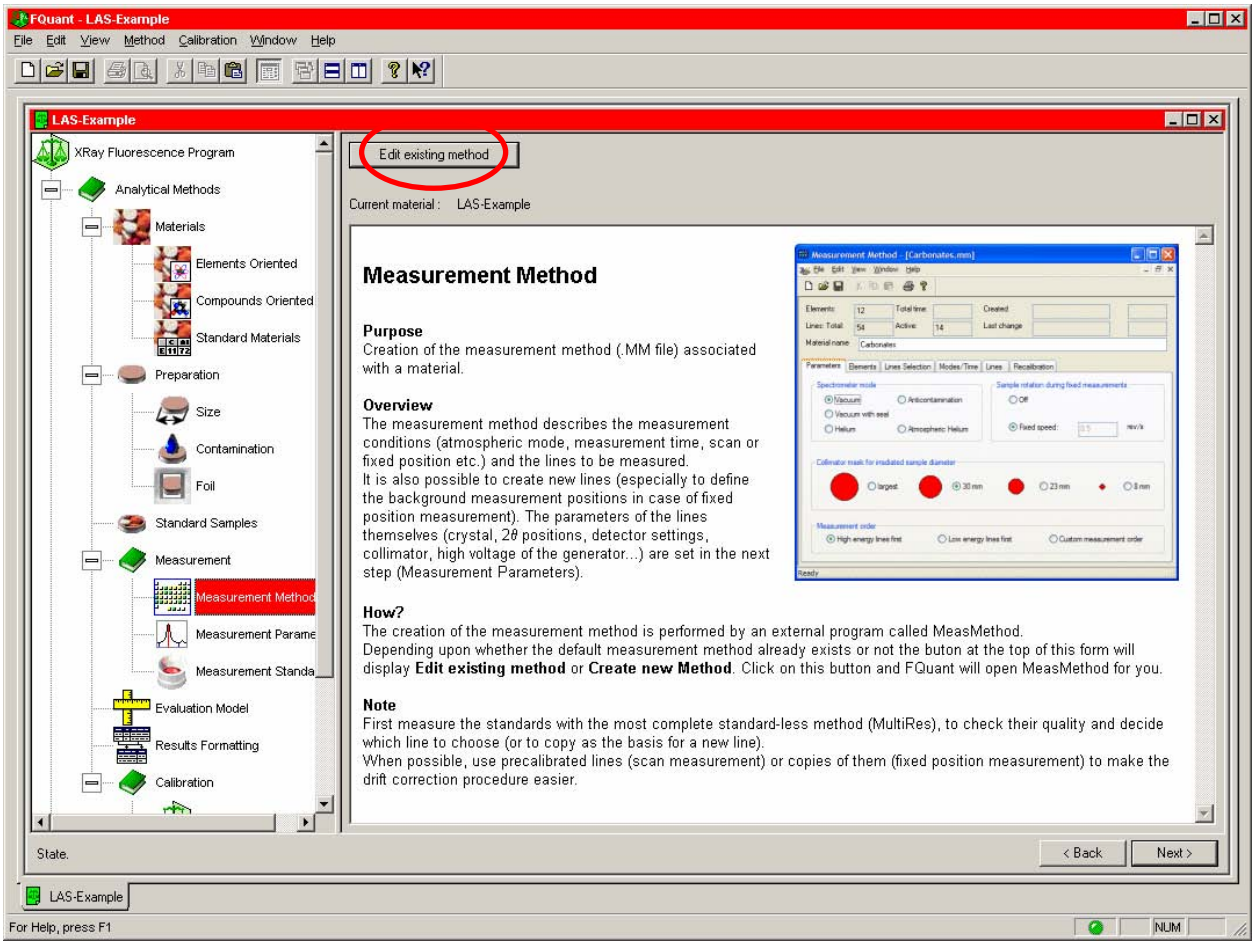


3.5 Creating a Measurement Method

In this step, a formal Measurement Method (*.mm) will be created. This defines which intensities to measure, and which spectrometer conditions to use when making these measurements.

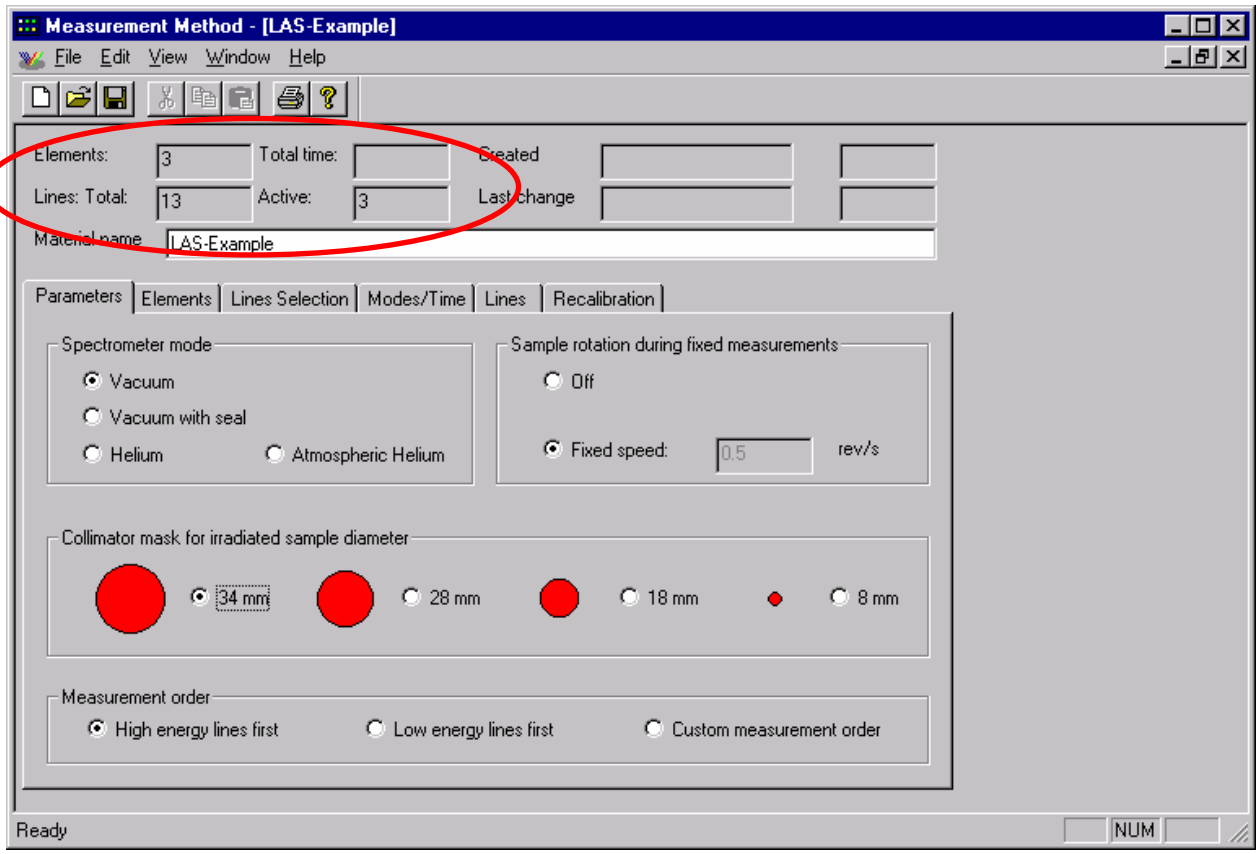
Launch

Click the **Edit Measurement Method** button to start the Measurement Method program.



SPECTRA^{plus} has already done most of the work.

For the 3 measured elements in our program (Ni, Cr and Mn) there were 13 possible lines in the Line Library of the system used here that could have been used to characterize the 3 elements. Of these 13 lines, SPECTRA^{plus} chose 3 lines, one for each element.



Parameters

Click on the **Parameters** tab to select it.

Vacuum

The “Vacuum” radio button should be selected under “Spectrometer mode”.

Fixed speed:

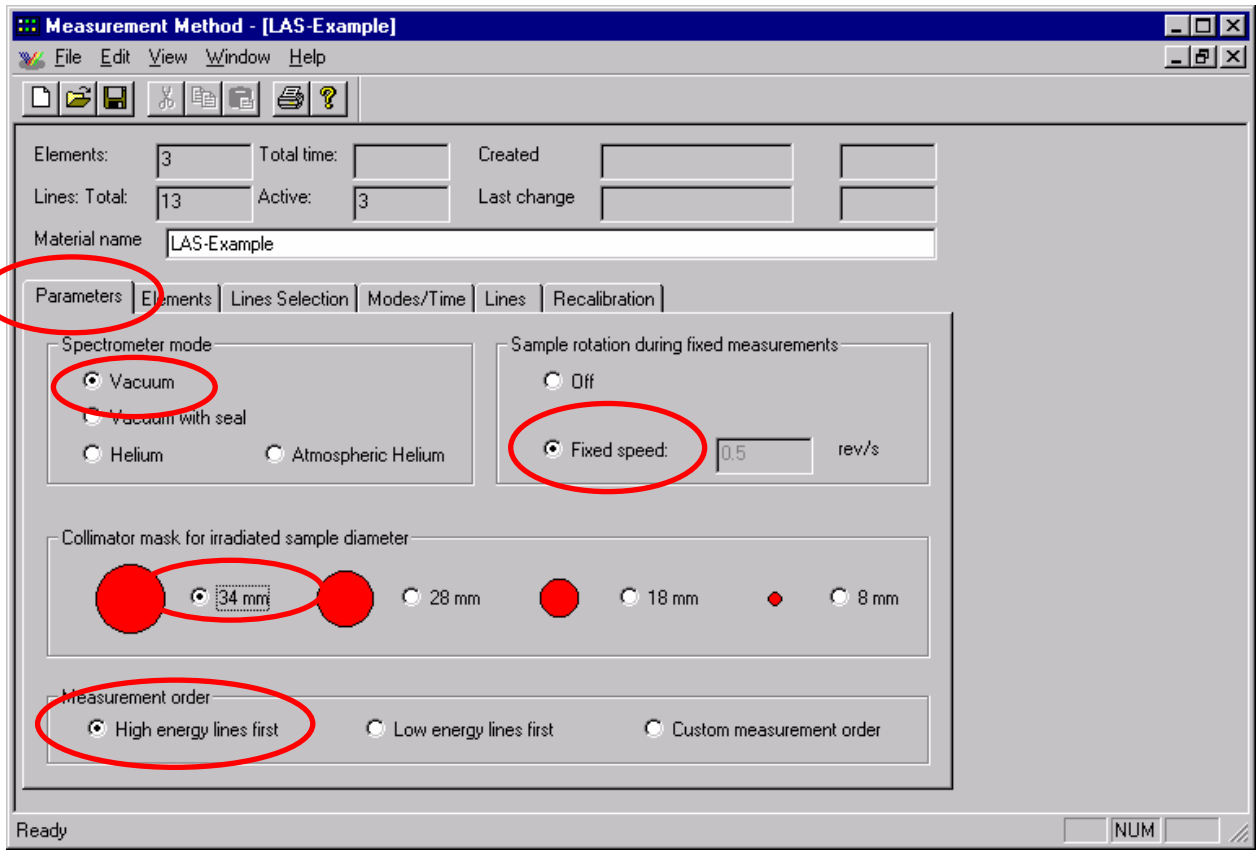
The “Fixed speed” radio button should be selected under “Sample rotation . . .”

34 mm

The “Mask” size radio button should be selected under “Collimator mask . . .” to match the sample holders being used.

High energy lines first

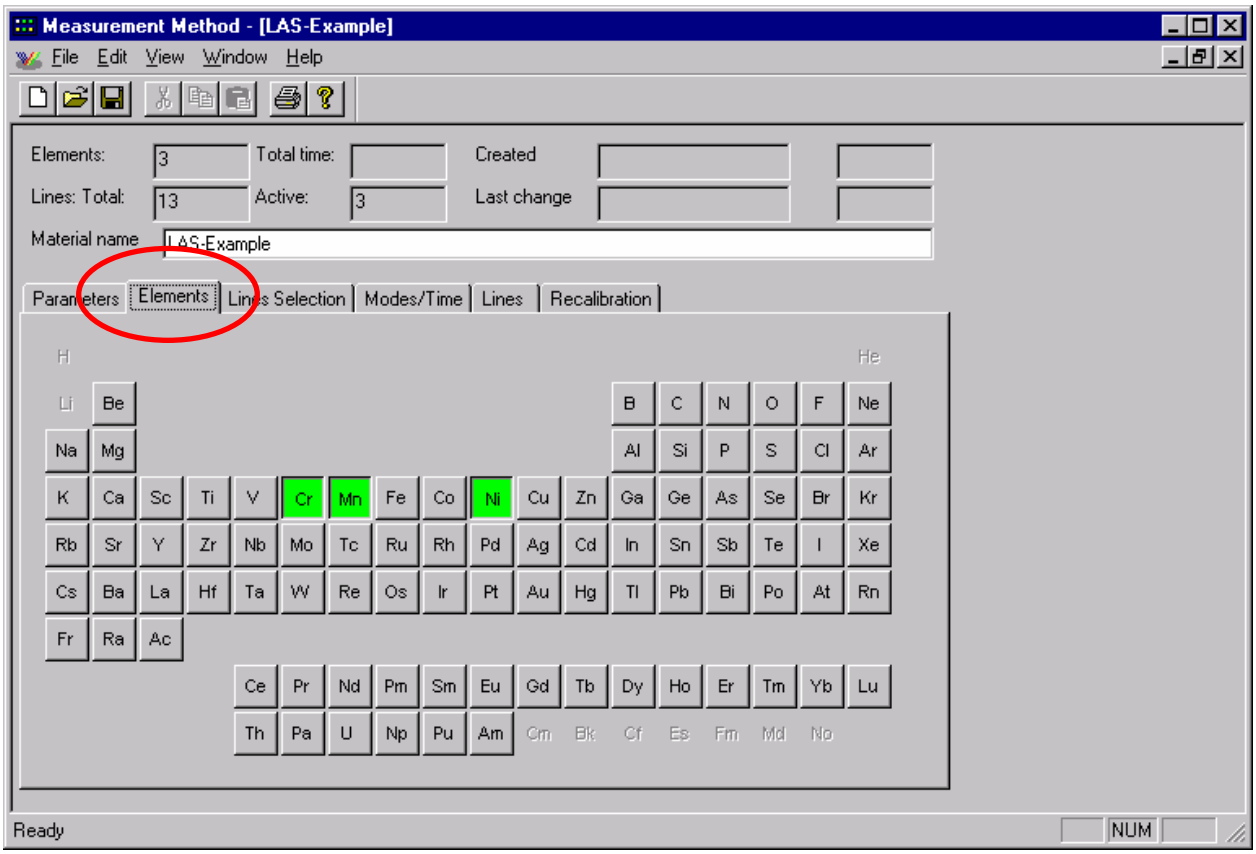
The “High energy lines first” radio button should be selected under “Measurement order”.



Click on the **Elements** tab to select it. There is nothing to do here, but the following should be noted:

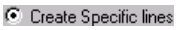
The measured elements (Cr, Mn and Ni) were automatically transferred from the Material defined in FQuant.

Fe was not transferred because it was declared as the “balance” element in FQuant.
(The column color was yellow in “Standard Materials”).



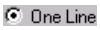
In the next step, we will make choices indicating how we want SPECTRA^{plus} to select the lines that are to be measured. SPECTRA^{plus} includes a complete Line Library with nearly all XRF lines already defined. Most lines have high resolution (HR) and high sensitivity (HS) versions defined, provided the hardware in the instrument supports these. Some lines are further classified into “Major” (Maj), “Trace” (Tra) and “Minor” (Min) categories. This allows SPECTRA^{plus} to select lines based on the major category (best resolution or best intensity), and then by the concentration level of each element in the prepared specimen (major, minor or trace). When these Library Lines are added to our Measurement Method they can be added directly (the line itself) or as a copy of the original line with the Material name appended to the end of the line name. Using copies of the Library Lines allows modification to the measurement conditions and independent drift correction of the lines selected.

Click on the **Lines Selection** tab to select it.



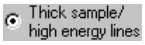
Select the “Create Specific lines” radio button.
This will give us an exact copy of the selected lines from the Line Library, except with the designation “LAS-Example” appended to the end of the line name. Because these are now separate lines in the Line Library, the measurement conditions used for these lines can be modified to make them optimal for these samples without affecting the other lines in the Line Library.

Enter the names of the Recalibration Samples or Drift Samples.
They were chosen because they will provide a reasonable low and high intensity for the three elements being measured.

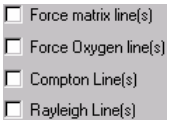


Select the “One Line” radio button.
This will cause only one line to be selected for each element.
SPECTRA^{plus} can select up to 3 lines per element.

Select the “Highest Intensity” radio button.
This will cause the “HS” (high sensitivity) lines to be selected from the Line Library.



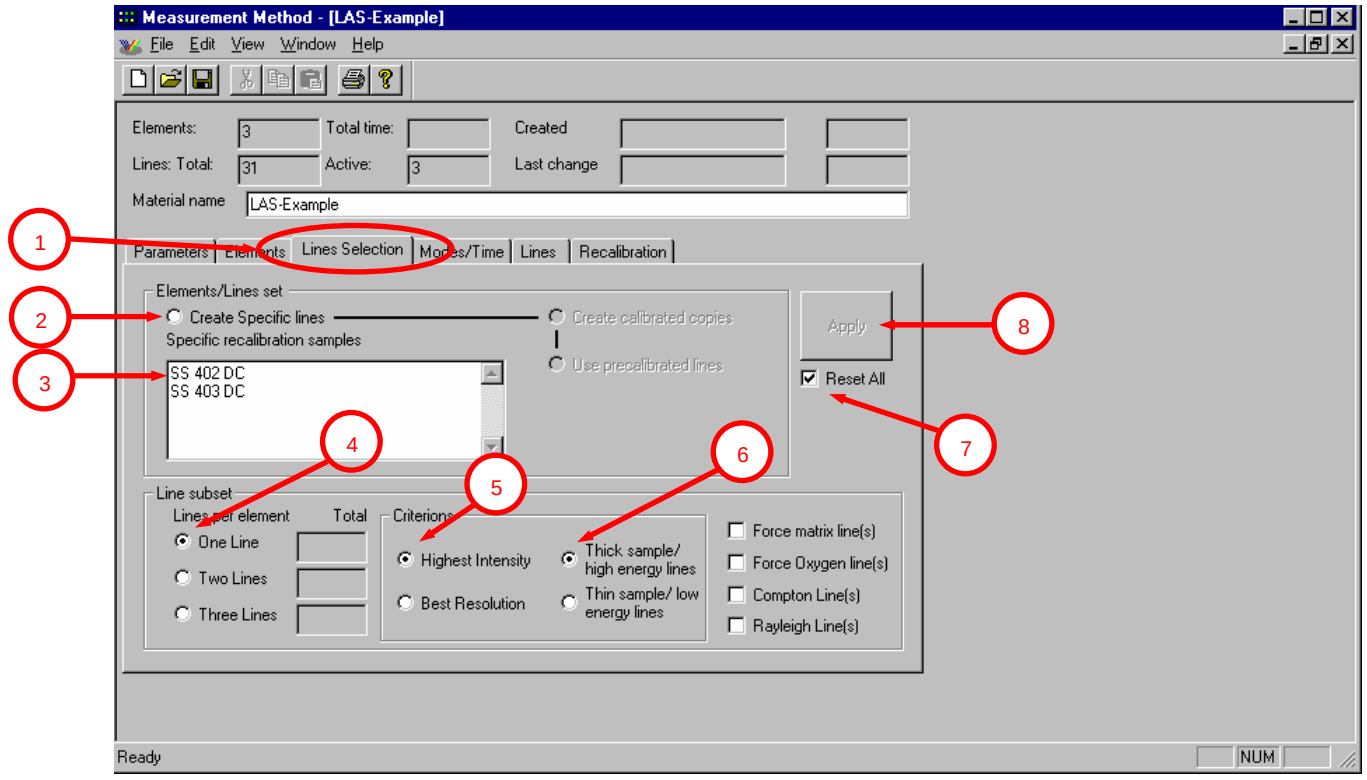
Select the “Thick sample/high energy lines” radio button.
This will cause K-lines to be selected instead of L-lines.



Uncheck the boxes to measure extra lines.
These allow tube scatter lines and matrix element lines to be measured.

Check the box to “Reset All” lines in the Measurement Method.
This will apply our selections to all the elements in our Measurement Method.

Click the **Apply** button to apply these selections to the Measurement Method.


Measurement Method - [LAS-Example]
File Edit View Window Help
Elements: 3
Total time:
Created
Lines: Total: 31
Active: 3
Last change
Material name
LAS-Example
Parameters
Elements
Lines Selection
Modes/Time
Lines
Recalibration
Elements/Lines set
Create Specific lines
Create calibrated copies
Specific recalibration samples
Use precalibrated lines
SS 402 DC
SS 403 DC
Apply
Reset All
Line subset
Lines per element
Total
One Line
Two Lines
Three Lines
Criterions
Highest Intensity
Best Resolution
Thick sample/ high energy lines
Thin sample/ low energy lines
Force matrix line(s)
Force Oxygen line(s)
Compton Line(s)
Rayleigh Line(s)
Ready
NUM
1
2
3
4
5
6
7
8

In the next step, we will make choices indicating how we want SPECTRA*plus* to measure the intensities from the lines that were selected. These choices include whether to measure the intensities as “scans” or at “fixed positions”, and the counting times to use. These choices can be set as “Global”, so they apply to all lines in the Measurement Method, or to “Local”, so they apply to certain lines.

For this example, we will measure all lines using fixed peak and fixed background positions. A counting time of 10 seconds will be used at each peak angle, and the same amount of time will be used at each of the background angles.

Click on the **Modes/Time** tab to select it.

Select the “Global parameters for method” radio button.
All lines will be measured the same.

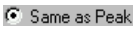
Make sure the **Measure at fixed Positions** button is pressed in “Peak Measurements” and “Background Measurements”.
All intensities will be measured using fixed positions.

Make sure the button for “Run scan measurements” is not pressed in “Peak Measurements” and “Background Measurements”.
No scan-based measurements will be made.

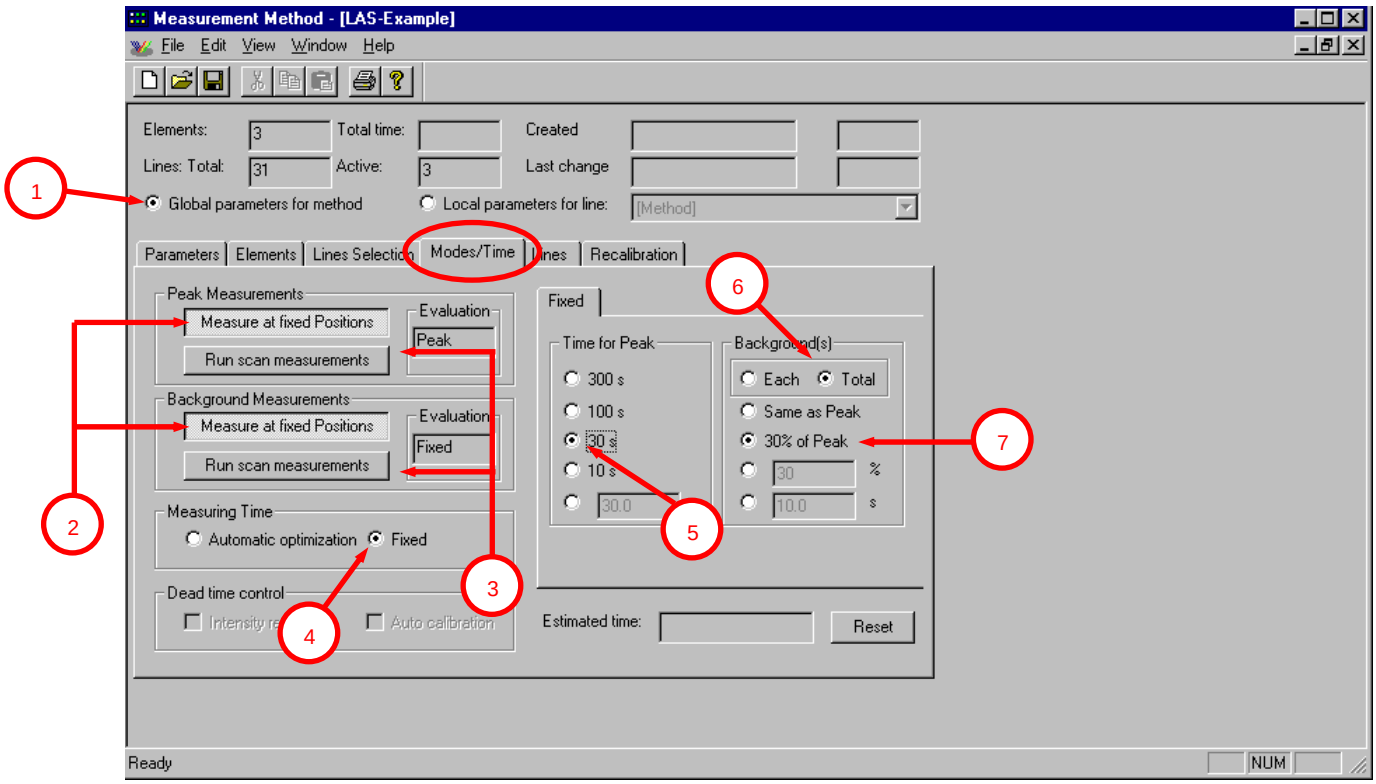
Select the “Fixed” radio button in “Measuring Time”.
All measurements will used fixed counting times, instead of counting to a set statistical error.

Select “30 s” as the counting “Time for Peak”.
Intensities will be measured for 30 seconds at each peak angle.

Select “Each” for the “Background(s)”.
Each background will have the same time, instead of splitting the background measurement time equally between each background position.



Select “Same as Peak” as the “Background(s)” time.
The background intensities will be counted for the same time as the peak intensities.



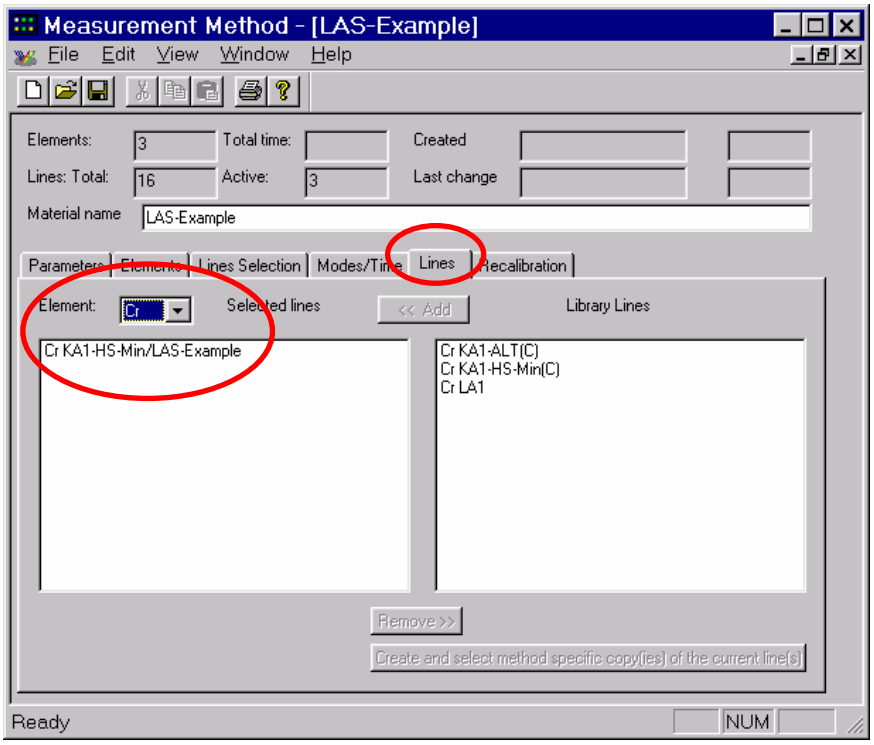
In the next step, we will look at the lines that were chosen by SPECTRA*plus* for inclusion in our Measurement Method. If we wanted to pre-select the lines so we could have a mix of “High Resolution” and “High Sensitivity” lines, we could have done this step before doing the “Lines Selection” step on page 3-36. The only thing we would need to do differently in the “Lines Selection” step would be to NOT CHECK “Reset All”.

Click on the **Lines** tab to select it.

Click on the drop-down arrow by “Element” and select “Cr” from the list of elements.

Note that Cr uses a “High Energy” line (K-alpha). The conditions are set to “High Sensitivity” (HS) for a minor concentration (Min). Also, the line is a copy of the original line with the Material name (LAS-Example) appended to it.

Click on the drop-down arrow by “Element” and look at the other two lines.



Click on the **Modes/Time** tab to select it.

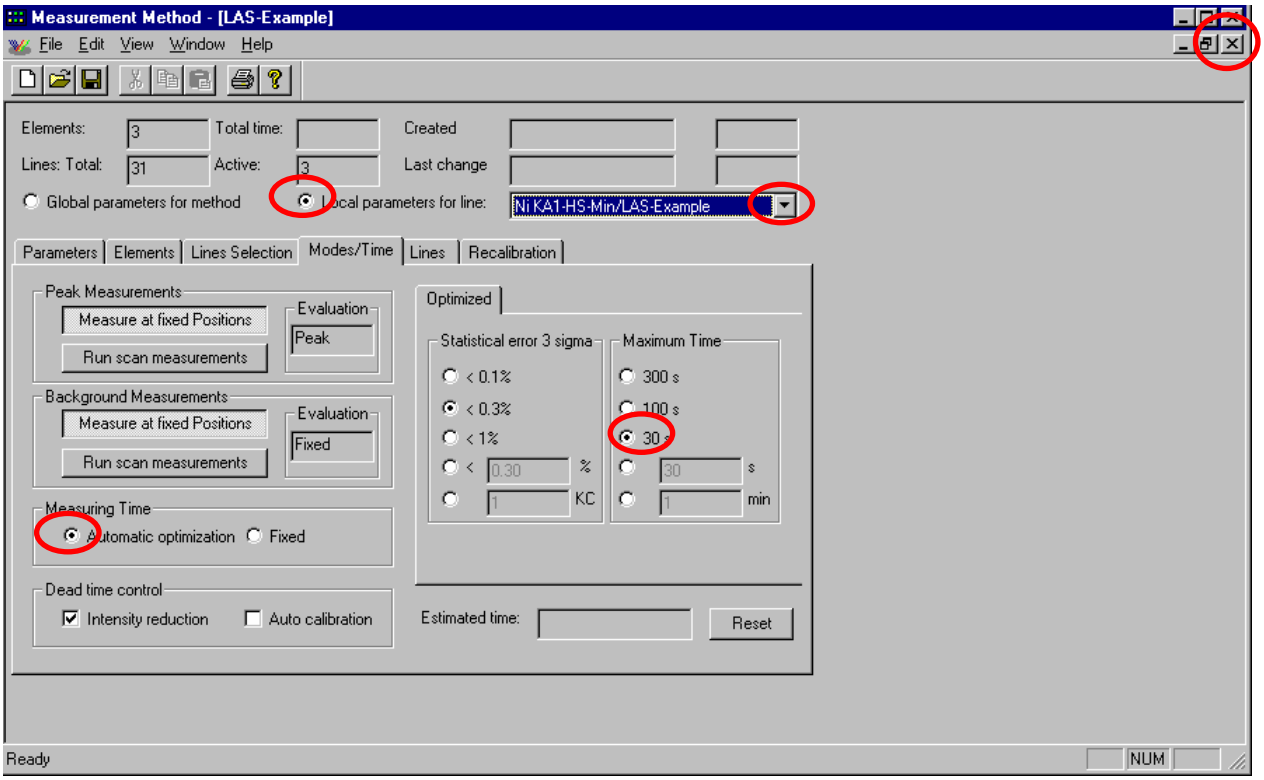
Select the “LOCAL parameters for method” radio button.
All lines will be measured the same.

Select the “Ni KA1 HS MIN” Line from the drop-down menu.

Select YES to select specific parameters.

Select the “Automatic” radio button in “Measuring Time”.
This measurement will be counting to a set statistical error.

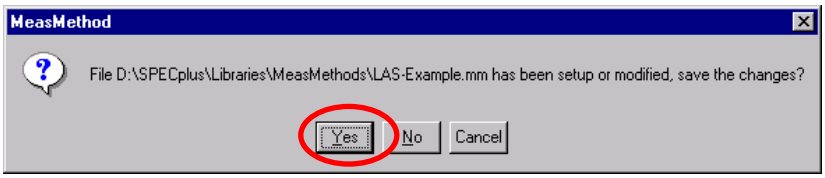
Select “30 s” as the maximum counting time.



Click the **Close** button to exit the Measurement Method program.

Yes

Click the **Yes** button to save the changes that were made to the Measurement Method file “LAS-Example.mm”.

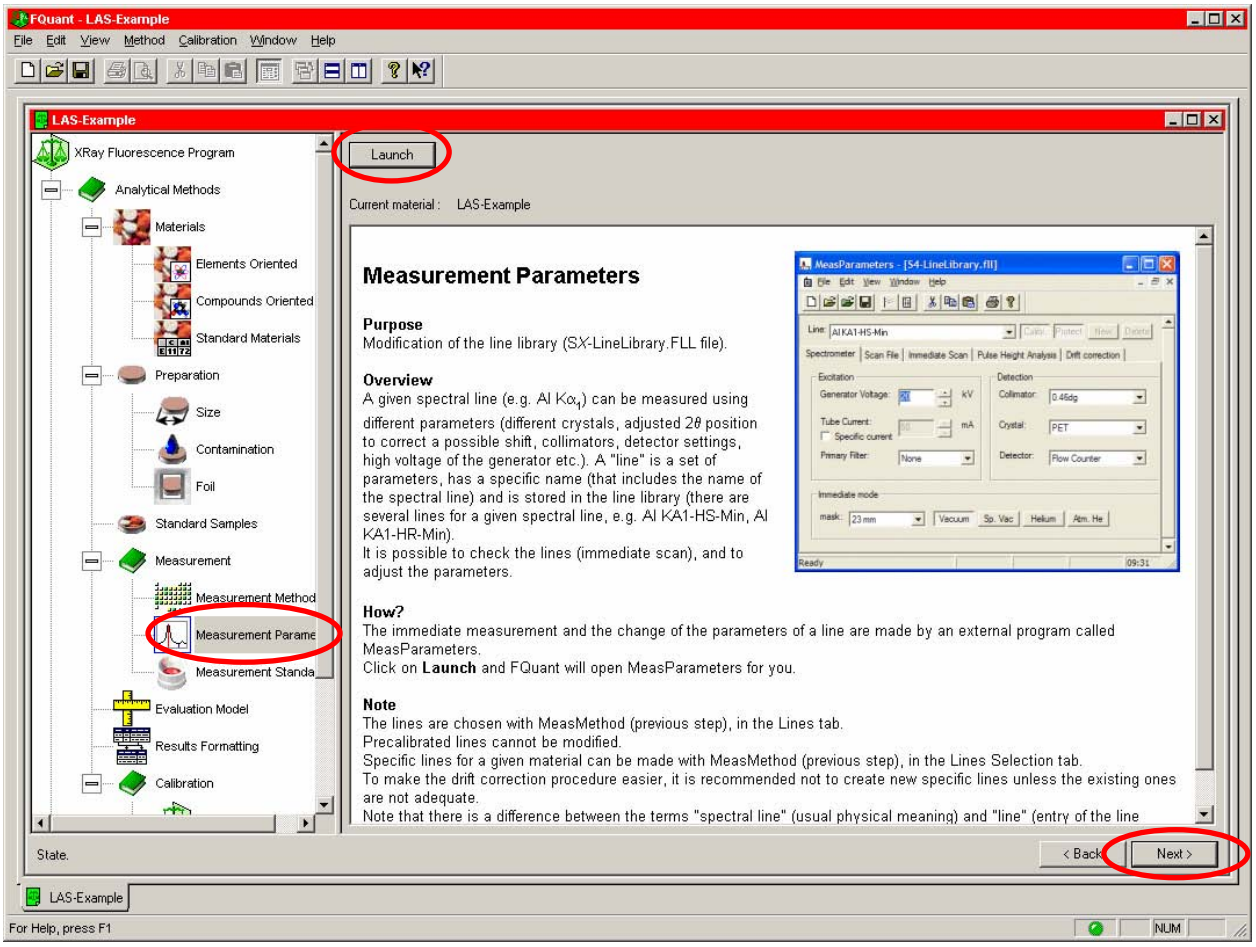


3.6 Optimizing the Measurement Conditions

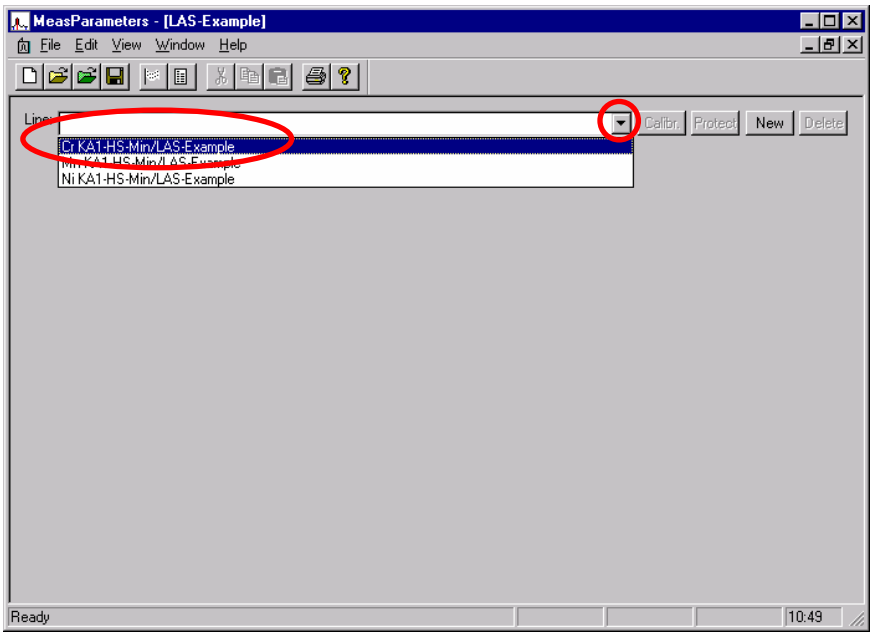
In this step, the measuring conditions used for the lines will be checked and optimized. This will be done by measuring pulse height distributions to optimize the detector discriminator settings, and by measuring 2-theta scans to optimize the peak and background measurement positions.

Click the **Next** button to move to **Measurement Parameters**.

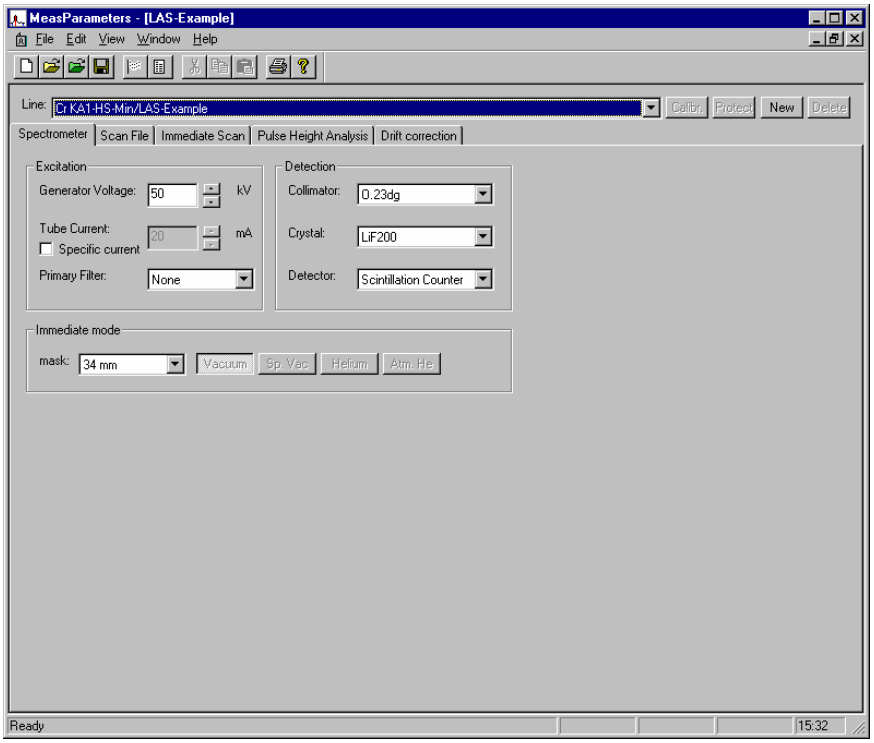
Click the **Launch** button to start the Measurement Parameters program.



Click the drop-down arrow by the “Line” box, and select the “Cr KA1-HS-Min/LAS-Example” line from the list of lines shown.



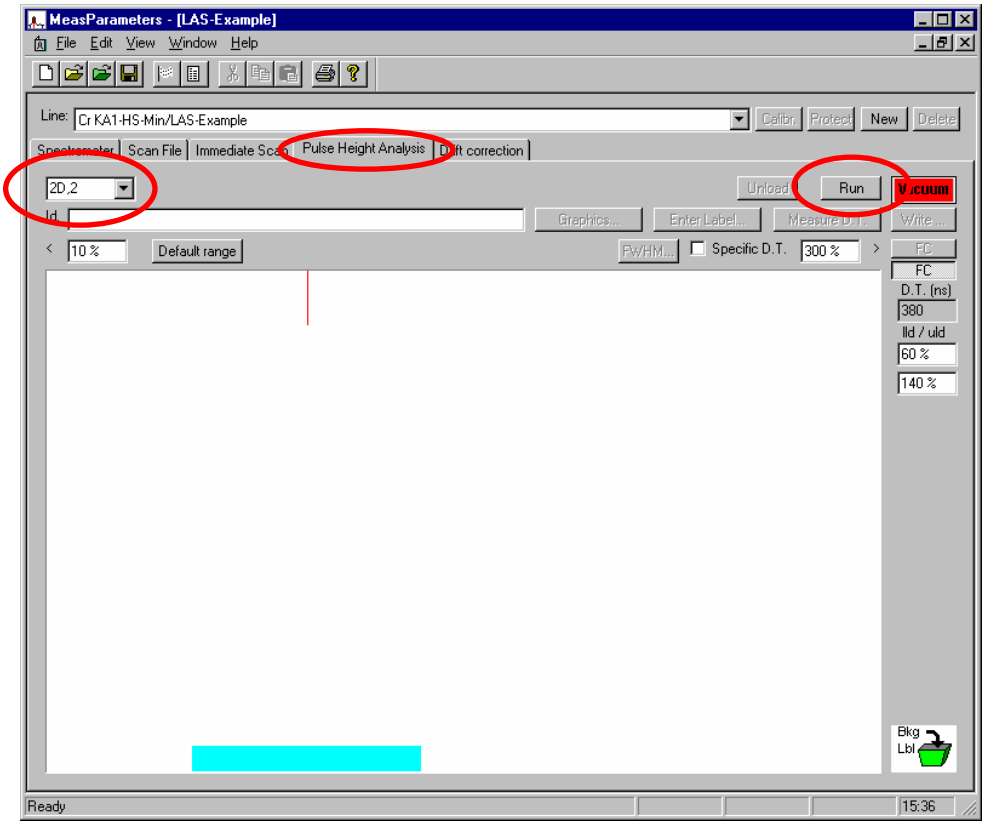
The **Spectrometer** tab should be selected. This shows the instrument operating parameters, except for detector discriminator settings, that will be used to measure this line. Any of the conditions shown could be changed from this tab.



Click on the **Pulse Height Analysis** tab to select it.

Enter the sample position where **SS 404** was placed on the sample loader.

Click the **Run** button to start the measurement.



The sample will now be transported into the spectrometer and measurement of an energy distribution will begin.

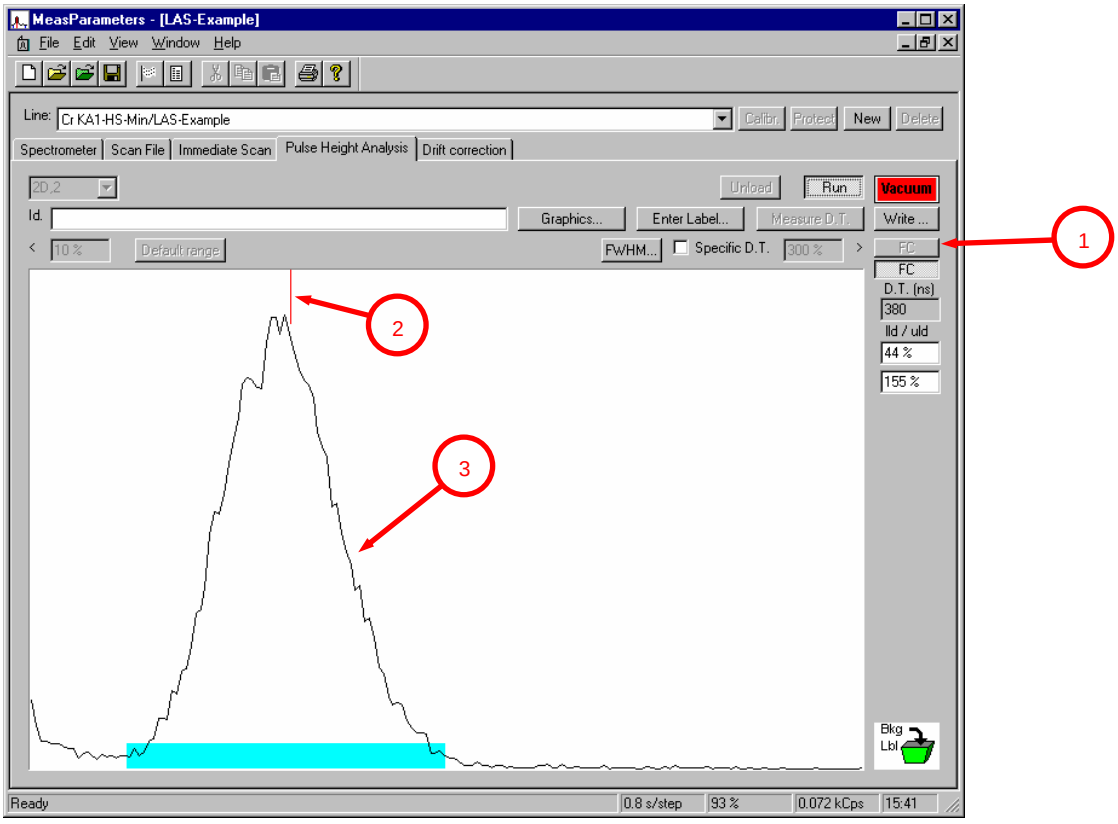
This energy distribution will be for one detector only, as selected by the detector buttons (“FC” or “SC”). These buttons will only be enabled if the corresponding detector is selected for this line.

The data will be plotted in real time as it is collected. The energy distribution should be centered approximately on the red line; if it is not, then the detector high voltages need to be reset.

The blue line shown below the energy distribution represents the detector discriminator settings, or “Lower-Level” (LLD) and “Upper-Level” (ULD). This should encompass all of the energies to be measured.

←→

If the detector discriminator settings (blue line) need to be adjusted, move the mouse over the end of the blue line that needs to be adjusted. The mouse pointer will change to a horizontal double arrow. Click and hold down the left mouse button, then drag the mouse to move the end of the line.



Click on the **Immediate Scan** tab to select it.

Click the **Run** button to start the measurement.

The tall vertical red or green line represents where the peak angle is currently set. Red means a user-set position, and green means a theoretical position.

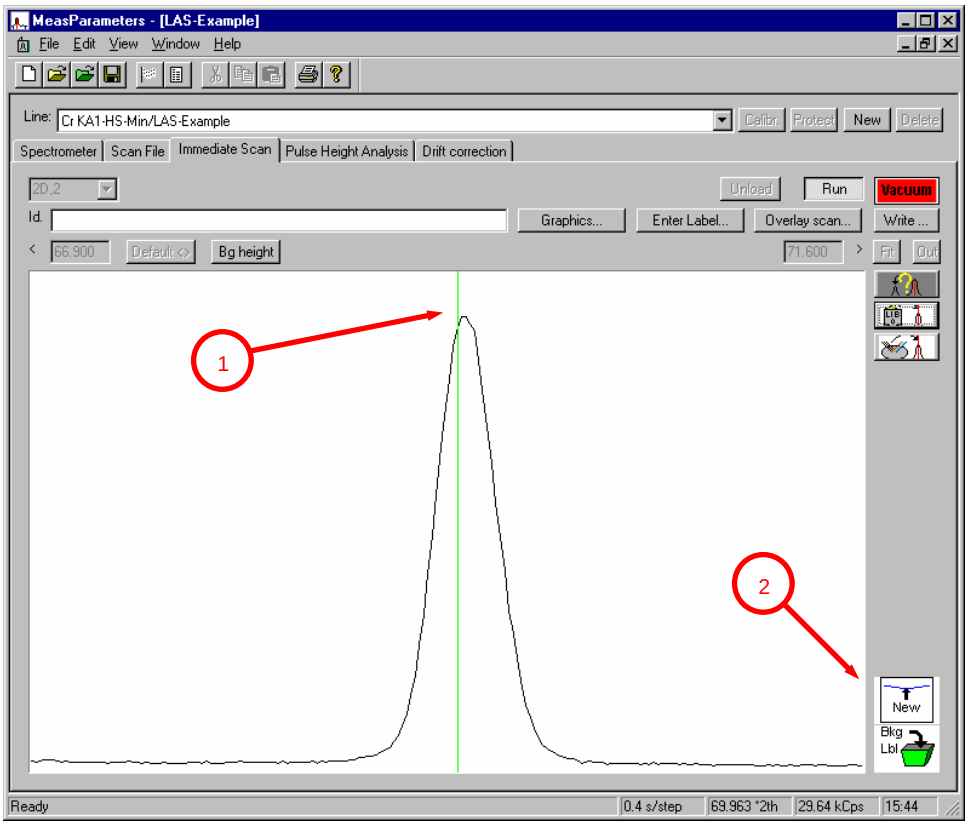
To set the peak angle, move the mouse pointer over the current position until it changes to a horizontal double arrow. Click and hold down the left mouse button, then drag the mouse to move the position of the line.

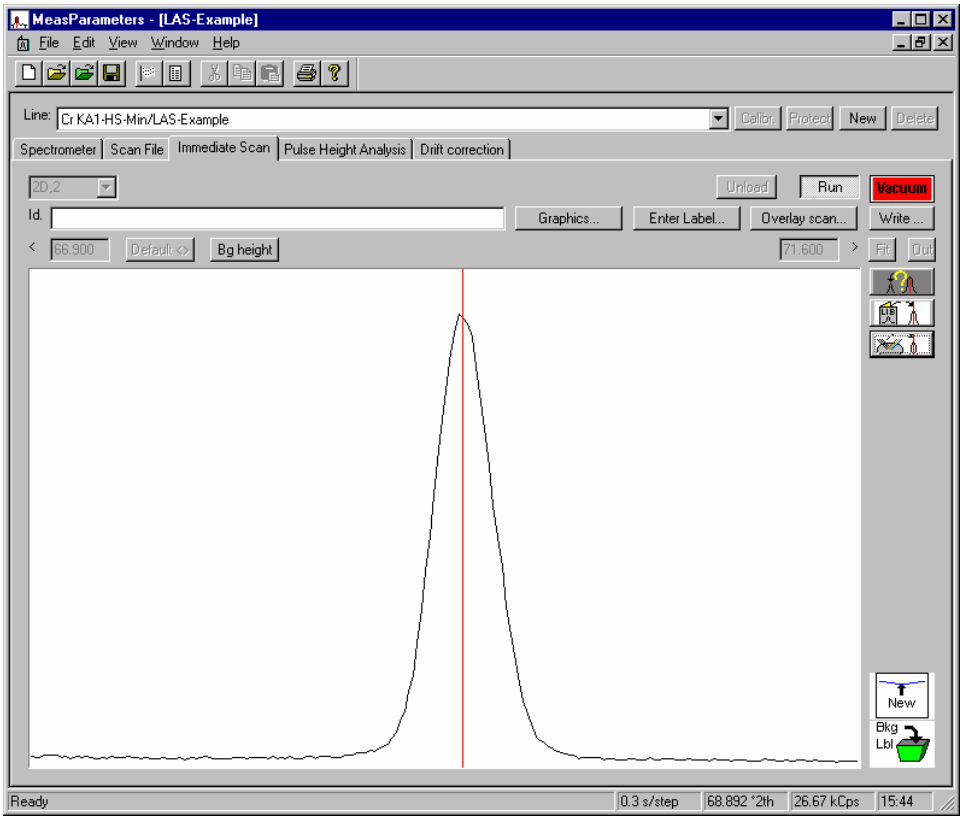
The short vertical blue line(s) represent where the background intensities will be measured.

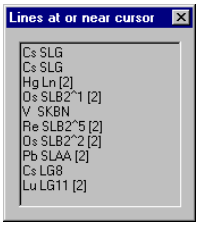
If there is more than one background position set, all but one should be removed. To do this, move the mouse pointer over the line to remove until the mouse pointer changes to a horizontal double arrow. Click and hold down the left mouse button, then drag the line to the Trash Can located in the lower right corner of the window.

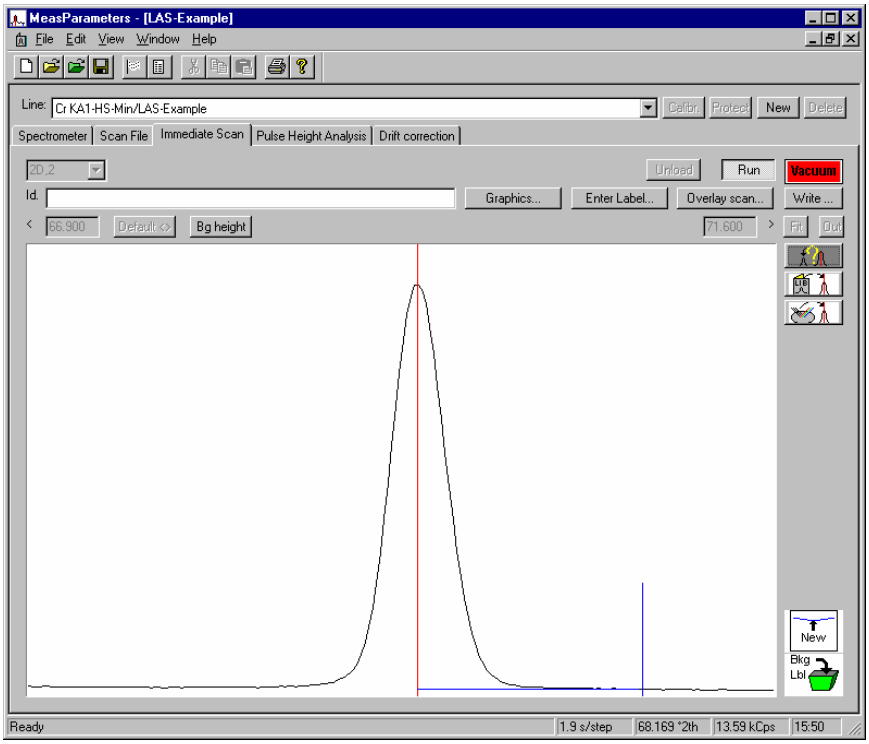
If there are no background positions set, then one should be set. To do this, move the mouse pointer over the New background icon in the lower right corner of the window. Click and hold down the left mouse button, then drag the mouse to position the background as indicated in the diagram below.

To move the background position to a new location, move the mouse pointer over the current position until it changes to a horizontal double arrow. Click and hold down the left mouse button, then drag the mouse to move the position of the line.

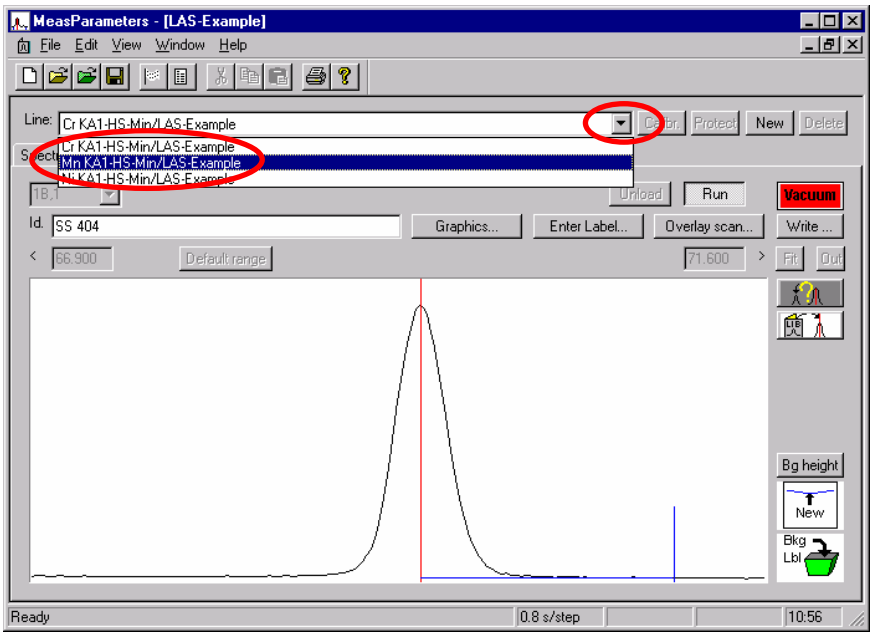

MeasParameters - [LAS-Example]
File Edit View Window Help
Line: Cr KA1-HS-Min/LAS-Example
Calibr. Protect New Delete
Spectrometer Scan File Immediate Scan Pulse Height Analysis Drift correction
2D.2
Unload Run Vacuum
Id.
Graphics... Enter Label... Overlay scan... Write ...
66.900 Default <> Bg height 71.600 Fit Out
1
2
New
Bkg
Lbl
Ready
0.4 s/step
69.963 °2th
29.64 kCps
15:44

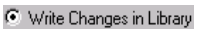

MeasParameters - [LAS-Example]
File Edit View Window Help
Line: Cr KA1-HS-Min/LAS-Example
Calibr. Protect New Delete
Spectrometer Scan File Immediate Scan Pulse Height Analysis Drift correction
2D.2
Unload Run Vacuum
Id.
Graphics... Enter Label... Overlay scan... Write ...
66.900 Default <> Bg height 71.600 Fit Out
New
Bkg
Lbl
Ready
0.3 s/step
68.892 °2th
26.67 kCps
15:44


Lines at or near cursor
Cs SLG
Cs SLG
Hg Ln [2]
Os SLB2^1 [2]
V SKBN
Re SLB2^5 [2]
Os SLB2^2 [2]
Pb SLAA [2]
Cs LG8
Lu LG11 [2]

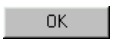

MeasParameters - [LAS-Example]
File Edit View Window Help
Line: Cr KA1-HS-Min/LAS-Example
Calibr. Protect New Delete
Spectrometer Scan File Immediate Scan Pulse Height Analysis Drift correction
2D.2
Unload Run Vacuum
Id.
Graphics... Enter Label... Overlay scan... Write ...
66.900 Default <> Bg height 71.600 Fit Out
New
Bkg
Lbl
Ready 1.9 s/step 68.169 °2th 13.59 kCps 15:50

Click the drop-down arrow by the “Line” box, and select the “Mn KA1-HS-Min/LAS-Example” line from the list of lines shown.

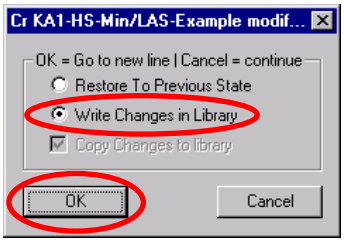




Click the “Write Changes in Library” radio button to select it.



Click the **OK** button to save the changes that were made.



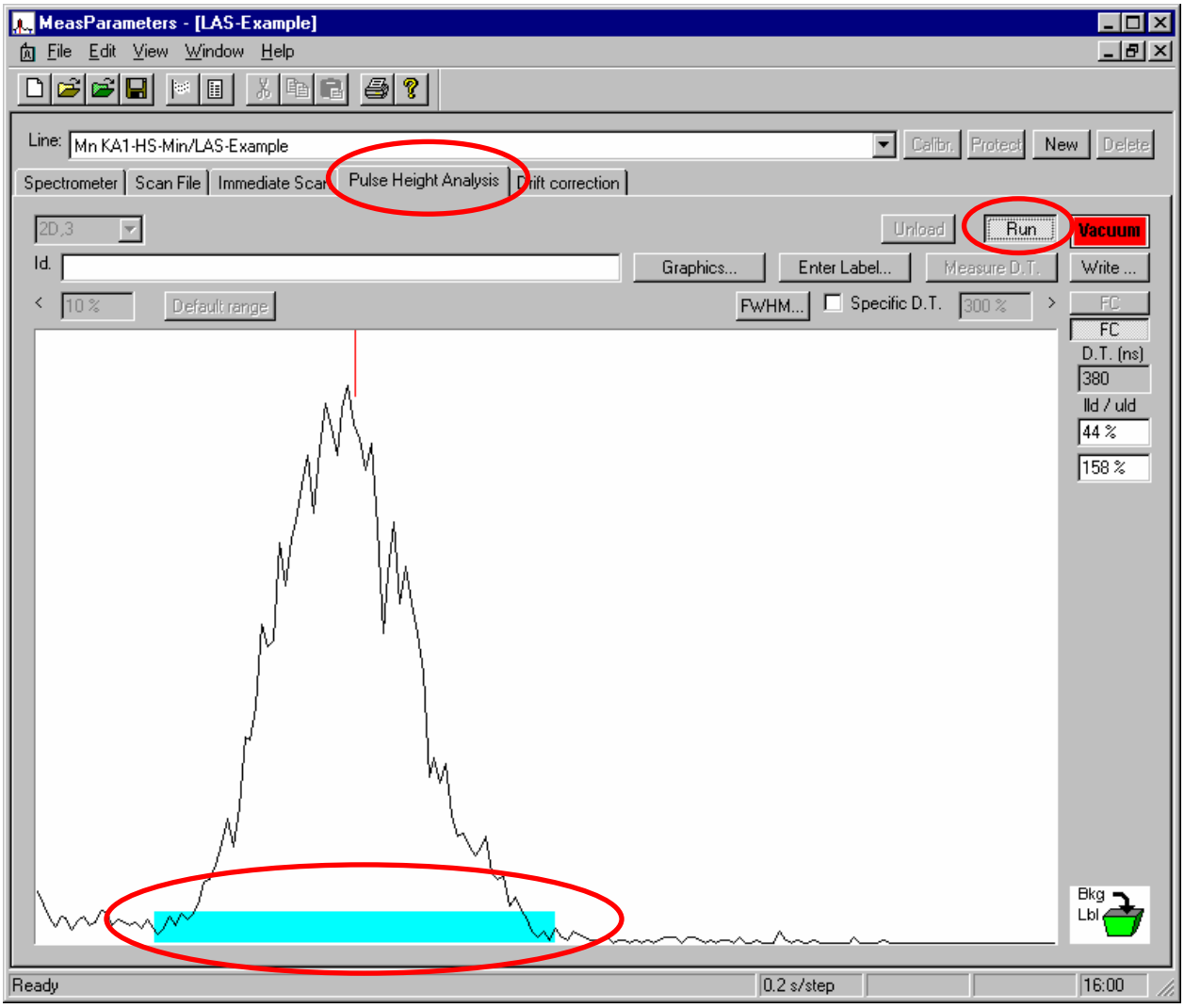
Pulse Height Analysis

Click on the **Pulse Height Analysis** tab to select it.

Run

The **Run** button should still be depressed, and the measurement should start; if not click the **Run** button.

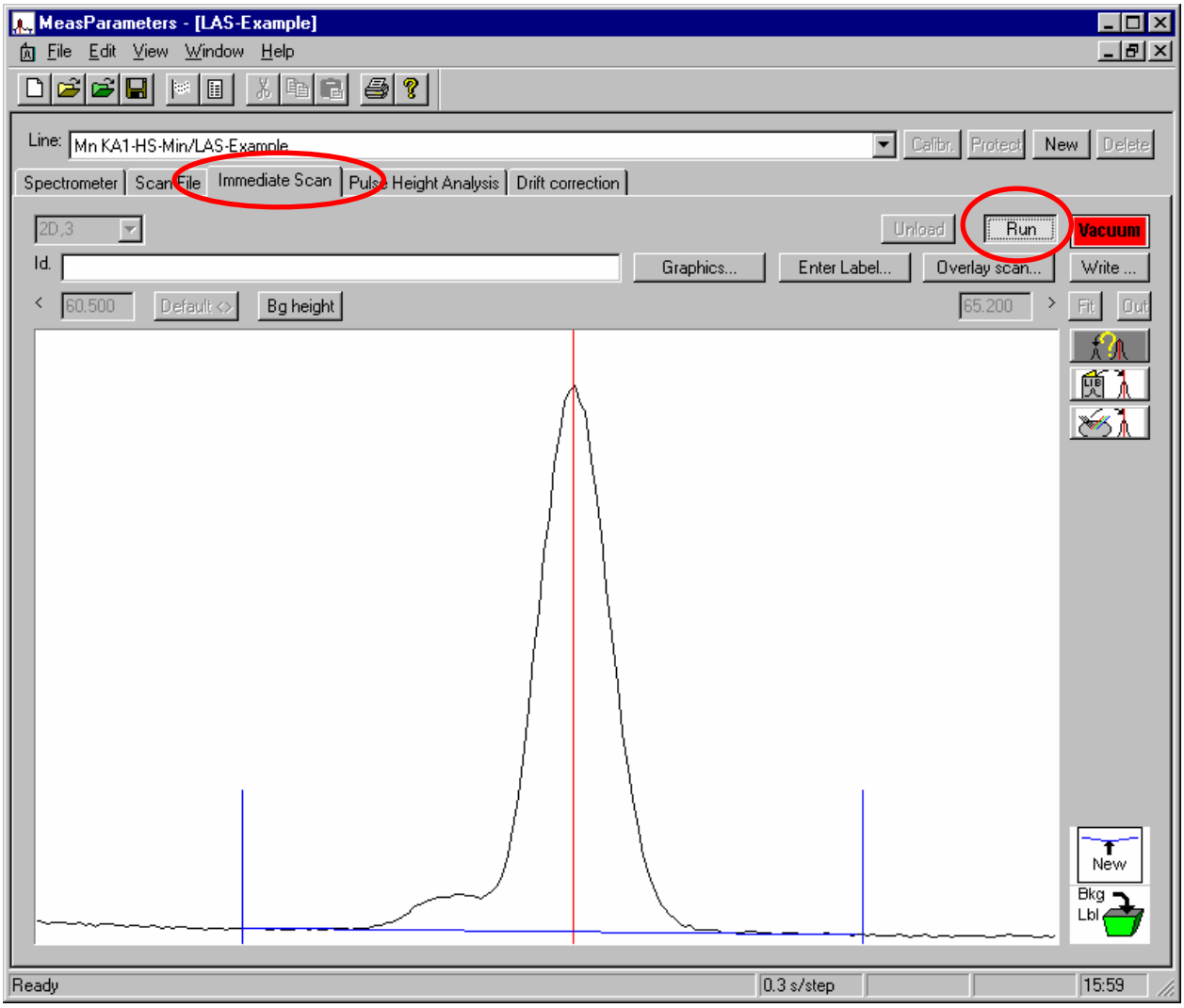
Set the discriminator settings to bracket the energy distribution for Mn.



Click on the **Immediate Scan** tab to select it.

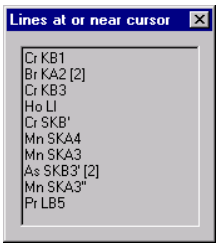
The **Run** button should still be depressed, and the measurement should start; if not, click the **Run** button.

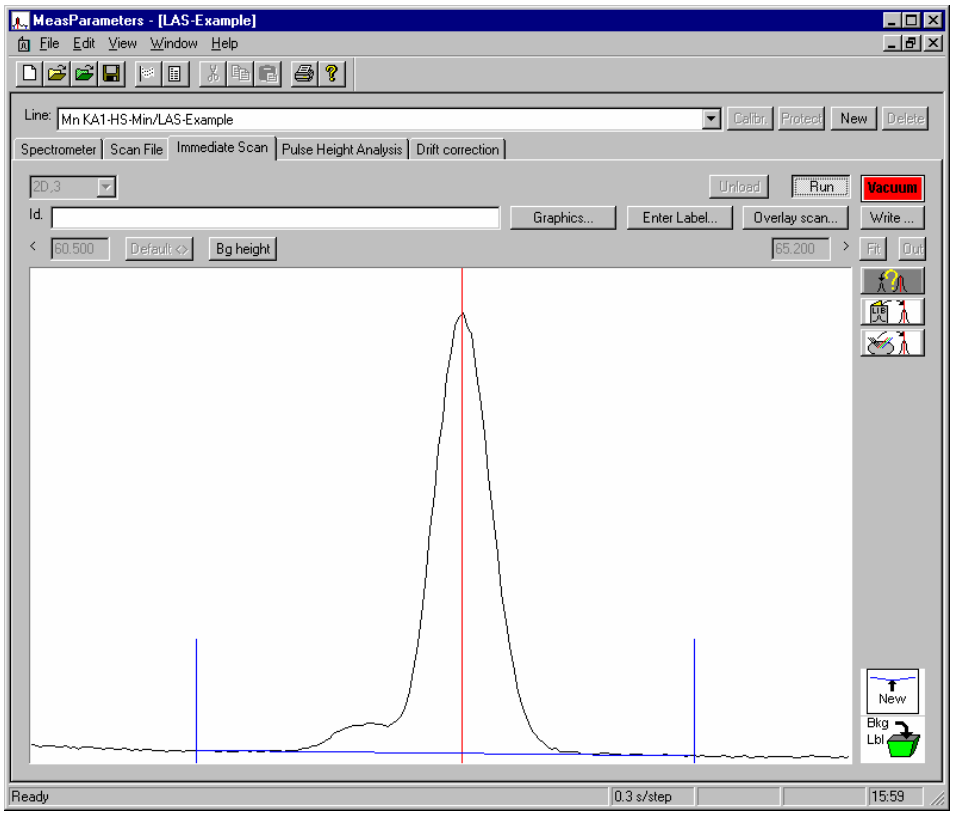
Set the peak angle and a single background position, as shown below.



To identify the peak seen to the low angle side of the Mn-Line, click on the **Identify Lines** icon, then move the mouse pointer over the peak.

A window will open, showing the lines around the mouse pointer's position.

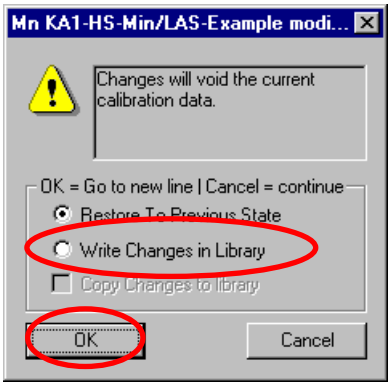




Click the drop-down arrow by the "Line" box, and select the "Ni KA1-HS-Min/LAS-Example" line from the list of lines shown.

Click the "Write Changes in Library" radio button to select it.

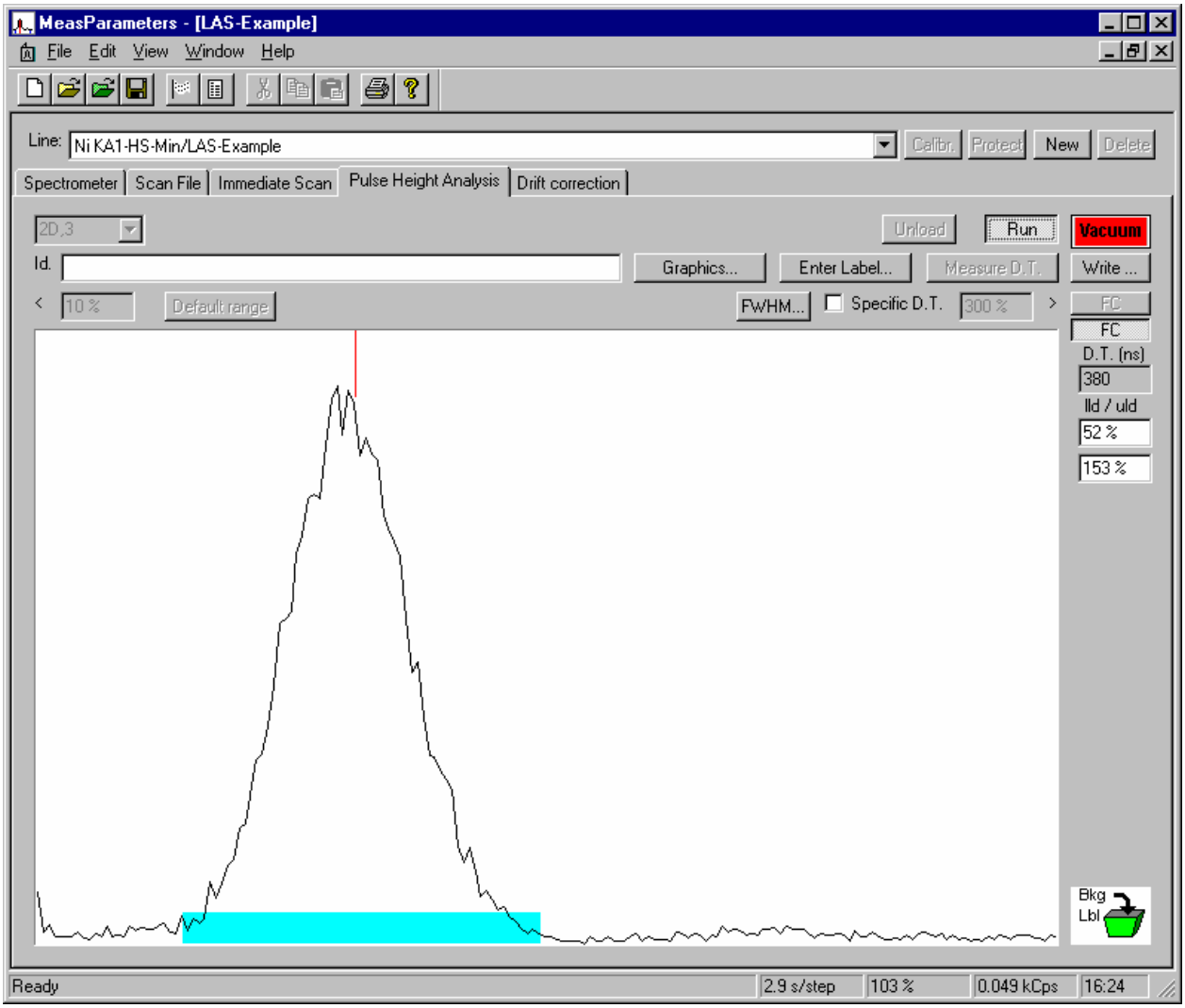
Click the **OK** button to save the changes that were made.



Click on the **Pulse Height Analysis** tab to select it.

The **Run** button should still be depressed, and the measurement should start; if not, click the **Run** button.

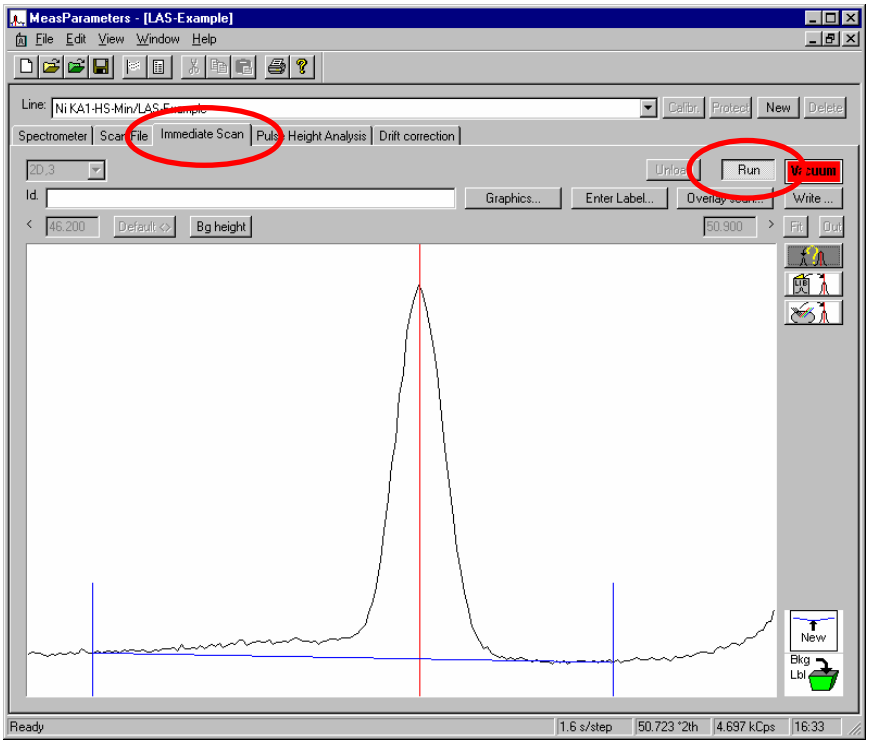
Set the discriminator settings to bracket the energy distribution for Ni.

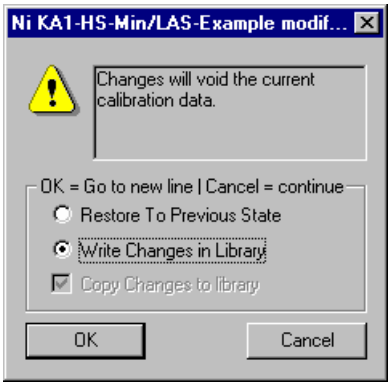


Click on the **Immediate Scan** tab to select it.

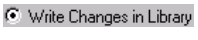
The **Run** button should still be depressed, and the measurement should start; if not, click the **Run** button.

Set the peak angle and a single background position, as shown below.





Select the “Cr KA1-HS-Min/LAS-Example” line.



Click the “Write Changes in Library” radio button to select it.

Click the **OK** button to save the changes that were made.

Line	Low Sample	High Sample
Mn KA1-HS-Min/LAS-Example	SS 402	SS 403
Cr KA1-HS-Min/LAS-Example	SS 402	SS 403
Ni KA1-HS-Min/LAS-Example	SS 402	SS 403

Table 1 - Drift correction samples for LAS-Example Measurement Method

Select the **Drift correction** tab.

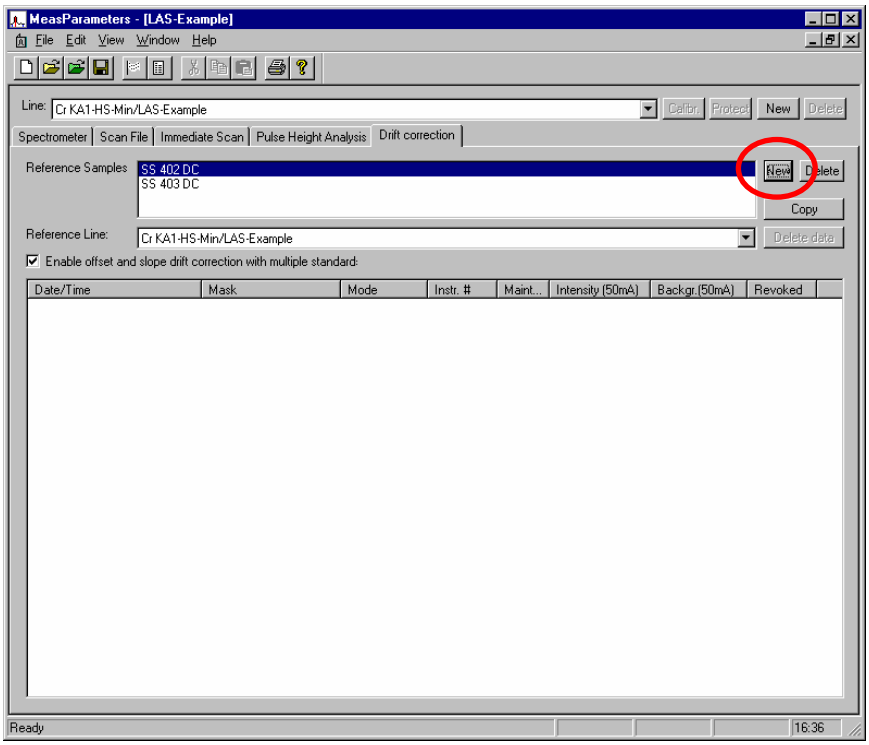
Check that there are two reference samples listed.

In some cases, the “drift correction samples” may be deleted from a line. This occurs if changes are made in the conditions used to measure the line, or significant changes are made in the discriminator settings.

If the Cr line does not have both “drift correction samples” listed, then:

New

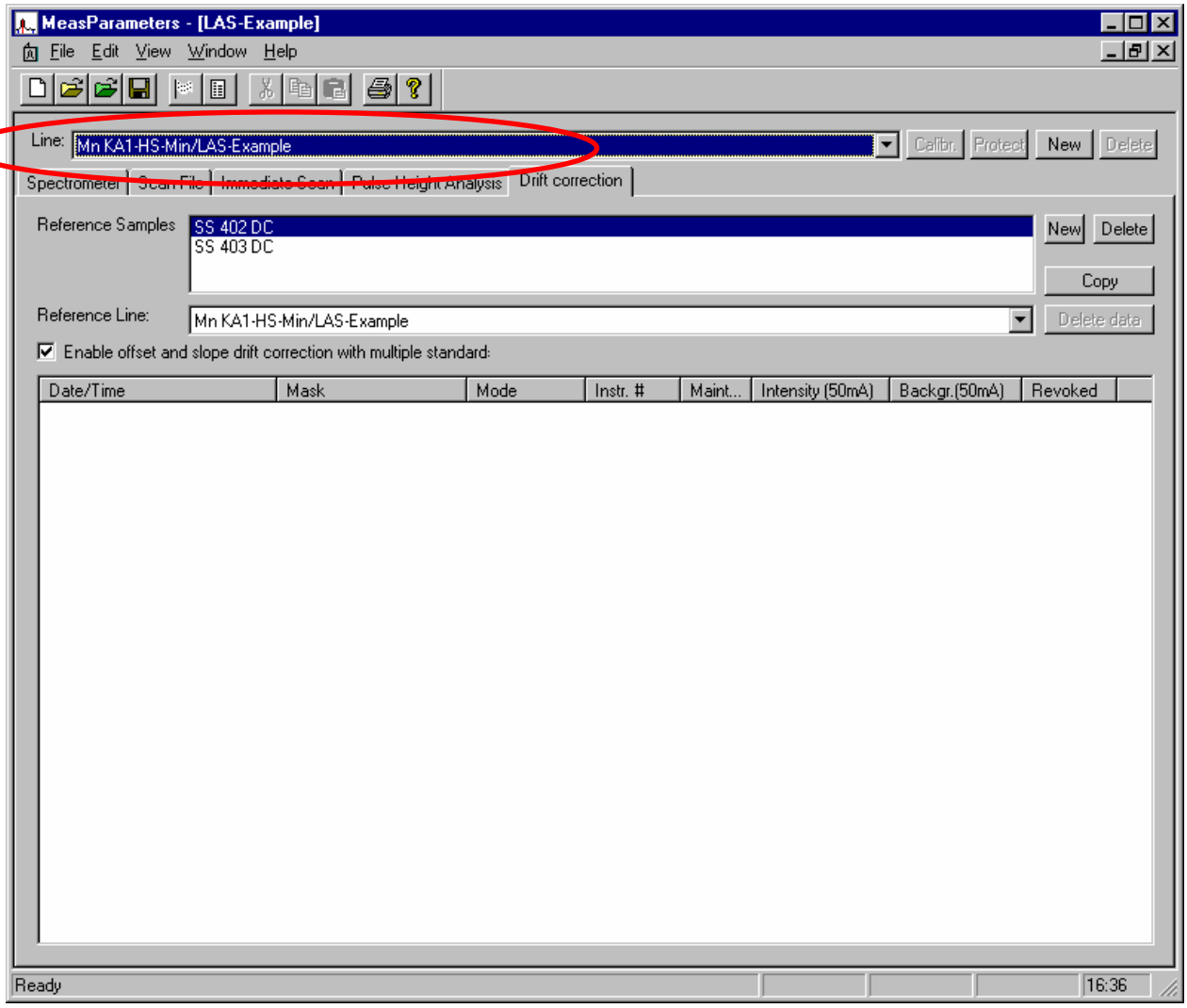
Click the **New** button.



Enter the name of the missing sample.

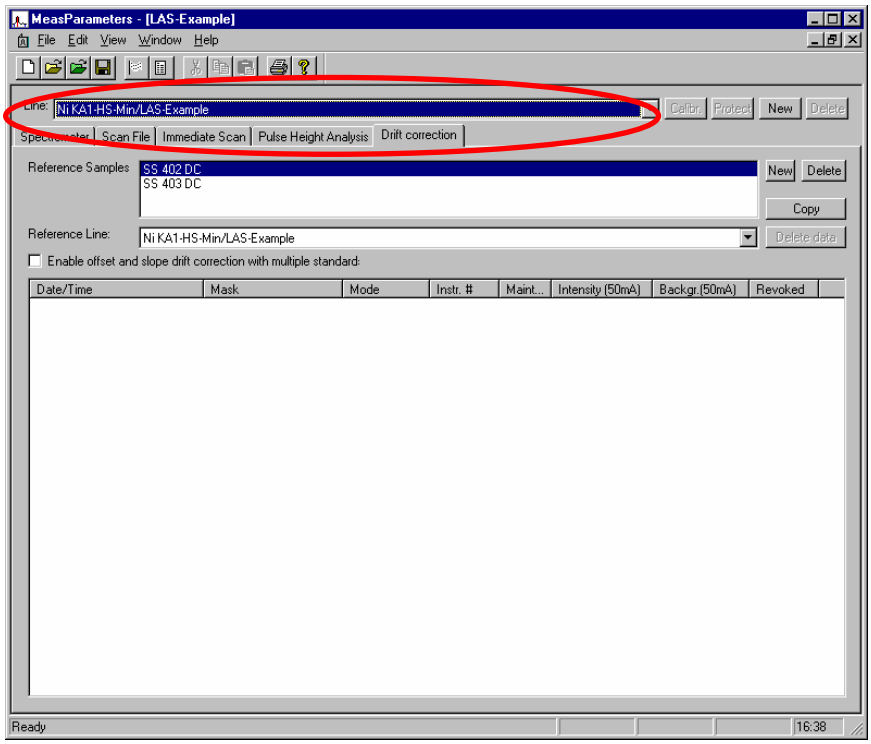
Click the **OK** button.

Switch to the “Mn KA1-HS-Min/LAS-Example” line and check the “Recalibration Samples” for it. Add back any missing ones.



Switch to the “Ni KA1-HS-Min/LAS-Example” line and check the “Recalibration Samples” for it. Add back any missing ones.

Click the **Close** button to exit the MeasParameters program.



OK

If measurements are still running, a message indicating that these will be interrupted will be given. Click the **OK** button to stop the measurements and unload the sample.

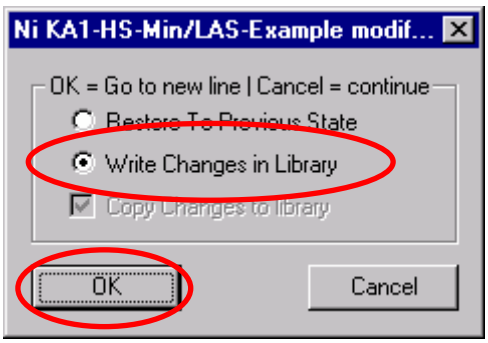
If any changes made have not been saved, the following window will be shown:

Write Changes in Library

Click the “Write Changes in Library” radio button to select it.

OK

Click the **OK** button to save the changes that were made.

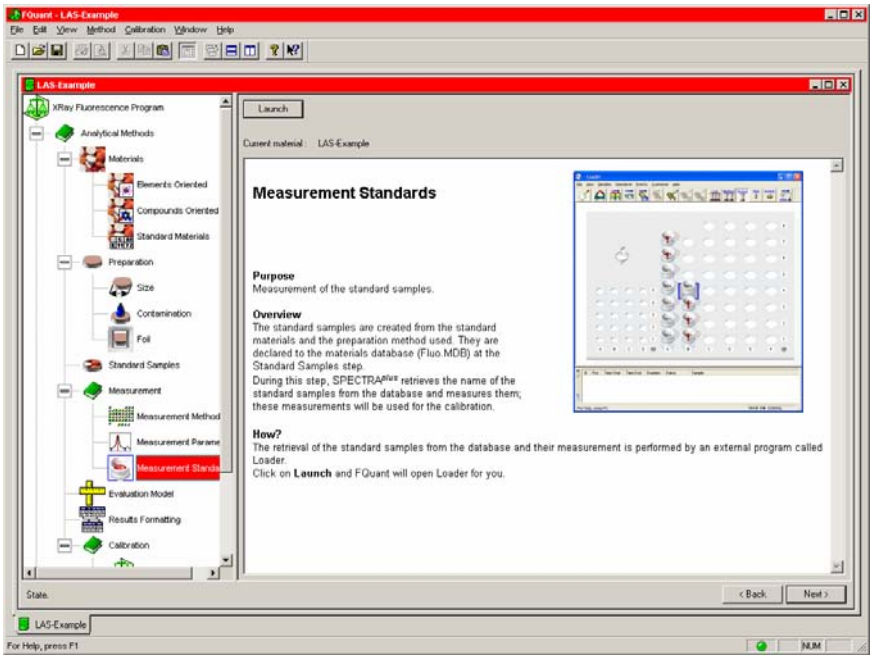


3.7 Measuring the Calibration Standards

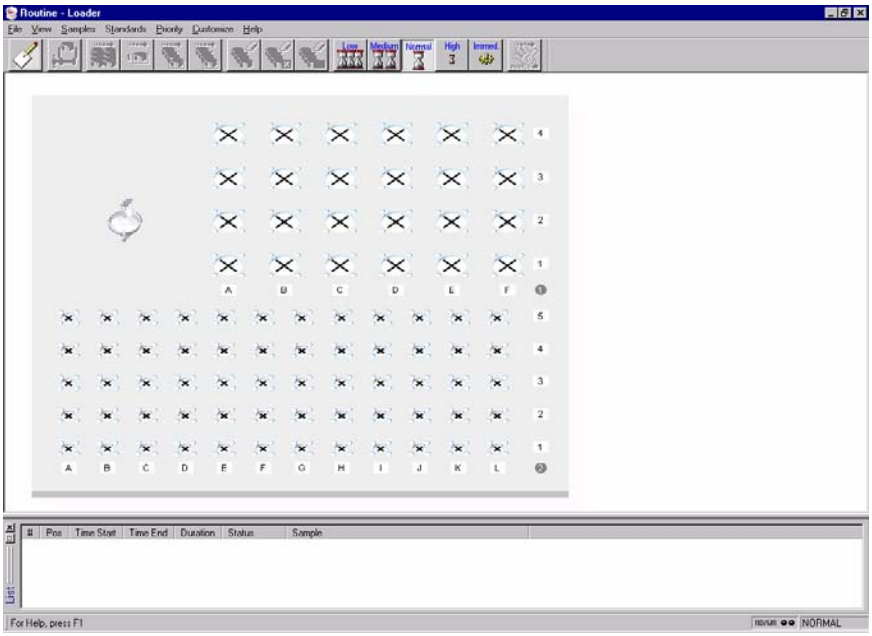
In this step, intensities will be measured on the five low-alloy steel calibration standards. Although the “Recalibration”, or “Drift Correction”, samples are the same as the “Calibration Standards,” the names used for them are different so an additional measurement needs to be made for them. The intensities that are measured will be stored in “Step Scan Data” files (“*.ssd”) in the folder “\SPECplus\Libraries\Materials\LAS-Example\Solid”.

Click the **Next** button to move to **Measurement Standards**.

Click the **Launch** button to start the Loader program.



When the Loader program first starts, all positions are blocked and have “X’s” displayed in them.

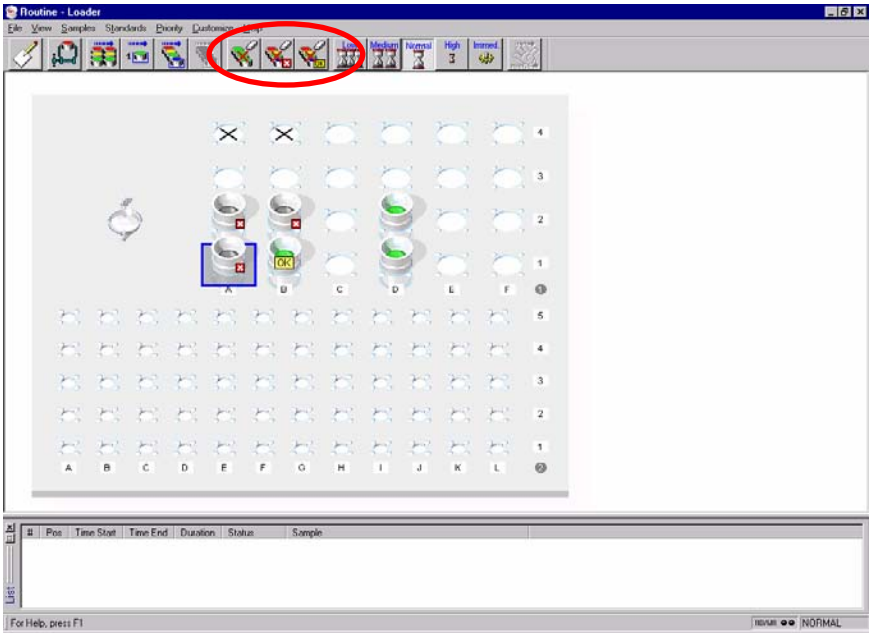


After a brief time, the “X’s” will disappear, and any cups that were measured in the past will be displayed on the Loader. Clear any cups using the following icons:

“Erase OK” cups.

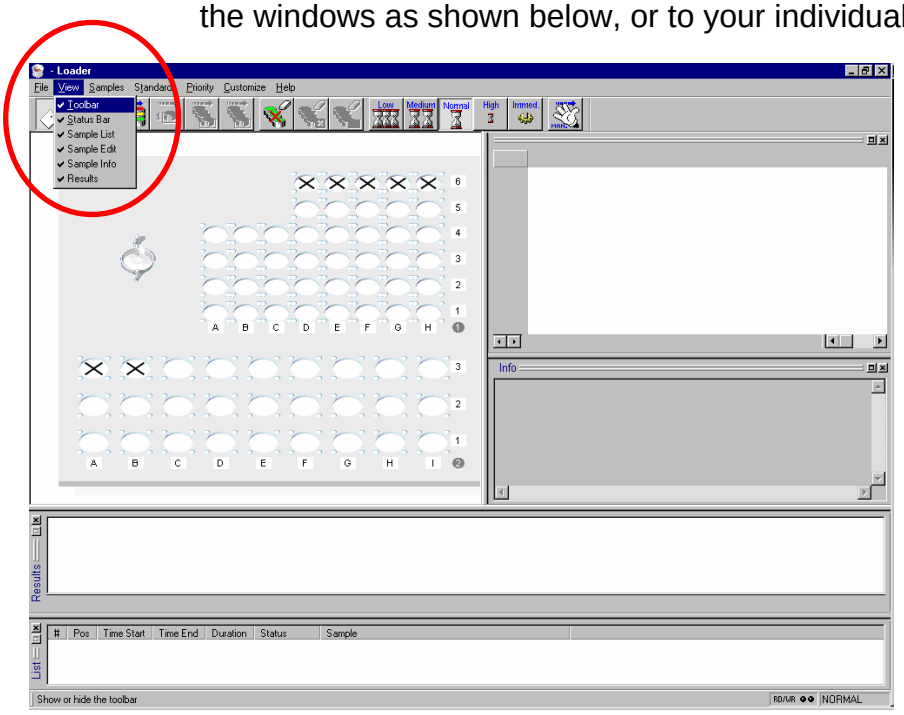
“Erase Error” cups.

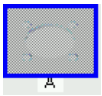
“Erase New” cups.



View

Click **View** on the menu. Make sure all options are checked, and arrange the windows as shown below, or to your individual tastes.

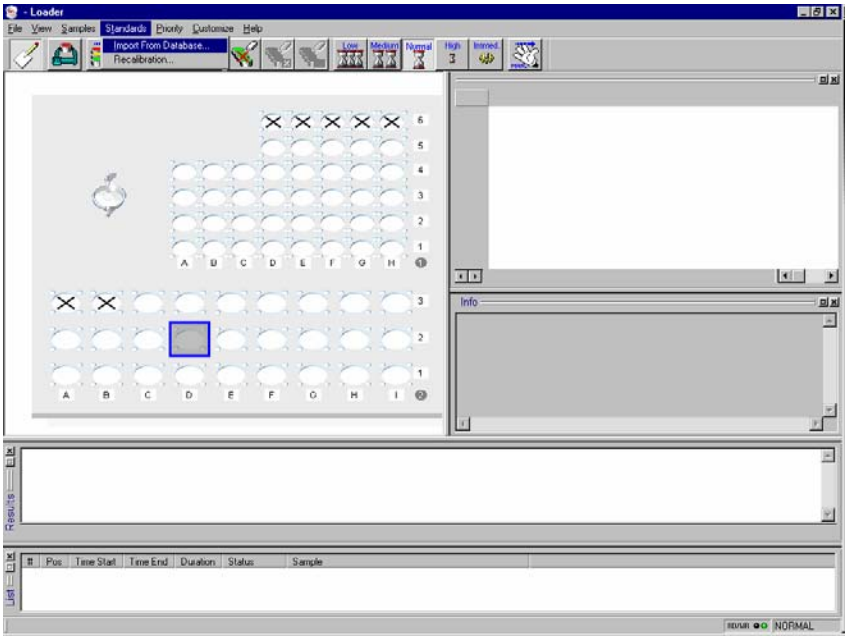




Click the position on the Loader where the first calibration standard is loaded.

Standards
Import From Database...

Select **Standards→Import From Database** from the menu.



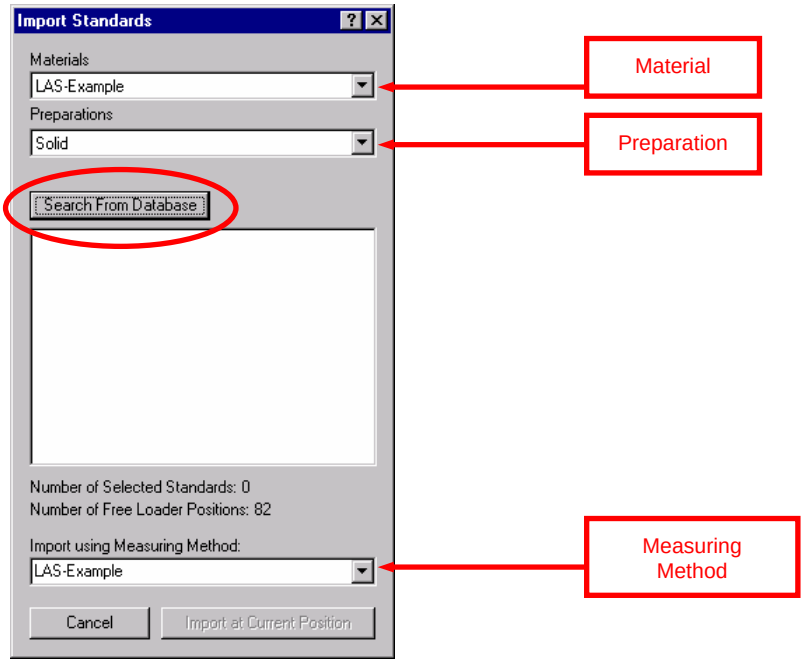
The “Material” should be set to “LAS-Example”. If not, use the drop-down control to change it.

The “Preparation” should be set to “Solid”. If not, use the drop-down control to change it.

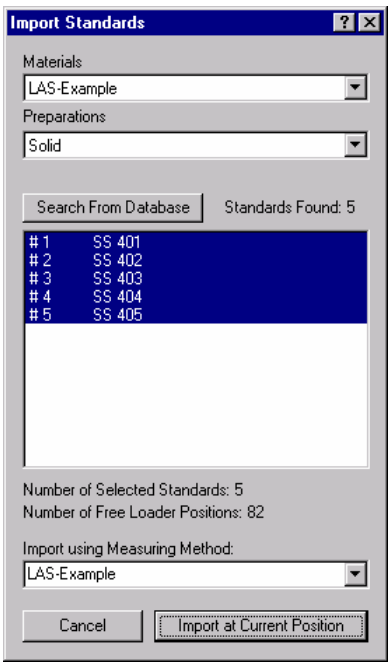
The “Import Using Measuring Method” should be set to “LAS-Example”. If not, use the drop-down control to change it.

Search From Database

Click the **Search From Database** button.



Make sure the standards are loaded in the order shown, and then click the **Import at Current Position** button.

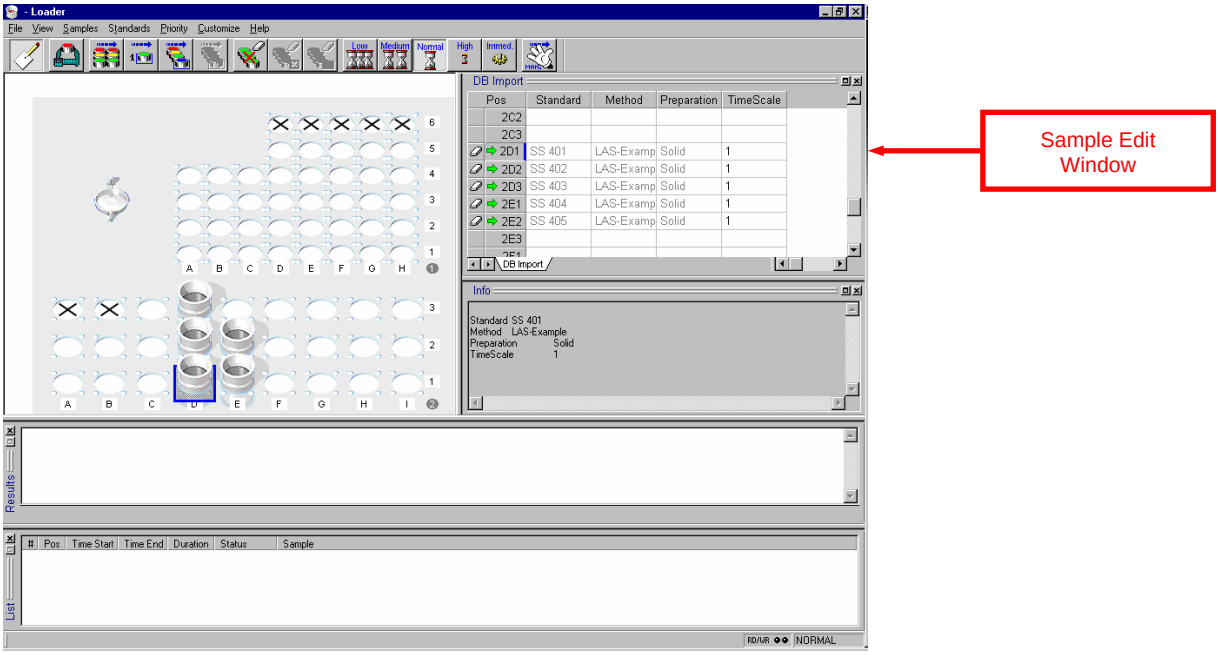


One sample holder per calibration standard should now be displayed on the loader.

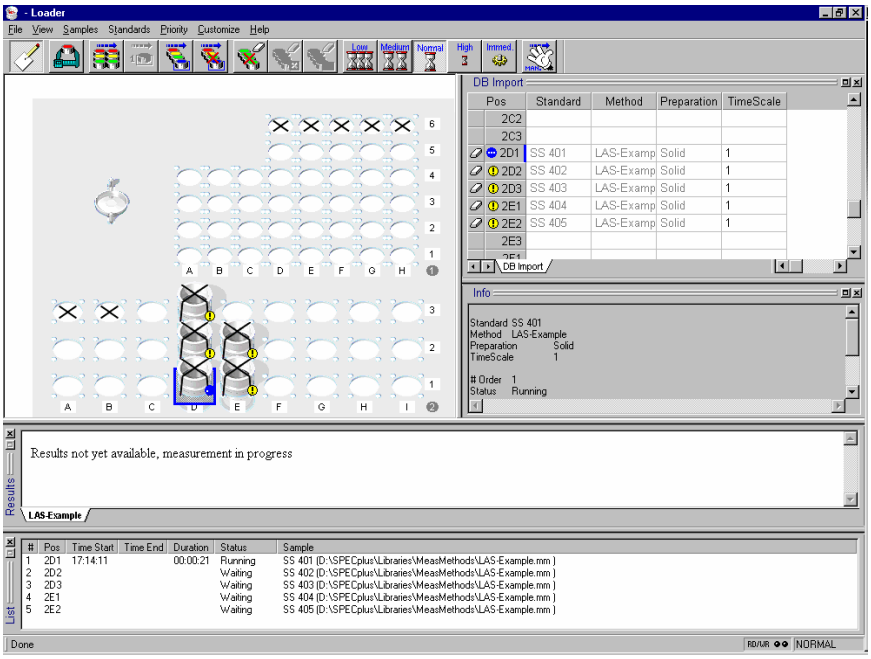
The measurement sequence is displayed in the Sample Edit window. Make sure the samples are loaded in this order; if not, reload them.

You may notice that the Type of sample is set to “Bead” instead of “Bulk”. This does not matter, so do not be too concerned about this.

Click the **Send all Samples** icon to start the measurements.



All the sample holders should have “lock-down bars” displayed on them. These indicate that the samples are committed to be measured, but have not actually been queued into the measurement sequence yet.



After a brief time, the samples will be queued into the measurement sequence. This is indicated by icons being shown on the cups and in the Sample Edit window, and by the samples being listed in the Sample List window.

The following icons can be displayed during the measurements:

The sample is queued for measurement, but the measurement has not started yet.

A sample cup could not be found in this position on the Loader.

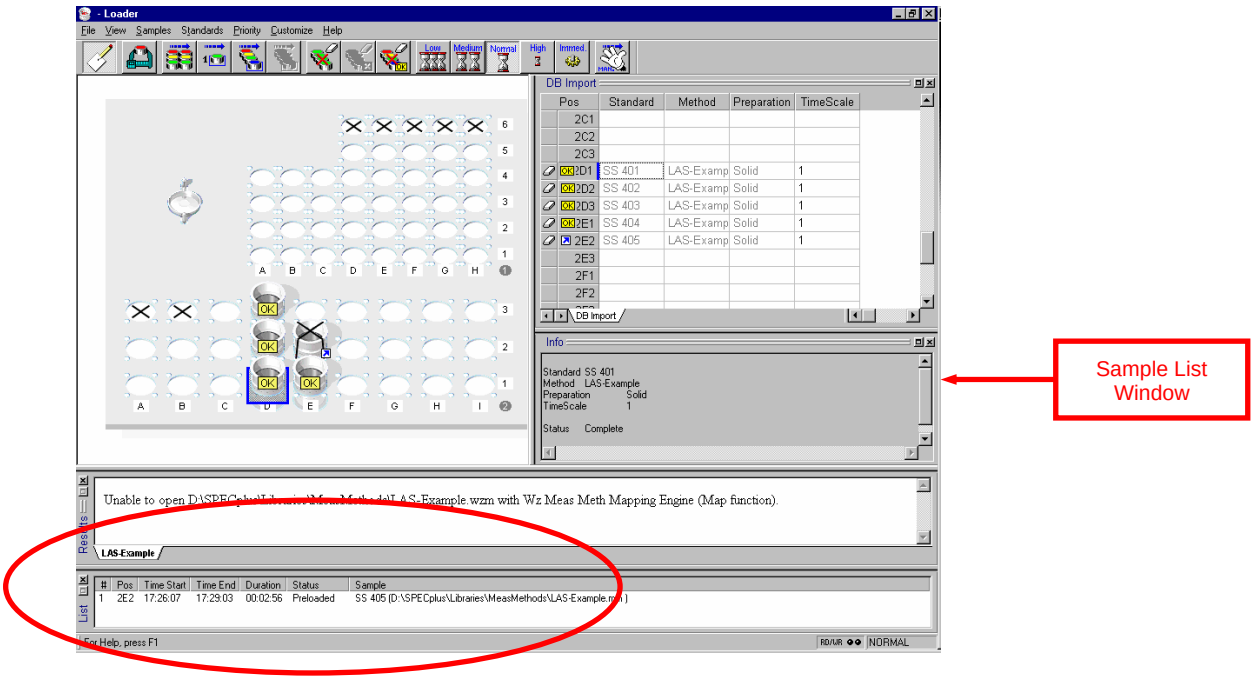
The sample is being loaded into the spectrometer, or is in the pre-load chamber of the spectrometer.

The sample is being measured.

The sample has finished being measured, and the measurement was successful.

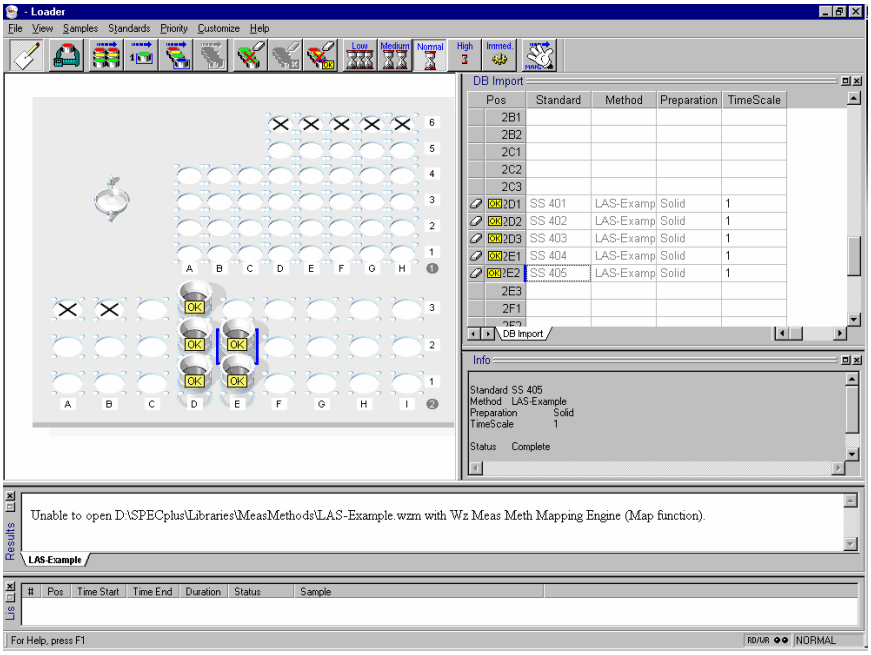
There was an error during the measurement of this sample, and the measurement did not complete.

After the first sample has finished being measured, a Duration and Time End will be displayed for the remaining samples.



All of the sample holders should have an “OK” icon displayed on them when all the measurements are finished.

Click the **Close** button to exit the Loader program.

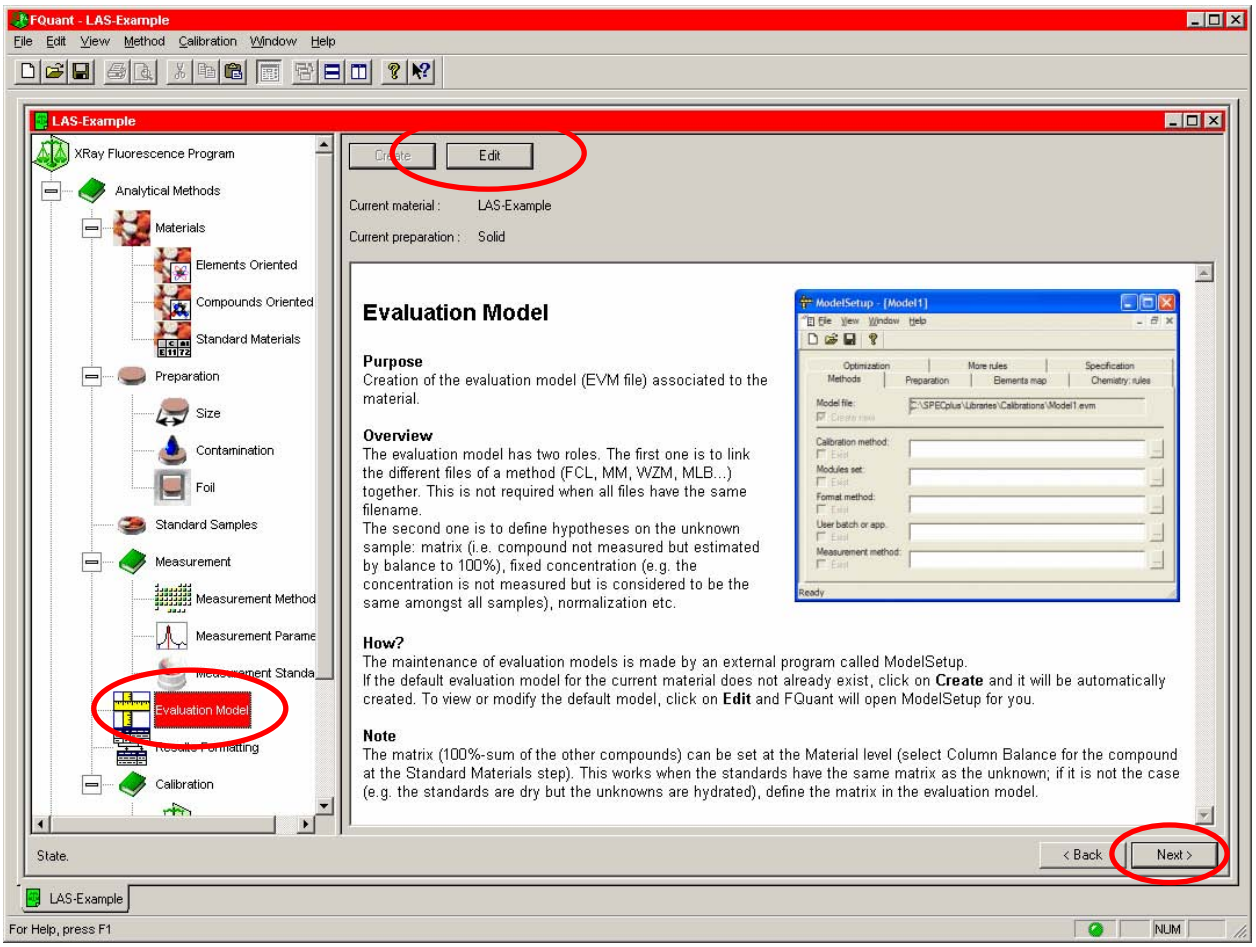


3.8 Creating an Evaluation Model

Next > Click the **Next** button to move to **Evaluation Model**.

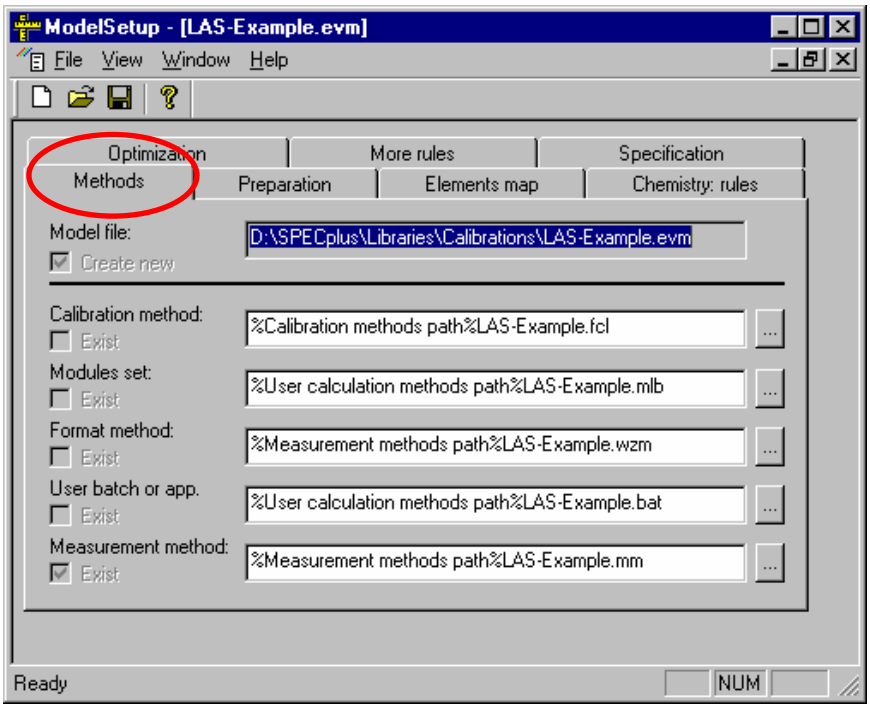
CREATE Click the **Create** button to create a default model.

EDIT Click on the **Edit** button to start the Model Setup program and edit the model.

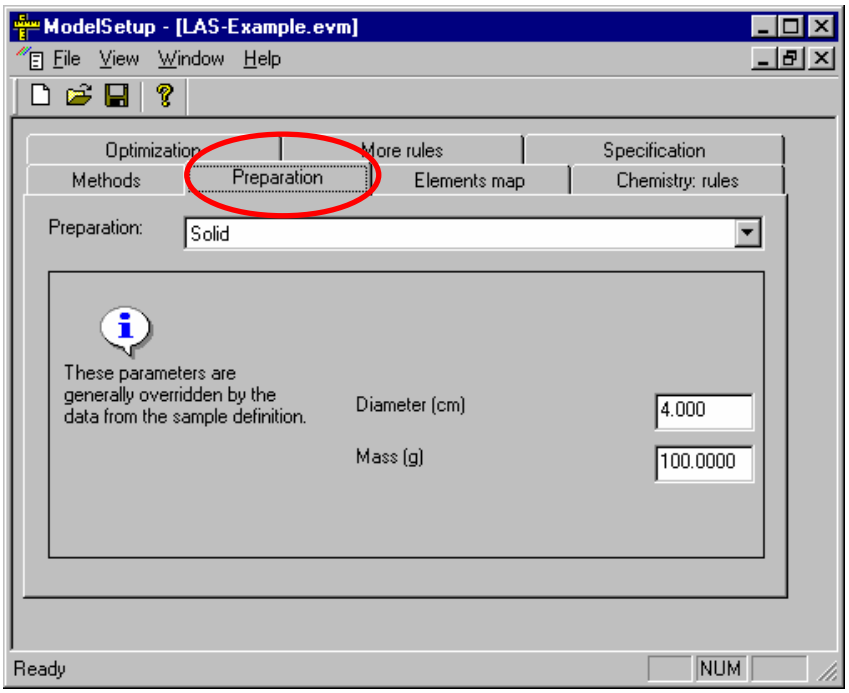


Click the **Methods** tab to select it. The names for each part of the total analytical method can be specified on this tab.

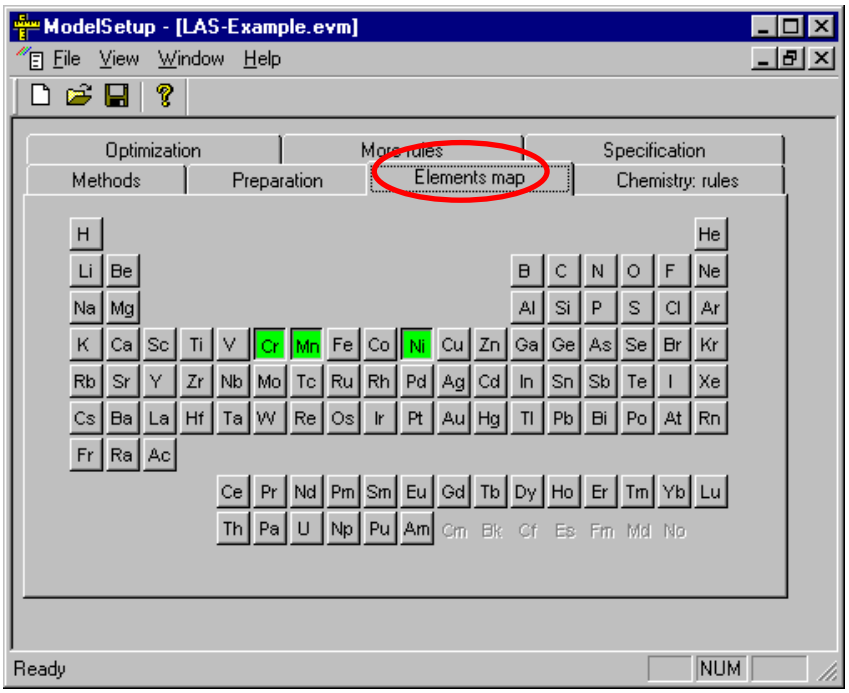
Calibration method:	File where calibration coefficients are stored, which is developed in Section 3.10.
Modules set:	File where extended calculations are stored. This is not described in this guide, but can be found in the SPECTRA^{plus} Reference Manual (M84-Exx025).
Format method:	File where output format for results is stored. This is developed in Section 3.9.
User calculations:	Program or batch file to be automatically started after SPECTRA^{plus} has calculated and reported the results for each sample. These are typically used for transferring results to a user's database, or for making specialized printouts.
Measurement method:	File describing which X-ray lines need to be measured by the XRF instrument, and how to measure them. This was described in Section 3.5.

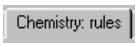


Click the **Preparation** tab to select it.
The specimen preparation used can be fixed on this tab so that it does not have to be given when measuring unknown samples.



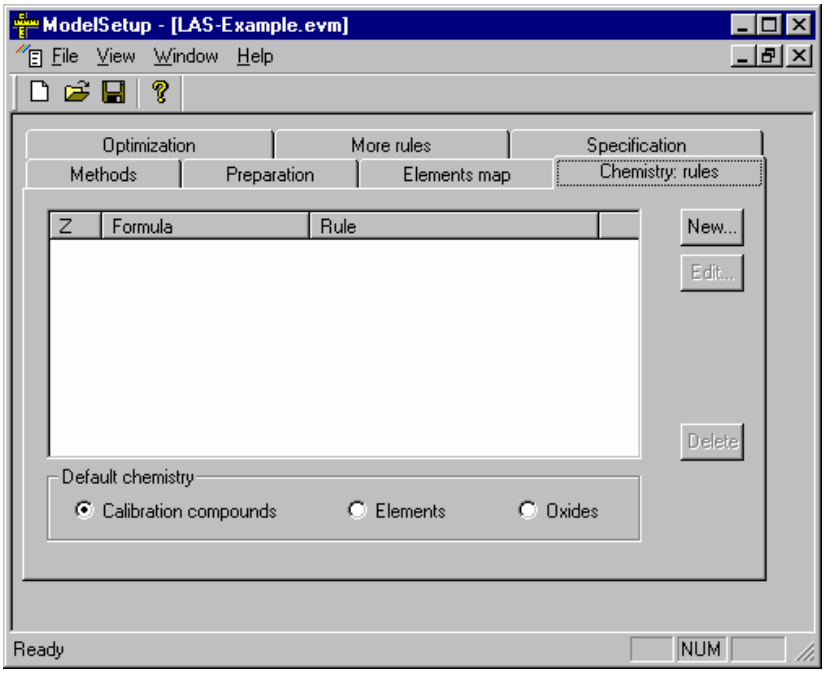
Click the **Elements Map** tab to select it.
The list of elements has been transferred from FQuant, and no further action is needed here.



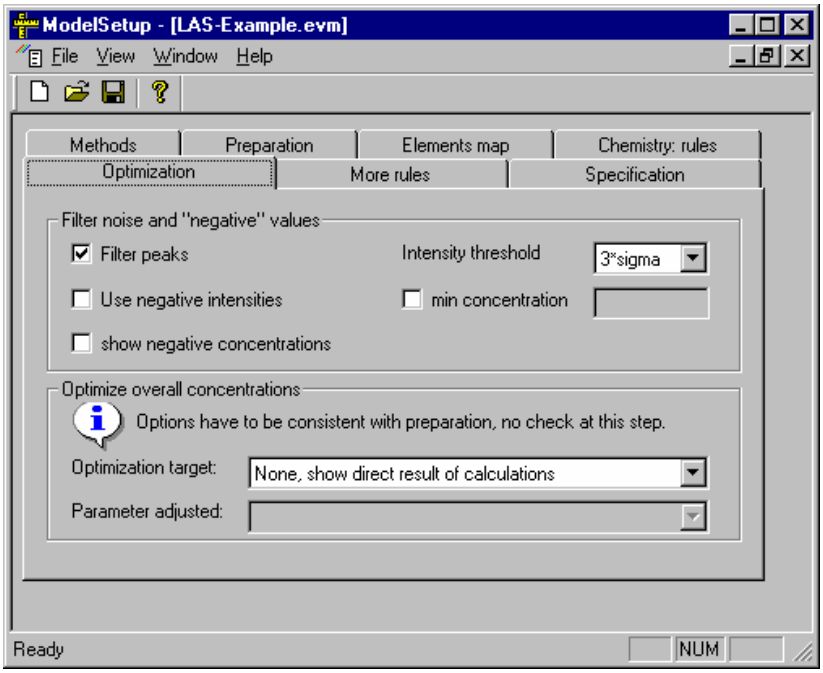


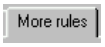
Click the **Chemistry: Rules** tab to select it.
This can be used to indicate special rules when using the Standardless program, like matrix element, compound formulas, etc.

The Default Chemistry has been set to “Calibration Compounds”, because this example will use a formal calibration to calculate sample composition.

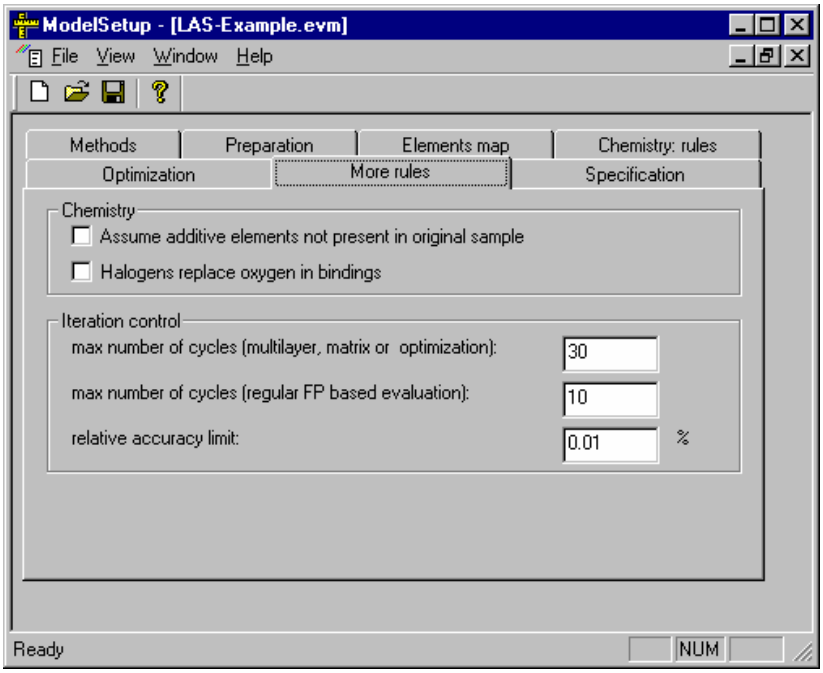


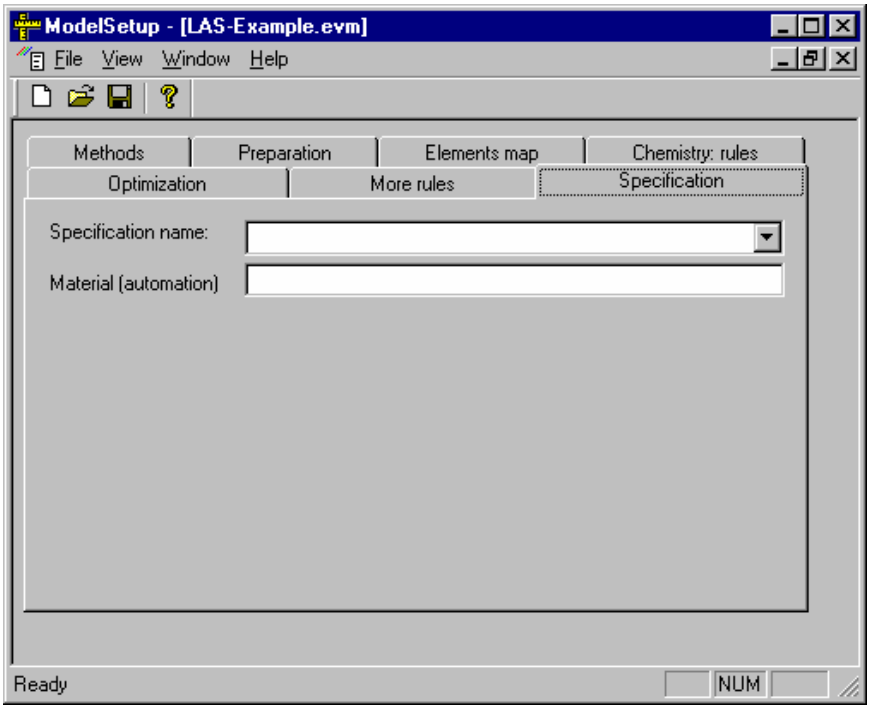
Click the **Optimization** tab to select it.
This can be used to force SPECTRA*plus* to show all concentrations including those with negative values, and those that are statistically insignificant.





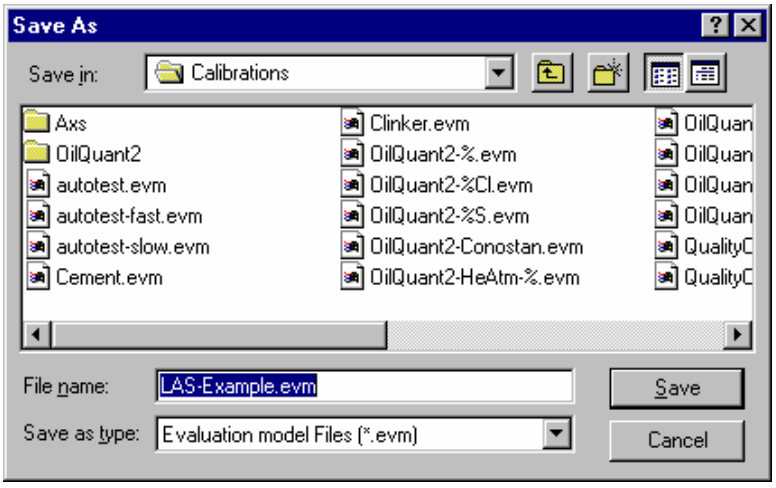
Click the **More rules** tab to select it.
This can be used to change the number of iterations SPECTRA*plus* uses when calculating the composition of a sample, and other special chemistry rules.





FILE Click **File→Save**.

SAVE Verify that you save into the Calibrations directory.



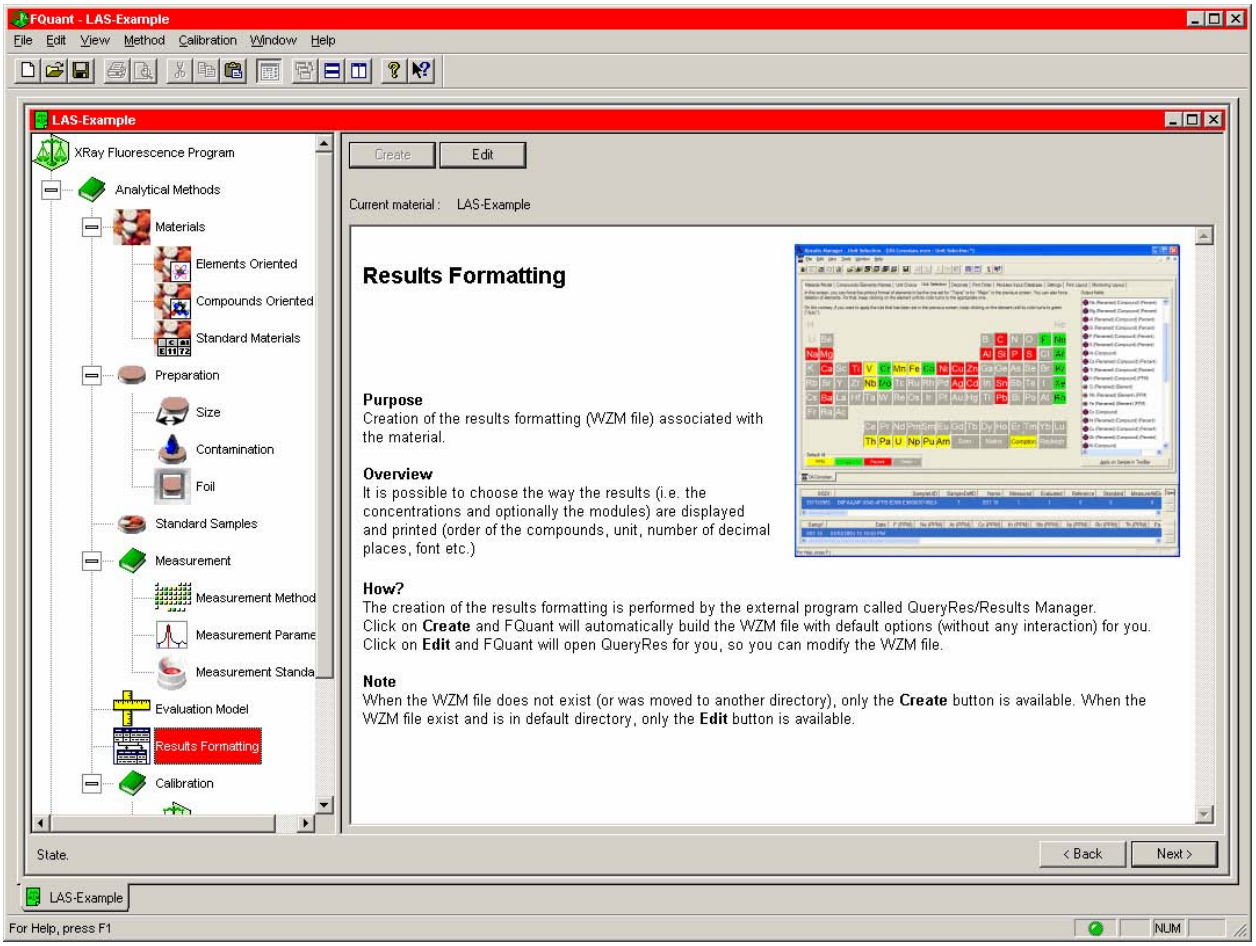
3.9 Creating a Results Format Method

In this step, the format for printing and displaying results will be specified.

Click the **Next** button to move to **Results Formatting**.

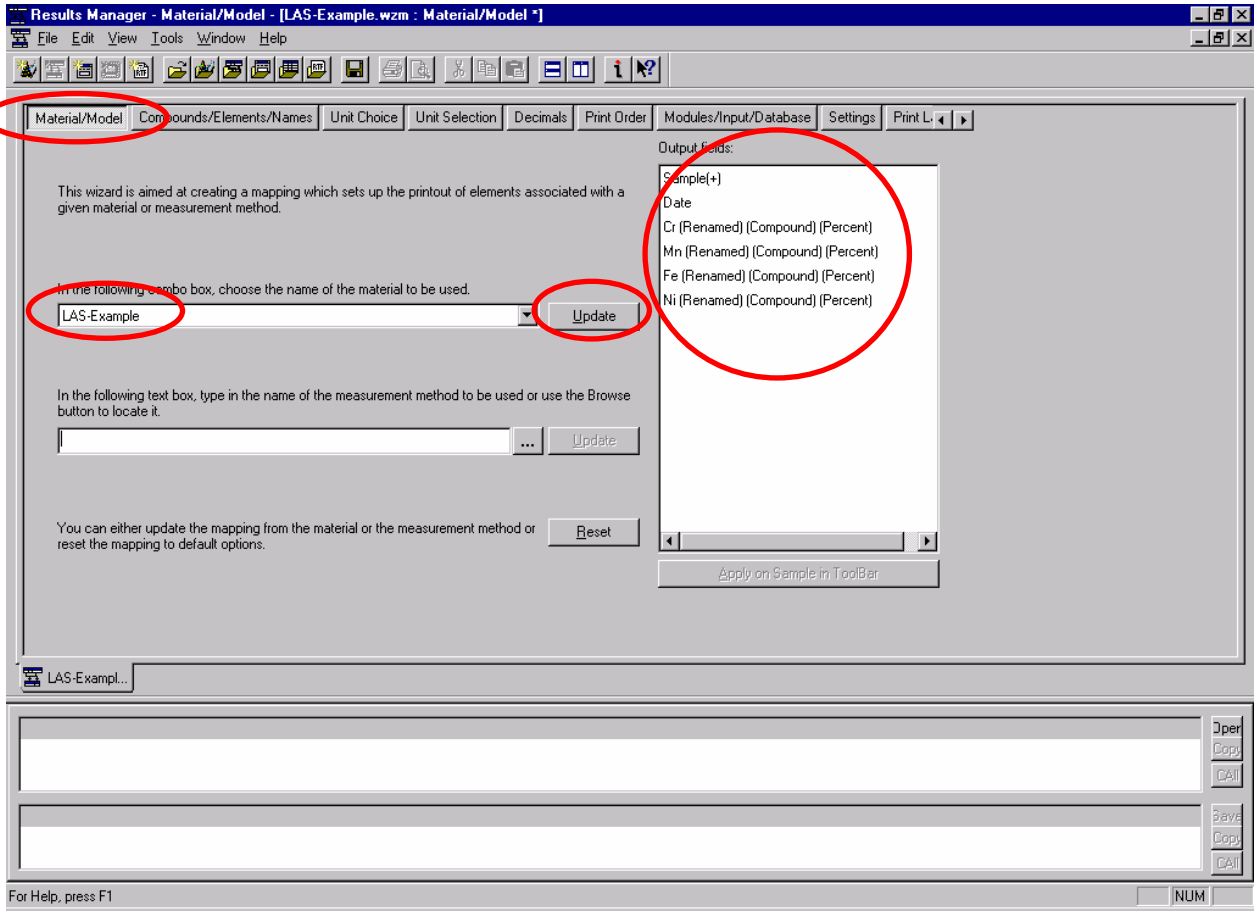
Click the **Create** button to create a default printout for the method. This will be enough to display the results the same way the standards were defined. It is not necessary to edit the format, unless it is desired to add more options.

Click **Edit** to modify the format.



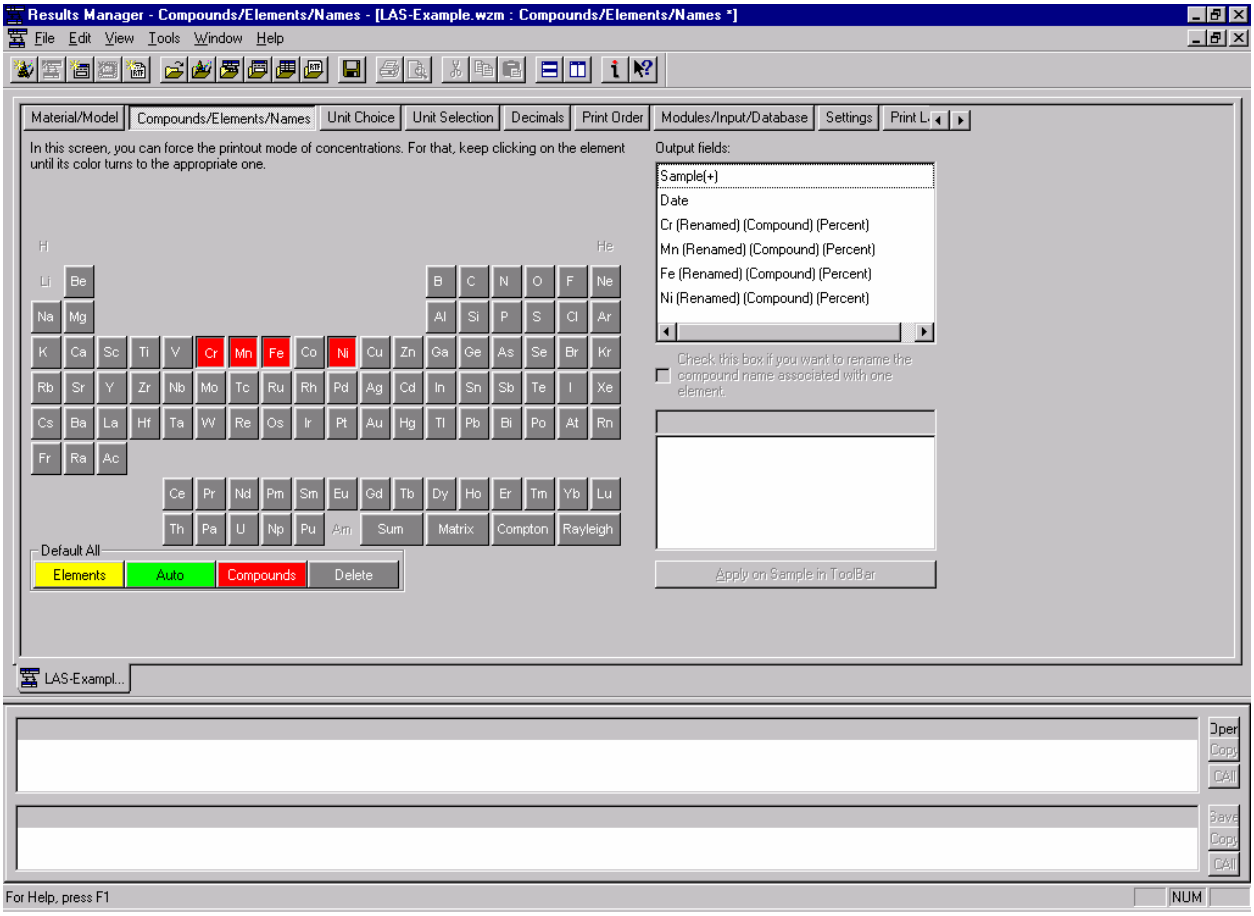
Material/Model

The **Material/Model** tab button should already be selected.
The information given in FQuant for our Material has been automatically transferred over.



Compounds/Elements/Names

Click the **Compounds/Elements/Names** button to select it.
This screen allows the user to select, on an element-by-element basis, whether to report the concentration as an “Element”, a “Compound”, or “Auto”. “Auto” reports the concentration as an element or a compound based on a concentration threshold that will be set later. In the context of this example, “Compound” refers to a calibrated compound. Since the calibration will be done as “Elements”, compound and element mean the same thing.

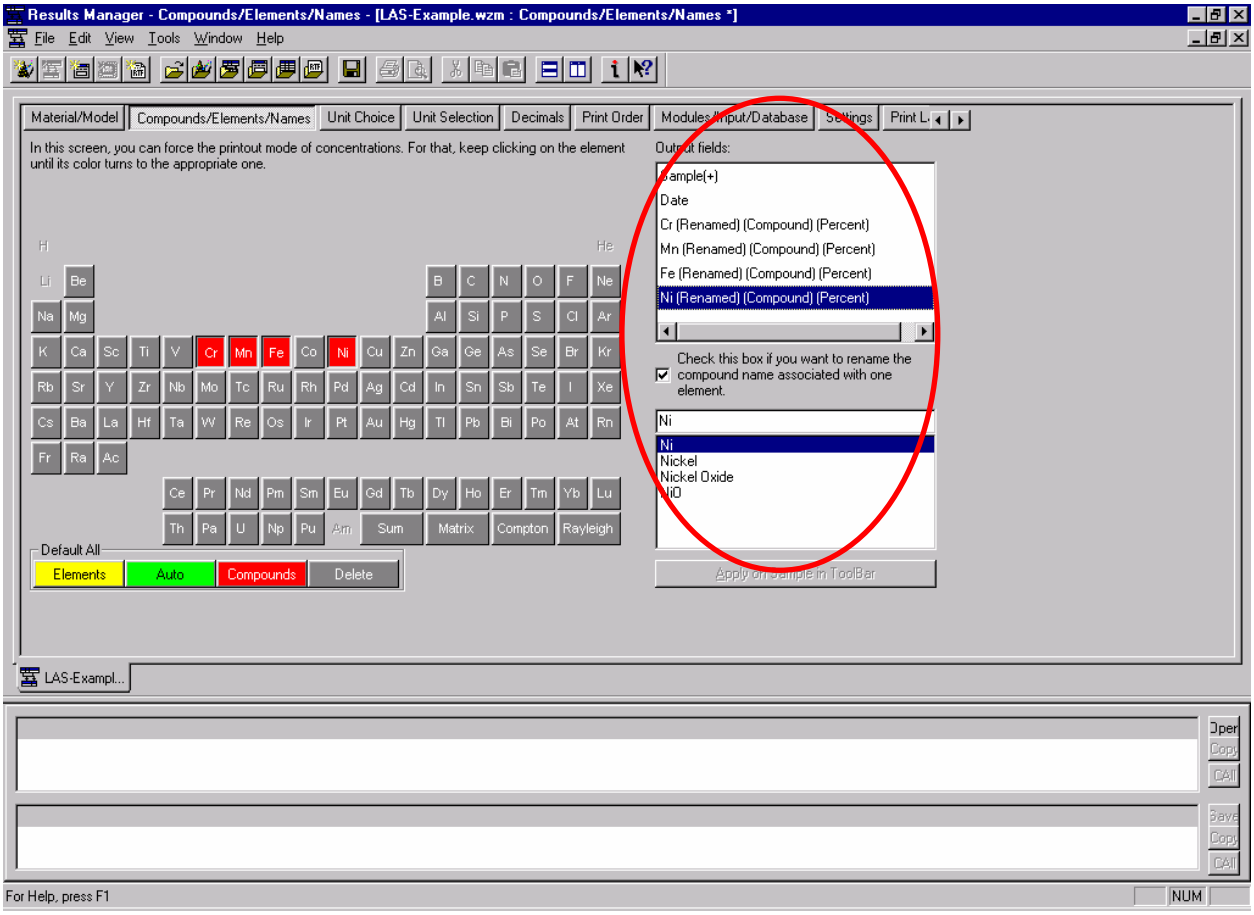


It is also possible to rename one of the constituents so that it will be reported using a different name, for example:

Click on **Ni** to highlight it

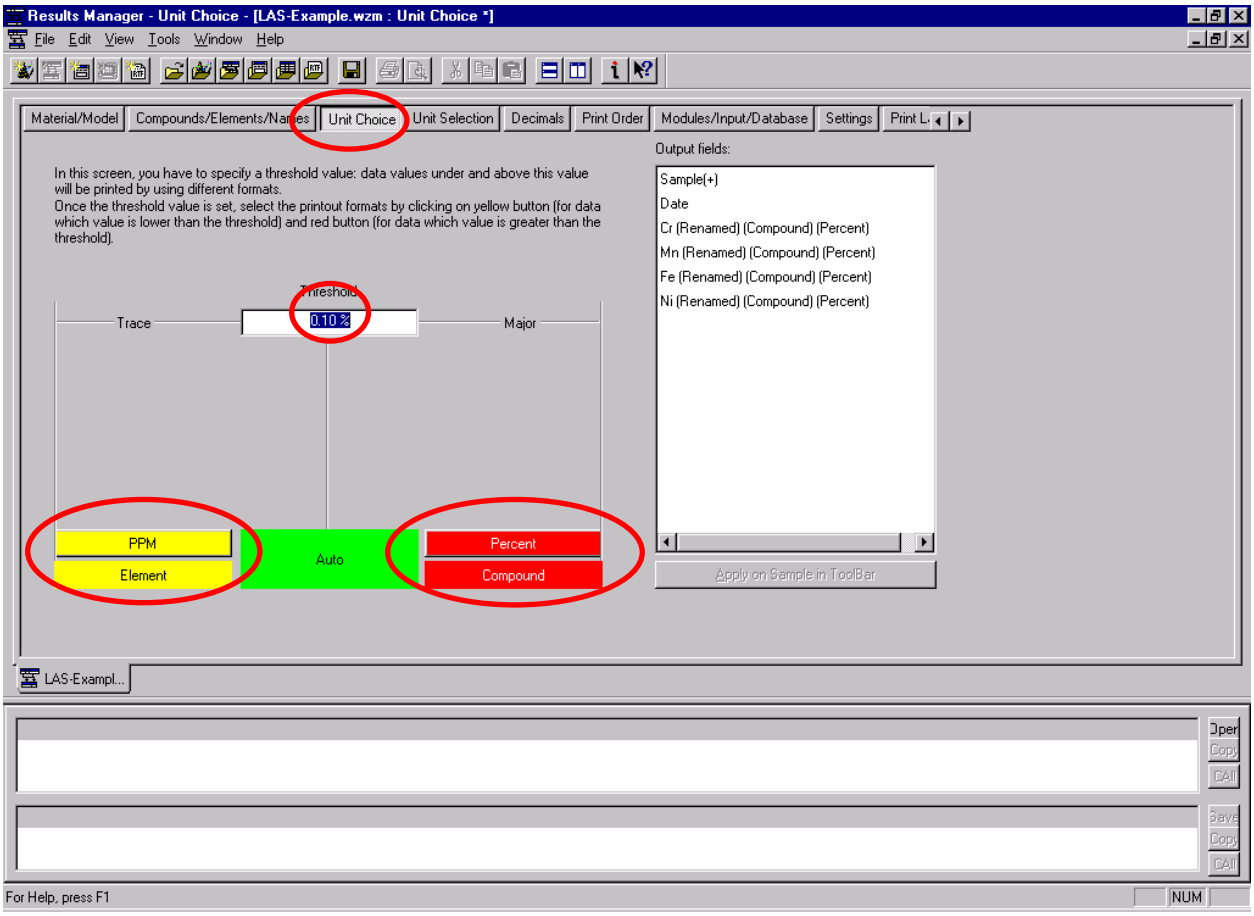
Check the box to rename it.

Type the new reporting name, or select the new name from the list of names given.



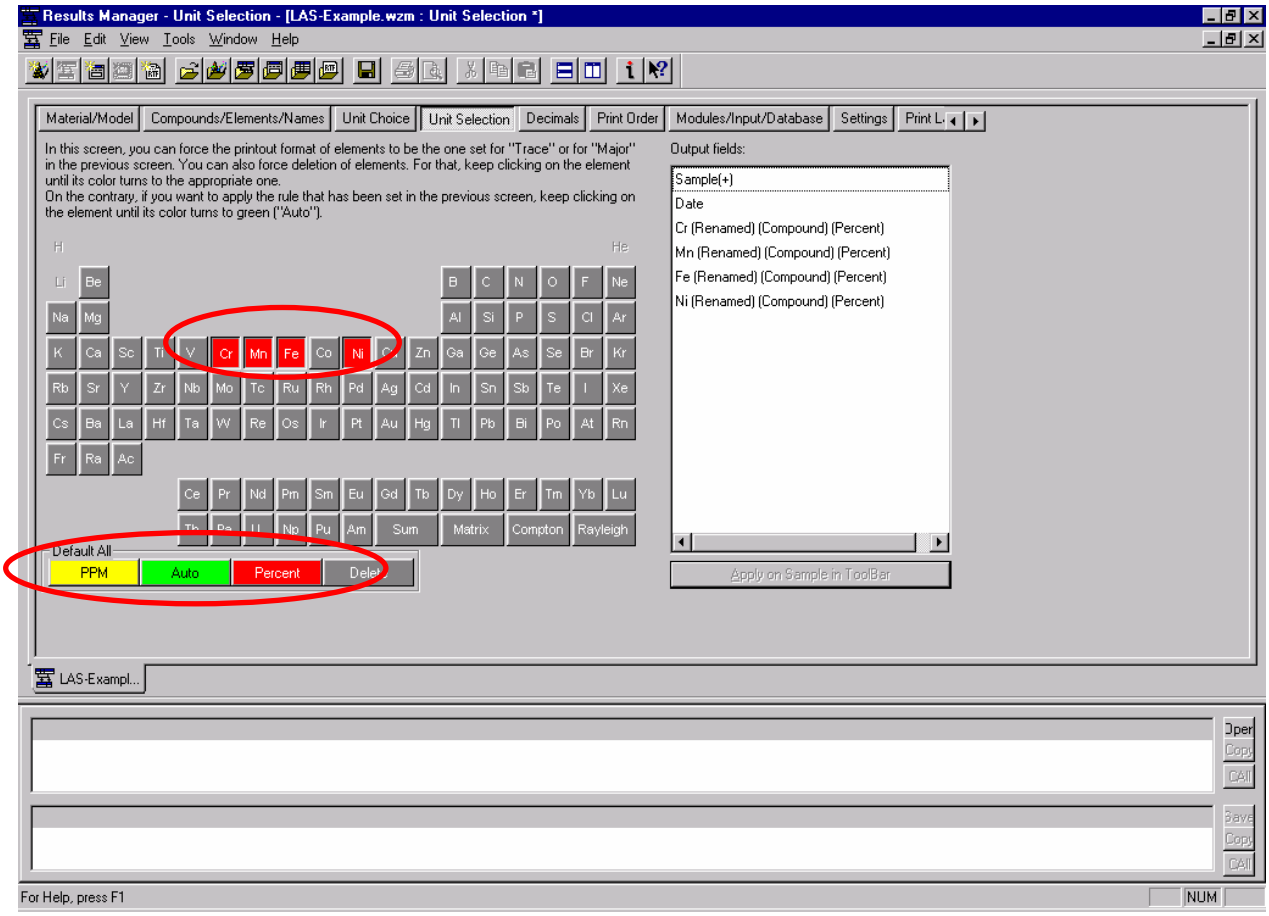
Unit Choice

Click on the **Unit Choice** button to select it.
SPECTRA*plus* uses two units for reporting concentrations. One is a “trace” unit, which by default is set to ppm. The other is a “major” unit, which by default is set to %. These default units can be changed by clicking on the “PPM” or “Percent” buttons.
This is also where the concentration threshold would be set to control the automatic (“Auto”) switching between the major and trace units.



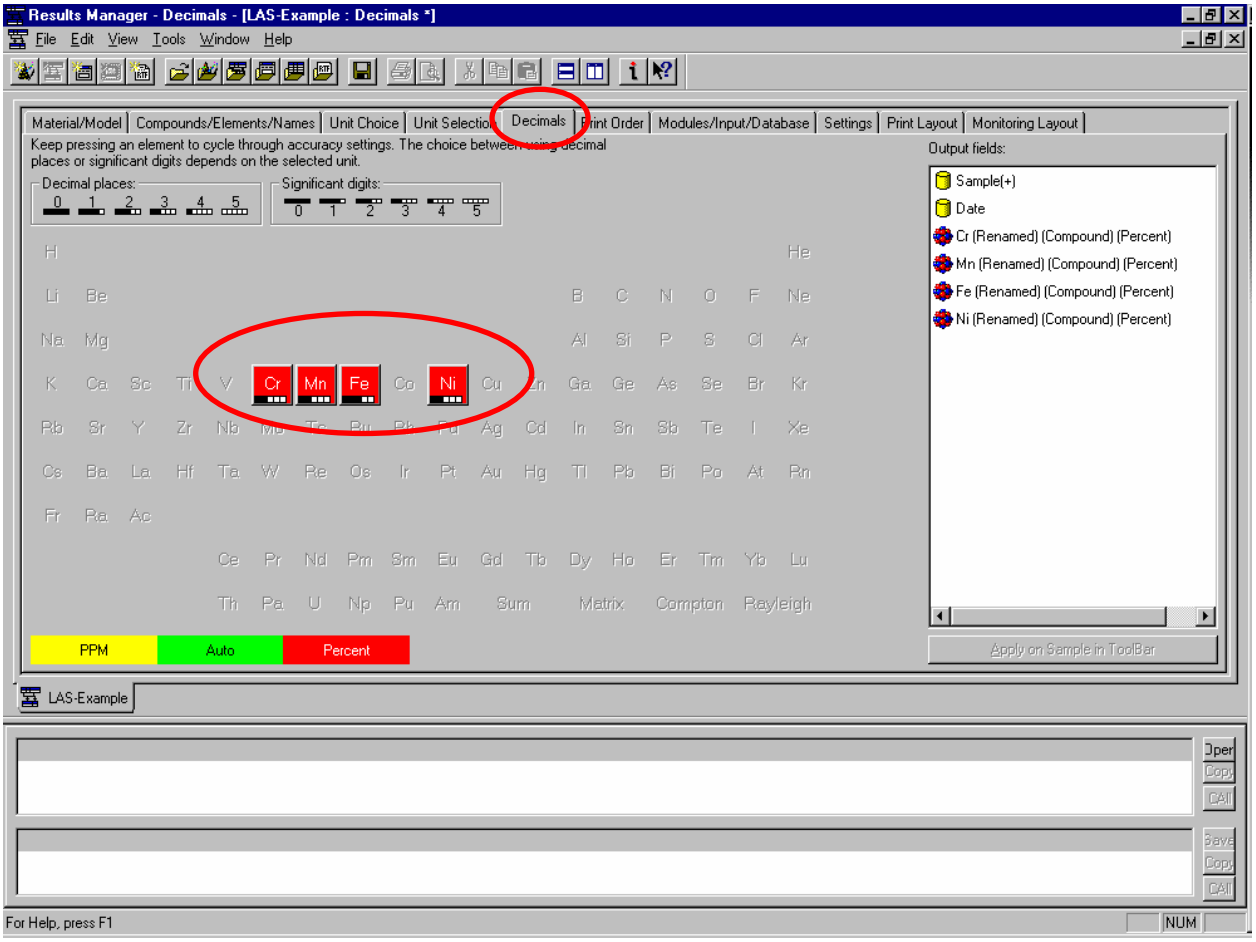
Unit Selection

Click on the **Unit Selection** button to select it.
This is where the reporting units for each element can be specified. Whatever units were defined in Section 3.2 will automatically be set here. These settings can be changed by clicking on the “PPM”, “Auto”, “Percent”, or “Delete” buttons to set all elements to the button selected. Individual elements can be changed by clicking an element symbol to match its color to one of the buttons.



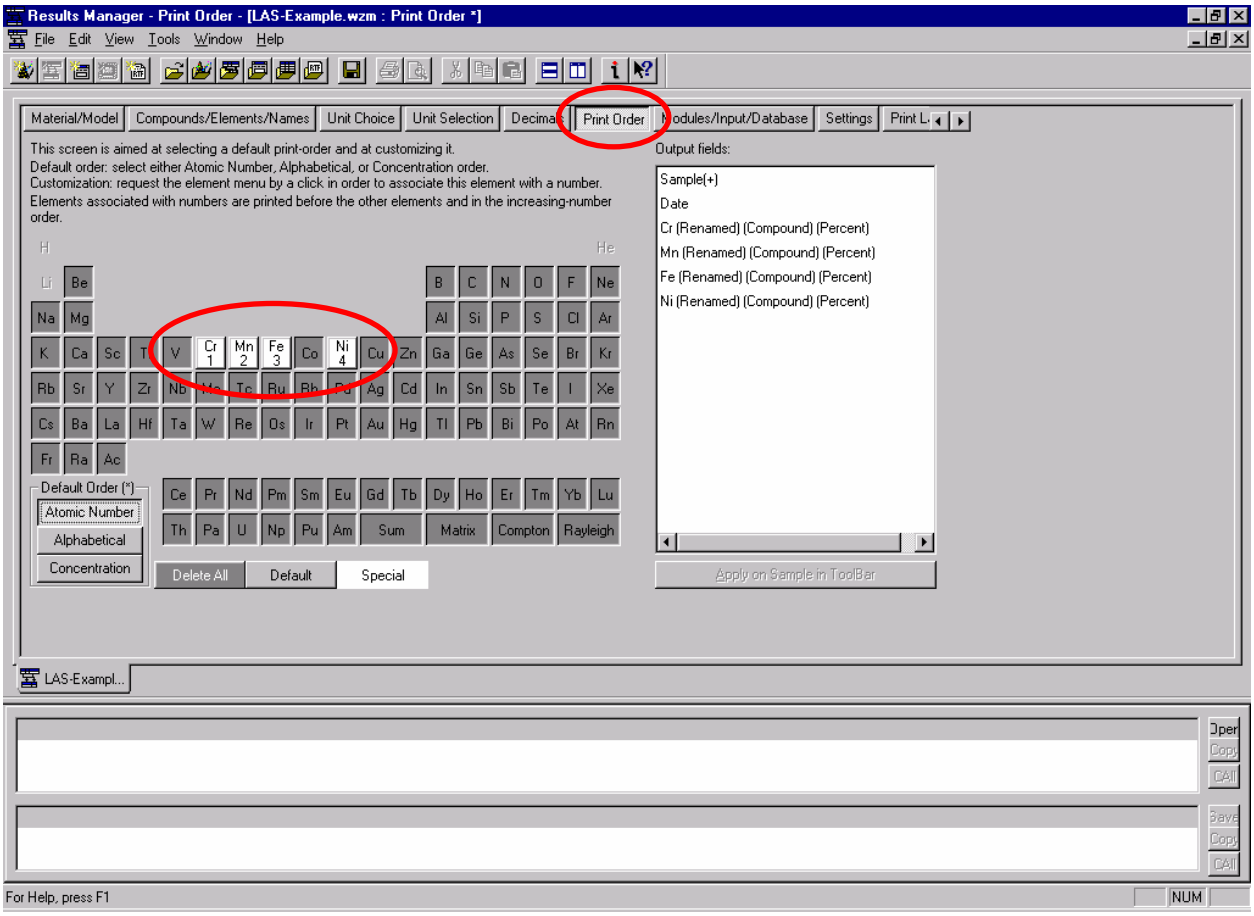
Click on the **Decimals** button to select it.
This is where the number of decimals to report can be given for each element. The decimals defined in Section 3.2 will automatically be set here.

The actual number of decimals is indicated by white dots shown below the element symbol. The number of decimals can be changed by clicking on an element symbol.



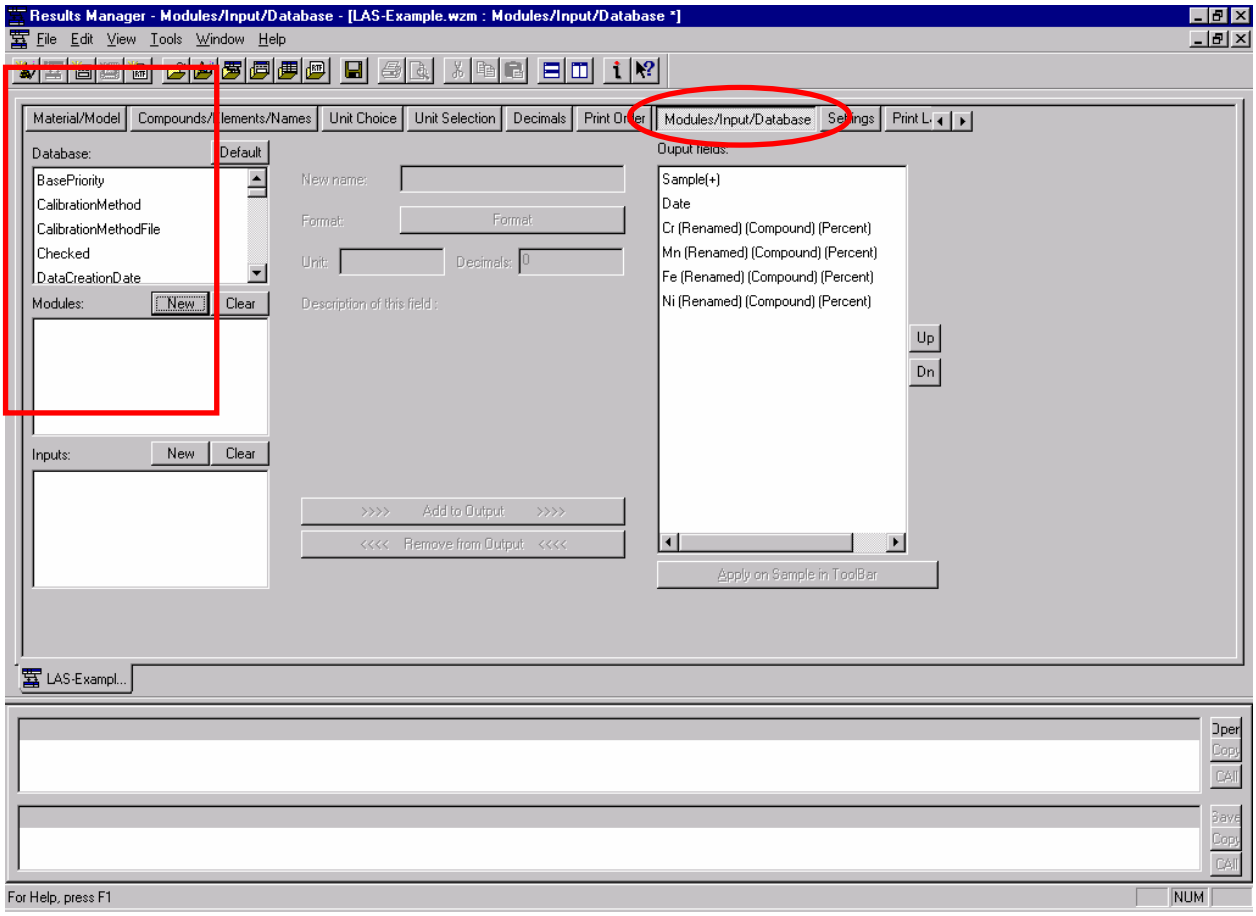
Click on the **Print Order** button to select it.
This is where the order in which to print the elements is given. The order elements were added to the Material in Section 3.2 will automatically be set here.

Individual element symbols can be clicked to give a different order.



Click the **Modules/Input/Databases** button to select it.

From here, additional information stored in the database "\SPECplus\Databases\Measure.mdb" can be added to the output. Results from a Modules calculation or manual inputs from a Sample Definition can also be added (see SPECTRA*plus* Reference Manual).

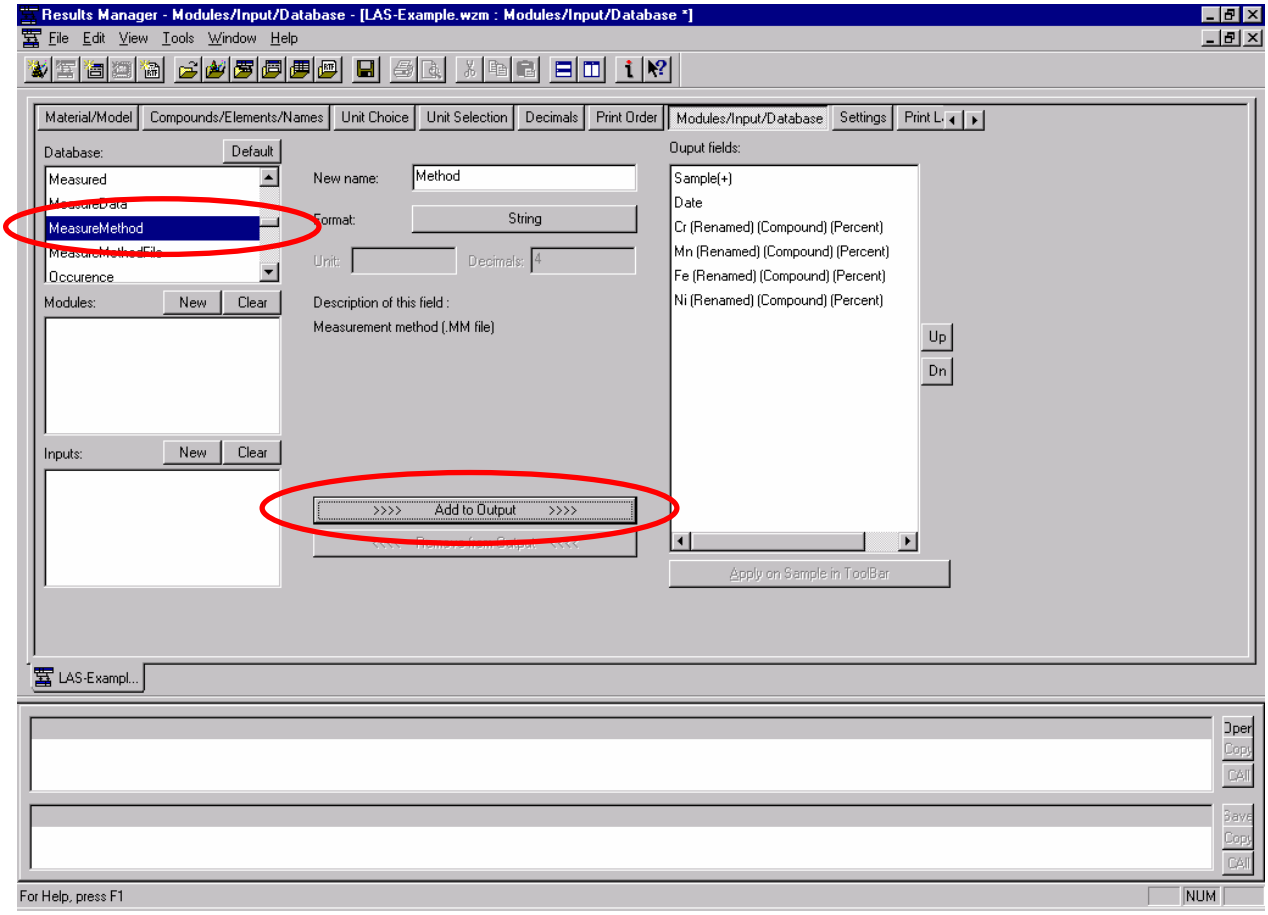


One of the useful items to add from the Database is the “Measurement Method Name”. Normally this is not displayed, but is good to have on reports so that it is clear how a sample was measured. To add this to the output:

Locate, then highlight **MeasureMethod** in the Database window.

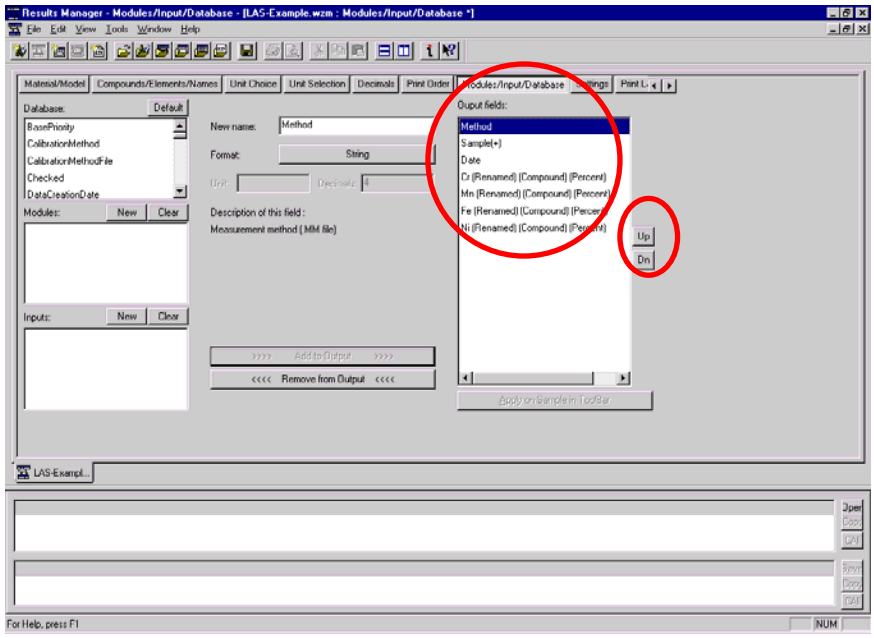
Type “Method” in the “New name:” box to give this field a shorter name.

Click the **Add to Output** button.



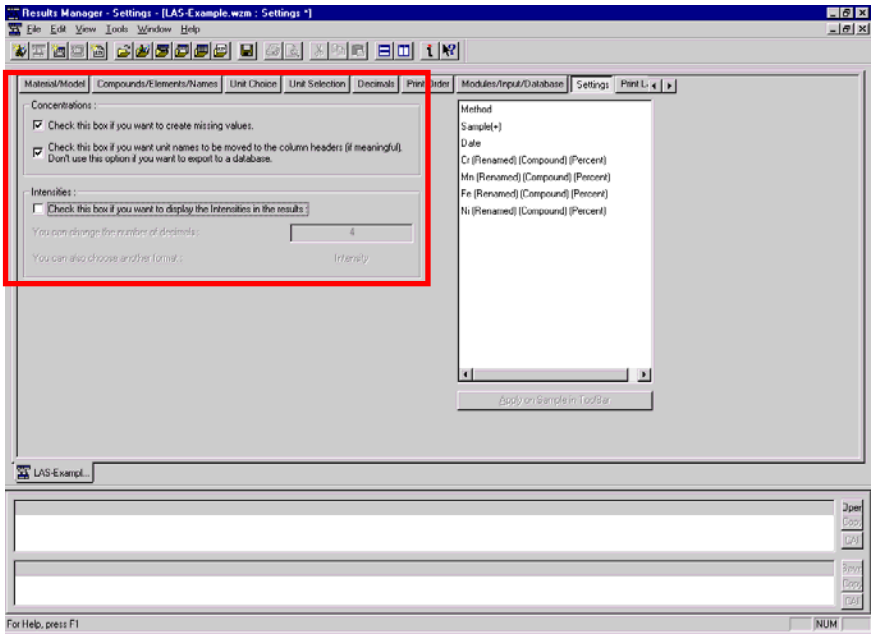
The Measurement Method will now be listed in the output under the heading of “Method”.

The **Up** and **Dn** (Down) buttons can be used to reorder items in the output.



Click the **Settings** button to select it.

This screen provides options for special settings, such as:
- Replacing missing values with zeroes
- Moving the concentration units to the header, instead of putting them in the cell with the value
- Displaying the intensities

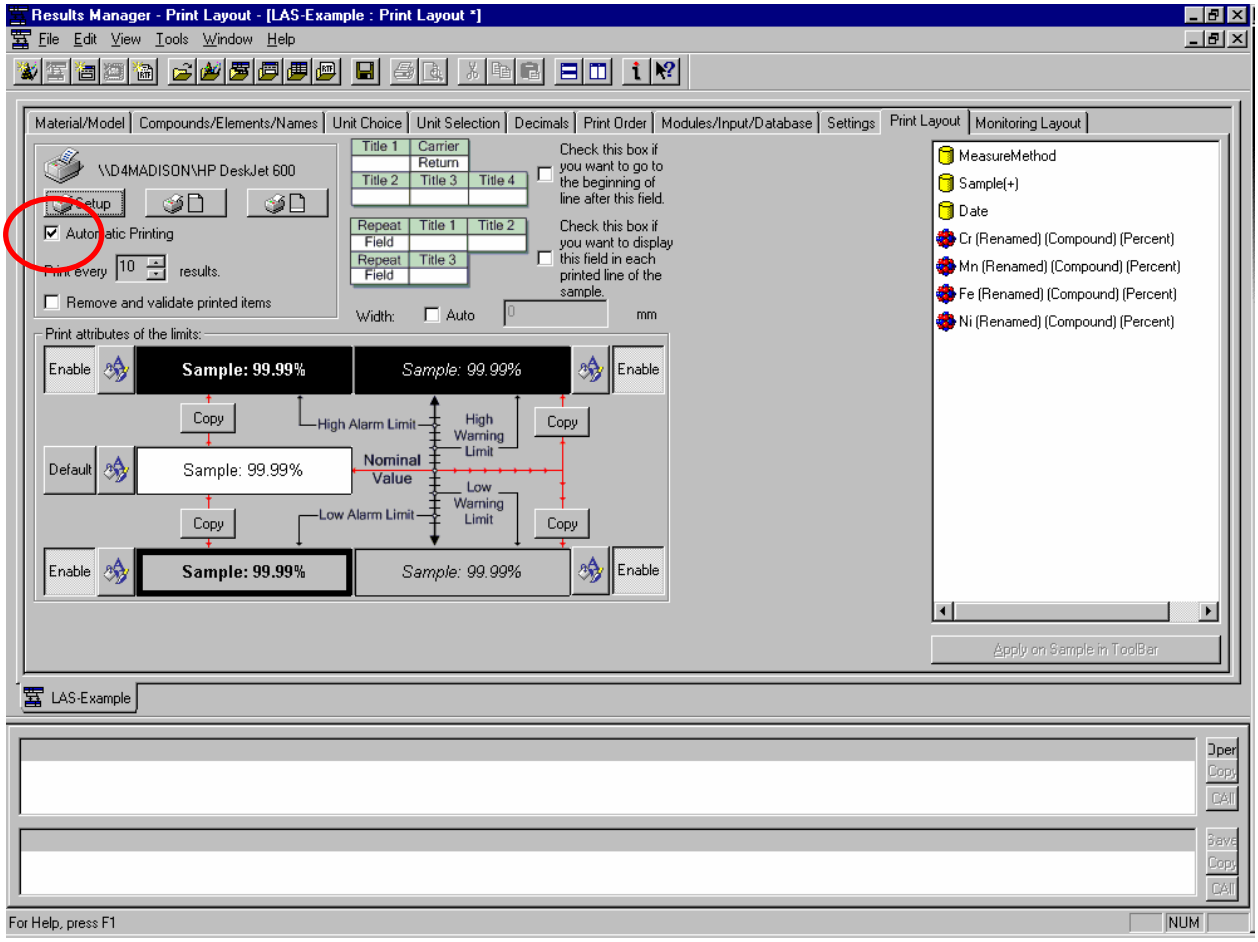


Print Layout

Click the **Print Layout** button to select it.
This screen allows giving options for the printouts, like page orientation, headers, etc. It also allows requesting automatic printouts after a fixed number of samples have been displayed.

In this example, automatic printing will not be used.

☐ Un-check the Automatic Printing box.



Click the **Monitoring Layout** button to select it.

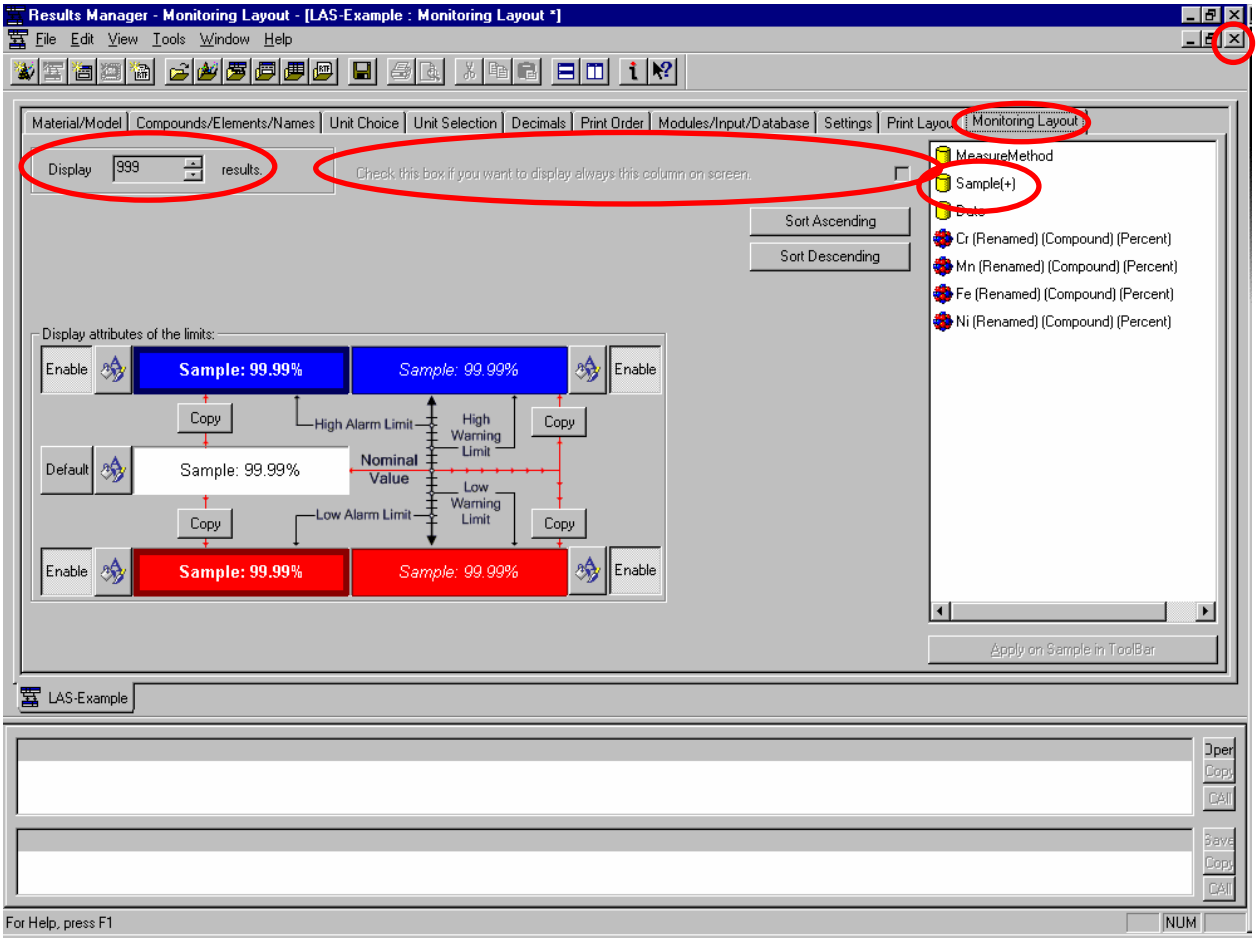
This screen is used to control the display of results on the monitor.

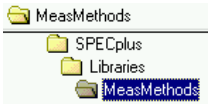
Use the arrows to increase the **Display** number to its maximum value or 999.
This controls the maximum number of samples that will be displayed at any given time.

Highlight **Sample** in the field list window.

Check the box to always display this column. This will keep the sample name on the screen when a long list of element concentrations is scrolled.

Click the **Close** button to exit the program.

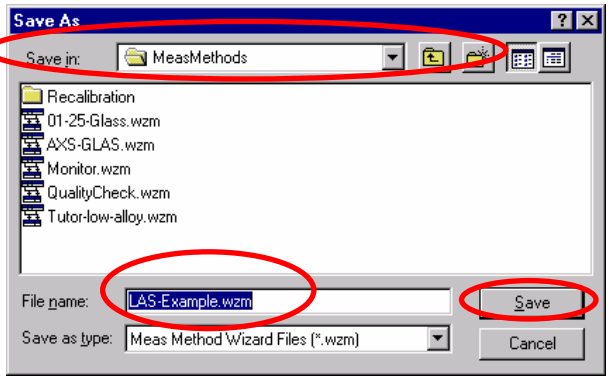




Make sure the directory is set to “\SPECplus\Libraries\MeasMethods” in the **Save In** box.

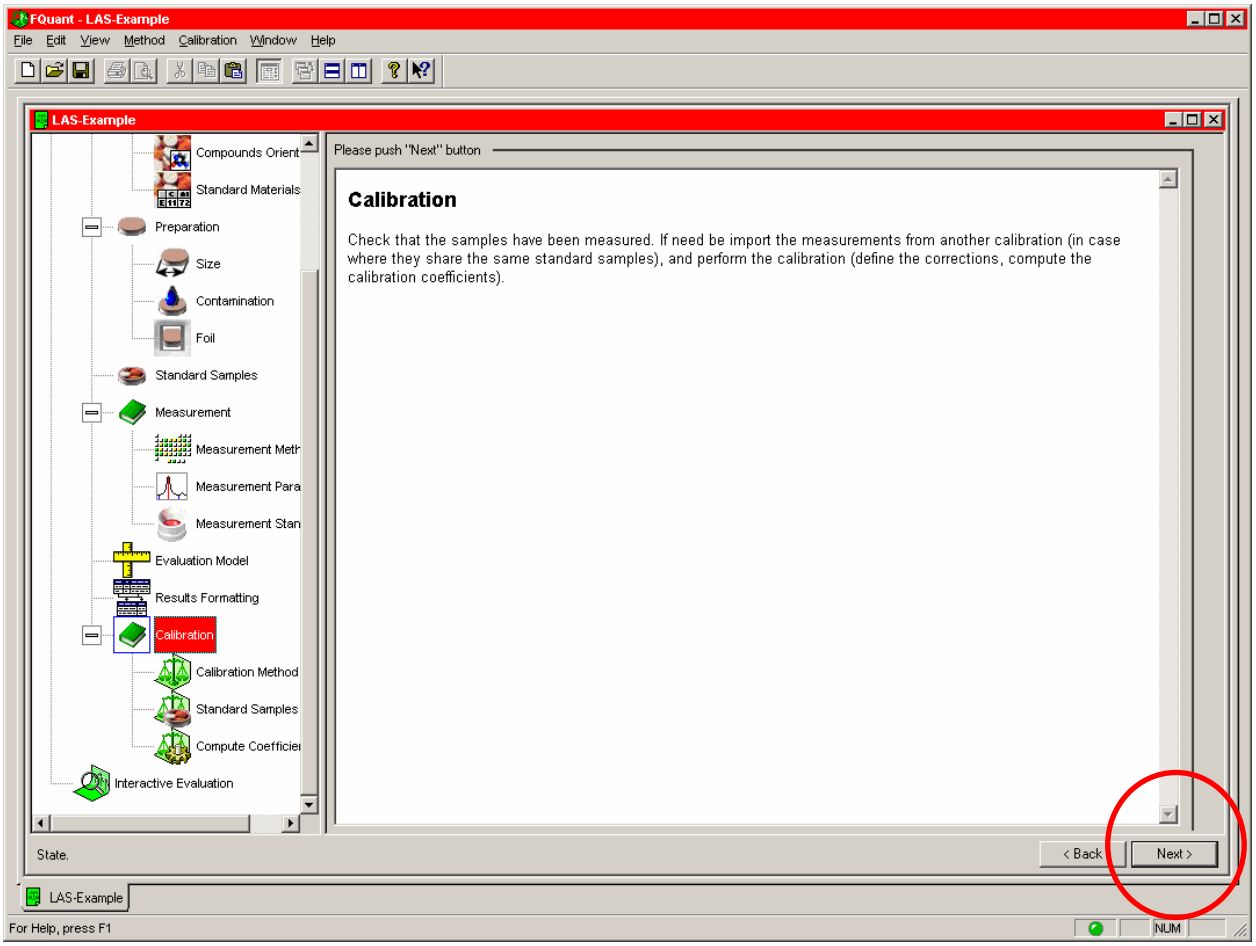
“LAS-Example.wzm” should be in the **File Name** box. Enter it if it is not.

Click the **Save** button.



3.10 Calculating Calibration Coefficients

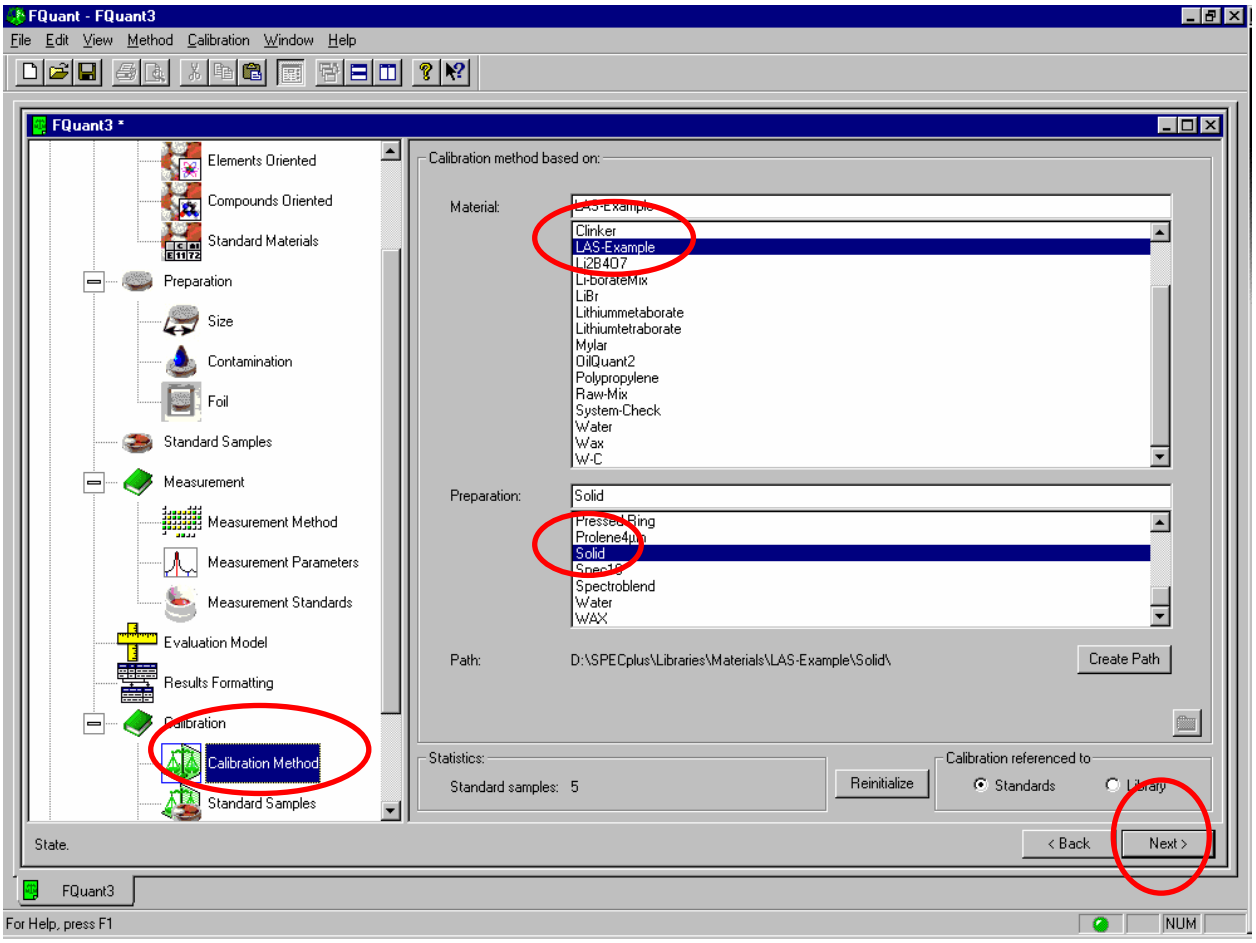
In this section, the intensities measured from the five calibration standards as outlined in Section 3.7 will be regressed against the concentrations entered in Section 3.2.

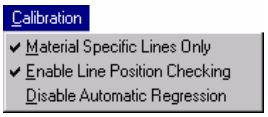


Next > Next > Click the **Next** button twice to move to **Calibration Method**.

“LAS-Example” should be selected in the Material box.

“Solid” should be selected in the Preparation box.


FQuant - FQuant3
File Edit View Method Calibration Window Help
FQuant3 *
Elements Oriented
Compounds Oriented
Standard Materials
Preparation
Size
Contamination
Foil
Standard Samples
Measurement
Measurement Method
Measurement Parameters
Measurement Standards
Evaluation Model
Results Formatting
Calibration
Calibration Method
Standard Samples
Calibration method based on:
Material:
Clinker
LAS-Example
Li2B4O7
Li-borateMix
LiBr
Lithiummetaborate
Lithiumtetraborate
Mylar
OilQuant2
Polypropylene
Raw-Mix
System-Check
Water
Wax
W-C
Preparation:
Solid
Pressed Ring
Prolene4µm
Solid
Spectroblend
Water
WAX
Path:
D:\SPECplus\Libraries\Materials\LAS-Example\Solid\
Create Path
Statistics:
Standard samples: 5
Reinitialize
Calibration referenced to
Standards
Library
State.
< Back
Next >
FQuant3
For Help, press F1
NUM



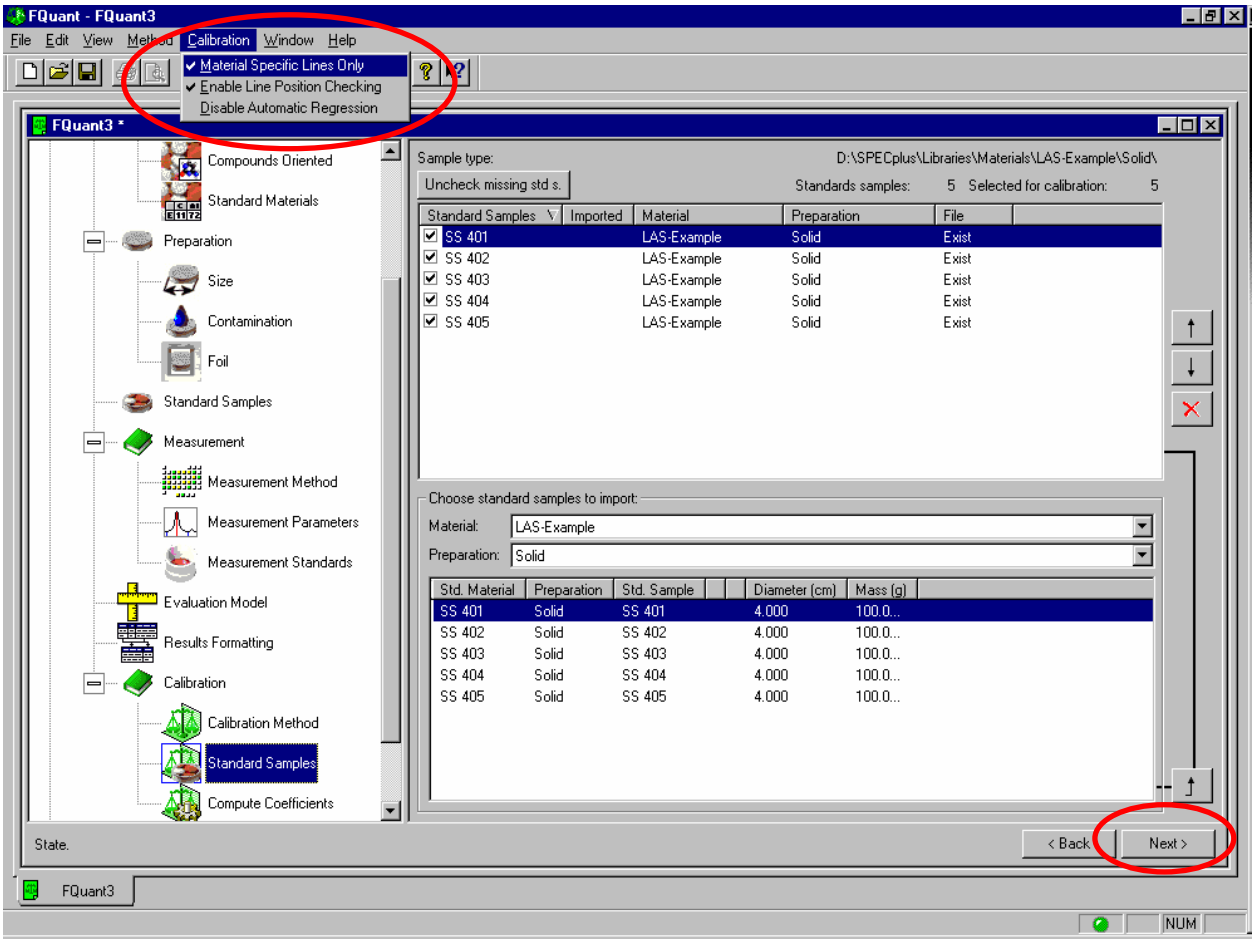
Click **Calibration** on the menu, and set the options as shown.

Material Specific Lines Only: Checked to use only lines with the Material name in them (“/LAS-Example”).

Enable Line Position Checking: Checked to make sure peak and background positions in the intensity data files match those stored in the Line Library.

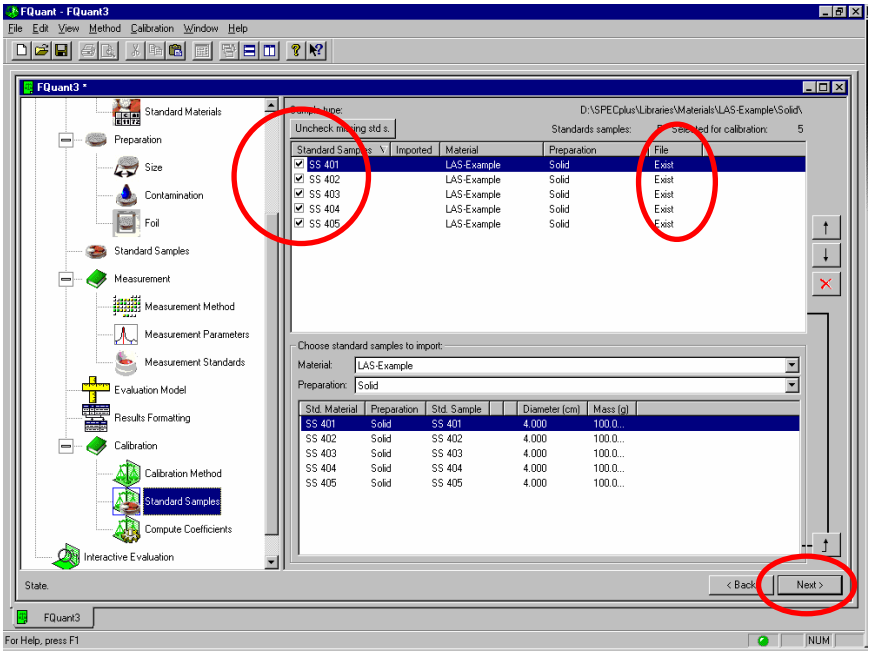
Disable Automatic Regression: Unchecked to automatically calculate the calibration coefficients as each element is visited.

Click the **Next** button to move to **Standard Samples**.



Five standard names should be listed with a check mark by their names. The column labeled “File” should have the word “Exist” for each sample. This means that the intensity data file was found.

Click the **Next** button to move to **Compute Coefficients**.



The **Calibration Toolbox** should be displayed on the screen. If it is not, press the F2 key to display it. This example shows “Cr” as the select element. Do not worry at this point which element is shown in your “Toolbox”. This toolbox is used to ask for different “views” of the data, like a graphical or tabular view. It is also used to move from element to element in the data set.

Make the initial settings to the toolbox as shown below:

The “E” or “Element” button is pushed in to show calibration data as elements. The “C” or “Compounds” button should be pushed in when doing compounds. In this case, either button could be pushed in.

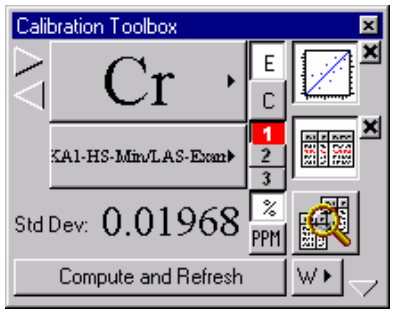
The “Show Graphical Display” button should be pushed in; a Close button is displayed to the right of it. This will show a graphical representation of the data in a separate window.

The “Show Tabular Display” button should be pushed in; a Close button is displayed to the right of it. This will show a tabular representation of the data in a separate window.

The “Show Comparison” button should not be pushed in; a Close button should not be displayed to the right of it.

The “%” button should be pressed in to display the concentrations in percent instead of ppm.

Click the “Show/Hide Additional Tools” arrow in the lower right corner to expand the Calibration Toolbox.



The “Show Tabular Display” button should be pushed in; a Close button is displayed to the right of it. This will show a tabular representation of the data in a separate window.

The “Show Overlaps” button should be pushed in; a Close button is displayed to the right of it. This will allow adding and removing of “spectral overlap” corrections.

The “Show Internal Standard” button should not be pushed in; a Close button should not be displayed to the right of it. No “internal standards” are used in this method.

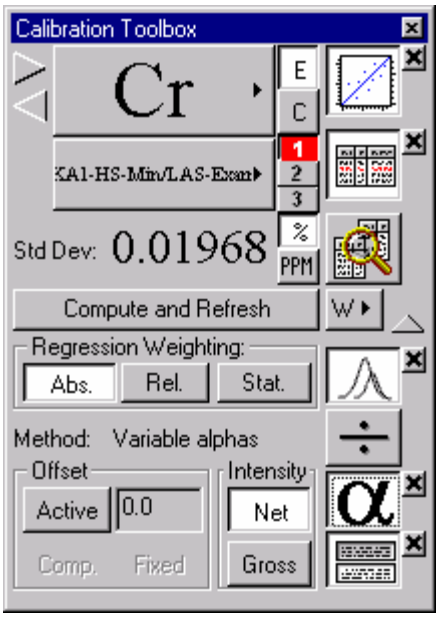
The “Show Coefficients” button should be pushed in; a Close button is displayed to the right of it. This will allow disabling the default “matrix” corrections from the calibration.

The “Show Summary” button should be pushed in; a Close button is displayed to the right of it. This will show a summary of the calibration data.

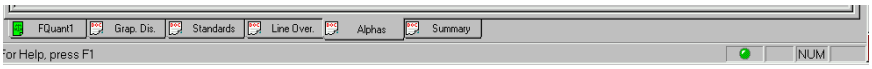
The “Minimize the Absolute Errors” button should be pushed in. This will make the regression program try to minimize the “absolute” errors between the calculated concentrations and the given concentrations.

The “Use Net Intensities” button should be pushed in. In this example, we want to subtract the background intensity from the gross peak intensity.

Click the “Show/Hide Additional Tools” arrow in the lower right corner to contract the Calibration Toolbox.



Note: As the icon buttons are pressed, requesting different views of the data, tabs will appear on the bottom of the main window. Switching to a different view can now be accomplished by clicking a tab on the bottom of the screen, or on the icon that opened that tab. Closing a view can be accomplished by clicking the Close button (“X”) to the right of the appropriate icon.

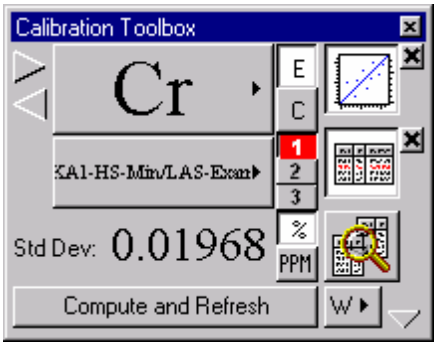


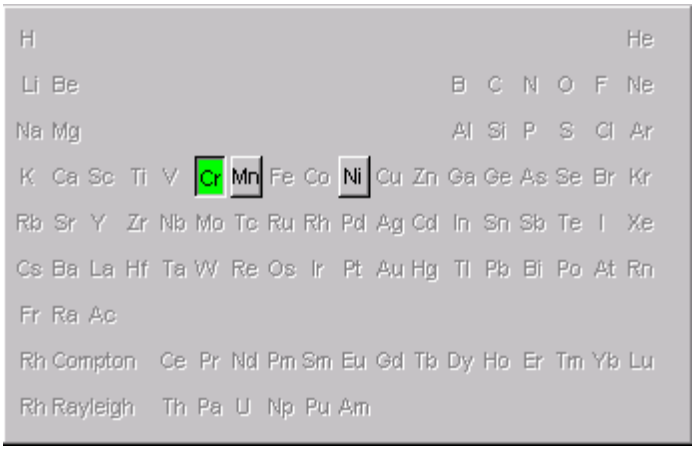
Using the Element Symbol control in the Toolbox, we can move from element to element within the data.

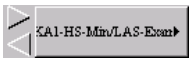
The Forward arrow moves to the next higher-atomic-number element in our list.

The Back arrow moves to the next lower-atomic-number element in our list.

The dark arrow to the right of the Element Symbol displays a periodic table that enables selection of any element in our list.







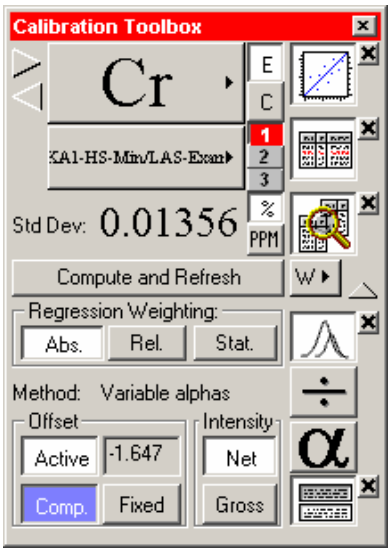
Using the Line Symbol control in the Toolbox, we can select which line to calibrate if multiple lines were measured. This example only measured one line per element.

The Forward arrow moves to the next line in our list of lines.

The Back arrow moves to the previous line in our list of lines.

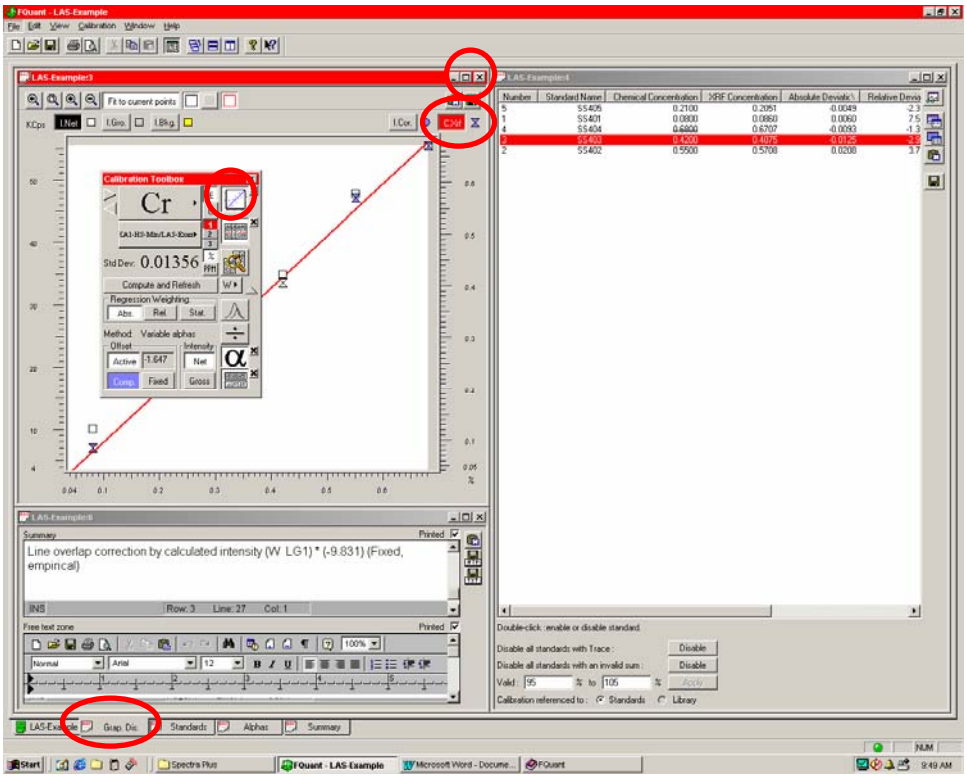
The dark arrow to the right of the element symbol displays the list of possible lines for calibration.

SPECTRA*plus* can be calibrated for up to three lines per element. Using the numeric control, we can designate each line as the default, secondary, or tertiary line. Our line should be designated as the primary line, so the “#1” should be pushed in, and its color should be red.

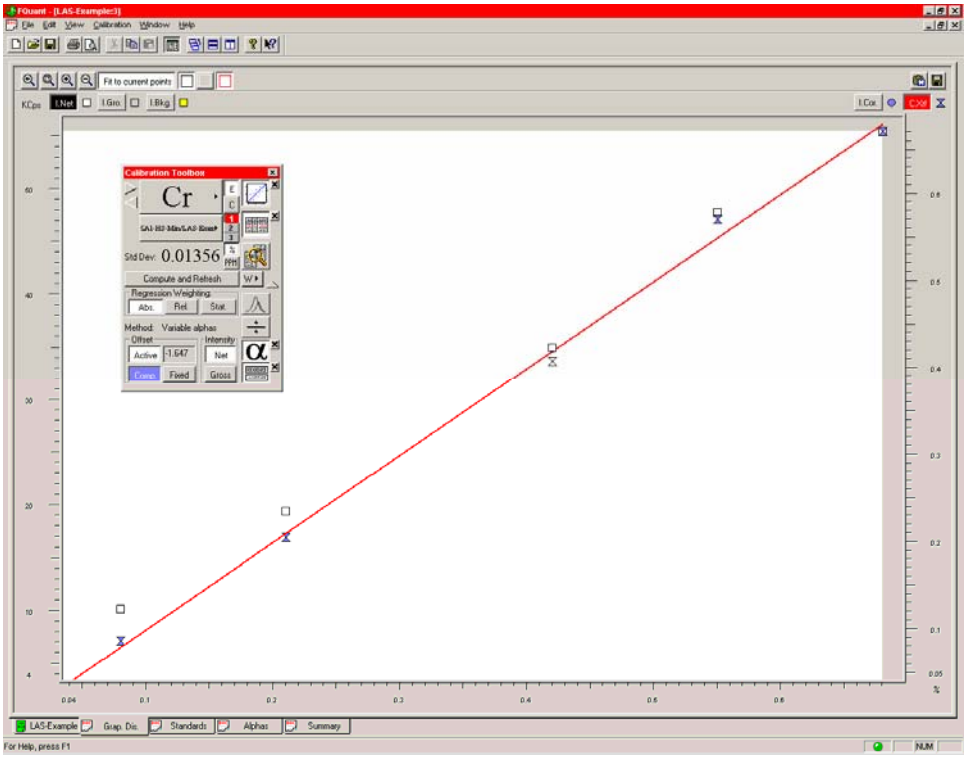


Select the **Graphical Display** tab.

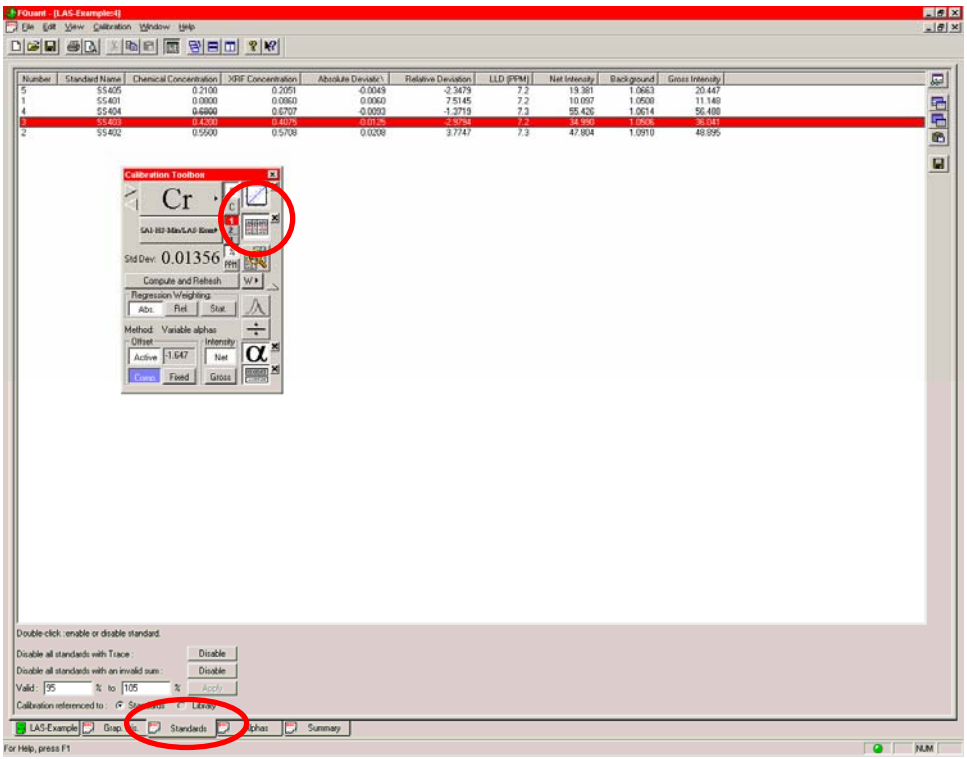
Check that C XRF is displayed on the right.



Expand the window to full screen.

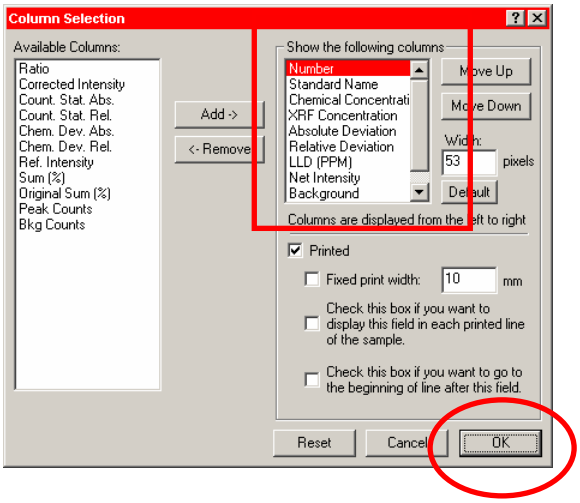


Select the Tabular/Standards display and expand it to full screen.

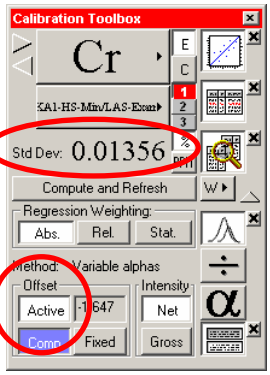


Select the Eyeglass button.

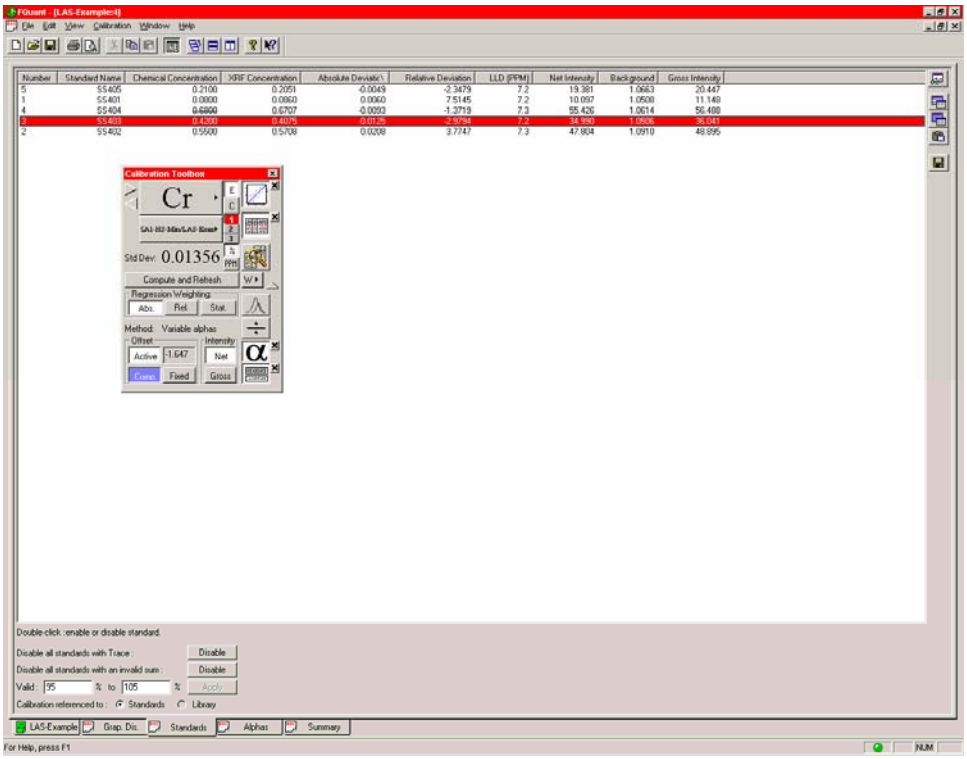
Select the following columns to be displayed: Number, Standard Name, Chemical C, XRF Conc, Abs dev, Rel Dev, Gross Int, Background, Net Intensity, LLD.



Activate Offset, check out the Standard deviation displayed.

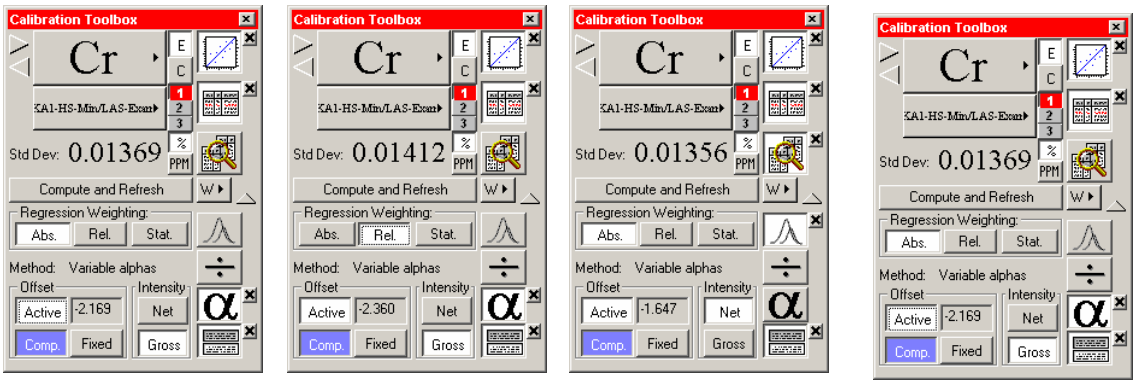


Compare the XRF with the Chemical Concentration in the Standards display.

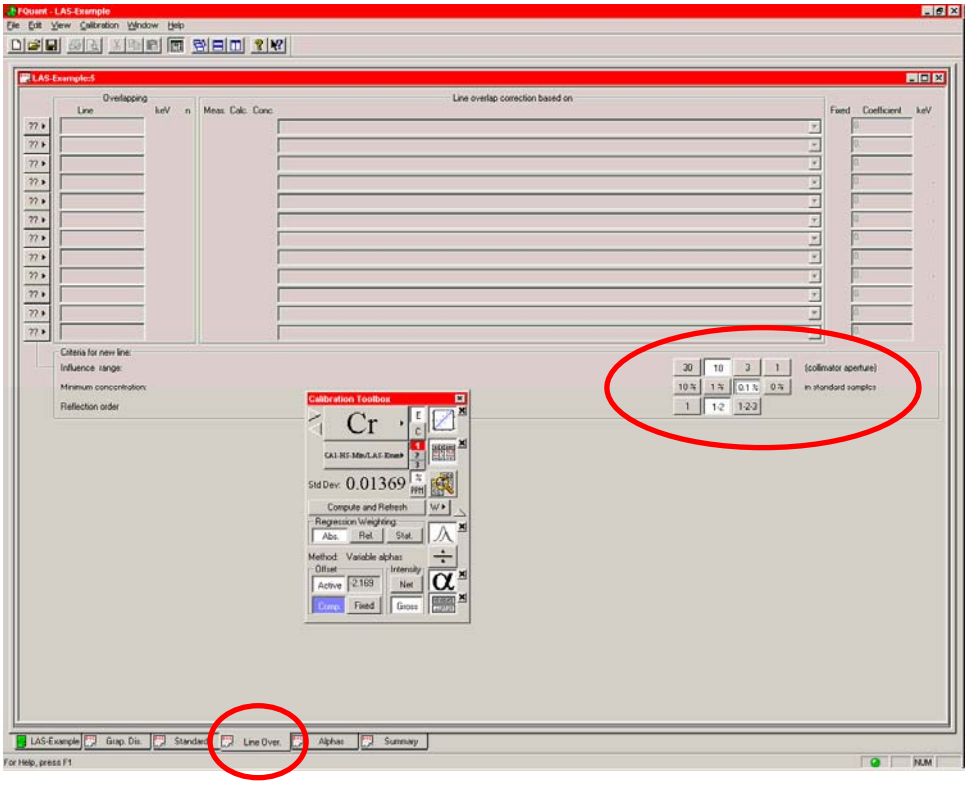


Select the **Compare** button, which shows you the function which logs the progress and all settings for each step performed so far.

Change from NET to GROSS on the Toolbox and see how it impacts the standard deviation displayed, change the regression weight from abs to relative and observe. At the end, switch back to NET and Active Offset.



Select the **Line Overlay** tab.



Select 10 for Influence Range.

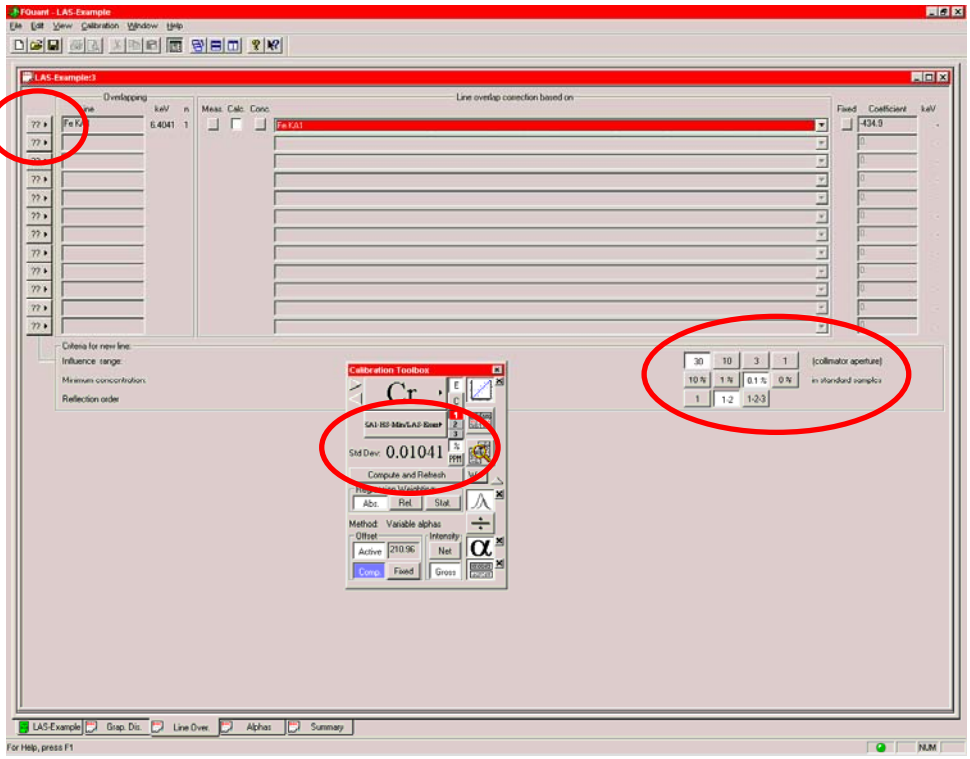
Select 0.1% in standard samples as minimum concentration.

Select 1-2 for Reflection Order.

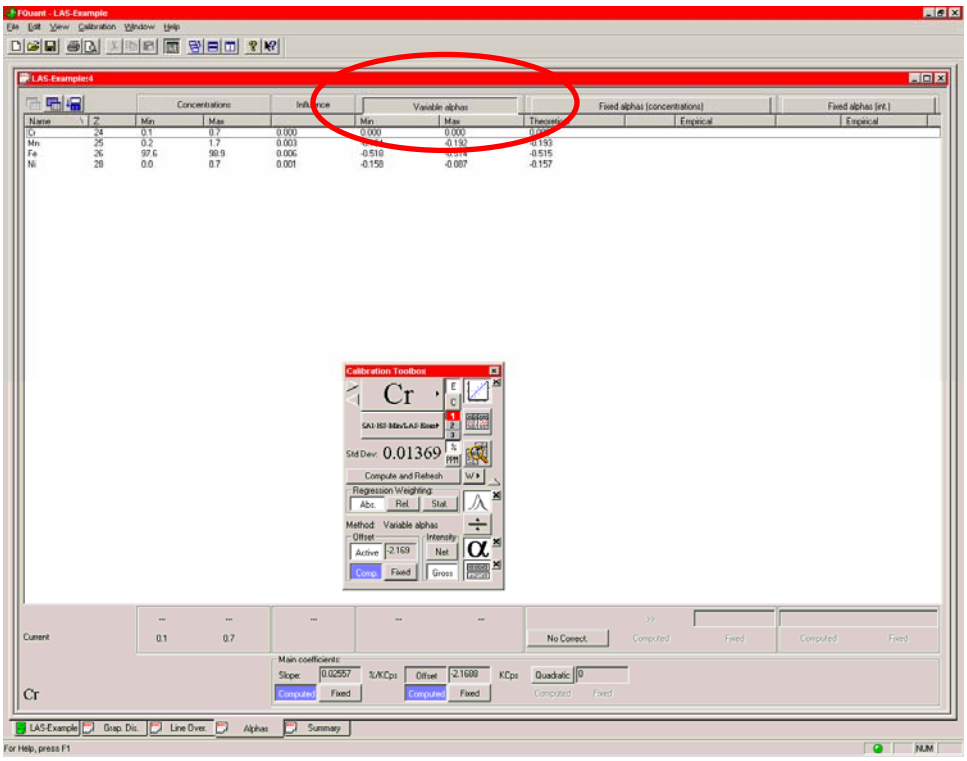
Press the first “???” button to have SPECTRA^{plus} search for line overlaps on this line.

Change the range to 30 and press the “???” button again.

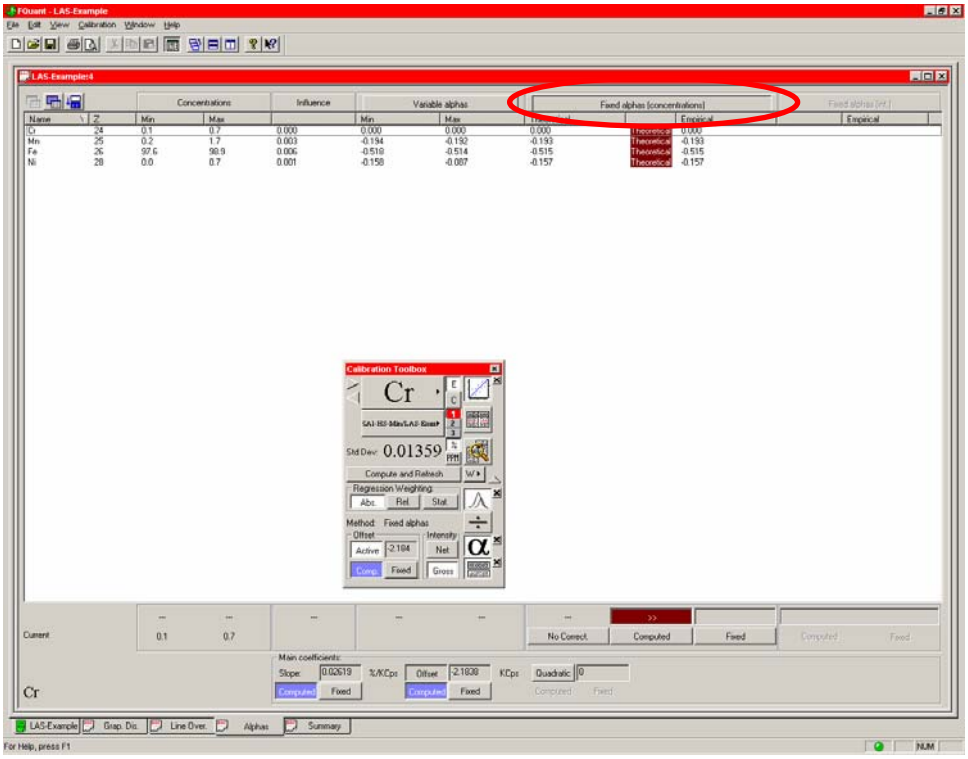
Select CALC and observe the Standard Deviation in the Toolbox.



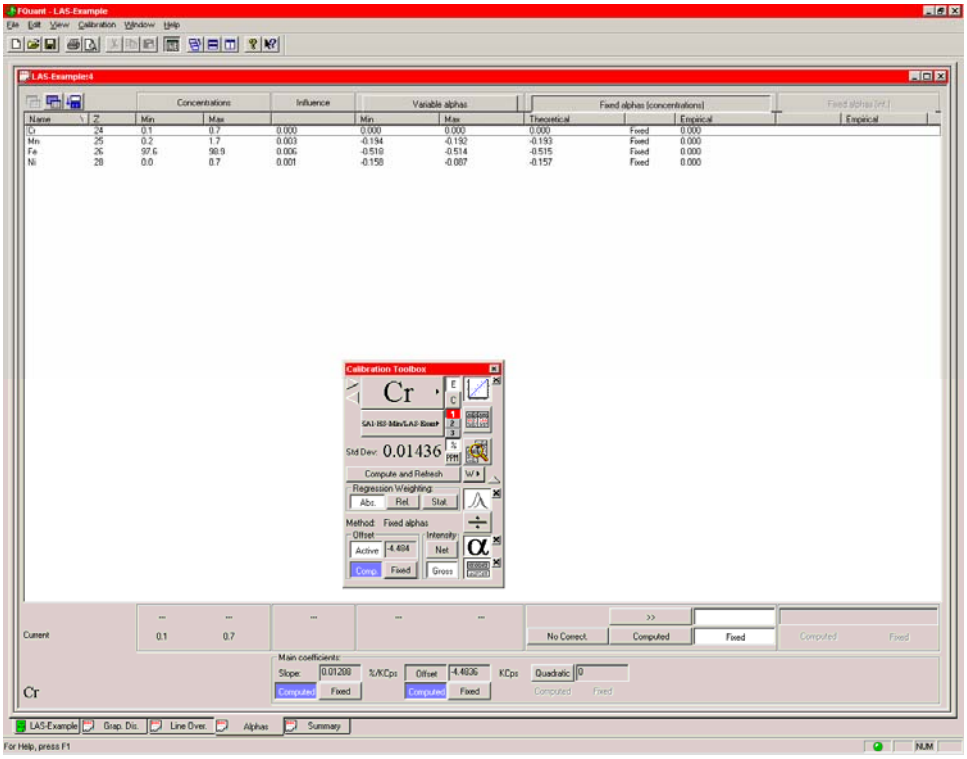
Select the **Alphas** tab. By default, VARIABLE alphas are selected (the best option).



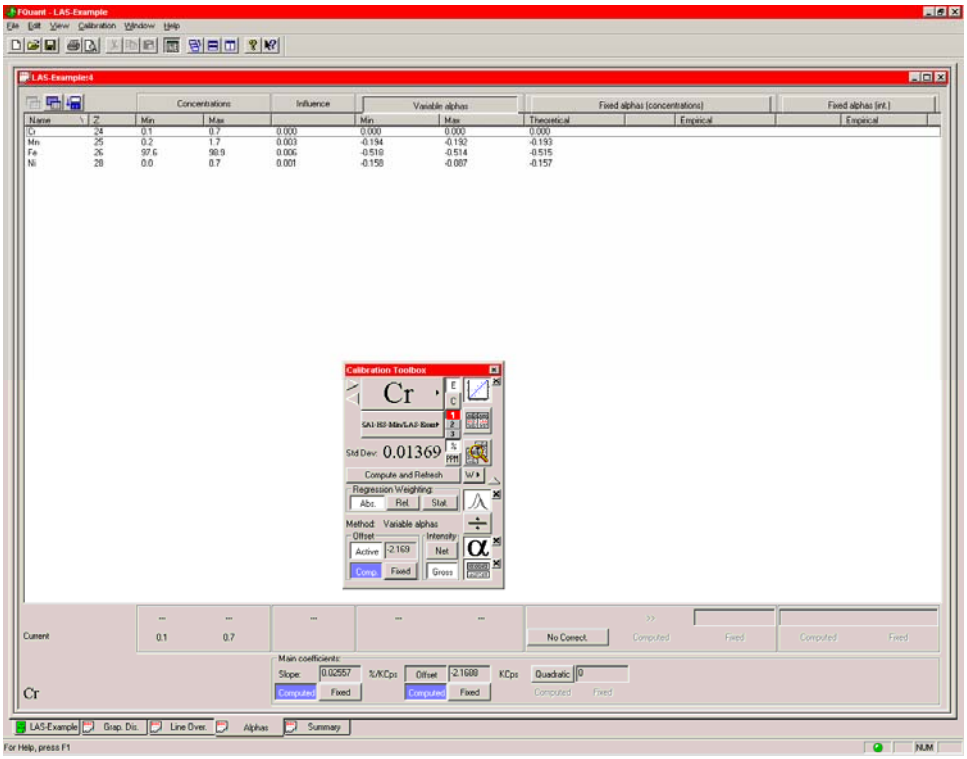
Change from variable to fixed alphas (theoretical) by pressing the **Fixed Alphas** button.



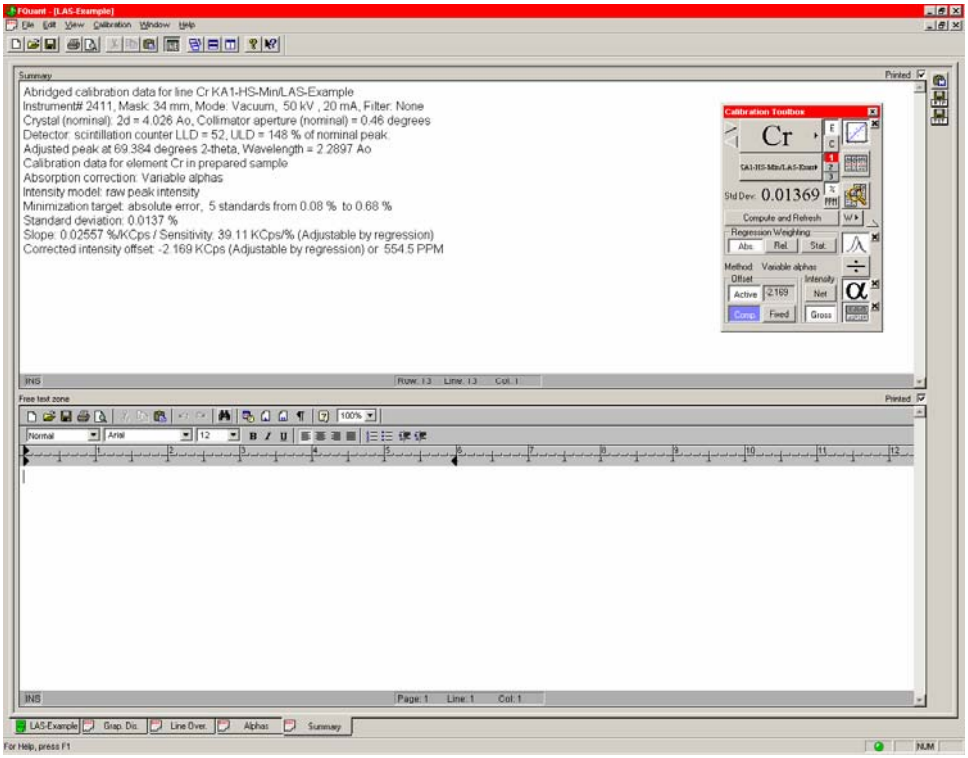
Select No Correction.



Select Variable Alphas.

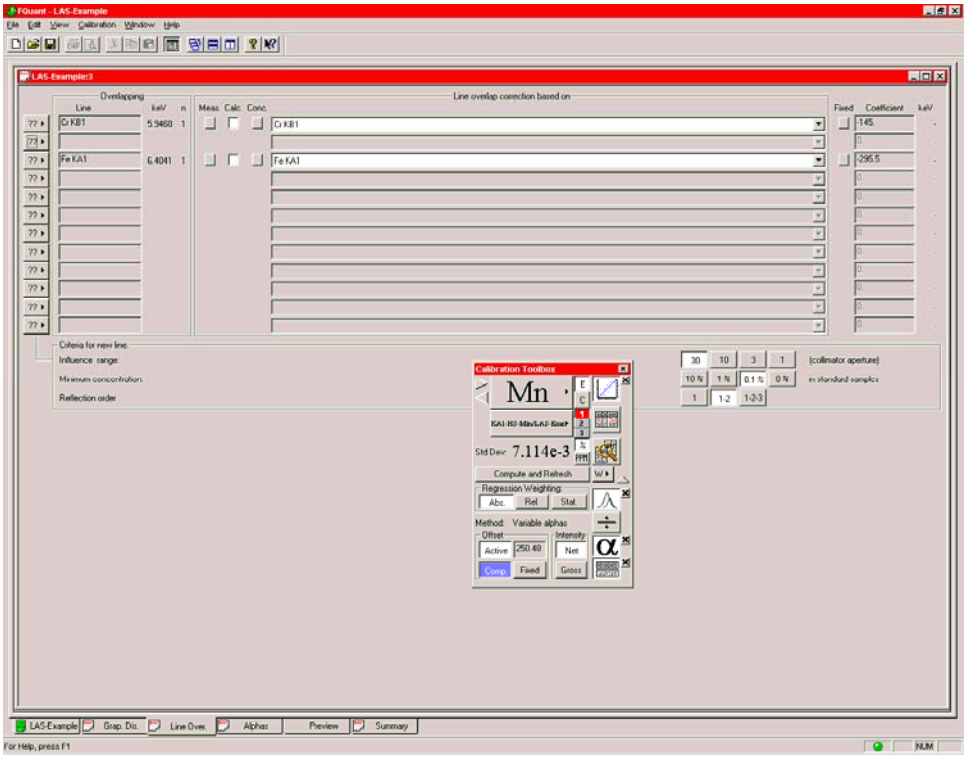


Select the **Summary** tab.

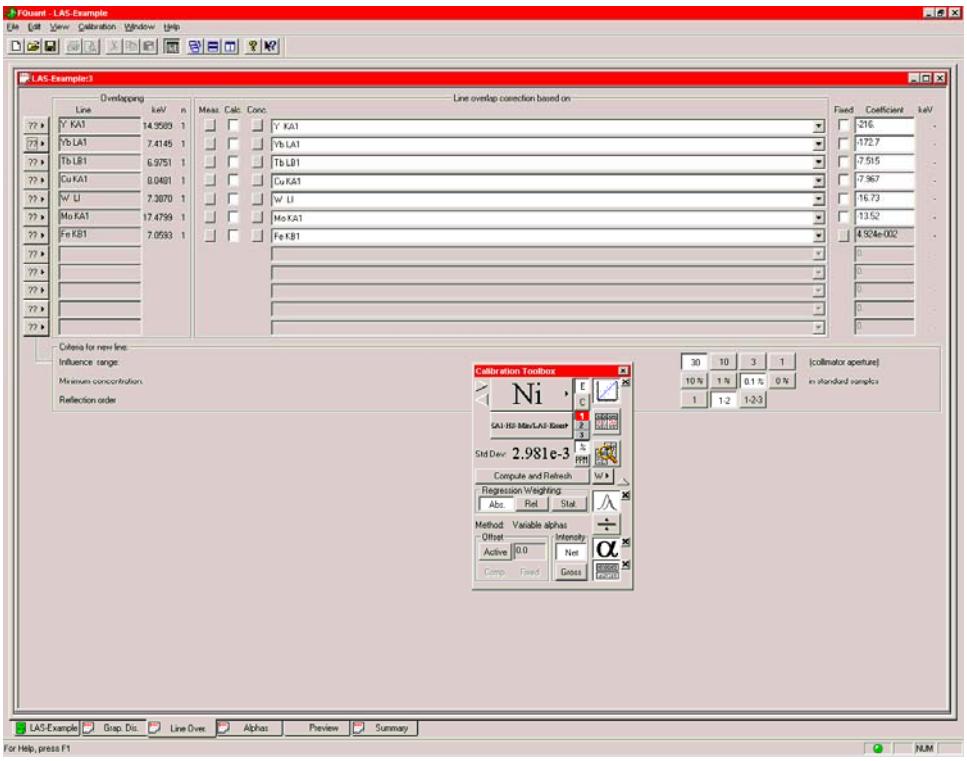


Select **Print Preview** and print the documentation for this line.

Select **Mn** from the Toolbox Element button and perform the same steps as for Cr.



Select **Ni** from the Toolbox element button and perform the same steps as for Cr.



Save the calibration.

3.11 Measuring the Drift Correction

Select the **Loader** from the Desktop folder.

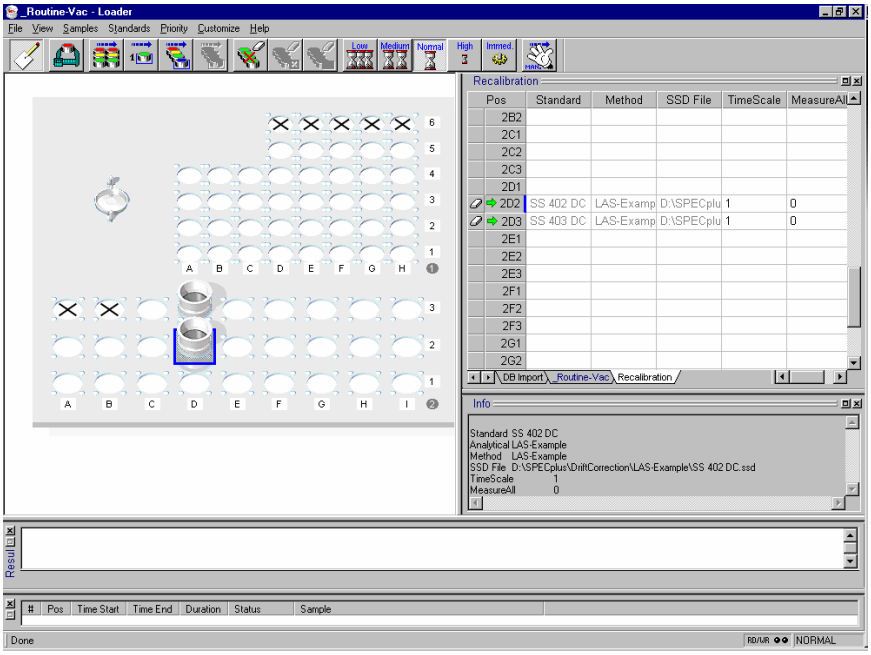
Select an available position on the Loader grid, where the drift correction samples will be placed.

Press the **Drift Correction** button, or select Standards→Drift correction.

Select “**LAS-Example**”.

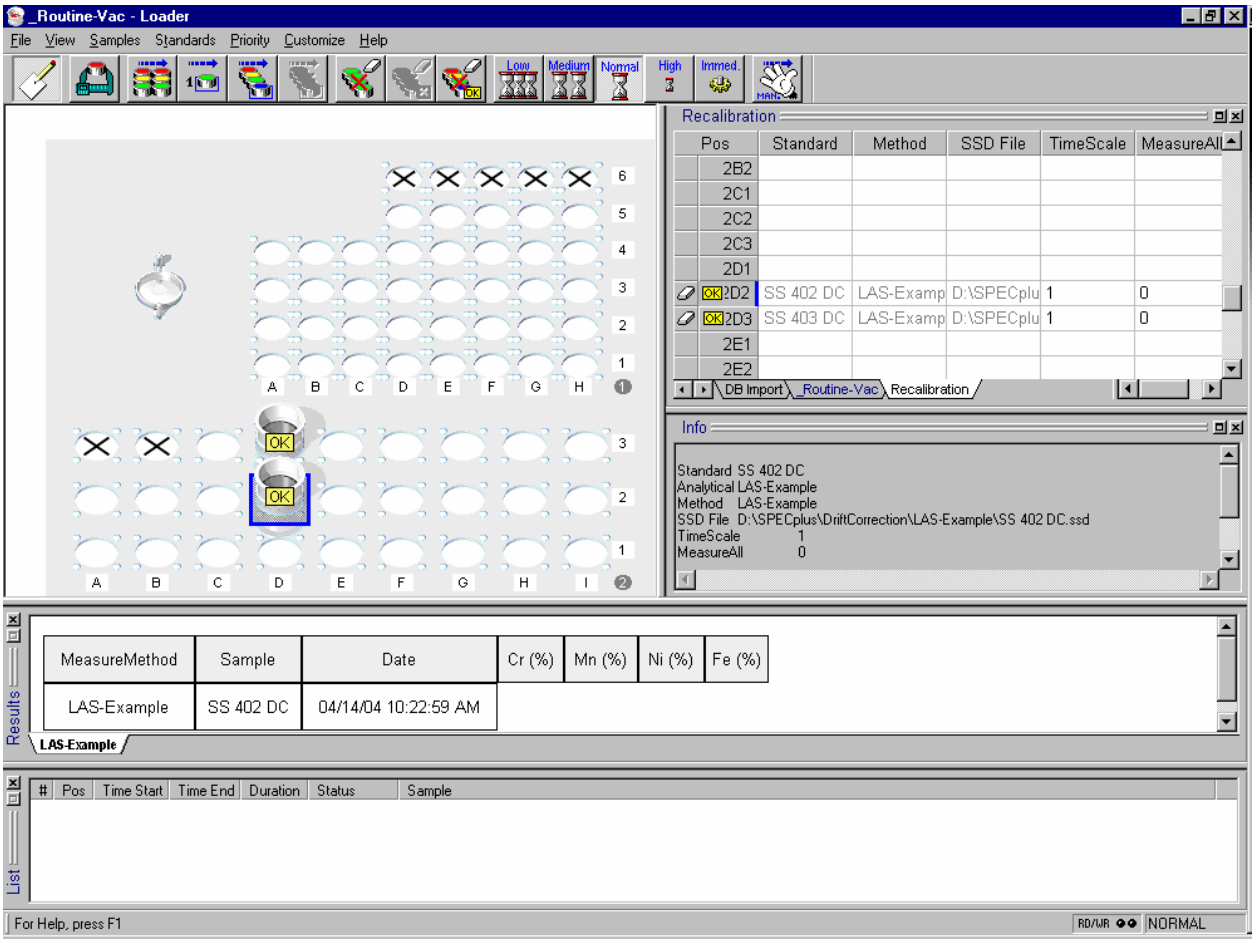
The designated drift correction samples are shown in the list.

Select **Import** to import the shown samples onto the Loader.



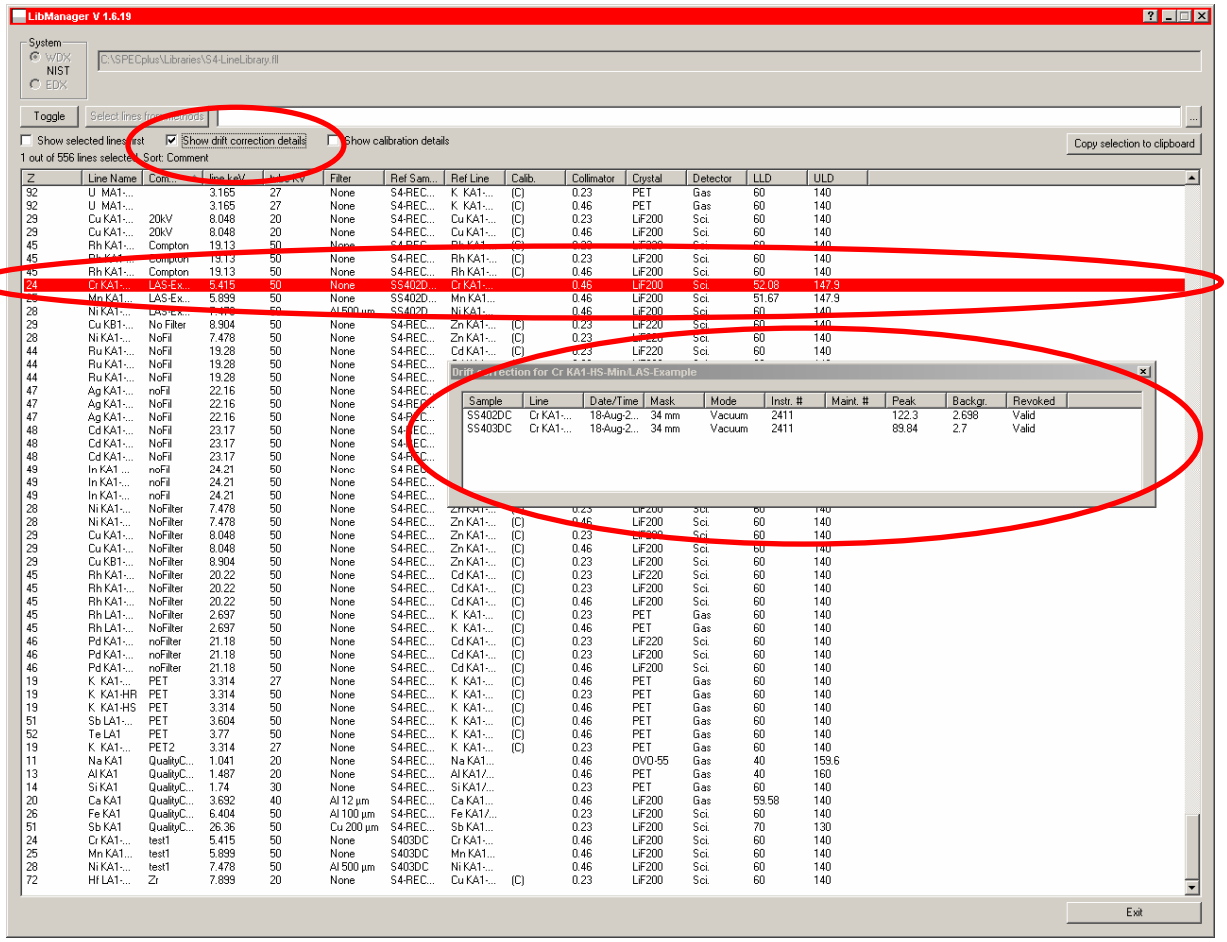
Press the **Send All Samples** button.

Wait for all samples to be measured. An OK flag indicates that the drift correction was stored. No concentrations will be displayed for drift corrected samples.



Select the **Libmanager** application from the SPECPLUS folder on your Desktop.

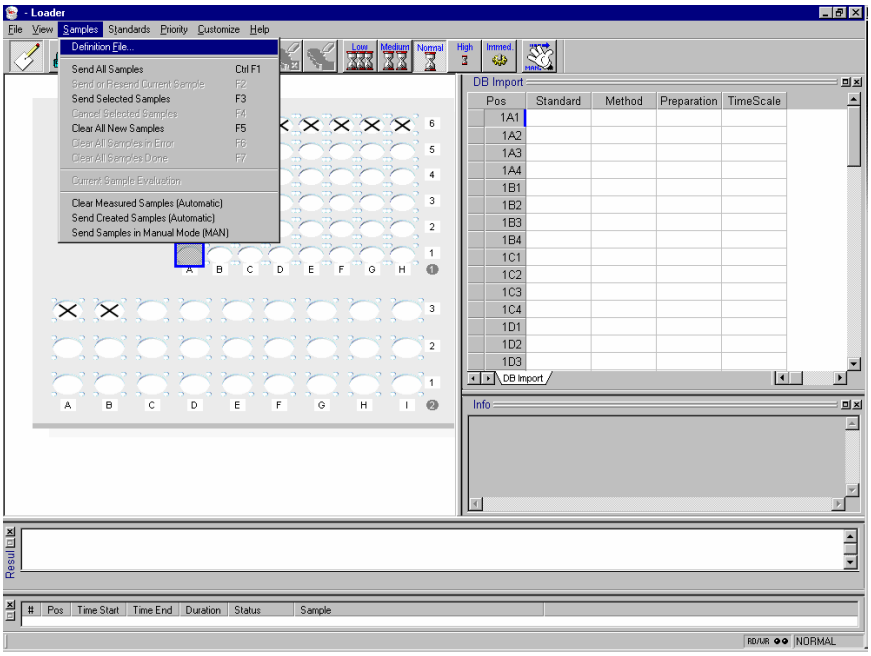
Sort for Extension name and click on a LAS –example line. Inspect the drift correction data by checking DC data. Validate that an entry was created for the measurement.



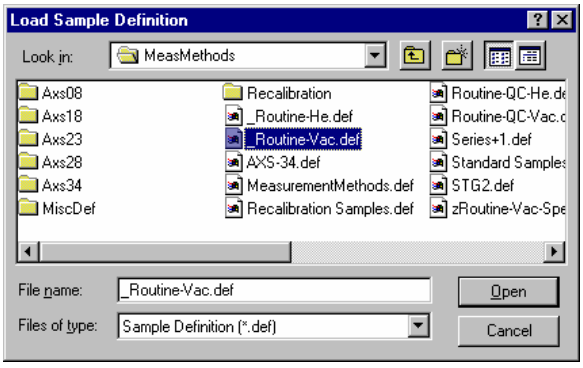
3.12 Measuring Sample as Unknown

Select **Loader**.

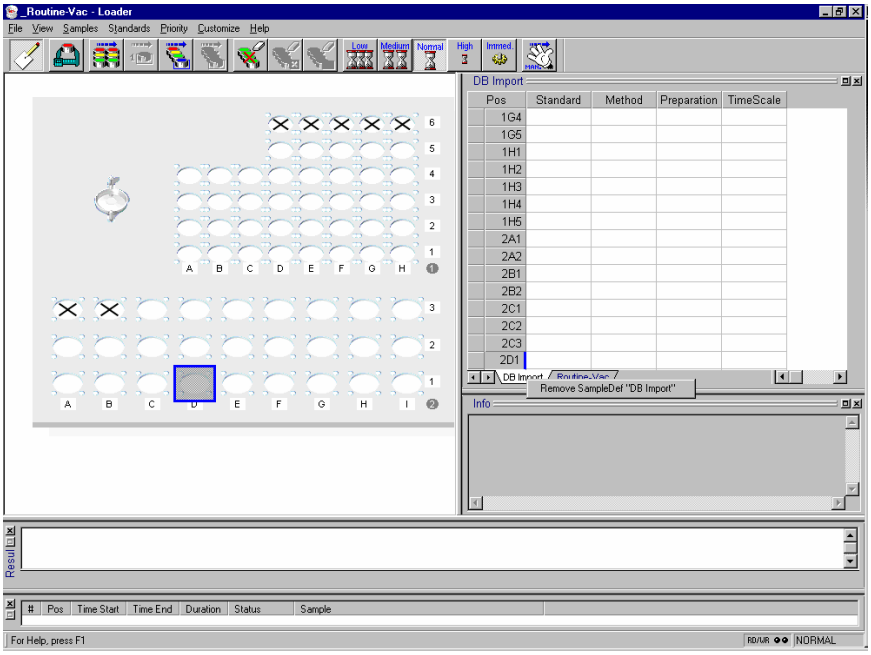
Select **Samples→Definition File**.



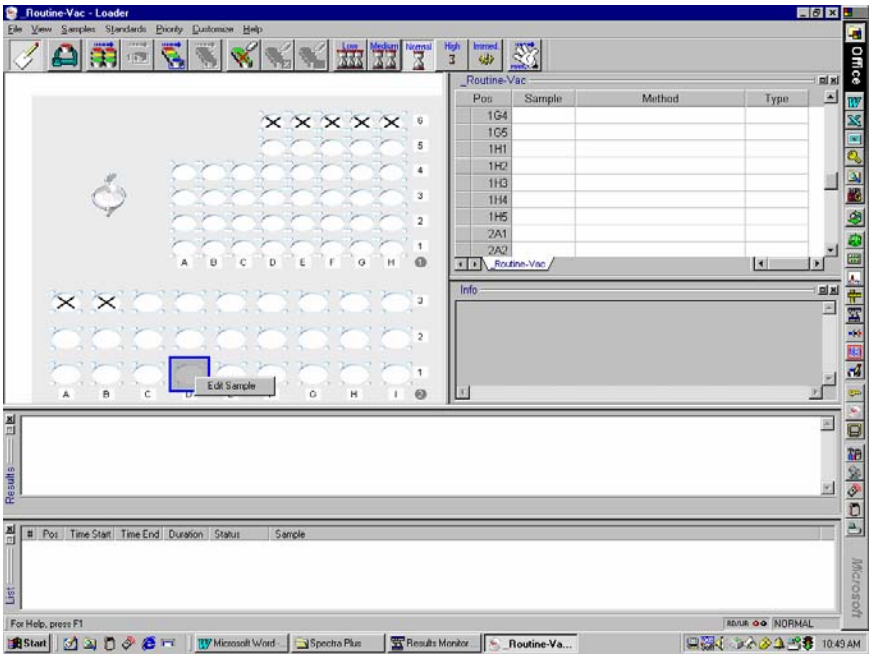
Select "**Routine-vac.def**".



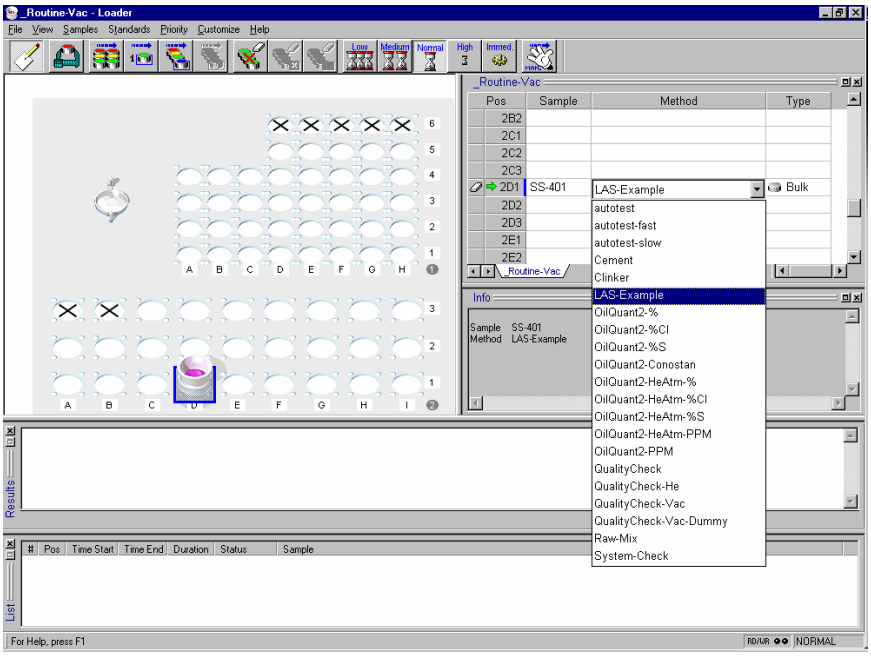
Right-click on any tab of the Sample Edit window to remove the tab.



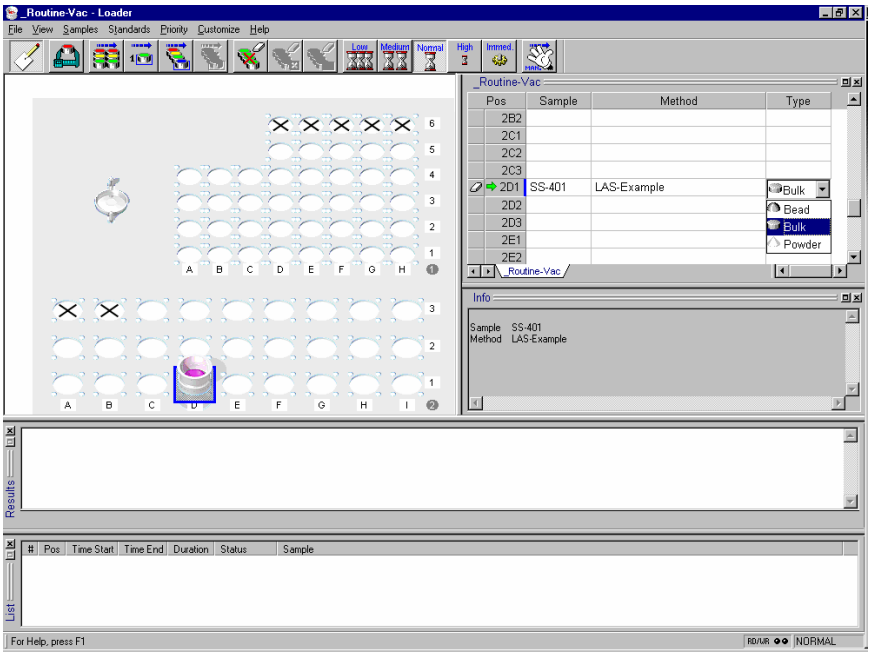
Right-click on the position where you placed a sample and select **Edit**.



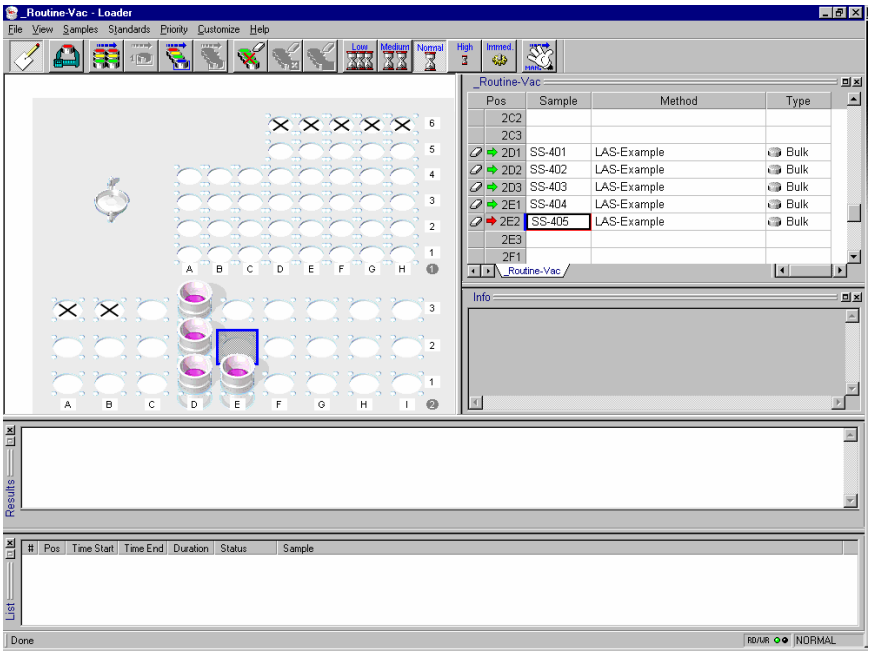
Enter the sample name and select the “LAS-Example” Method from the drop-down menu under “Method”.



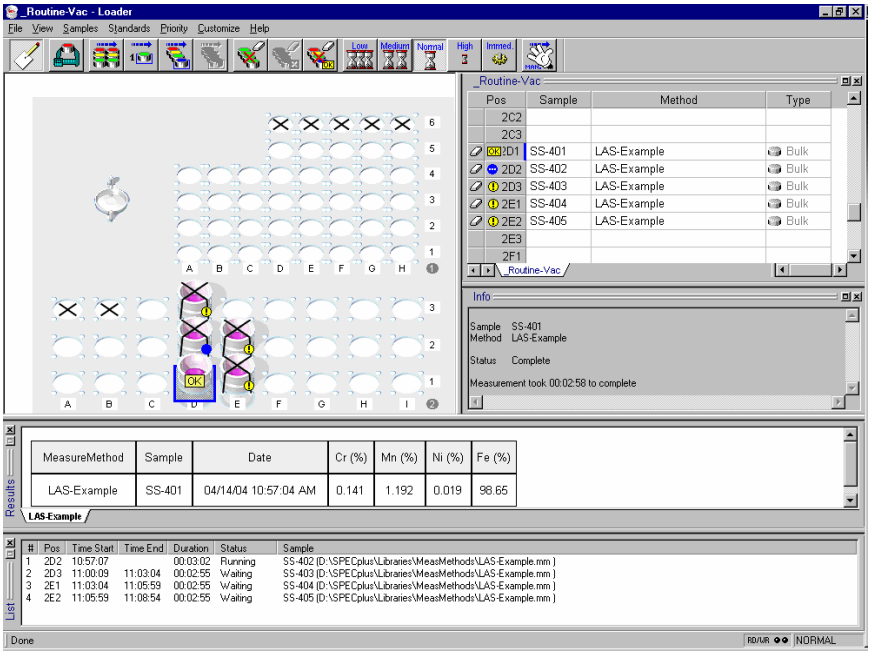
If a Sample Type column is shown, select **Bulk**.



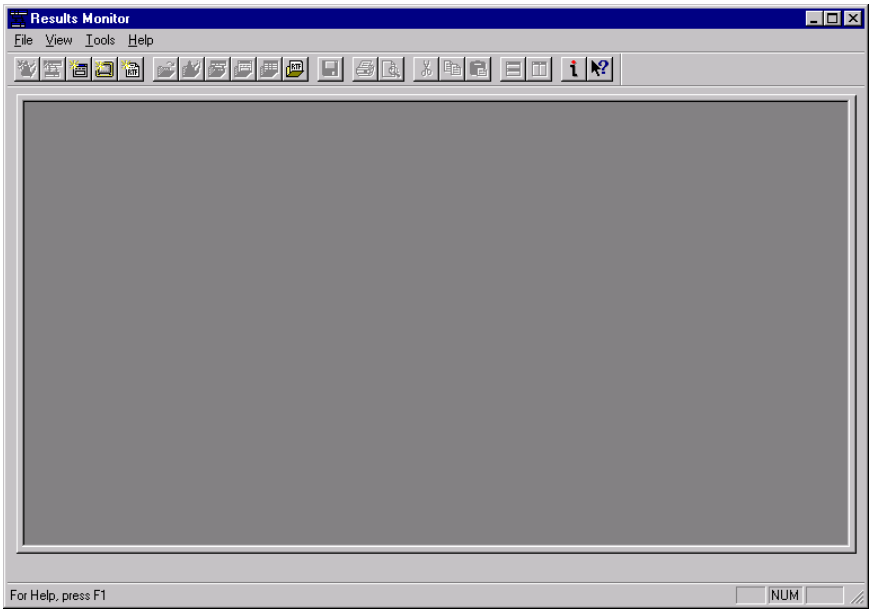
Repeat procedure until you have entered all 5 samples.



Send All Samples and look at the results.

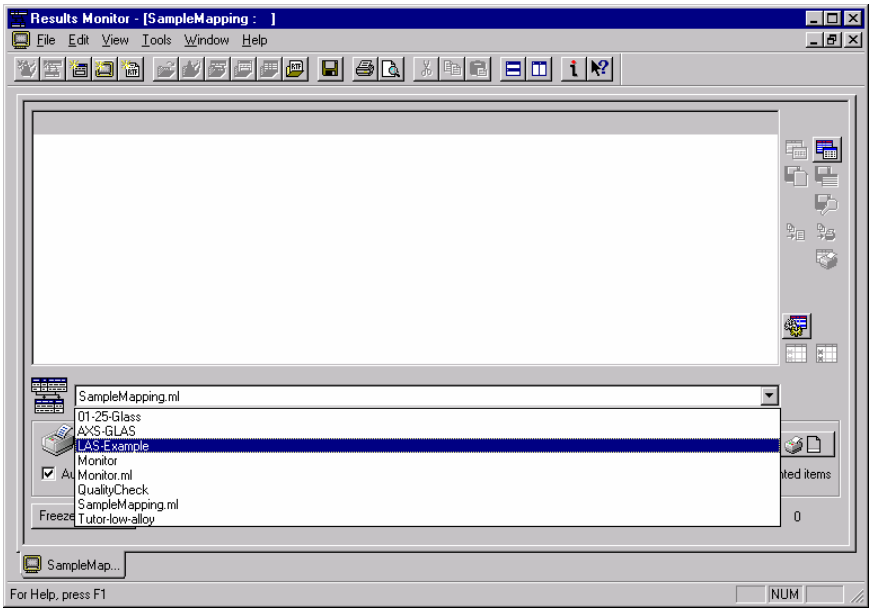


Select the **Results Monitor**.



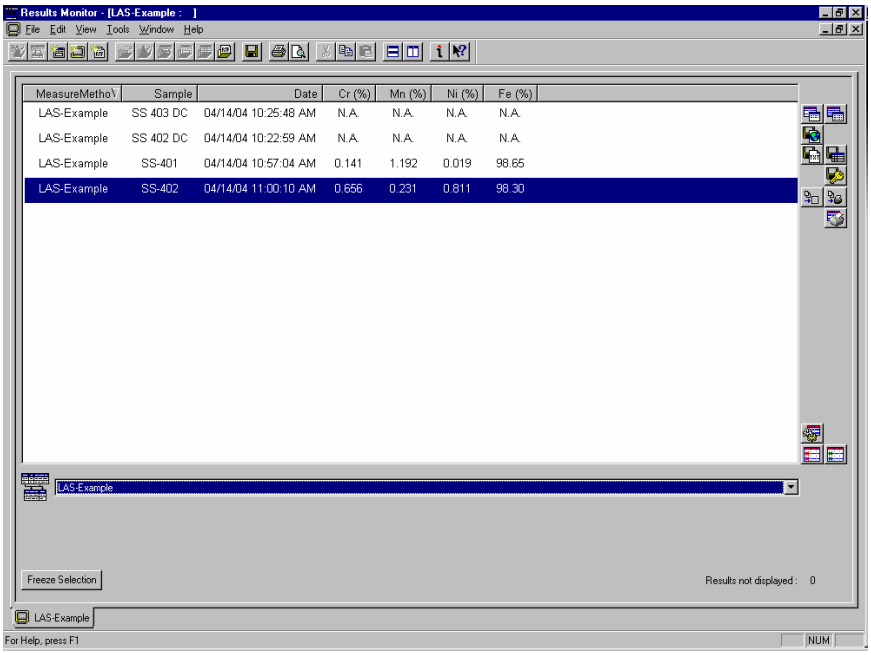
Select **File→New Monitoring View**.

From the Mapping List, select the “LAS-EXAMPLE” by clicking on the down arrow.



Click on the table with the **Gears** icon to update the view.

In this view (Monitoring), SPECTRA^{plus} will display the last *n* results (as set in the Monitoring section of the Results program) for the selected format.



Click on a column header to change the sort order, e.g., for sorting by date.