Probe for EPMA v. 9.4.1

User's Guide to Getting Started

Xtreme Edition

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Probe Software, Inc.

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Conventions Used in this Guide

The screenshots used in this guide are consistent with Probe For EPMA version 9.4.1, released April 2013. Some screenshots might display older version information, but the contents of these dialogs and windows have not been changed since then.

The following conventions are used in this document; **Menu Commands** and **Dialog Box (Windows) Names and buttons** are bold-faced whenever they occur in the text. *Dialog Box Options* are italicized and FILE NAMES are capitalized.
Several tips for saving time/steps include:

Context sensitive HELP is available in any window by pressing the F1 key.
Pressing <Enter> (or <Return> <↵ > on international keyboards) on the keyboard is identical to clicking the **OK** button.
Pressing the <Esc> key on the keyboard is identical to clicking the **Cancel** command.
To select a range of items in *Multi-Select* list boxes, click on the first item, move to the last and hold the <Shift> key down while clicking on the last item.
To select individual items in *Multi-Select* list boxes, hold down the <Ctrl> key down while clicking on the item.
De-select items in *Multi-Select* list boxes by holding the <Ctrl> key down and clicking the item.

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Creating the Default Standard Database File

PROBE FOR EPMA requires a database of microprobe standards for use in quantitative analysis. This standard database can store up to 32768 standards each with up to 72 elements per standard. All standard information is stored in a file designated STANDARD.MDB. MDB is an abbreviation for Microsoft DataBase and represents a Microsoft Access v. 3.5 database file. In addition to the default standard database, four other standard databases are supplied as ASCII files and as MDB files. These are:

DHZ.MDB	Deer, Howie and Zussman
ORE.MDB	Dana's Mineralogy (Sulfides)
SRM.MDB	NIST standard reference alloys and glasses
AMCSD.MDB	American Mineralogist Crystal Structures

The DHZ.MDB is a database of all of the analyses listed in the first edition of "Rock Forming Minerals" by Deer, Howie and Zussman.

The ORE.MDB is a database composed of sulfide minerals from Dana's Mineralogy entered in ideal formulas.

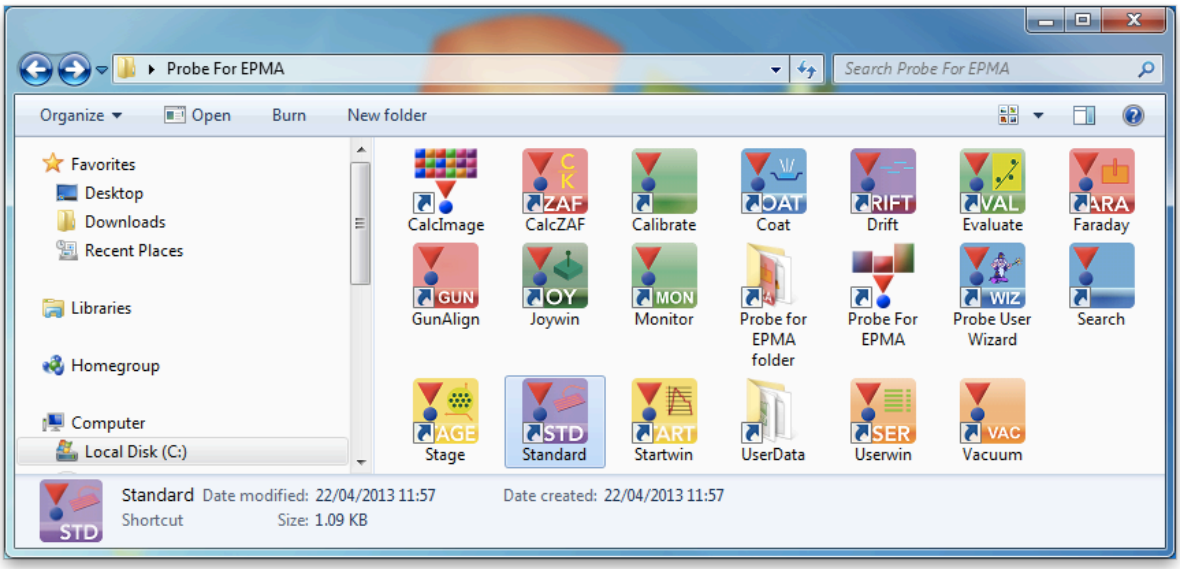
The SRM.MDB is a database of SRM (Standard Reference Materials) alloys and glasses from the NIST SRM catalog.

The AMCSD.MDB is the American Mineralogist Crystal Structure Database, which contains over 9500 compositions based on formula stoichiometries.

All of these database files can be used for reference and compositional matching purposes through the Standard menu.

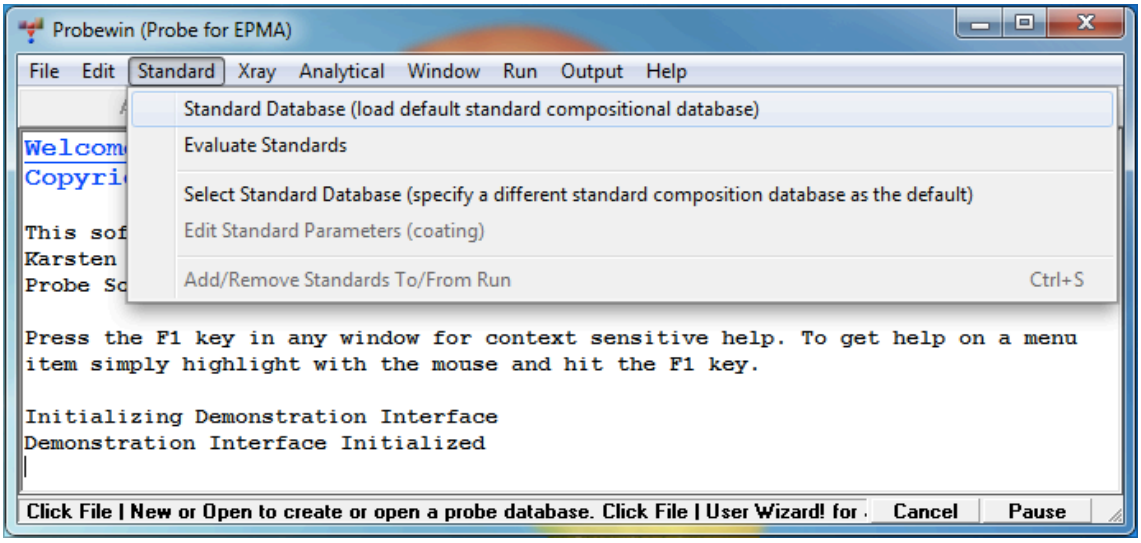
The following procedure illustrates how to create a new default standard database and enter standard compositions into it.

Open the STANDARD application. If available, double click on the yellow Probe for EPMA software folder on the desktop. Then double click on the **Standard** icon:

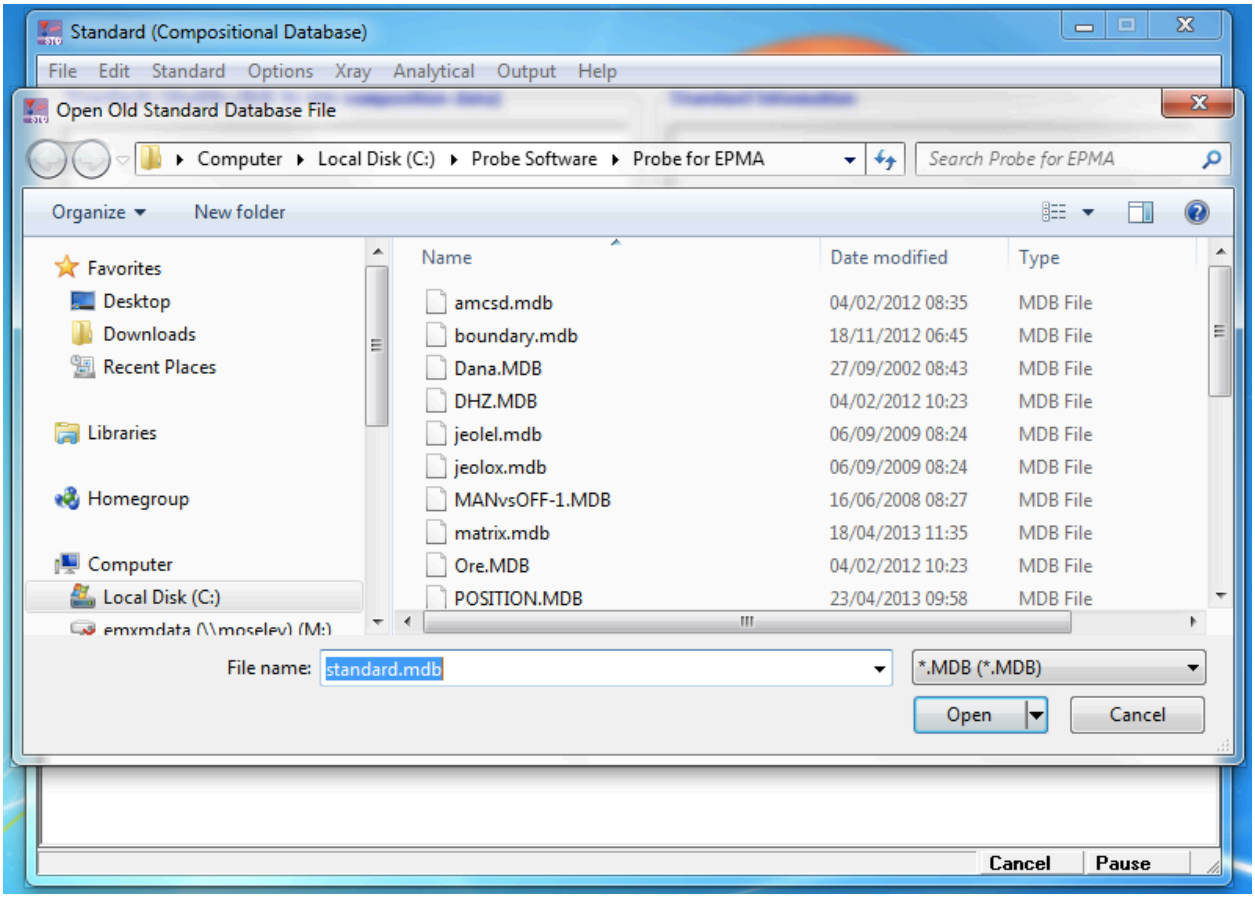


Alternatively, select **Standard** from the Probe Software group in the Windows Start Menu, or locate and double click on the STANDARD application in the Probe for EPMA application directory, which is usually C:\Probe Software\Probe for EPMA under Windows Vista and Windows 7, or C:\Program Files\Probe Software\Probe for EPMA for older operating systems.

STANDARD can also be launched by selecting **Standard Database** in the **Standard** menu of Probe for EPMA:

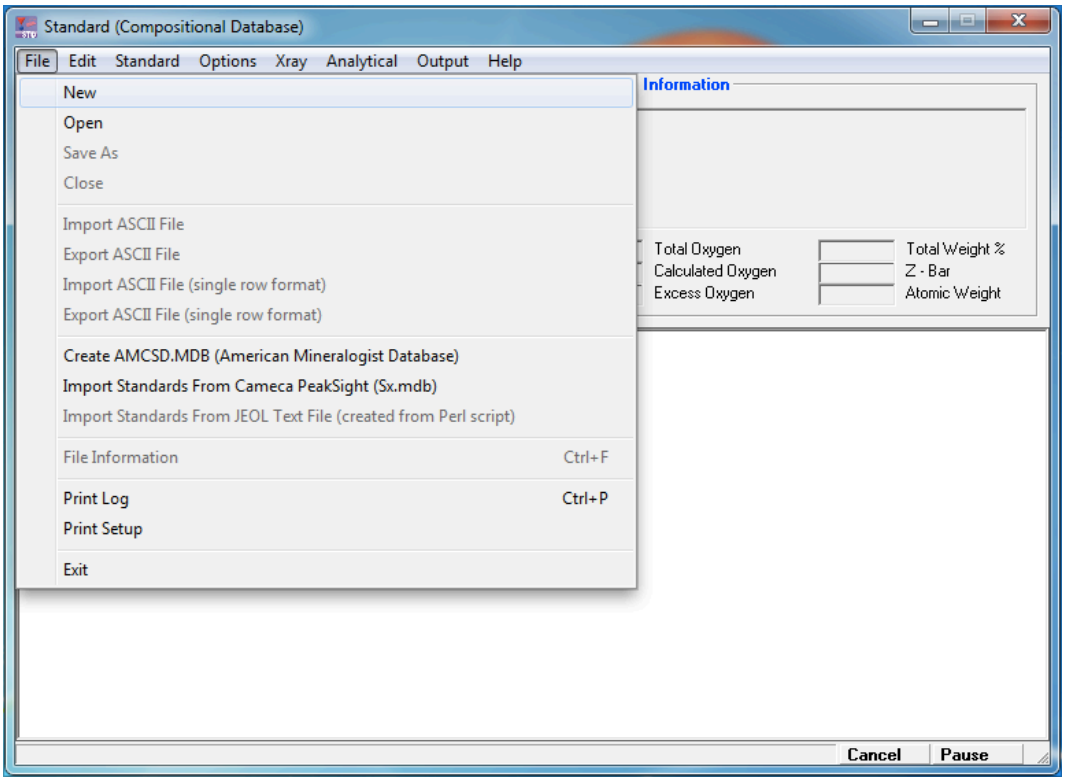


This action launches the STANDARD (Compositional Database) program and opens the **Open Old Standard Database File** dialog box.

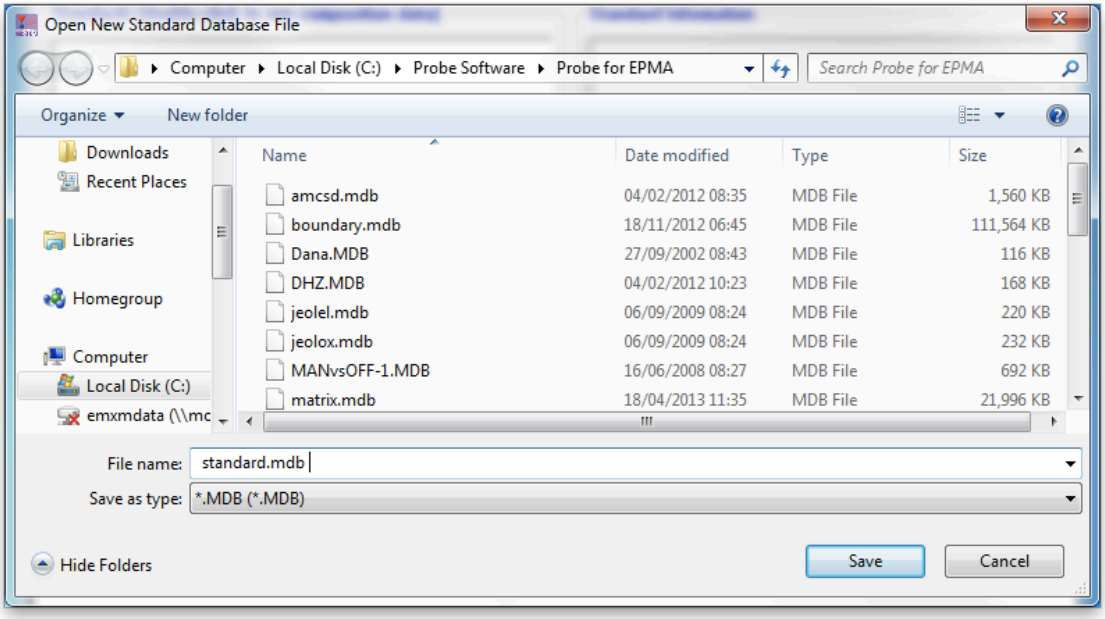


To create a new standard database, click on the **Cancel** button to close the **Open Old Standard Database File** dialog box.

Select **File** from the menu bar and then click on **New** from the menu.

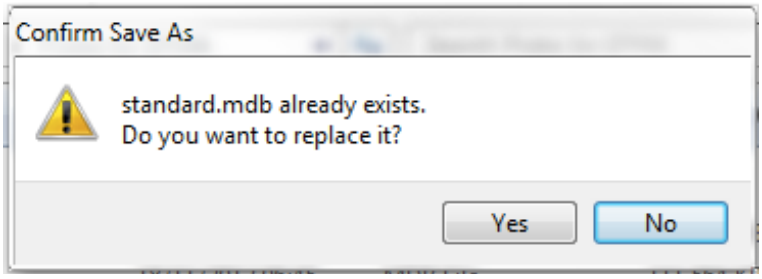


This opens the **Open New Standard Database File** dialog box.



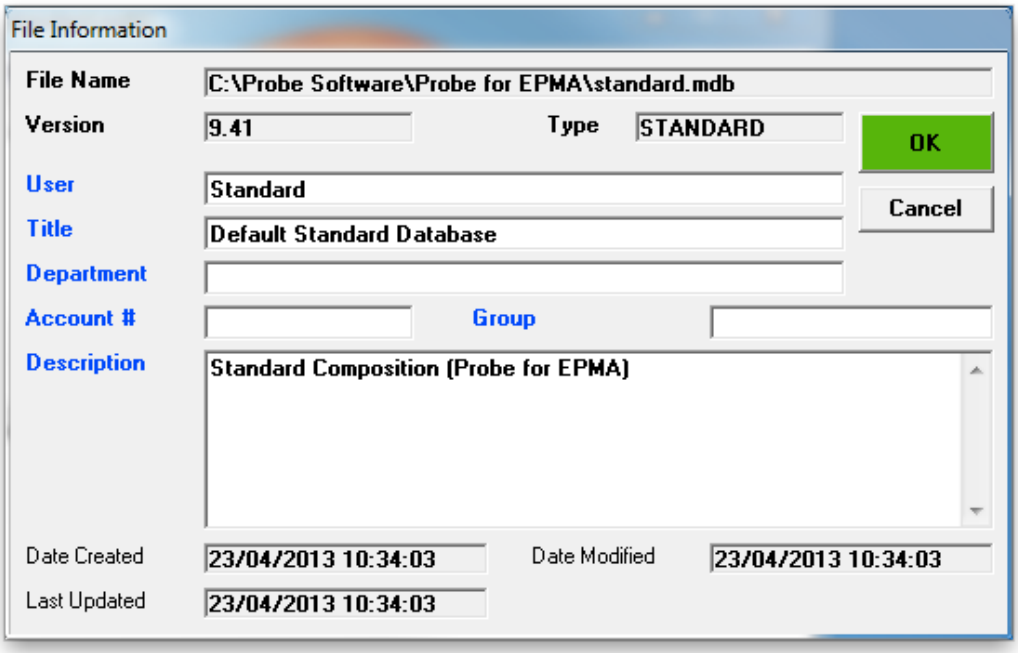
Click the **Save** button to open a new default standard database (STANDARD.MDB).

The **Confirm Save As** window appears. Click **Yes**.

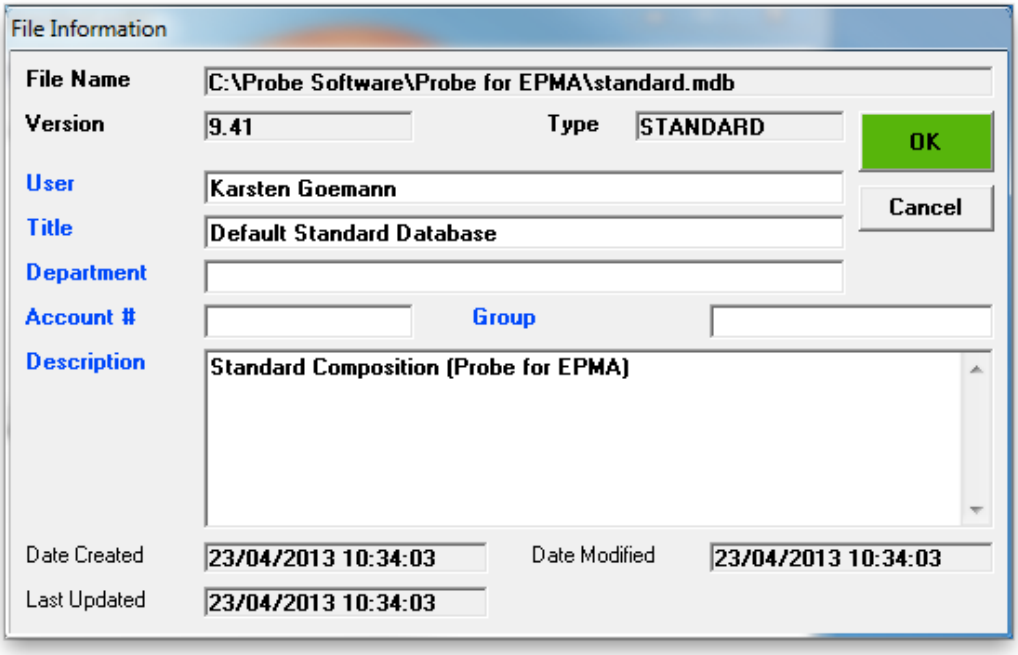


The default standard database supplied with the installation is J. Donovan's standard listing. The user has a different set of standards typically, so the choice is to overwrite the supplied database. Click the **Yes** button to confirm overwriting the existing default database. Note: the supplied demonstration files JEOLEL.MDB and JEOLOX.MDB will no longer be usable after this operation.

The **File Information** window opens.

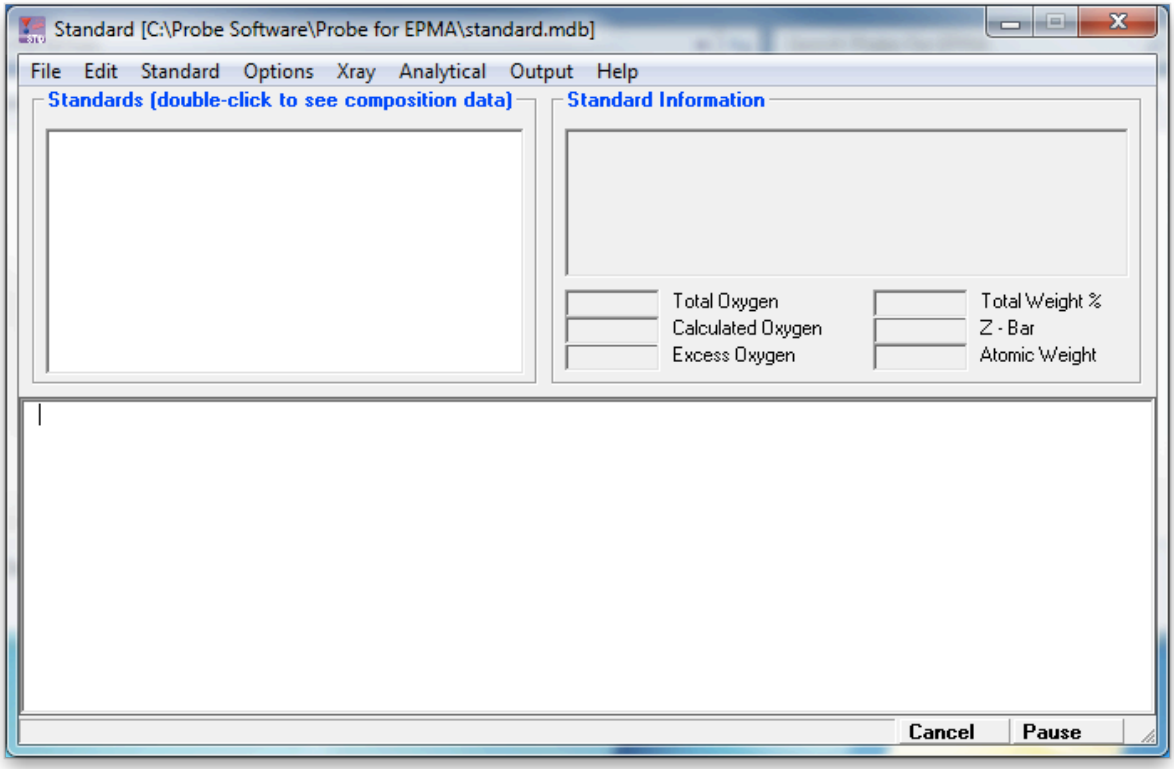


Enter the relevant information into the *User*, *Title*, and other *Description* text boxes shown in the **File Information** dialog box displayed below. Use the <tab> key to move between text boxes.

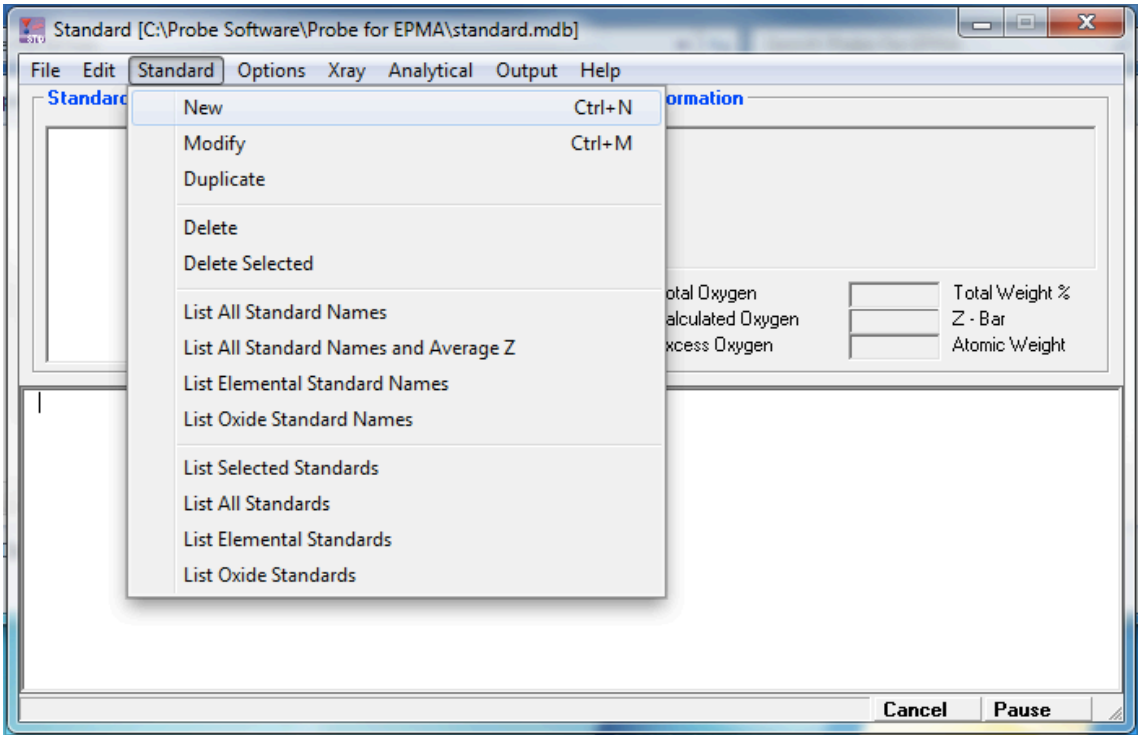


When finished, click the **OK** button.

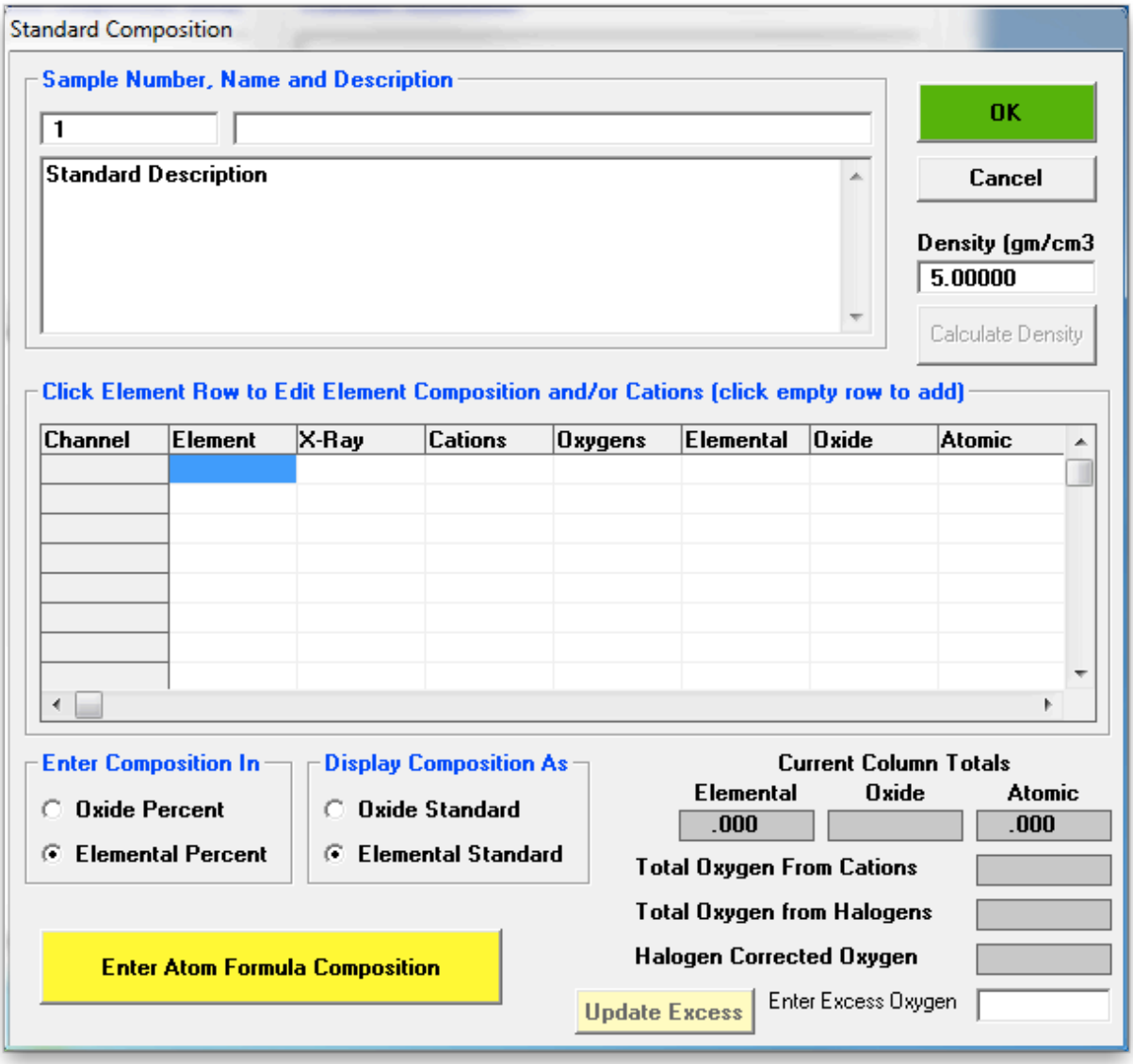
The user now has an empty database ready to accept standard composition data.



To enter standards into this database, select **Standard** from the menu bar and click on **New** from the menu.

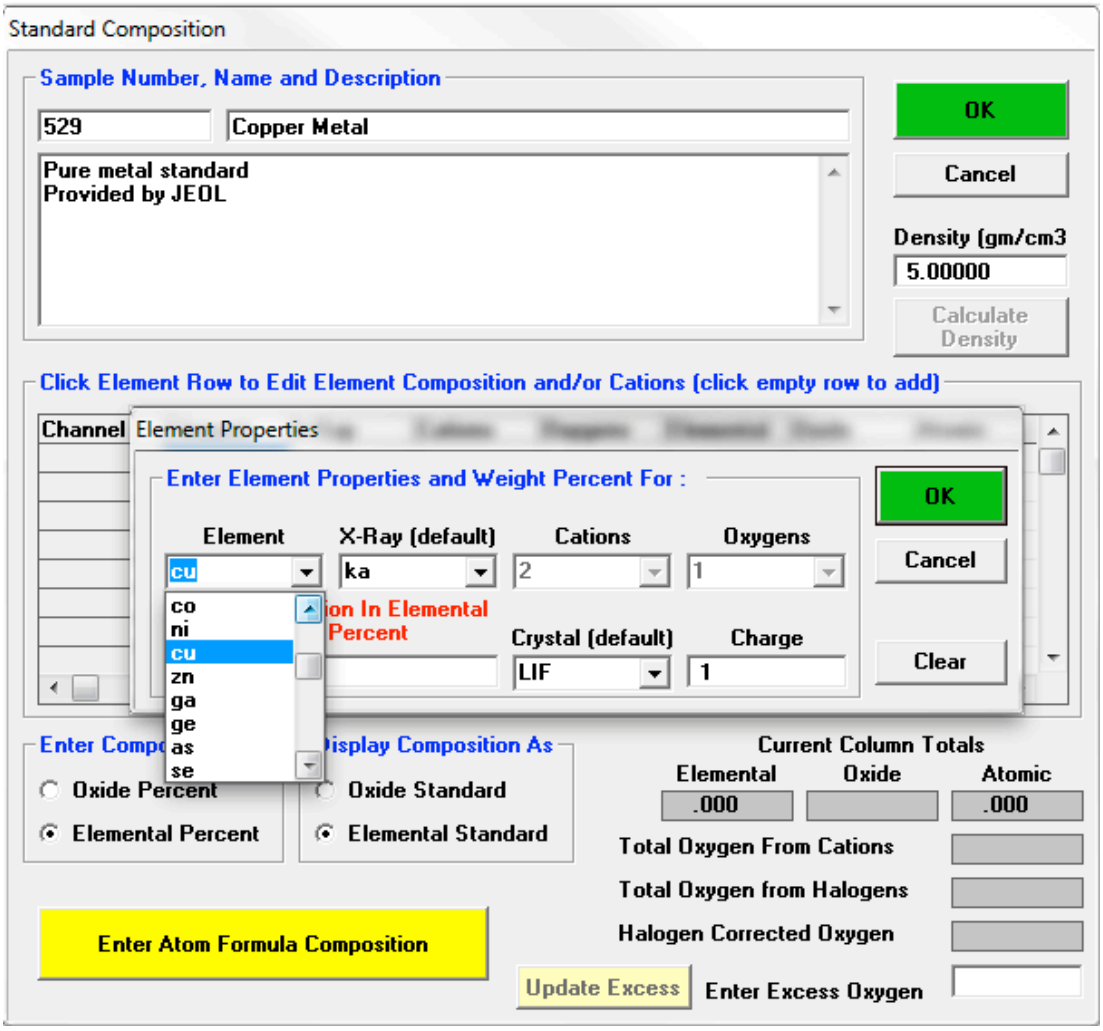


This action opens the **Standard Composition** dialog box. Type the appropriate *Sample Number*, *Standard Name*, and *Standard Description* into the text boxes. The software automatically loads the next available number by default. Choose standard numbers that will allow grouping of standards into various functional sets. Standard numbers may range from 1 to 32768, however to avoid conflict with the supplied NIST SRM, DHZ, and Dana ORE sample databases select numbers below 2000.

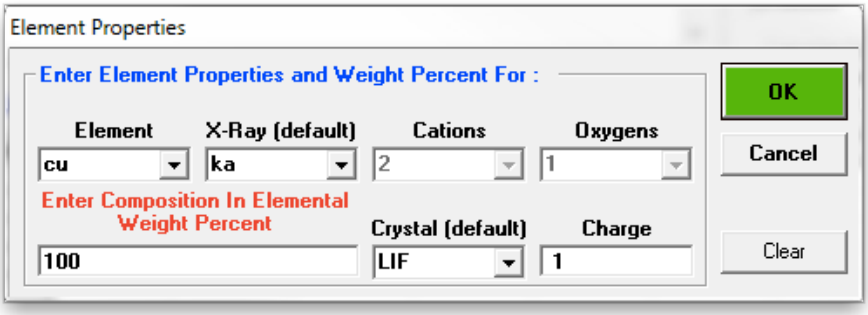


Click the *Elemental Percent* and *Elemental Standard* buttons under *Enter Composition In* and *Display Composition As* respectively, as necessary. All standard compositions are saved in the standard database as elemental concentrations. If oxygen is present in the standard then the user must enter oxygen as an element and its concentration into the standard entry. See the silicate example in this manual for details.

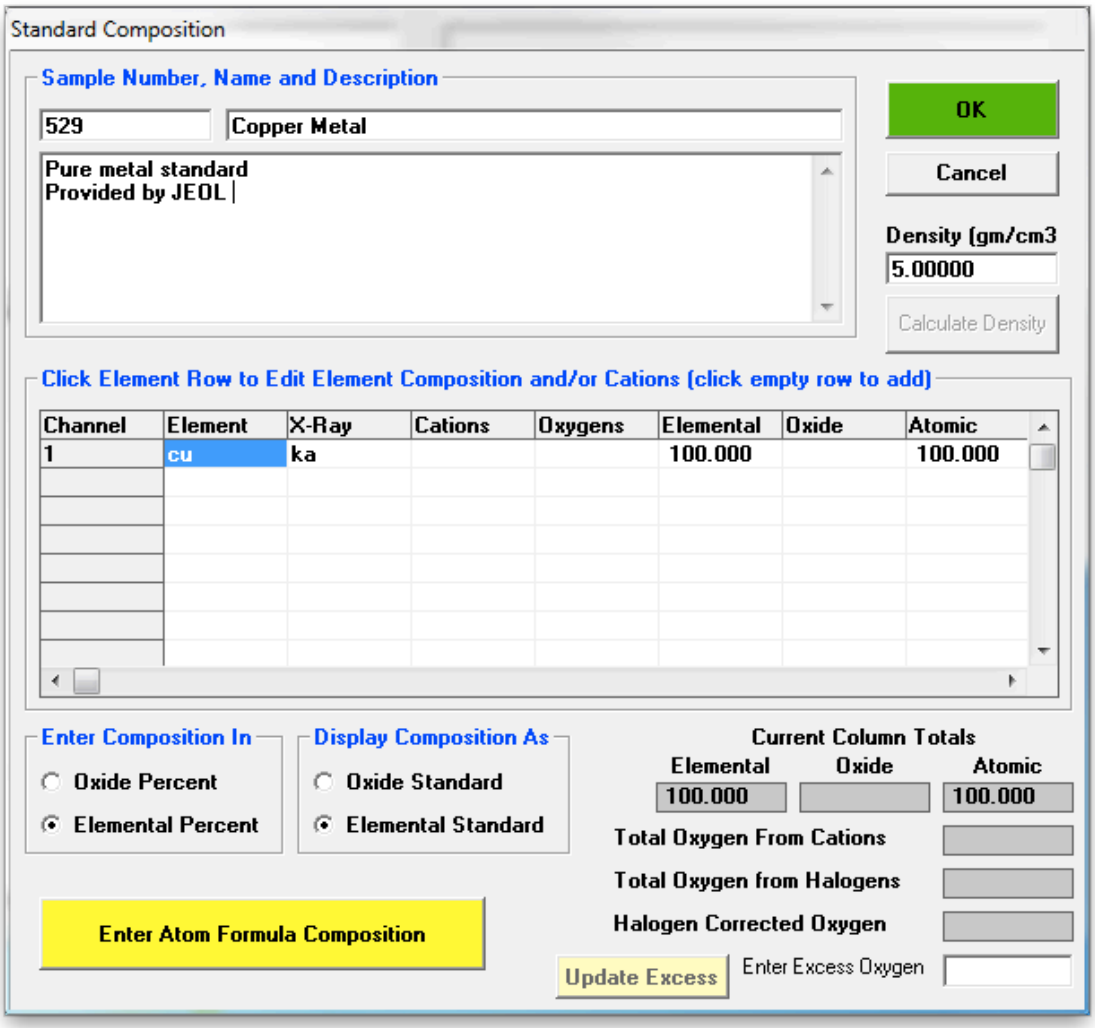
The first example will illustrate the entry of an elemental metal standard. Click on any empty row in the spreadsheet. This opens the **Element Properties** dialog box. In the *Element* field either type in the first element in the standard or use the drop-down list box to select the element symbol. Continue by choosing the correct *X-Ray* line, *Cations*, and *Oxygens*. The *X-Ray* line is used for modeling purposes only. When entering properties and concentrations for elements in elemental mode, the program grays out the *Cations* and *Oxygens* text boxes, as no editing of these text boxes is necessary.



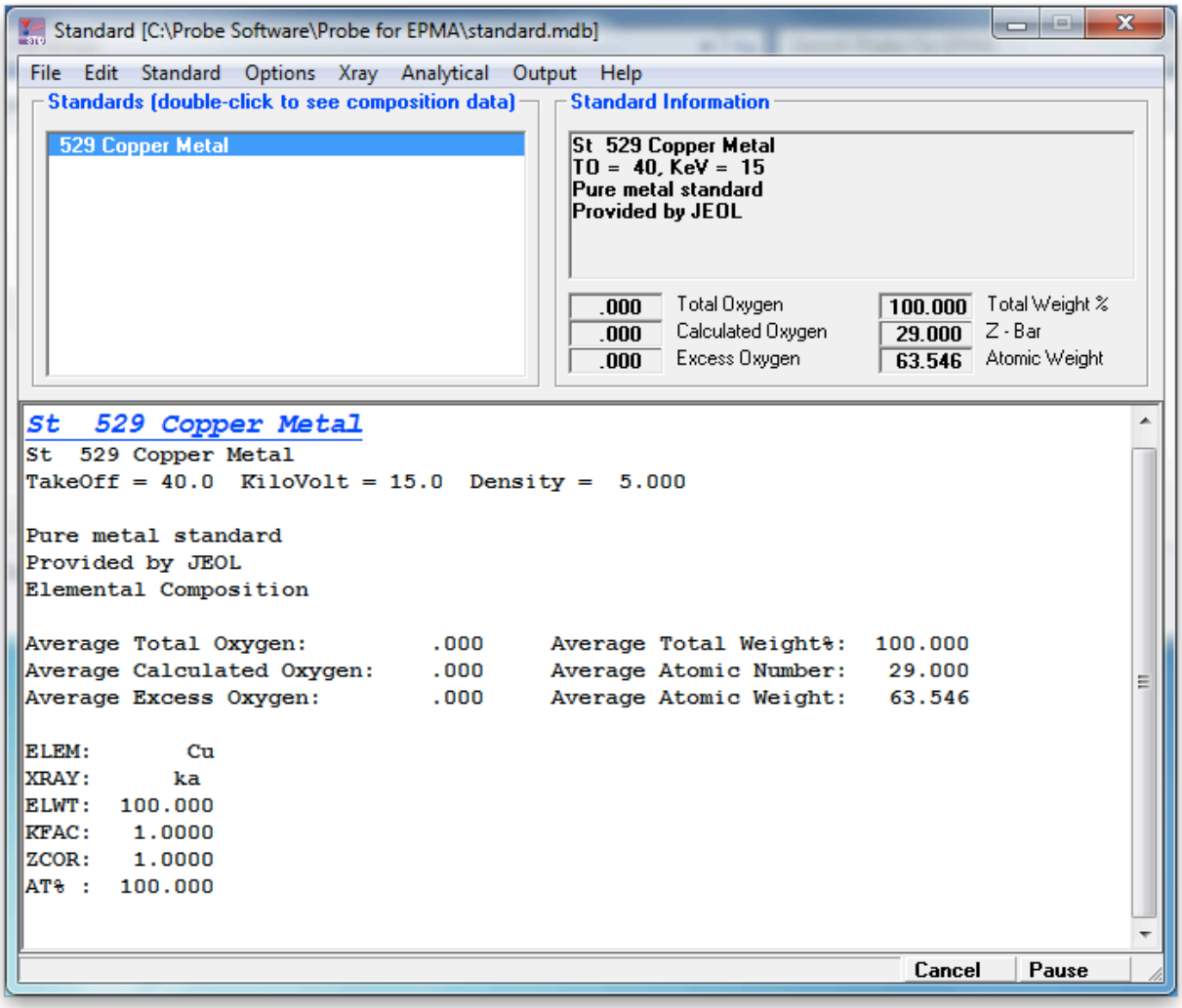
Enter the elemental weight percent for copper into the *Enter Composition In Elemental Weight Percent* text box. Finish by clicking the **OK** button of the **Element Properties** dialog box.



The program returns to the **Standard Composition** dialog box.

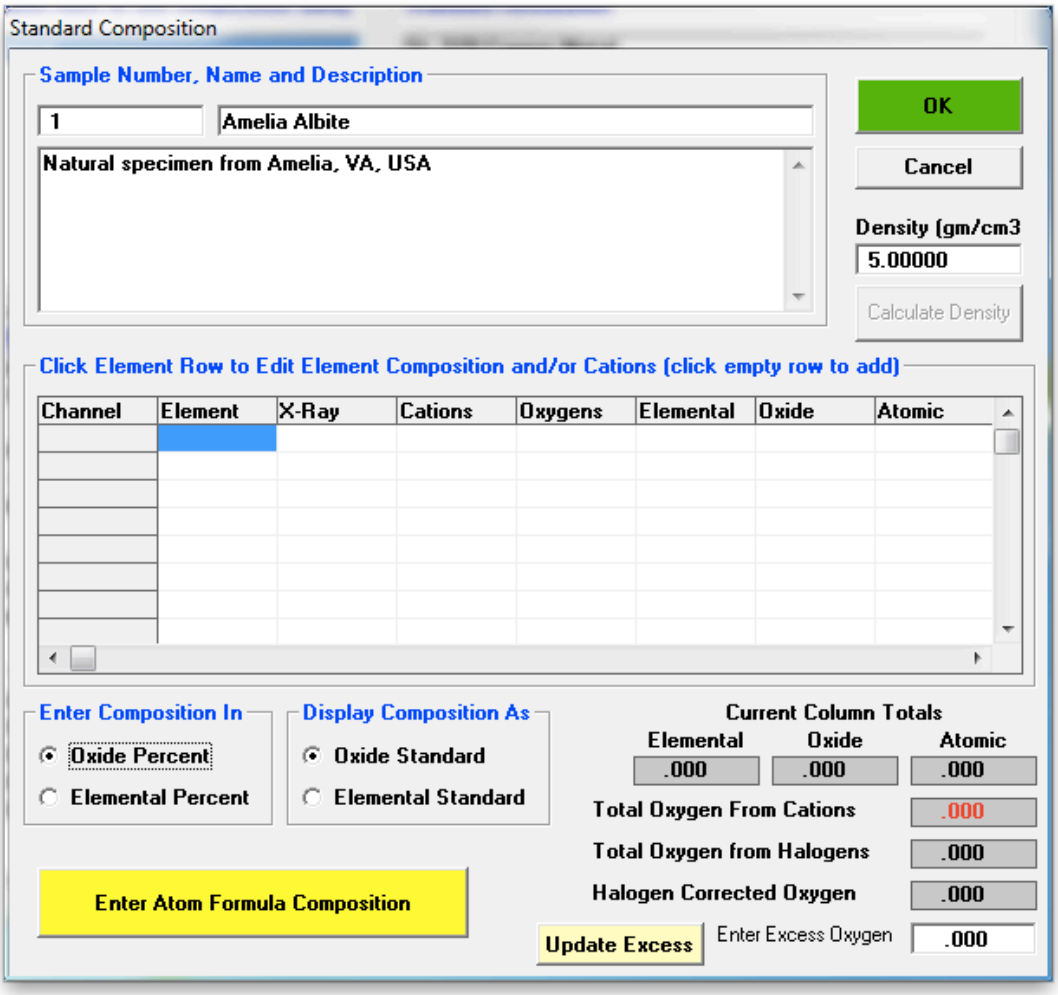


If there are more elements (compound standards) in the standard, click the next empty *Element* row and repeat the data entry process. When all elements are entered, click the **OK** button on the **Standard Composition** dialog box. This concludes the entry of a standard into the standard database and results in the following log window output.



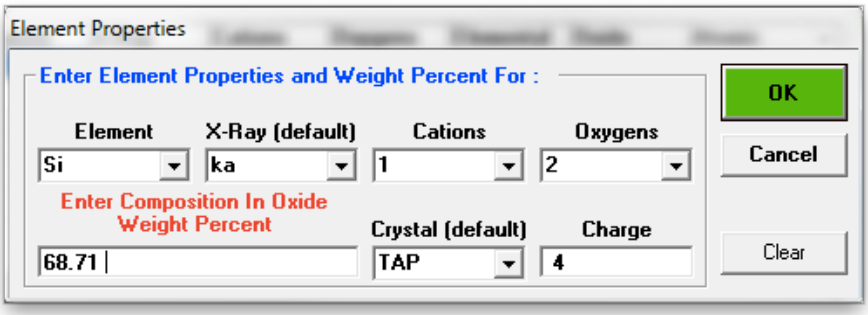
Many standards contain oxygen in their compositions. Since all standard compositions are saved to the standard database as elemental concentrations, it is necessary to enter the oxygen concentration if oxygen is present in the compound. This applies to all standards, even those that are entered and/or displayed as oxide concentrations. The following example illustrates a silicate (oxygen bearing) standard entry into the database.

From the main STANDARD log window, select **Standard** from the menu bar and click on **New** from the menu choices. This action opens the **Standard Composition** dialog box. Type the appropriate *Sample Number*, *Standard Name*, and *Standard Description* into the text boxes. Click the *Oxide Percent* and *Oxide Standard* buttons under the *Enter Composition In* and *Display Composition As* boxes.

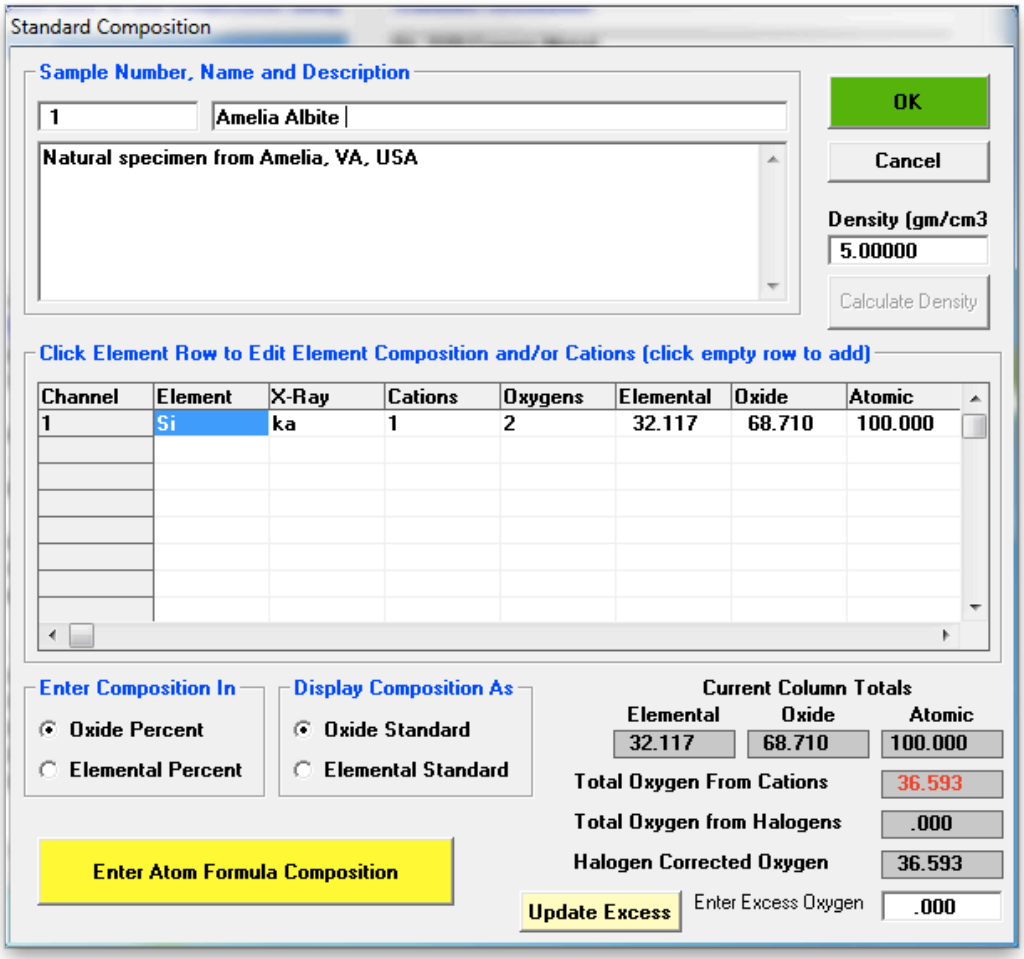


Click on any empty row in the spreadsheet.

This opens the **Element Properties** dialog box. In the *Element* field either type in the first element in the standard or use the drop-down list box to select the element symbol. Continue by choosing the correct *X-Ray* line, *Cations*, and *Oxygens*. Finally, enter the weight percent for SiO_2 into the *Enter Composition In Oxide Weight Percent* text box.

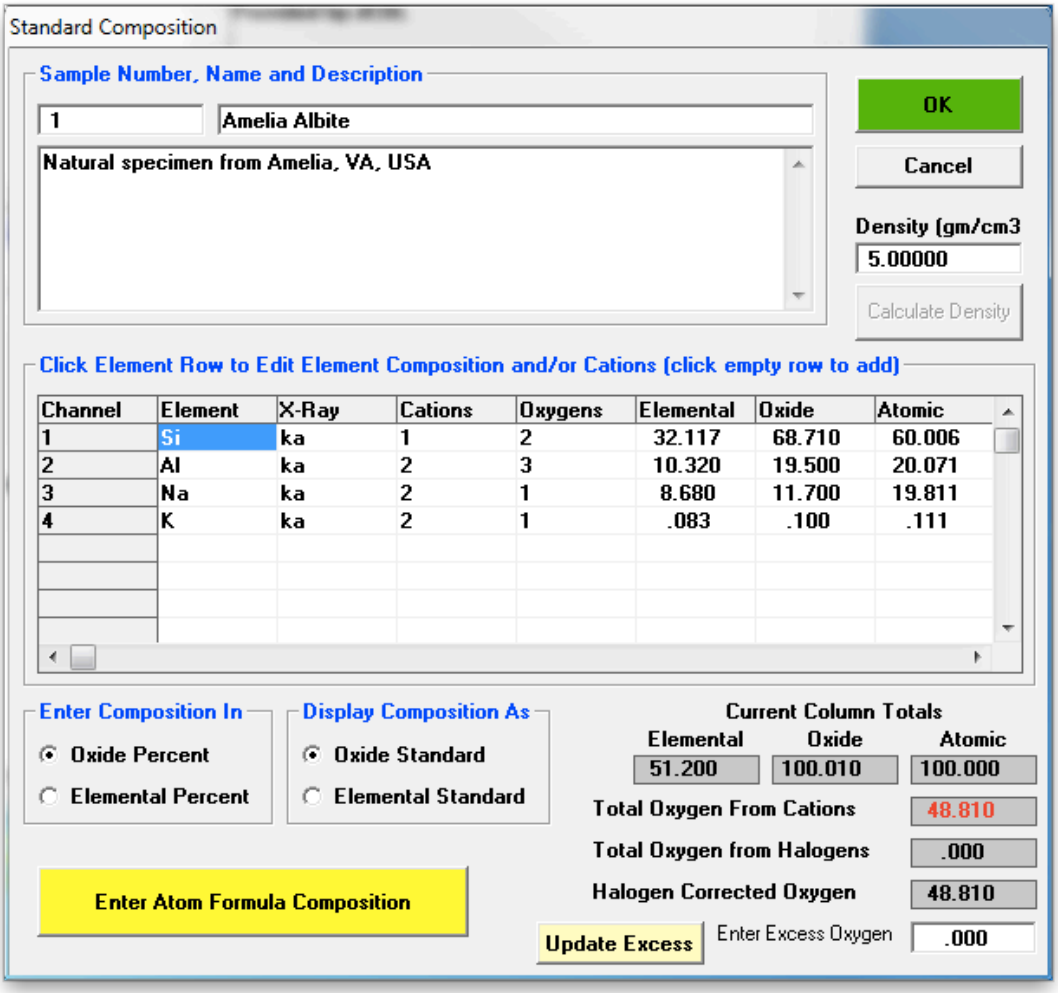


Finish by clicking the **OK** button of the **Element Properties** dialog box. This results in the following **Standard Composition** dialog box.

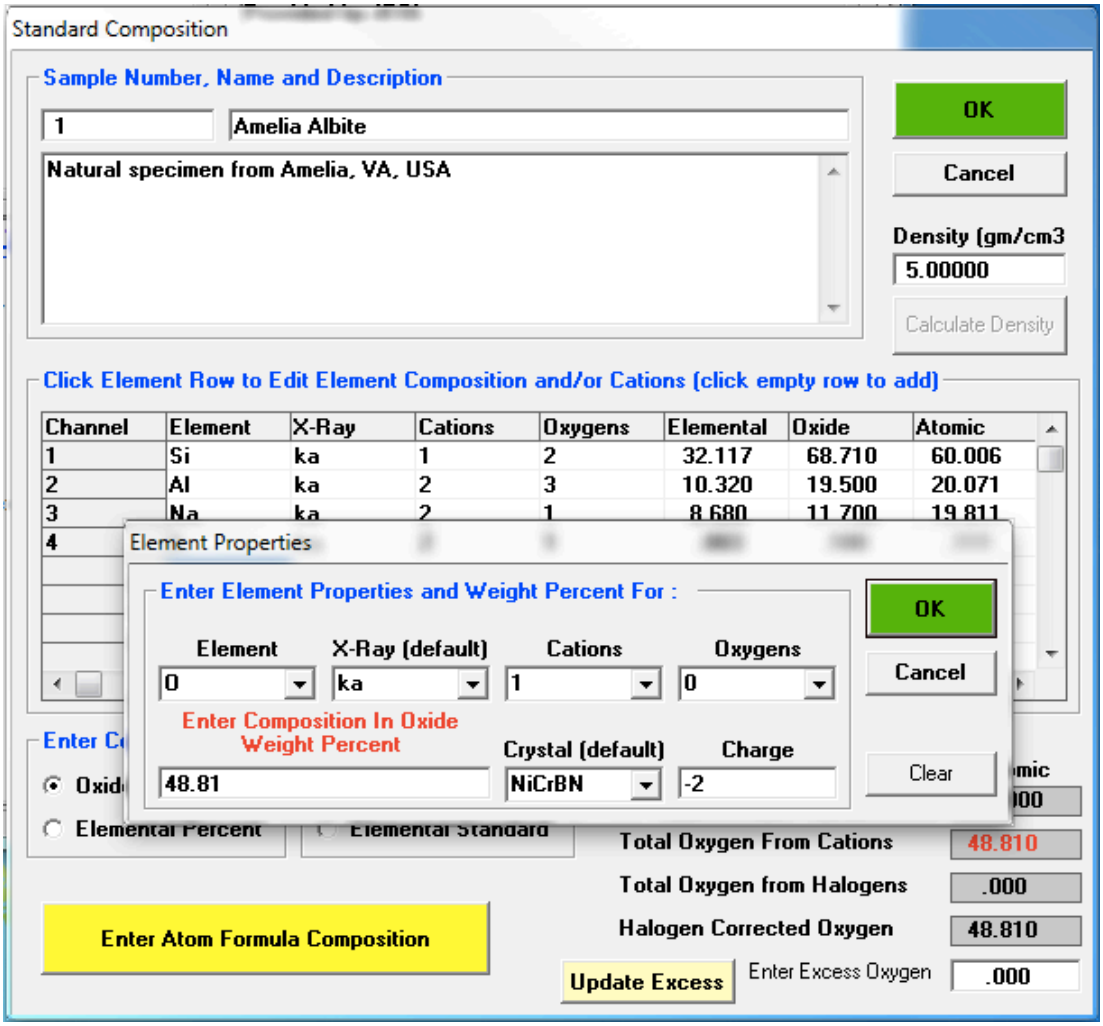


Note: to facilitate the data entry for the oxygen concentration of standard compositions which are entered as oxide concentrations, the program will display a running total in the text box designated *Total Oxygen From Cations.*

Continue the data entry process for the remaining elements (as oxides).

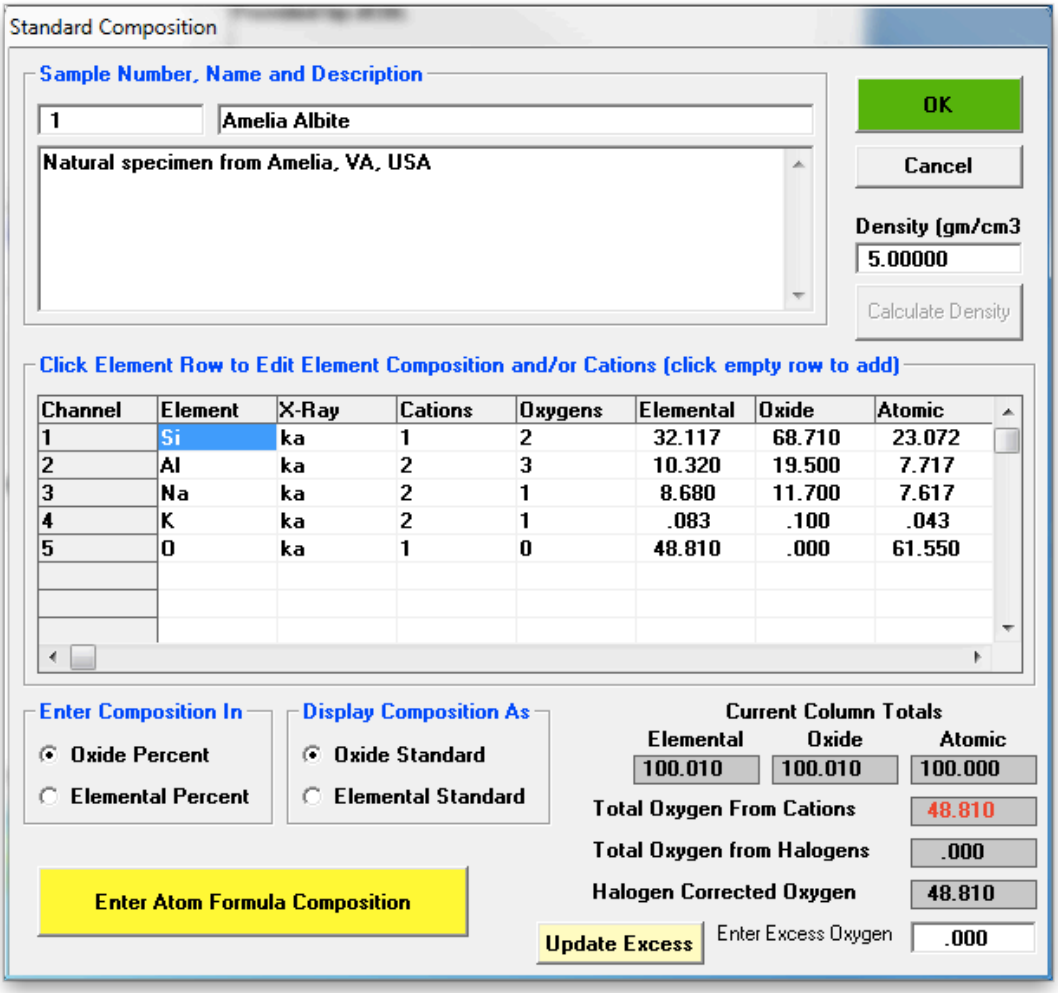


To complete the standard entry into the standard database, enter oxygen as the last element in the standard. Click on any empty row in the spreadsheet. This opens the **Element Properties** dialog box. In the *Element* field type in the element symbol for oxygen. Check for the appropriate *X-Ray* line, *Cations*, and *Oxygens*. Finally, enter the running total from the *Total Oxygen From Cations* text box into the *Enter Composition in Oxide Weight Percent* text box.

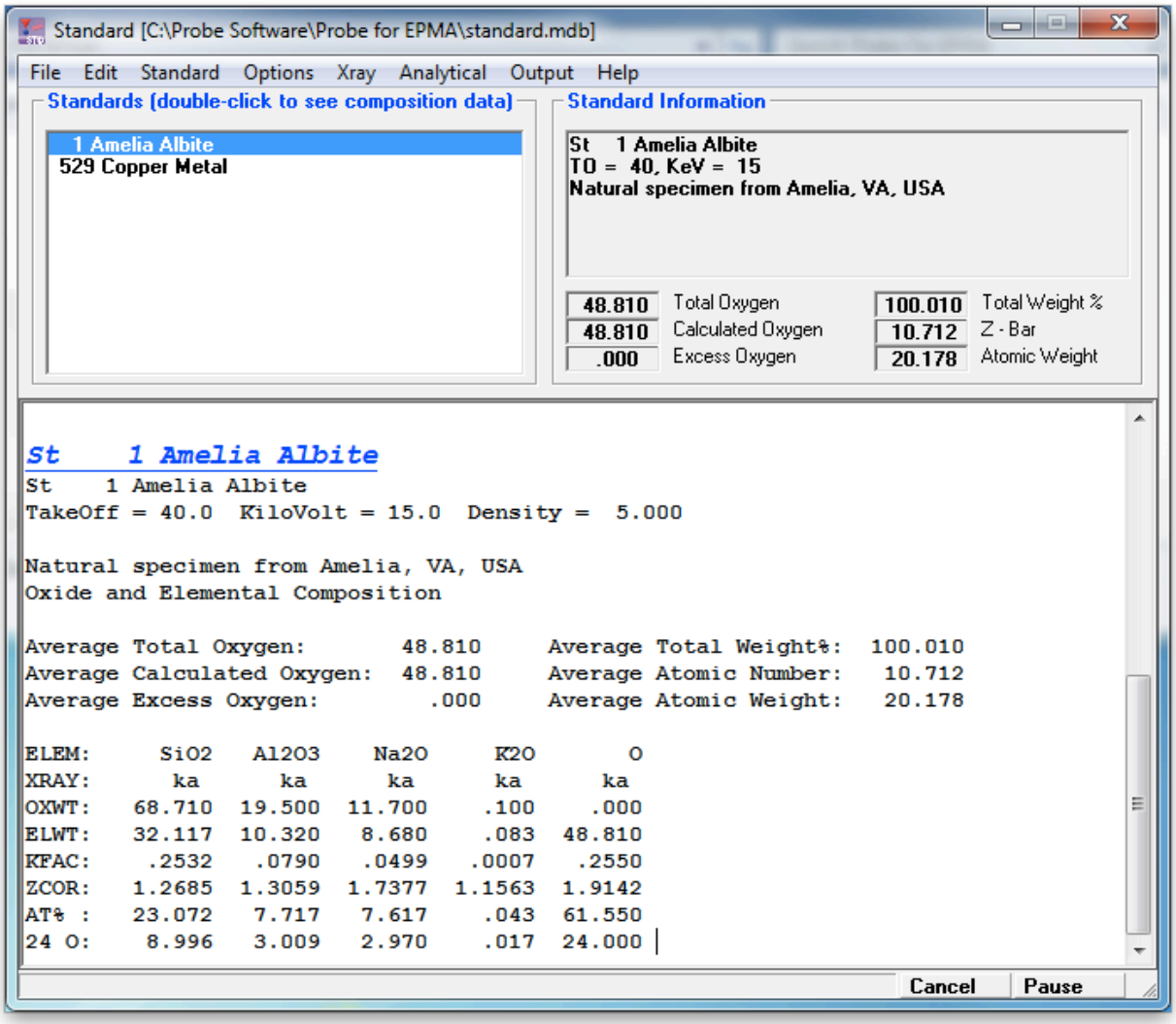


Click the **OK** button of the **Element Properties** dialog box.

The following **Standard Composition** dialog box illustrates the completed five-element silicate standard, Albite.

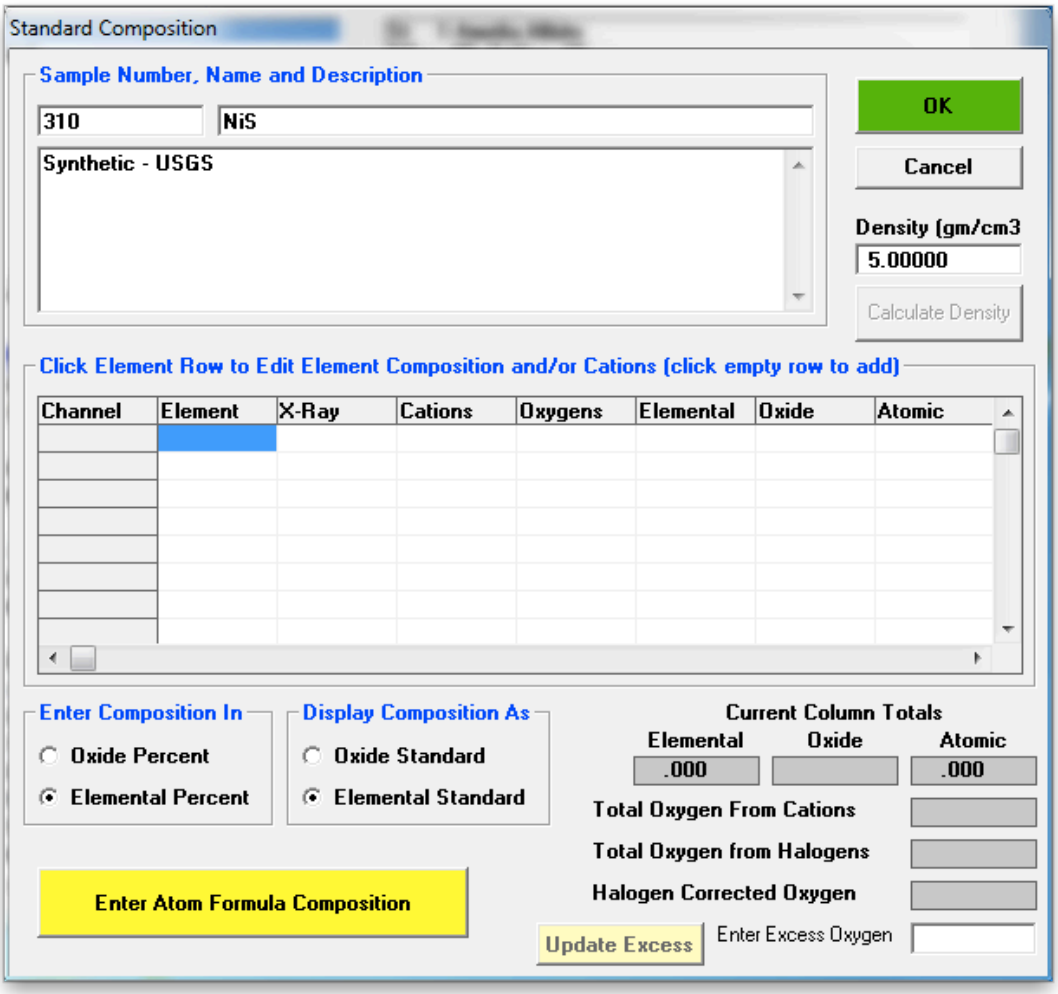


The compositional data of any standard entered into the standard database may be reviewed by simply double-clicking on the standard of interest from the scrollable *Standards* list box. The following window contains two standards with the compositional data of Albite displayed in the log window in oxide form.



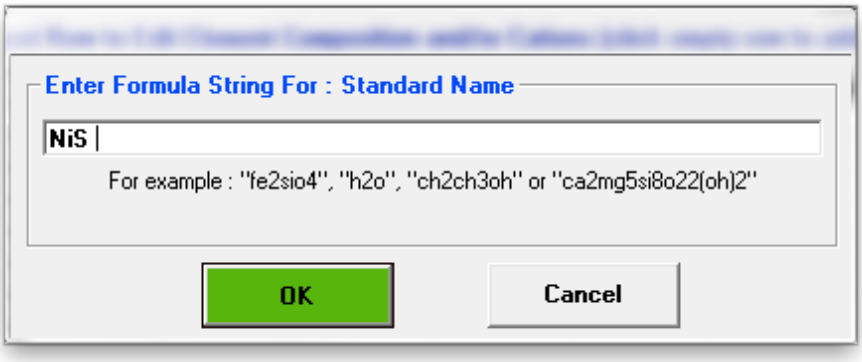
Some standards are simple end-member compounds or stoichiometric phases. These standards may be entered as a formula string.

Select **Standard** from the menu bar and click on **New** from the menu. This action opens the **Standard Composition** dialog box. Type the appropriate *Sample Number*, *Standard Name*, and *Standard Description* into the text boxes.

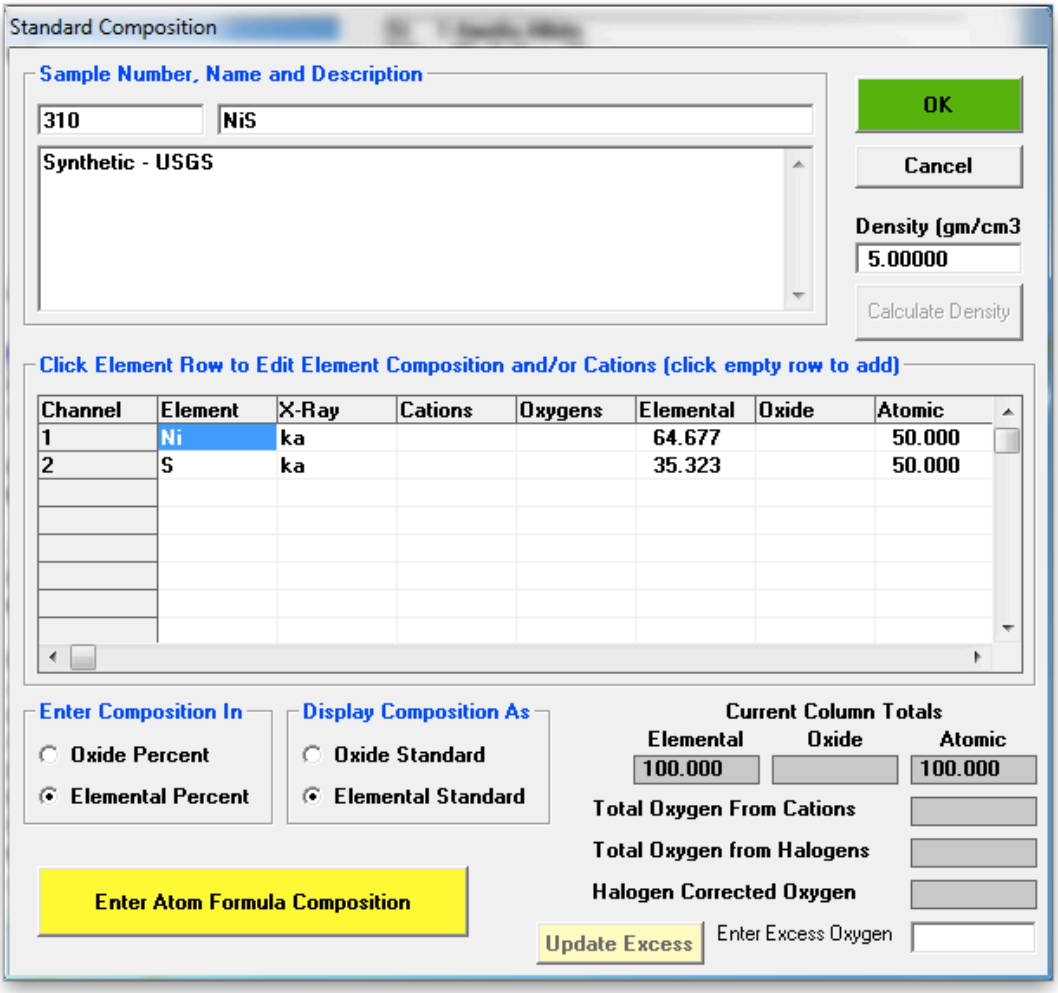


Click the **Enter Atom Formula Composition** button and enter the formula string into the text box.

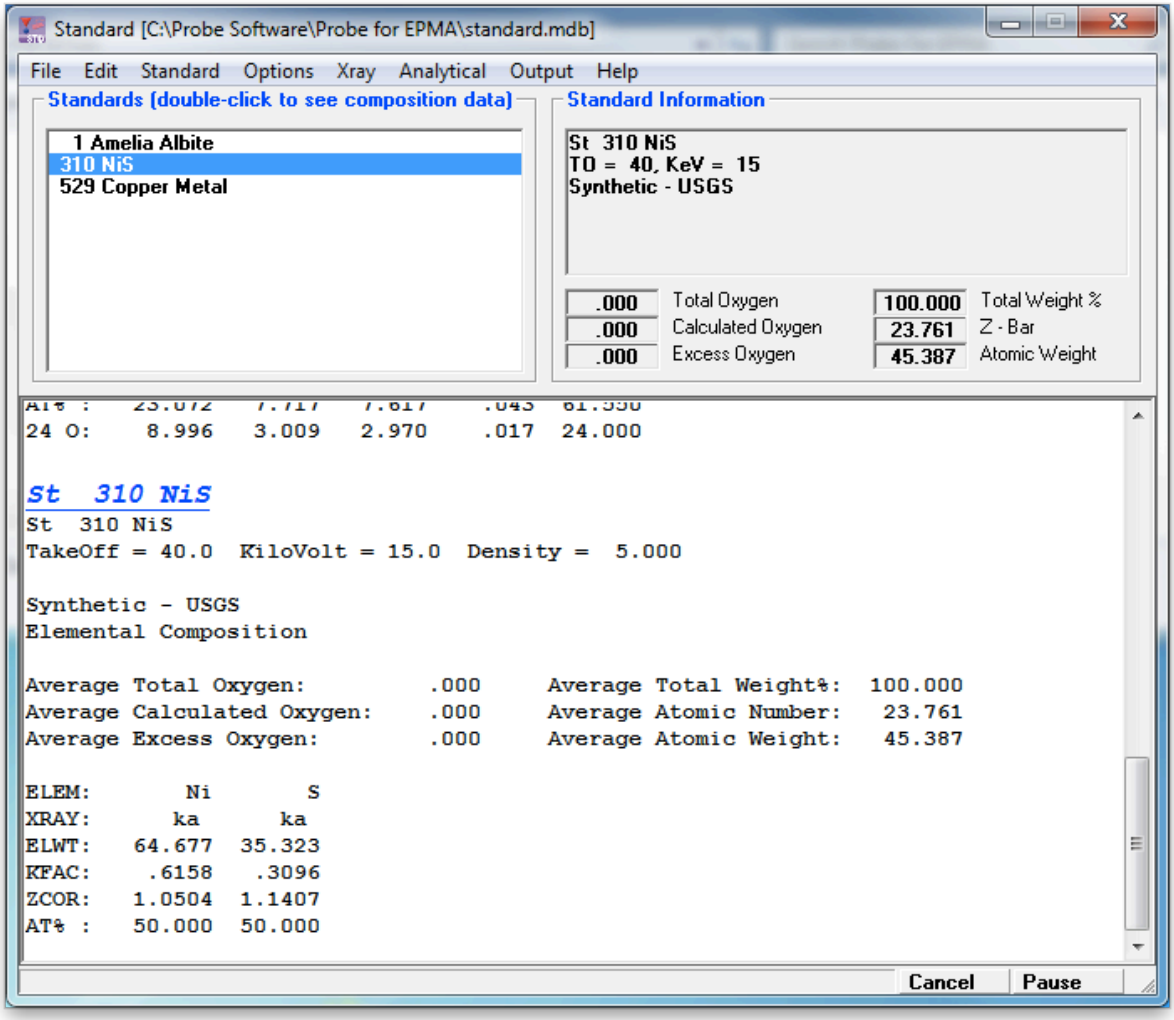
In this example NiS for nickel sulfide was entered.



Click the **OK** button. The stoichiometric phase NiS is entered into the **Standard Composition** window.



Click the **OK** button closing the **Standard Composition** window and returning to the main STANDARD log window.



To modify a particular standard, select the standard in the *Standards* list box. Click **Standard** from the menu bar and select **Modify** from the menu. Edit the appropriate fields in the **Standard Composition** window as described previously.

After entering all of the standard compositions in your standard collection, copy this important file (STANDARD.MDB) to another directory on the hard disk and likewise to another storage media for archival purposes.

Note: the takeoff, kilovolt, x-ray, and cation ratio parameters displayed here are used only for nominal calculations of the k-factors and ZAF corrections within the program STANDARD. The PROBE FOR EPMA quantitative analysis will calculate the quantitative standard k-factors based on the actual conditions.

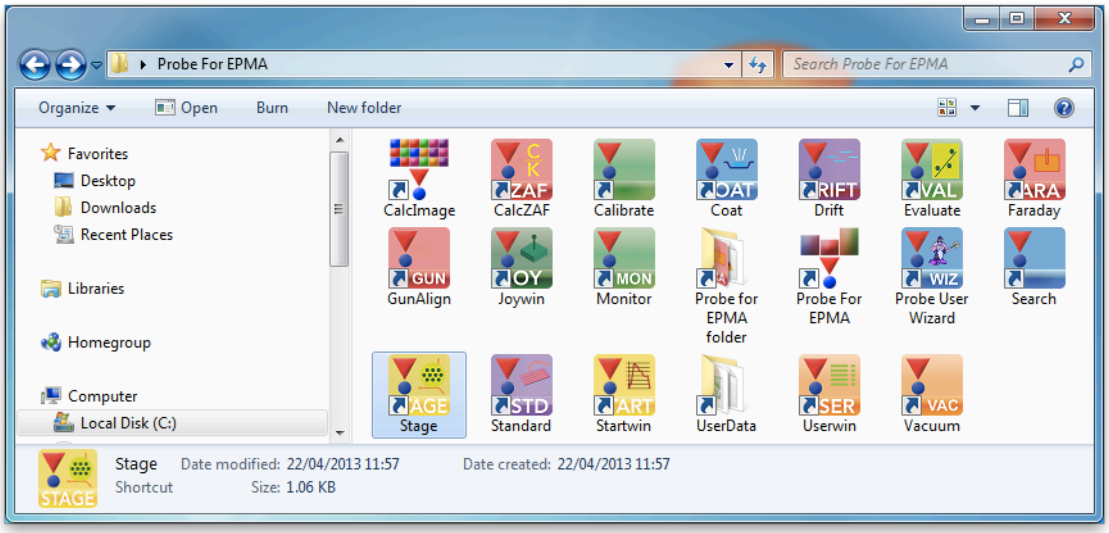
Creating Standard Position Files

The program STAGE is used to digitize your standard mounts to create pre-digitized standard coordinate files. These files are necessary for automated acquisition and standardization. The standard coordinates are digitized in three dimensions (X, Y, and Z) as well as the W stage position (JEOL multi-position specimen stages only) and are typically referenced to three physical fiducial marks on the standard mount surface. These coordinate files should be digitized with the standard mount located in the position where it is typically found.

The following procedure illustrates how to create a new standard position file. In this example, four carbonate standards will be digitized. These standards must already be entered into the standard database, using program STANDARD.

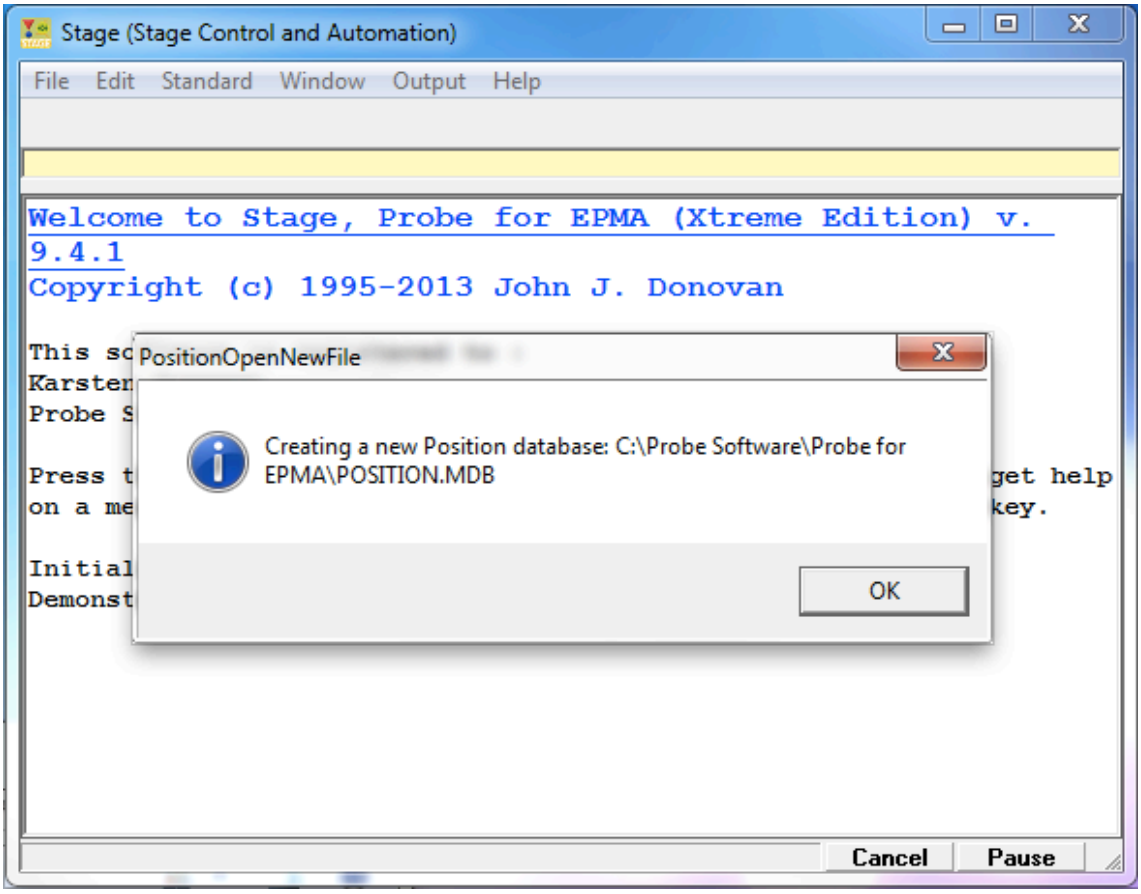
When creating digitized standard files for standard mounts containing more than 64 standards, a slightly different procedure than outlined below must be followed. Concise instructions on how to bypass the current 64 standard limit in the STAGE digitize feature are outlined in the reference documentation. To find these instructions, open the PROBEWIN.HLP program from the Probe for EPMA folder. Click the **Index** button and type in digitize in the text box. Highlight the topic entitled *Digitizing Standard Mounts with More than 64 Standards* and click the **Display** button.

Open the STAGE application (Stage Control and Automation), if available by double clicking on the **Stage** icon in the Probe for EPMA Software group.



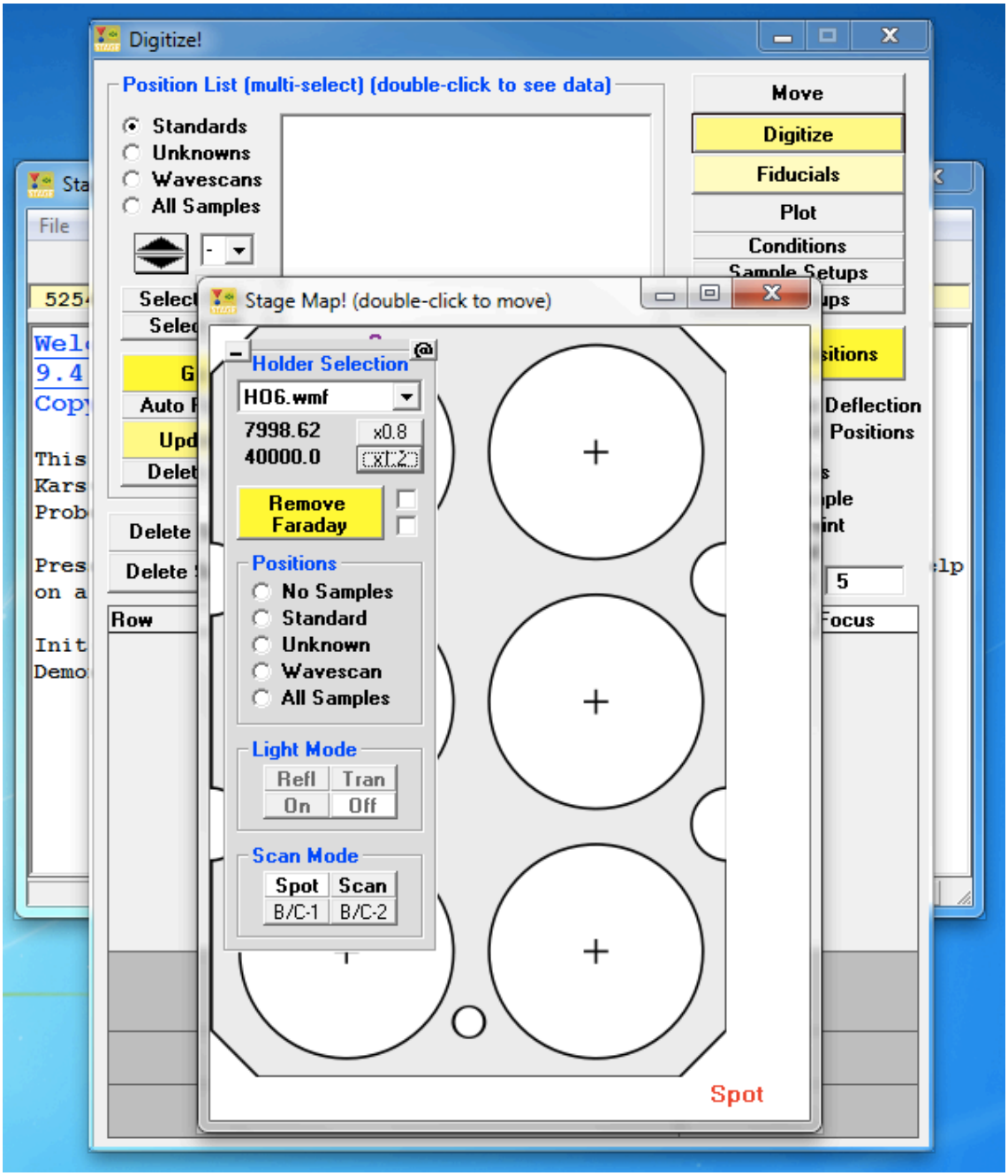
Alternatively, select **Stage** from the Probe Software group in the Windows Start Menu, or locate and double click on the STAGE application in the Probe for EPMA application directory, which is usually C:\Probe Software\Probe for EPMA under Windows Vista and 7, or C:\Program Files\Probe Software\Probe for EPMA for older operating systems.

This starts the STAGE program and opens its main log window. If no POSITION.MDB is detected, the **PositionOpenNewFile** dialog opens.

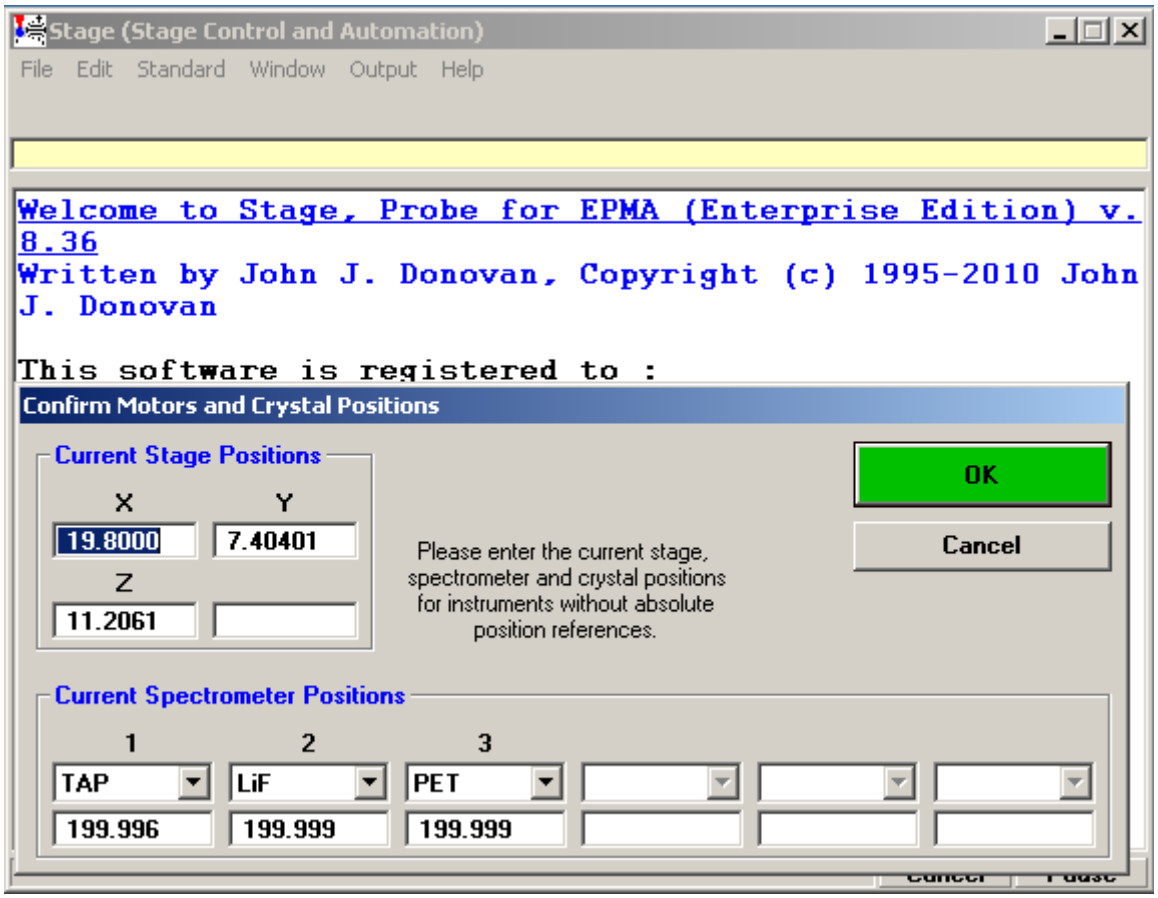


Clicking the **OK** button creates a new Position database.

If the POSITION.MDB exists then STAGE opens the **Stage Map!** window and the **Digitize!** dialog as seen below.



Older microprobes with Advanced MicroBeam automation systems will first see the **Confirm Motor and Crystal Positions** dialog box when STAGE is first opened. Confirm that all of the motors (stage and spectrometer positions) and crystal designations are correctly calibrated. If there is disagreement between the mechanical positions (actual) and the software values, adjust the software values. Use the <tab> key to move between the various *Stage* and *Spectrometer Positions* boxes.

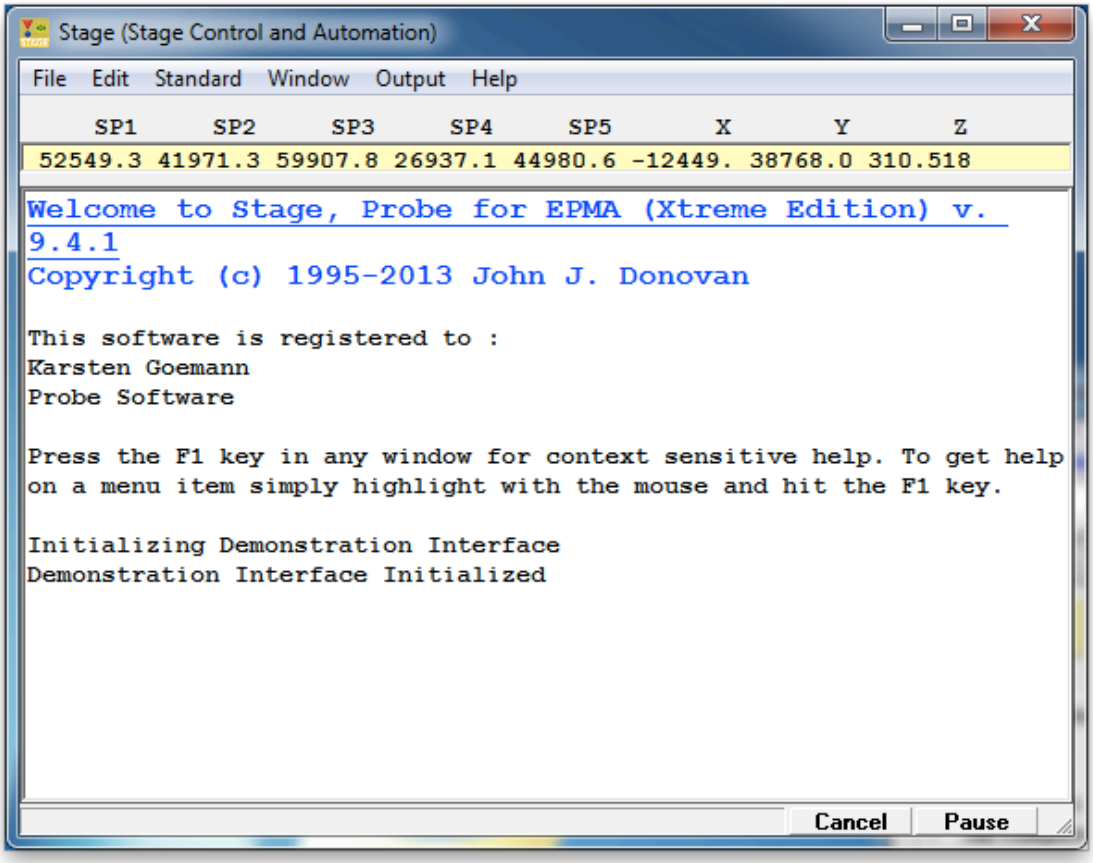


Click the **OK** button to close the **Confirm Motor and Crystal Positions** dialog box when done.

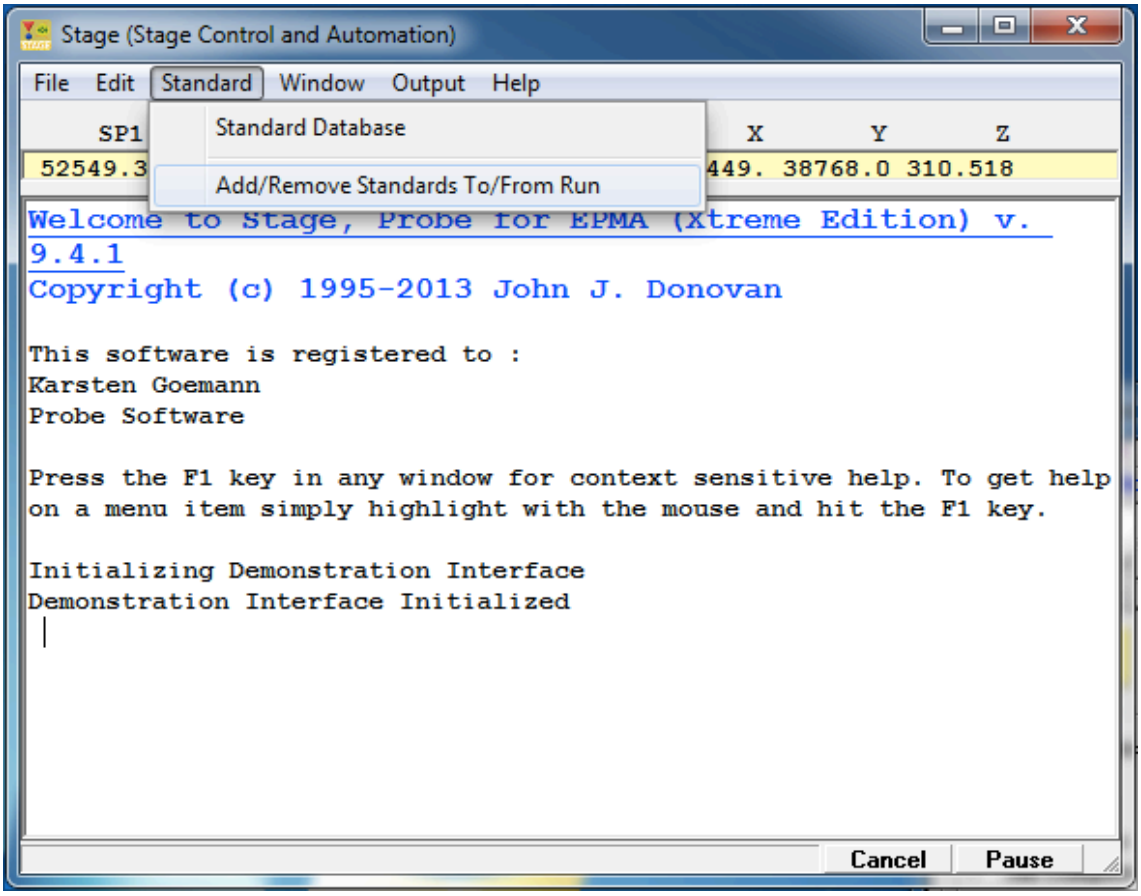
The **PositionOpenNewFile** dialog opens if no POSITION.MDB exists and a new POSITION.MDB is created upon clicking the **OK** button.

Two additional windows then open; the **Stage Map!** window and the **Digitize!** dialog as described on the previous page.

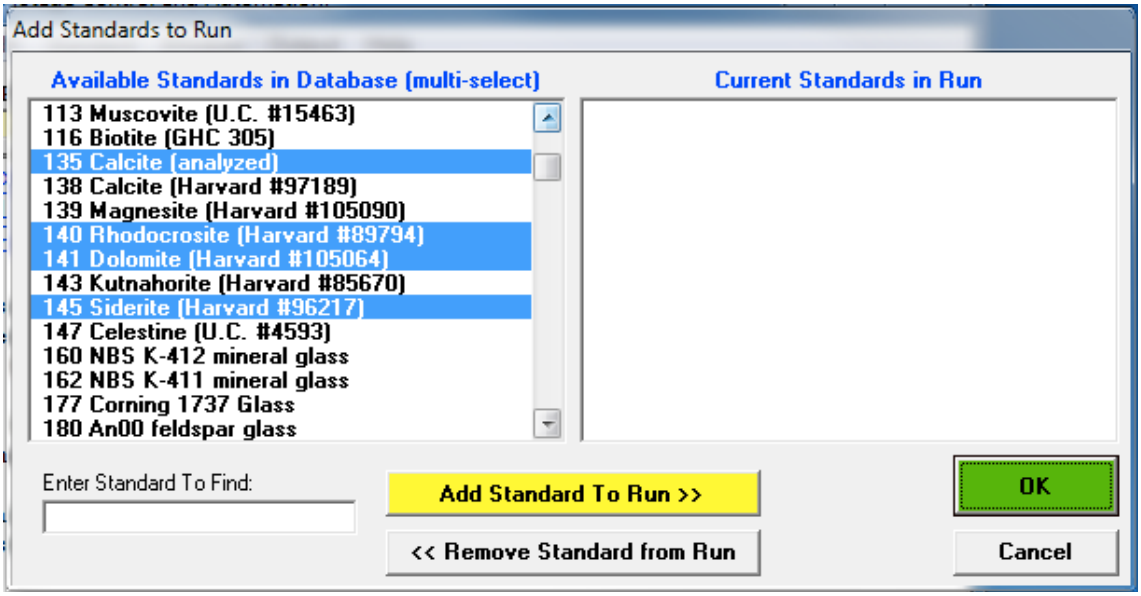
The following display illustrates the STAGE log window.



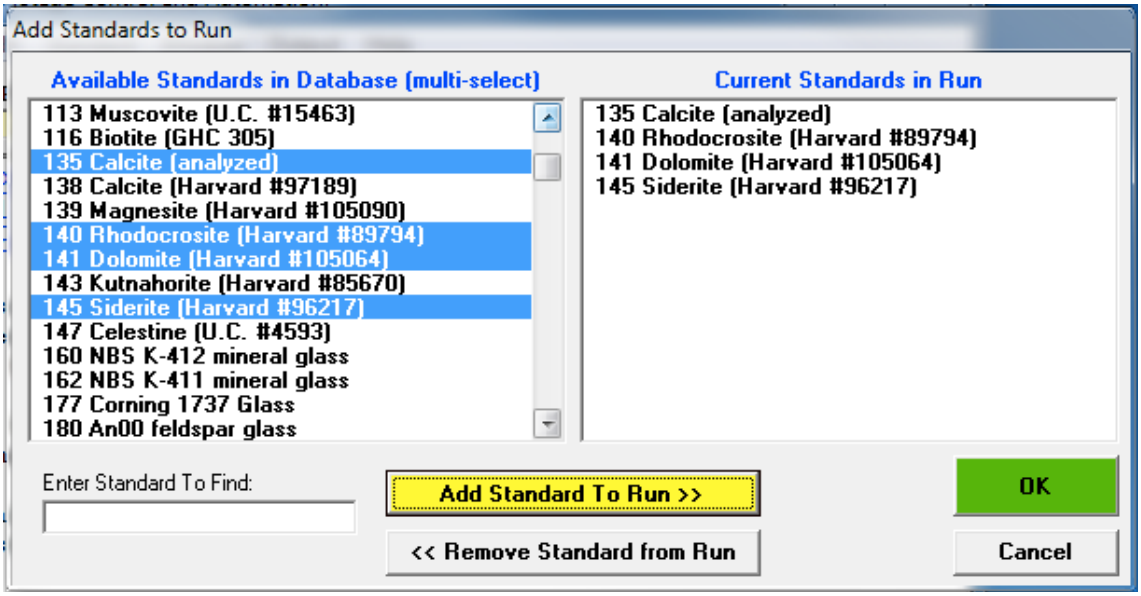
Select **Standard** from the menu bar and click on **Add/Remove Standards To/From Run** from the menu choices.



This action opens the **Add Standards to Run** dialog box. Click on the name of each of the standards in the standard block to be digitized from the *Available Standards in Database* list box.

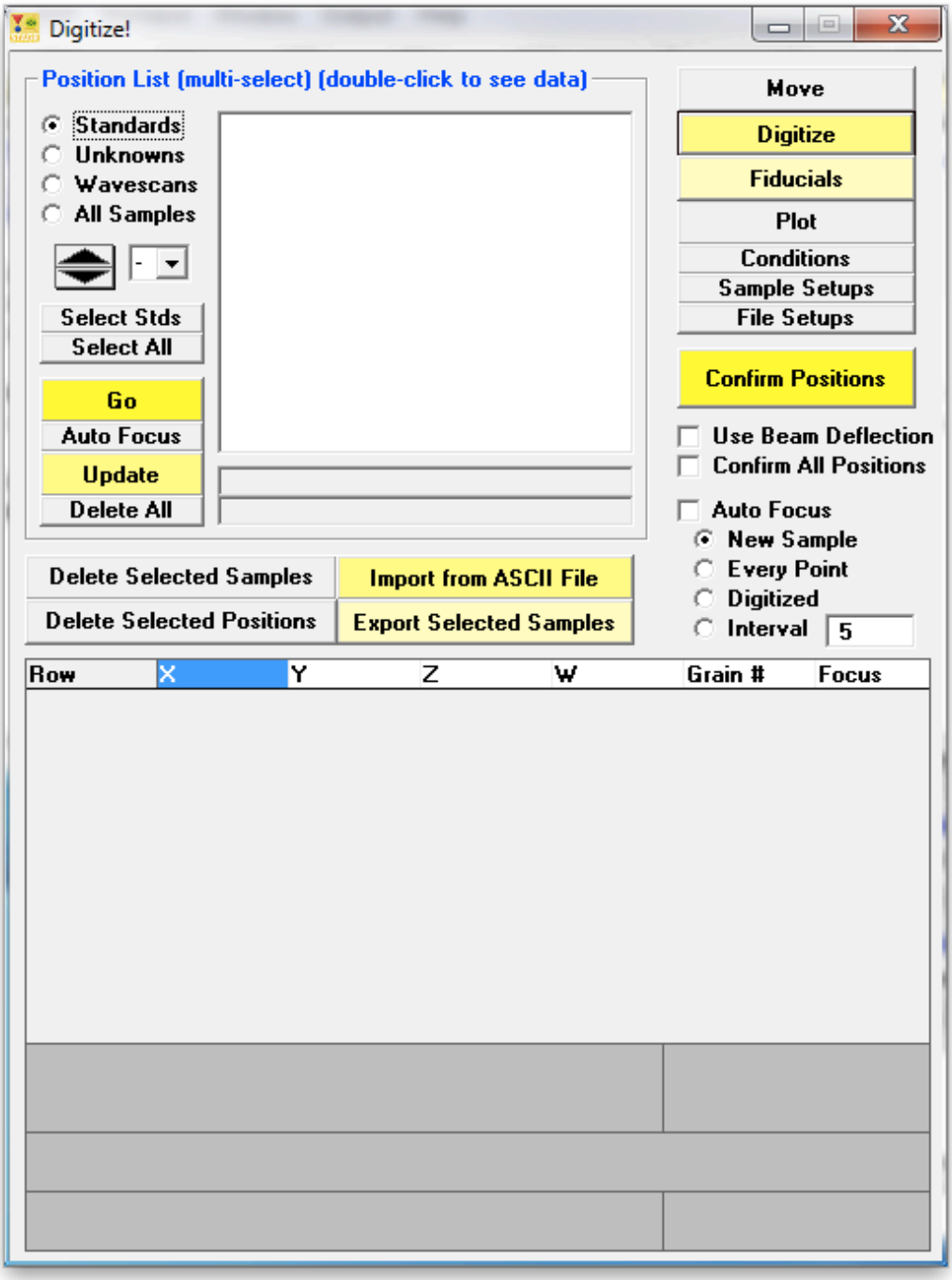


Click the **Add Standard to Run** >> button to move these standards into the current run. Standards may be added one at a time or the user may multi-select standards by holding down the <Ctrl> button on the keyboard as standards are selected. Double clicking each entry will also send the standard to the other column in the dialog window.



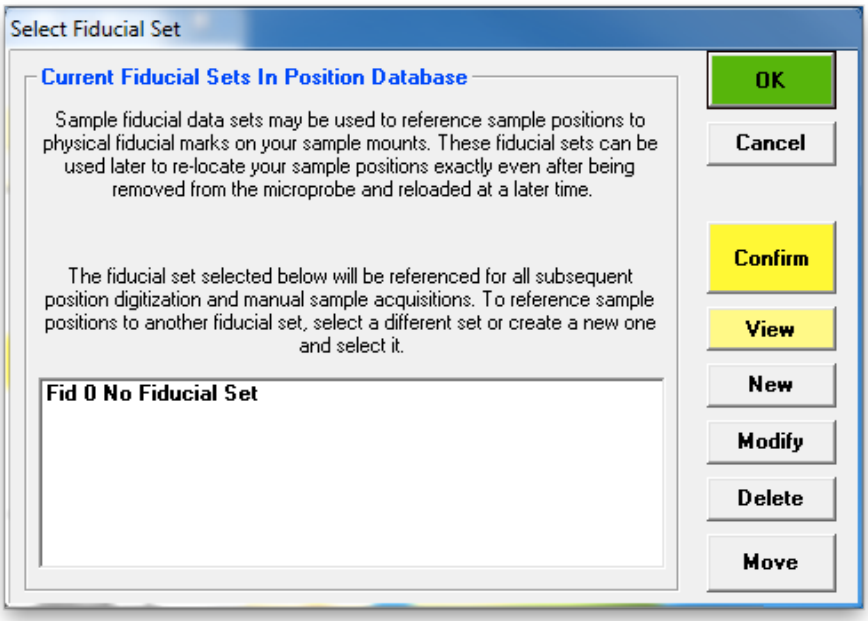
Click the **OK** button of the **Add Standards to Run** dialog box when finished.

The previously opened **Digitize!** dialog box should be brought forward.

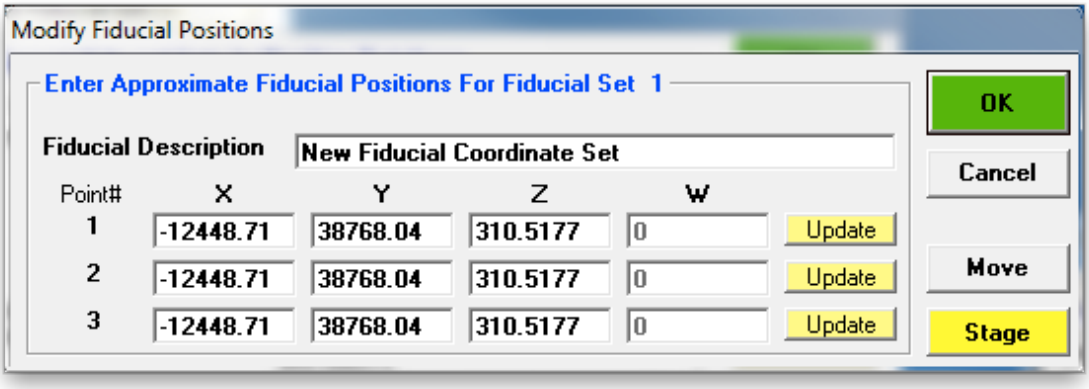


Click the **Fiducials** button.

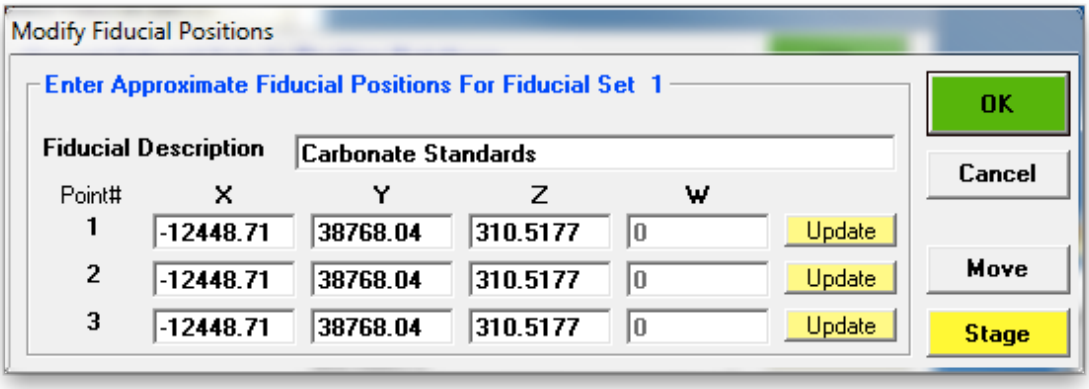
This opens the **Select Fiducial Set** window.



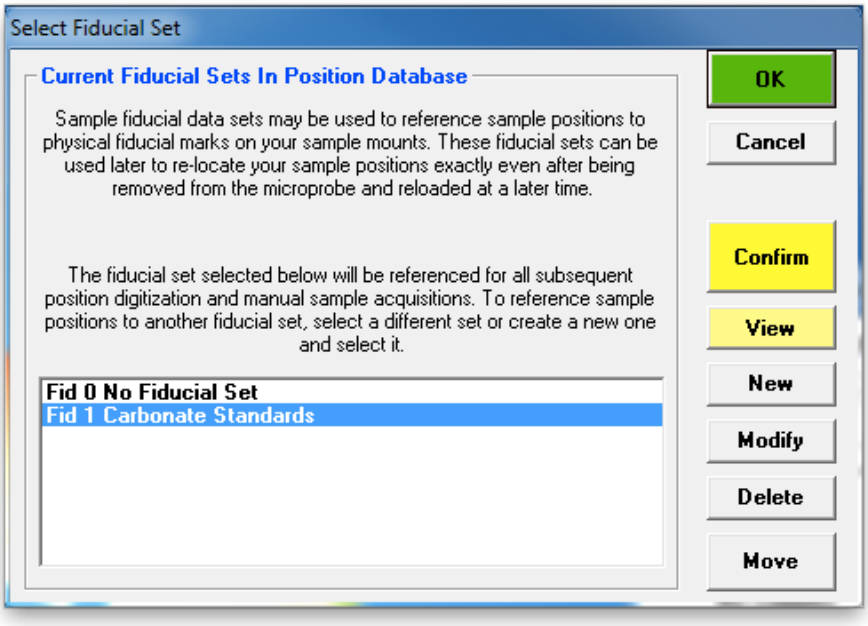
Click the **New** button. This opens the **Modify Fiducial Positions** window. The current stage coordinates are loaded by default.



Type in a *Fiducial Description*. Enter the nominal coordinates or move to each of the three fiducial marks on the standard mount, determining their approximate coordinates, and enter those values into the appropriate fields. On JEOL 733 microprobes, the W stage position needs to be recorded as well. The following window will result.

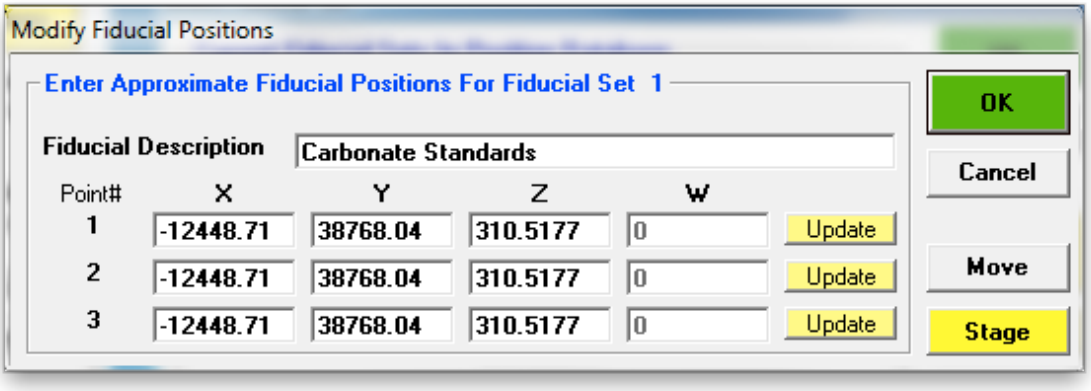


Click the **OK** button when done. This creates a new entry in the **Select Fiducial Set** list box as shown below.

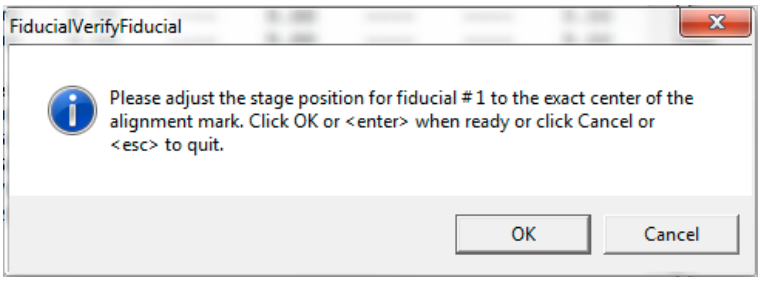


Select (highlight) the new fiducial set and click the **Confirm** button to initiate a precise centering of the three fiducial marks.

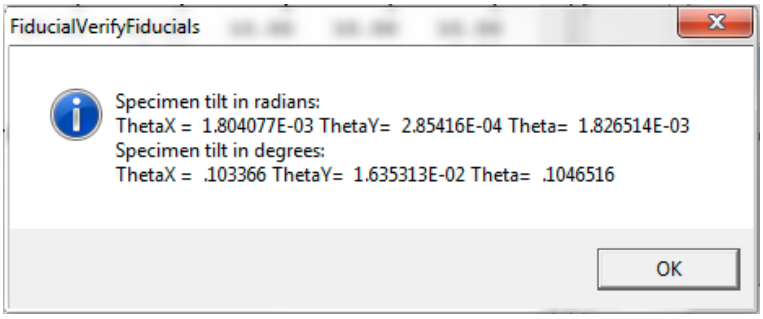
The **Modify Fiducial Positions** window opens displaying the originally entered fiducial coordinates. Click the **OK** button to initiate the centering process.



The computer then drives the stage to each fiducial mark and displays the **FiducialVerifyFiducial** window. Adjust the stage position to center the fiducial mark and click the **OK** button.

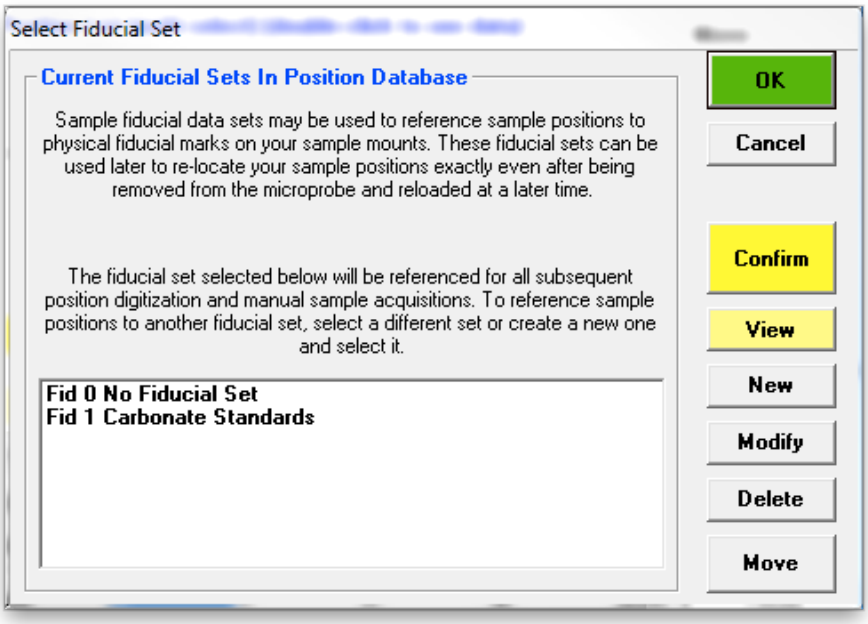


After centering the third fiducial mark and clicking the **OK** button, the **FiducialVerifyFiducials** window opens to display the specimen tilt in radians and degrees. A warning will be given if the sample is tilted at more than 0.5 degrees.

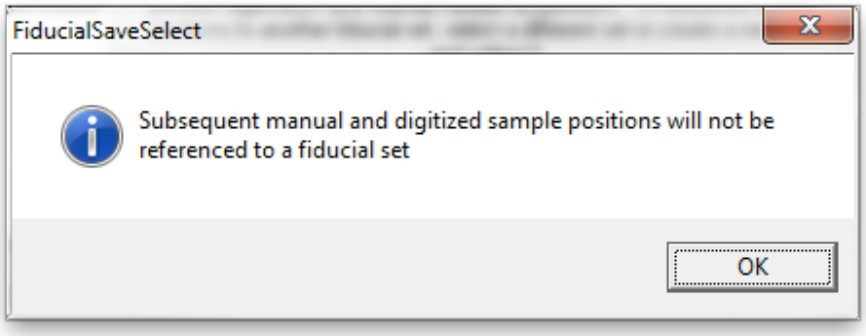


Click this **OK** button.

Closing the **FiducialVerifyFiducials** window returns to the **Select Fiducial Set** dialog box.

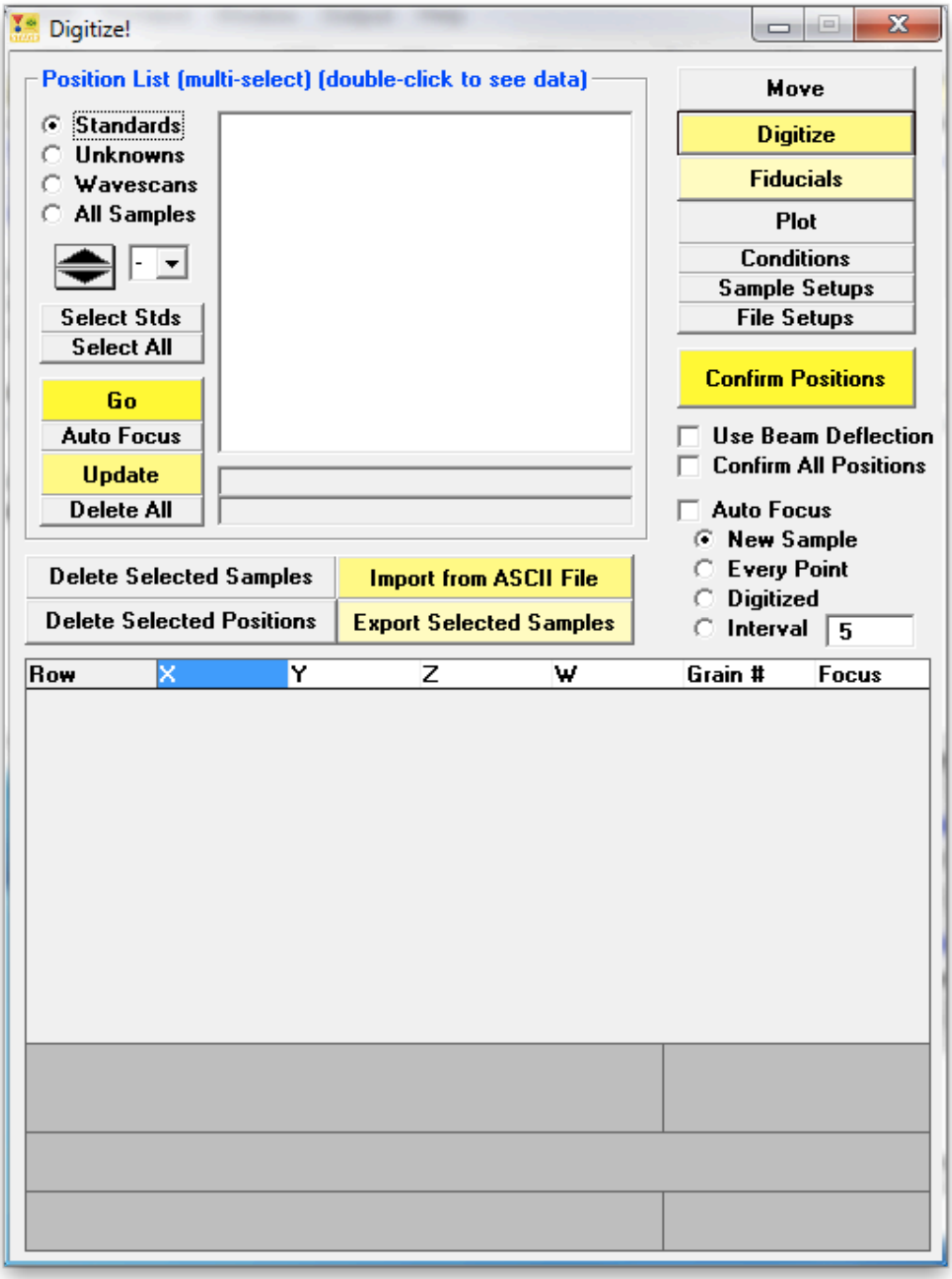


Finally, click the **OK** button on the **Select Fiducial Set** dialog box. This opens the **FiducialSaveSelect** window to confirm the currently selected fiducial set.



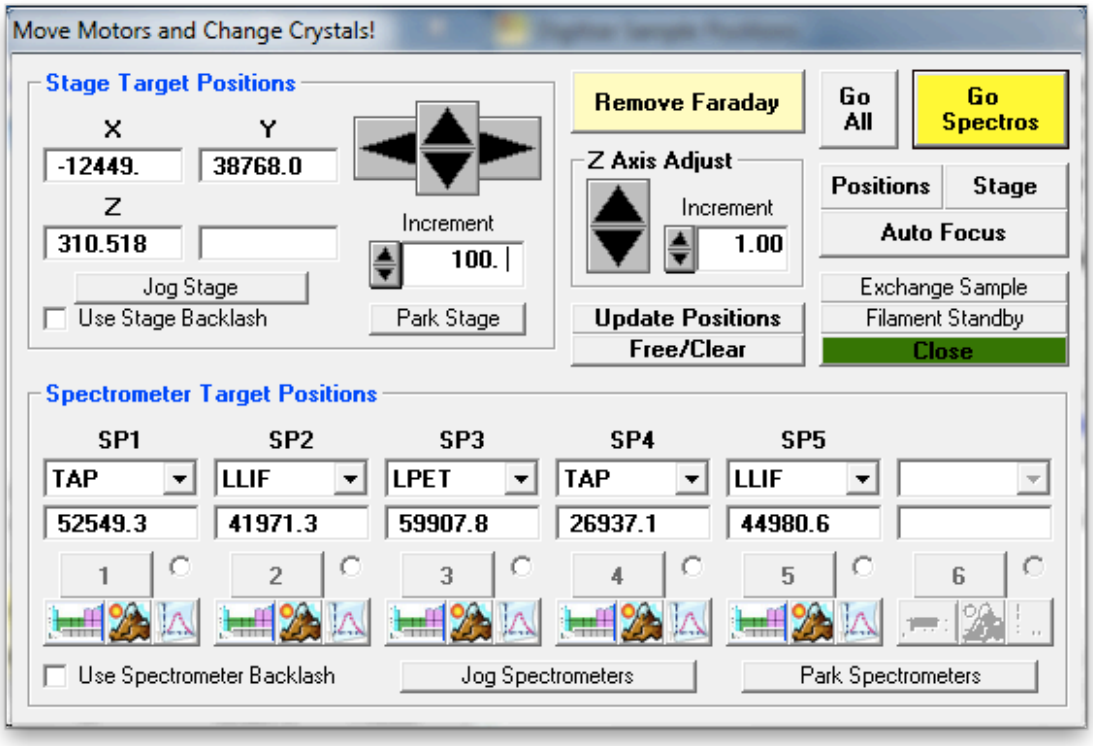
Click the **OK** button of the **FiducialSaveSelect** window.

The fiducial coordinate positions are recorded to disk and the **Digitize!** dialog box returns.



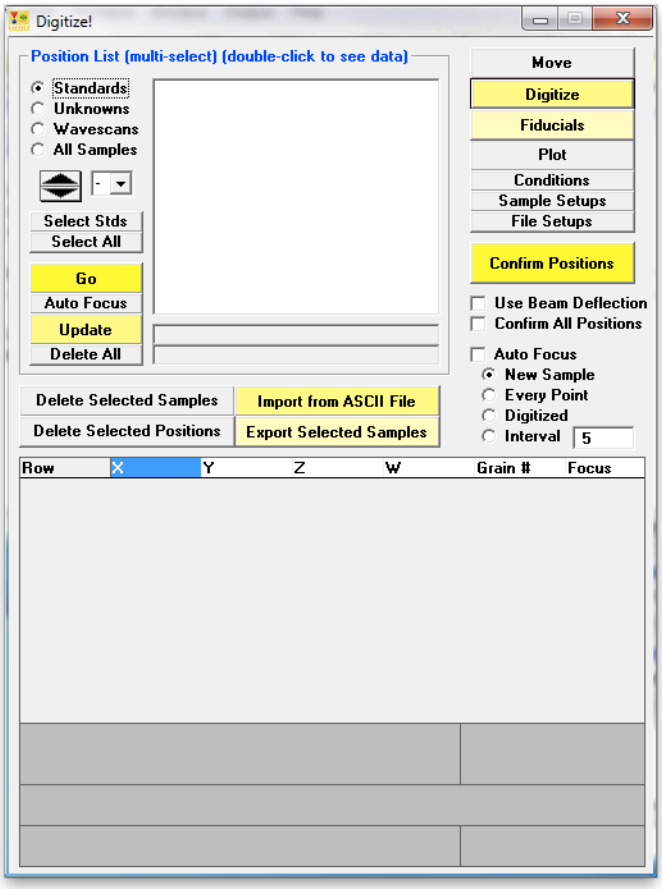
The position of each of the standards in this standard mount must now be digitized. Move to the first standard; either by turning the motor controls manually or, if possible with your instrument, by using the joystick via the JOYWIN (Joystick Control for Stage and Spectrometers) program, or by using the **Move** button in the **Digitize!** window. Clicking the **Move** button opens the **Move Motors and Change Crystals** dialog box.

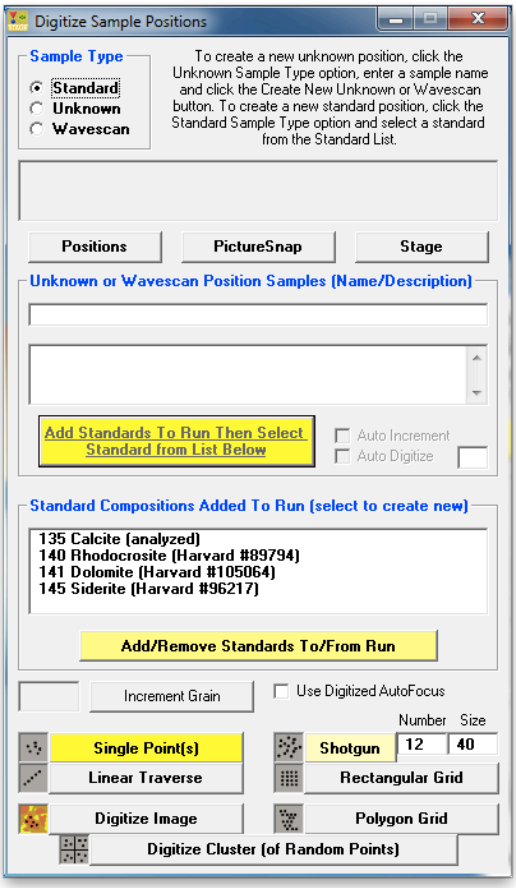
Type in the appropriate target coordinates in the *Stage Target Positions* boxes for the first standard. Use the <tab> key to move between entries.



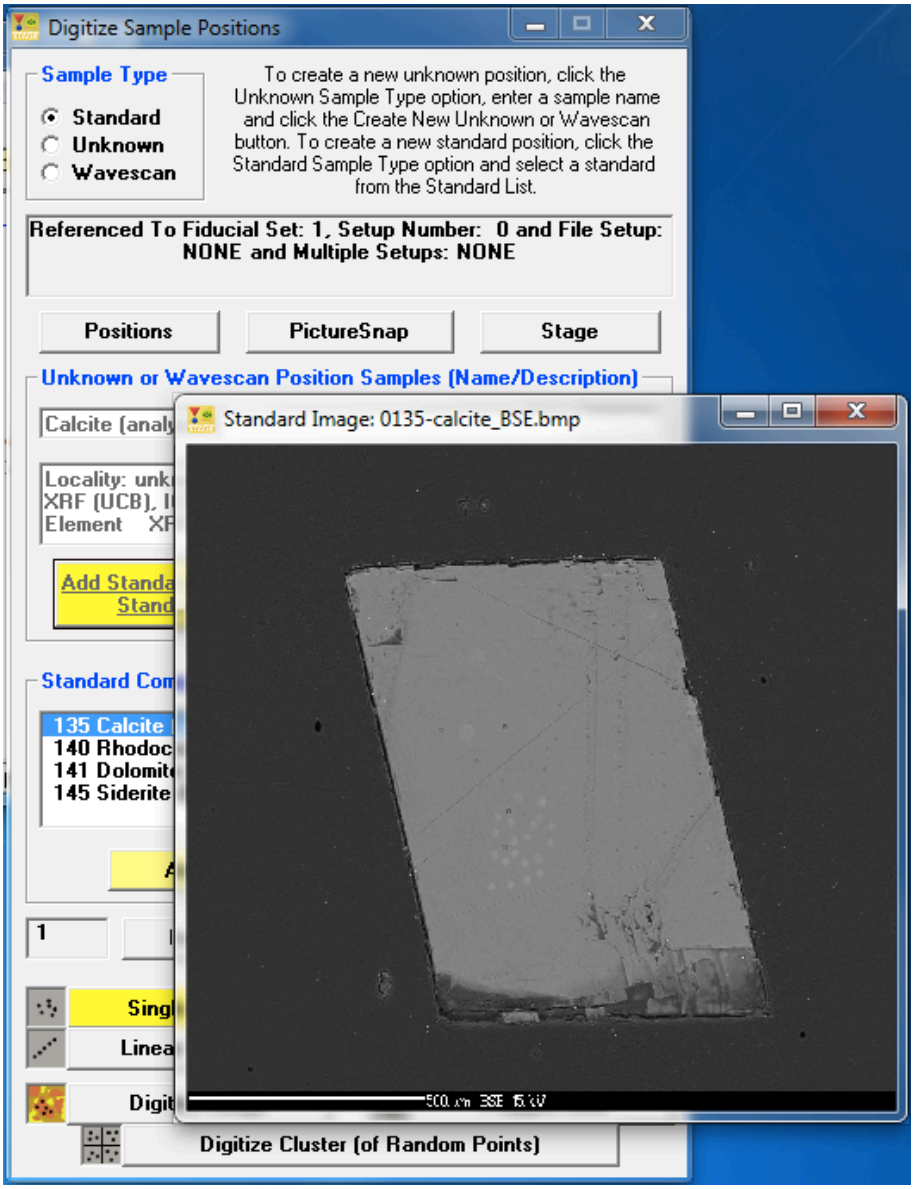
Click **Go All.** This will drive the stage to the target positions. Check the position and optical focus.

Click the **Digitize** button of the **Digitize!** dialog box. This activates the **Digitize Sample Positions** dialog box. The *Standard Position Samples* list box contains the standards already added to the run.

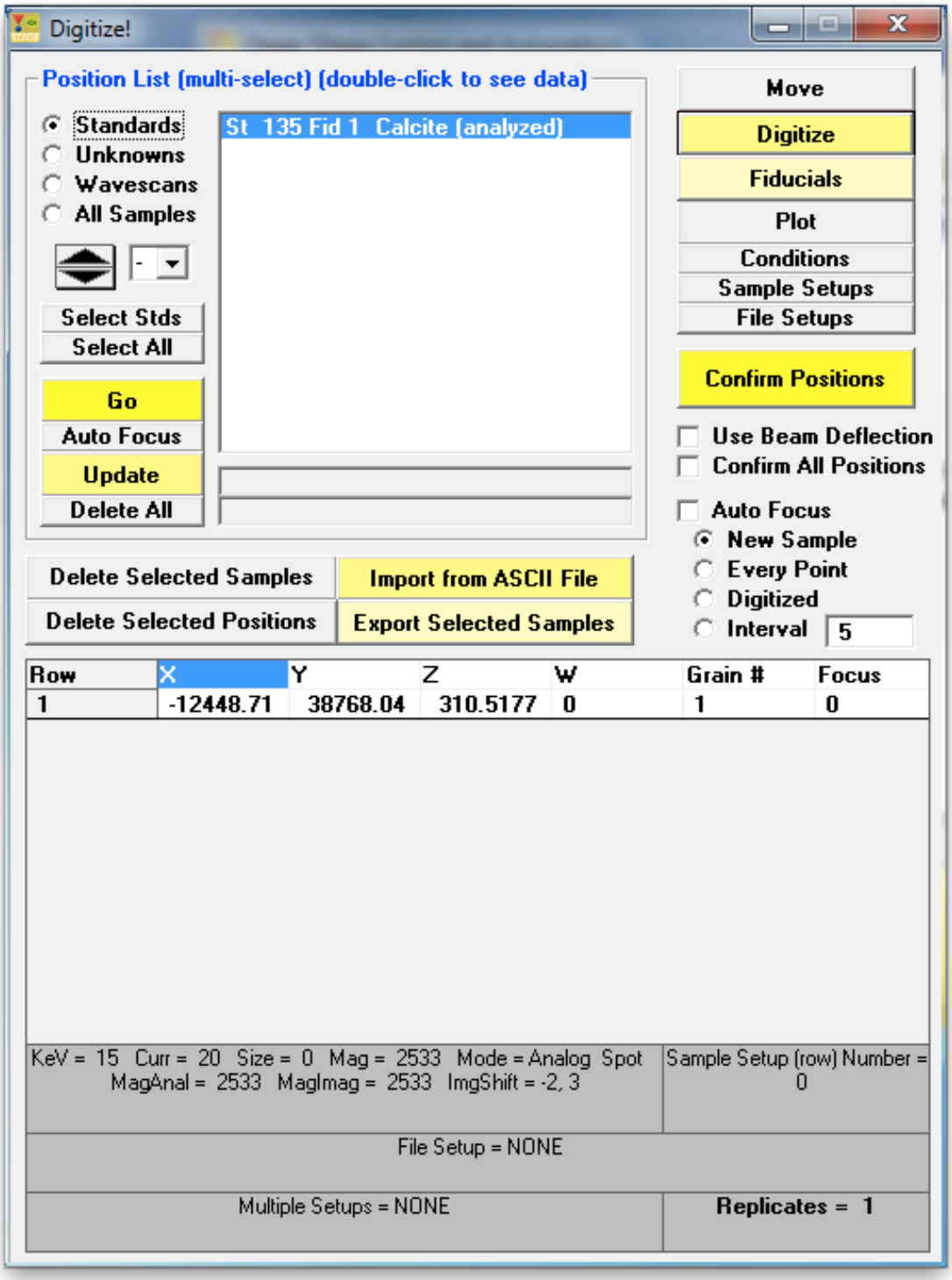




Select (highlight) the first standard to digitize from the *Standard Position Samples* list box. The standard will be added automatically to the **Digitize!** *Position List*. If a BMP electron image file exists for the standard, then it too will be displayed. This can be used to check whether you are on the correct standard. Further, the image could be annotated to suggest places or grains to avoid during the standard digitization or acquisition process.

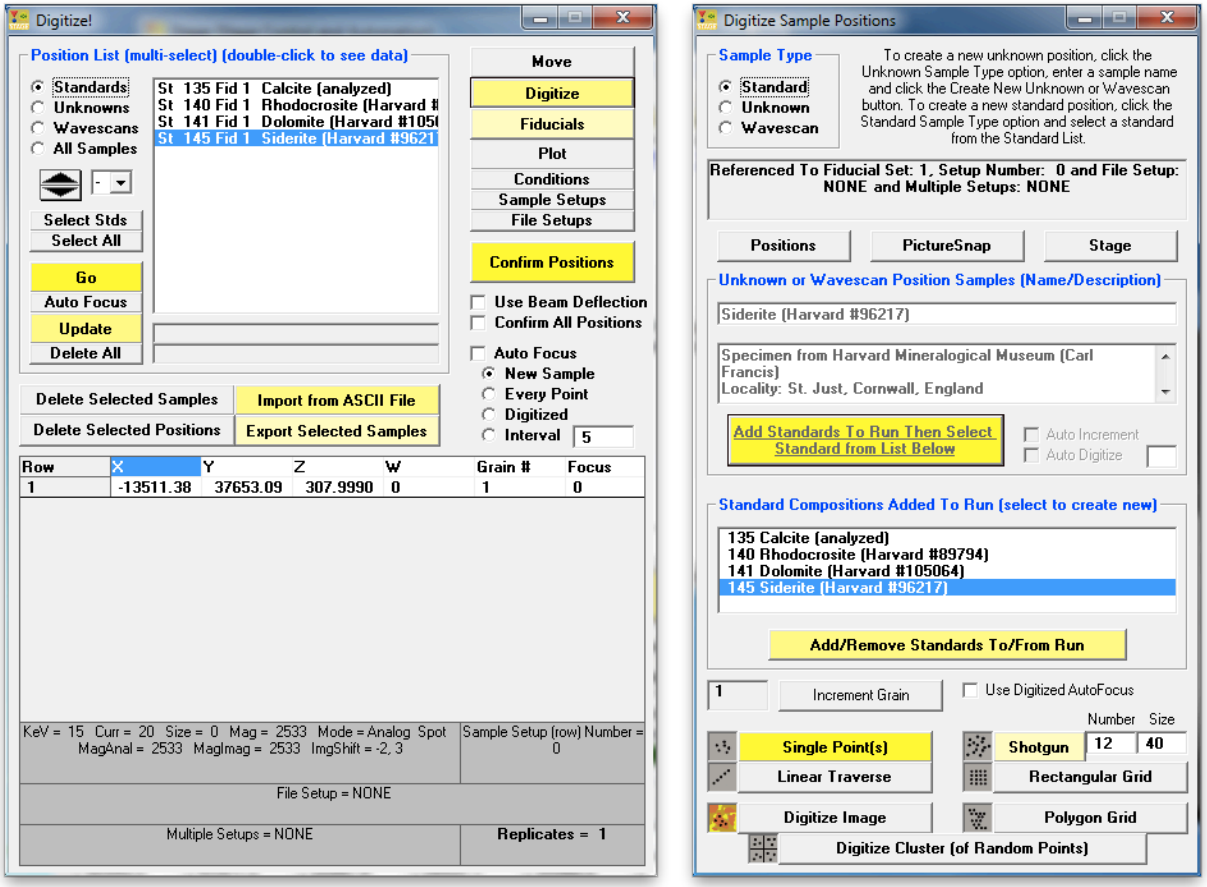


To digitize a random point on this standard, click the **Single Point(s)** button of the **Digitize Sample Positions** dialog box to record the current coordinates (X, Y, Z, and W) for this grain. The coordinates of this standard in the **Digitize!** dialog box are seen below.



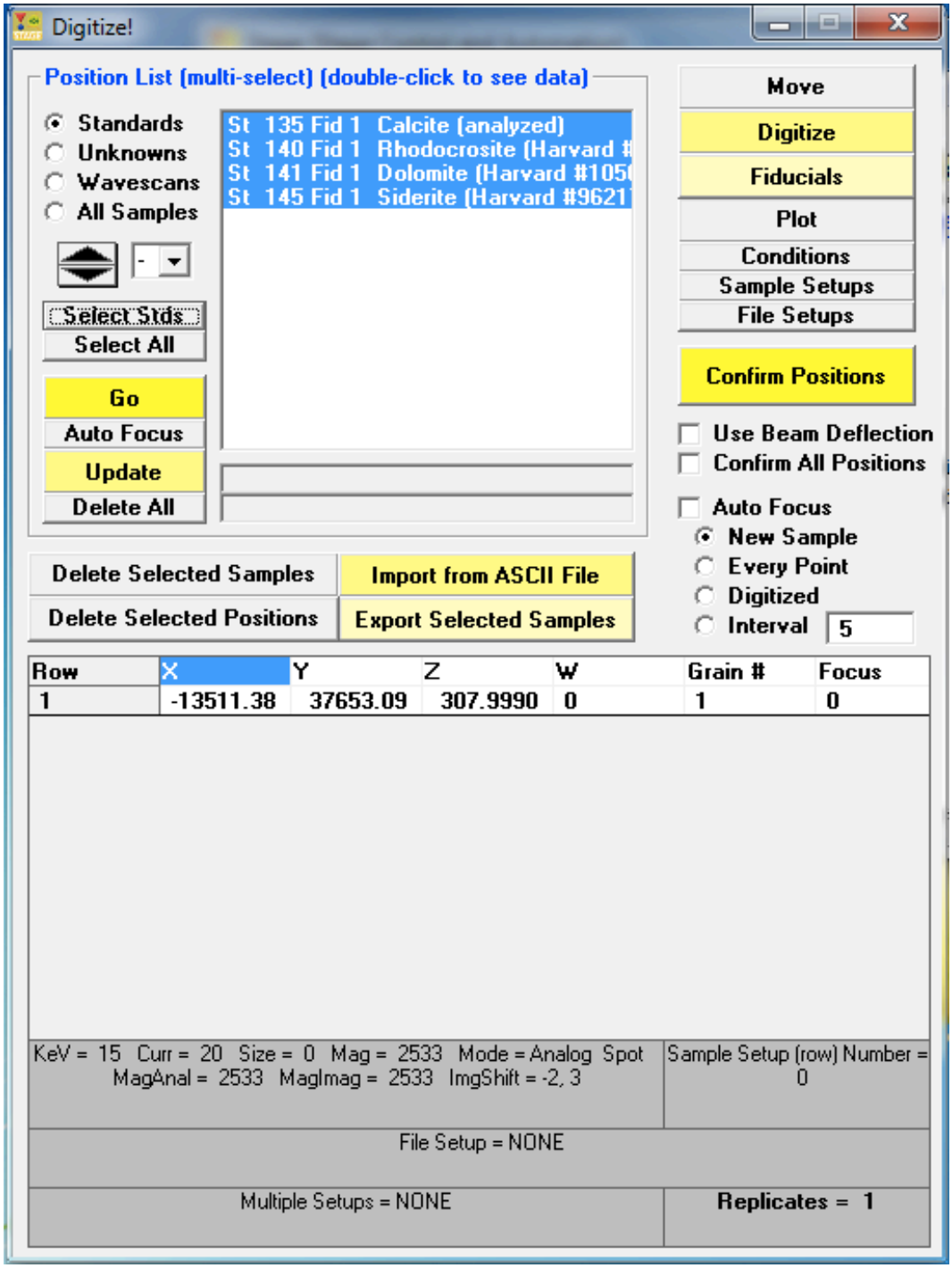
Note, that although only one position per standard need be digitized, if additional points are digitized, PROBE FOR EPMA will automatically utilize them. Otherwise, PROBE FOR EPMA will simply increment the stage X position for each additional acquisition required.

Move the stage to the next standard, select the standard from the list box in the **Digitize Sample Position** window and click the **Single Point(s)** button again. The standard position will be digitized. Continue until all of the remaining standards in the standard block are digitized. In this example the **Digitize!** and **Digitize Sample Positions** dialog boxes would appear as follows.



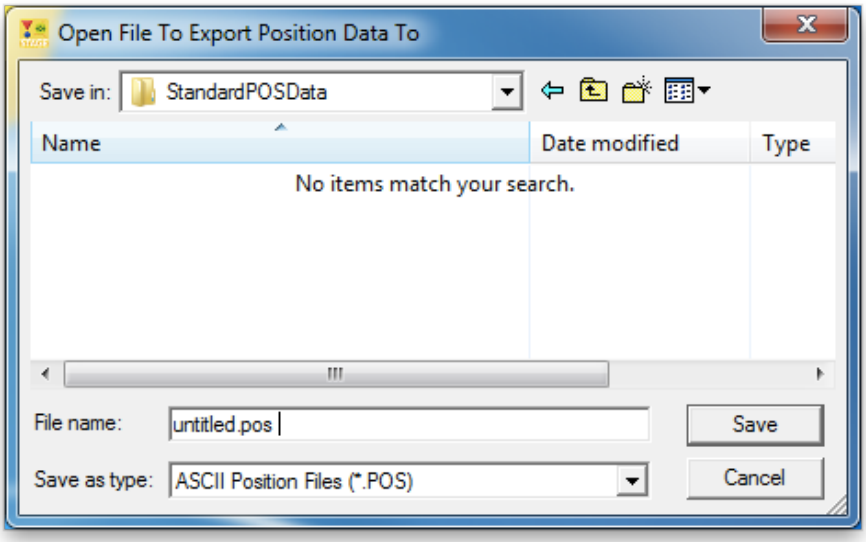
Close the **Digitize Sample Positions** dialog box by clicking the **Close** button in the upper right corner.

Finally, store the new pre-digitized standard coordinates to disk as an ASCII position file (.POS). Select all of the standards using the **Select Stds** button of the **Digitize!** dialog box.

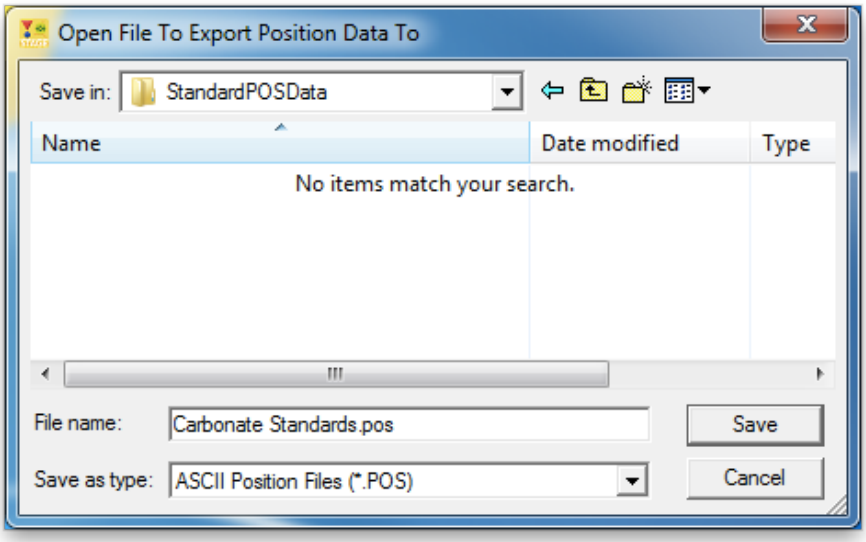


Click the Export Selected Samples button.

This action opens the **Open File To Export Position Data To** window. The default *Save in:* location is specified by the StandardPOSFileDirectory keyword in the PROBEWIN.INI file.

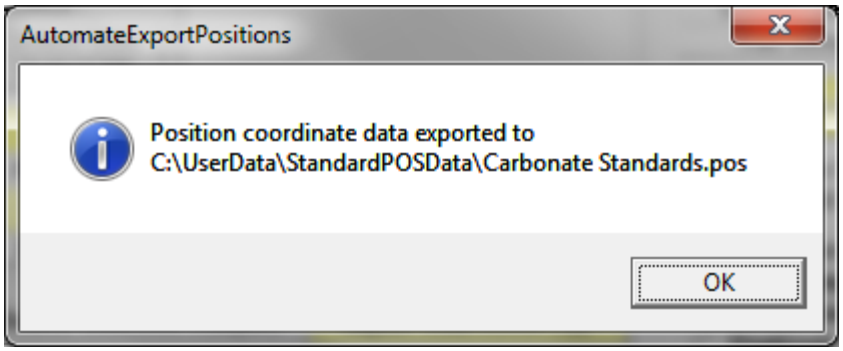


Type in an appropriate *File name:*

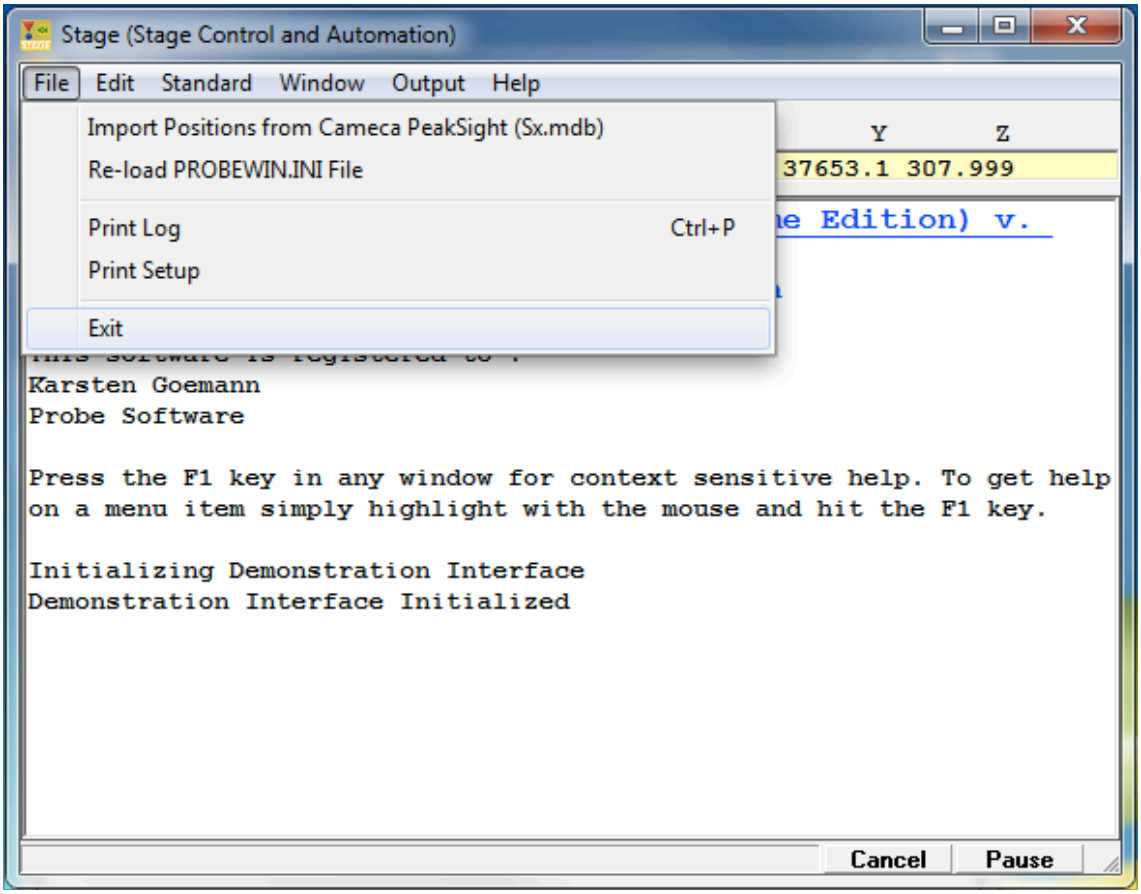


Click the Save button of the **Open File To Export Position Data To** window.

After the positions are written to disk, the **AutomateExportPositions** window appears.



Click the **OK** button to confirm the exported position coordinate data to disk in the **AutomateExportPositions** window. Next, close the **Digitize!** dialog box by clicking the **Close** button. Finally, close STAGE by clicking the **File | Exit** menu.

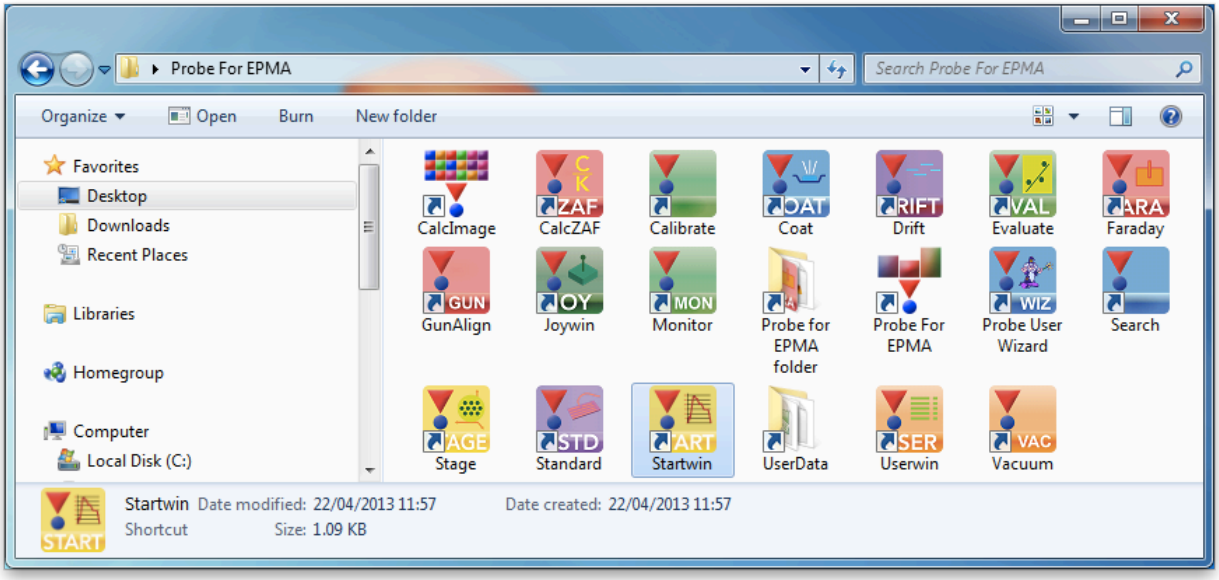


After digitizing all of the standards on the standard mounts and creating various *.POS files, copy these files to another directory and to a backup media.

Beam and Detector Stability

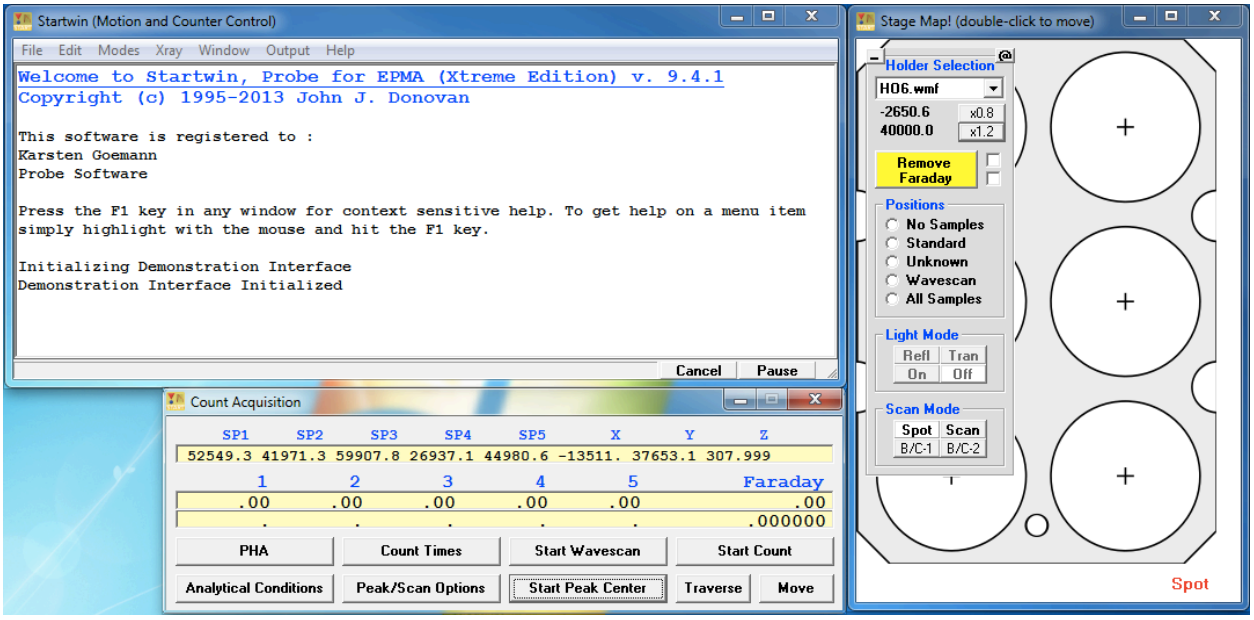
Testing beam (drift) stability is an important step prior to acquiring any quantitative data. The following step-by-step procedure illustrates how to monitor and plot beam current with time. On JEOL microprobes the FARADAY module may also be run for measurement of the beam (see the User's Guide and Reference documentation for further details).

From the Desktop, double click on the yellow Probe for EPMA Software folder, if available. Then, double click on the **Startwin** icon.

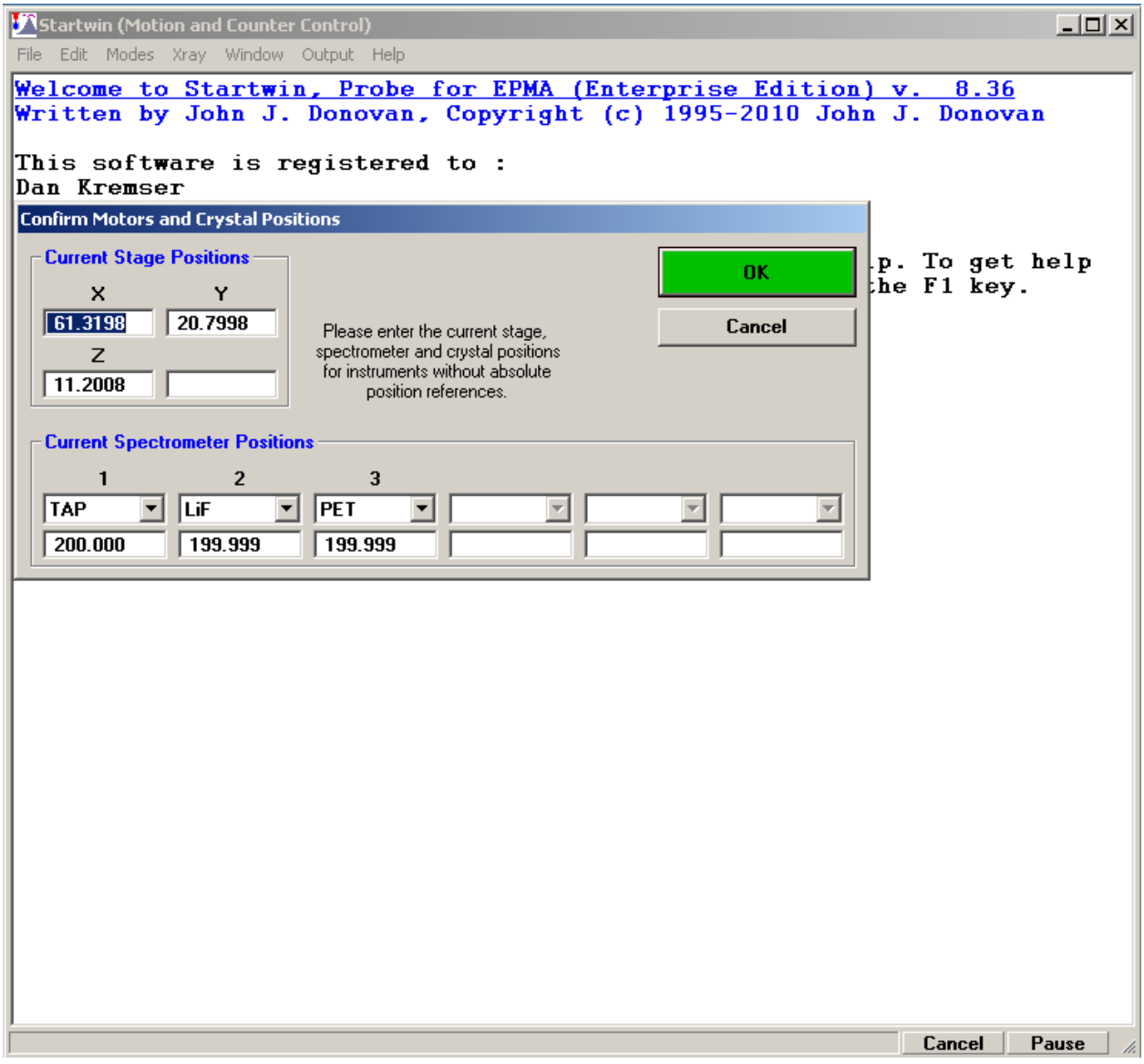


Alternatively, select **Startwin** in the Probe Software group of the Windows Start Menu, or locate and double click on STARTWIN in the Probe for EPMA application directory, which is usually C:\Probe Software\Probe for EPMA under Windows Vista and Windows 7, or C:\Program Files\Probe Software\Probe for EPMA for older operating systems.

This action launches the STARTWIN (Motion and Counter Control) program and opens three windows. The main STARTWIN log window, the **Count Acquisition** window and the first **Stage Map!** are displayed.



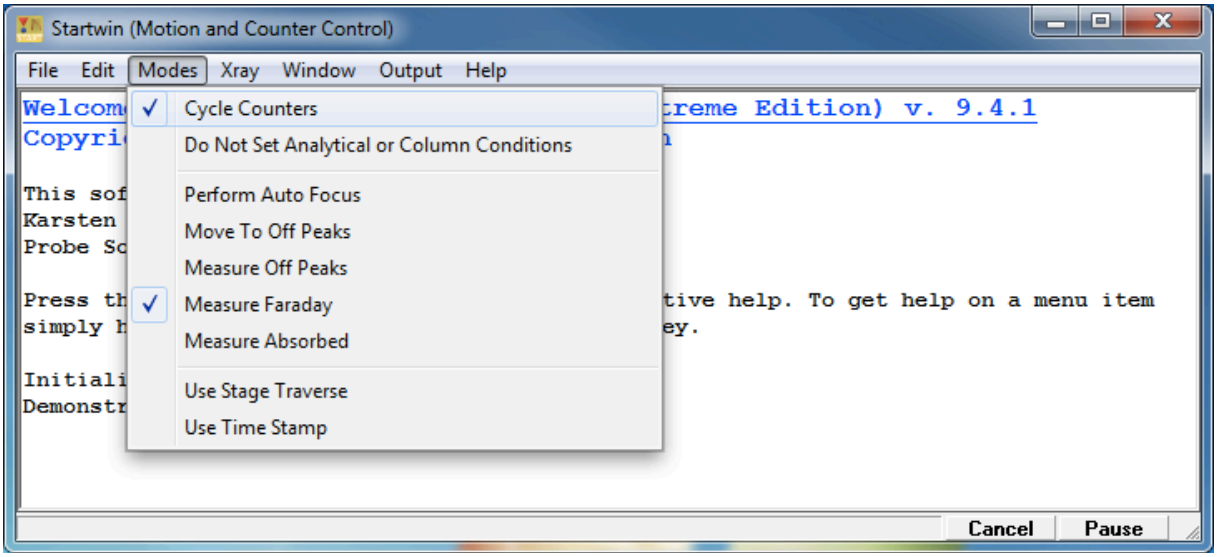
Older microprobes with Advanced MicroBeam automation systems will first see the **Confirm Motor and Crystal Positions** dialog box. Confirm that all of the motors (stage and spectrometer positions) and crystal designations are correctly calibrated. If there is disagreement between the mechanical positions (actual) and the software values, adjust the software values. Use the <tab> key to move between the various *Stage* and *Spectrometer Positions* boxes.



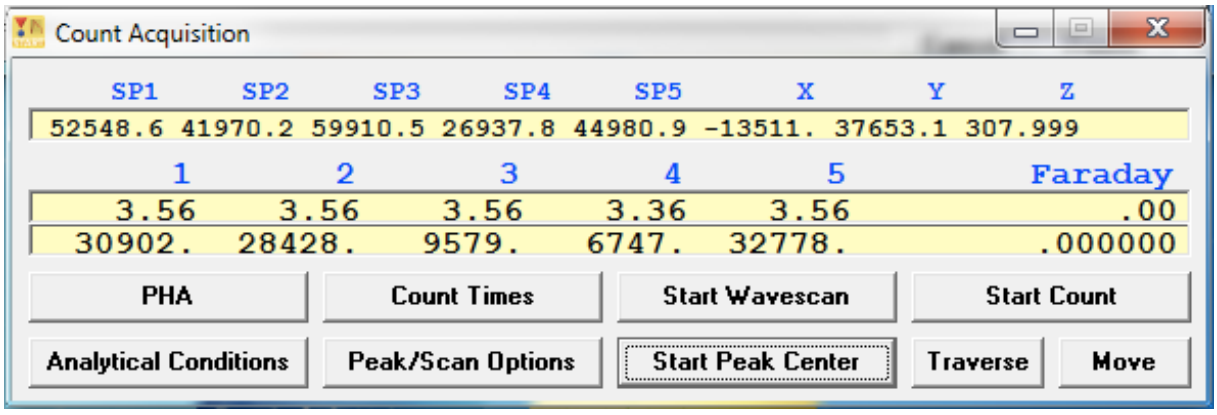
Click the **OK** button to close the **Confirm Motor and Crystal Positions** dialog box when done.

This causes both the **Count Acquisition** window and the **Stage Map!** window to open.

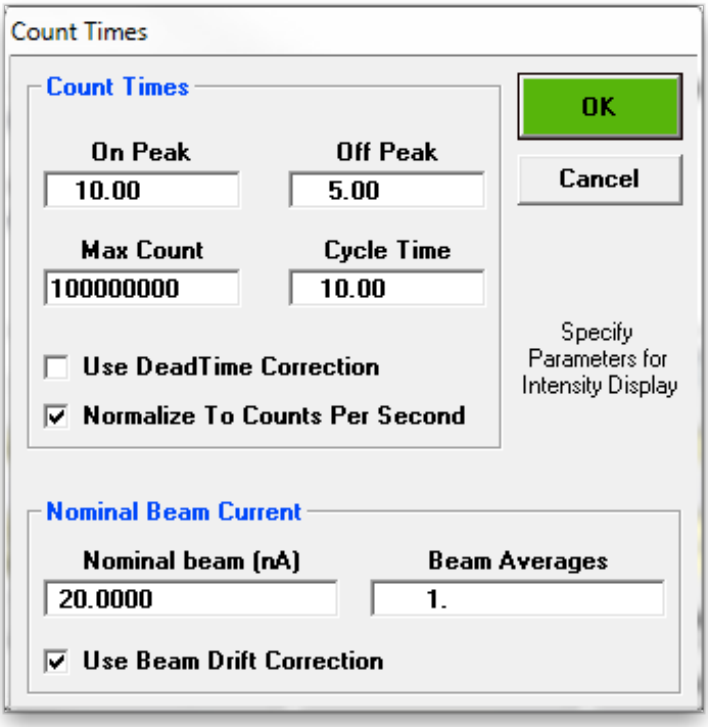
From the STARTWIN log window, select **Modes** from the menu bar and click on **Cycle Counters** from the menu choices. The **Measure Faraday** menu should also be selected.



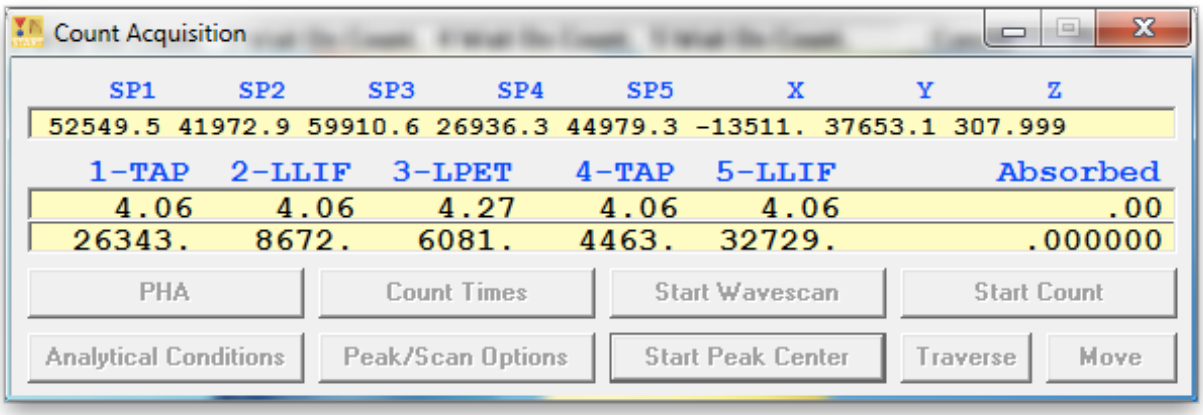
Next, click the **Count Times** button in the **Count Acquisition** window.



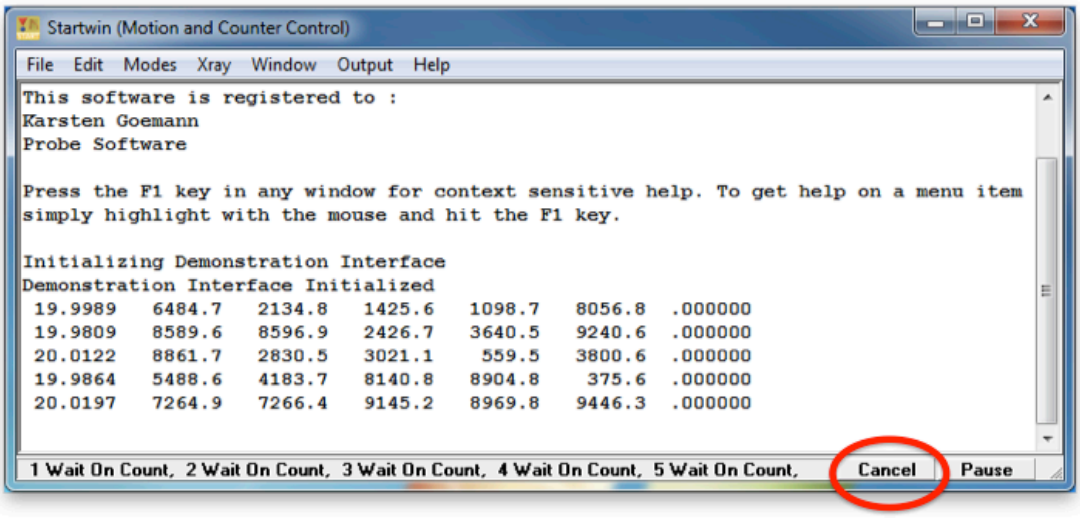
This opens the **Count Times** dialog box. Choose an *On Peak* and *Cycle Time* count time. The *On Peak* time is the time the scalers will count and the *Cycle Time* is the interval of time between successive measurements. Thus, the sum of both numbers is the time between measurements of the beam. Finally, disable the beam drift correction; confirm that the *Use Beam Drift Correction* box is unchecked.



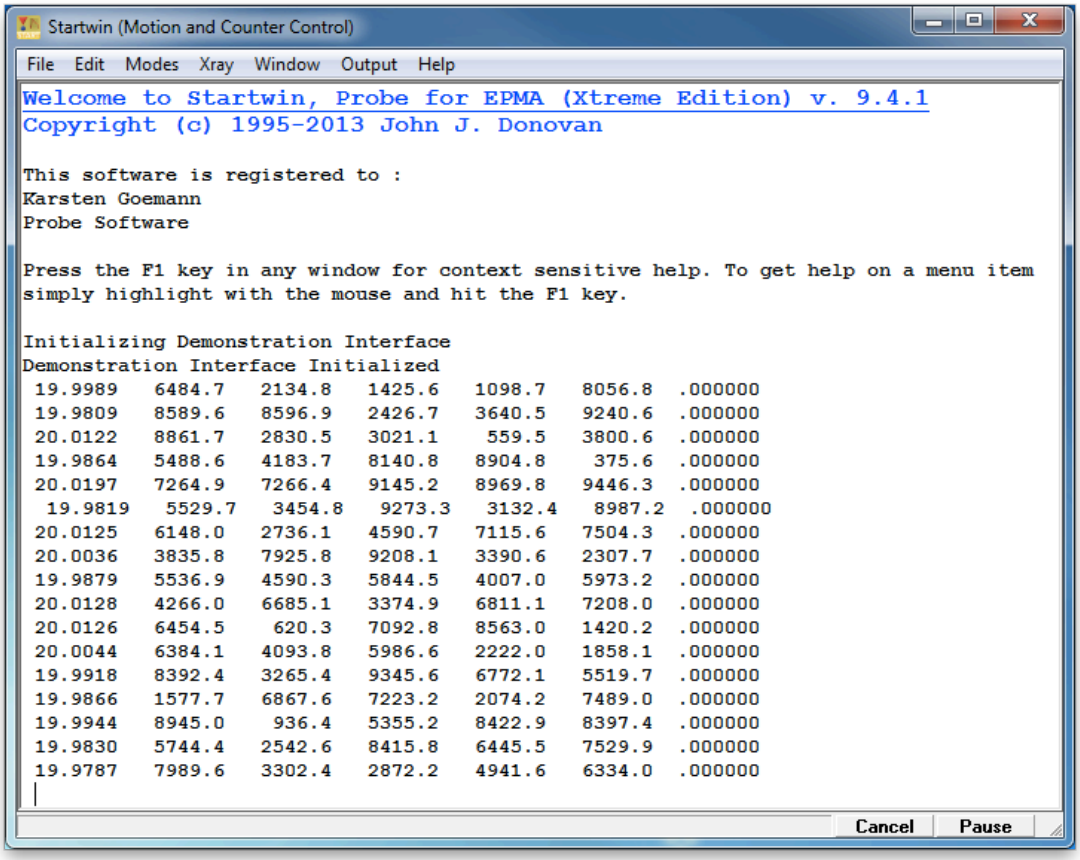
Click the **OK** button returning to the **Count Acquisition** window. Click the **Start Count** button to initiate a continuous cycle of beam current measurements. In this example, a ten second scaler count will be done, followed by a thirty-second countdown and then a Faraday current measurement. This process repeats until the user cancels the loop.



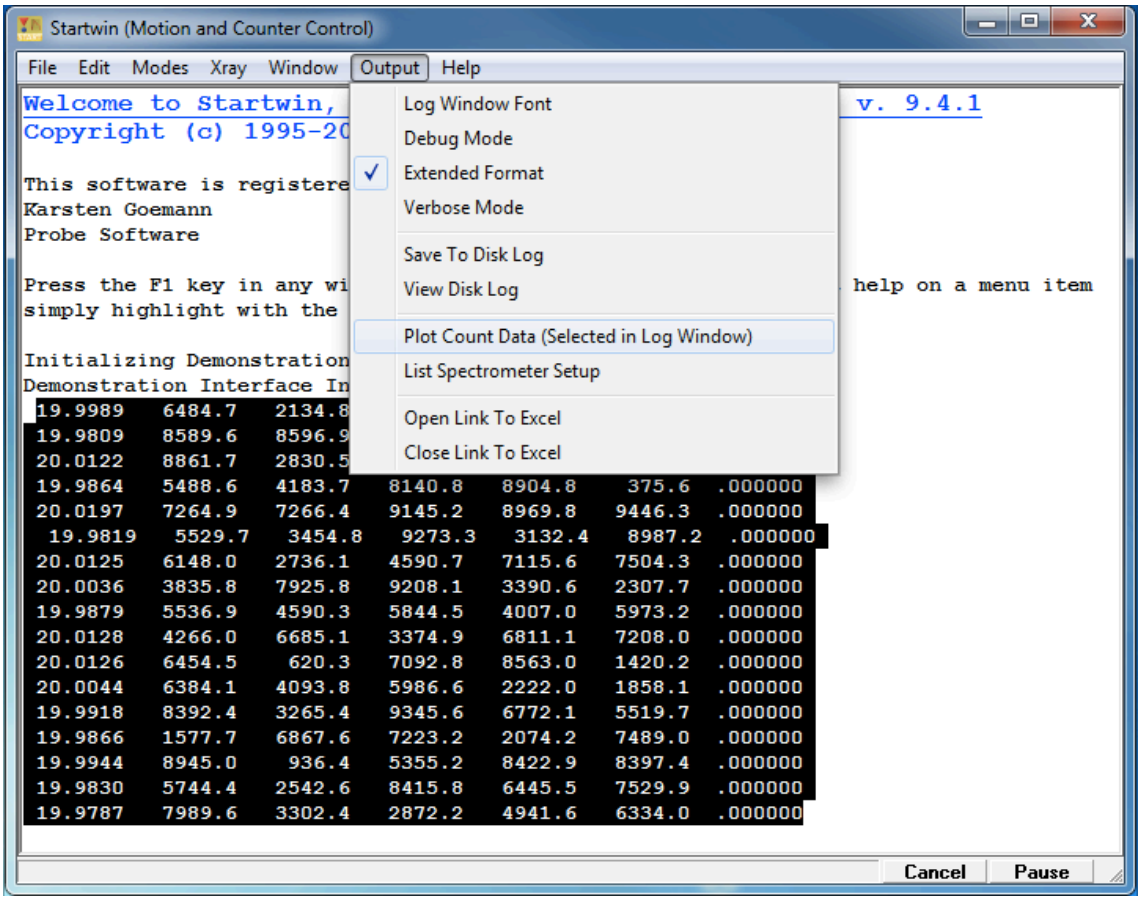
When the user has acquired a suitable number of beam current measurements, click the **Cancel** button in the **Automation Status** bar located at the bottom right side of the STARTWIN log window to stop the acquisition cycle. All log windows in any PROBE FOR EPMA program will have a **Cancel** and **Pause/Continue** buttons in the lower right portion of the window.



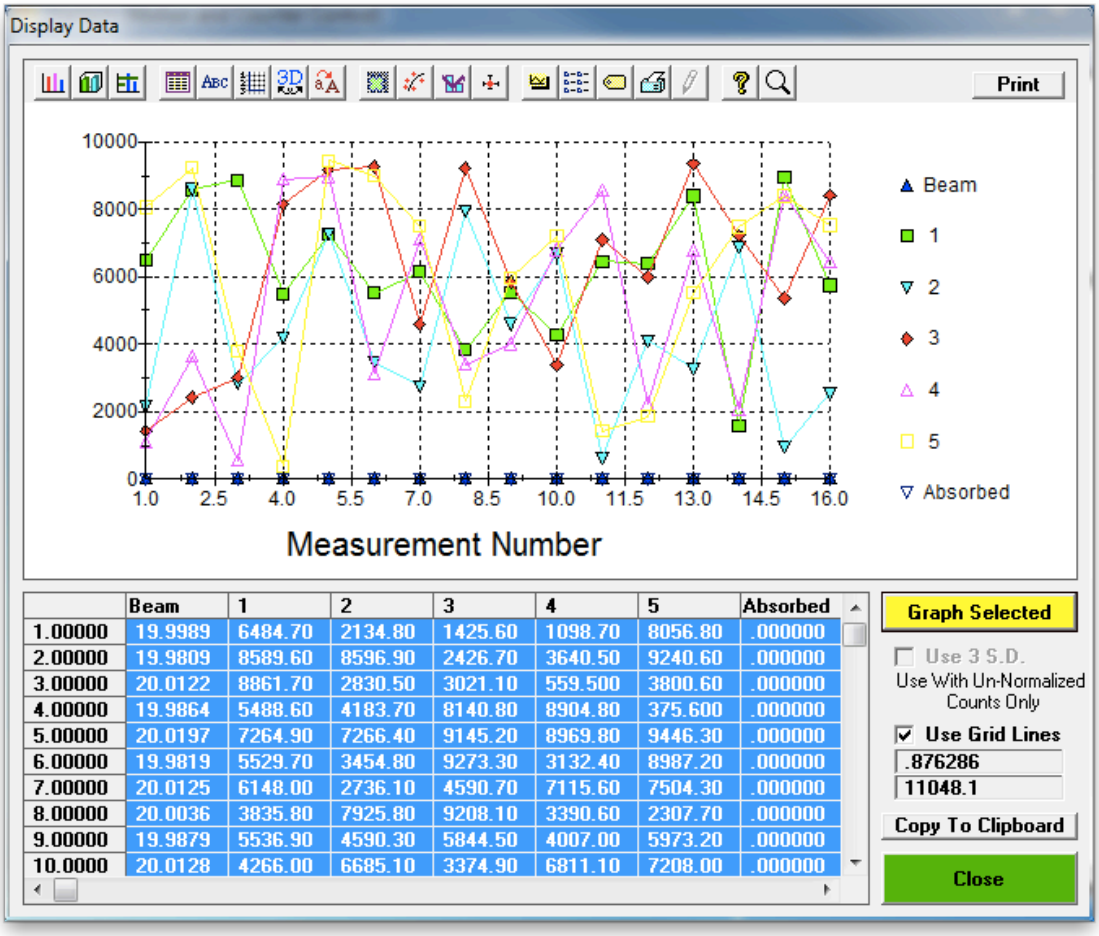
The STARTWIN log window will contain the beam current data acquired so far (in this example, reported in nanoamps). The other five columns represent counts in counts per second acquired by each spectrometer, which is in this case randomly generated by the demonstration mode of the software:



Evaluating the trend between beam current and time may best be viewed in graphical format rather than looking at a long series of numbers. Use the mouse to select the data set to plot. Then, select **Output** from the menu bar and click **Plot Count Data (Selected in Log Window)** from the drop-down menu choices.



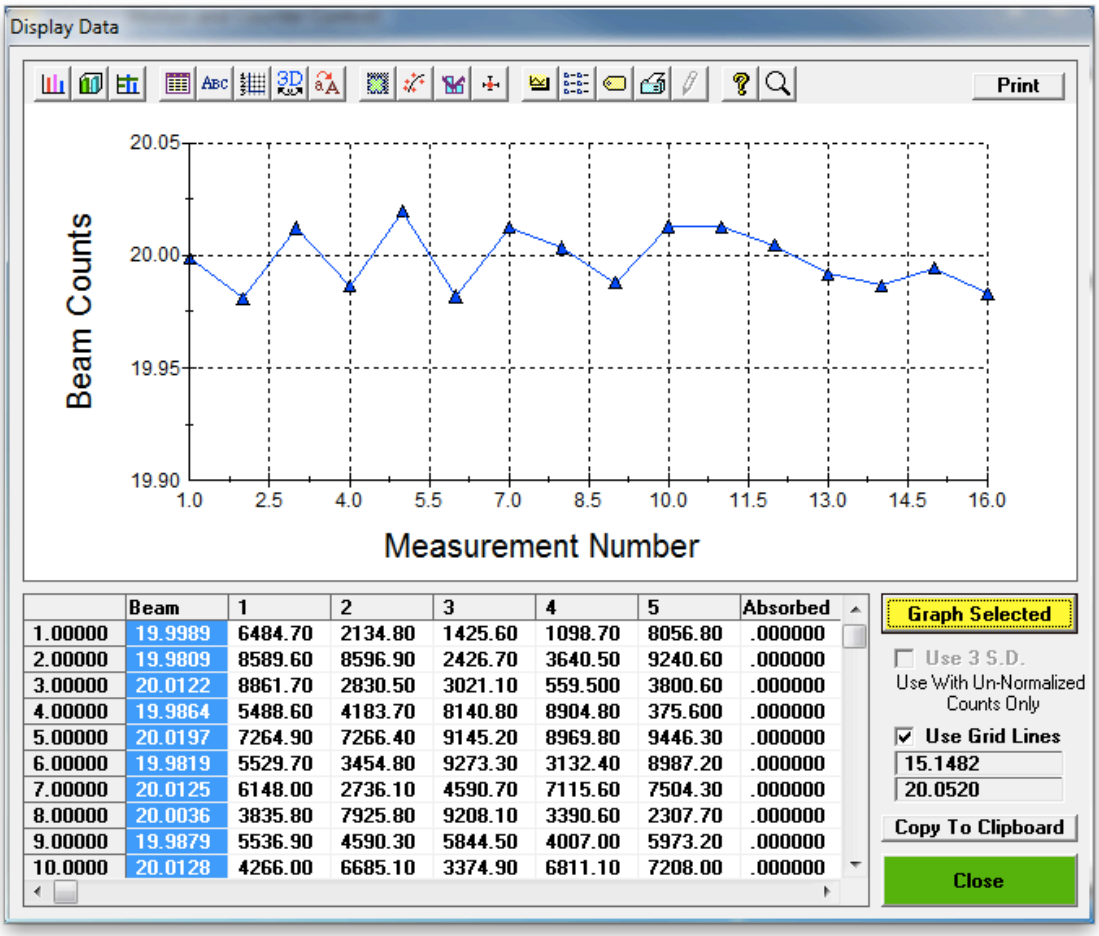
This opens the **Display Data** window.



	Beam	1	2	3	4	5	Absorbed
1.00000	19.9989	6484.70	2134.80	1425.60	1098.70	8056.80	.000000
2.00000	19.9809	8589.60	8596.90	2426.70	3640.50	9240.60	.000000
3.00000	20.0122	8861.70	2830.50	3021.10	559.500	3800.60	.000000
4.00000	19.9864	5488.60	4183.70	8140.80	8904.80	375.600	.000000
5.00000	20.0197	7264.90	7266.40	9145.20	8969.80	9446.30	.000000
6.00000	19.9819	5529.70	3454.80	9273.30	3132.40	8987.20	.000000
7.00000	20.0125	6148.00	2736.10	4590.70	7115.60	7504.30	.000000
8.00000	20.0036	3835.80	7925.80	9208.10	3390.60	2307.70	.000000
9.00000	19.9879	5536.90	4590.30	5844.50	4007.00	5973.20	.000000
10.0000	20.0128	4266.00	6685.10	3374.90	6811.10	7208.00	.000000

While all data columns were selected by the mouse operation previously, the user may plot a single column of data by clicking the column label of the desired data and then clicking the **Graph Selected** button.

Below, *Beam Counts* versus *Measurement Number* (time) are graphed and the overall beam stability with time may be judged. Here, the beam measurements fluctuate slightly around a value of 20 nA.



	Beam	1	2	3	4	5	Absorbed
1.00000	19.9989	6484.70	2134.80	1425.60	1098.70	8056.80	.000000
2.00000	19.9809	8589.60	8596.90	2426.70	3640.50	9240.60	.000000
3.00000	20.0122	8861.70	2830.50	3021.10	559.500	3800.60	.000000
4.00000	19.9864	5488.60	4183.70	8140.80	8904.80	375.600	.000000
5.00000	20.0197	7264.90	7266.40	9145.20	8969.80	9446.30	.000000
6.00000	19.9819	5529.70	3454.80	9273.30	3132.40	8987.20	.000000
7.00000	20.0125	6148.00	2736.10	4590.70	7115.60	7504.30	.000000
8.00000	20.0036	3835.80	7925.80	9208.10	3390.60	2307.70	.000000
9.00000	19.9879	5536.90	4590.30	5844.50	4007.00	5973.20	.000000
10.0000	20.0128	4266.00	6685.10	3374.90	6811.10	7208.00	.000000

The numeric value of any point on the graph may be read by placing the mouse cursor over the data point and viewing its value in the two windows directly above the **Close** button (bottom right).

Click the **Close** button to return to the STARTWIN log window.

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Quantitative Measurement Run

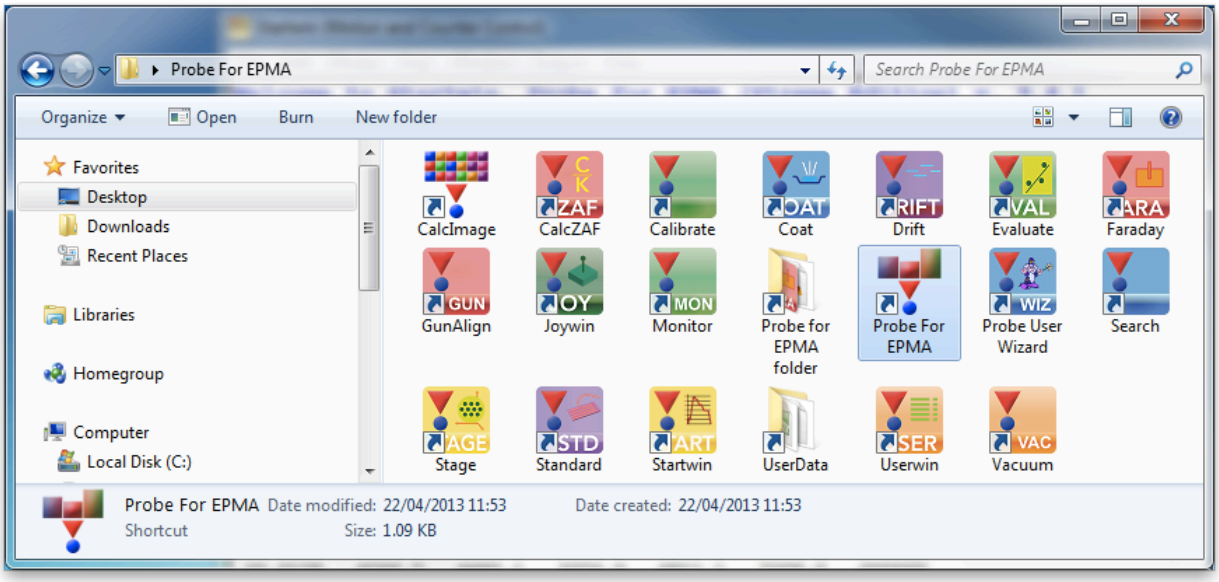
Introduction

This chapter illustrates step by step how to set up a new PROBE FOR EPMA quantitative run and how to analyze ten elements in an unknown pyroxene, which is a type of silicate mineral. This documentation was produced using a five spectrometer Cameca SX100 electron microprobe and on a computer running in demo mode. Your particular run may look very different depending on the specific configuration of your microprobe. This document should be used in conjunction with the User's Guide and Reference documentation, on-line help, Probe for EPMA Quick Start Guide and the PROBEUSERWIZARD program.

This run will demonstrate some of the basic and more advanced features of the PROBE FOR EPMA program. These include the use of manual and automated spectrometer peaking, manual and automated standard count and unknown sample acquisition, mean atomic number (MAN) background corrections, and automated spectral interference corrections. The use of pre-digitized standard positions, the unique wavescan option, off-peak adjustment capabilities and data output methods will be illustrated.

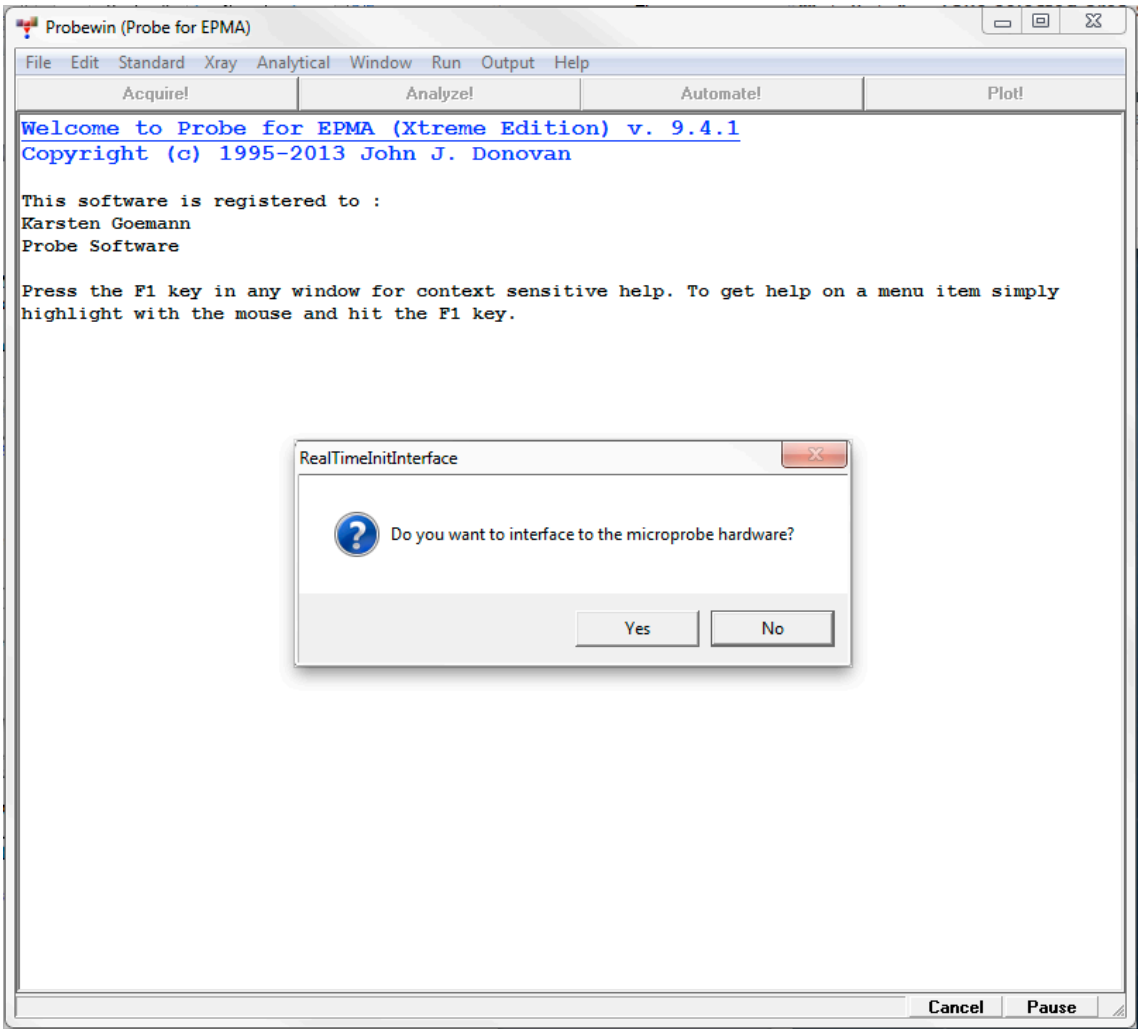
Opening Probe for EPMA

From the Desktop, double-click on the yellow Probe for EPMA Software folder, if available. Double click on the **Probe for EPMA** icon.



Alternatively, select Probe For EPMA from the Probe Software Group in the Windows Start Menu, or locate and double click on PROBEWIN in the Probe for EPMA application directory, which is usually C:\Probe Software\Probe for EPMA under Windows Vista and Windows 7, or C:\Program Files\Probe Software\Probe for EPMA for older operating systems.

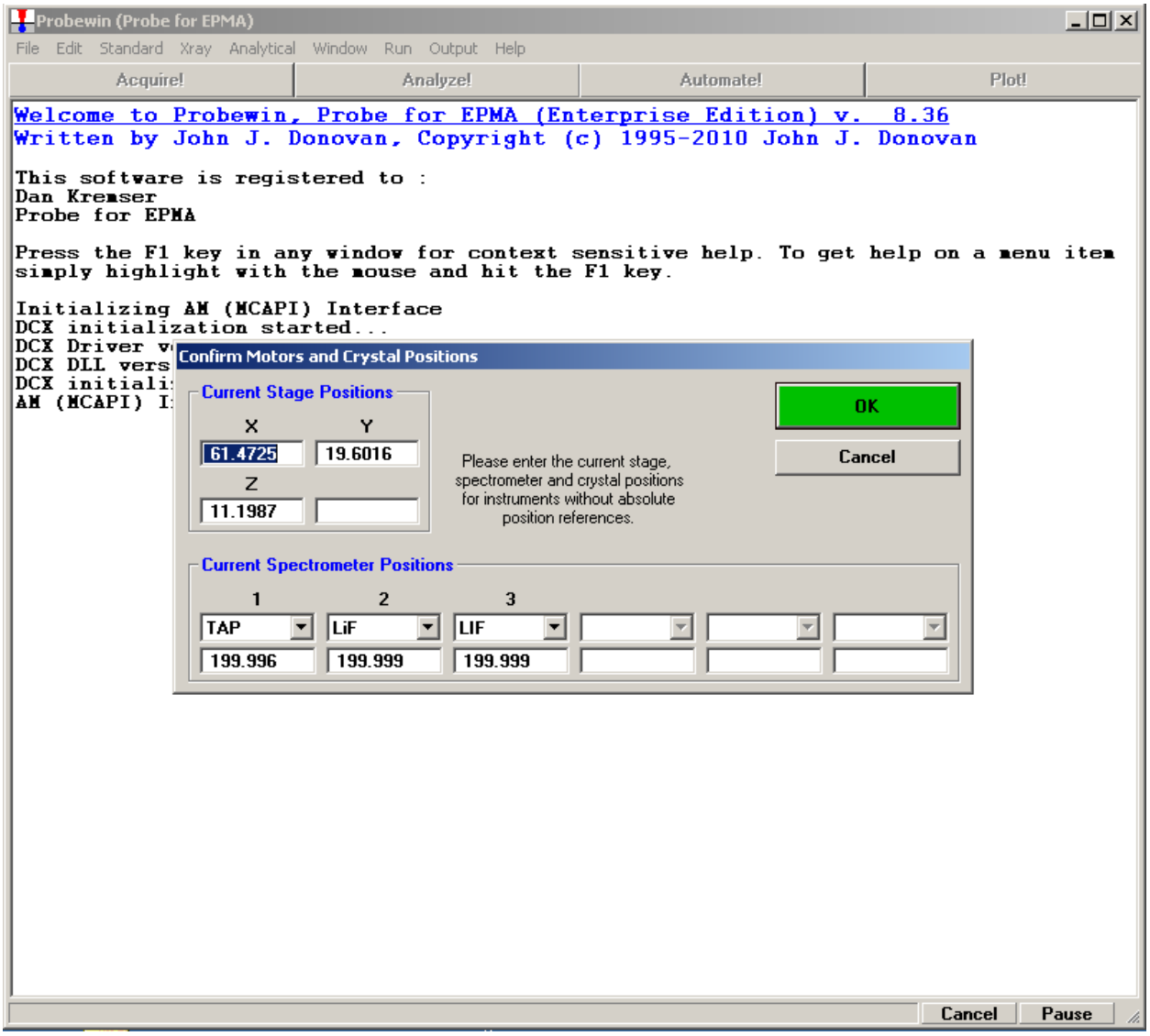
Upon launching PROBEWIN (Probe For EPMA), the main log window appears along with the **RealTimeInitInterface** window as illustrated below. To collect real time data click the **Yes** button. The program can also be run off-line without the microprobe interface to re-process previously acquired data or on another computer.



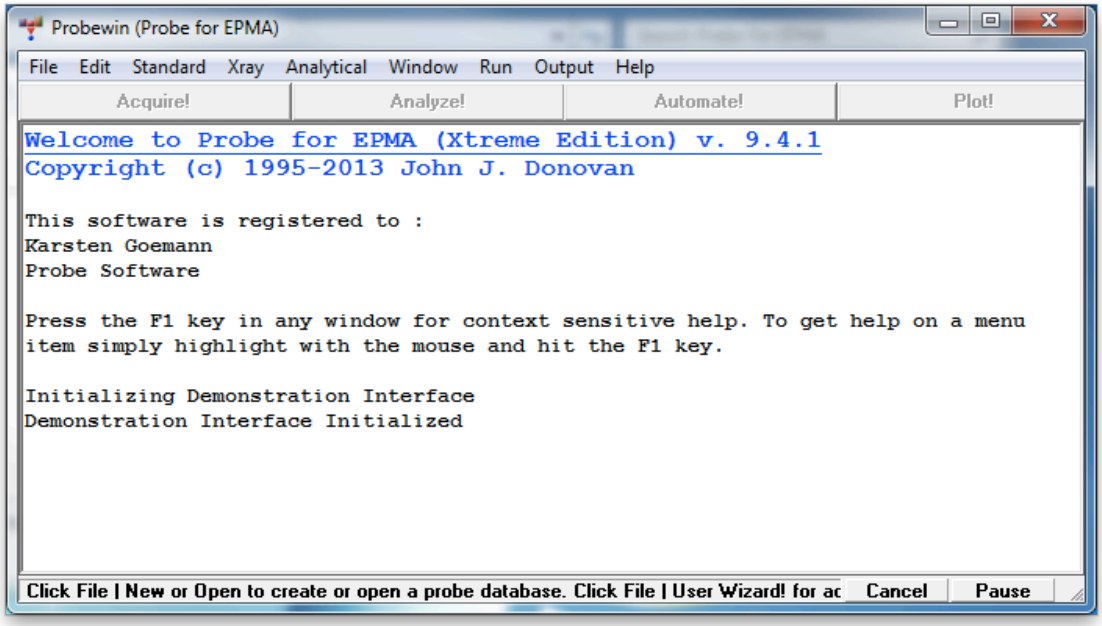
Note if this is the first time running PROBEWIN, several database files are created; SETUP.MDB, SETUP2.MDB, and SETUP3.MDB via the **SetupOpenNewFile** window. Click **Yes** to create these database files if prompted.

Older microprobes with Advanced MicroBeam automation systems will next see the **Confirm Motor and Crystal Positions** dialog box. Confirm that all of the motors (stage and spectrometer positions) and crystal designations are correctly calibrated. If there is disagreement between the mechanical positions (actual) and the software values, adjust the software values. Use the <tab> key to move between the various *Stage* and *Spectrometer Positions* boxes.

Click the **OK** button after you have finished to close the **Confirm Motor and Crystal Positions** dialog box.

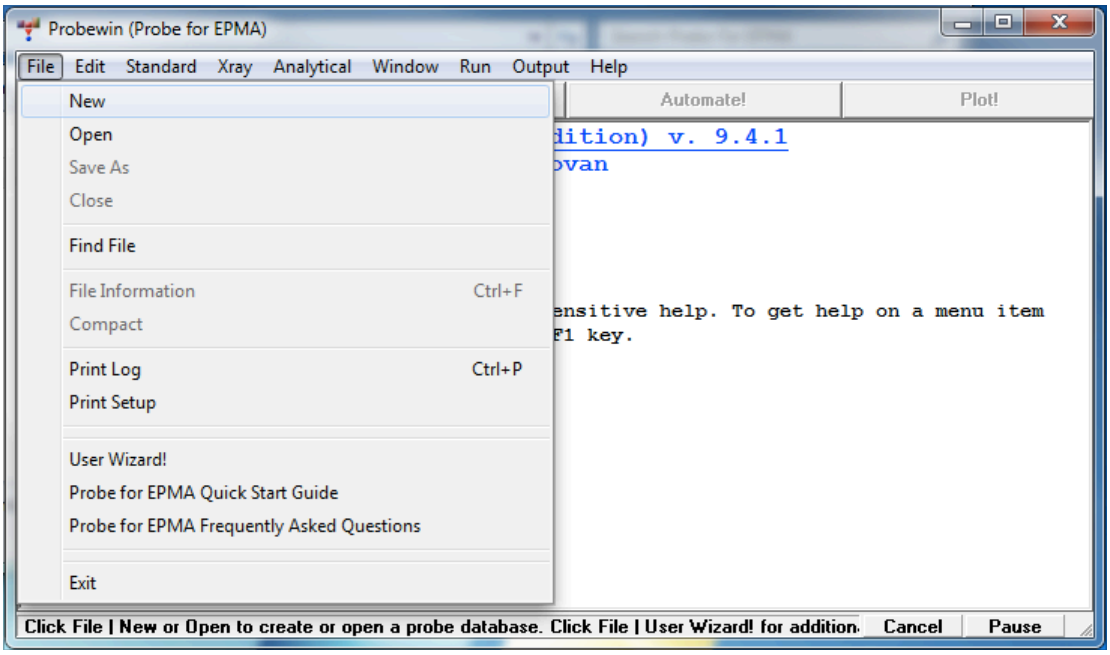


The main PROBE FOR EPMA log window is now visible as seen below.

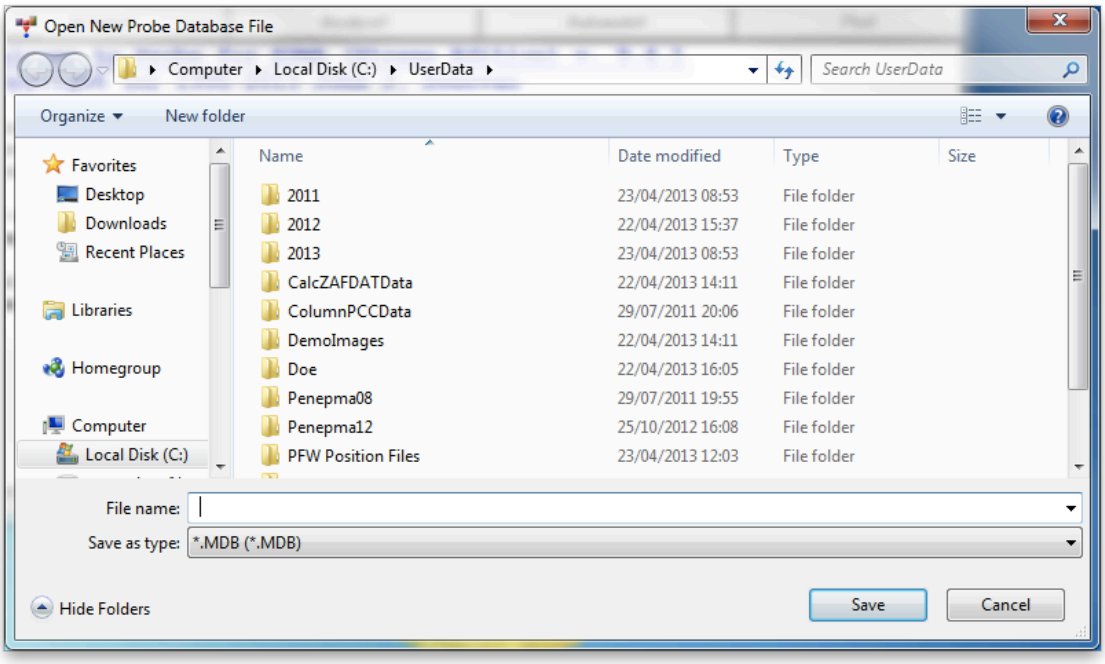


Creating a New Run

To create a new sample run, select **File** from the menu bar and click **New** from the menu.



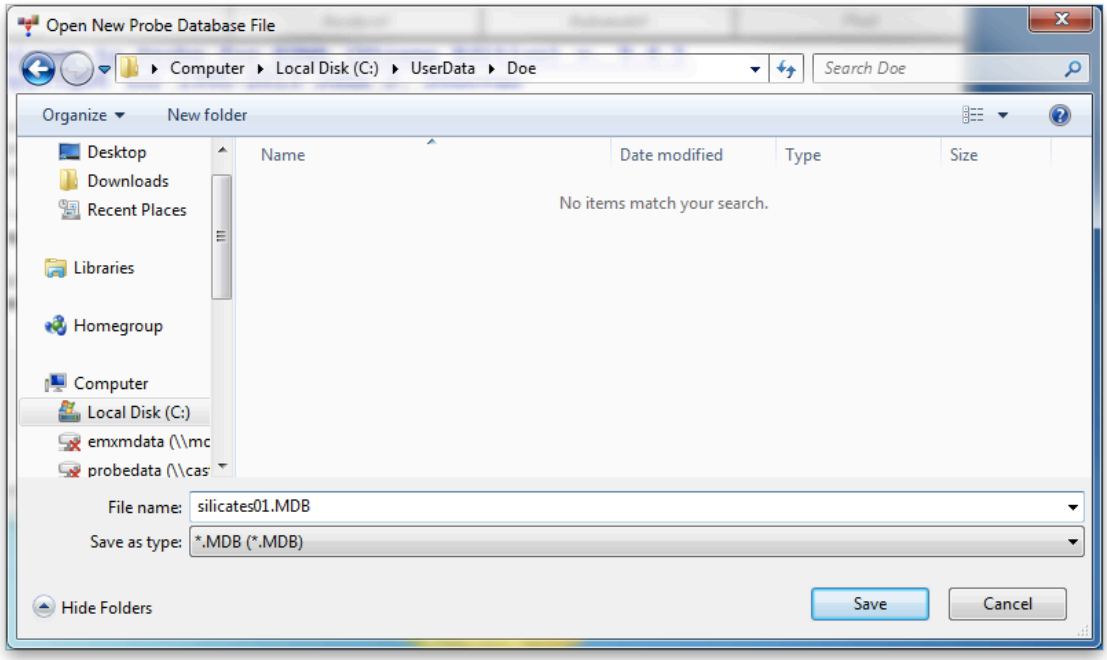
The **Open New Probe Database File** dialog box opens.



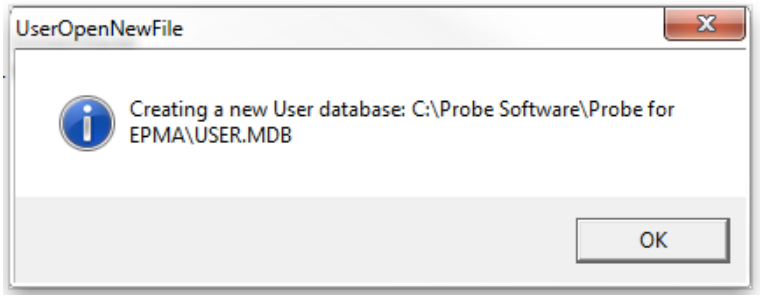
Change the directory if desired and type an appropriate run name into the *File name:* text box.

The initial *Save in:* location is specified by the UserDataDirectory keyword in the PROBEWIN.INI file.

In this example, a new file designated SILICATES01.MDB will be created in the Doe directory. Any existing old runs may be re-opened to acquire additional data or used as a "setup" file for starting a new run. This will be the first .MDB file in this folder.

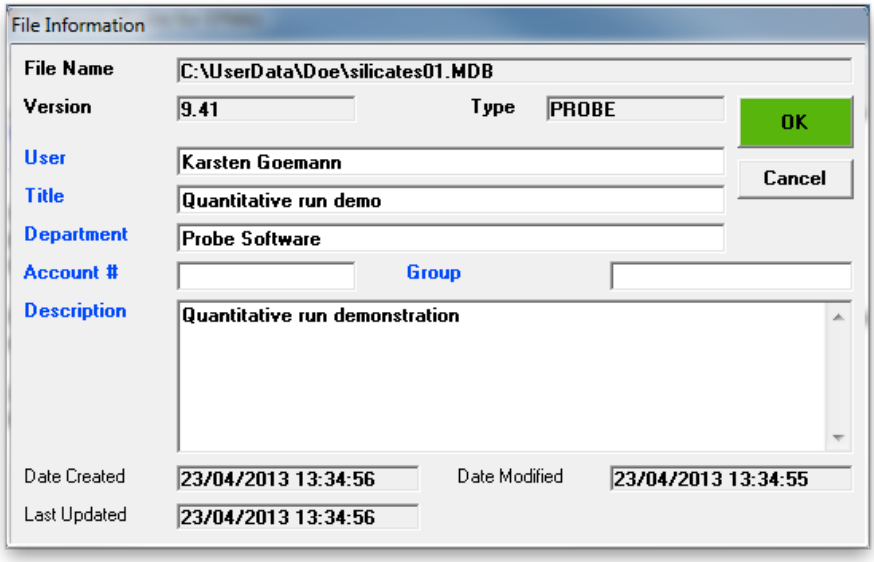


Close the **Open New Probe Database File** window by clicking the **Save** button. First time users will see the creation of a new User database as shown below.

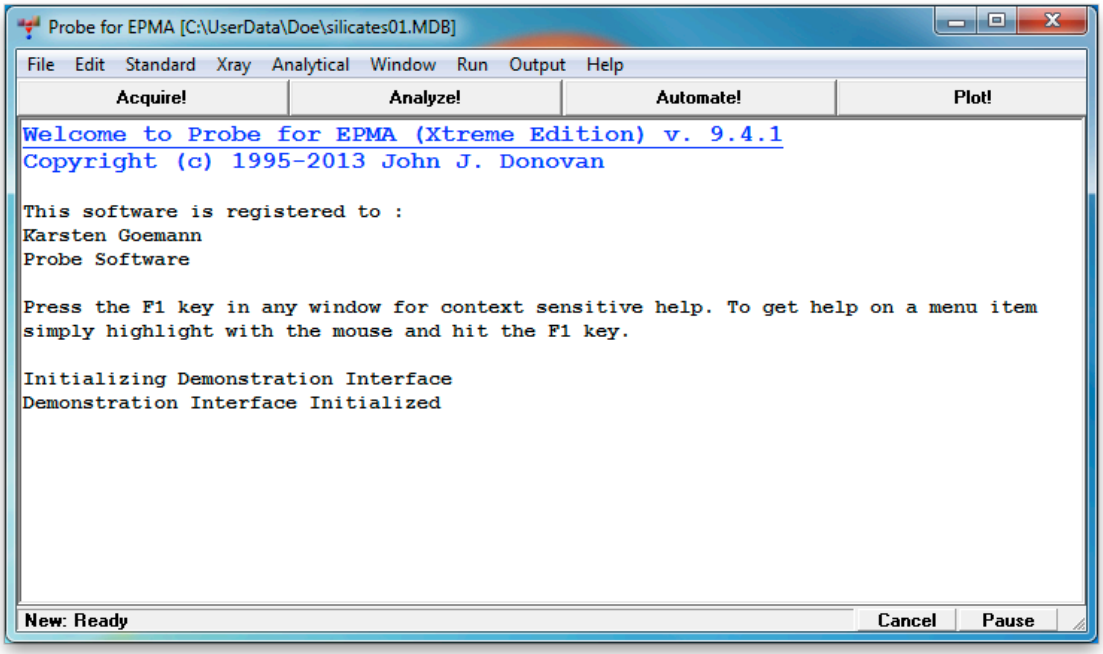


Clicking the **OK** button opens the **File Information** dialog box.

Enter the relevant information for the new run into the *User*, *Title*, and other *Description* text boxes. Use the <tab> key to move between text boxes. When finished, click the **OK** button.



This returns the program to the main PROBE FOR EPMA log window. Now the four main Probe buttons **Acquire!**, **Analyze!**, **Automate!**, and **Plot!** become active.

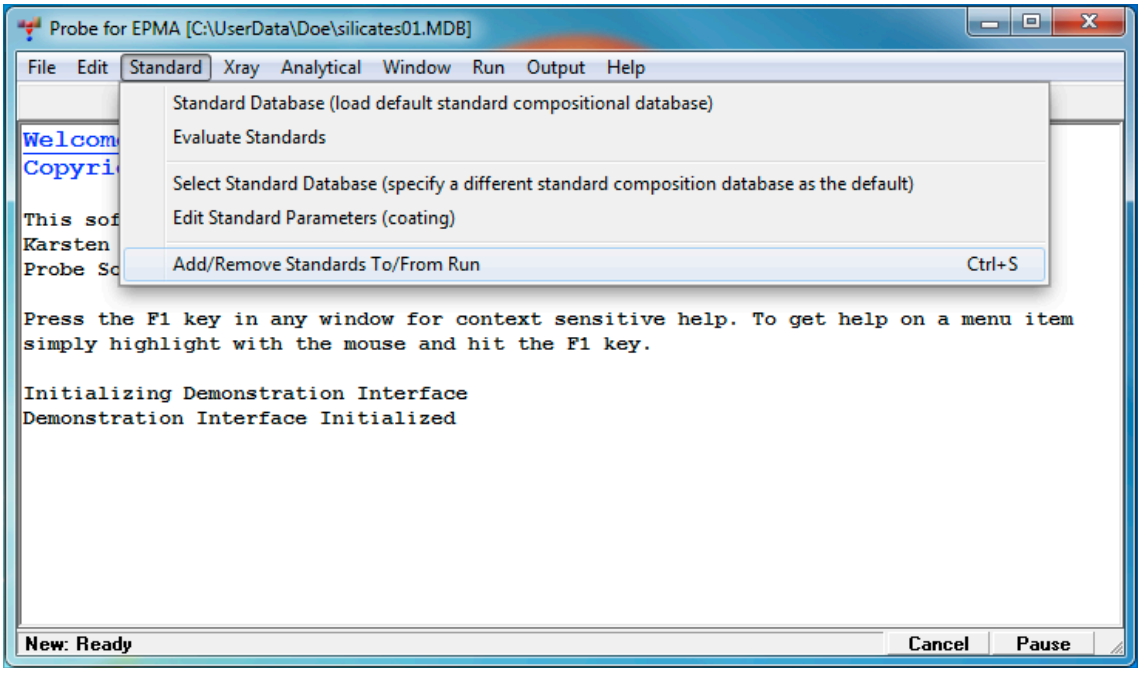


It is assumed that the user has previously chosen the elements to be analyzed, decided on initial standard assignments and has worked through the assignment of spectrometers for each element to be collected. Of course, additional elements and standards may be added or deleted at any time.

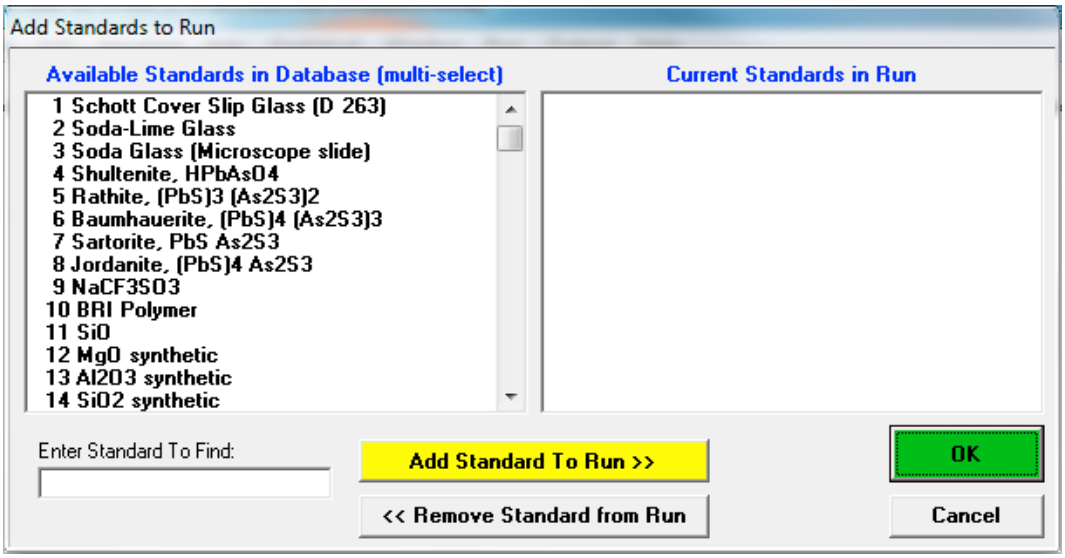
Parameter Initialization

Analytical Standard Selection

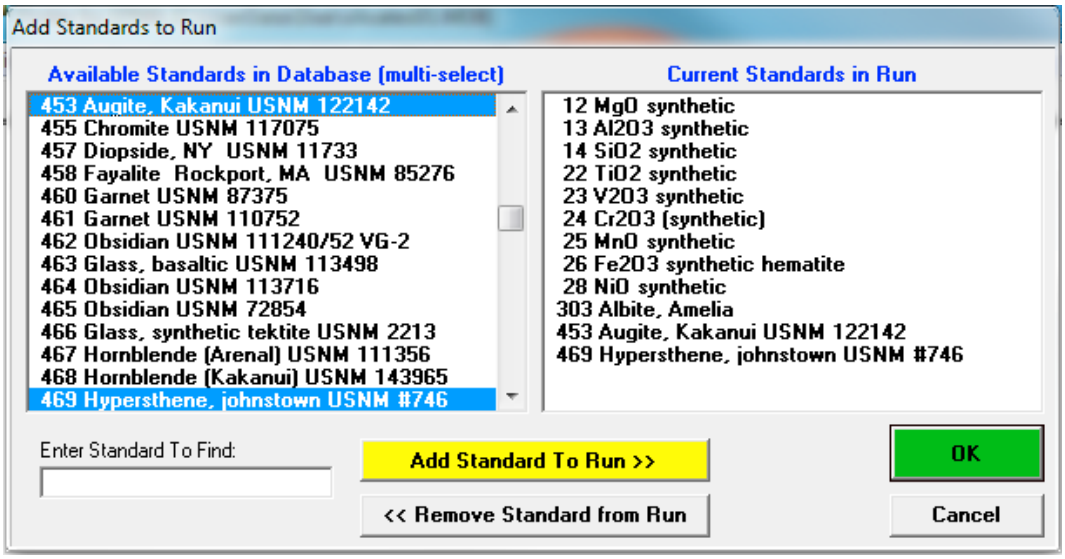
Select the analytical standards to be used in the new probe run. From the main PROBE FOR EPMA log window, click **Standard** from the menu bar and select **Add/Remove Standards To/From Run** from the menu.



This opens the **Add Standards to Run** dialog box.



All previously entered standards in the default standard database are accessible. Scroll through the *Available Standards in Database* list box to find the standards to be used in this run. Select primary analytical standards, secondary standards for method validation, and the MAN background standards. The latter are used for background calculation and must not contain the element they are used for, so for example a few silicon free standards should be selected for Si background calculation. Standards may serve more than one purpose, e.g. pure silica could be used as Si primary standard and MAN standard for all other elements. Select each and click the **Add Standard To Run** >> button to add each to the *Current Standards in Run* list box.

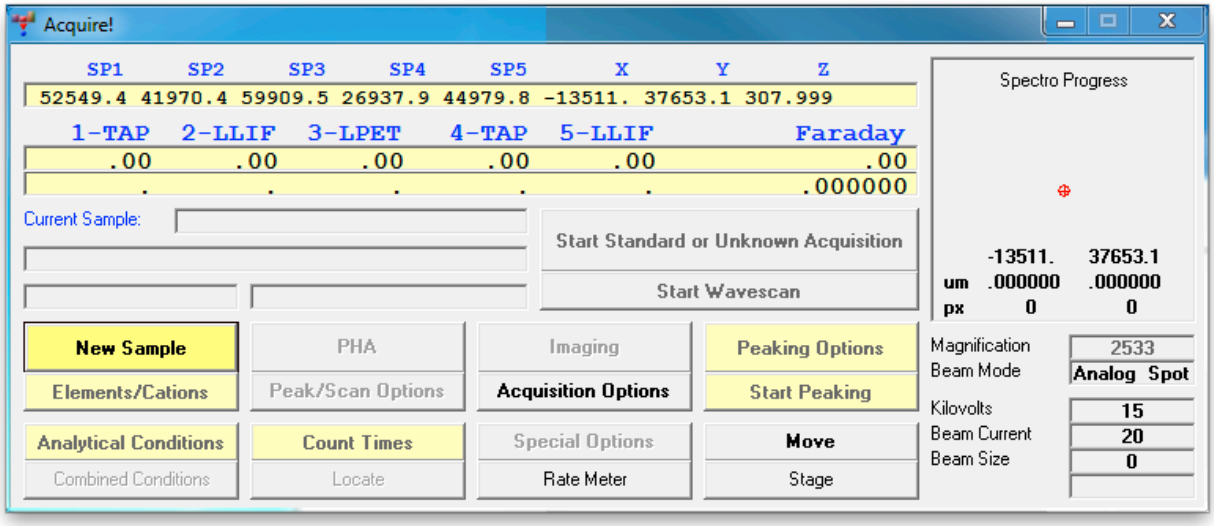


Click the **OK** button of the **Add Standards to Run** window when finished selecting standards. This returns the program to the main log window.

Creating a New Sample

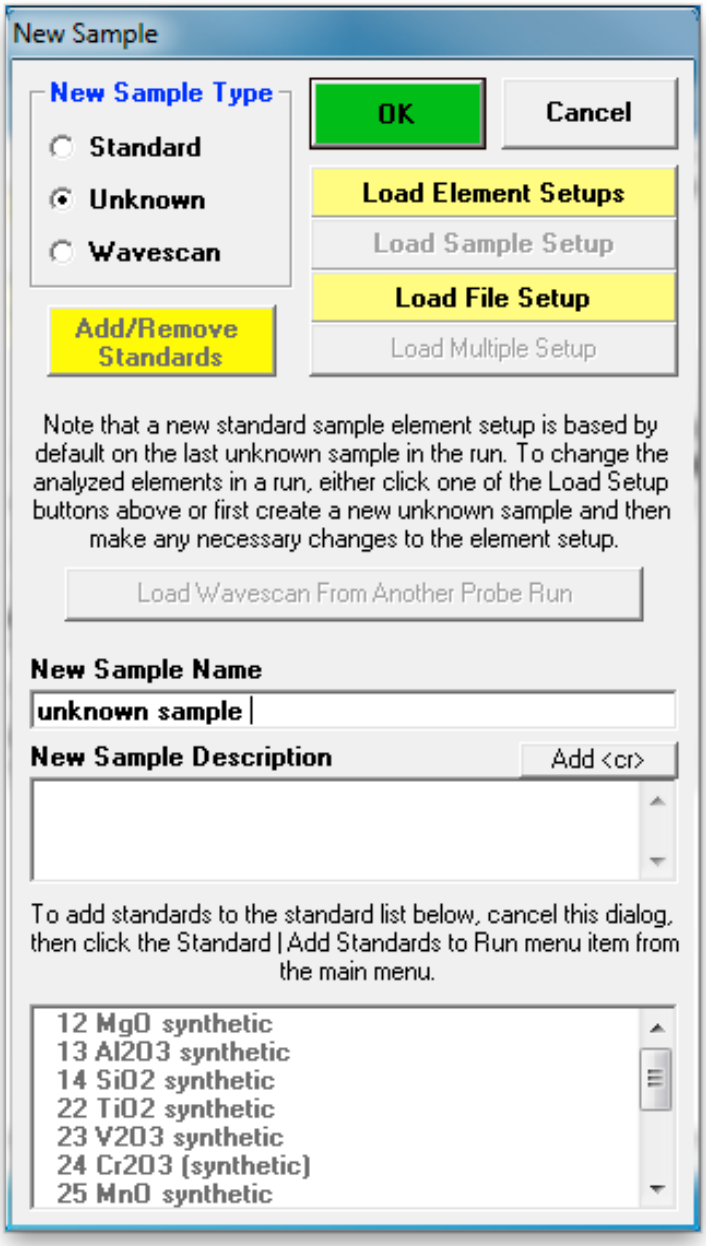
Click the **Acquire!** button in the main PROBE FOR EPMA log window. This action opens the **Acquire!** dialog box. Note, not all buttons are active.

The first task is to create an initial sample as a template that can be populated with the elements the user wishes to analyze.

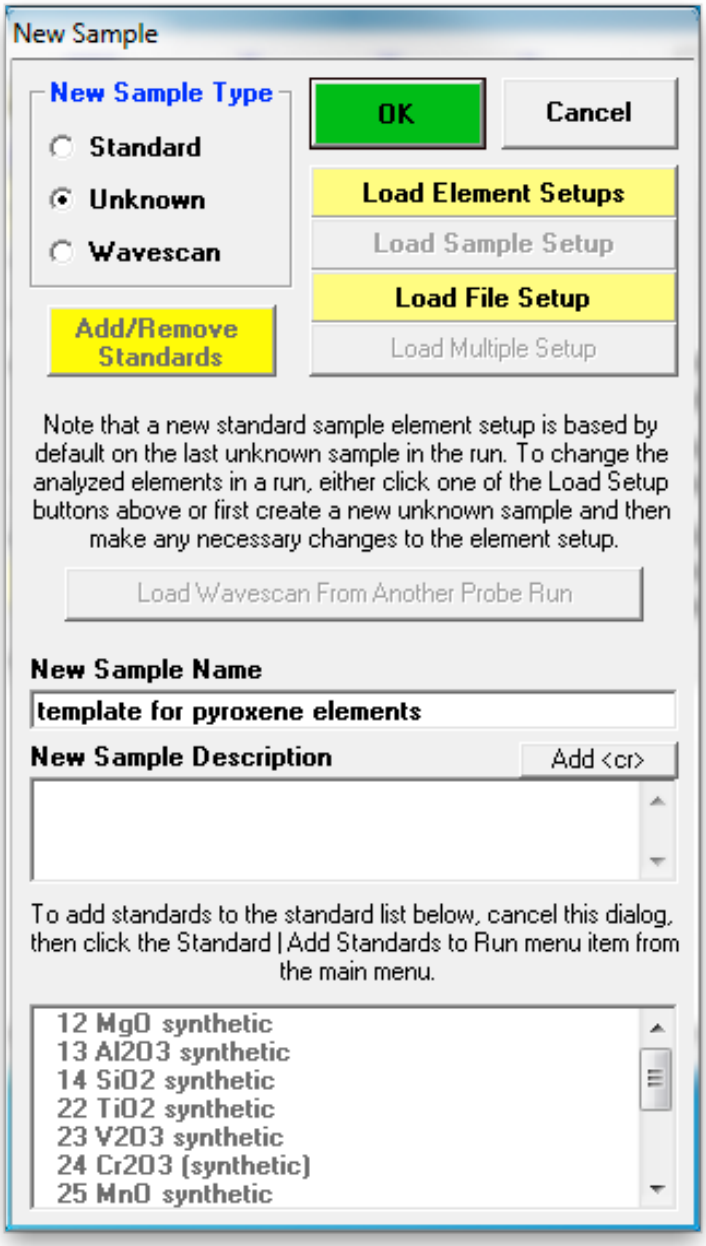


Click the **New Sample** button of the **Acquire!** dialog box.

This opens the **New Sample** dialog box.

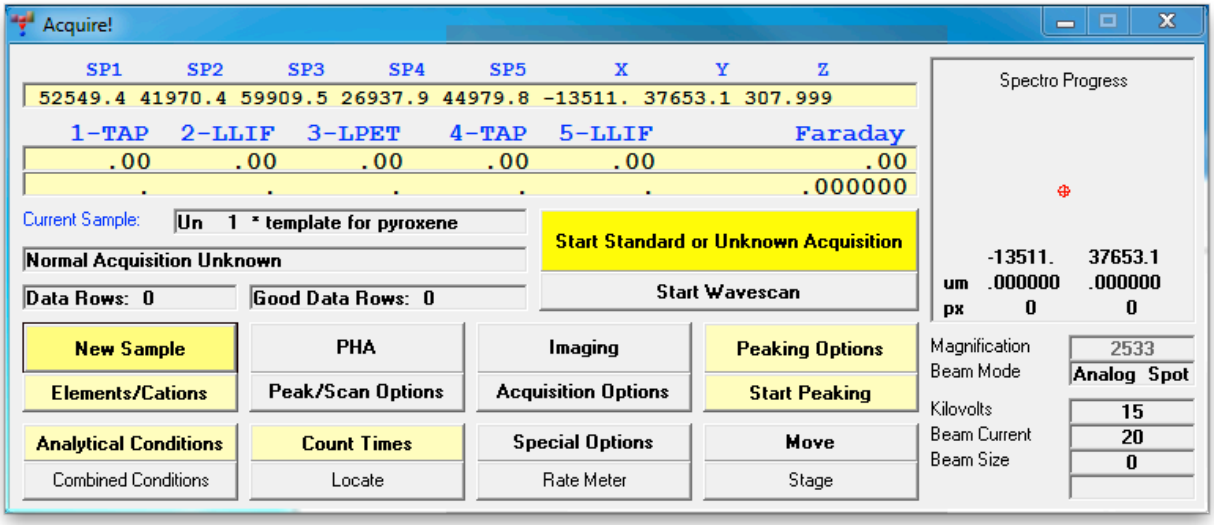


Select *Unknown* from the *New Sample Type* buttons. Type an appropriate sample name and description into the *New Sample Name* and *New Sample Description* text boxes. This first sample will be used as a "template", only to establish the analysis parameters.



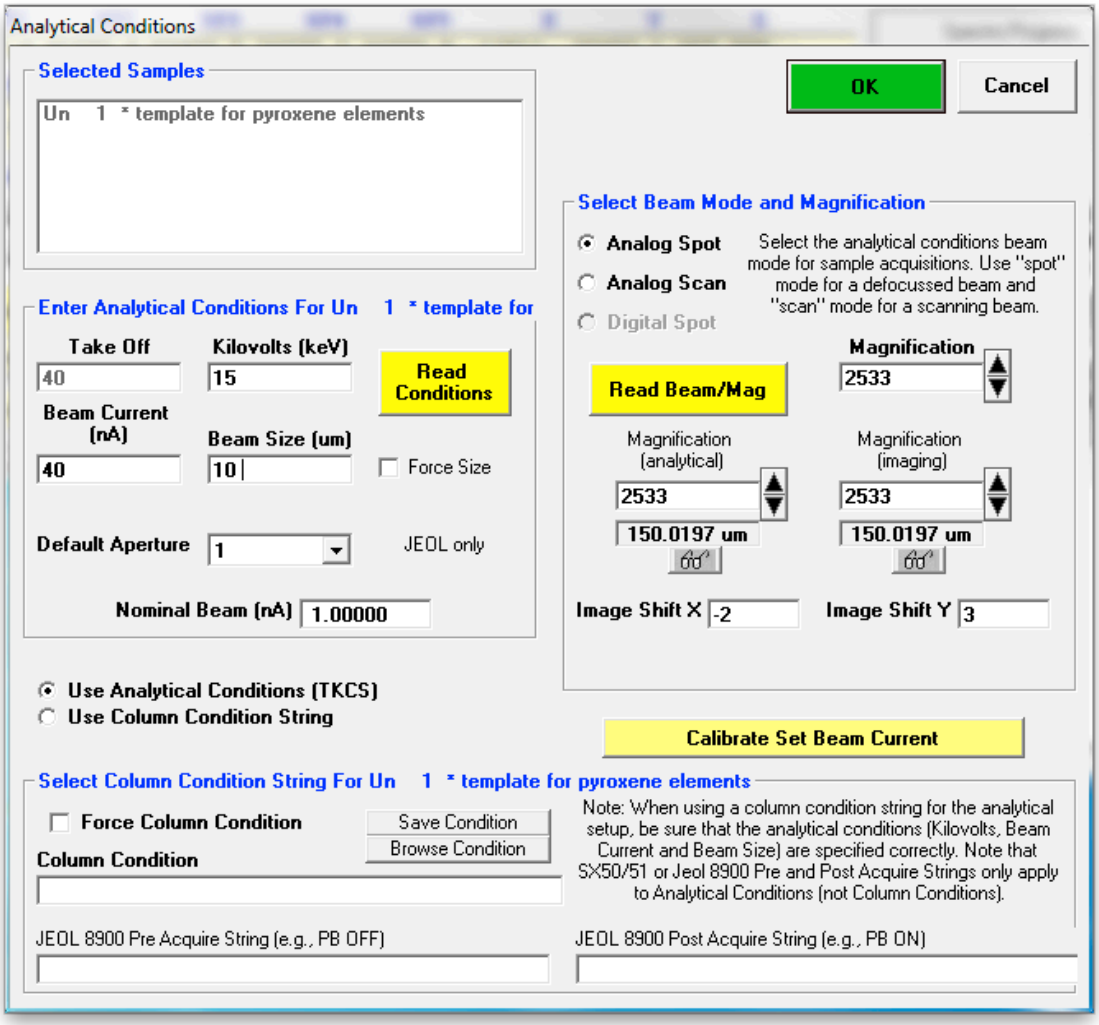
Click the **OK** button of the **New Sample** dialog box.

The program returns to the **Acquire!** window. Notice that the first sample designated *Un 1 * template for pyroxene elements* is now listed in the *Current Sample* text box. The * symbol indicates that no data has been collected for this sample yet. Note, all of the buttons in **Acquire!** are now available.



Setting Analytical Conditions

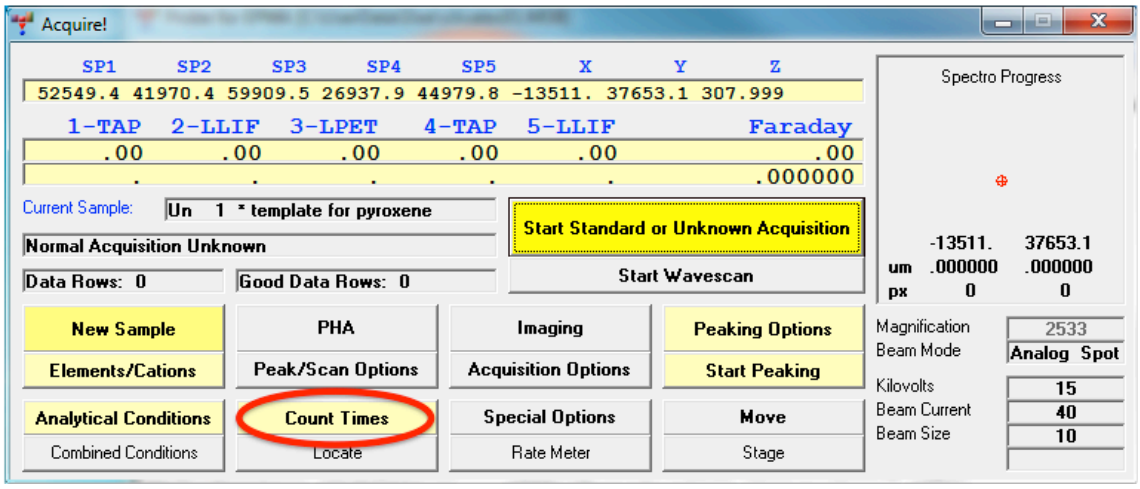
Click the **Analytical Conditions** button to open the **Analytical Conditions** dialog box. Enter the appropriate numbers into the *Kilovolts*, *Beam Current*, and *Beam Size* text boxes for the currently *Selected Sample*. The *Kilovolts*, *Beam Current*, and *Beam Size* will need to be manually adjusted if a column digital interface is not present. If a hardware interface is supported, the user may specify a column condition string to indicate the desired analytical conditions of the instrument. Clicking the **Read Conditions** button will cause PROBE FOR EPMA to check the current analytical conditions and reload them if the conditions have changed.



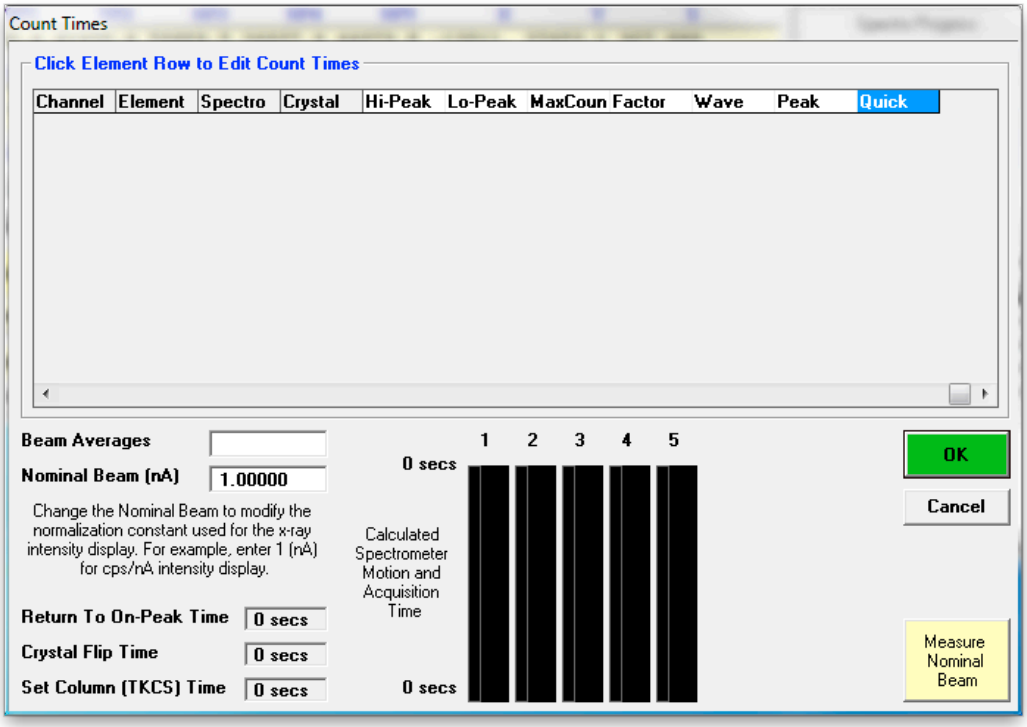
Click the **OK** button when done. The **Analytical Conditions** dialog box closes, returning to the **Acquire!** window.

Nominal Beam Current Measurement

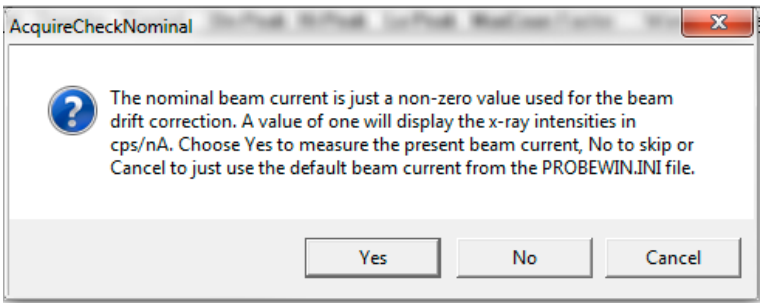
The nominal beam current is not the actual measured beam current but a close approximation that is used to calibrate the magnitude of the beam drift correction. If the nominal beam current is close to the actual measured beam current then the correction is close to 1.0. The beam drift corrected counts displayed in the main log window will be close in magnitude to the counts displayed on the screen scalers. The nominal beam can be adjusted in several ways. Click the **Count Times** button of the **Acquire!** dialog box.



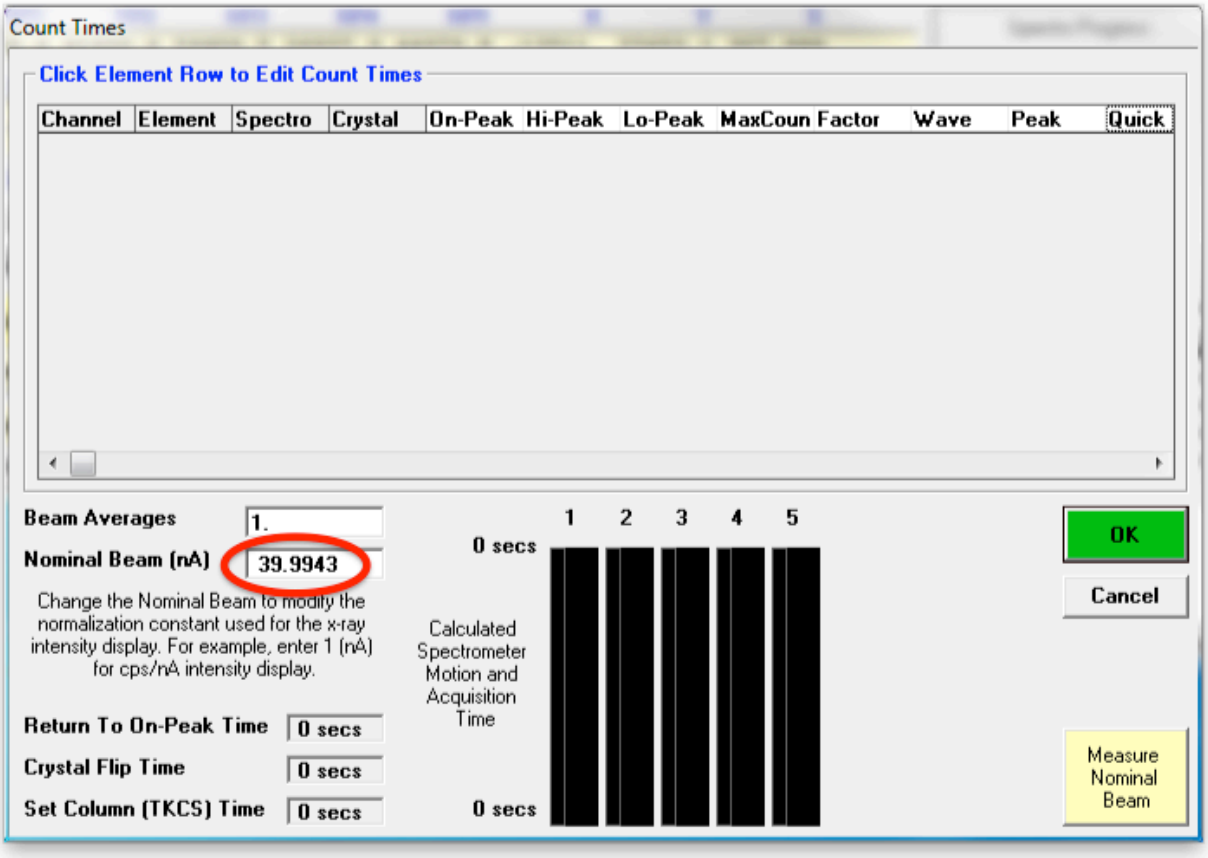
This opens the **Count Times** dialog box.



Any value desired may be directly entered into the *Nominal Beam* text box (1 nA value is stored in the PROBEWIN.INI file) or the user may measure the present beam current by clicking the **Measure Nominal Beam** button. The **AcquireCheckNormal** dialog box appears, choose the **Yes** button to measure the present beam current for use in the beam drift correction.



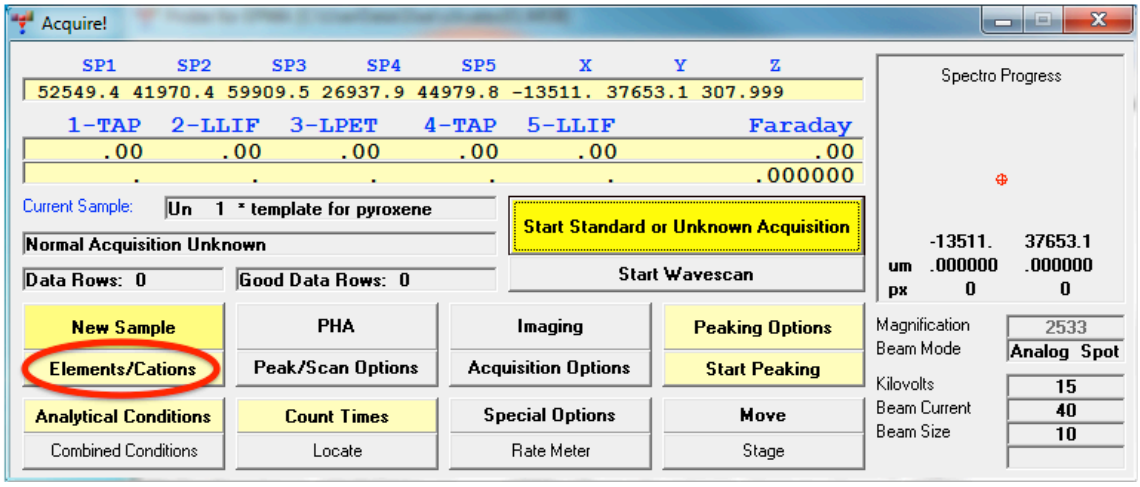
The current value of the faraday beam is measured and reported to both the **Acquire!** window and the *Nominal Beam* text box in the **Count Times** window as seen below.



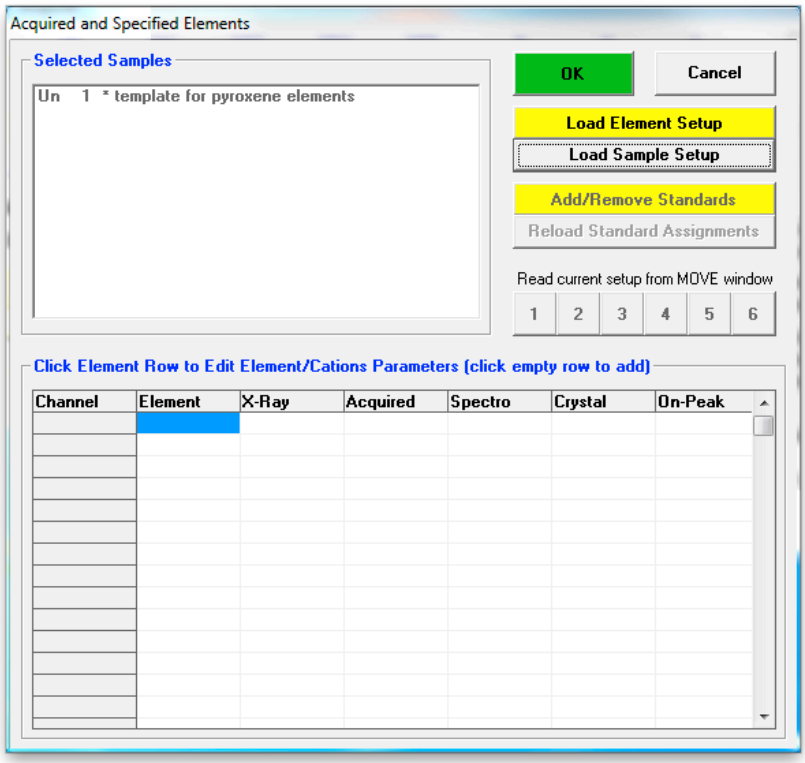
Close the **Count Times** window by clicking the **OK** button.

Element, X-Ray Line and Spectrometer Parameters Selection

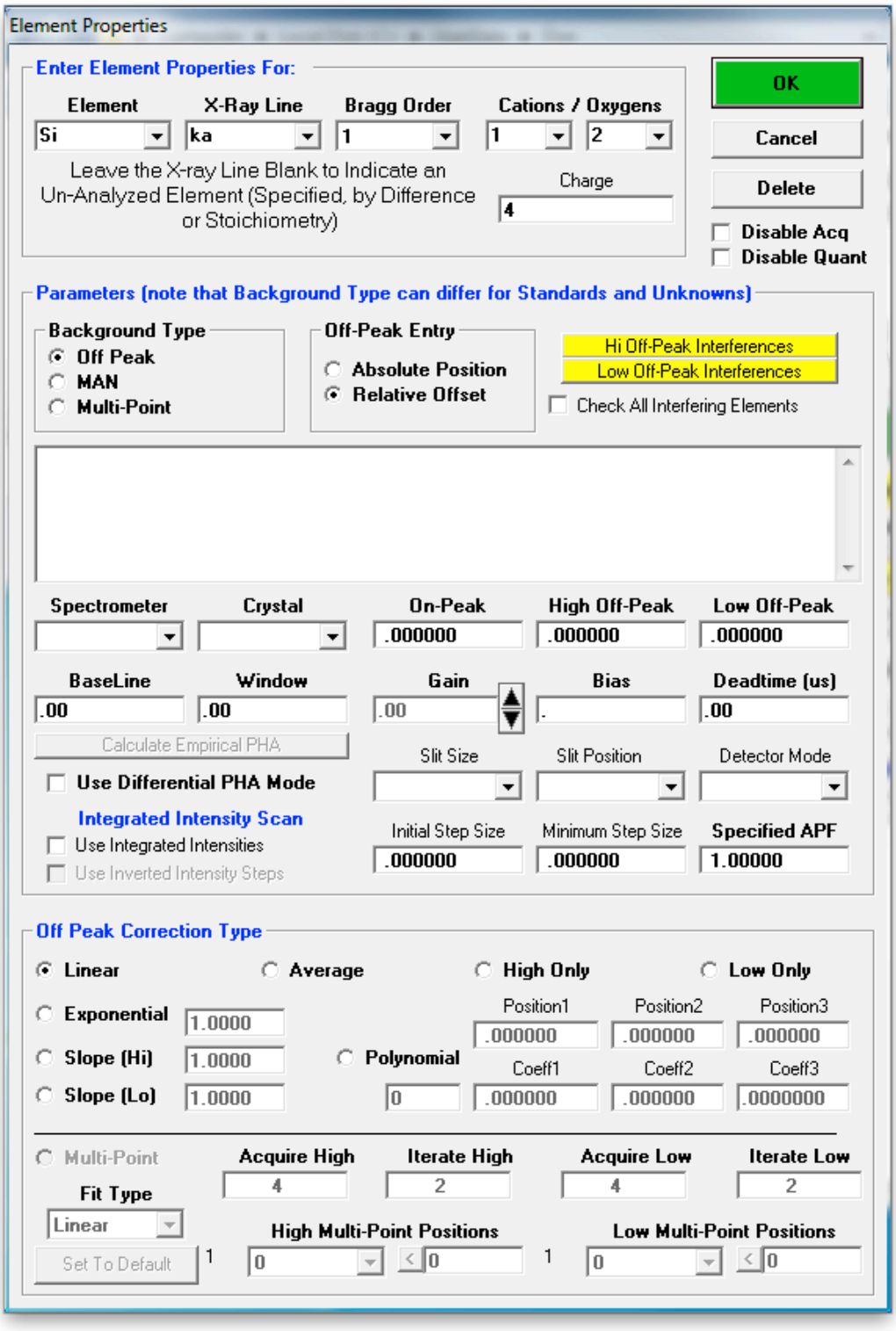
Next, the user specifies the elements to be analyzed. Click the **Elements/Cations** button of the **Acquire!** window.



This action opens the **Acquired and Specified Elements** dialog box. Click on the first empty row under the element column to enter the first element to analyze. The user may enter the analyzed elements in any order; however, the analysis output will follow the order in which the elements were entered here.



This opens the **Element Properties** dialog box. In the *Element* field type in the first element to analyze. Certain default values listed in this window are based on parameters entered into the previously established configuration files.



Under the *Enter Element Properties For:* section (top of the **Element Properties** dialog box), choose the correct *X-Ray Line*, *Cations*, and *Oxygens* for the first element. Both alpha and beta lines are now supported as well as the ability to analyze the same element on all relevant spectrometers.

Continue by selecting the *Background Type*. Three background correction methods are available to the user; off-peak, MAN (mean atomic number), and the multi-point method (see the User's Guide and Reference documentation for a complete discussion of these three types).

Short Note on Background Types

The ***off-peak*** method entails measuring the background conventionally on each element in the sample of interest with the spectrometer adjusted to a position, typically on each side of the analytical peak. This method, while somewhat time-consuming, can accurately determine the background contribution for major, minor, and trace element concentrations. Sophisticated modeling routines are available for precisely fitting backgrounds around analytical peaks (see User's Guide and Reference documentation for details).

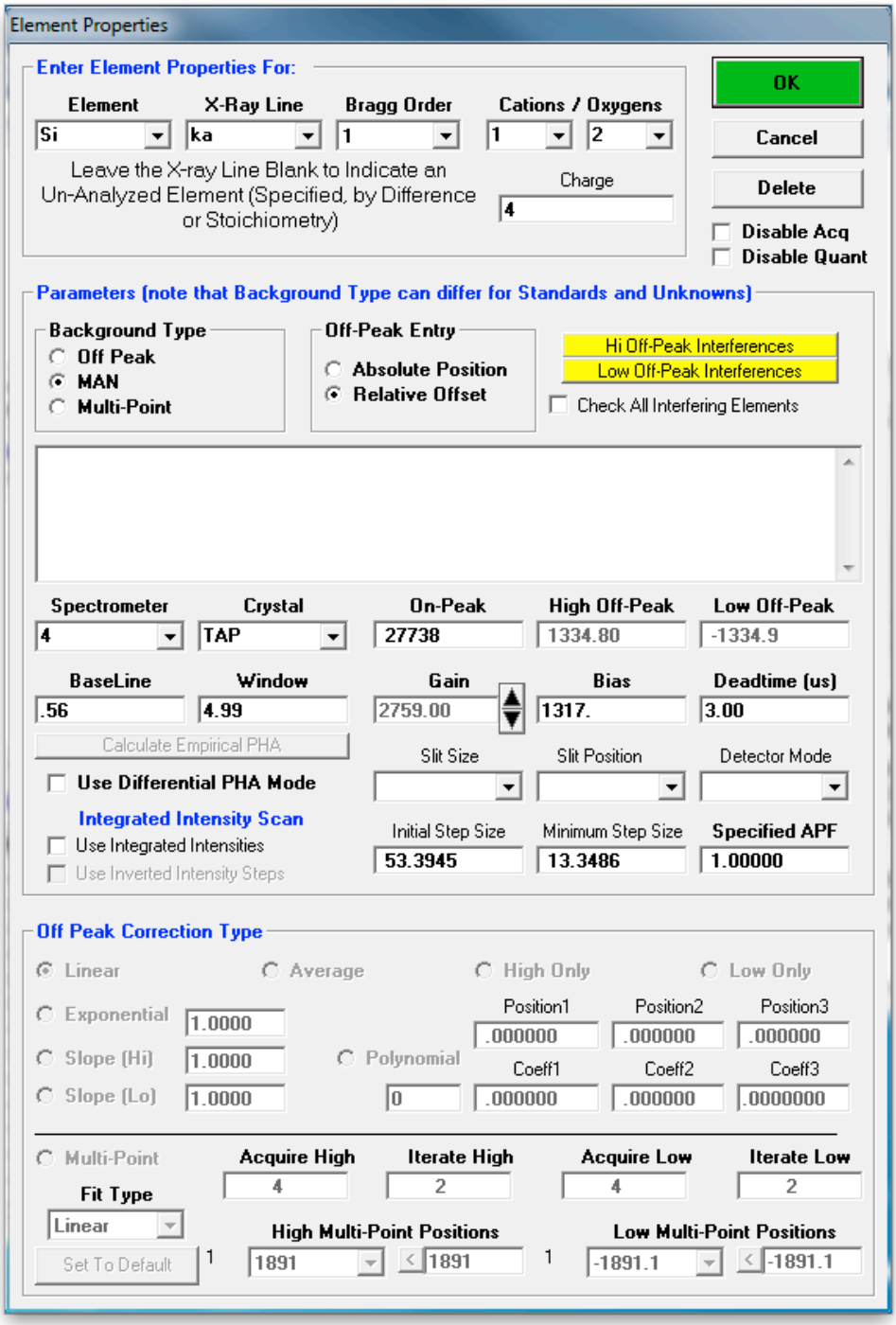
The ***MAN*** method relies on the fact that most of the background (continuum) production in the sample is directly proportional to the average atomic number of the sample. The MAN correction is an empirical calibration curve method involving the measurement of standards of known composition (hence average atomic number). If many samples are to be analyzed for their major and minor element concentrations then substantial time may be saved using the MAN method. However, if the user is required to measure high atomic number samples and/or trace concentrations, more accurate data may be obtained with off-peak background corrections.

Finally, a new third background method is now available for high accuracy trace element analysis, called the ***multi-point*** background method. PROBE FOR EPMA automatically acquires a number of off-peak intensities distributed on each side of the analytical peak (user specified) so that at least a few of the background measurements will not be affected by the unpredicted presence of various other elements in the sample.

In this exercise, we will use MAN for the major pyroxene elements Si, Fe, Mg, Ca, and off-peak for Ti, Al, Cr, V, Mn, Na, which might only be present at trace levels. Continue by selecting MAN for the Si *Background Type* in the *Parameters* section. This deactivates the *Off Peak Correction Type* buttons as well as the *High* and *Low Off-Peak* boxes.

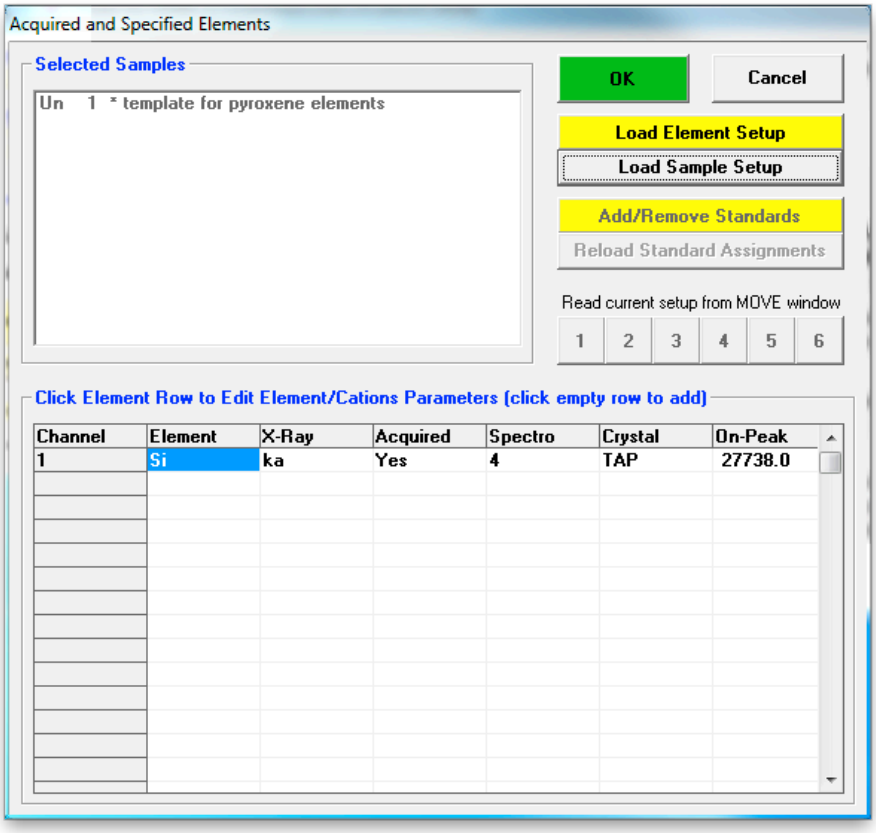
Next, use the drop-down menu to select or click the text box under *Spectrometer* and enter the appropriate spectrometer number that will be used to analyze the first element. Choosing a spectrometer number loads various parameters from the configuration files. Each of these parameters in this window should be inspected and edited as needed (use the <tab> key to move between boxes).

The next screen shows the edited **Element Properties** dialog box for silicon.

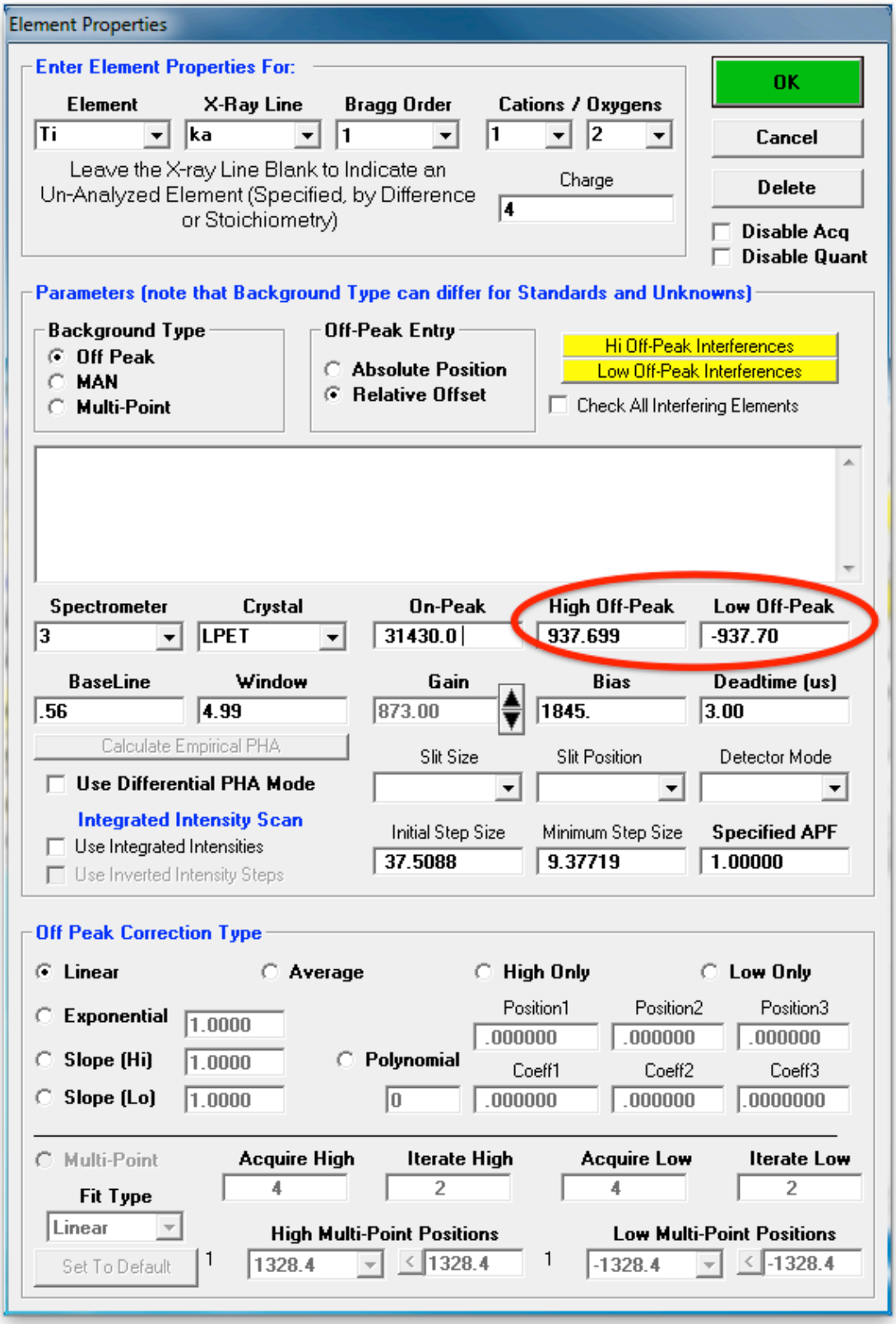


Click the **OK** button of the **Element Properties** dialog box to accept these element parameters for silicon.

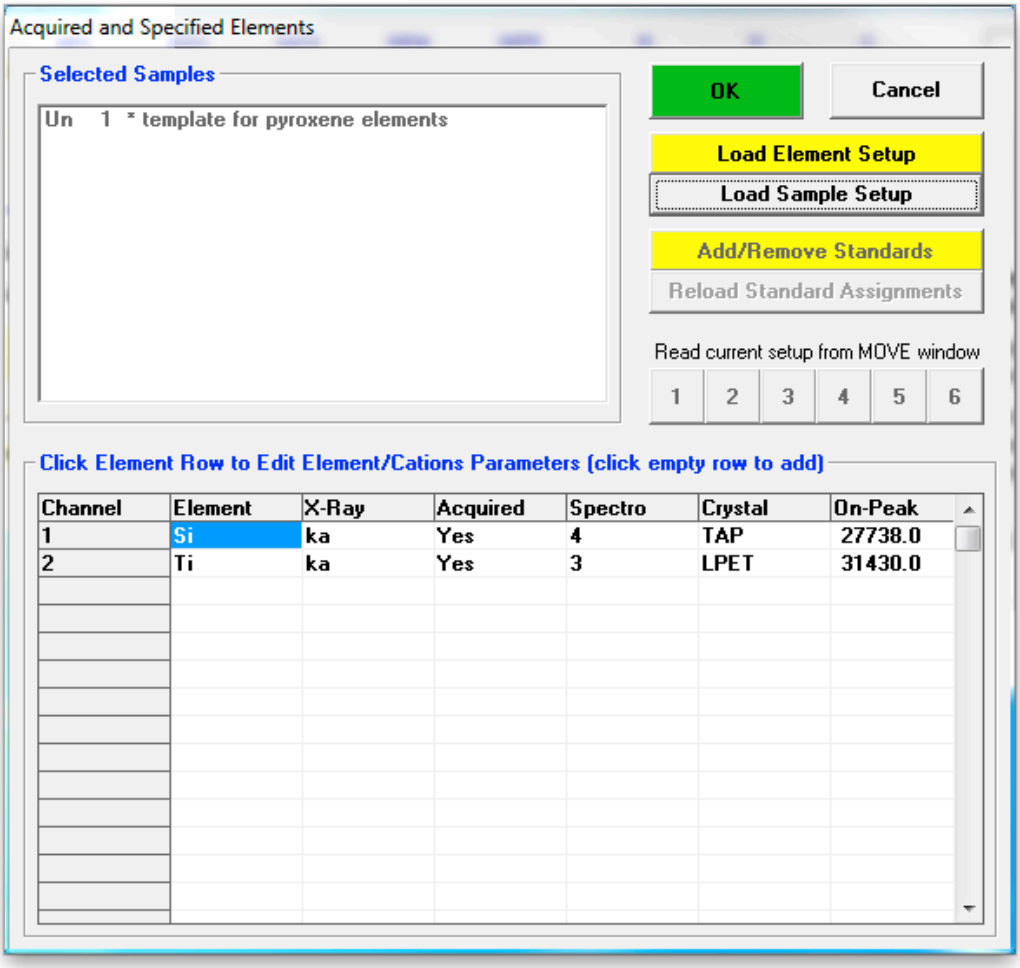
The program returns to the **Acquired and Specified Elements** window with silicon now entered into the *Element/Cations Parameters* table.



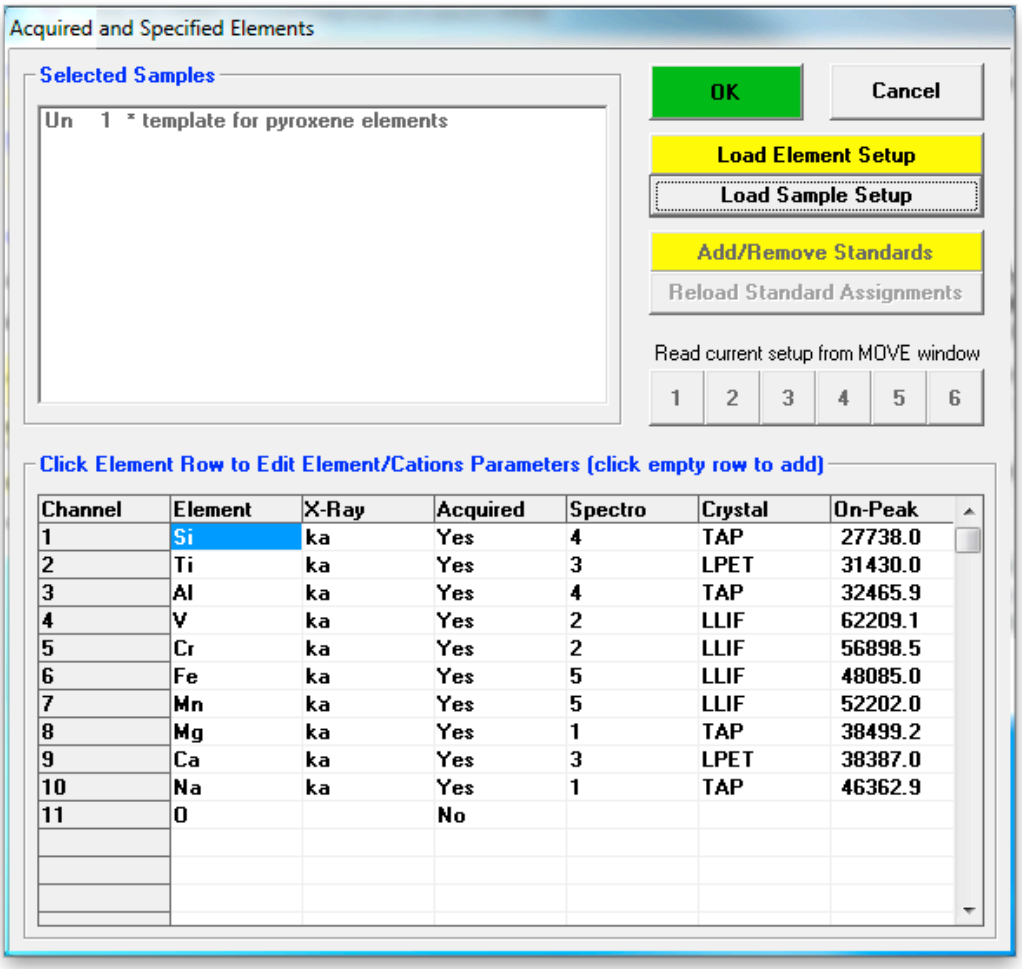
Enter titanium as the next element in the run by clicking on the next empty row of the **Acquired and Specified Elements** window. This opens the **Element Properties** dialog box again. Enter the appropriate *Element, Spectrometer, and Crystal* and adjust all other text boxes and buttons. Choose *Background Type* off-peak this time. The software calculates default high and low off-peak positions as shown below. Leave these as they are for the moment. They can be changed later if required.


Element Properties
Enter Element Properties For:
Element
X-Ray Line
Bragg Order
Cations / Oxygens
Ti
ka
1
1
2
Leave the X-ray Line Blank to Indicate an Un-Analyzed Element (Specified, by Difference or Stoichiometry)
Charge
4
OK
Cancel
Delete
Disable Acq
Disable Quant
Parameters (note that Background Type can differ for Standards and Unknowns)
Background Type
Off Peak
MAN
Multi-Point
Off-Peak Entry
Absolute Position
Relative Offset
Hi Off-Peak Interferences
Low Off-Peak Interferences
Check All Interfering Elements
Spectrometer
Crystal
On-Peak
High Off-Peak
Low Off-Peak
3
LPET
31430.0
937.699
-937.70
BaseLine
Window
Gain
Bias
Deadtime (us)
.56
4.99
873.00
1845.
3.00
Calculate Empirical PHA
Use Differential PHA Mode
Slit Size
Slit Position
Detector Mode
Integrated Intensity Scan
Use Integrated Intensities
Use Inverted Intensity Steps
Initial Step Size
Minimum Step Size
Specified APF
37.5088
9.37719
1.00000
Off Peak Correction Type
Linear
Average
High Only
Low Only
Exponential
1.0000
Slope (Hi)
1.0000
Slope (Lo)
1.0000
Polynomial
0
Position1
Position2
Position3
.000000
.000000
.000000
Coeff1
Coeff2
Coeff3
.000000
.000000
.0000000
Multi-Point
Fit Type
Linear
Set To Default
Acquire High
Iterate High
Acquire Low
Iterate Low
4
2
4
2
High Multi-Point Positions
1328.4
1328.4
Low Multi-Point Positions
-1328.4
-1328.4

Click the **OK** button of the **Element Properties** to enter titanium into the *Element/Cations Parameters* table of the **Acquired and Specified Elements** window.

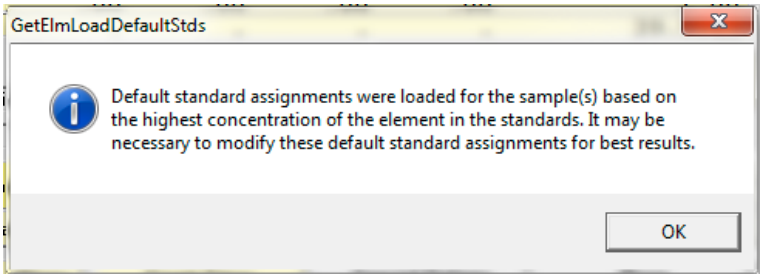


Continue adding the remaining elements in the desired order, choosing *Background Type* **MAN** or **off-peak** as indicated above. The remaining eight element entries are not shown here to save space. Finally, oxygen is added to the element list as a not analyzed element for subsequent formula calculations. This is done by entering O (for oxygen) in the *Element* text box and leaving the *X-Ray Line* text box empty (see User's Guide and Reference documentation for more details).



Click the **OK** button of the **Acquired and Specified Elements** window when done entering elements in the run.

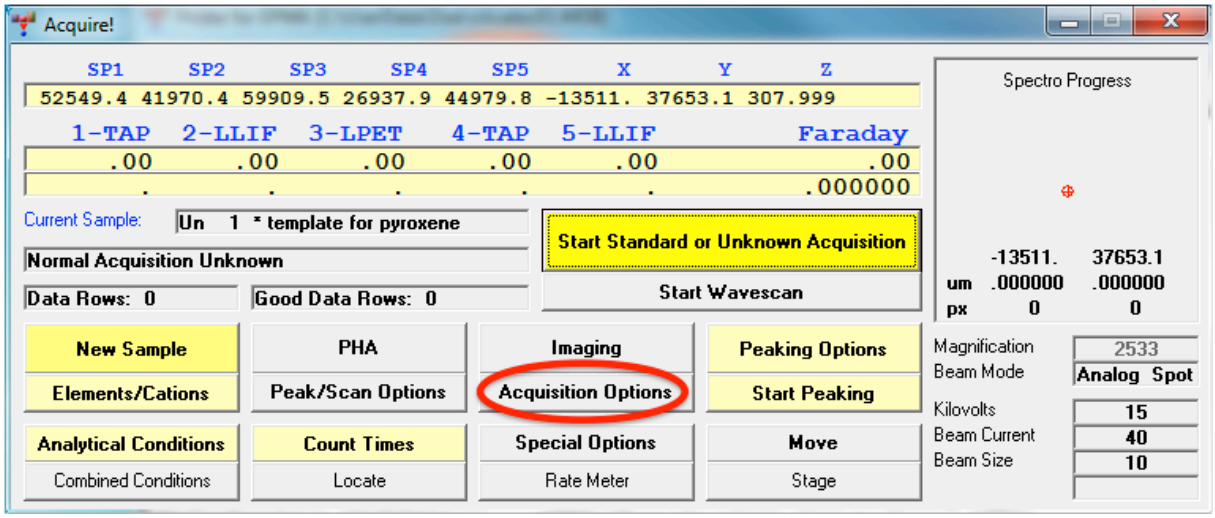
The **GetElmLoadDefaultStds** window opens to inform the user that standard assignments have been made based on elemental concentrations. The user will edit these choices shortly.



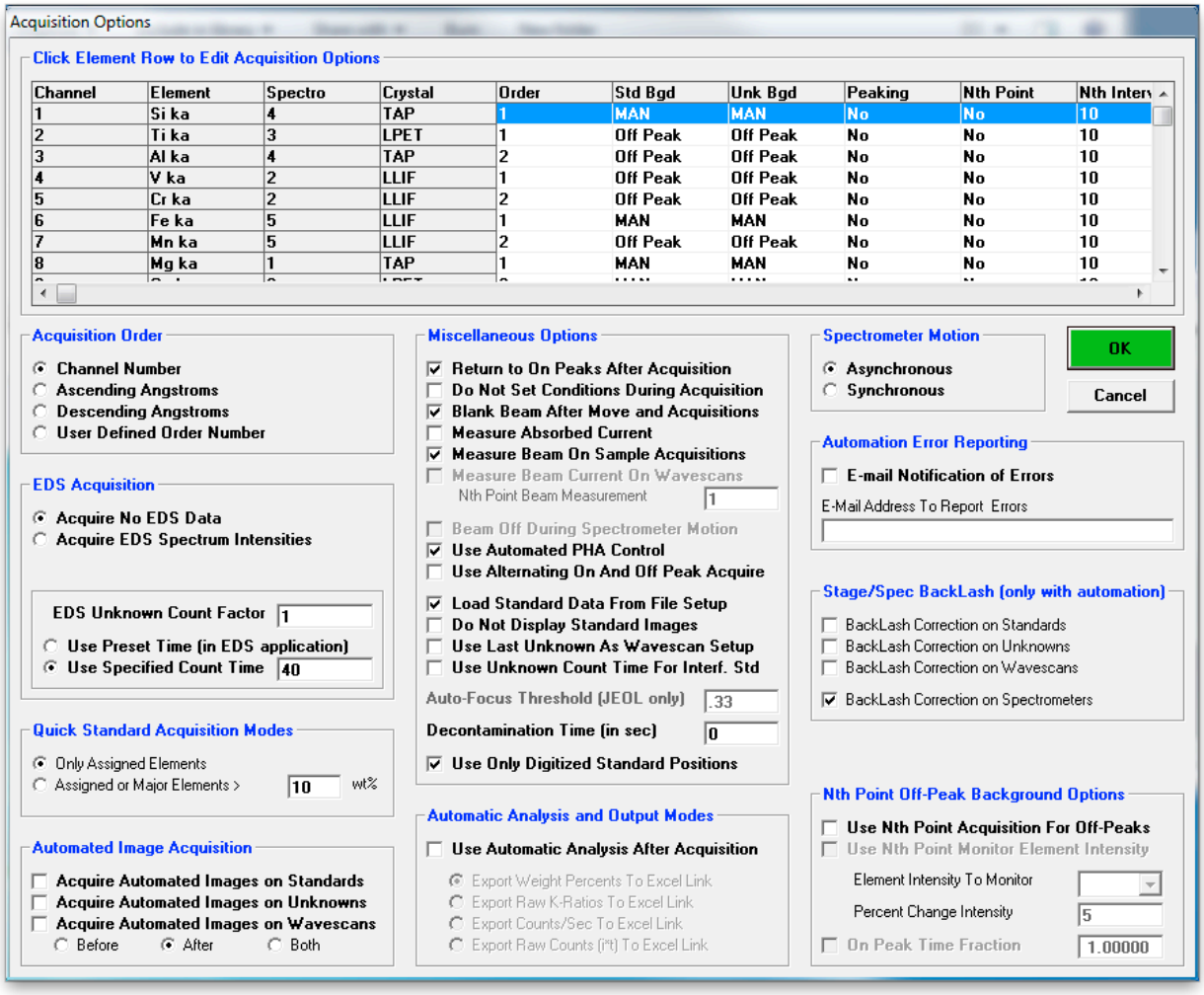
Click **OK** to return to the main **Acquire!** window.

Editing Acquisition Options

The user may change the element acquisition order of the spectrometers by clicking the **Acquisition Options** button in the **Acquire!** dialog box.

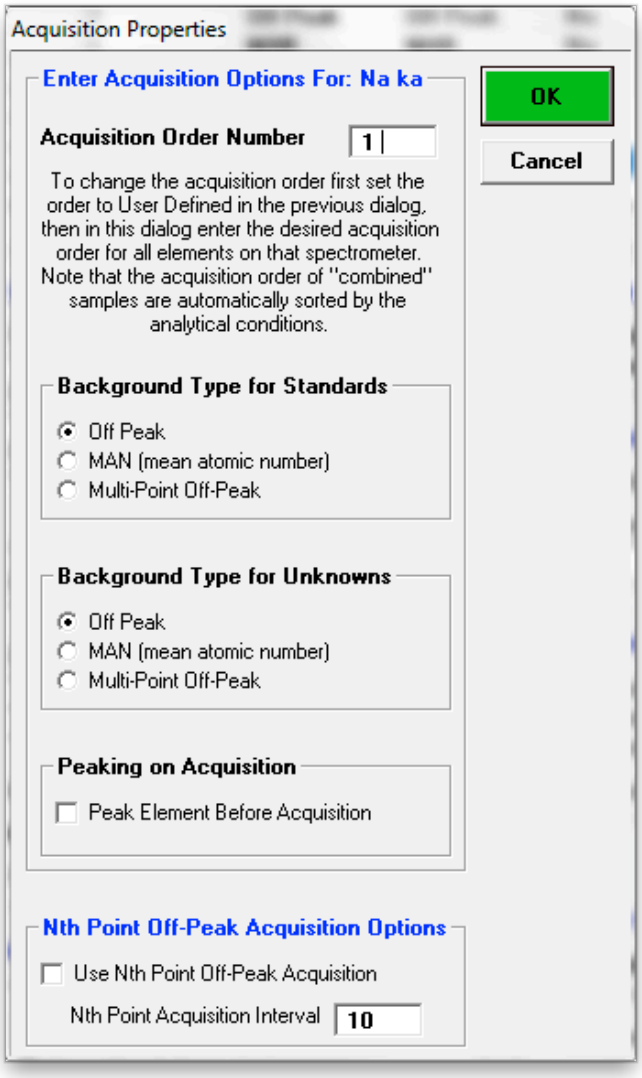


This opens the **Acquisition Options** dialog box.



To change the order that the spectrometer measures an element, select the *User Defined Order Number* button under *Acquisition Order* and click the row of the element to edit.

This opens the **Acquisition Properties** dialog box, seen below. Here, the user will re-define sodium (Na) to be counted on the first spectrometer pass due to its susceptibility to being volatilized by long exposure to the electron beam. In samples containing volatile elements the user may wish to consider running the time dependent intensity calibration routine (see User's Guide and Reference documentation and/or Advanced Topics manual).

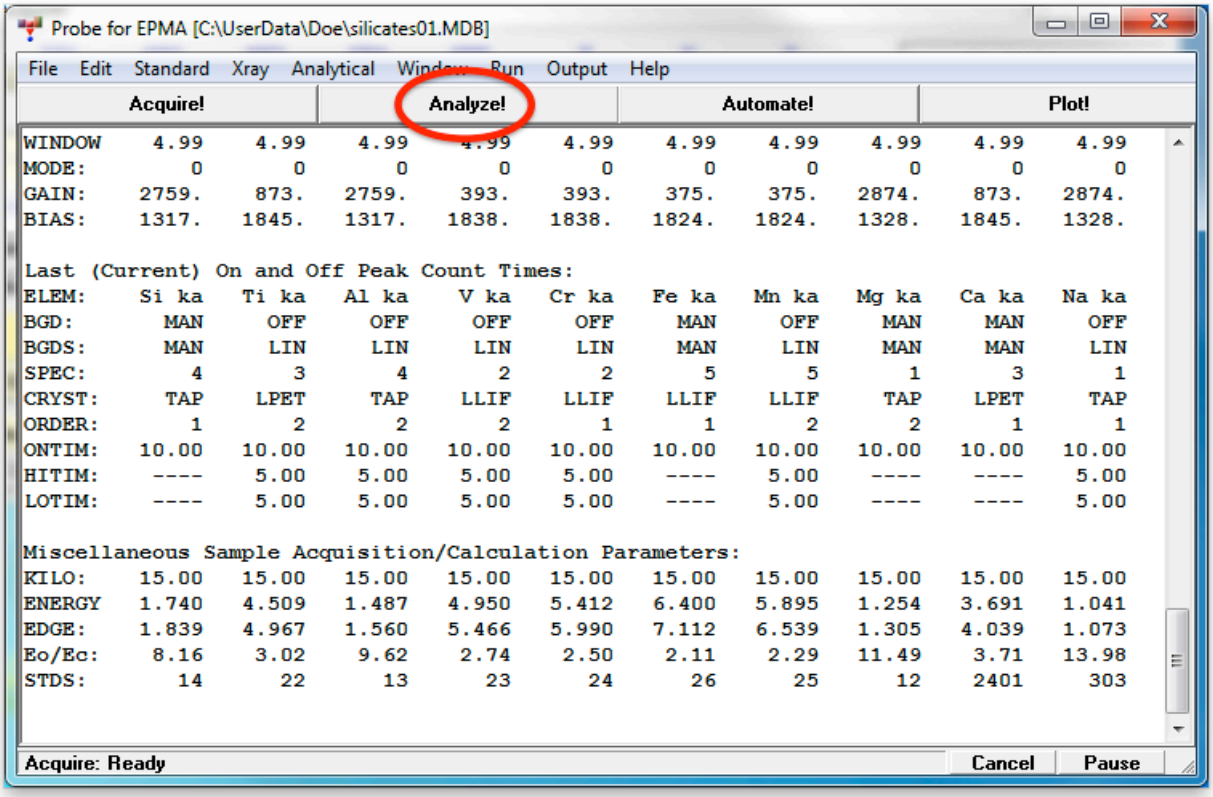


Edit the *Spectrometer Order Number* for all elements to change the acquisition order, e.g. change Na to 1 and Mg, which is measured on the same spectrometer, to 2. Further, to use the same background correction method for both standards and unknowns edit the *Background Type for Standards* to *MAN* for Si, Fe, Mg, and Ca. Click the **OK** button returning to the **Acquisition Options** window.

Click the **OK** button of the **Acquisition Options** window to return to the **Acquire!** window.

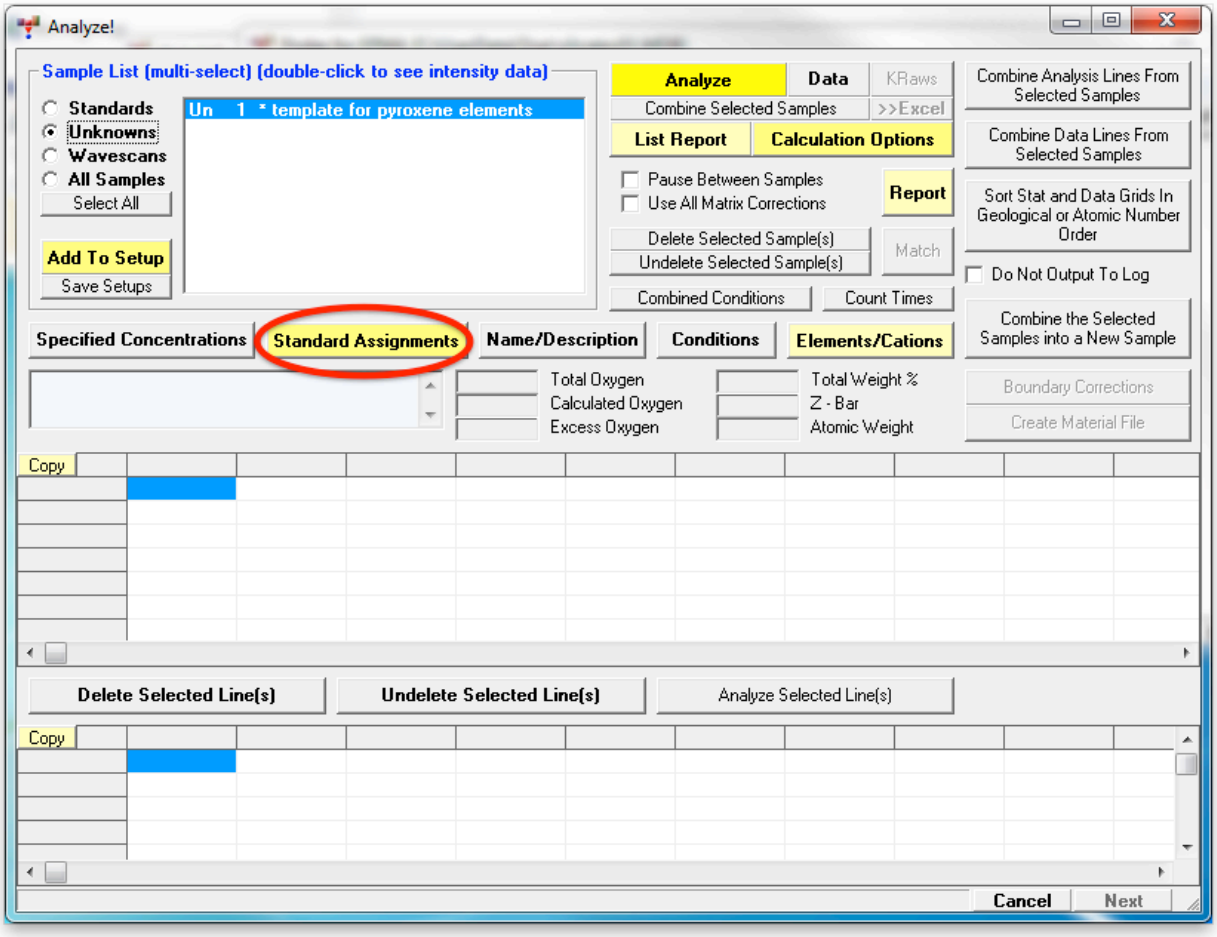
Modifying Standard Assignments

The standard assignments chosen by PROBE FOR EPMA may be inspected and edited by clicking the **Analyze!** button from the main log window.



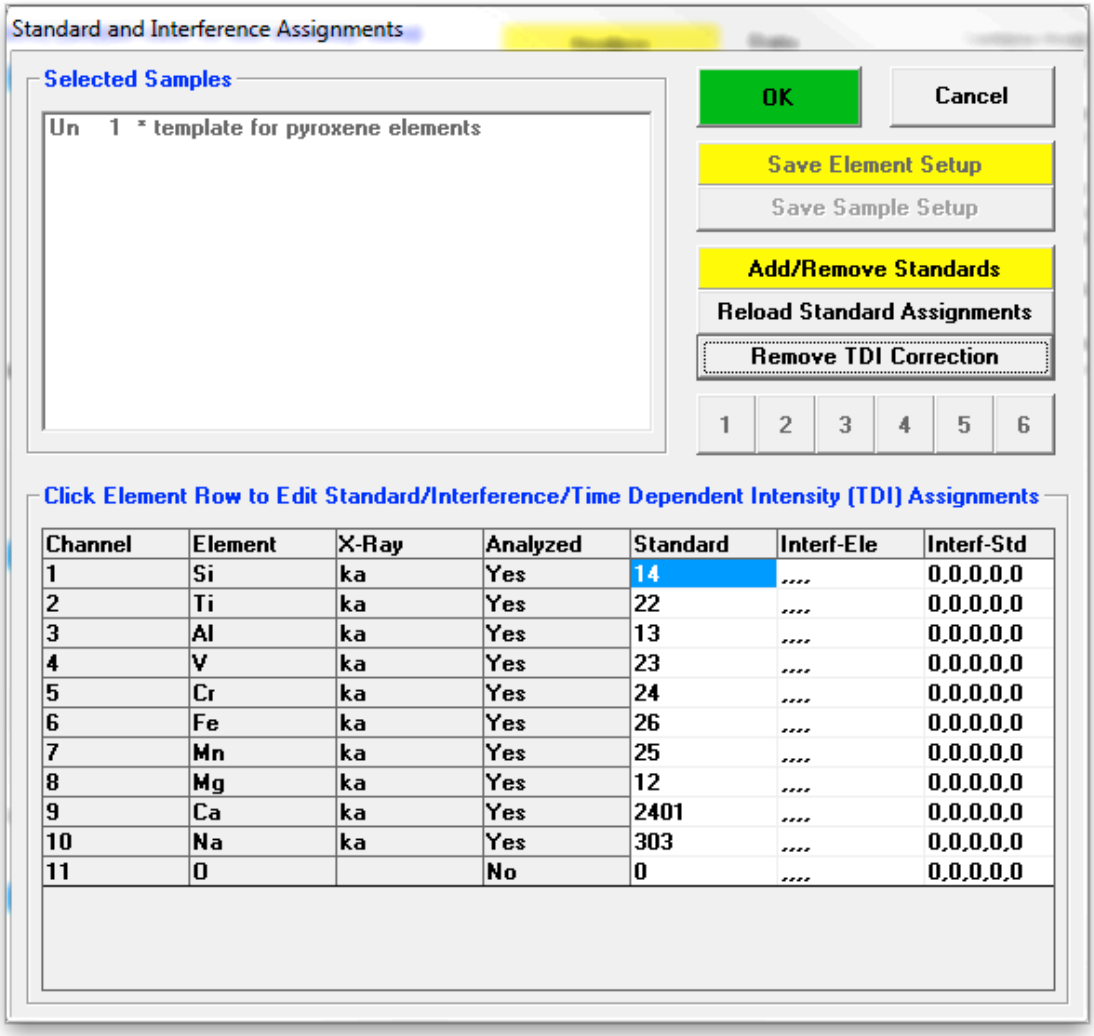
Note that the program automatically wraps element data output to eight elements per line. If the extended format menu is checked, (activated from the **Output** menu) the data is written out (in log window and to disk file, if enabled) as far as necessary to the right.

This opens the **Analyze!** dialog box.



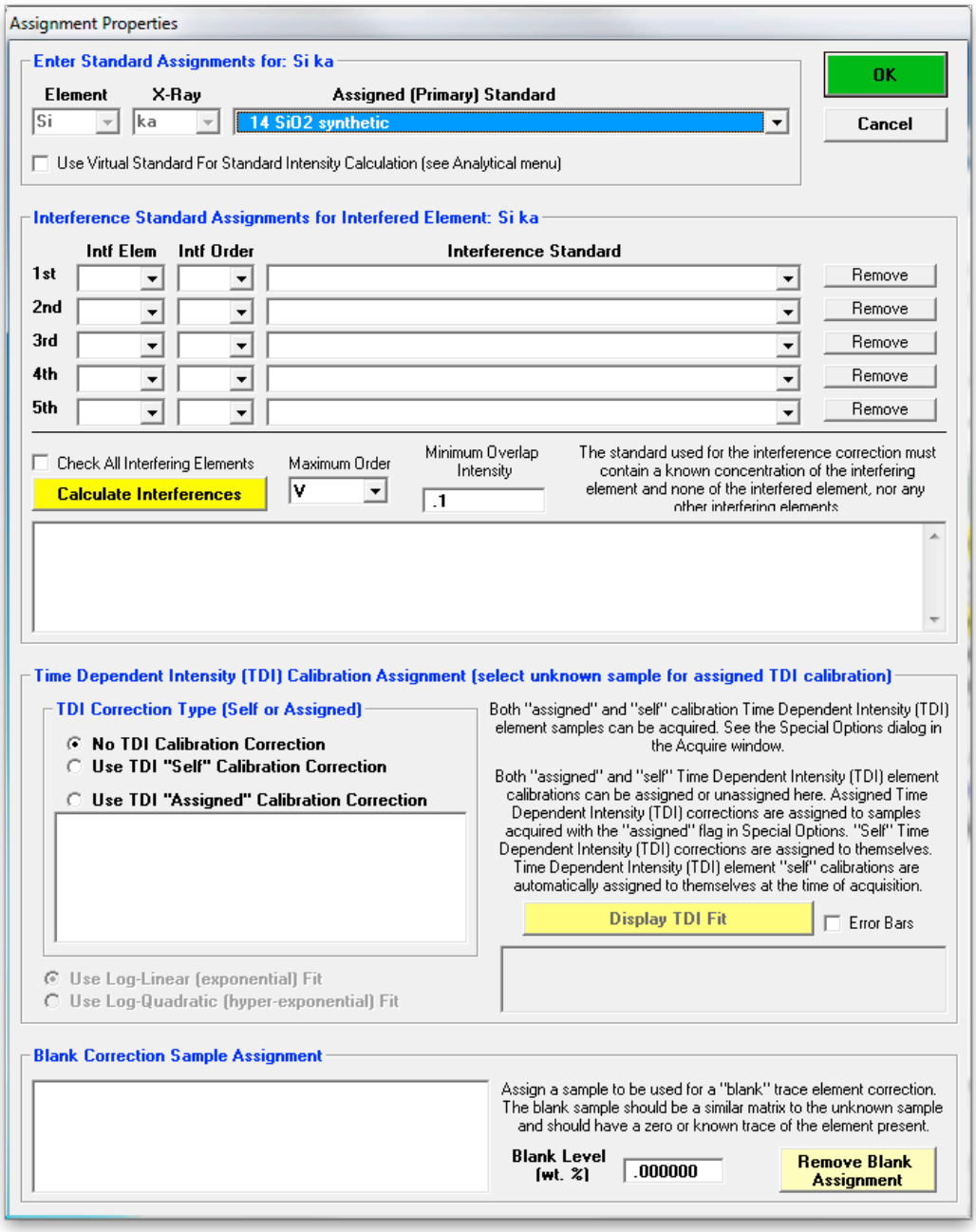
Click the **Standard Assignments** button.

The **Standard and Interference Assignments** dialog box opens.

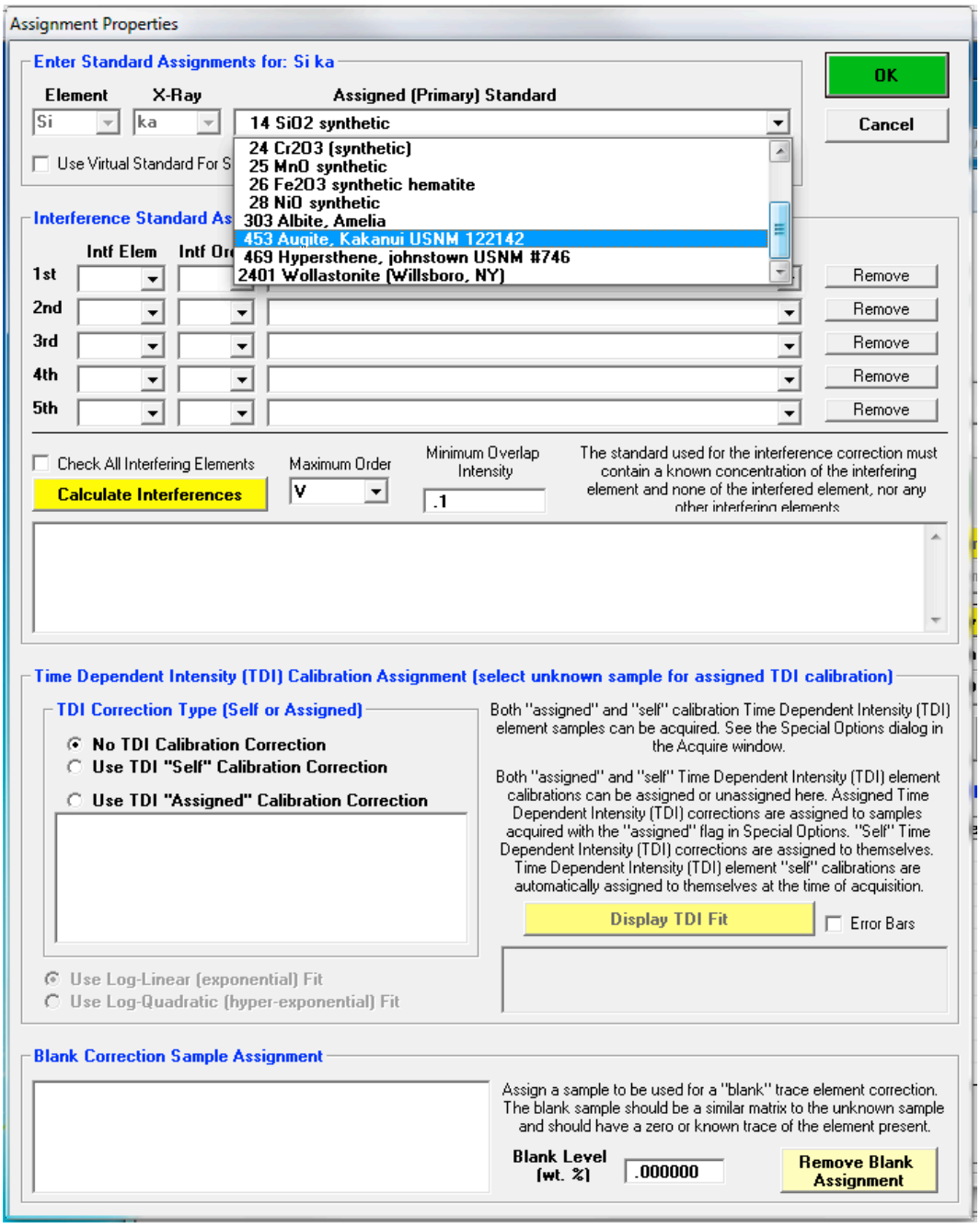


Click the row of an element to change the respective standard assignment, e.g. Si.

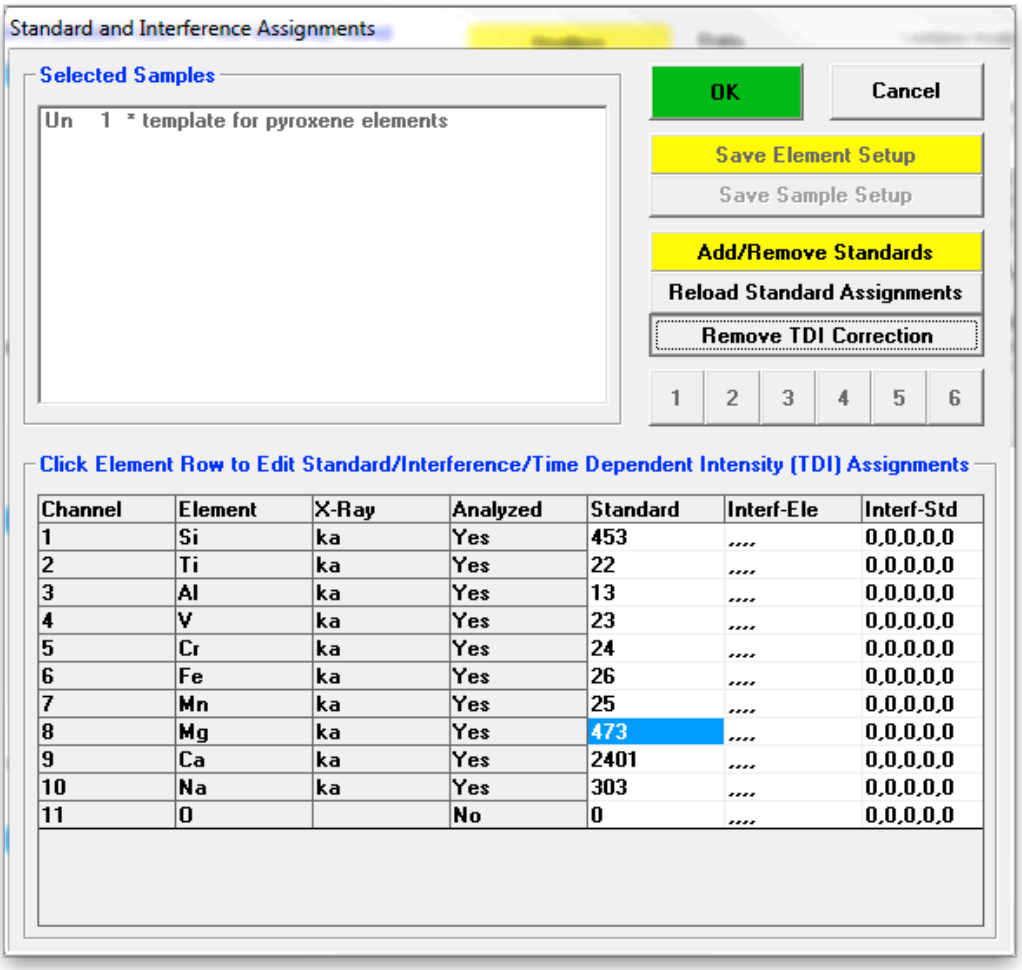
This opens the **Assignment Properties** dialog box. The default standard assignments are based on the highest concentration of the element in the standards currently in the run. In addition to standard assignments, the user may assign spectral interference corrections and time dependent intensity element calibrations from this window.



Click the *Assigned Standard* menu box. A scrollable list of all standards added to the current run is displayed. Select a new standard for element Si.



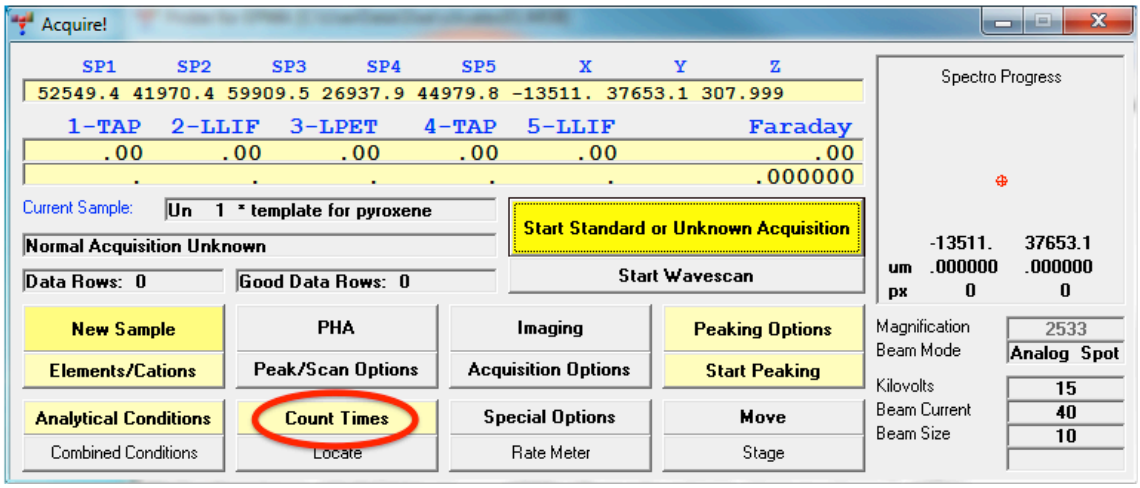
Click the **OK** button returning to the **Standard and Interference Assignments** dialog box. Repeat these editing steps until all necessary element standard assignments have been modified. In this example, the standard assignments for Si and Mg are edited, resulting in the following window.



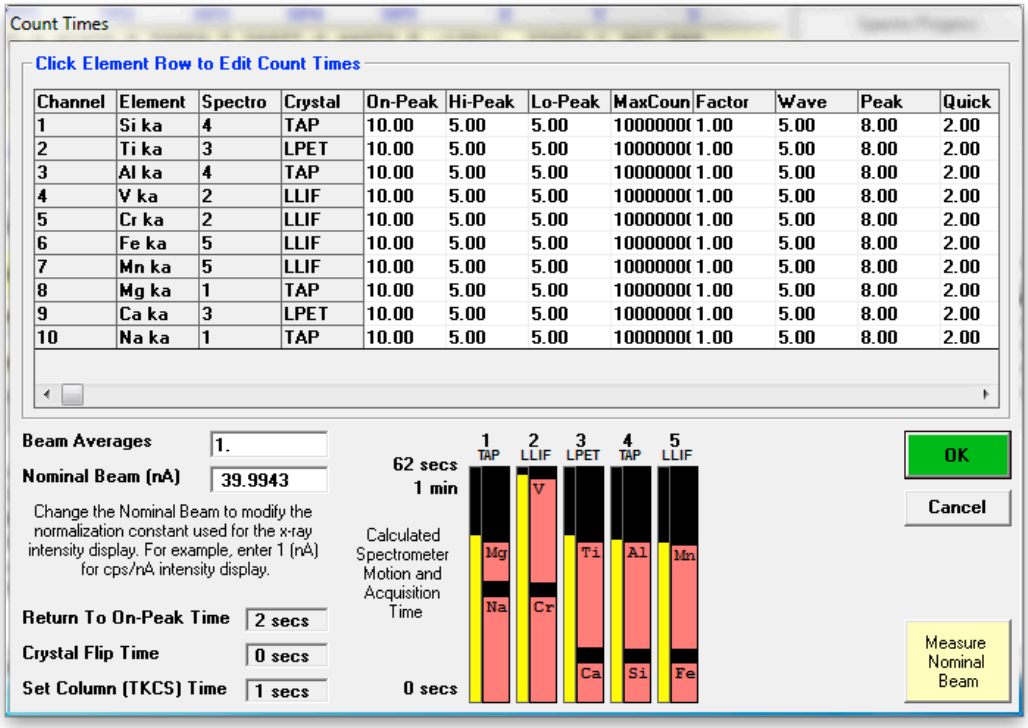
Click the **OK** button of the **Standard and Interference Assignments** dialog box returning to the **Analyze!** window.

Setting Count Times

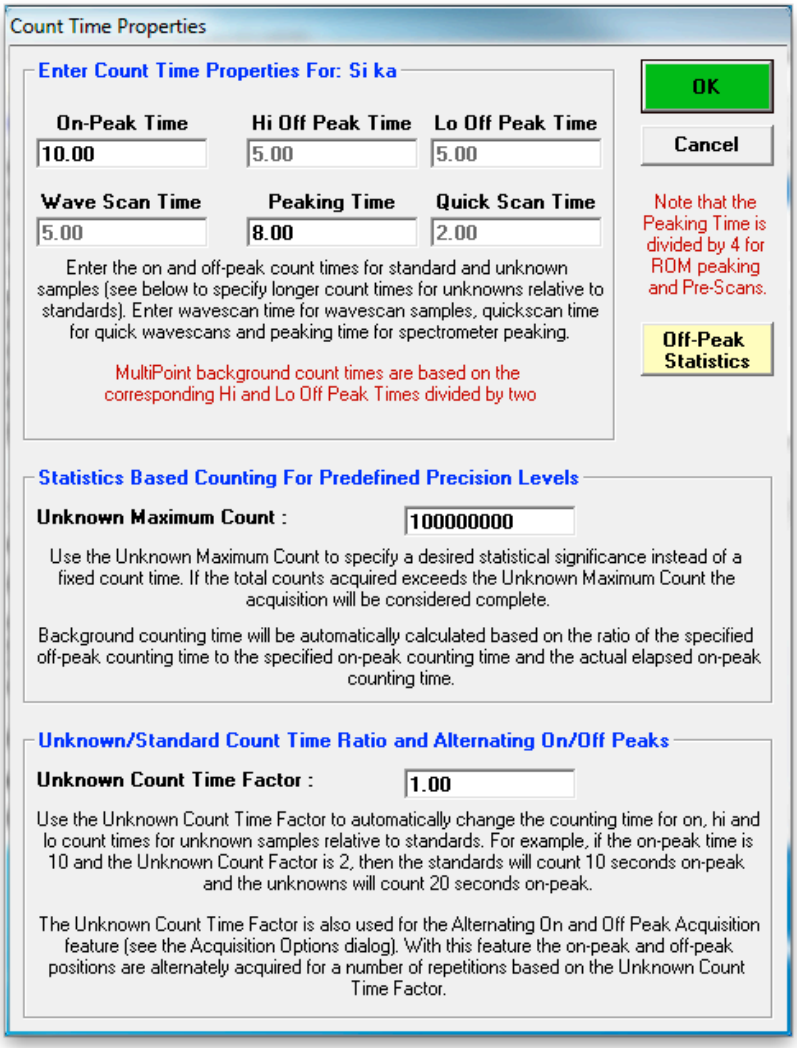
Click the **Count Times** button of the **Acquire!** window.



This opens the **Count Times** dialog box. Here various parameters relating to counting times can be adjusted. Initially *On-Peak* count time is set for 10 seconds and both *Hi-Peak* and *Lo-Peak* times are set for 5 seconds based on the configuration file defaults. For elements measured with Background Type MAN only the *On-Peak* setting is relevant. Note: Real time spectrometer motion and acquisition time is graphically displayed.



To edit the count times for any element click that row in the spreadsheet. This opens the **Count Time Properties** dialog box.



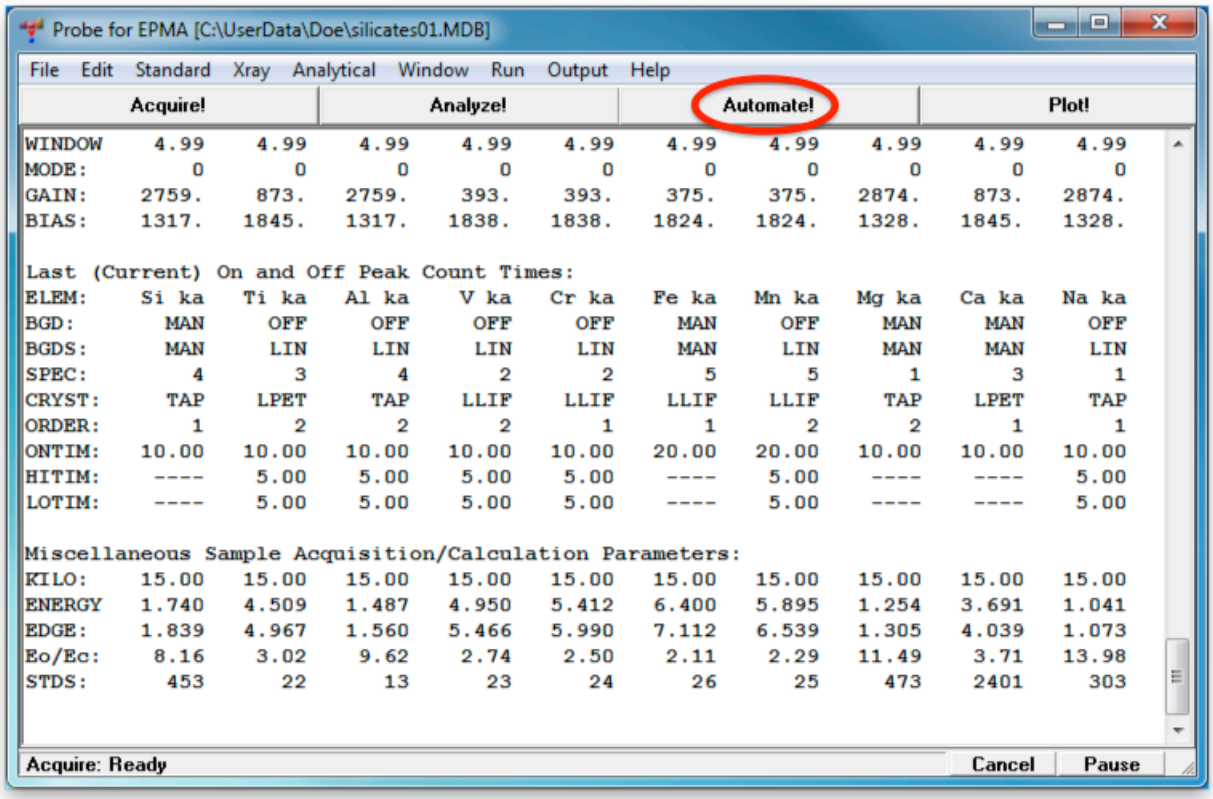
Edit the *Count Time* text boxes with new times. To adjust the count times on unknowns, change the *Unknown Count Time Factor*. This is the multiplicity factor for acquiring unknown sample elements relative to the count times specified for the standards.

The *Unknown Maximum Count* text box is used to specify a statistics based count time. This is most useful if the user wishes to count for 30 seconds or 40000 counts, whichever comes first. For samples with high count rate elements, the actual analysis time would be shorter.

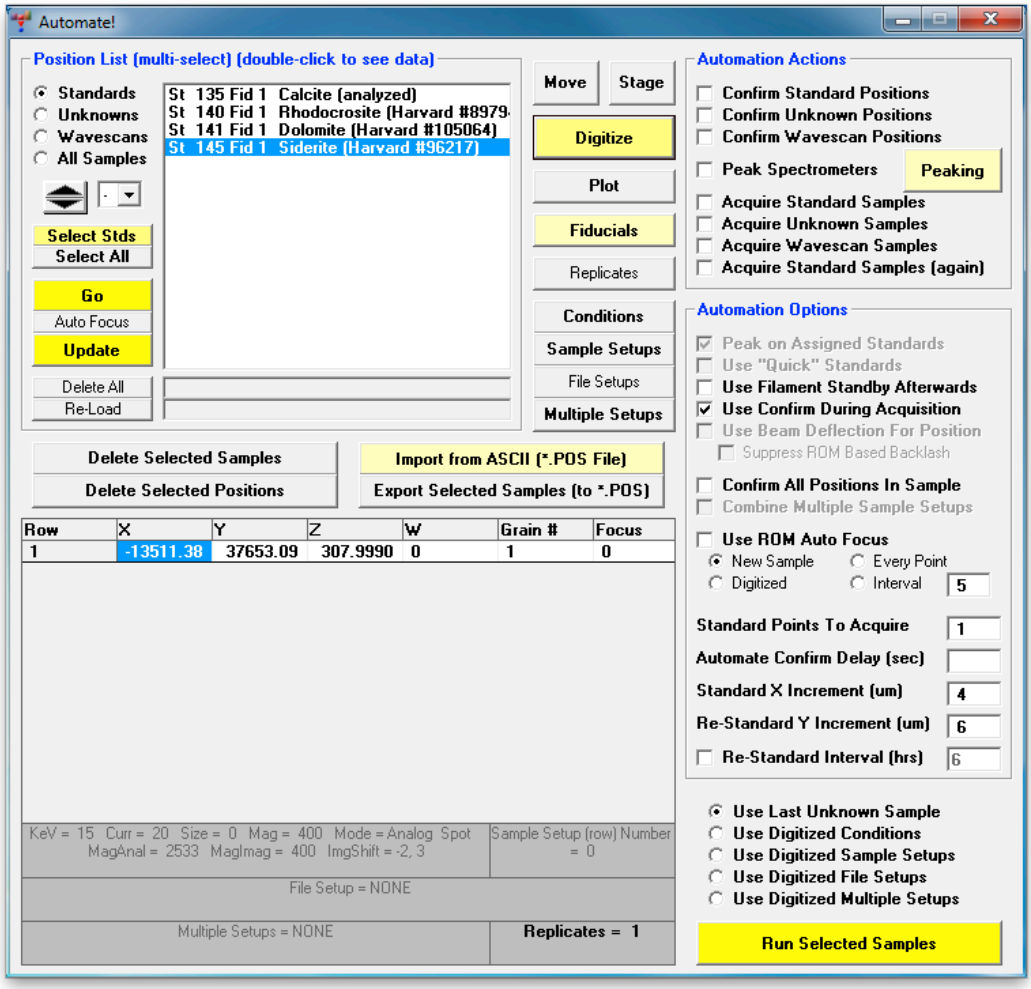
Click the **OK** button of the **Count Time Properties** window. Finally, click the **OK** button of the **Count Times** dialog box to accept any modified count times and return to the **Acquire!** window.

Loading Standard Position Files

Running standards using automation requires PROBE FOR EPMA to know the physical location of all the standards for this run. Click the **Automate!** button from the main PROBE FOR EPMA log window.

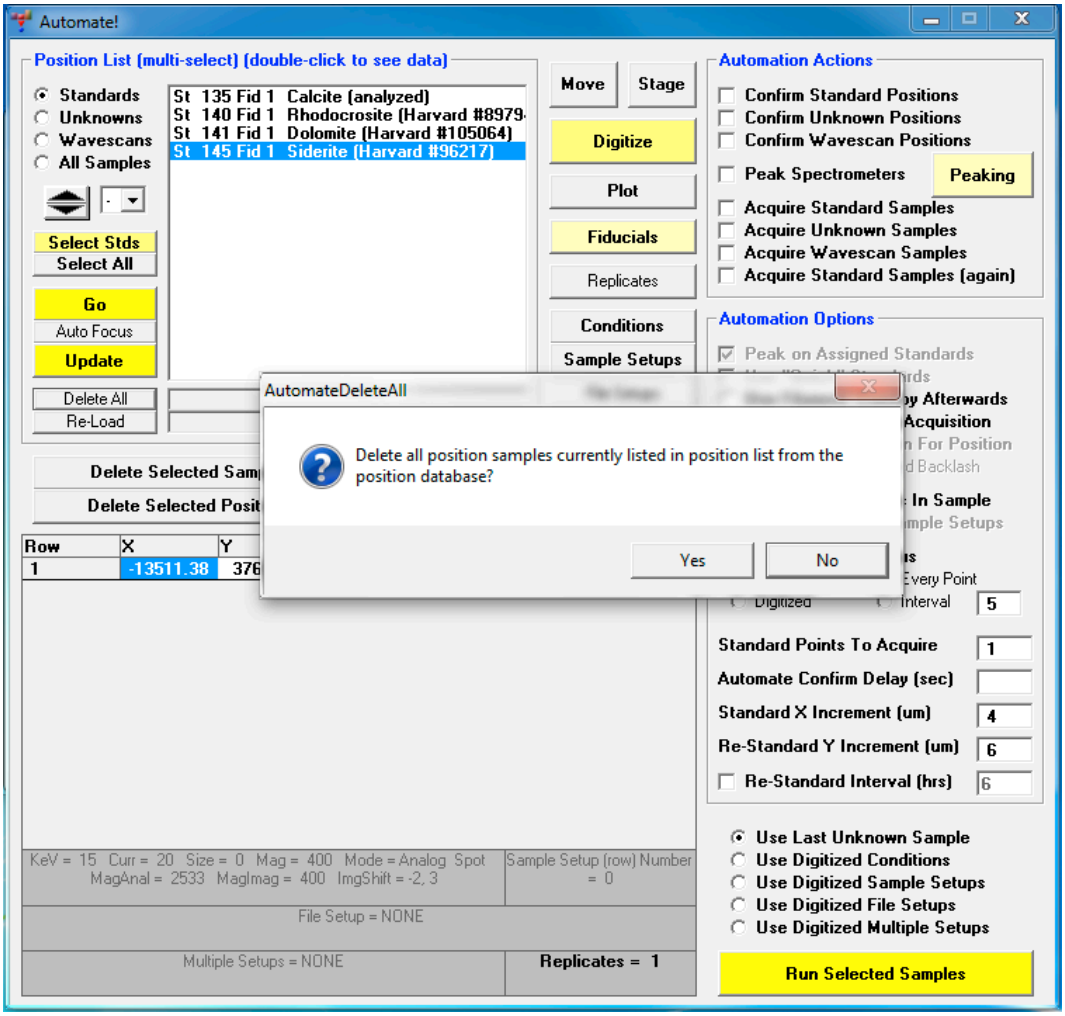


This opens the **Automate!** dialog box shown below.

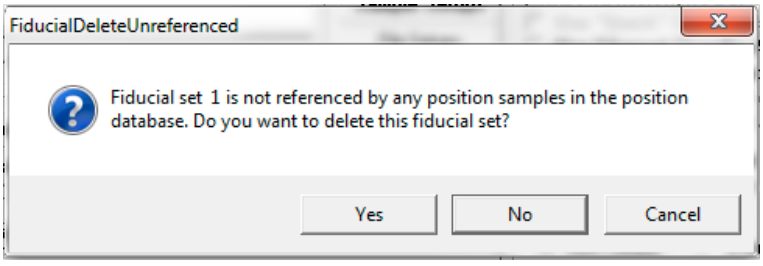


The last set of digitized standards used is visible in the *Position List* list box of the **Automate!** window. Currently, the carbonate standards digitized previously are listed. These will be deleted and replaced by the appropriate standard position file(s).

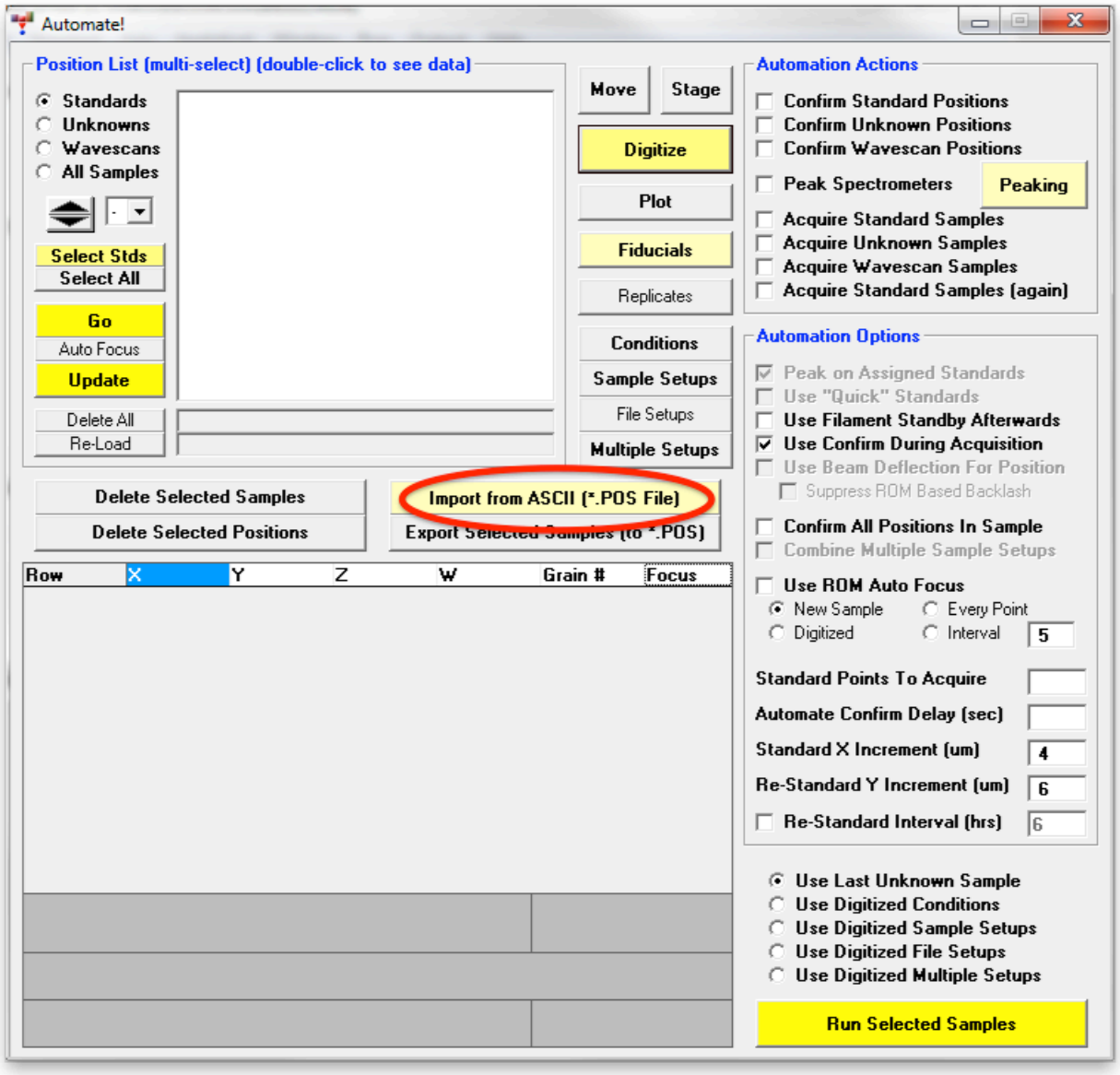
Click the **Delete All** button. This opens the **AutomateDeleteAll** window. Click the **Yes** button of the **AutomateDeleteAll** window to clear the *Position List* list box of all displayed position samples.



The **FiducialDeleteUnreferenced** window opens. Click the **Yes** button to clear the fiducial coordinate set from the position database.



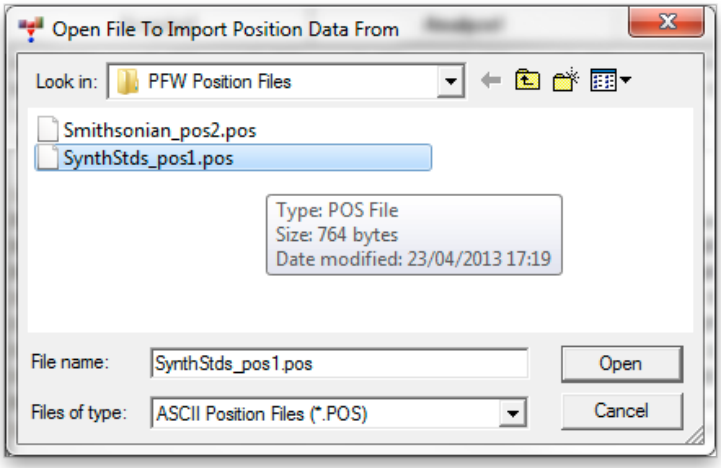
Click the **Import from ASCII File (*.POS File)** button of the **Automate!** dialog box to import position samples from a previously saved ASCII file.



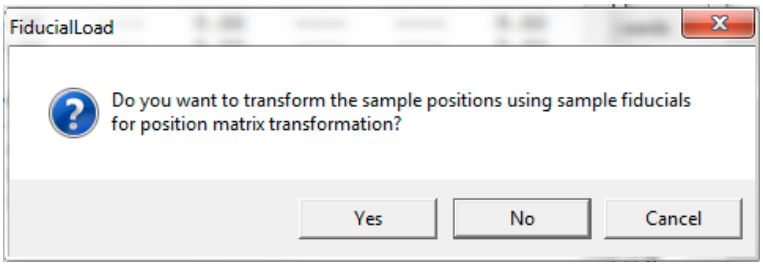
This action opens the **Open File To Import Position Data From** window. The user previously digitized all standard blocks and created a variety of *.POS files. Two *.POS files will be loaded for the pyroxene run documented here: SynthStds_pos1.pos, which contains a range of synthetic simple oxides and silicates, and Smithsonian_pos2.pos, which contains a selection of Smithsonian microbeam standards.

The default location for *.POS files is at C:\Probe Software\Probe for EPMA\PFW Position Files, but this can be changed in the PROBEWIN.INI file.

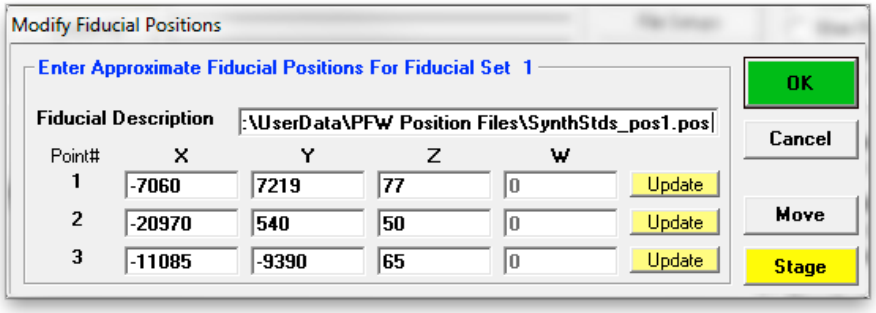
Select the file in the list and click the **Open** button.



This action opens the **FiducialLoad** window. Click the **Yes** button to do a fiducial transformation on this pre-digitized standard block to obtain an accurate set of standard positions.

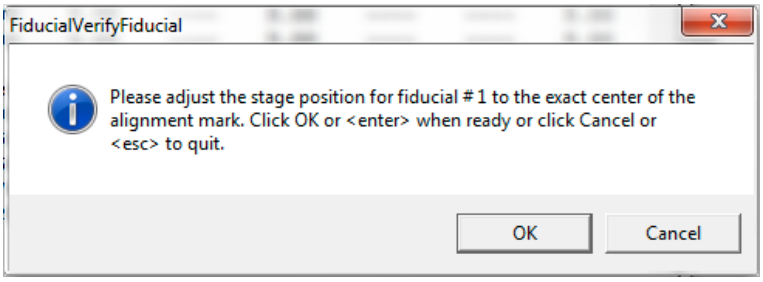


The **Modify Fiducial Positions** window opens. Normally the user would simply accept the defaults or edit the position text boxes for each point, including the appropriate stage location number (JEOL 733 use appropriate W stage position). When done, click the **OK** button.

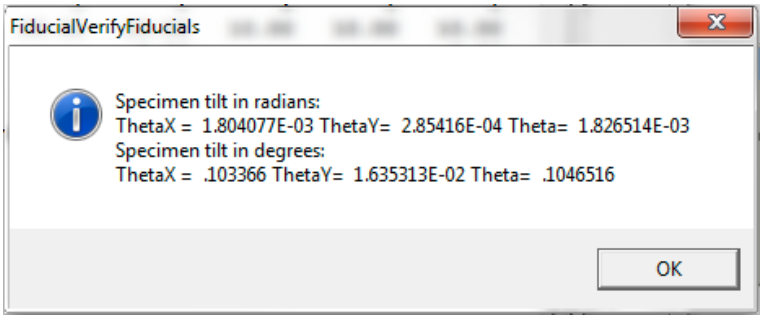


This action causes the stage motors to drive to the first fiducial coordinate in its lookup table.

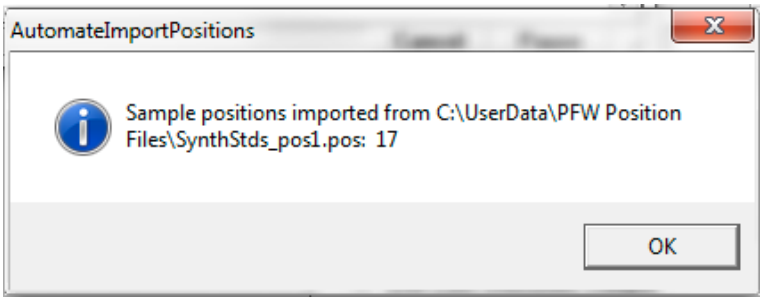
The **FiducialVerifyFiducial** window appears. Adjust the stage motors to center the first fiducial mark, click the **OK** button.



The computer will drive to each of the three fiducial marks for centering. Clicking the **OK** button after the third fiducial mark opens the **FiducialsVerifyFiducials** window. Click this **OK** button.

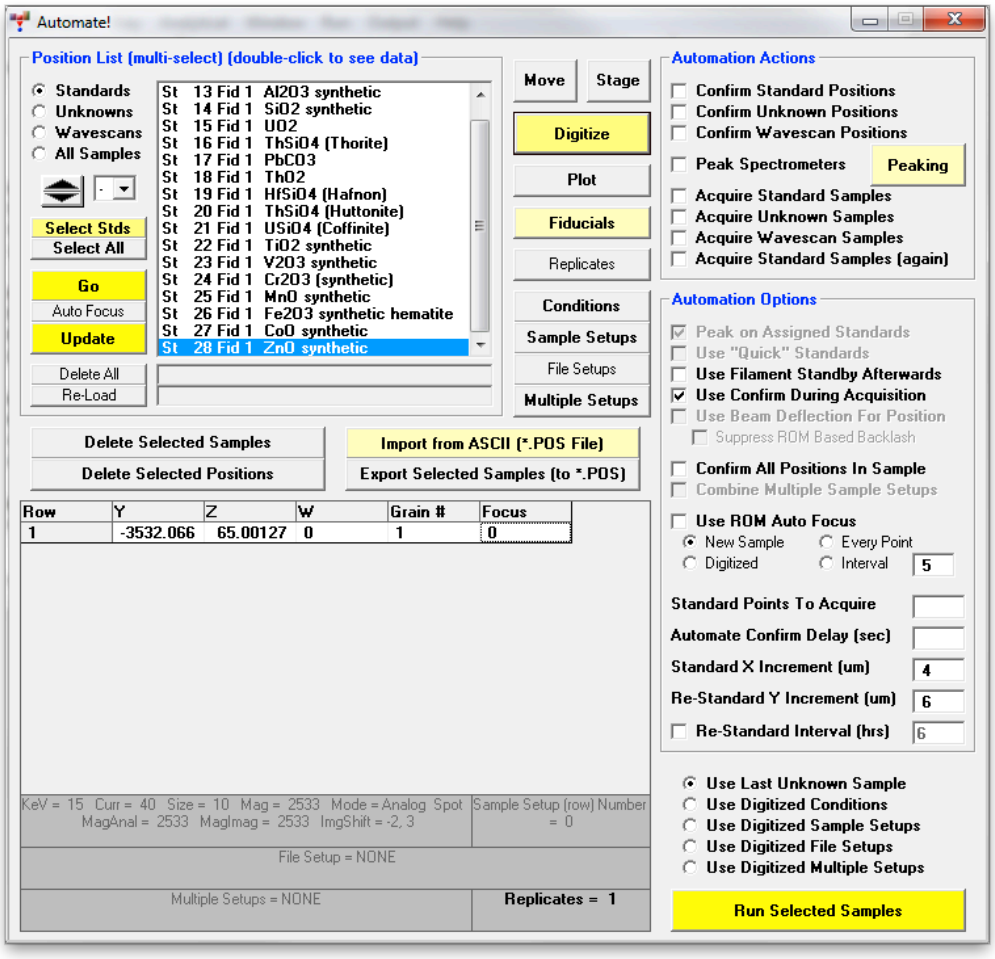


The program then imports and updates the position coordinates of all of the standards in the pre-digitized standard position file. The **AutomateImportPositions** window opens.

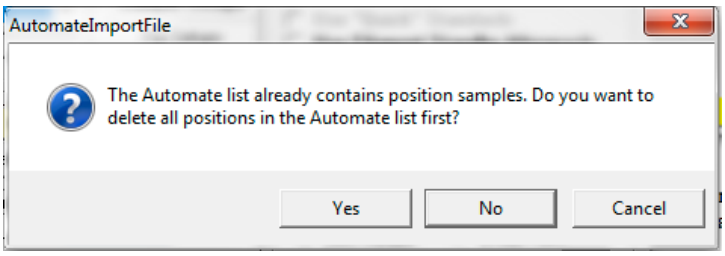


Click the **OK** button returning to the **Automate!** window.

The **Automate!** window would appear as below. The currently transformed standard position file is listed in the *Position List* list box.

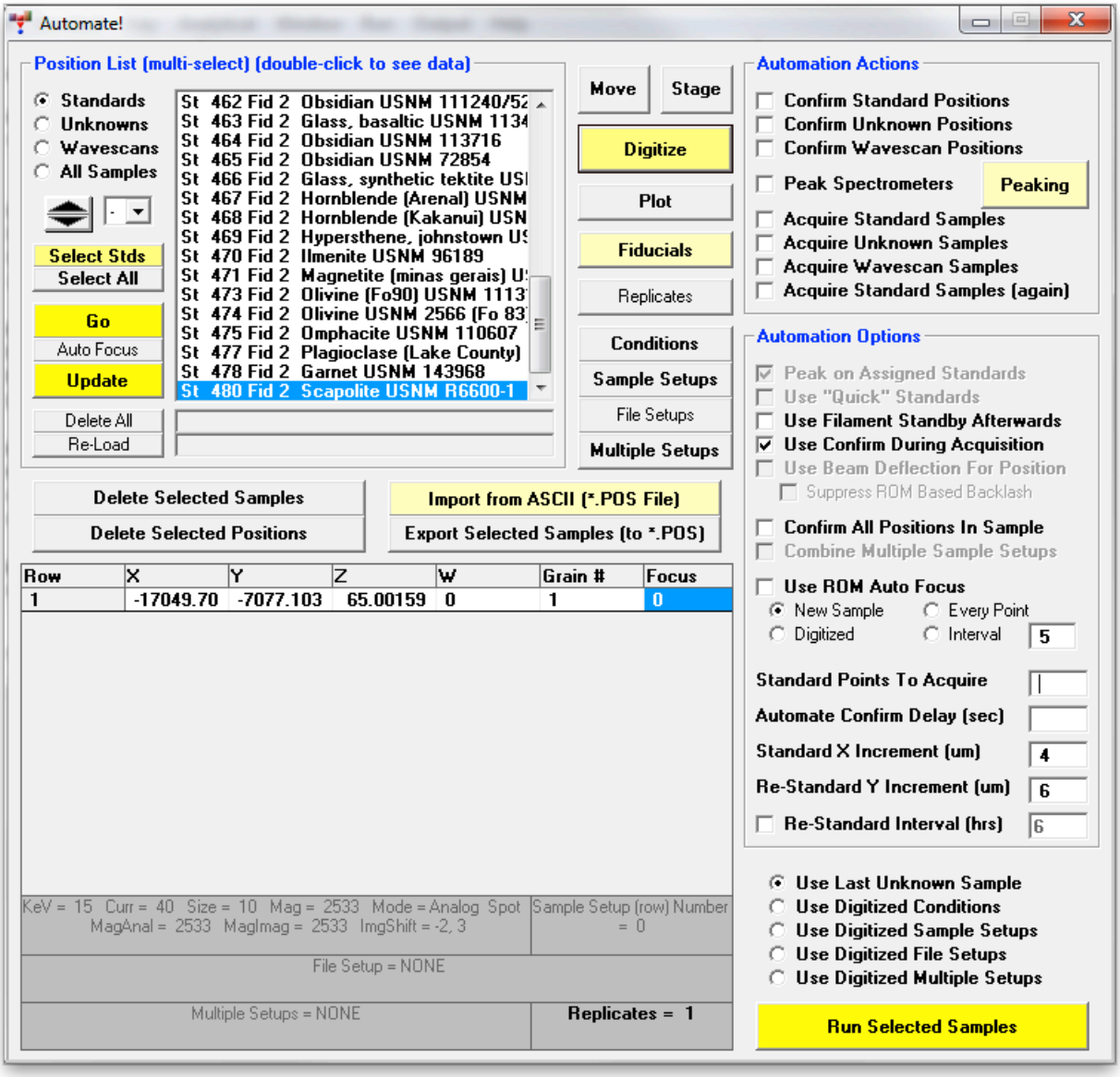


Repeat the same loading procedure for the other standard position files required for use in the automation. After clicking the **Import from ASCII File** button, the **AutomateImportFile** window opens.



Typically, when using more than one standard mount, the user would not delete all positions in the *Position List*, instead appending the additional position files to the first file. Select **No** and import additional standards.

All of the standards loaded are listed in the *Position List* list box of the **Automate!** window. These may now be accessed by the program during any automation action. For instance, it is now possible to drive to any standard located on the imported blocks by double clicking on the standard in the list first and then double clicking on the coordinate row.

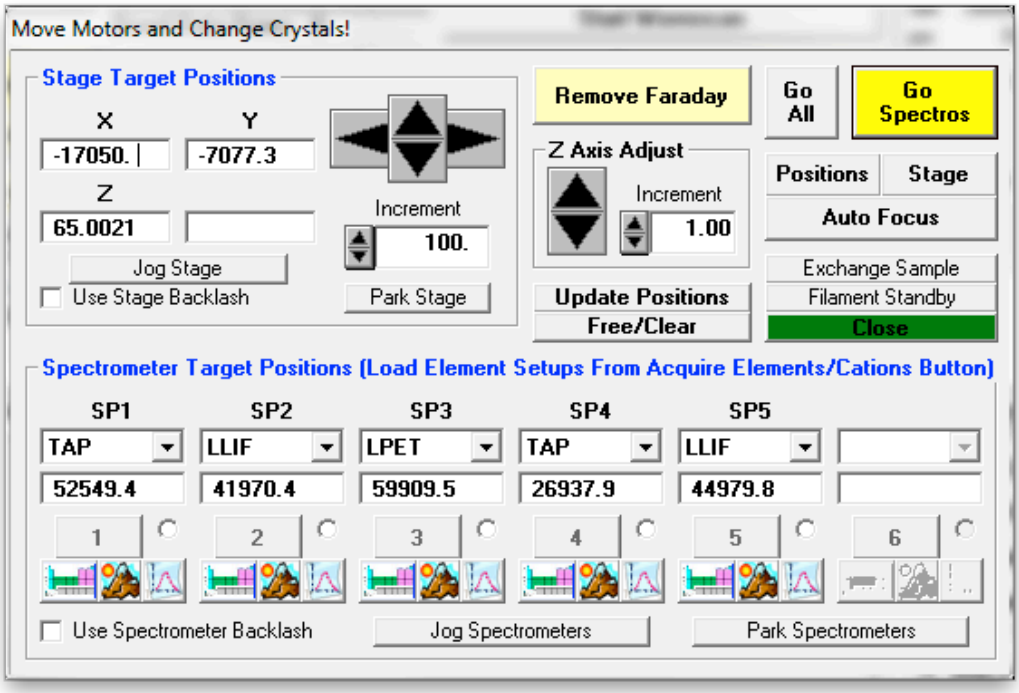


This concludes the initial parameter setup portion of PROBE FOR EPMA.

Manual Peaking and PHA using the Acquire! Window

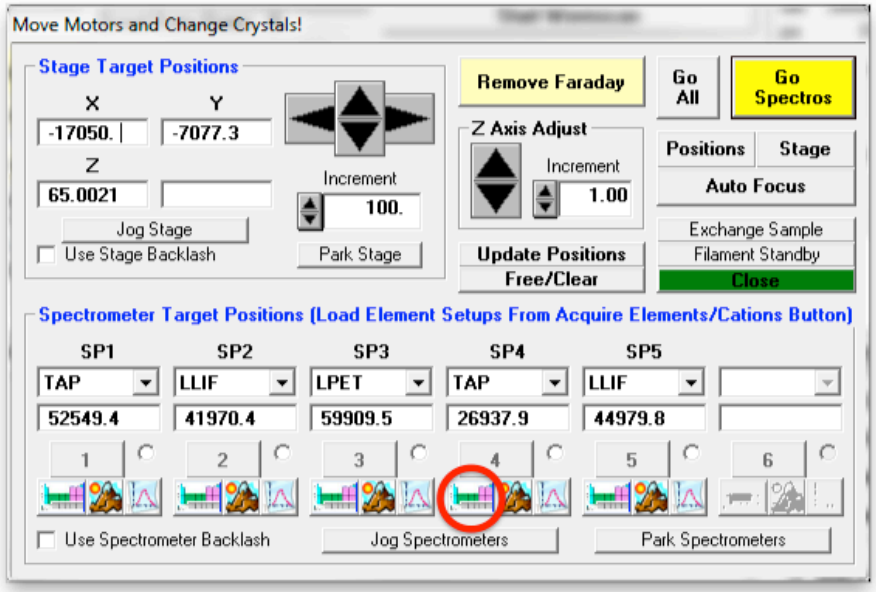
The user may now manually determine the peak positions from the **Acquire!** window.

Move to the silicon standard either by double clicking in the **Automate!** window as described on the previous page, or by clicking the **Move** button found in many windows. This opens the **Move Motors and Change Crystals!** dialog box. Enter the coordinates of the standard into the *Stage Target Positions* text boxes. Click the **Go All** button.

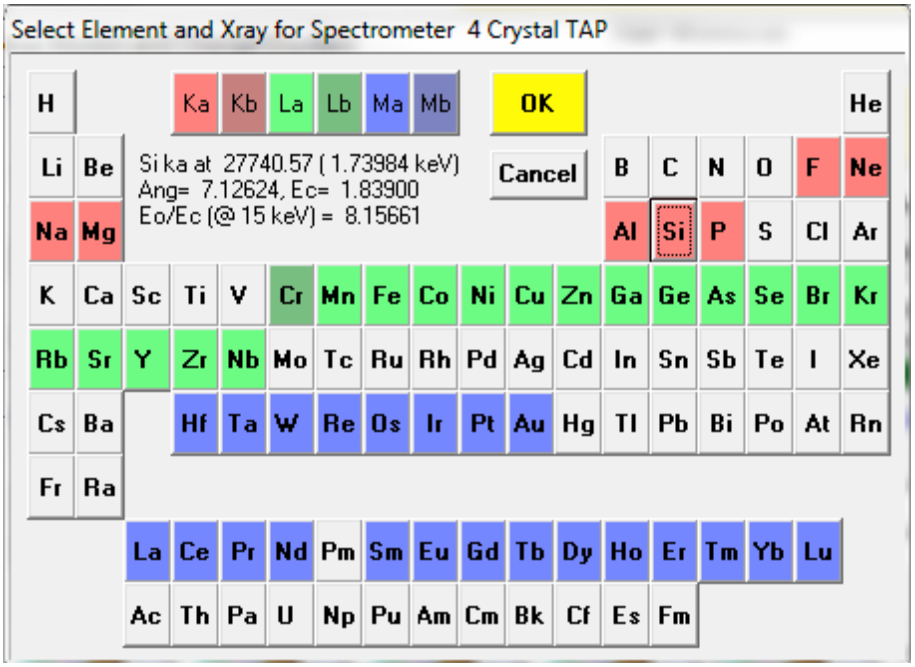


The stage motors will move the stage to the expected position of the standard. Inspect the final X, Y location, adjust if necessary and check the focus.

Inspect the spectrometer crystal type and position text boxes, edit if required. The user may also select the element and x-ray line from the Periodic Table function. Click on the **Periodic Table** () button for spectrometer 4, where silicon is measured in this method.

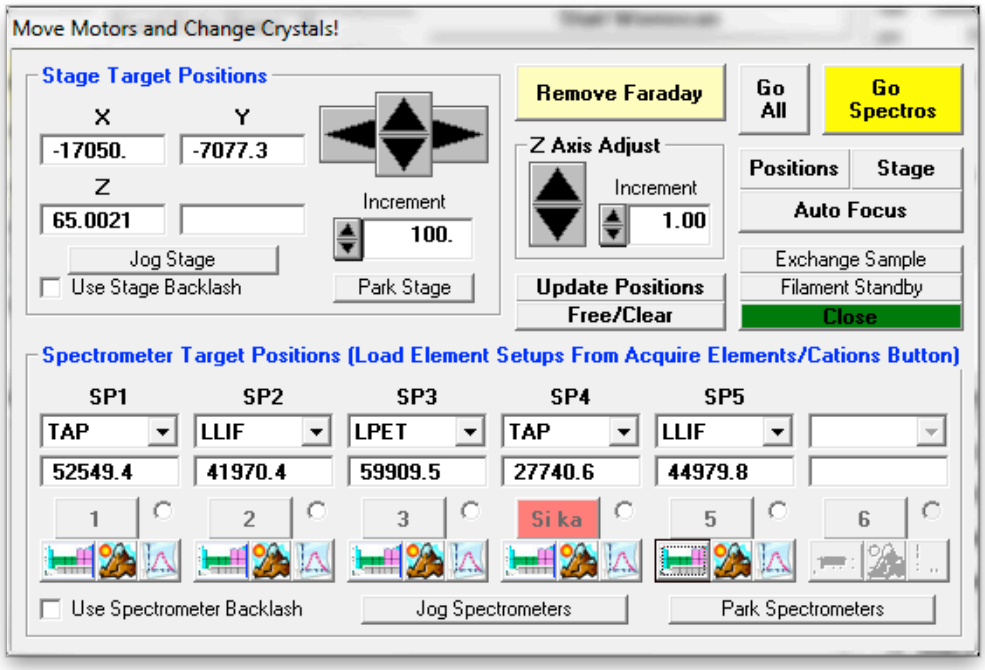


The **Select Element and Xray for Spectrometer** window opens, click on Si in the periodic table.



Click the **OK** button to return to the **Move Motors and Change Crystals!** window.

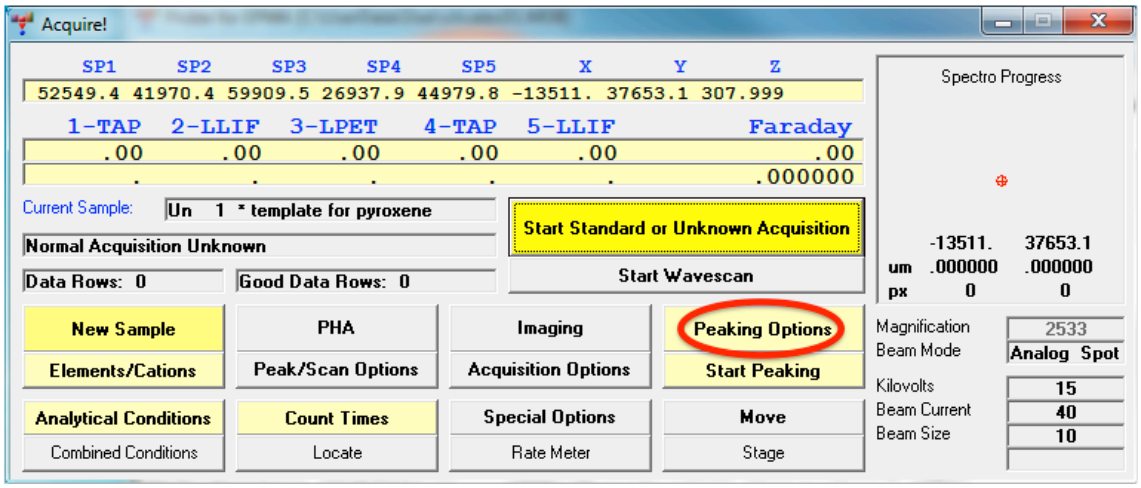
The **Move Motors and Change Crystals!** window appears as below.



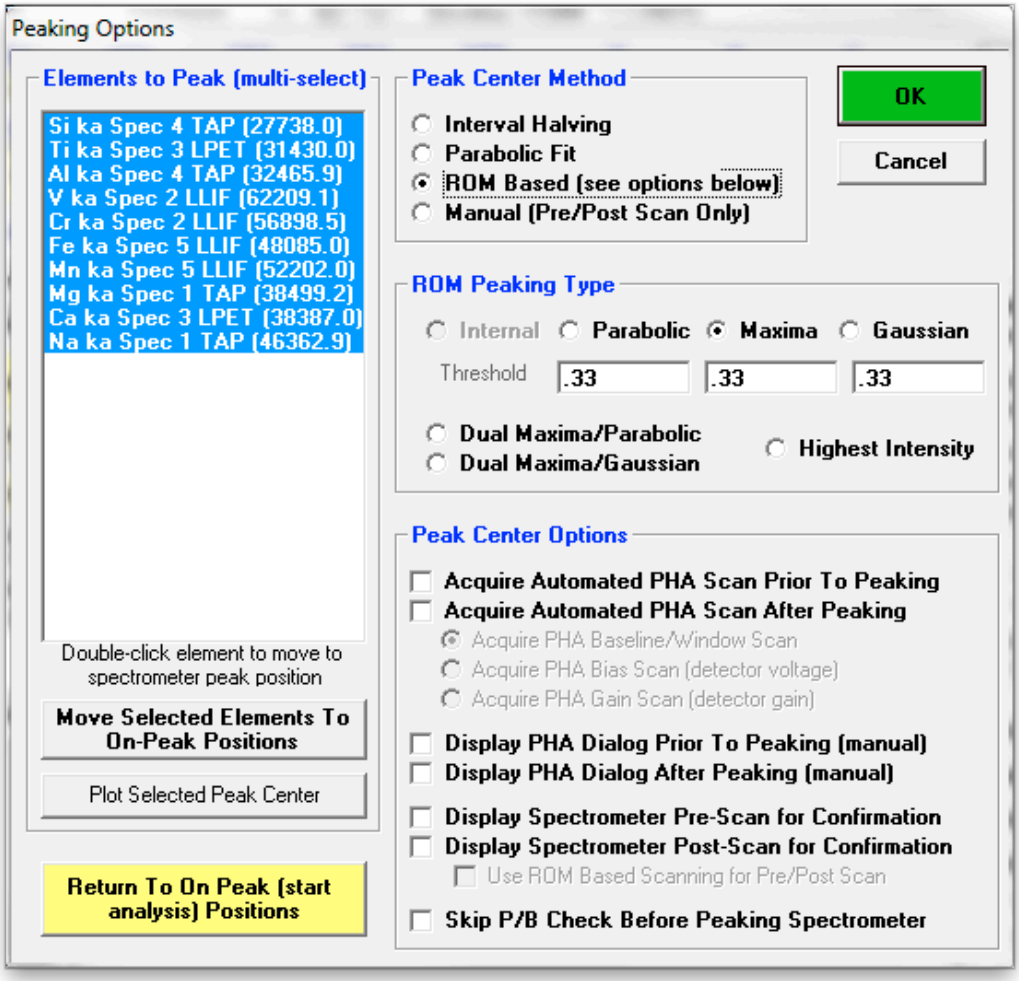
Send the spectrometer directly to the theoretical position by clicking the **Go Spectros** button.

The user could peak spectrometers as well as adjust the PHA parameters (baseline, window, gain, and bias) from the **Move Motors and Change Crystals!** window by clicking on the respective Peaking ( ) and PHA ( ) buttons next to the Periodic Table buttons, but in this exercise the peaking functionality of the **Acquire!** window will be used for this purpose.

Click the **Peaking Options** button of the **Acquire!** window.

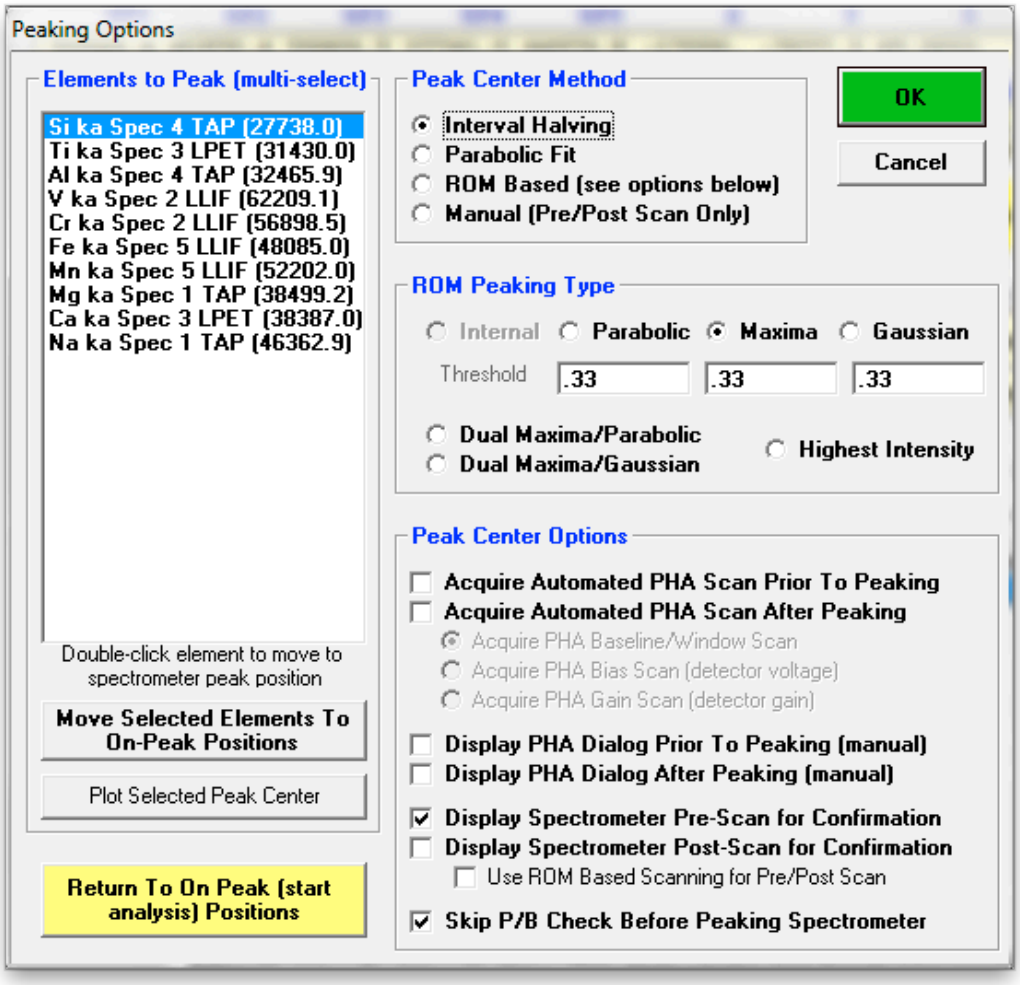


This opens the **Peaking Options** dialog box.



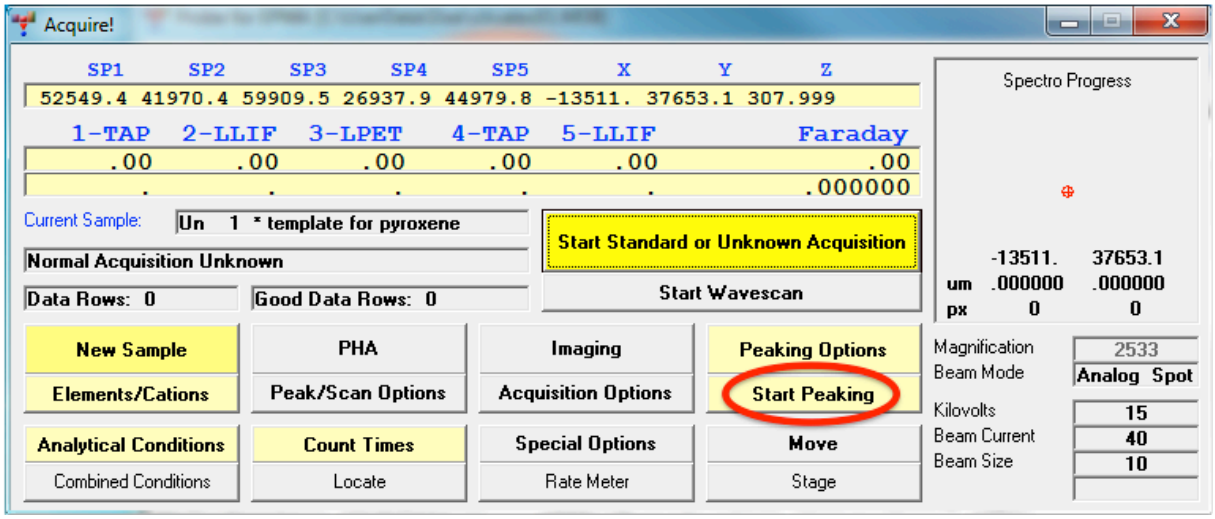
From the *Peak Center Method* group, choose *Interval Halving* (see User's Guide and Reference documentation for discussion of various Peak Center methods) and click *Display Spectrometer Pre-Scan for Confirmation* from the *Peak Center Options* choices. Finally, select the *Si kα Spec 4 TAP (27738.0)* selection under the *Elements to Peak* list box.

The **Peak Center** window should appear as follows. An example of the more commonly used ROM based peaking will be displayed as well.

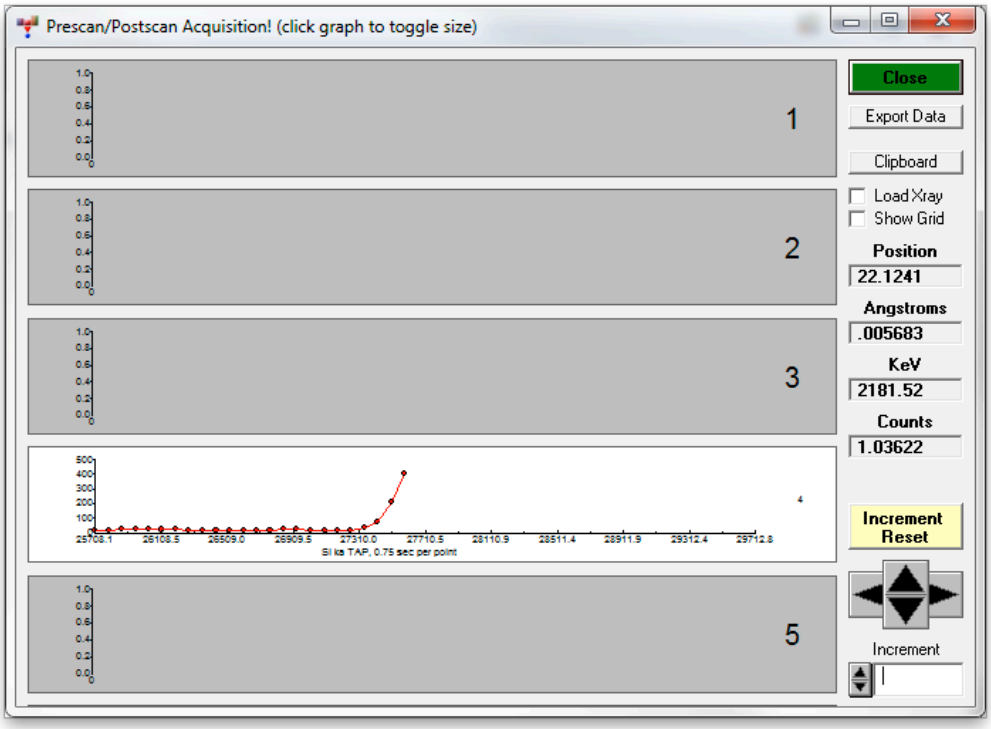


Click the **OK** button to close the **Peaking Options** dialog box.

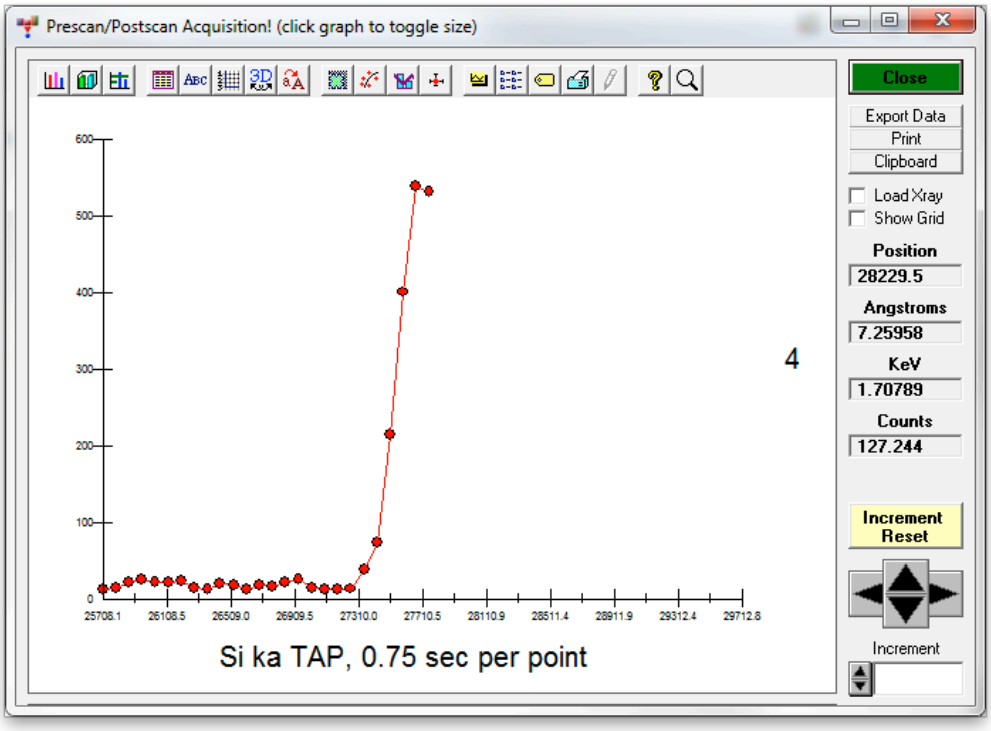
Click the **Start Peaking** button in the **Acquire!** window.



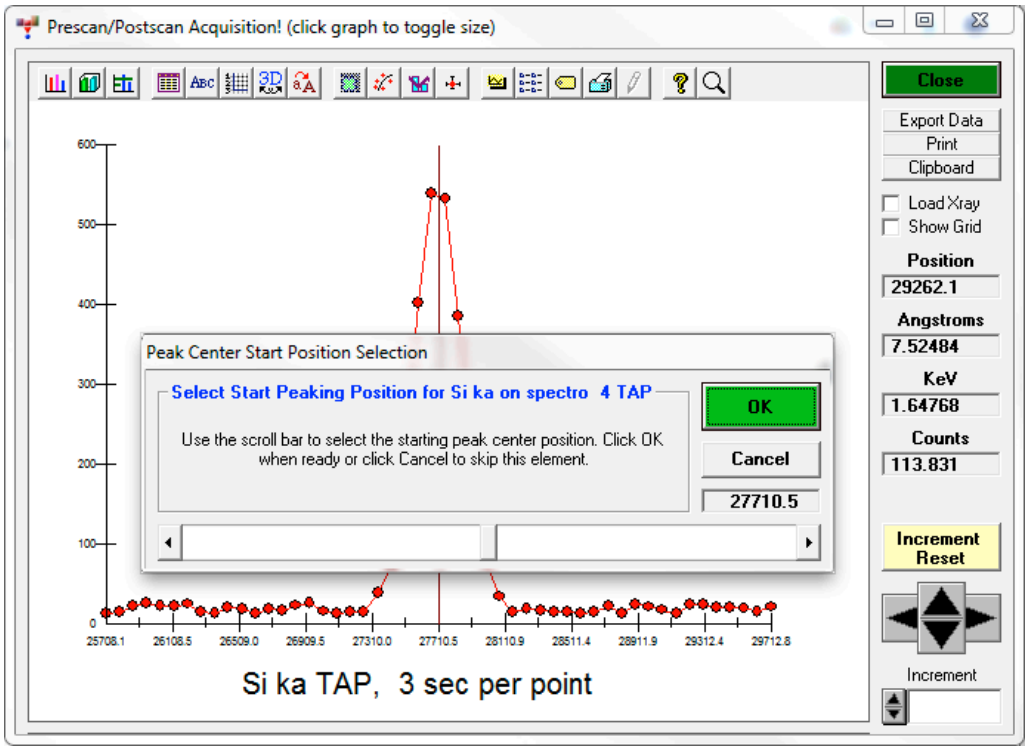
This action opens the **Prescan/Postscan Acquisition** window. The software then performs a peak pre-scan (40 step, user defined parameter) on spectrometer 4 in the Si Kα region.



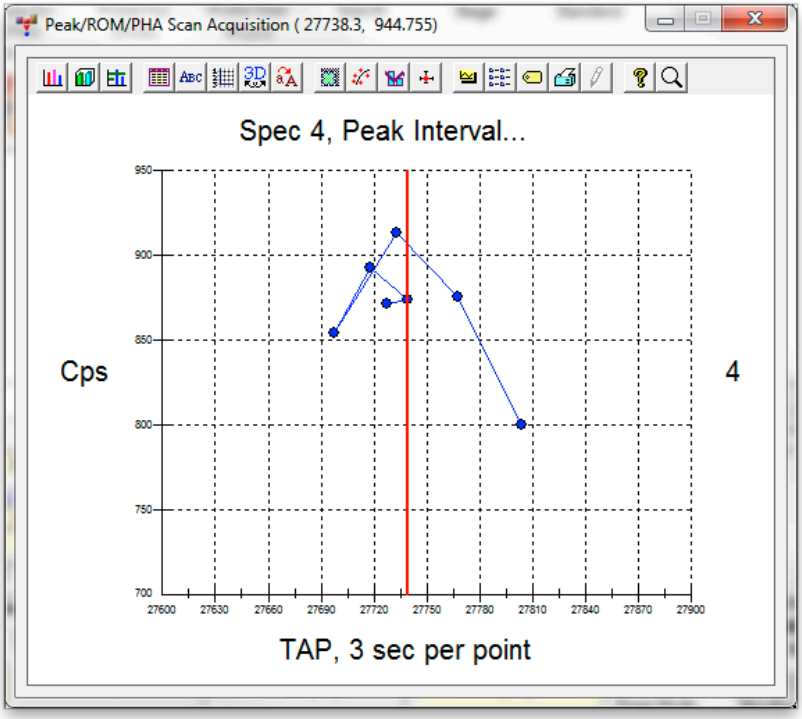
Click on a graph to toggle the display between graphs for all spectrometers and a single spectrometer.



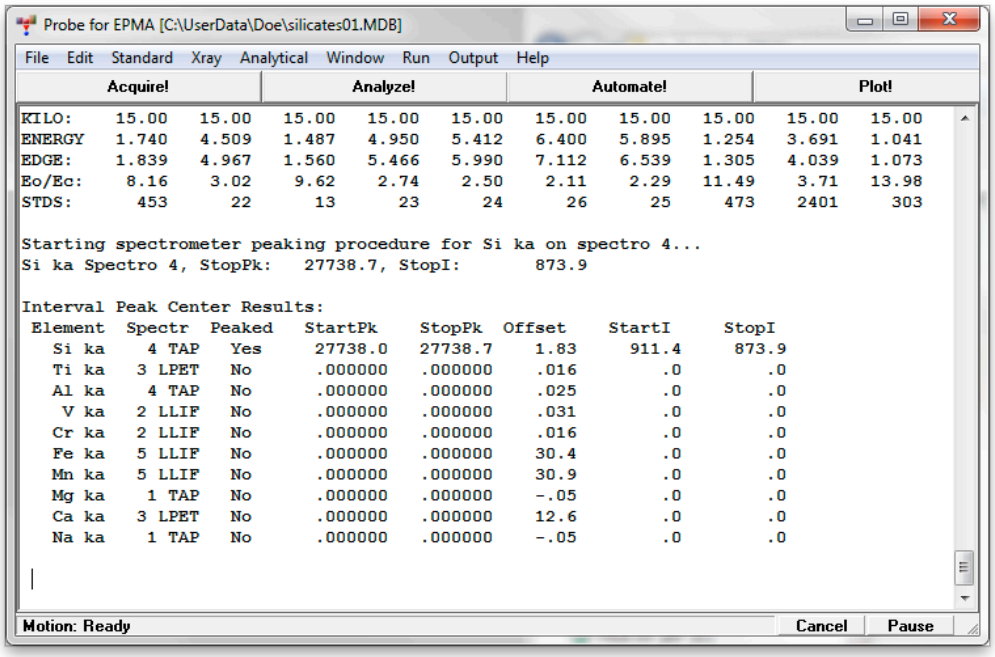
Upon completion of the spectrometer pre-scan the **Peak Center Start Position Selection** window opens. Slide the scroll bar to move the vertical (maroon) peak line to match the actual x-ray maximum position. This selects a starting peak center position for the peaking routine.



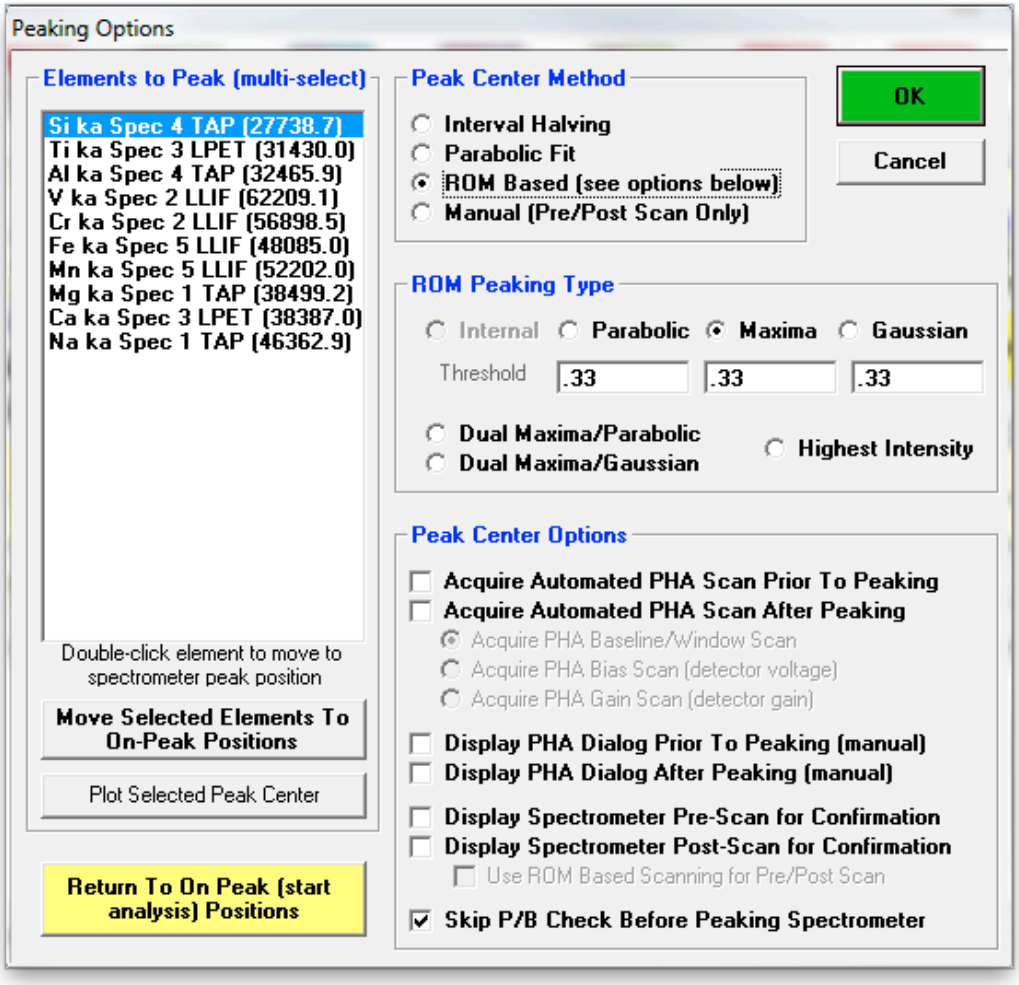
Click the **OK** button when manually centered. This initiates a peak center routine to locate the precise peak center. The **Peak/ROM/PHA Scan Acquisition** window opens and real time peaking can be viewed.



The results appear in the main log window, displayed below.

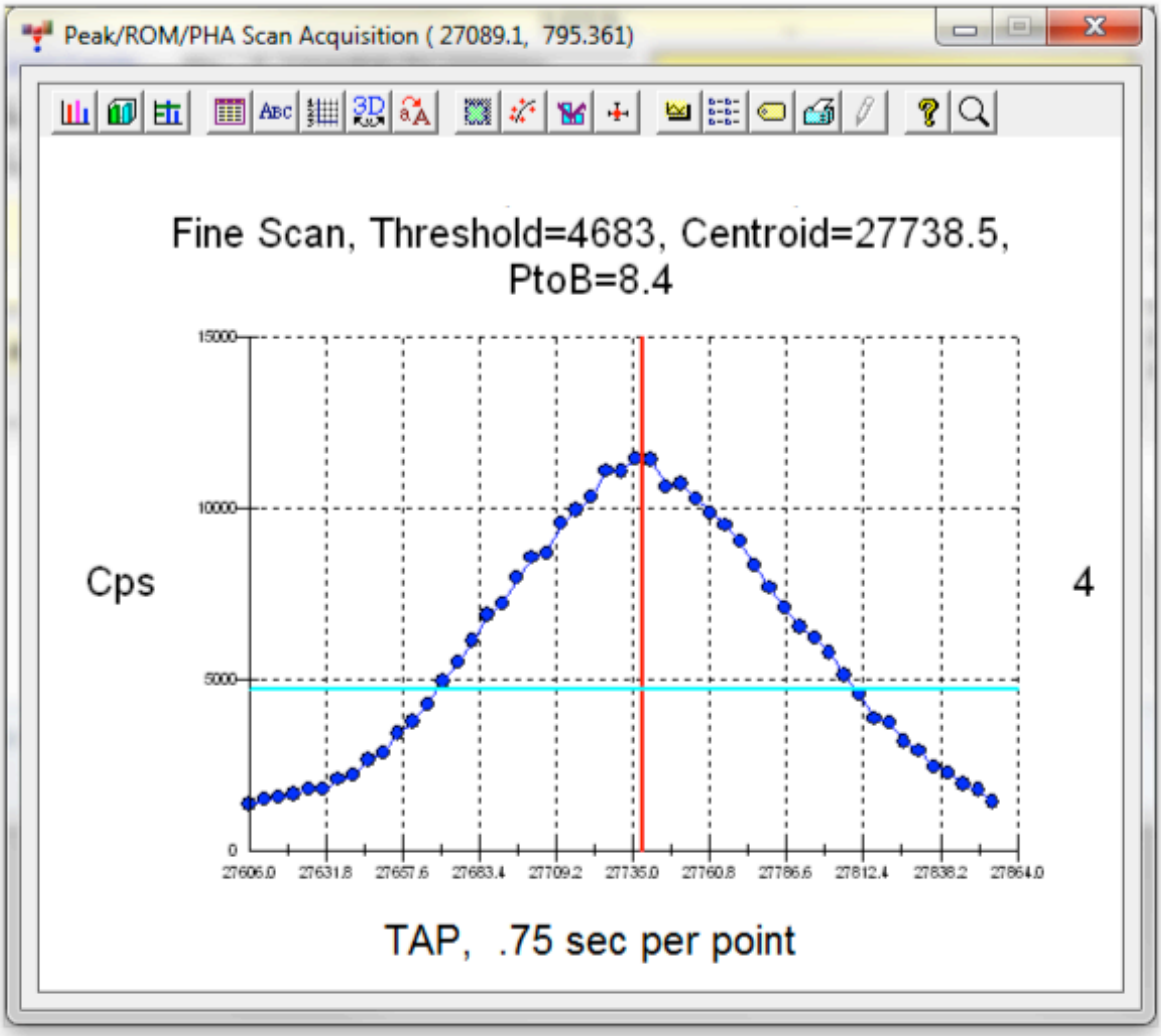


While *Interval Halving* is the most accurate peaking method, it is also the slowest. Many microprobes support *ROM Based* peak center scans. An illustration is provided below. Select the *ROM Based* method and highlight the appropriate elements in the *Elements to Peak* list. Click the **OK** button.

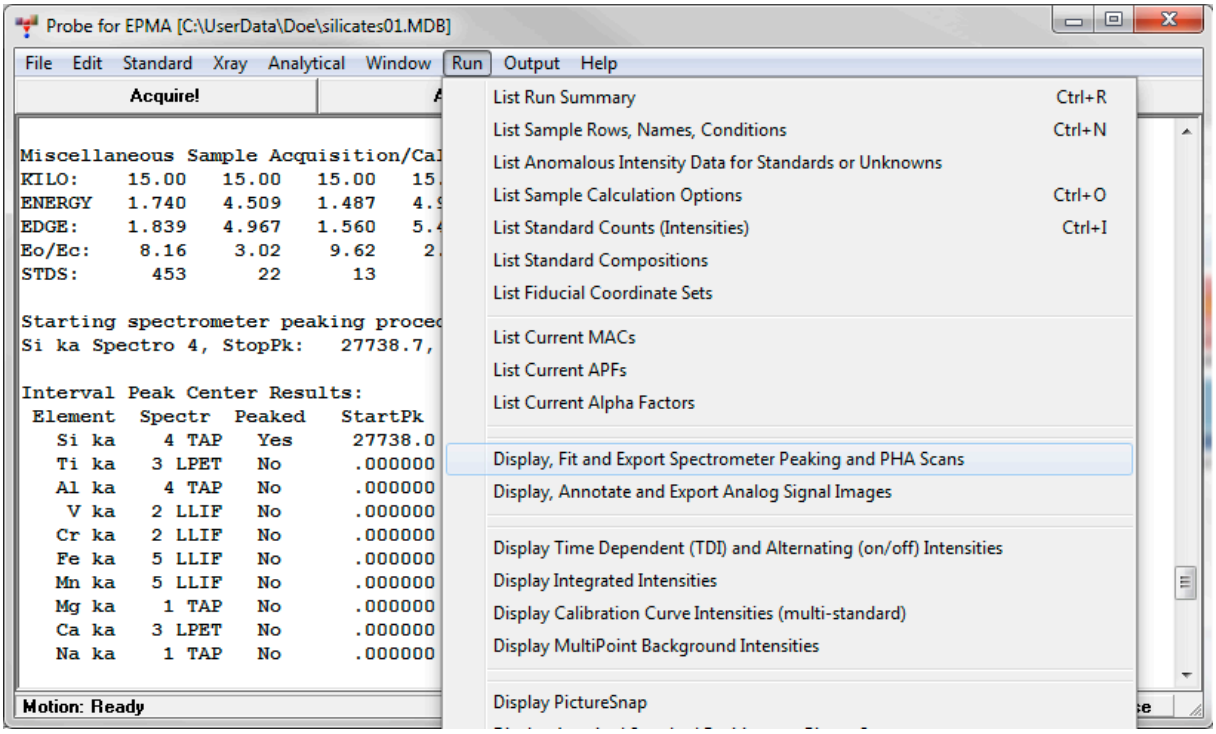


Start the peak process by clicking the **Start Peaking** button in the **Acquire!** window.

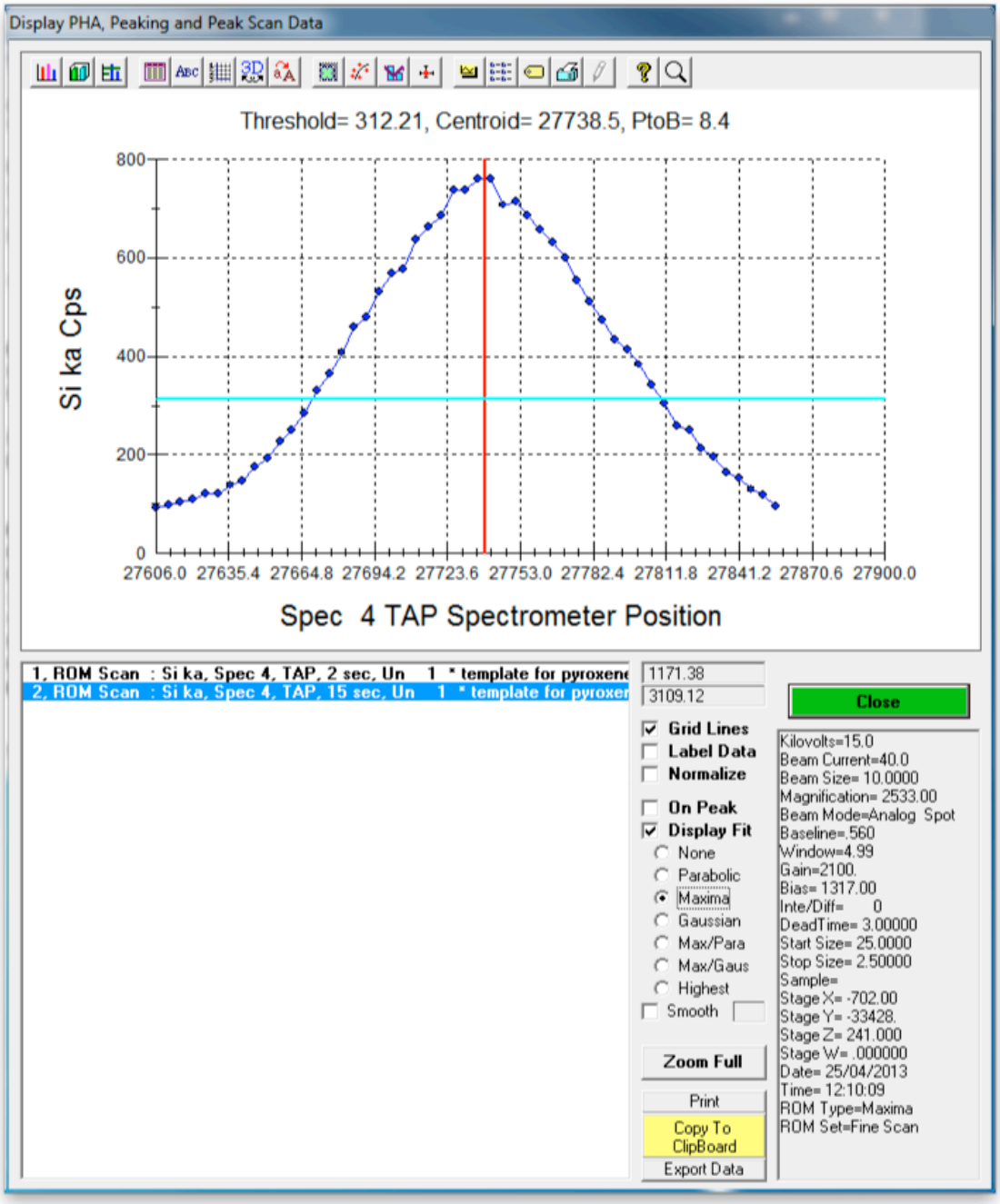
Real time peaking may be observed (in the **Peak/ROM/PHA Scan Acquisition** window) as the ROM scan progresses. After completion, the **Peak/ROM/PHA Scan Acquisition** window opens again and the peak is displayed.



All spectrometer peaking and PHA scans are automatically saved to the probe run. These can be reviewed by selecting the **Run | Display, Fit and Export Spectrometer Peaking and PHA Scans** menu in the main Probe for EPMA log window.



The **Display PHA, Peaking and Peak Scan Data** window opens.



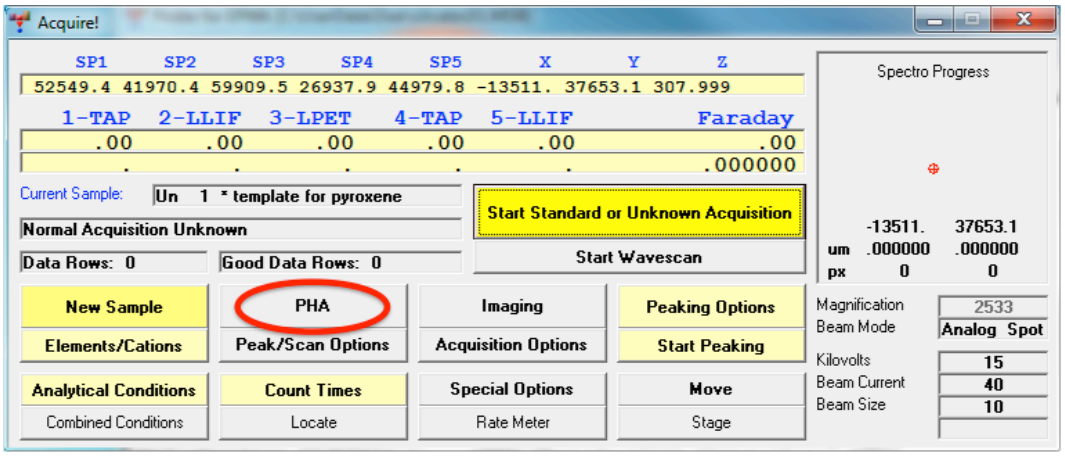
The user has the option to check the fitting (change radio buttons in list) and view the threshold values (set in the PROBEWIN.INI file as well as edited in the **Peaking Options** window). As well as Export capabilities.

Click the **Close** button to exit this dialog.

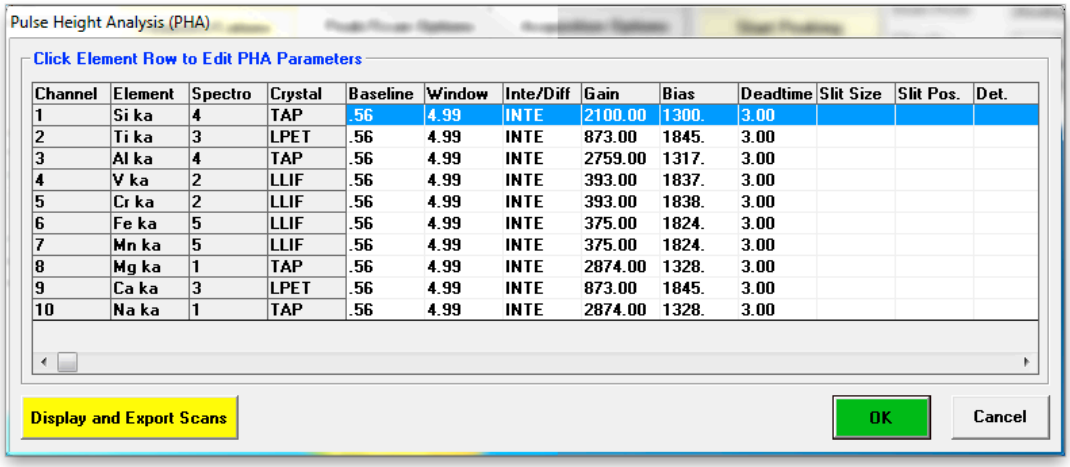
Next, the PHA properties for each element need to be checked. Each spectrometer has a single channel analyzer that selects pulses of interest (the amplitude of the pulse from the detector is proportional to the energy of the incident x-ray photon) and then outputs this pulse to the counting electronics. PROBE FOR EPMA allows adjustment of the baseline, window, whether the SCA is run in integral or differential mode, and the gain and/or bias voltage of the counter.

Most microprobe automation systems have gain and bias hardware interfaces. However, JEOL instruments typically run a fixed gain setting and allow bias scans on a per element basis. The object is to place the pulse height peak near 4 volts by adjusting the bias. Each element may have a slightly different bias value. On Cameca instruments both bias and gain scans can be performed and the gain value can be adjusted to change the position of the pulse height maximum, typically to a value around 2-2.5 volts.

Click the **PHA** button in the **Acquire!** window.

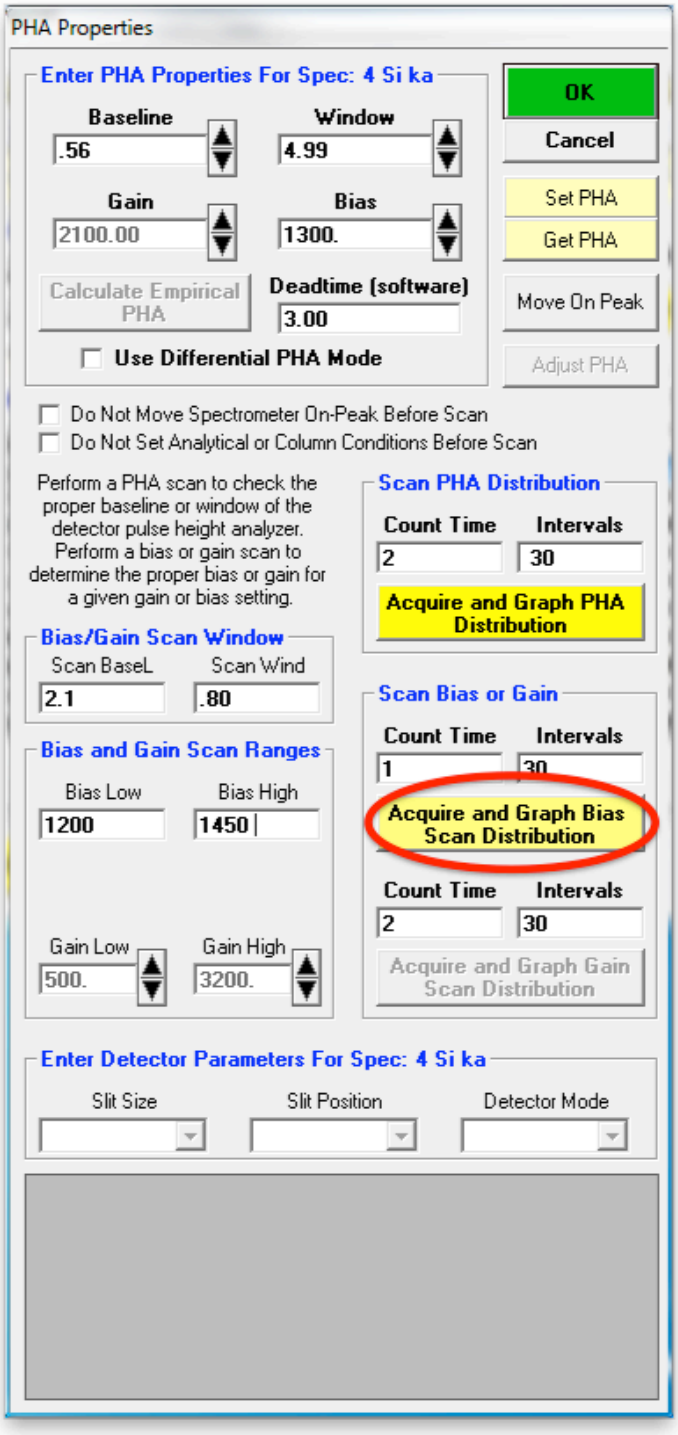


The **Pulse Height Analysis (PHA)** window opens.

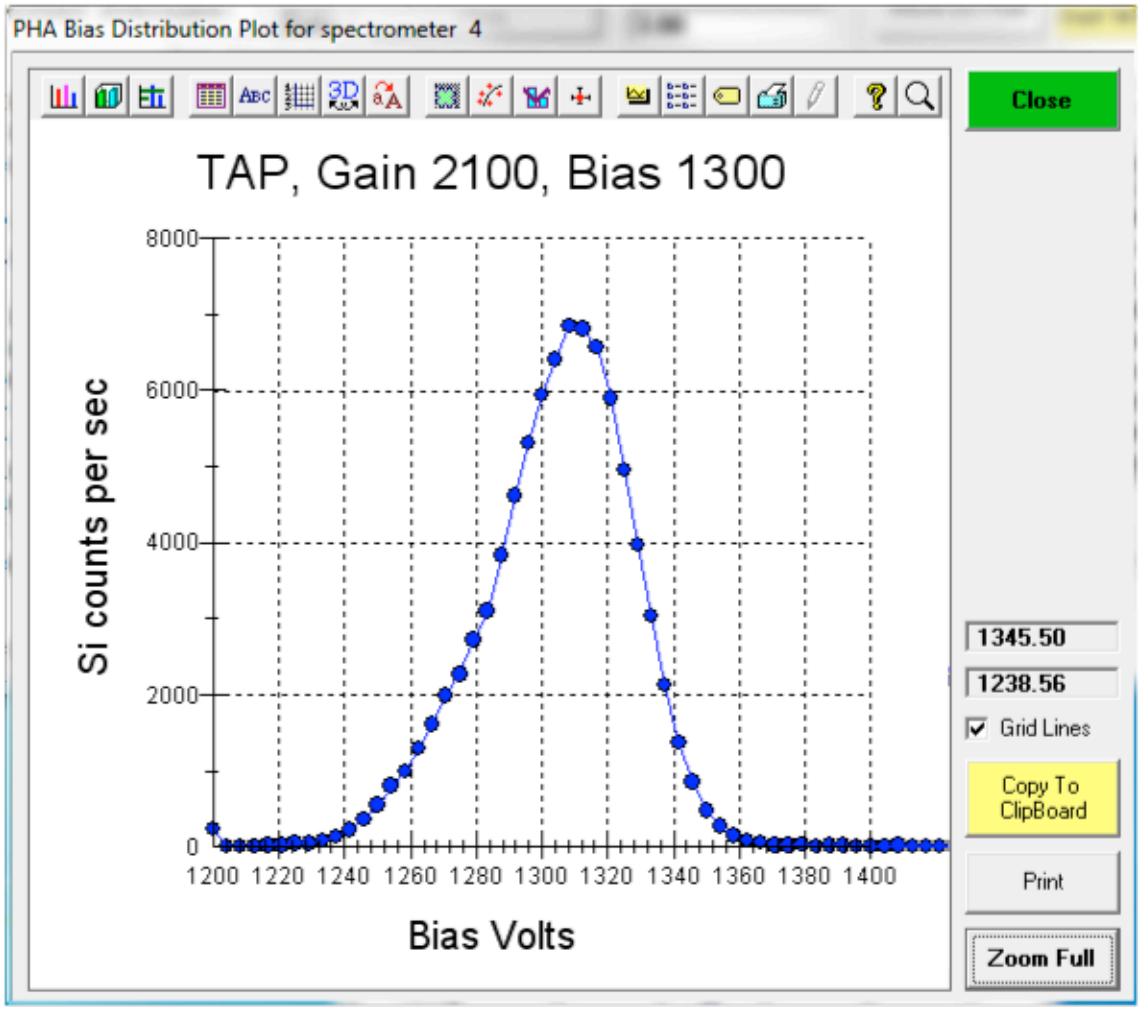


To select the first element to evaluate (Si), click its element row.

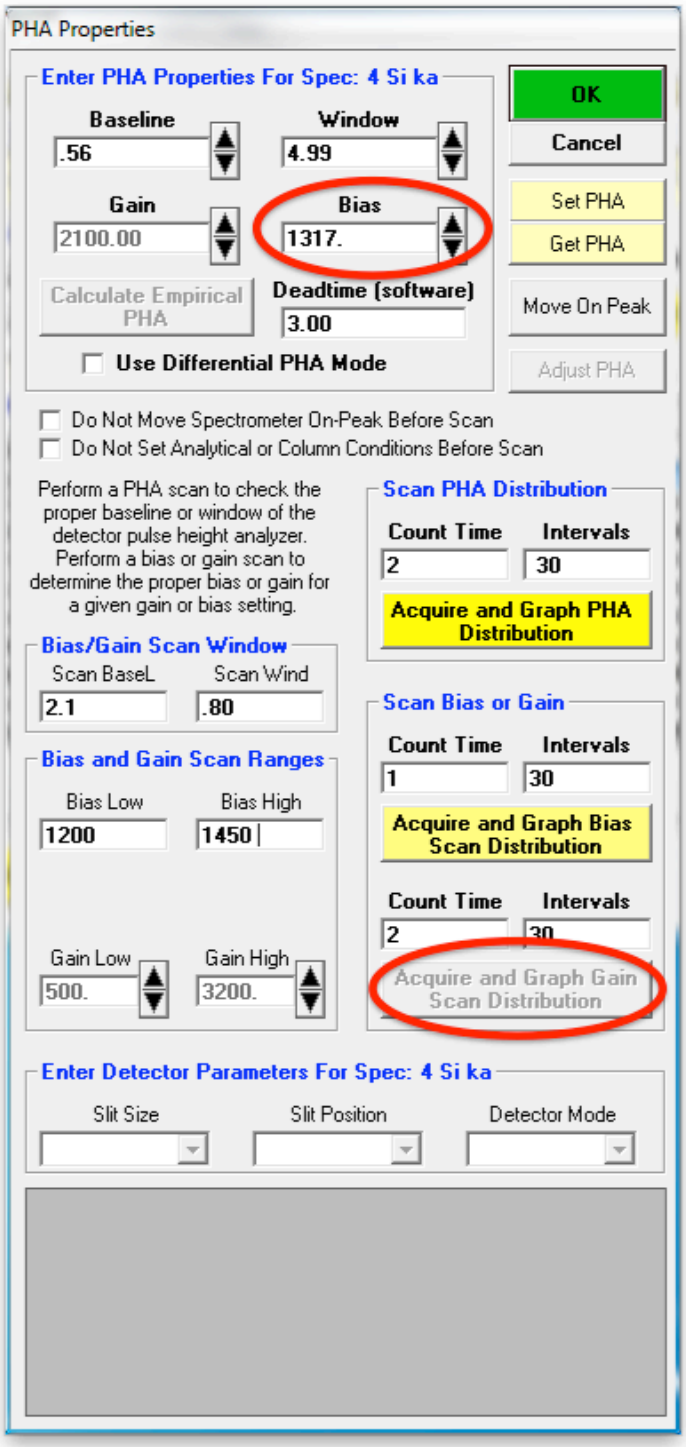
The **PHA Properties** window appears. Run a Bias scan for Si by clicking on the **Acquire and Graph Bias Scan Distribution** button.



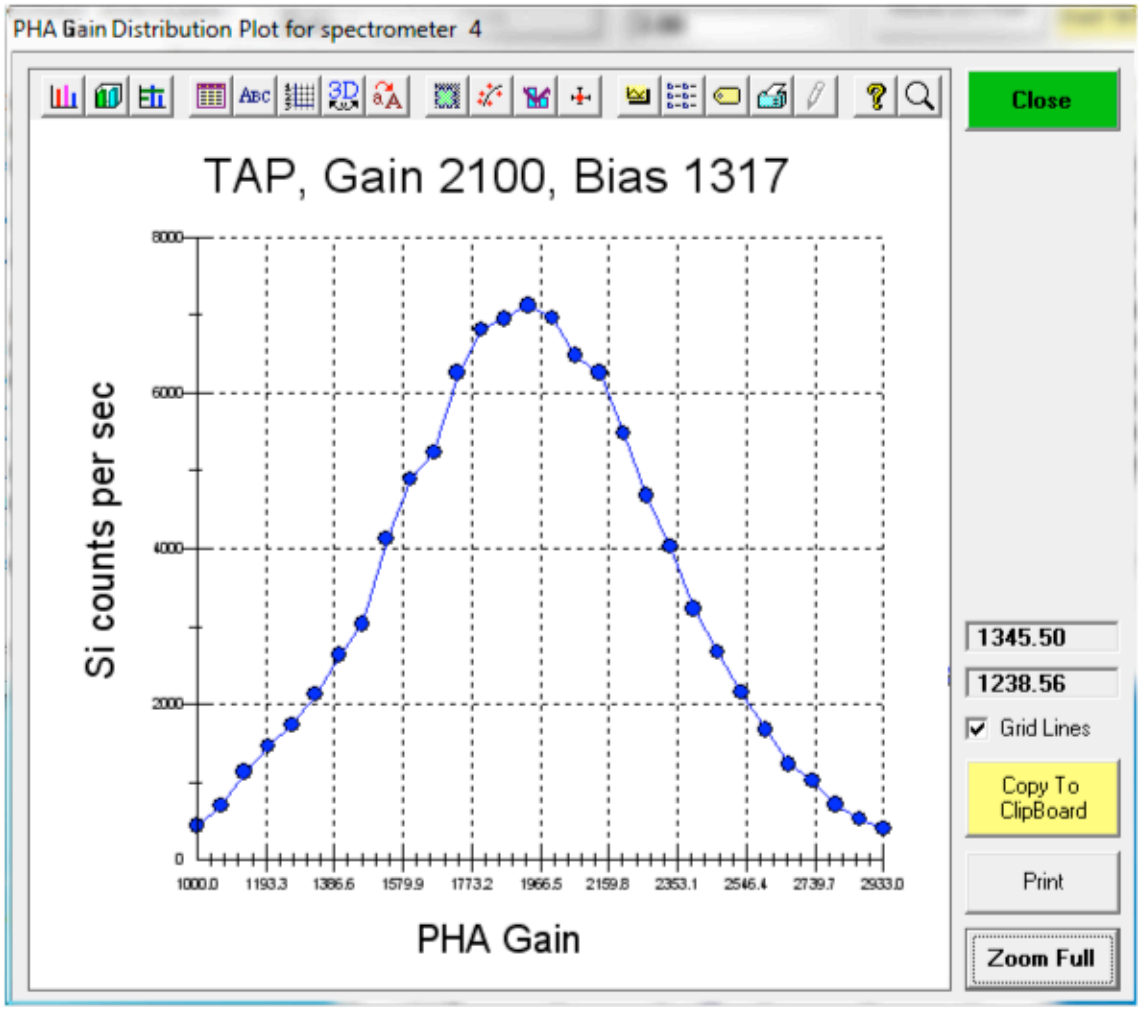
At the completion of the bias scan the **PHA Bias Distribution Plot for spectrometer 4** window will be visible. Read the bias value for the maximum Si count rate from the plot and click the **Close** button.



Edit the *Bias* text field in the **PHA Properties** window with the appropriate value, here 1317. On Cameca instruments only, run a gain scan by clicking on the **Acquire and Graph Bias Scan Distribution** button.



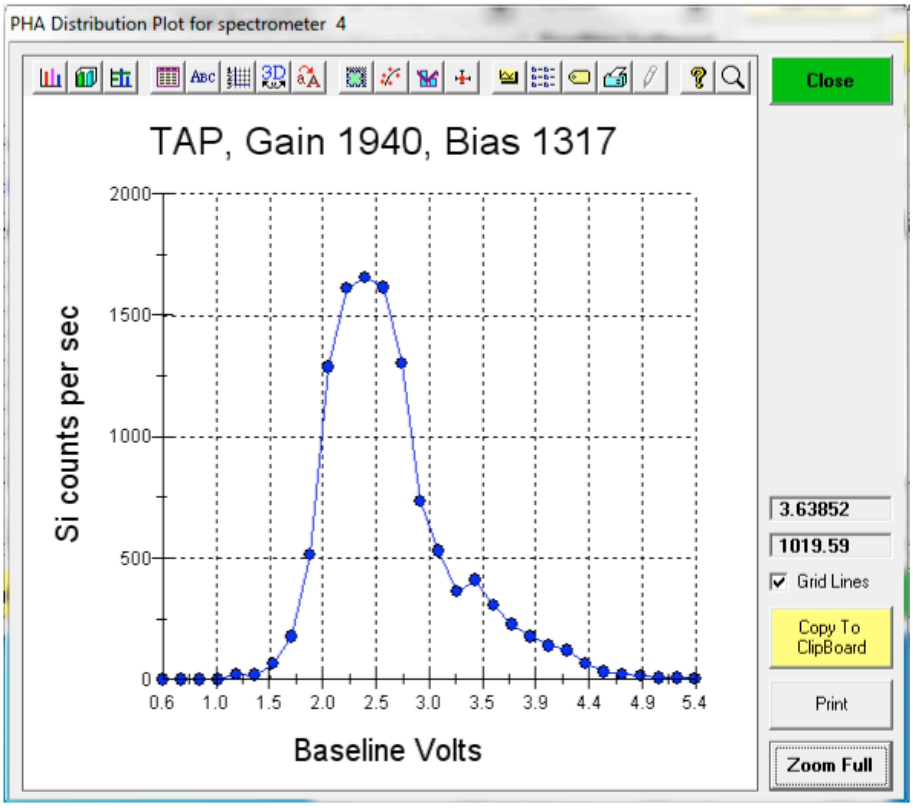
At the completion of the gain scan the **PHA Gain Distribution Plot for spectrometer 4** window will be visible. Read the gain value for the maximum Si count rate from the plot and click the **Close** button.



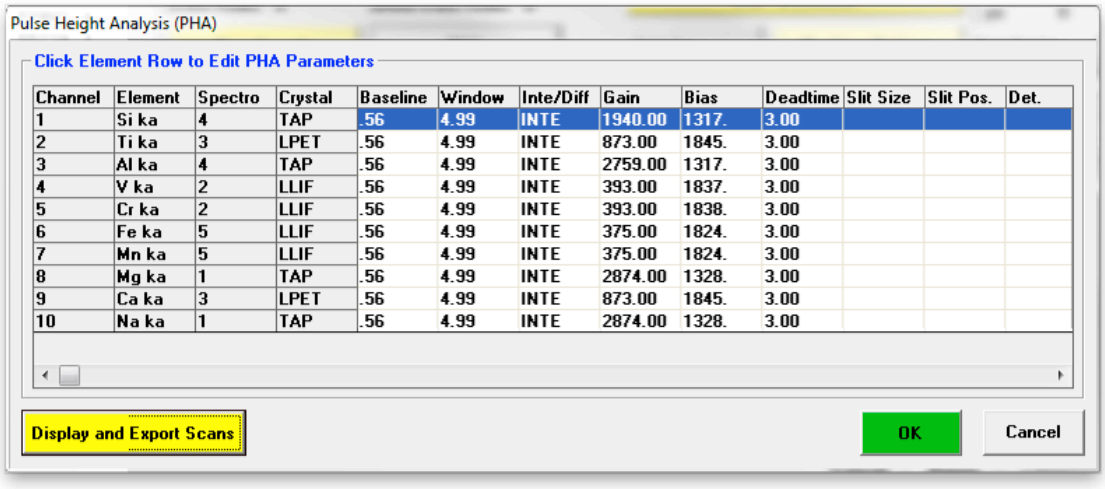
Edit the *Gain* text field in the **PHA Properties** window with the appropriate value, here 1940.

Next, run a PHA scan to evaluate the appropriate baseline and window (if in differential mode operation) settings as well as gain and bias settings for the element of interest. Click the **Acquire and Graph PHA Distribution** button in the **PHA Properties** window.

The **PHA Distribution Plot** window will display at the completion of the scan.



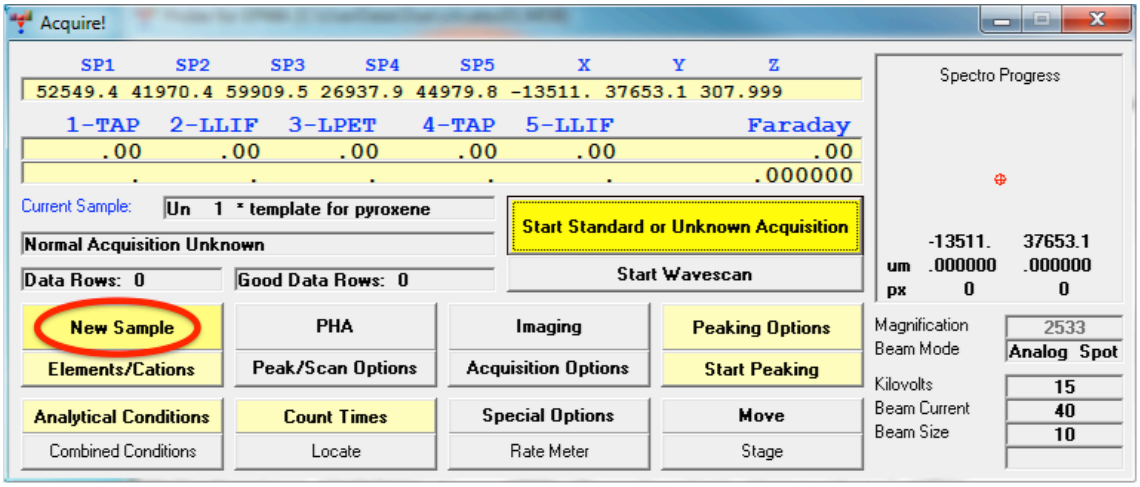
Click the **Close** button to return to the **PHA Properties** window. Click the **OK** button to close the **PHA Properties** window. Note the new *Bias* and *Gain* values for Si in the **Pulse Height Analysis (PHA)** window.



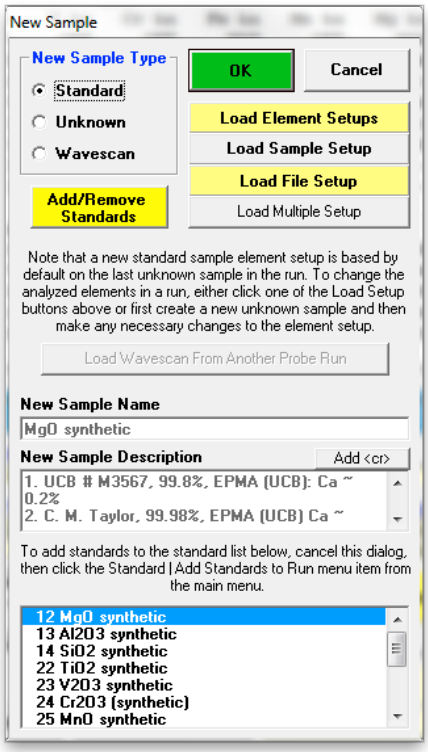
Click the **OK** button to close the **Pulse Height Analysis (PHA)** window returning to the **Acquire!** window.

Manual Count Acquisition using the Acquire! Window

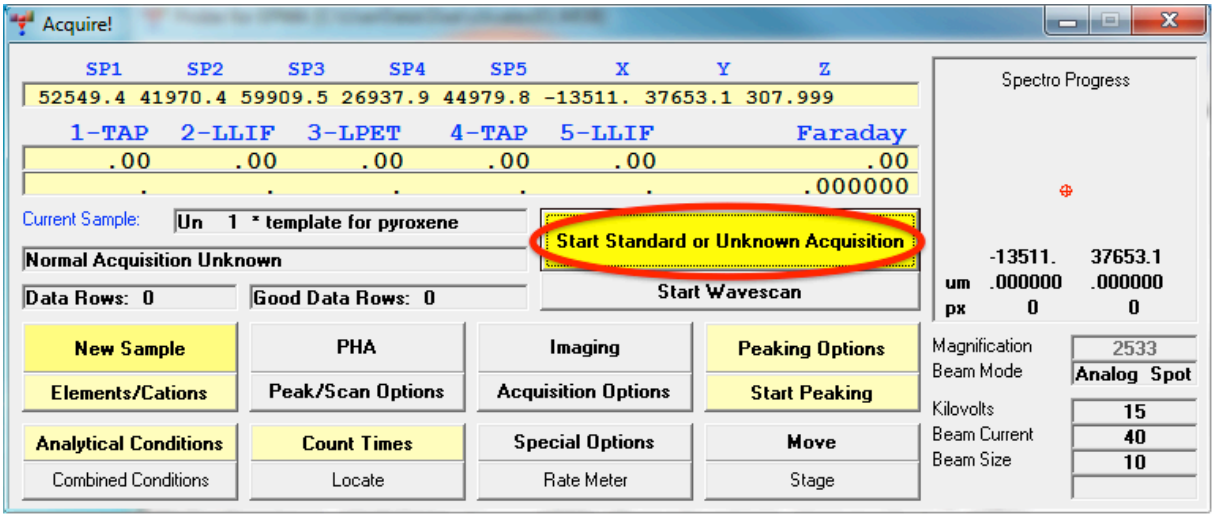
To acquire a single point of x-ray count data for a standard proceed as follows. From the **Acquire!** dialog box click the **New Sample** button.



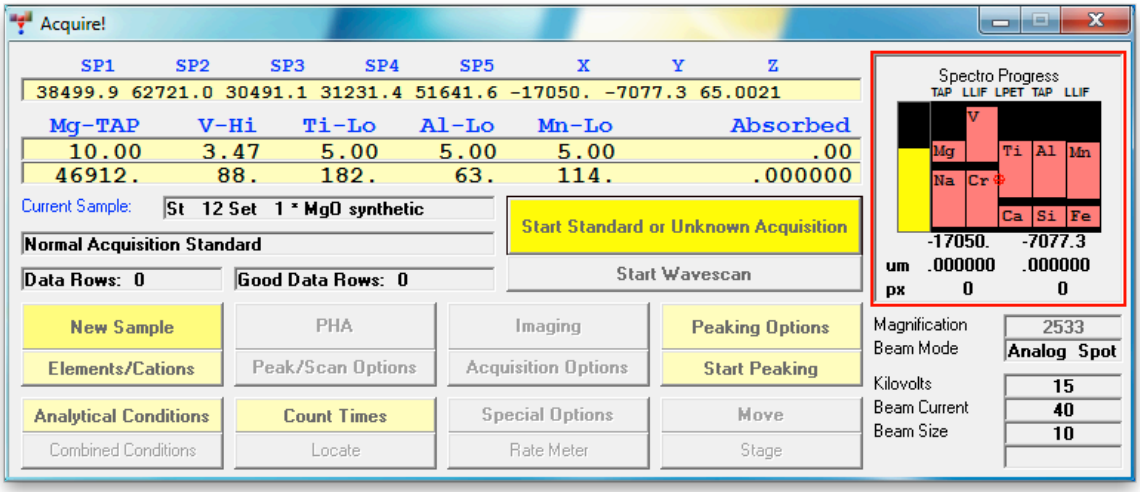
This opens the **New Sample** window. Click on *Standard* from the *New Sample Type* buttons. This allows the user to specify a standard from the list now active at the bottom of the *New Sample* dialog box. Click *12 MgO synthetic*, its name now appears under *New Sample Name*. Click the **OK** button when done.



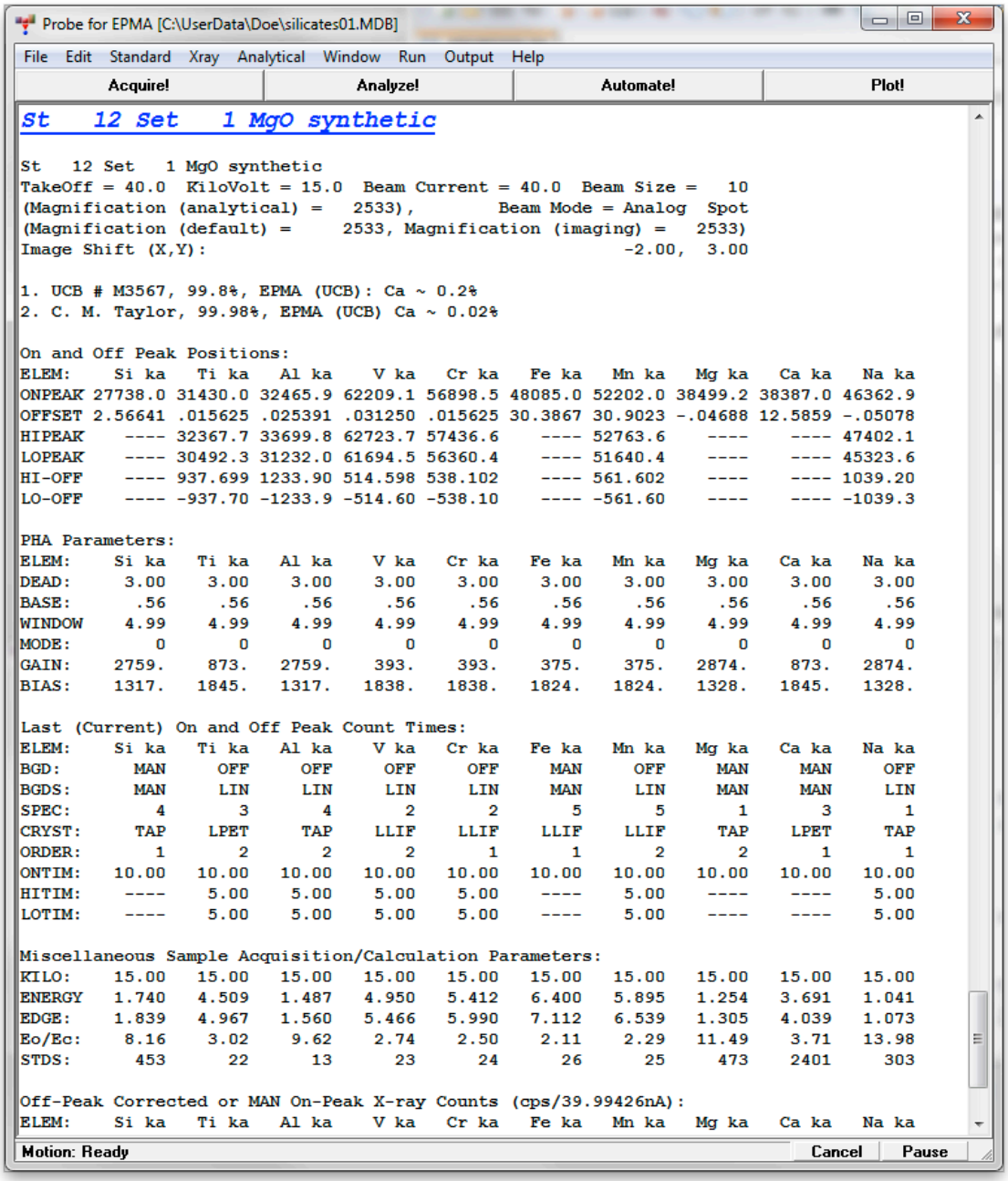
Check the optical focus on the standard and click the **Start Standard or Unknown Acquisition** button of the **Acquire!** window to initiate the data acquisition.



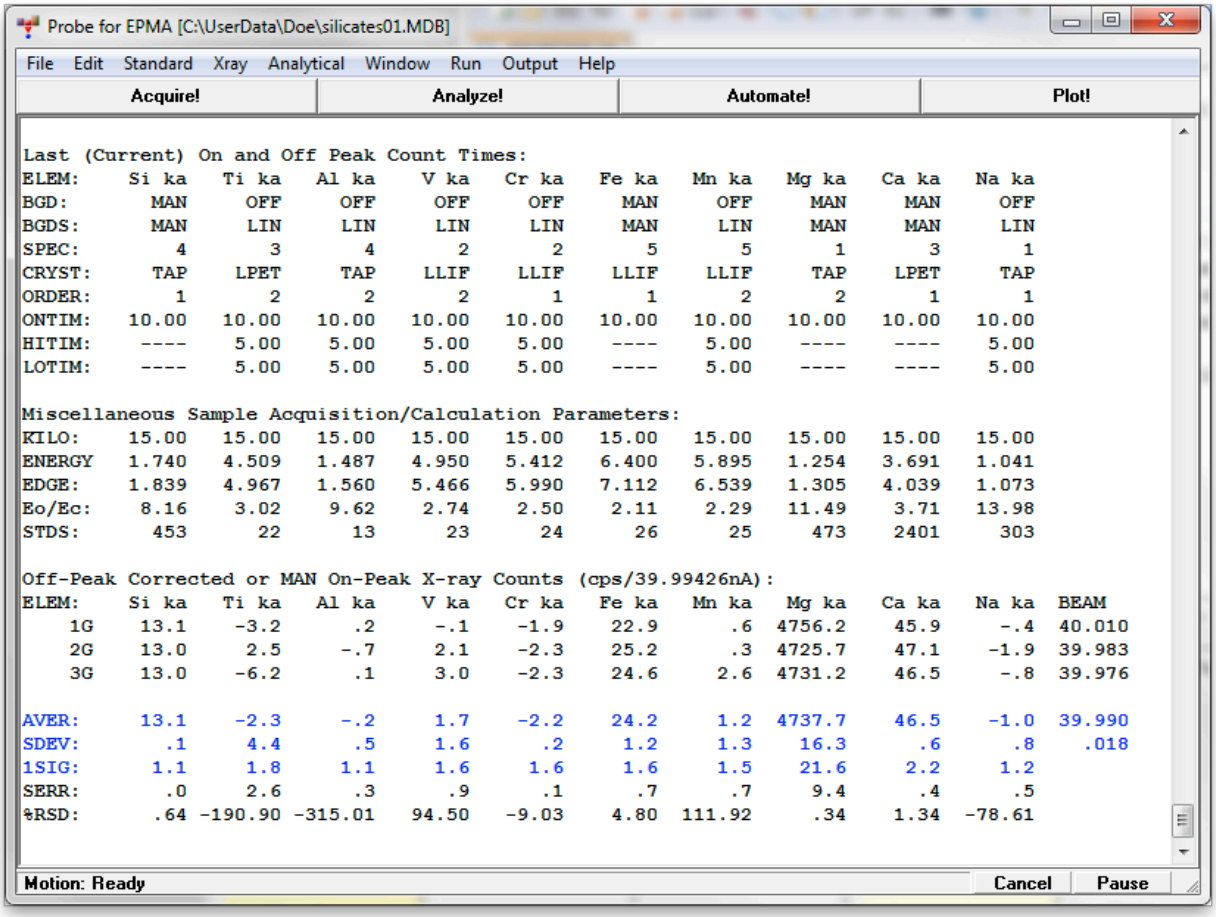
The progress of all data acquisition may be viewed in the **Acquire!** window. The current sample is displayed in the **Acquire!** window and the spectrometers move to their respective peak positions for the first elements and count on peak and off peak for times specified earlier in the **Count Times** window. Off peak measurements are not performed for Si, Fe, Mg, and Ca as the MAN background type was selected earlier. The Faraday cup is also measured.



After completion of acquisition, the results are written to the log window.



Repeated clicking of the **Start Standard or Unknown Acquisition** button acquires additional intensity data. The following log window illustrates the acquisition of three data points on the MgO standard.



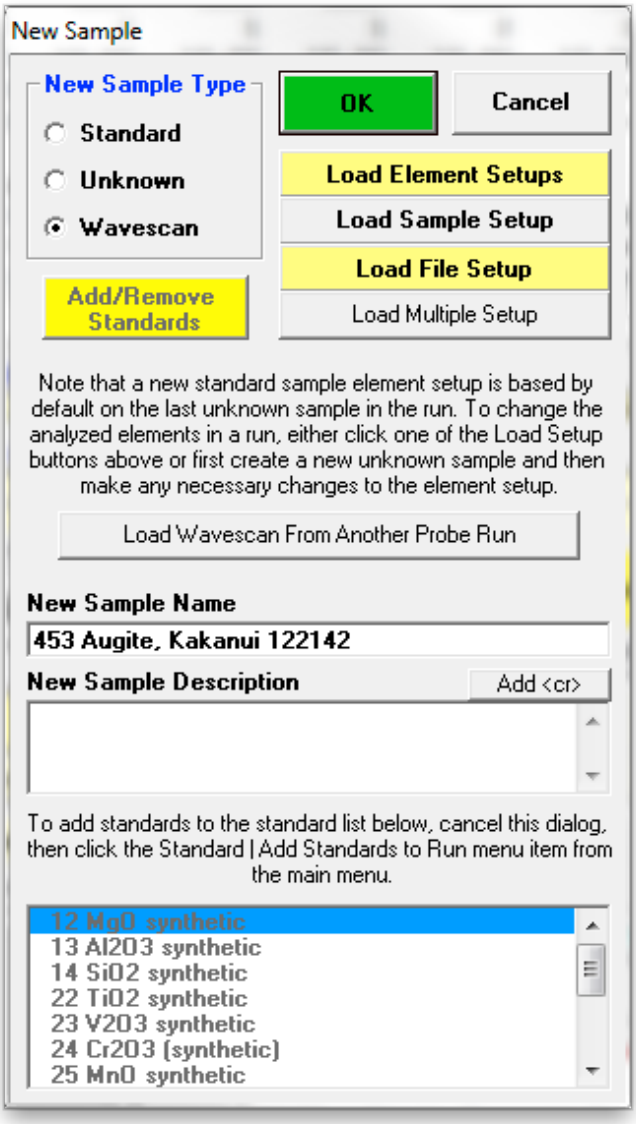
Similarly, x-ray counts can be acquired on the other standards. Move to the next standard position via the **Move** button and inspect the location and focus. Click the **New Sample** button, select the next standard from the standard list, and click the **OK** button when done.

Wavescan Acquisitions and Off-Peak Adjustments

Wavescans can be performed for example to check for spectral interferences, presence of minor elements, or to check and adjust off-peak positions.

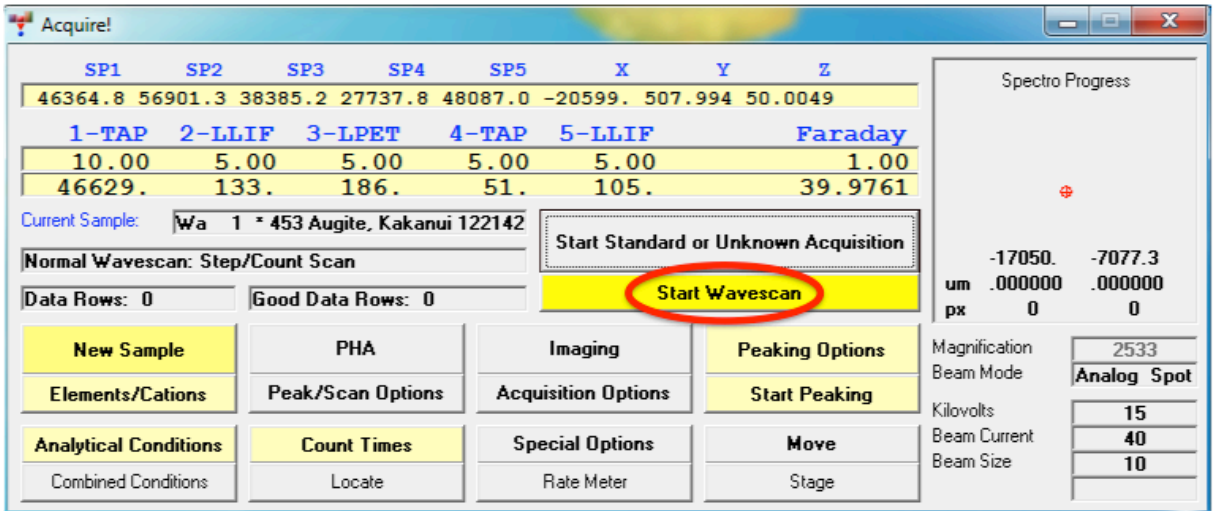
Move to standard 453 (Augite, Kakanui USNM 122142), which is a pyroxene expected to be similar in composition to the unknowns, using the **Move Motors and Change Crystals!** or the **Automate!** window as explained before.

Click the **New Sample** button. Select *Wavescan* under *New Sample Type*, edit the *New Sample Name* and *New Sample Description* text boxes, as desired.



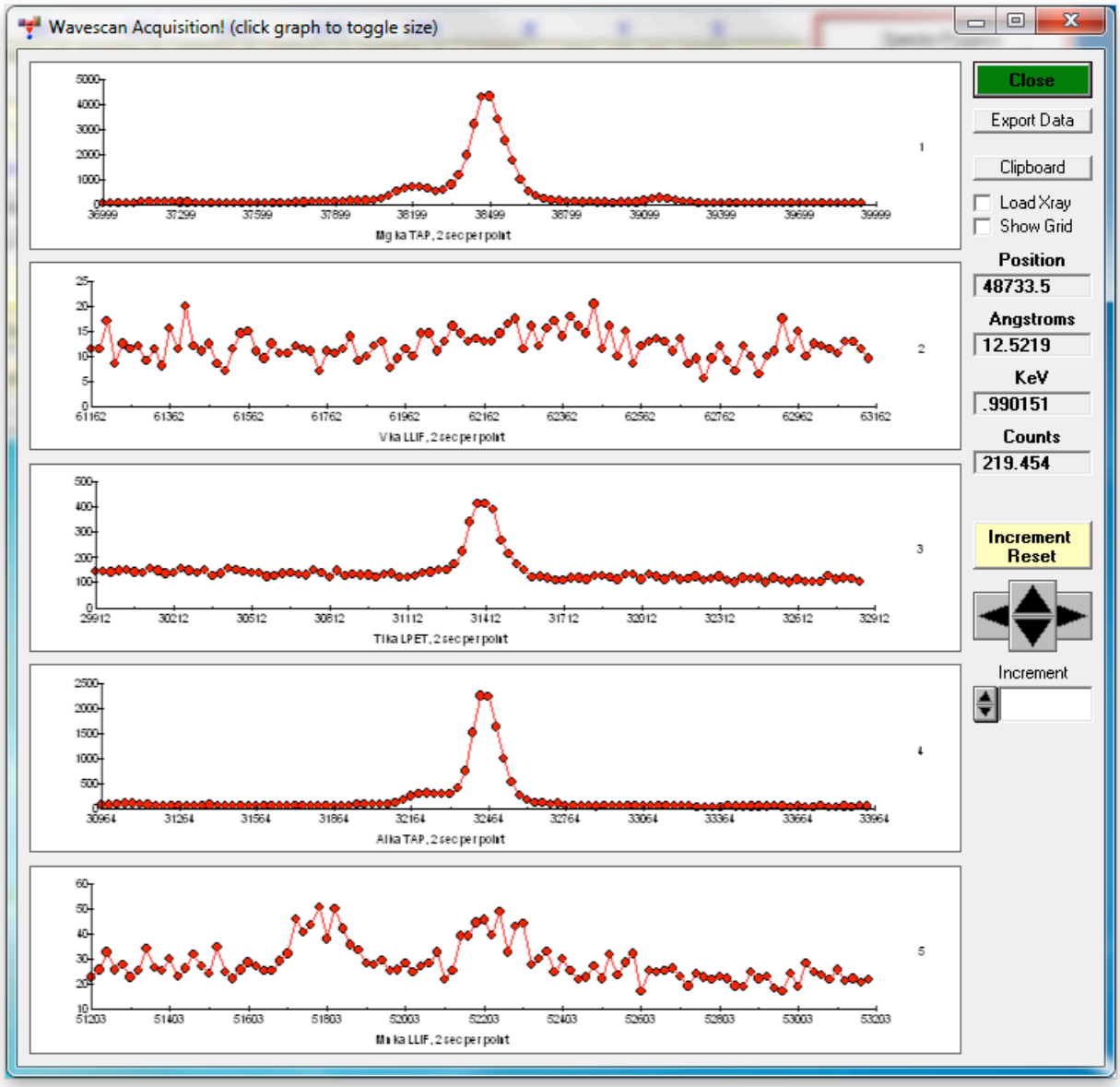
Click **OK** when done.

Click the **Start Wavescan** button of the **Acquire!** window.

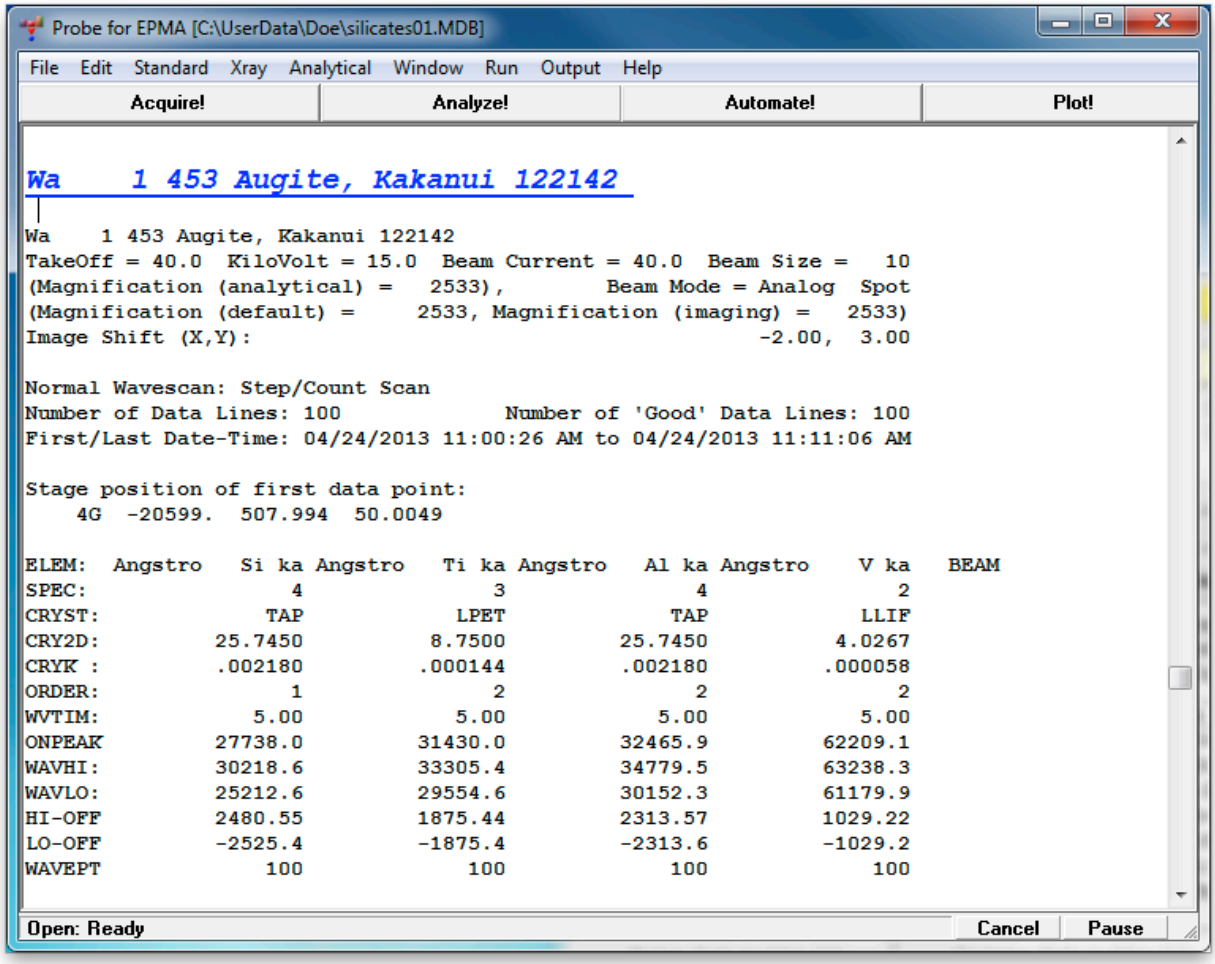


This action opens the **Wavescan Acquisition** window and automatically initiates a 100 step (user defined) wavelength scan for all of the elements entered into the current sample.

Graphical output of the completed scan via the **Wavescan Acquisition** window can be seen below for the second set of elements on the respective spectrometers, Mg, V, Ti, Al, and Mn. The Augite Kakanui standards contains Mg and Al as major elements, Ti and Mn as minor elements, and may contain a trace amount of V. Additional peaks are present which will be discussed later.

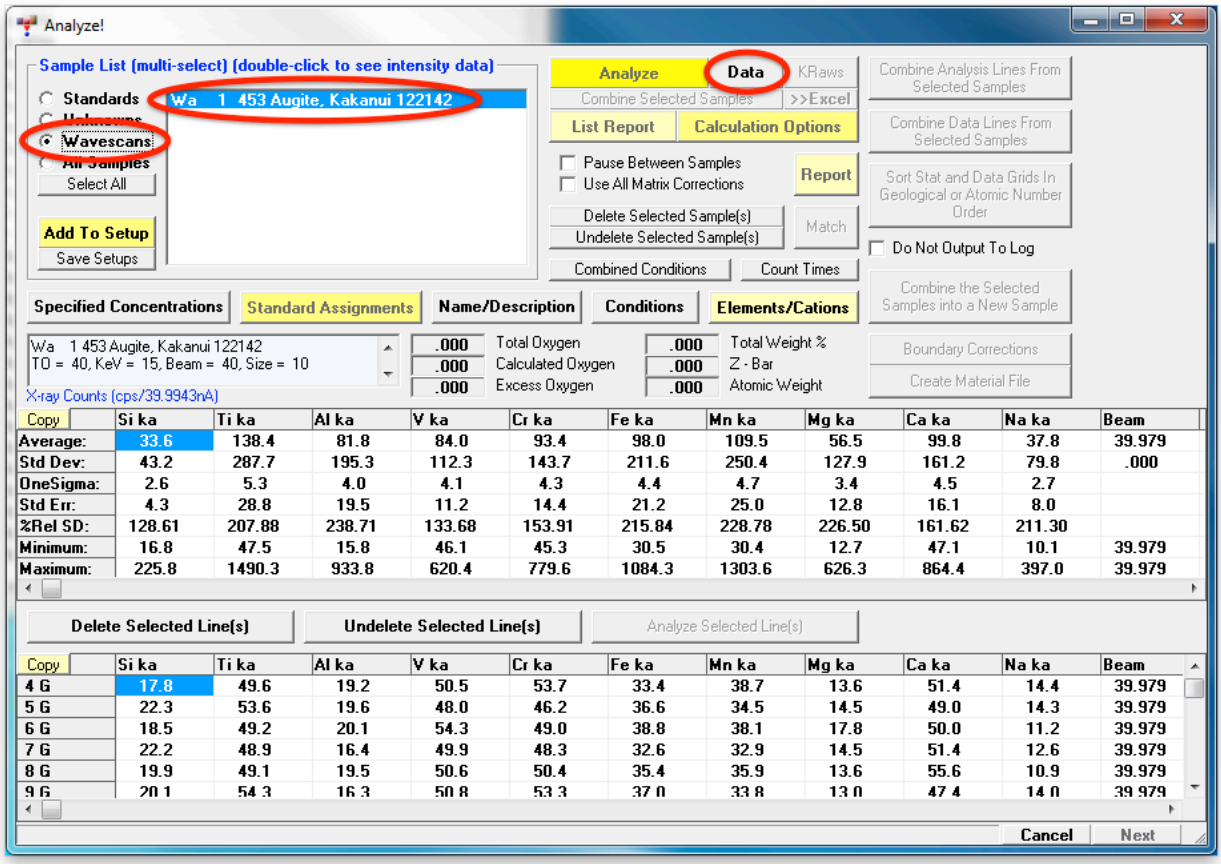


The wavescan labels appear in the main PROBE FOR EPMA log window.

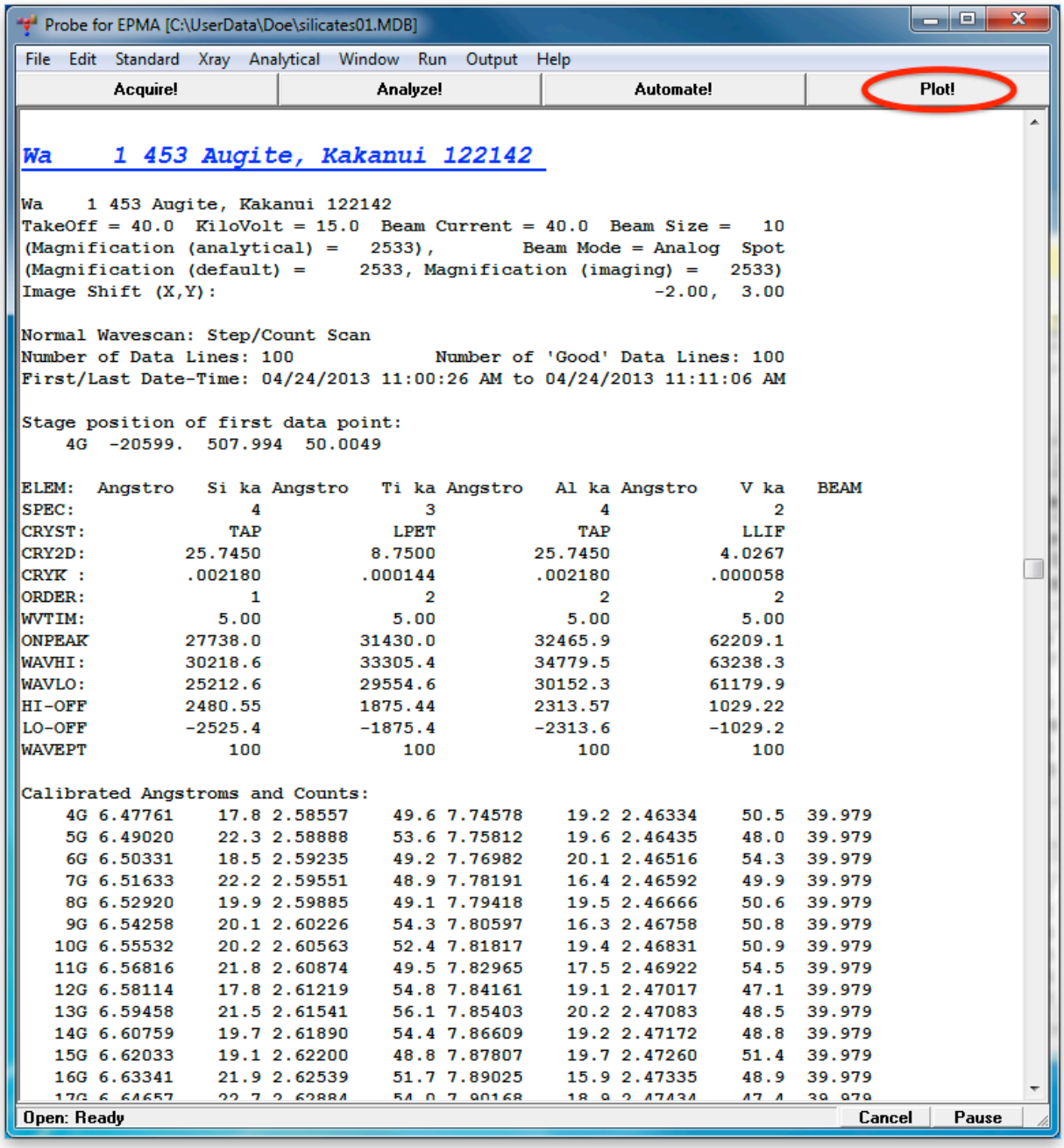


The wavescan positions and counts may be displayed in the main log window by clicking the **Analyze!** button opening the **Analyze!** window.

Select the *Wavescans* radio button, highlight the *Wa 1 453 Augite, Kakanui 122142* sample and finally click the **Data** button to write the data to the main log window.

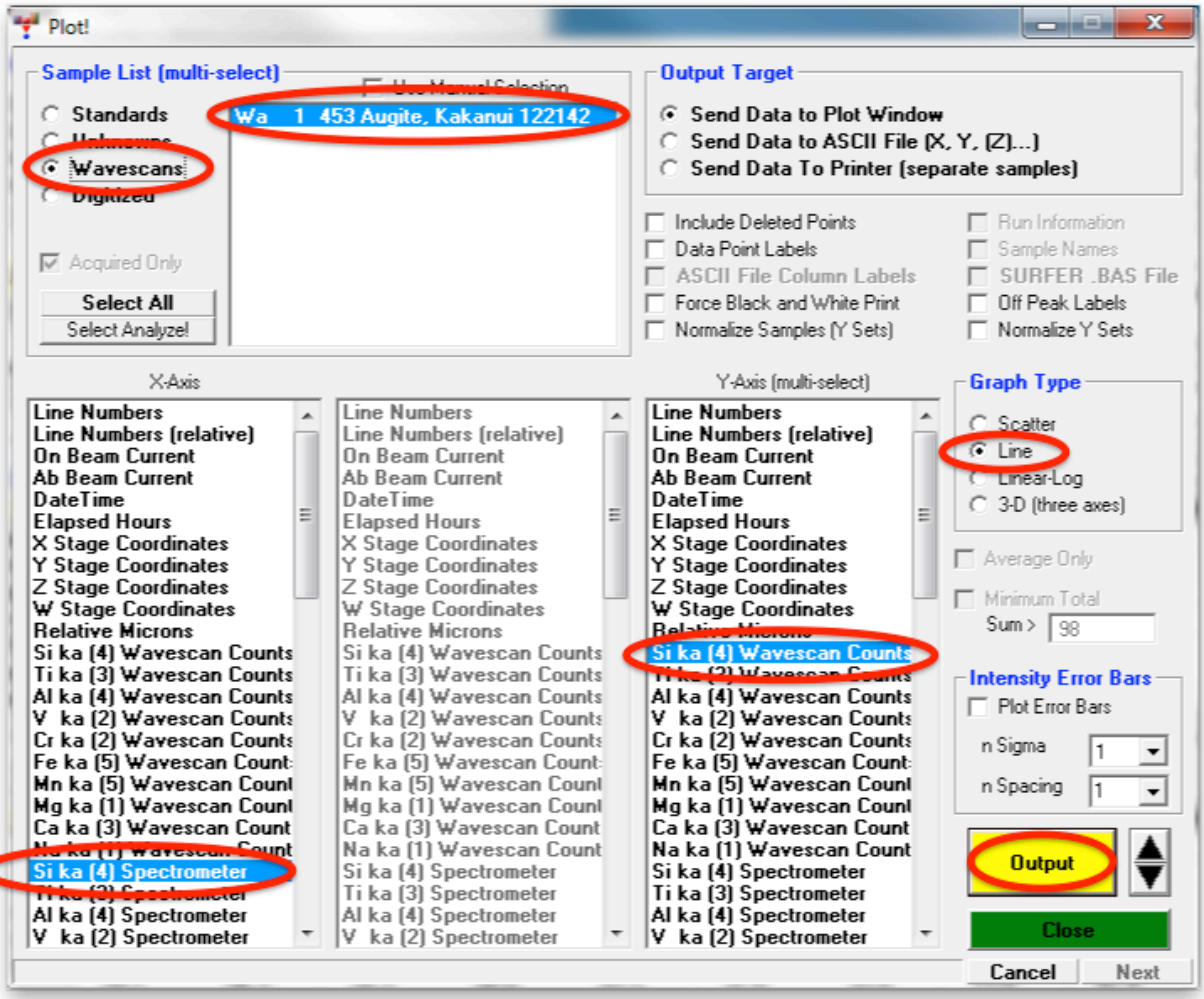


The output of the first lines of the wavescan data is shown below. Graphical display of these wavescans may be accomplished using the **Plot!** window, where they can also be used to check and adjust off-peak background positions. Click the **Plot!** button

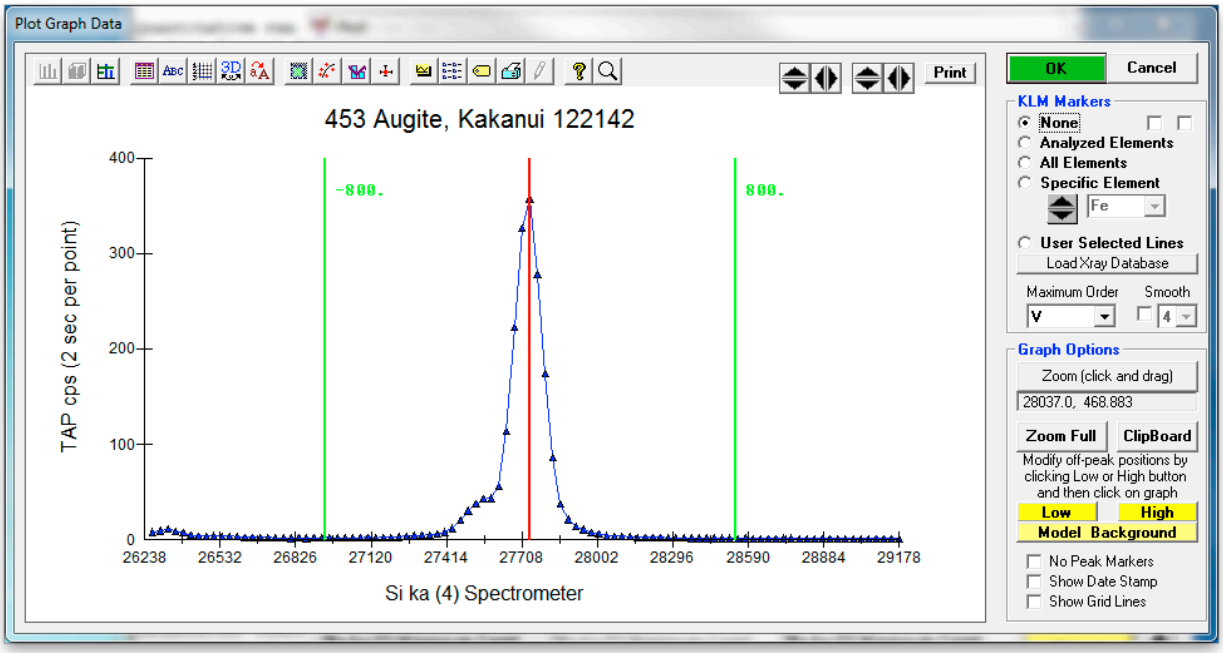


The **Plot!** window opens. Click the *Wavescans* radio button under *Sample List*. Click to highlight the *Wa 1 453 Augite, Kakanui 122142* wavescan sample.

Click on *Si ka (4) Spectrometer* from the *X-Axis* list and *Si ka (4) Wavescan Counts* from the *Y-Axis* list selections. The number following the element label (4, in this case) designates which spectrometer collected the data. The same element may be run on multiple spectrometers (see User's Guide and Reference documentation for additional details). Choose a *Graph Type*, click the *Line* button and an *Output Target* of *Send Data to Plot Window*. Finally, click the **Output** button to view the graph.



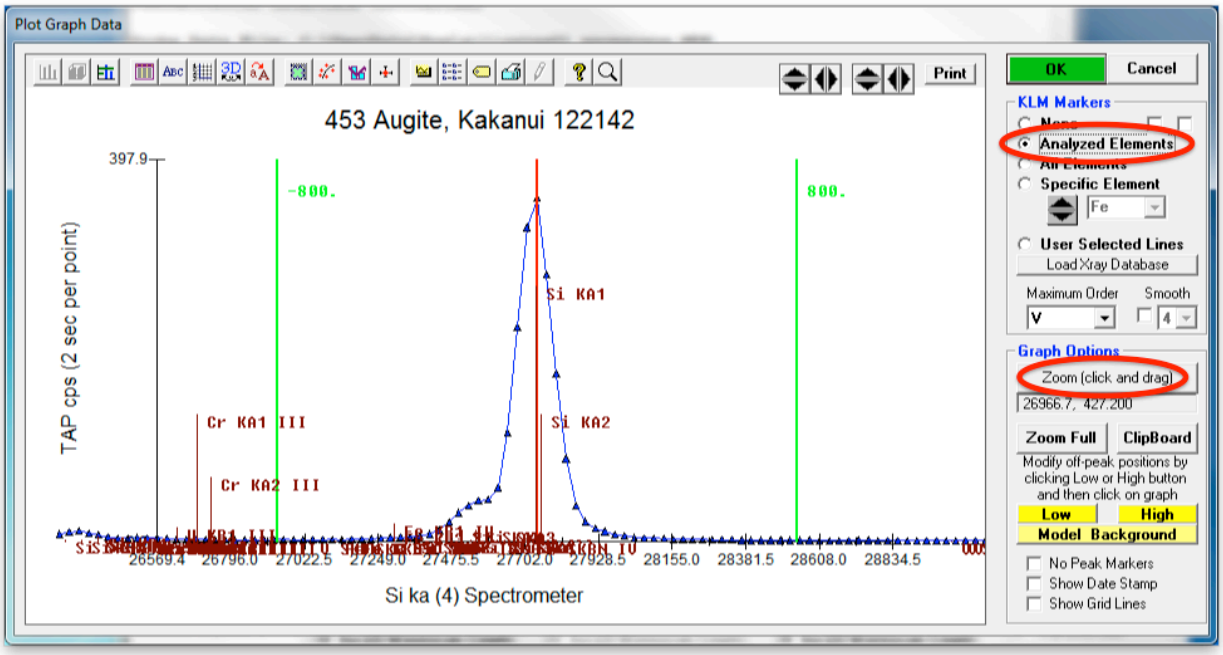
The program then loads the selected data into the **Plot Graph Data** window.



The **Plot Graph Data** module allows a more robust treatment of the wavescan data. The graph of *Si ka (4) Spectrometer* position versus *Si (4) Wavescan Counts* (labeled as *TAP cps*) is plotted as well as the locations of the on-peak (red vertical line) and both off-peaks (green vertical lines).

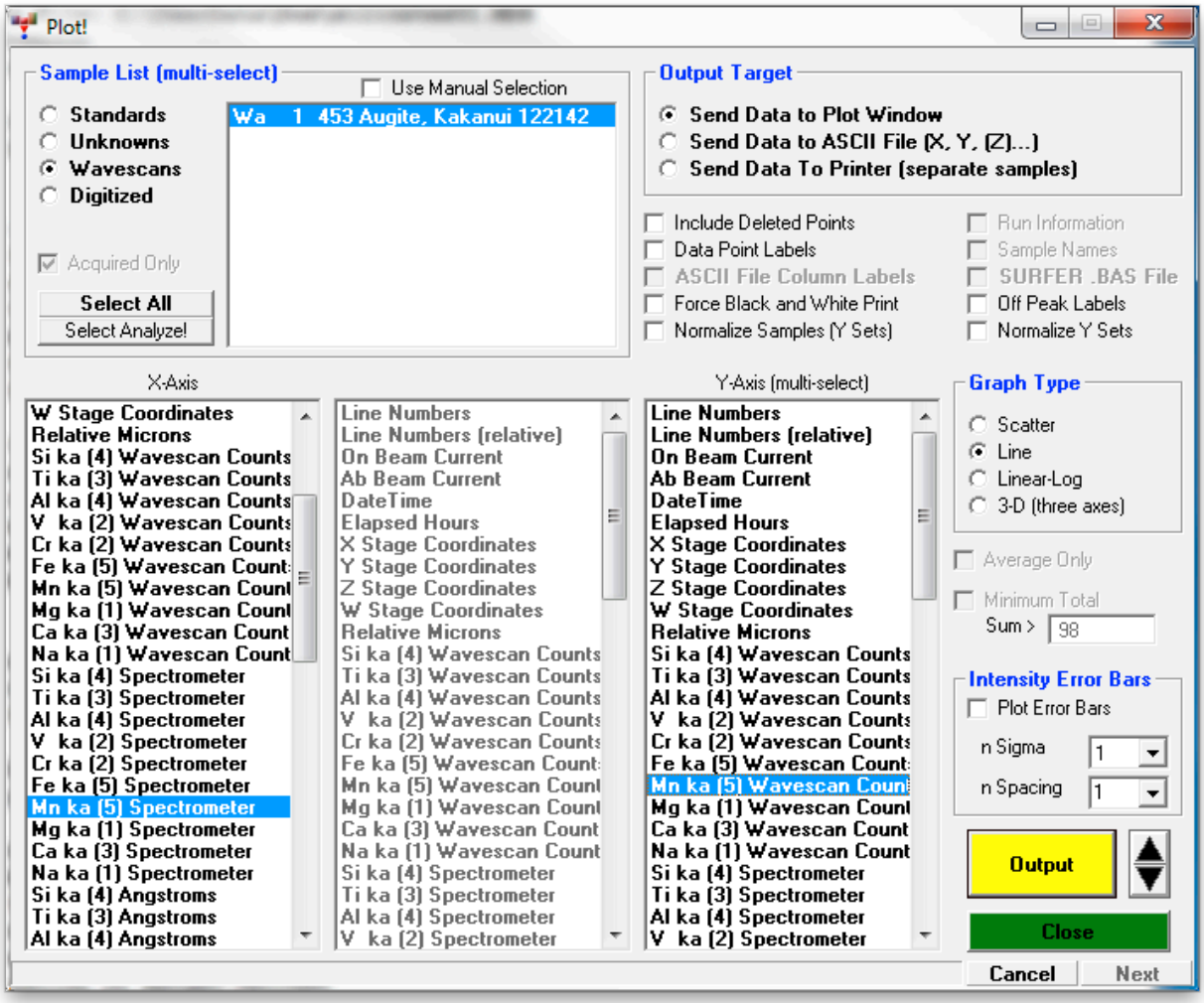
Various options are available for evaluation of the data. Besides click and drag *Zoom* capabilities, a large selection of *KLM Markers* options may be enabled to plot theoretical x-ray line positions. Further, a model background option is available (see User's Guide and Reference documentation for a complete discussion of this feature).

With the **Zoom** button active, click and drag the mouse over the region the user wishes to magnify. The *Analyzed Elements* button of the *KLM Markers* may be selected, plotting the various x-ray line positions for all analyzed elements in the current spectrum region.

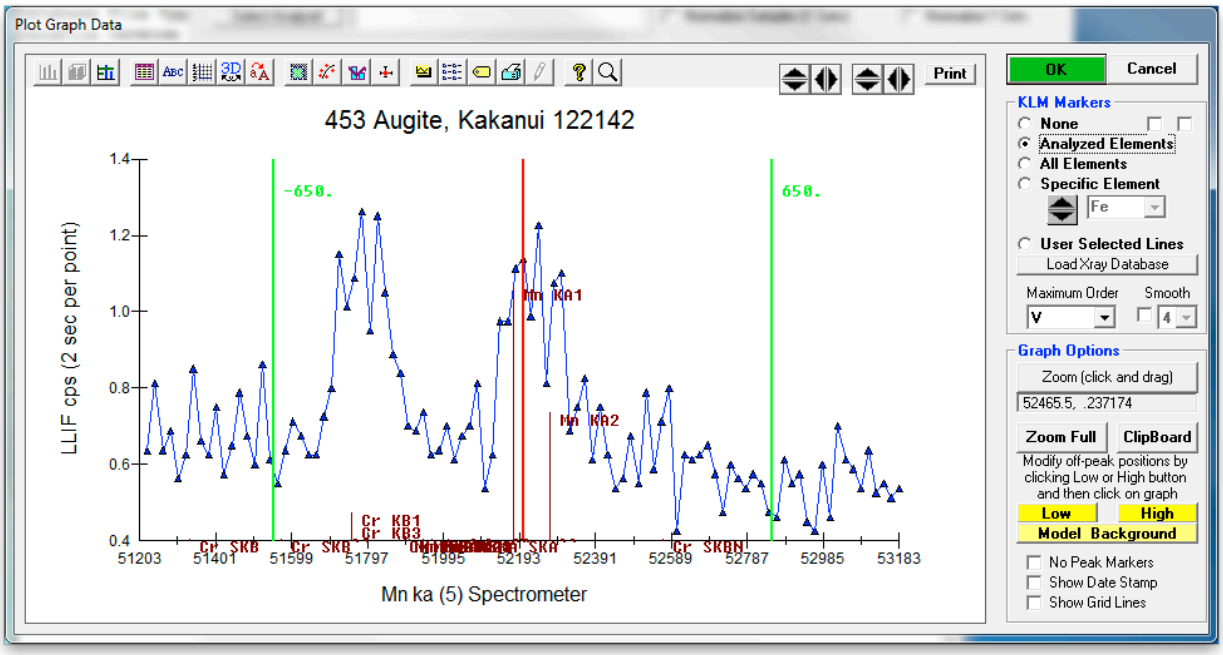


The default choices for both silicon background positions (green vertical lines) appear sound as no analyzed element lies nearby and the background counts near these peaks are low. Click the **OK** button of the **Plot Graph Data** window to return to the **Plot!** dialog box.

Next, the user evaluates the same data set for manganese. From the **Plot!** dialog box, select *Mn ka (5) Spectrometer* from the *X-Axis* list and *Mn ka (5) Wavescan Counts* from the *Y-Axis* list selections. Click the **Output** button.



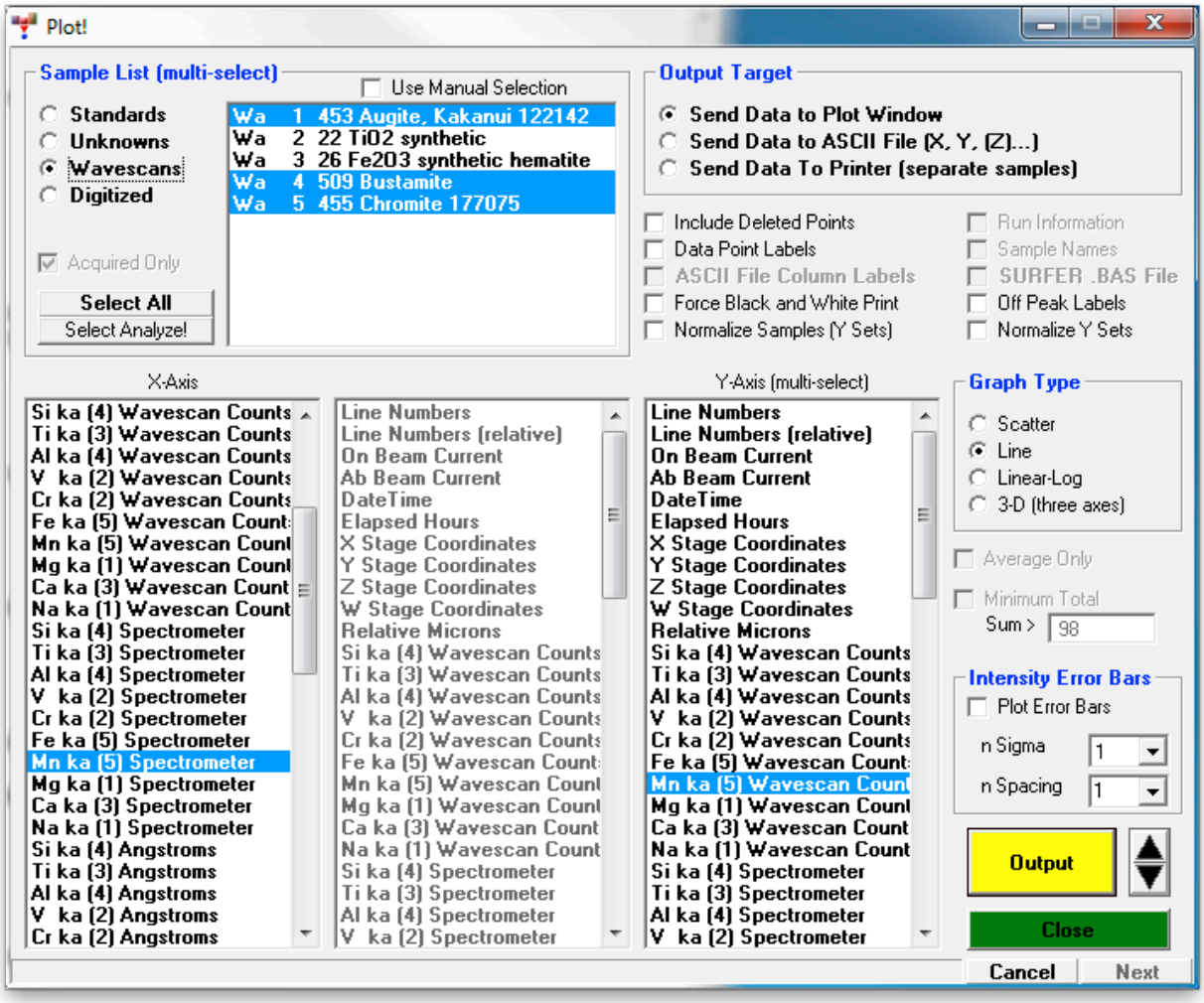
The **Plot Graph Data** window for the manganese data set is shown below. Two peaks are visible and the user observes that the low background position lies close to the second unknown peak, which is identified as Cr $K\beta_{1,2}$.



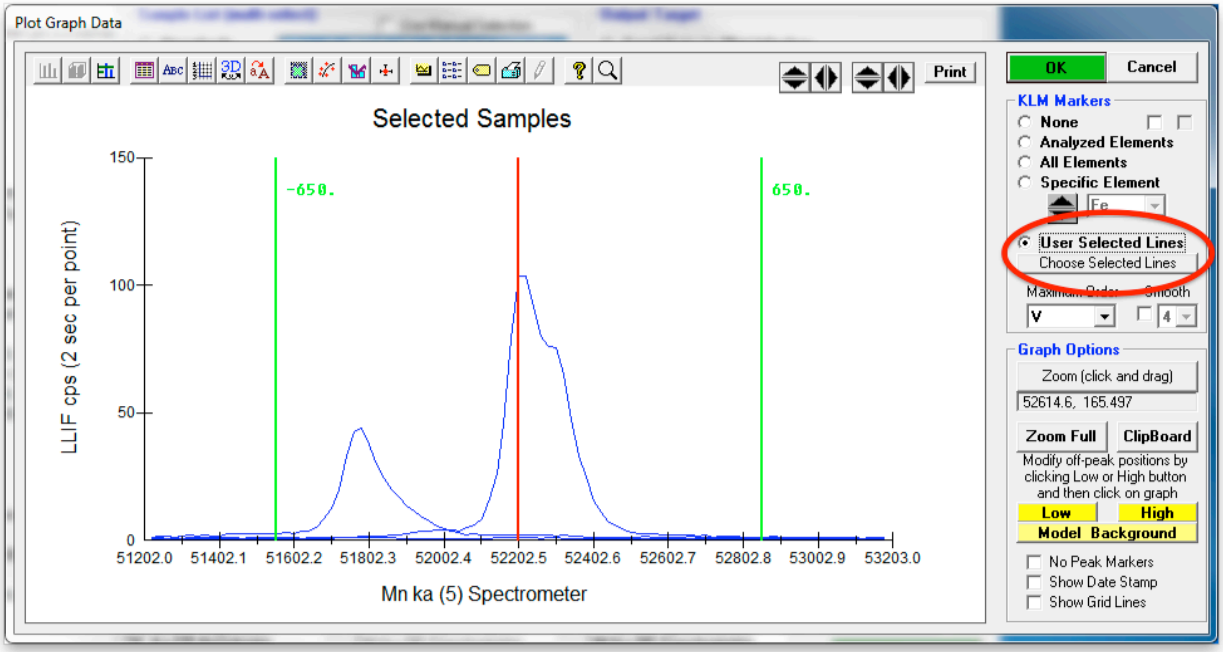
As both Mn and Cr are only present as minor elements in the Augite Kakanui standards, the count rates are low. To better assess the situation, additional wavescans need to be collected in the same way on standards high in chromium and manganese, respectively.

Click **OK** to return to the **Plot!** window.

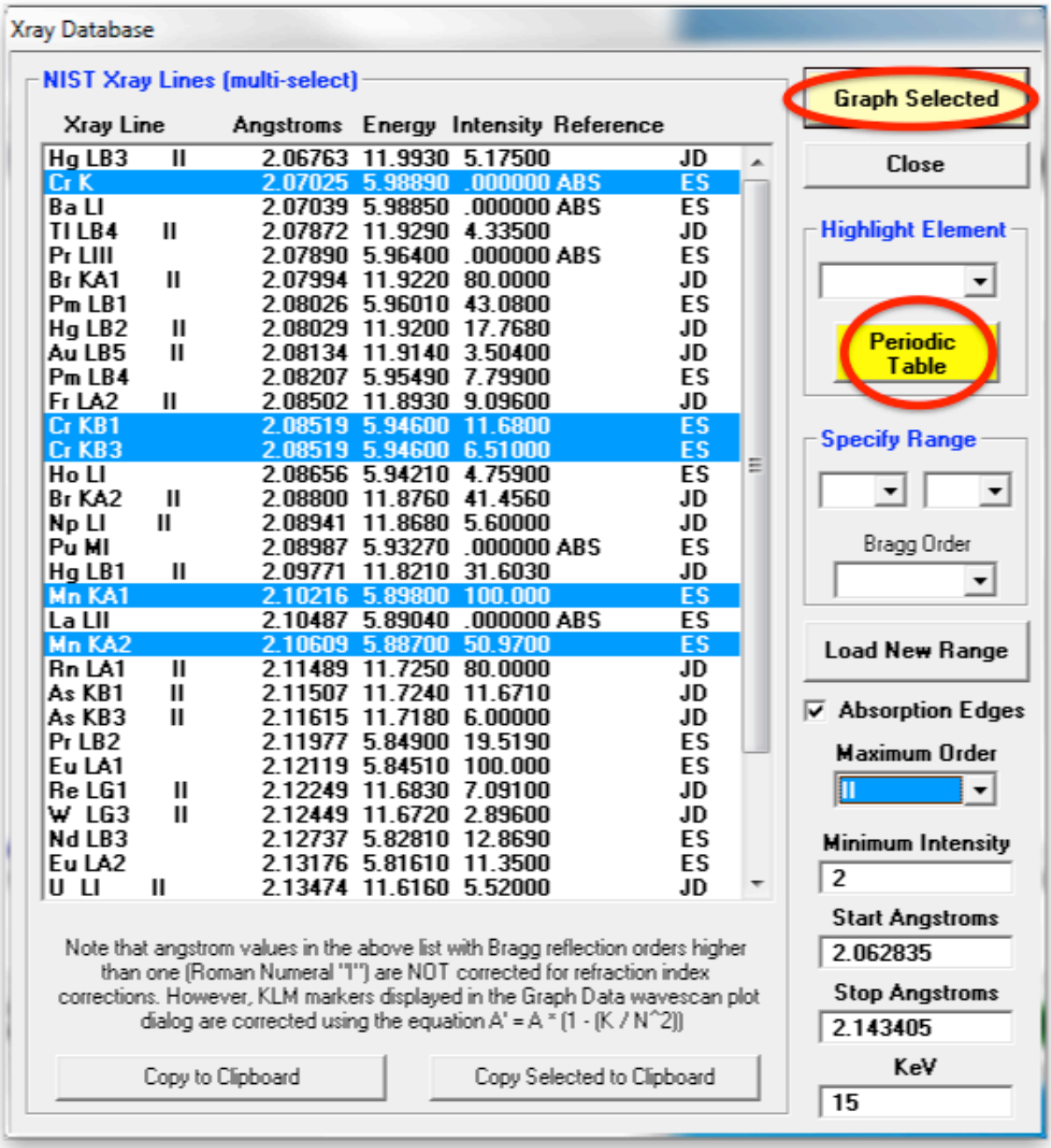
Additional wavescans are collected using the same procedure, which is not shown again to save space. The Plot! window is now shown below with additional wavescans acquired on standards, including Bustamite for Mn and Chromite for Cr. Hold the <ctrl> key and click on the Augite, Bustamite, and Chromite standards to multi-select all three. Again, select *Mn ka (5) Spectrometer* from the *X-Axis* list and *Mn ka (5) Wavescan Counts* from the *Y-Axis* list selections. Click the **Output** button.



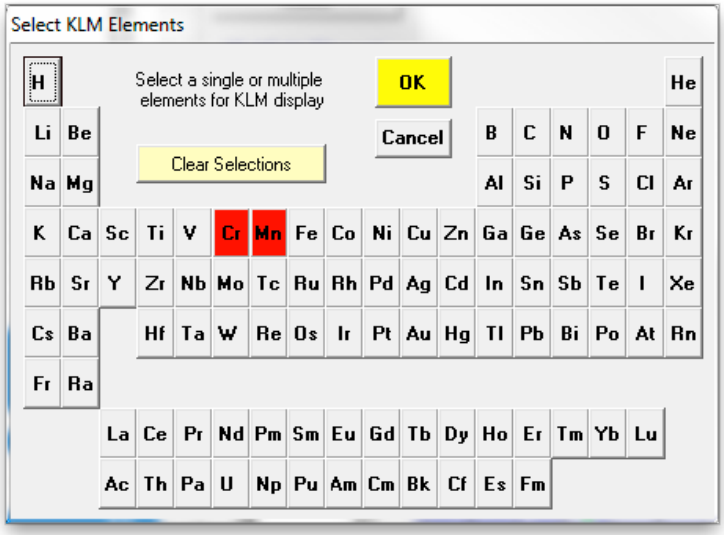
The **Plot Graph Data** window for the manganese data set is shown below. Due to the better counting statistics, the Mn and Cr peak shapes can now be much better assessed for off-peak positioning. To make a more detailed selection for the KLM Markers to be displayed, click the *User Selected Lines* radio button. Then click the **Choose Selected Lines** button below.



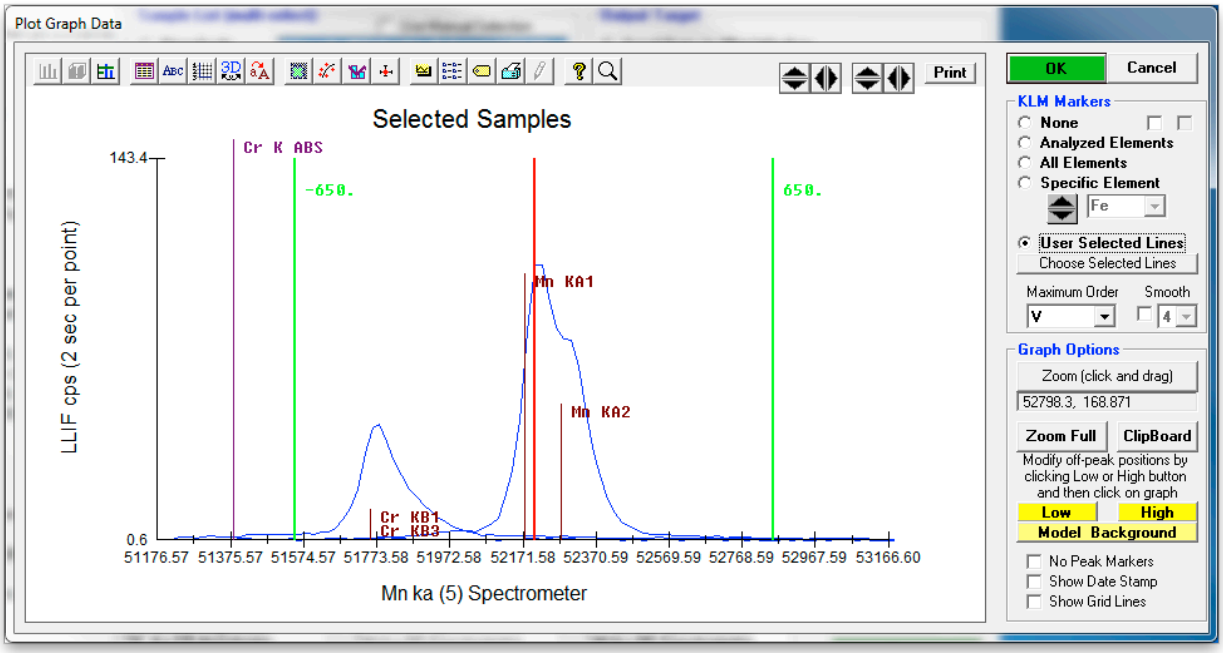
The **Xray Database** window opens containing the NIST x-ray line catalog. The user may select or multi-select any x-ray line to plot in the **Plot Graph Data** window simply by highlighting (a) line(s) and clicking the **Graph Selected** button. To select all lines for certain elements, click the **Periodic Table** button.



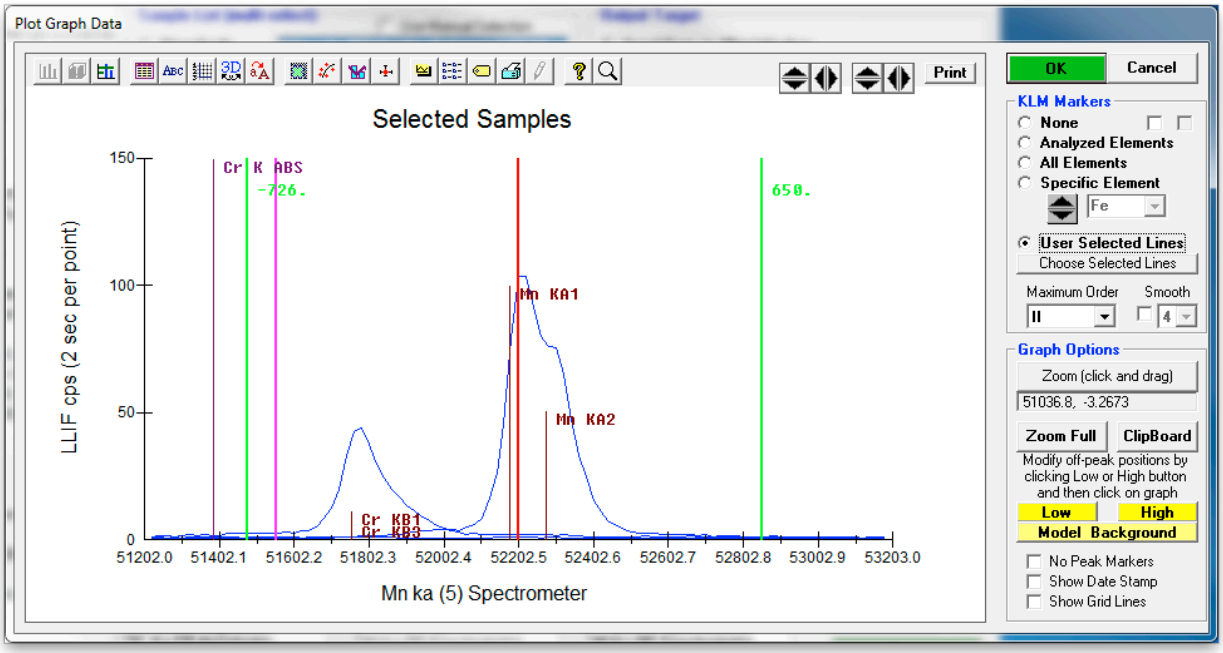
This opens the **Select KLM Elements** window. Click to highlight Cr and Mn.



Click **OK** to return to the **Xray Database** window when finished. Click the **Graph Selected button** again in the **Xray Database** window to plot the new selection, then click the **Close** button to return to the **Plot Graph Data** window.

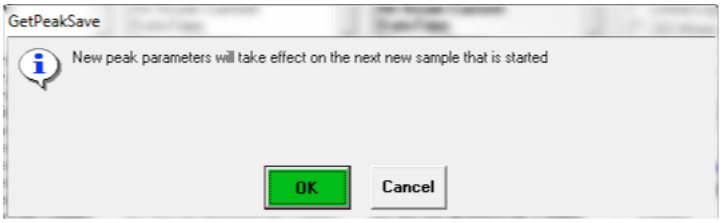


The low off-peak position for Mn appears to be slightly on the slope of the Cr Kβ peak. To adjust the low off-peak position further away from this peak, click the **Low** button, creating a crosshair on the graph window. Move the crosshair to a new low background position and click the mouse. The color of the initial off-peak position changes to pink and a new vertical green line appears, indicating the new off-peak position.



If high Cr samples are analyzed, the user should not move the off-peak position close or past the Cr K edge depicted by the purple *Cr K ABS* marker. It might become necessary to measure the Mn background using a high off-peak only with a slope factor (see User's Guide and Reference manual for details). The Cr Kβ peak also interferes slightly with the Mn Kα on-peak position, potentially requiring an interference correction, which will be discussed further below.

Click the **OK** button to update this background position in the run and close the **Graph Data** window. The **GetPeakSave** window appears and the user is notified that new parameters (off-peak position) will take effect on the next new sample.



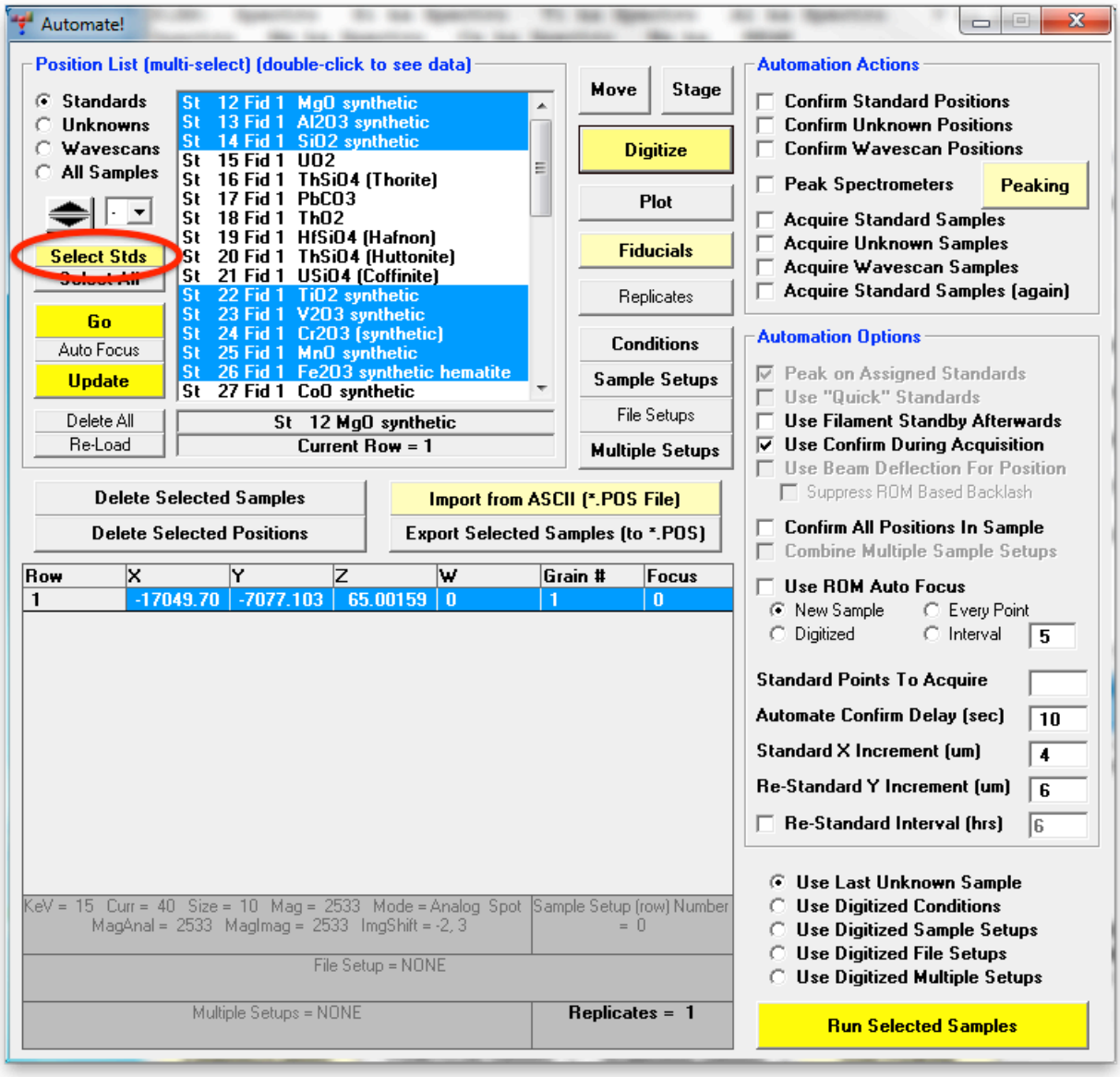
Click this **OK** button, returning to the **Plot!** window. Finally, click the **Close** button to exit the **Plot!** window returning to the main PROBE FOR EPMA log window.

Automation Actions

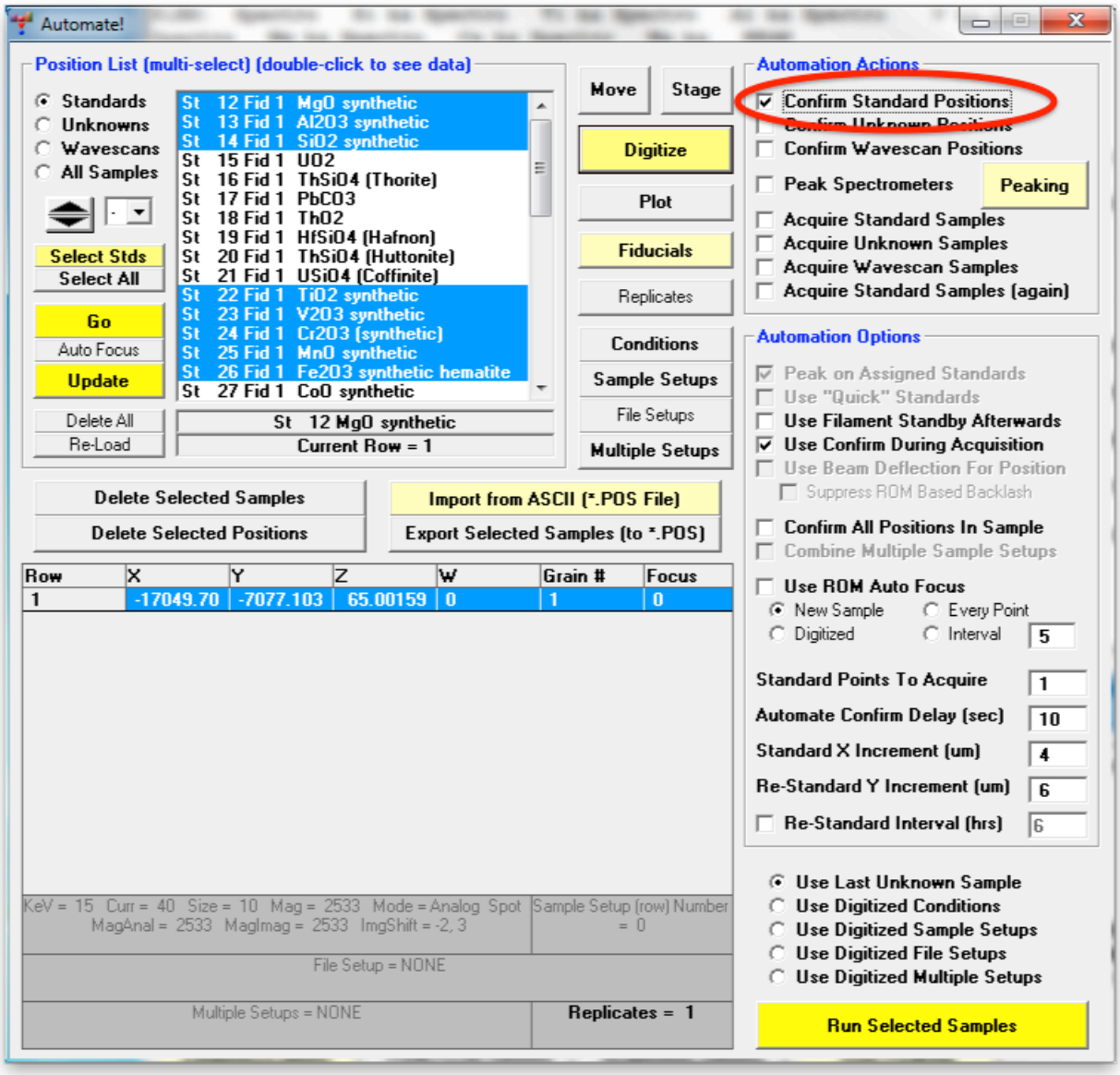
Confirm Standard Positions

All of the basic position confirmation, peak centering and x-ray count acquisition procedures may be automated. This is accomplished via the **Automate!** window.

Click the **Select Stds** button of the **Automate!** dialog box. All standards that have been added to the current run will be highlighted in the *Position List* list box.

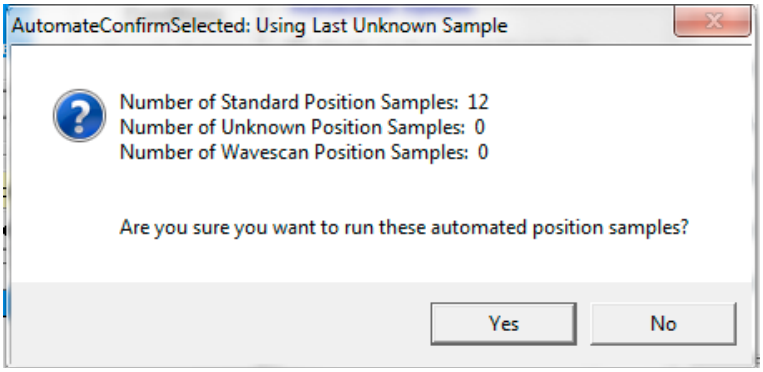


The user might start by checking the location and focus of each standard selected for the automated analysis. Click the box for *Confirm Standard Positions* under *Automation Actions*.



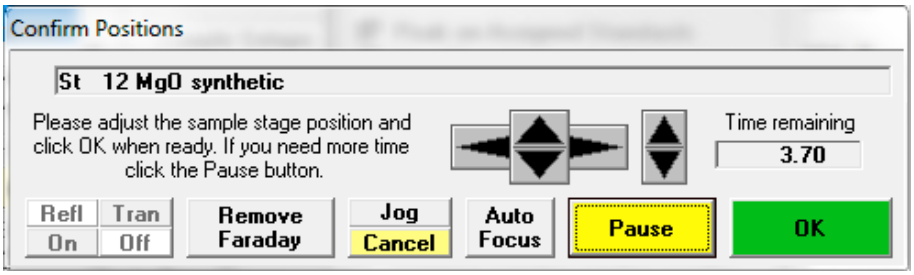
Click the **Run Selected Samples** button in the bottom right corner of the window.

The **AutomateConfirmSelected** window opens informing the user how many standards were chosen and asks if the user wants to run these automated samples. Click **Yes**.

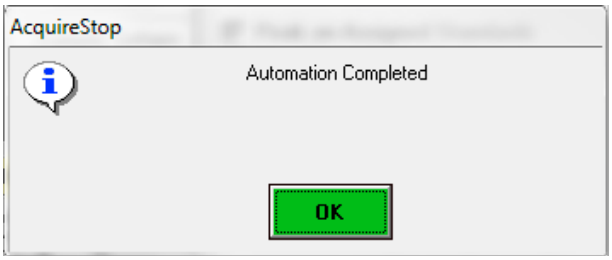


The program then sends the stage motors to the fiducial transformed coordinates for the first selected standard and opens the **Confirm Positions** window. Clicking the two-way **Pause/Continue** button suspends the 10 second countdown (user defined in the PROBEWIN.INI file). Adjust the stage motors (X , Y, and Z) to a new, clean analysis position. Click the **OK** button of the **Confirm Positions** window when done, sending the stage to the next standard to confirm its position. Again, the **Confirm Positions** window opens, allowing the user to pause the countdown and adjust the sample position.

If more than one position is digitized, the software moves to the first position and updates all positions for that sample by the same X, Y, and Z offset.



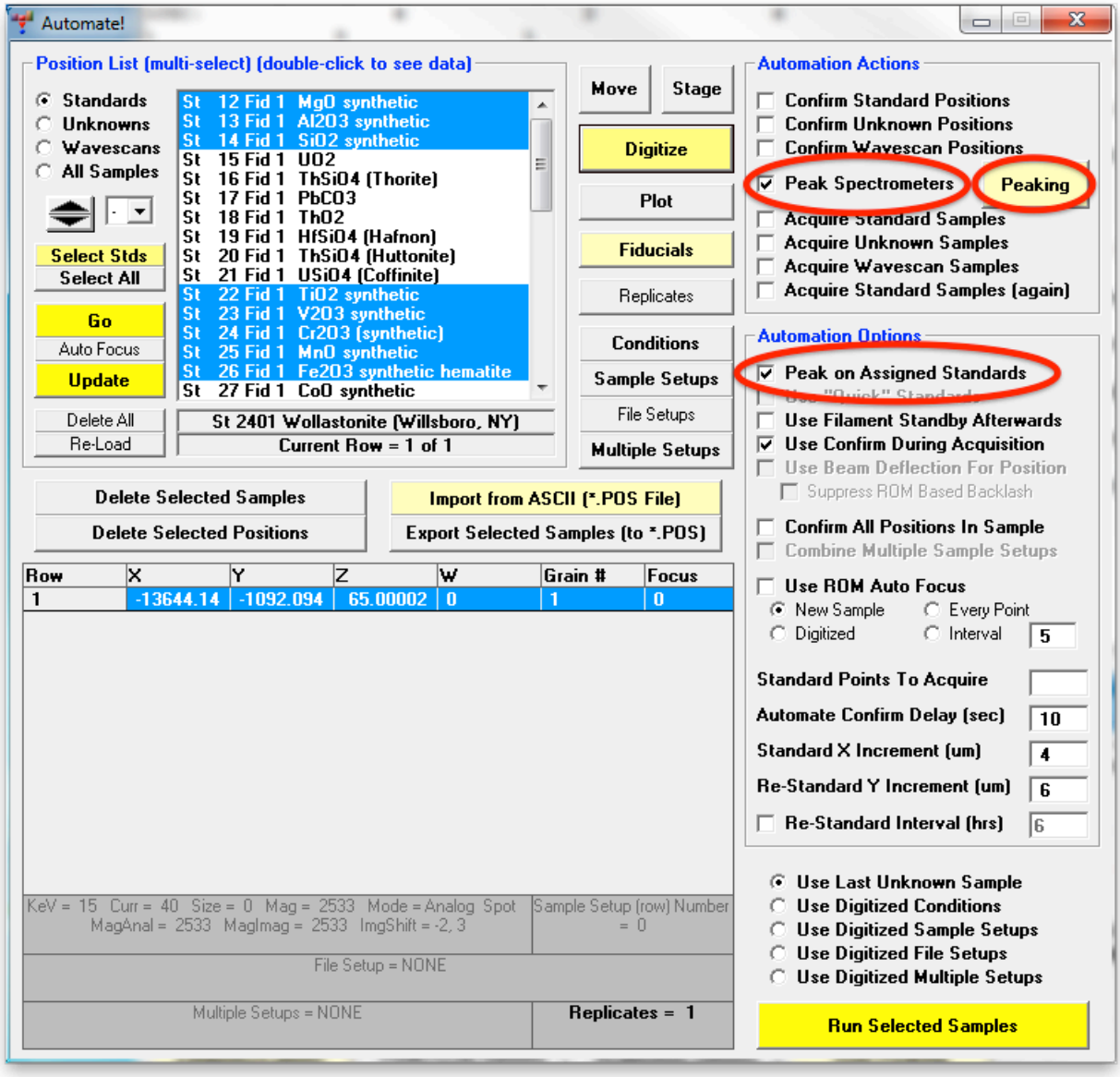
After the final standard is confirmed, the **AcquireStop** window appears. Click this **OK** button to return to the **Automate!** dialog box.



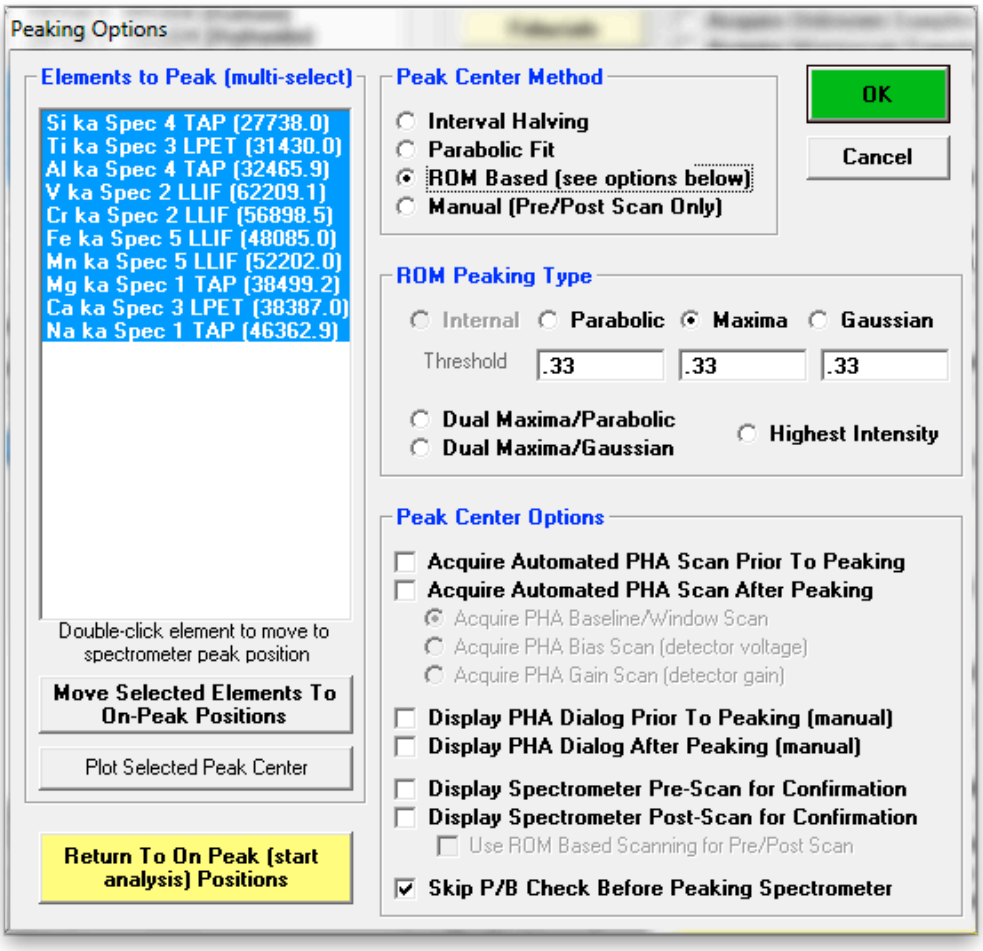
Calibrate Peak Positions

X-ray peaking may be automated from the **Automate!** window as follows. Under *Automation Actions* click only the *Peak Spectrometers* box. Under *Automation Options* click the *Peak on Assigned Standards* box. This option causes the program to attempt a peak center on a standard position sample if the standard is assigned as the primary standard for that element. If the element has no assigned standard, then the program will attempt to assign one automatically based on the highest concentration of the elements present among the standards in the run.

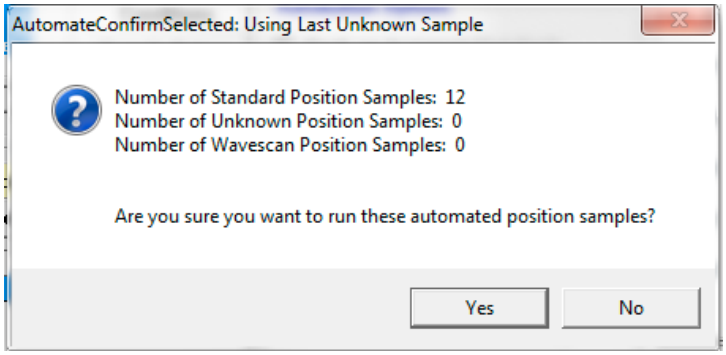
Next, click the **Peaking** button to open the **Peaking Options** dialog box.



In the **Peaking Options** dialog box, highlight (select) all of the elements in the *Elements to Peak* list box, and click on a *Peak Center Method*. A spectrometer pre-scan is useful if that element has not been run recently or if maintenance has occurred on the spectrometer.



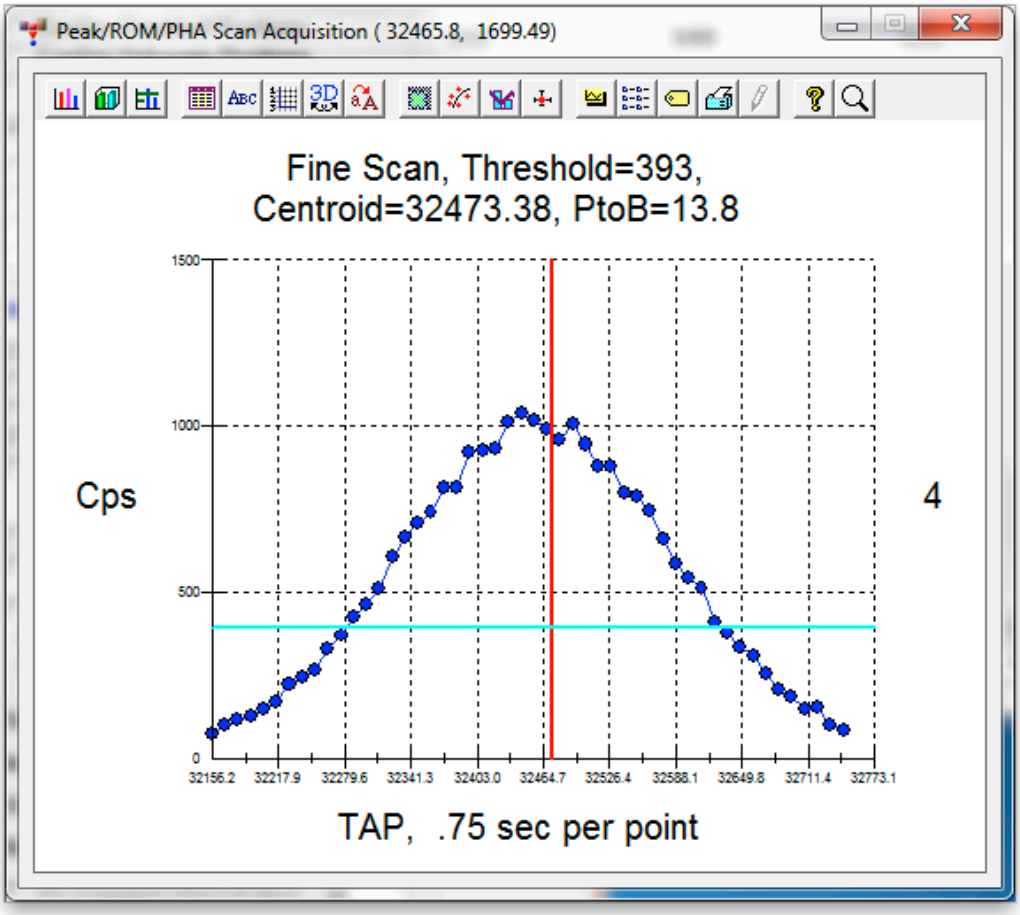
Click the **OK** button of the **Peaking Options** window. Click the **Run Selected Samples** button from the **Automate!** window. This opens the **AutomateConfirmSelected** window. To run these automated samples, click **Yes**.



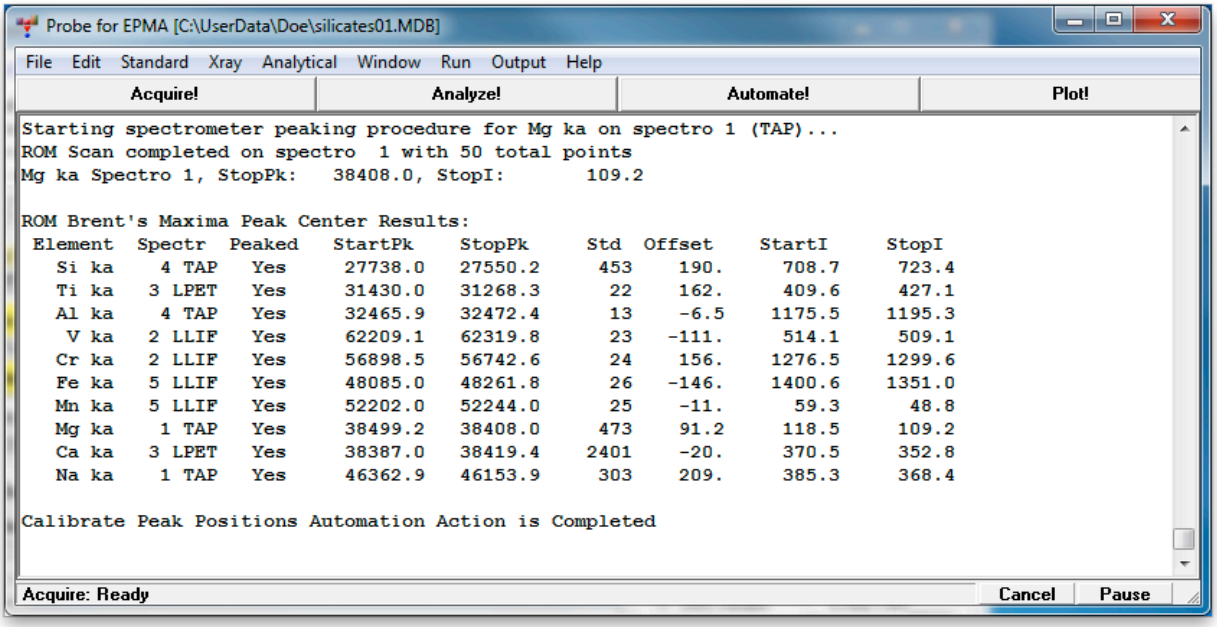
The steps of the automated procedure are similar to the manual peaking procedure described in the section "Manual Peaking and PHA using the Acquire! Window".

The stage motors move to the position coordinates of the first standard in the *Position List* list box. If the *Use Confirm During Acquisition* box under *Automation Options* is checked then the computer automation will pause at each standard (**Confirm Positions** window will open) for some user defined amount of time (usually 10 seconds) to allow the operator to adjust the stage position and focus. The **Peak/ROM/PHA Scan Acquisition** window opens and the spectrometers go through the peaking routine to peak center the spectrometer position to the intensity maximum for all the elements assigned to that standard. After finding a new peak position and reporting the results to the graph window and the main log window, the stage motors move on to the coordinates of the next standard highlighted in the *Position List* list box. Once situated on this standard, the spectrometers peak center those elements assigned to it. This procedure continues until all standards are done. When all automation action is complete, the **AcquireStop** window appears and requests the user to click the **OK** button.

An example for the graph displayed in the **Peak/ROM/PHA Scan Acquisition** window for Al Ka on TAP for Al2O3, using the ROM based peak center method and maxima fitting, is given below.



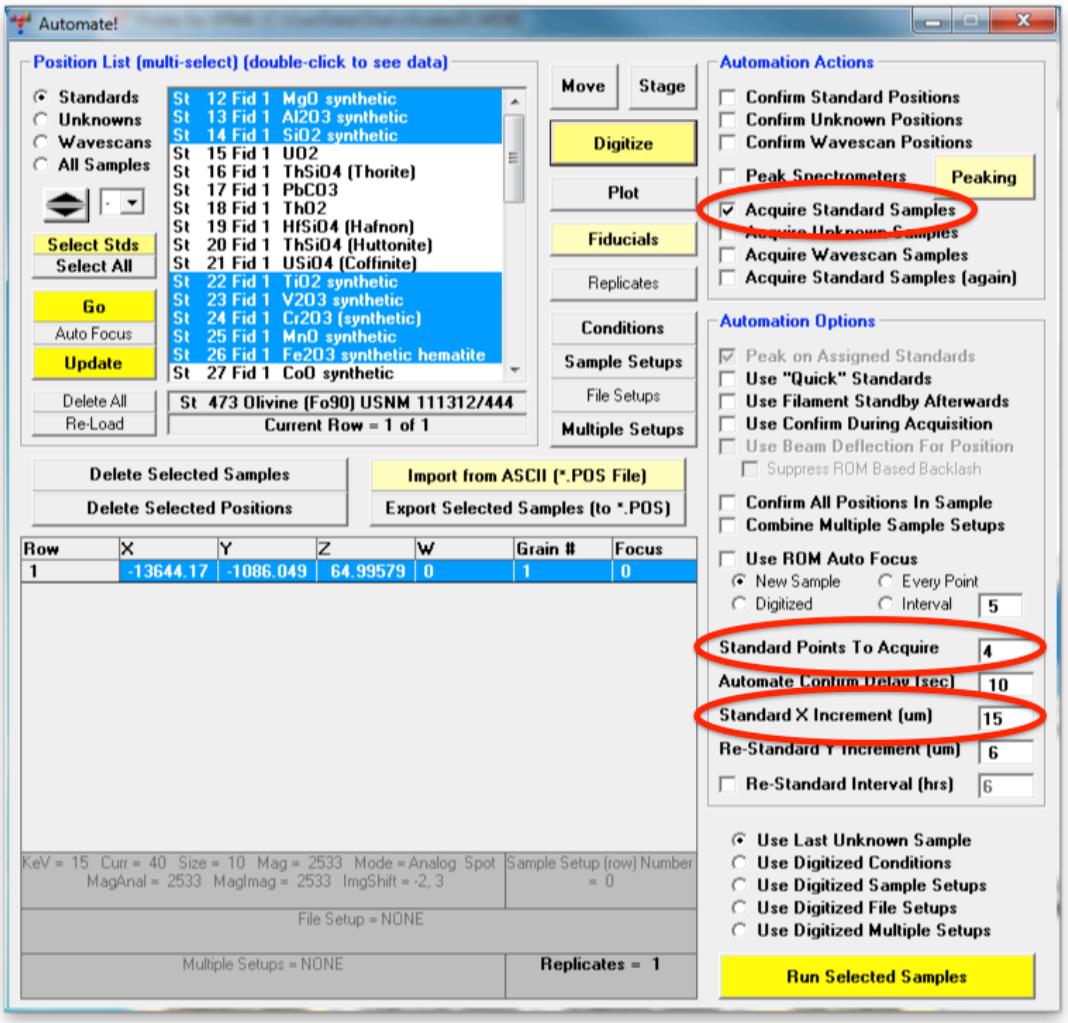
The following summary of the peak automation for two standards is found in the main log window.



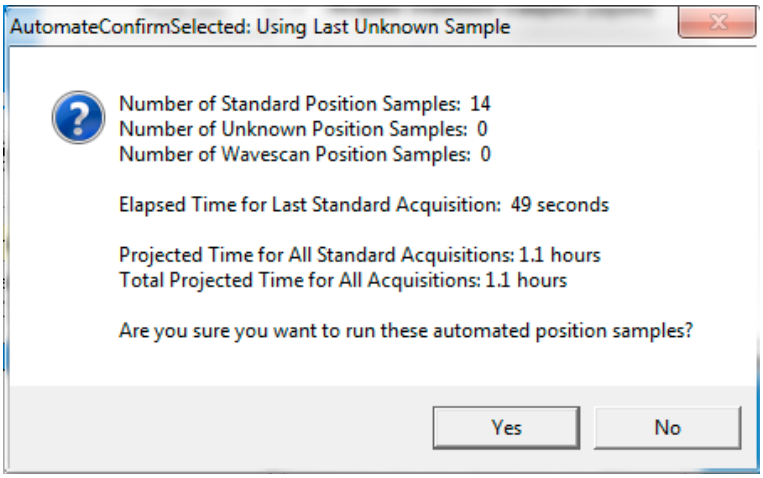
Acquire Standard Samples

The next step is to automate the acquisition of x-ray counts on all primary and MAN background standards as calibration for the unknown samples. Again, click the **Select Stds** button in the **Automate!** dialog box to select all current standards in the run, highlighting them in the *Position List* list box. Next, under *Automation Actions*, click only on the *Acquire Standard Samples* box. From the *Automation Options* choices select the number of *Standard Points To Acquire* and whether to *Use Confirm During Acquisition.*

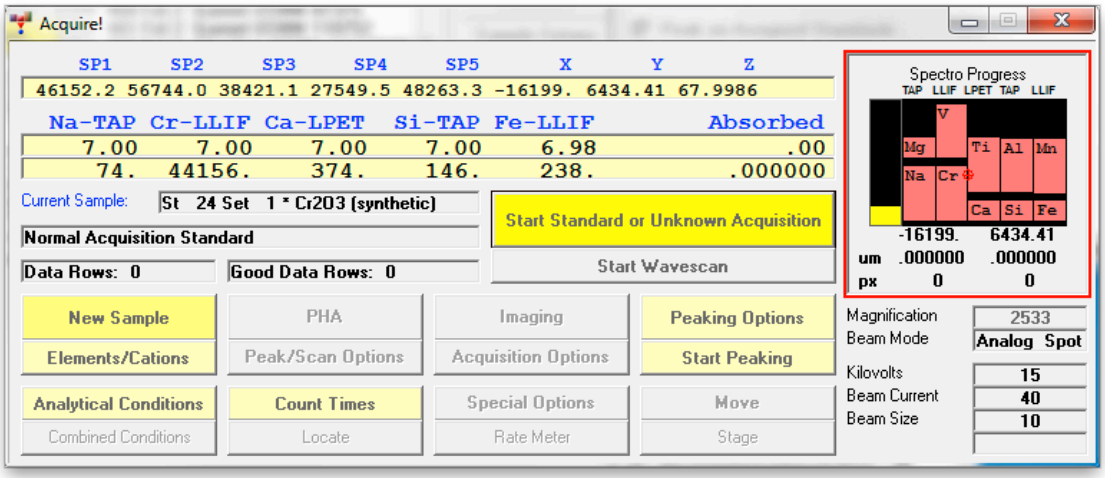
In this example, only one XYZ(W) position was digitized for each standard, but four standard points are chosen along with a *Standard X Increment* of 15 µm. This means that the first measurement will be performed in the digitized position, and for each consecutive measurement the X position will be shifted by +15 µm. This value should be sufficiently larger than the beam diameter (here 10 µm) to prevent overlapping measurements. The user has to ensure that sufficient space is available in the X direction on the standard grains for this operation. Alternatively, additional measurement positions could be digitized for each standard.



Finally, click the **Run Selected Samples** button. The **AutomateConfirmSelected** window opens again, stating how many standards are chosen and how long the run is expected to take and asking if these automated position samples should be run. Click **Yes**.

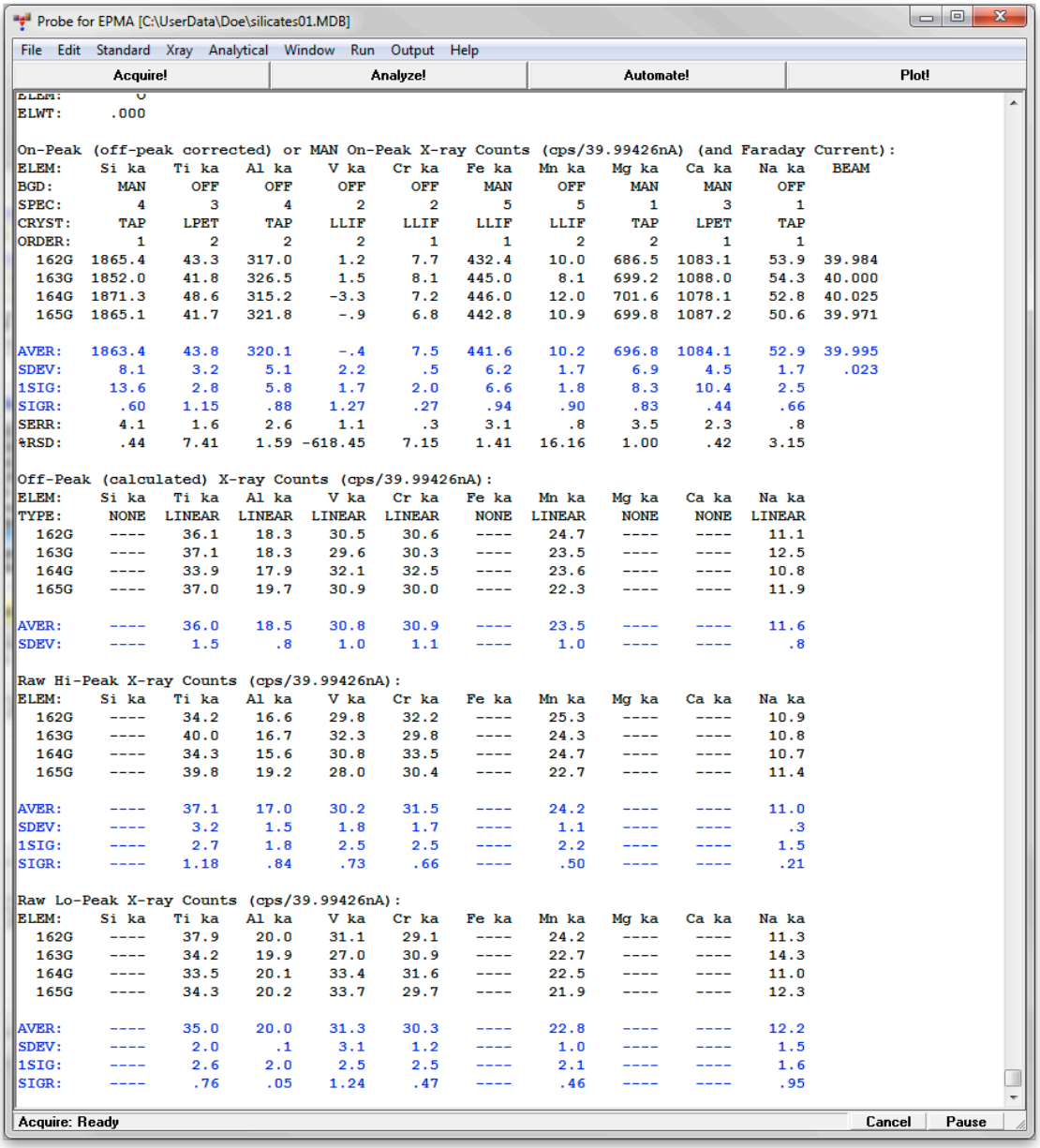


The stage moves to the coordinates of the first standard in the *Position List* list box. If *Use Confirm During Acquisition* was chosen, the **Confirm Positions** window opens, allowing a readjustment of the stage position and optical focus. X-rays are counted on peak and off peak positions for all elements as specified earlier. Progress can be monitored in the **Acquire!** window.



Finally, the Faraday cup is measured. The stage moves 15 µm in the X direction and the procedure is repeated for the remaining three points as specified in the *Automation Options* section of the **Automate!** dialog box. After completing data collection on the first standard, the stage travels to the next standard in the list and acquires four complete analyses on that standard. After finishing the automation schedule the familiar **AcquireStop** window opens and requires the user to click the **OK** button, thereby returning to the **Automate!** window.

The log window result for the x-ray count acquisition for the Augite Kakanui standard is seen below.



Probe for EPMA [C:\UserData\Doe\silicates01.MDB]

File Edit Standard Xray Analytical Window Run Output Help

Acquire! | Analyze! | Automate! | Plot!

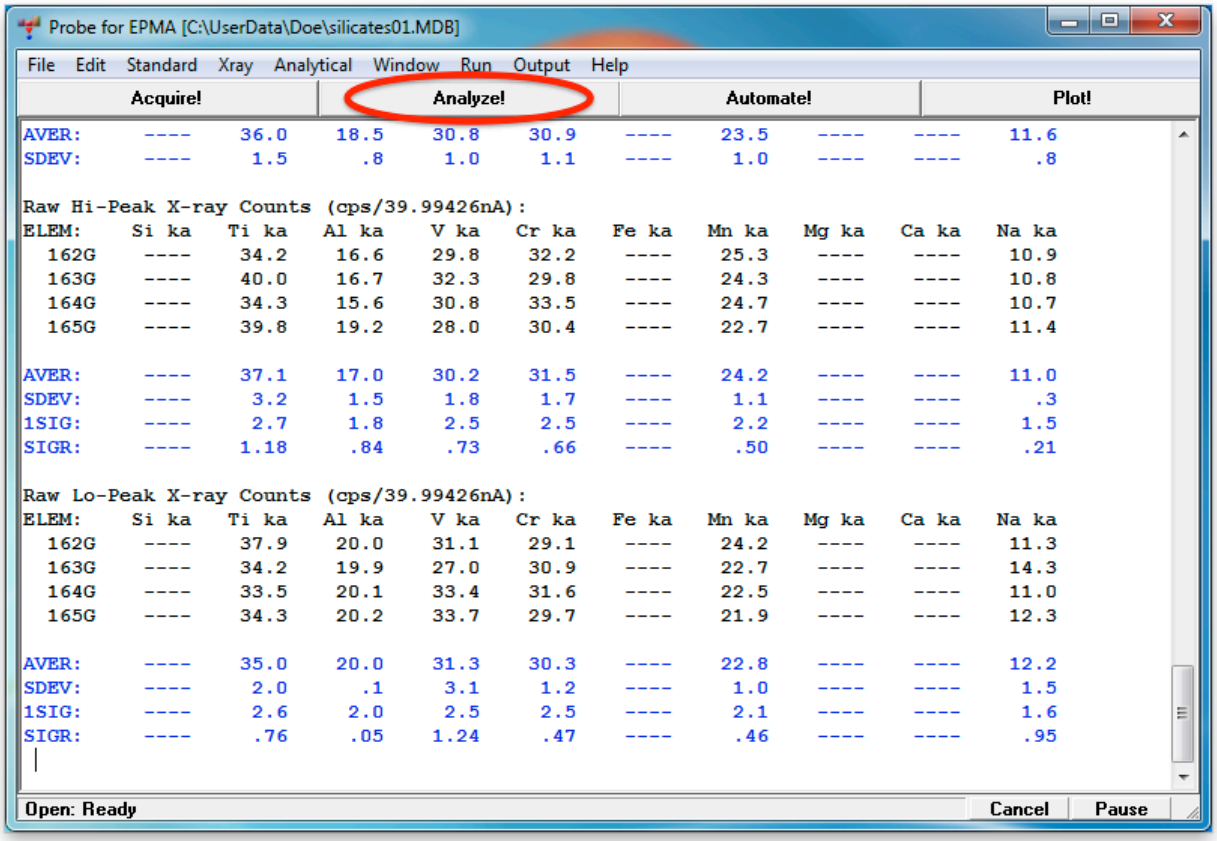
```
ELEM:        O
ELWT:     .000

On-Peak (off-peak corrected) or MAN On-Peak X-ray Counts (cps/39.99426nA) (and Faraday Current):
ELEM:    Si ka   Ti ka   Al ka    V ka   Cr ka   Fe ka   Mn ka   Mg ka   Ca ka   Na ka   BEAM
BGD:       MAN     OFF     OFF     OFF     OFF     MAN     OFF     MAN     MAN     OFF
SPEC:        4       3       4       2       2       5       5       1       3       1
CRYST:     TAP    LPET     TAP    LLIF    LLIF    LLIF    LLIF     TAP    LPET     TAP
ORDER:       1       2       2       2       1       1       2       2       1       1
  162G  1865.4    43.3   317.0     1.2     7.7   432.4    10.0   686.5  1083.1    53.9  39.984
  163G  1852.0    41.8   326.5     1.5     8.1   445.0     8.1   699.2  1088.0    54.3  40.000
  164G  1871.3    48.6   315.2    -3.3     7.2   446.0    12.0   701.6  1078.1    52.8  40.025
  165G  1865.1    41.7   321.8     -.9     6.8   442.8    10.9   699.8  1087.2    50.6  39.971

AVER:   1863.4    43.8   320.1     -.4     7.5   441.6    10.2   696.8  1084.1    52.9  39.995
SDEV:      8.1     3.2     5.1     2.2      .5     6.2     1.7     6.9     4.5     1.7    .023
1SIG:     13.6     2.8     5.8     1.7     2.0     6.6     1.8     8.3    10.4     2.5
SIGR:      .60    1.15     .88    1.27     .27     .94     .90     .83     .44     .66
SERR:      4.1     1.6     2.6     1.1      .3     3.1      .8     3.5     2.3      .8
%RSD:      .44    7.41    1.59 -618.45    7.15    1.41   16.16    1.00     .42    3.15

Off-Peak (calculated) X-ray Counts (cps/39.99426nA):
ELEM:    Si ka   Ti ka   Al ka    V ka   Cr ka   Fe ka   Mn ka   Mg ka   Ca ka   Na ka
TYPE:     NONE  LINEAR  LINEAR  LINEAR  LINEAR    NONE  LINEAR    NONE    NONE  LINEAR
  162G    ----    36.1    18.3    30.5    30.6    ----    24.7    ----    ----    11.1
  163G    ----    37.1    18.3    29.6    30.3    ----    23.5    ----    ----    12.5
  164G    ----    33.9    17.9    32.1    32.5    ----    23.6    ----    ----    10.8
  165G    ----    37.0    19.7    30.9    30.0    ----    22.3    ----    ----    11.9

AVER:     ----    36.0    18.5    30.8    30.9    ----    23.5    ----    ----    11.6
SDEV:     ----     1.5      .8     1.0     1.1    ----     1.0    ----    ----      .8

Raw Hi-Peak X-ray Counts (cps/39.99426nA):
ELEM:    Si ka   Ti ka   Al ka    V ka   Cr ka   Fe ka   Mn ka   Mg ka   Ca ka   Na ka
  162G    ----    34.2    16.6    29.8    32.2    ----    25.3    ----    ----    10.9
  163G    ----    40.0    16.7    32.3    29.8    ----    24.3    ----    ----    10.8
  164G    ----    34.3    15.6    30.8    33.5    ----    24.7    ----    ----    10.7
  165G    ----    39.8    19.2    28.0    30.4    ----    22.7    ----    ----    11.4

AVER:     ----    37.1    17.0    30.2    31.5    ----    24.2    ----    ----    11.0
SDEV:     ----     3.2     1.5     1.8     1.7    ----     1.1    ----    ----      .3
1SIG:     ----     2.7     1.8     2.5     2.5    ----     2.2    ----    ----     1.5
SIGR:     ----    1.18     .84     .73     .66    ----     .50    ----    ----     .21

Raw Lo-Peak X-ray Counts (cps/39.99426nA):
ELEM:    Si ka   Ti ka   Al ka    V ka   Cr ka   Fe ka   Mn ka   Mg ka   Ca ka   Na ka
  162G    ----    37.9    20.0    31.1    29.1    ----    24.2    ----    ----    11.3
  163G    ----    34.2    19.9    27.0    30.9    ----    22.7    ----    ----    14.3
  164G    ----    33.5    20.1    33.4    31.6    ----    22.5    ----    ----    11.0
  165G    ----    34.3    20.2    33.7    29.7    ----    21.9    ----    ----    12.3

AVER:     ----    35.0    20.0    31.3    30.3    ----    22.8    ----    ----    12.2
SDEV:     ----     2.0      .1     3.1     1.2    ----     1.0    ----    ----     1.5
1SIG:     ----     2.6     2.0     2.5     2.5    ----     2.1    ----    ----     1.6
SIGR:     ----     .76     .05    1.24     .47    ----     .46    ----    ----     .95
```

Acquire: Ready | Cancel | Pause

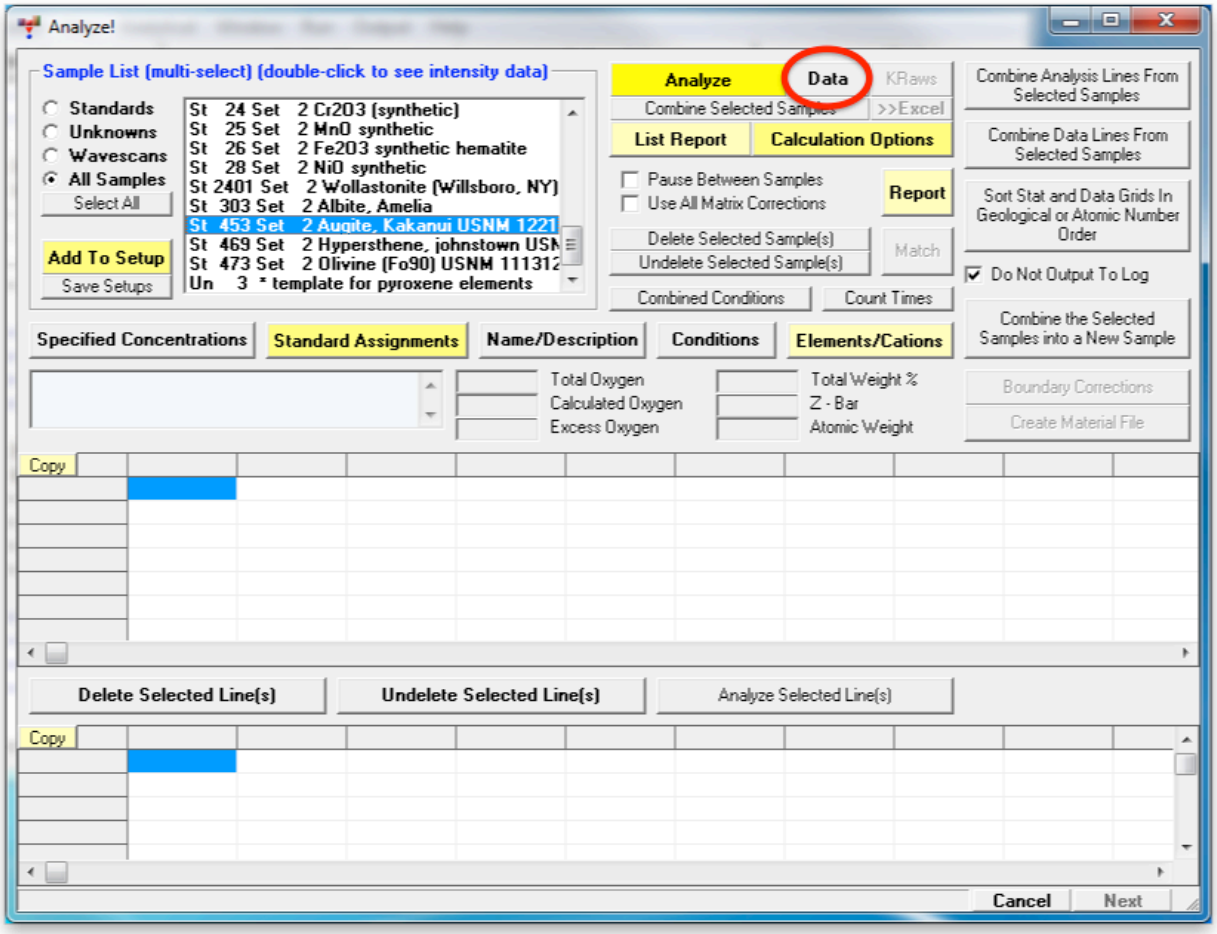
Off-peak counts were not collected for Si, Fe, Mg, and Ca, where MAN background correction was specified. In addition to the four individual lines of count data, the *AVER*, *SDEV*, *1SIG*, *SERR*, and *%RSD* are calculated. *AVER* is the average intensity reading of each element column. *SDEV* is the standard deviation of these results, *1SIG* (one sigma) is the predicted standard deviation, and *SERR* (standard error) is essentially the precision of the average. *%RSD* (percent relative standard deviation) is the *SDEV* divided by the *AVER* times 100. See the User's Guide and Reference documentation for exact equations. The output of the raw data counts for the remaining standards is not shown here to save space.

Evaluate Standard Count Data

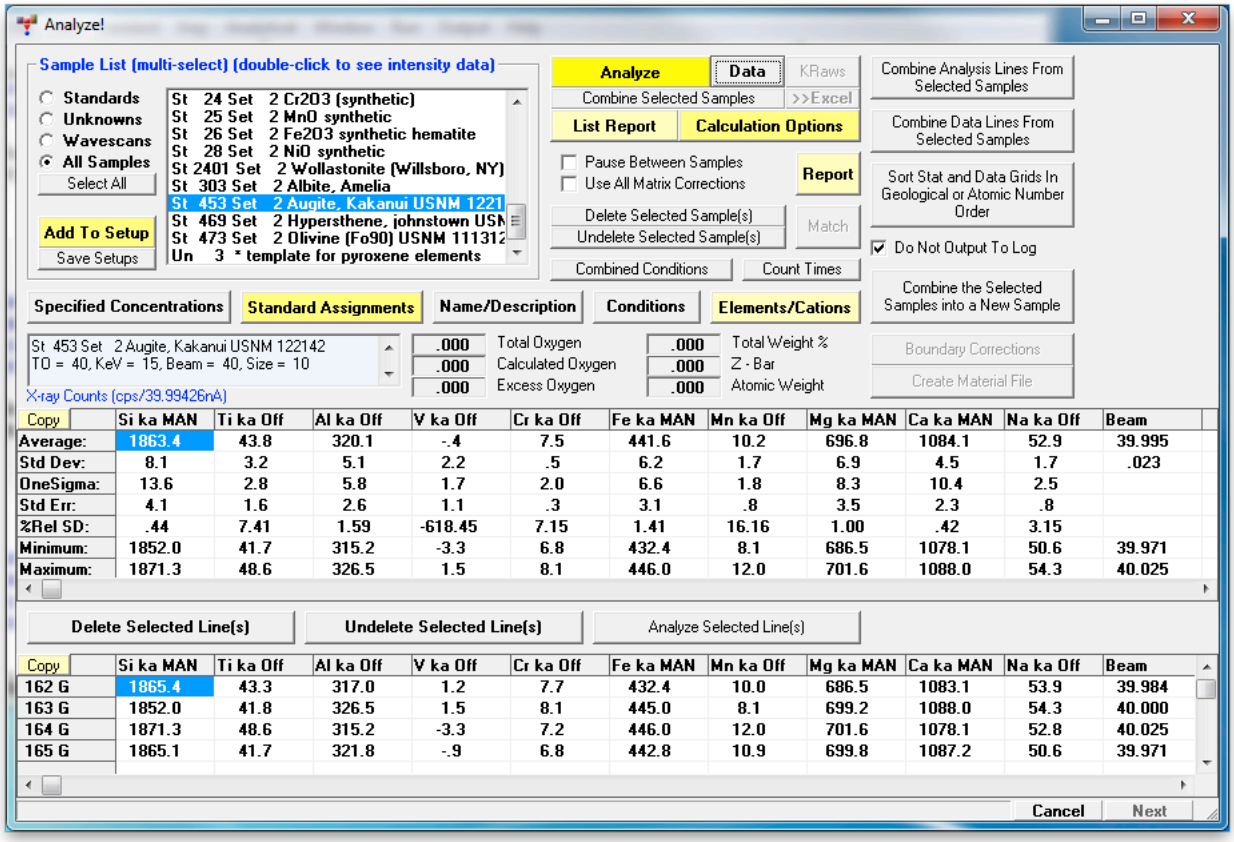
After standard data is acquired it is useful to check the data for agreement among standards and for possible interferences. Click the **Analyze!** button in the main PROBE FOR EPMA log window.



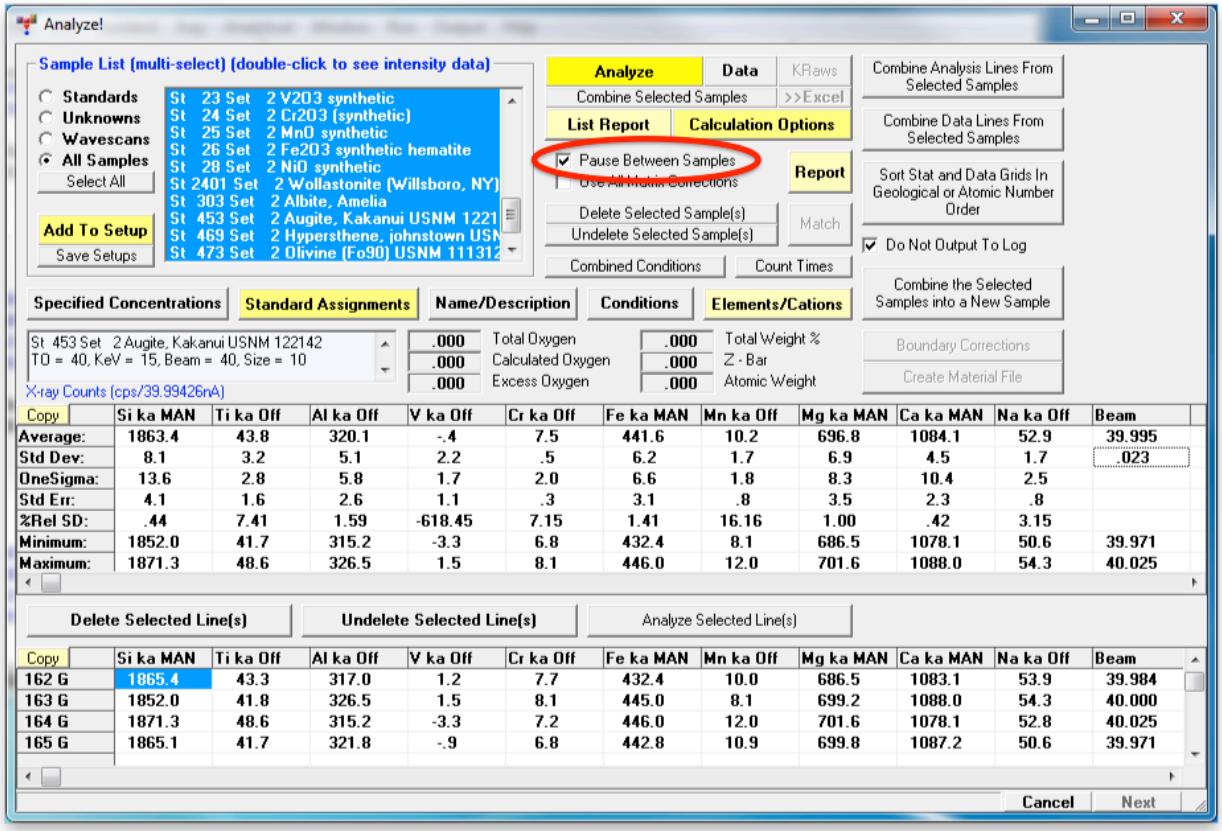
This opens the **Analyze!** dialog box. The *Sample List* list box contains the list of the standards that on which data has been acquired. To examine the raw count data acquired on any standard run under automation, first select the standard of interest and click the **Data** button.



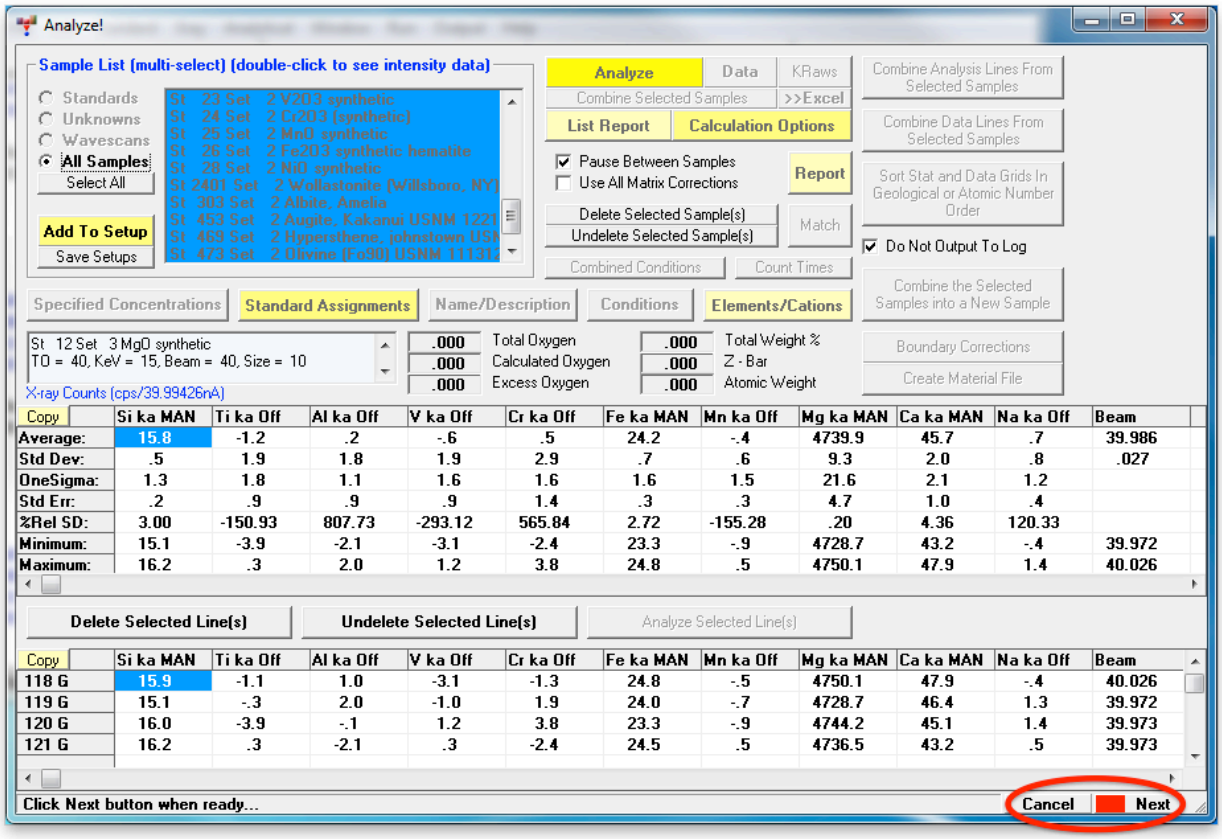
The raw count data for the four automated measurements of the Augite Kakanui standard are shown below. Each individual line (162 G to 165 G) is illustrated along with the *Average*, *Std Dev*, *OneSigma*, *Std Err*, *%Rel SD*, and *Minimum* and *Maximum* of the acquired points. This count data is also printed to the log window.



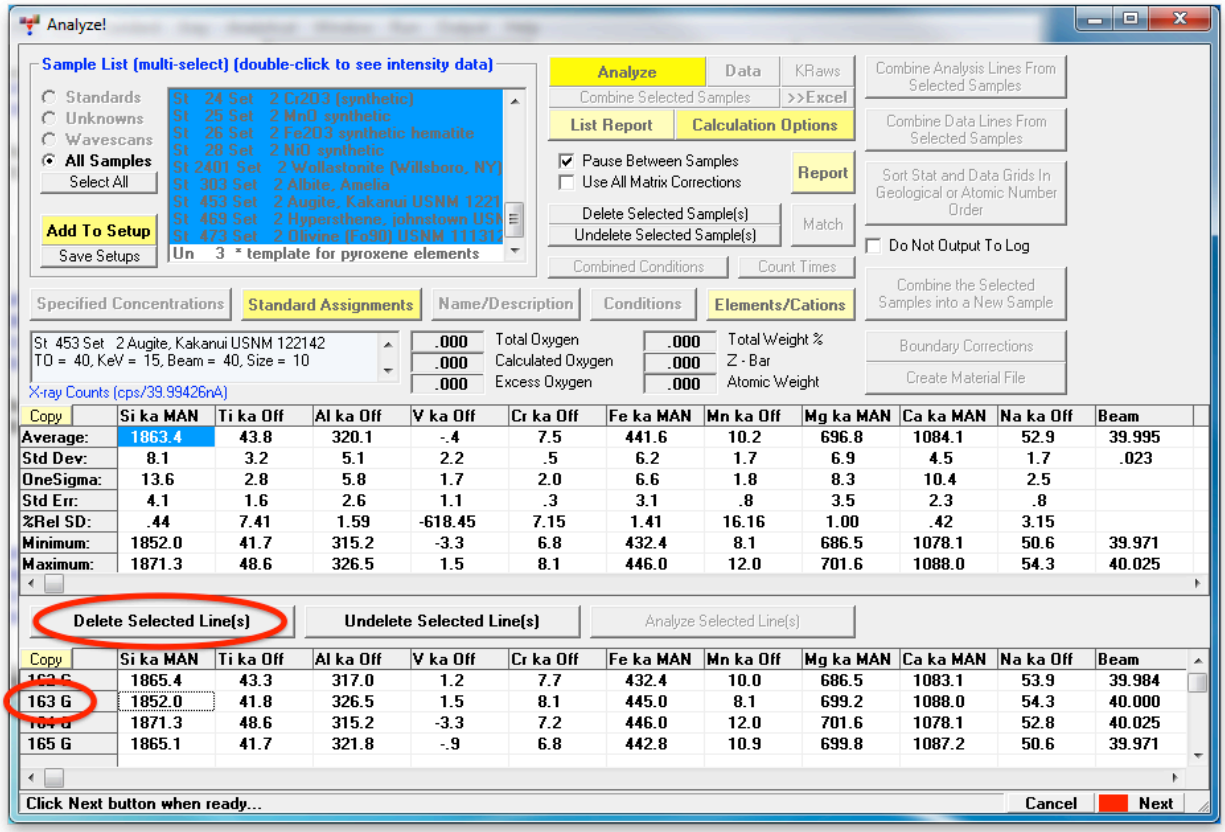
Examine the raw count data for each standard. If more than one sample/standard is selected for assessment, select the *Pause Between Samples* check box.



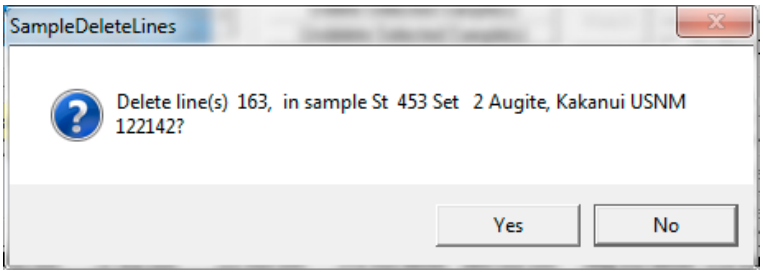
When this box is checked, the program will automatically pause after displaying each analysis until the user clicks the **Cancel** or **Next (red flashing)** buttons on that are located at the bottom of the log window.



If there are any bad data points, use the **Delete Selected Line(s)** button to flag a line of data as bad. In the Augite Kakanui standard, seen below, line 163 G (good) shows the lowest Si and highest Al counts which could indicate contribution of a small inclusion to the analysis. To delete, click on the line number in the first column, highlighting the line. Next click the **Delete Selected Line(s)** button.

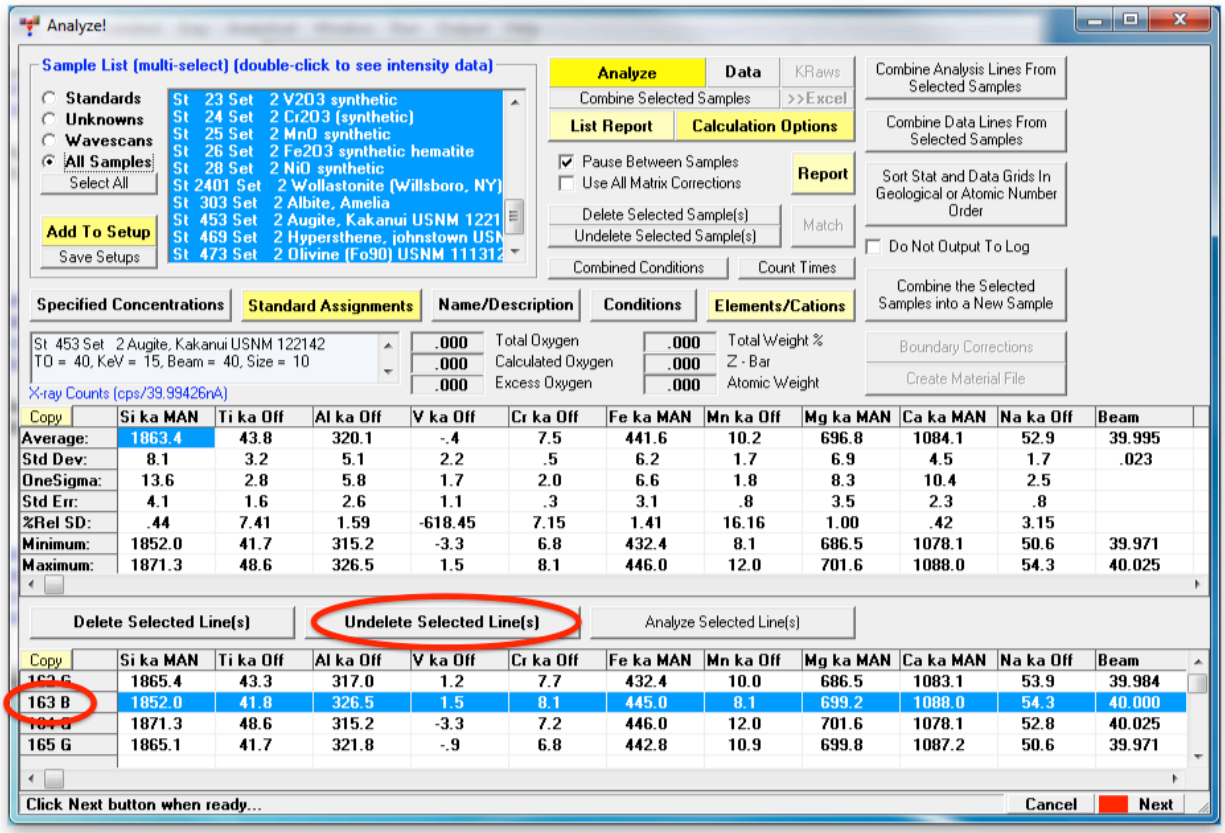


This opens the **SampleDeleteLines** window.



Click the **Yes** button.

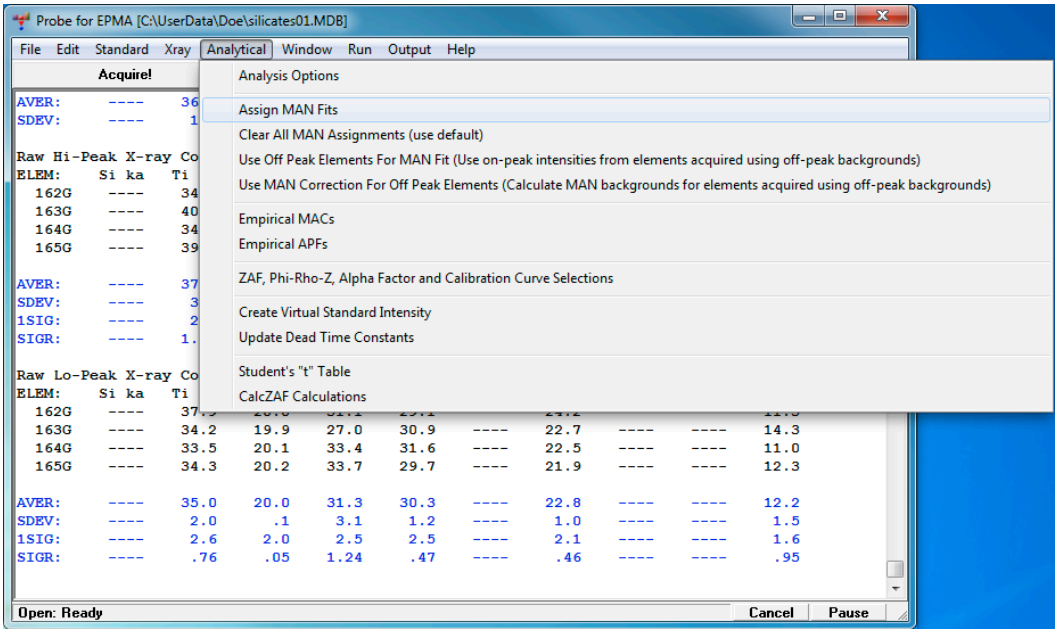
The computer will flag this line with a B (bad) and ignore this data for any subsequent calculations, but the raw point data is still kept in the SILICATES01.MDB data file. Note that data lines can always be undeleted later with the **Undelete Selected Line(s)** button.



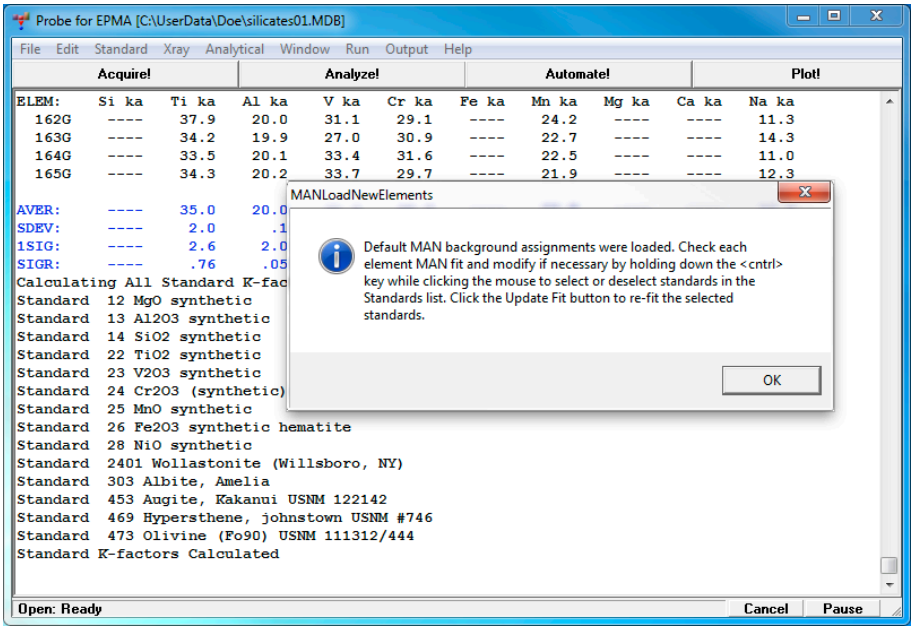
At this point, the user has collected all standardization data and is ready to make MAN background assignments for the elements Si, Fe, Mg, and Ca.

Assign MAN Background Calibrations

From the main PROBE FOR EPMA log window, select **Analytical** from the menu bar and click **Assign MAN Fits** from the menu choices.

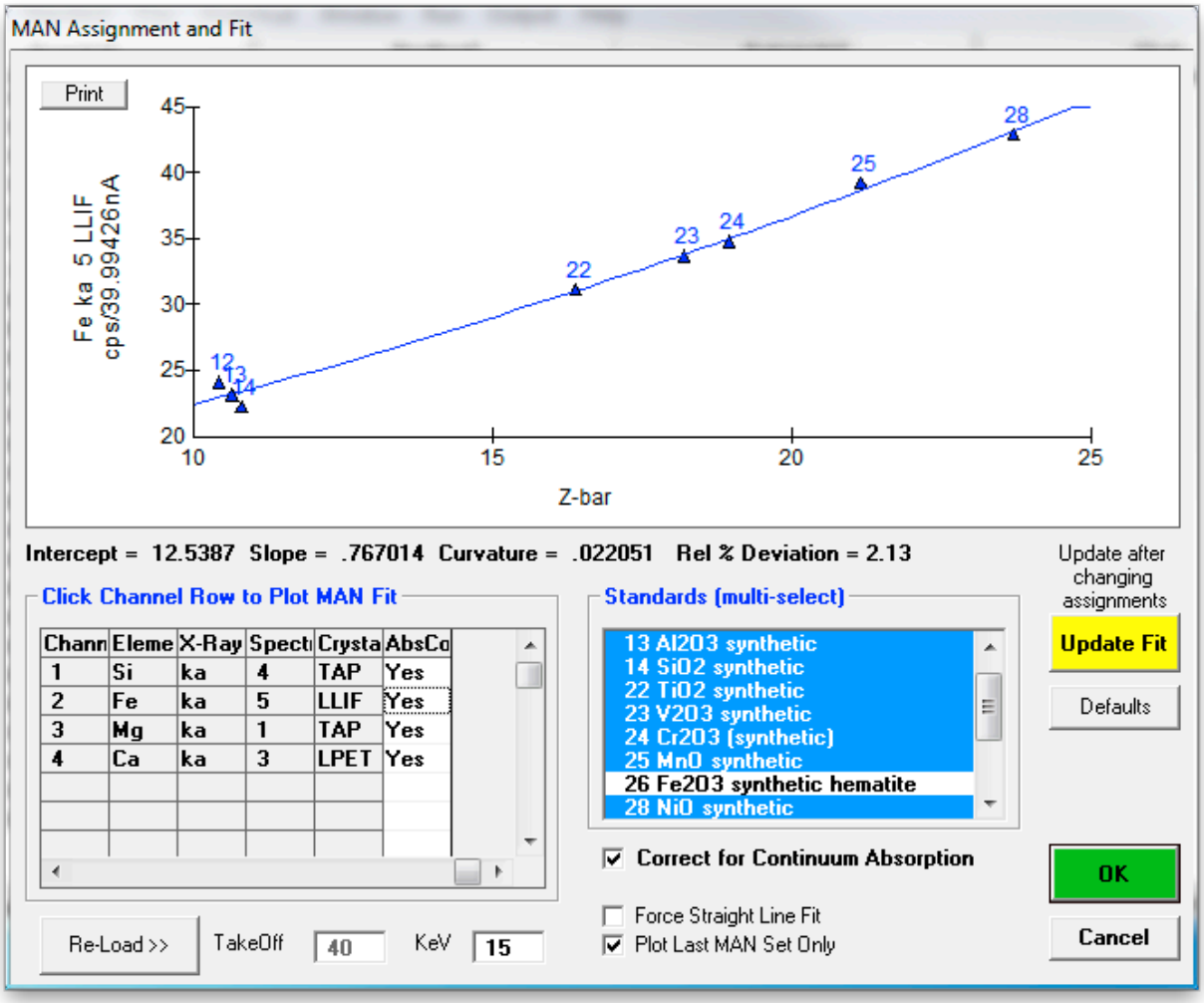


This opens the **MANLoadNewElements** window. Default standards have now been assigned for the background correction of each element based on the standard database.



Click the **OK** button.

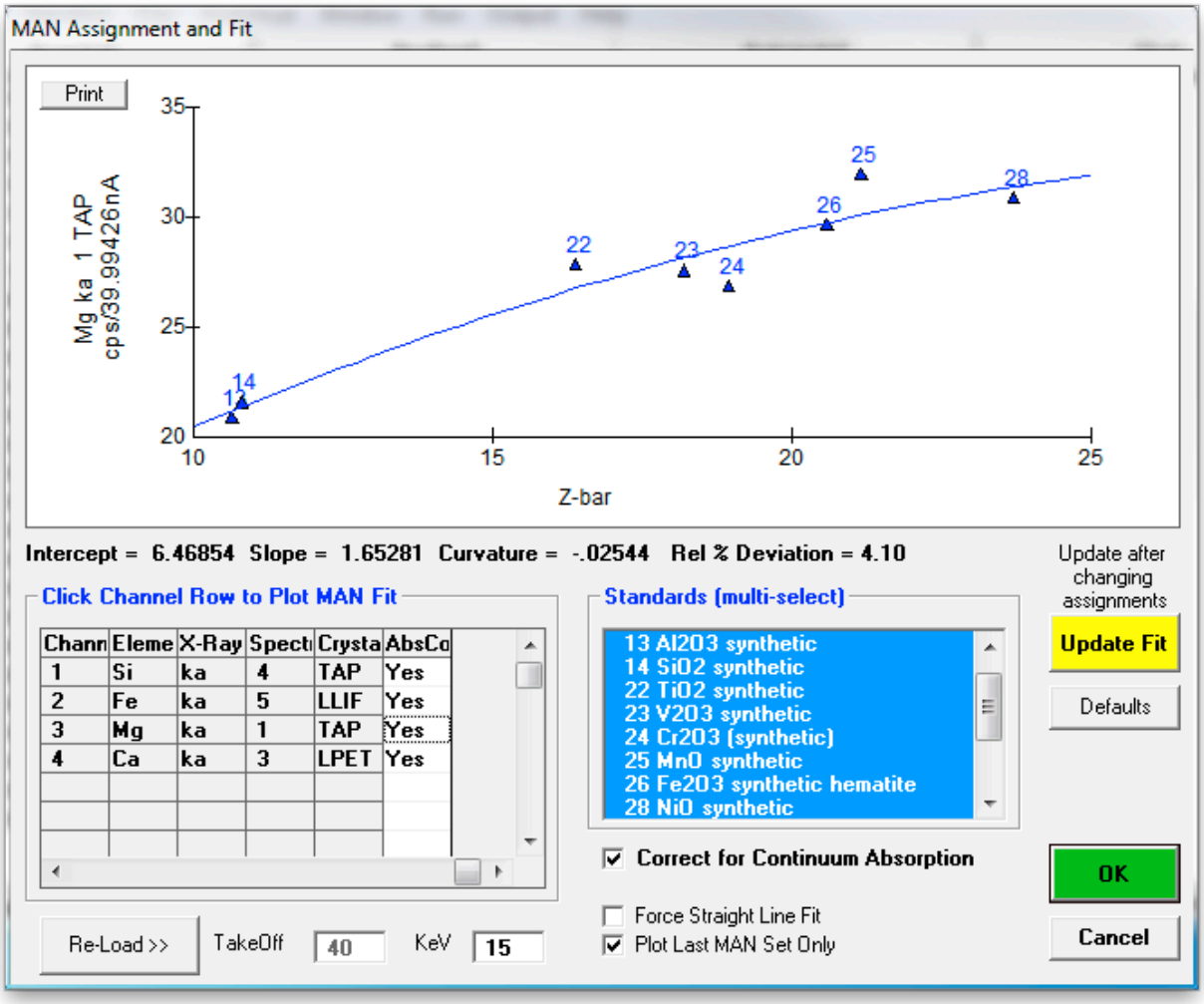
This opens the **MAN Assignment and Fit** dialog box. The second element in the list, iron (Fe), is shown below.



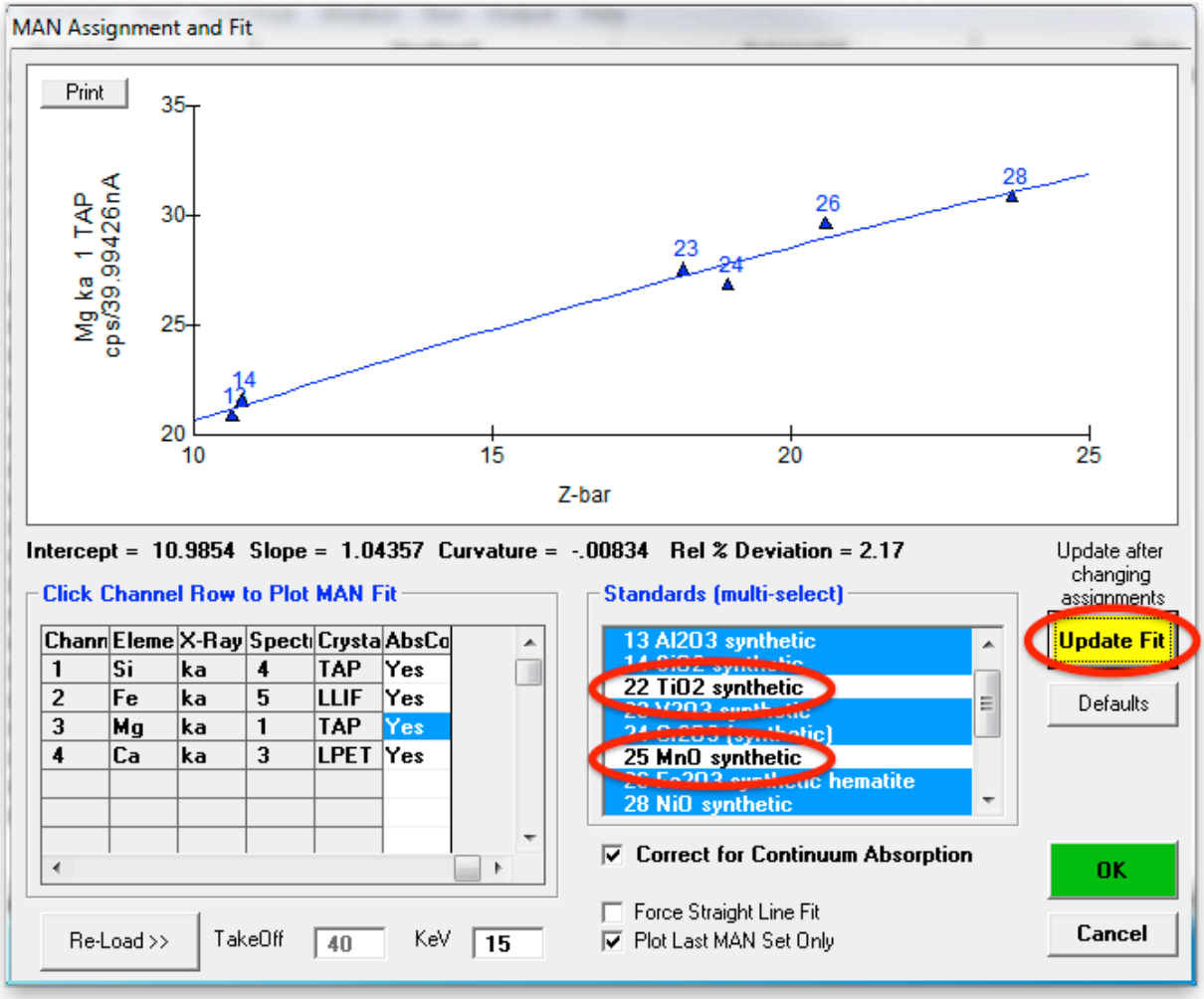
From this dialog box, the user may display and modify the MAN background assignments and fits used for the background correction of all elements in the current run. The advantage of this method is that it requires only a simple calibration of the analyzing channel over a range of atomic number. Substantial time may be saved when many samples are to be analyzed. However, if measuring high atomic number samples and/or trace concentrations, the off-peak background correction technique is usually superior.

To review the MAN assignments, select the row for each element in the *Click Channel Row to Plot MAN Fit* section. The measured background counts are plotted as a function of the mean atomic number (MAN or Z-bar). Select standards from the *Standards* list box that do not contain the element itself. Use the shift key and the control key together with the left mouse button to select a range or additional individual standards, respectively. Choose at least five standards per element and click the **Update Fit** button to update the graph. Choose between a second-order polynomial or force a straight line fit. For further details and suggestions, see the User's Guide and Reference documentation.

Standards 22 (TiO2 synthetic) and 25 (MnO synthetic) plot slightly above the fit curve for magnesium. This could indicate a spectral interference or, more likely in this case, a minor amount of Mg in these standards which was so far not documented in the standards database.



Use <ctrl>-click to deselect these two standards in the *Standards* list and click the **Update Fit** button to update the graph. This change only applies to the MAN assignments for the current element, in this case Mg.

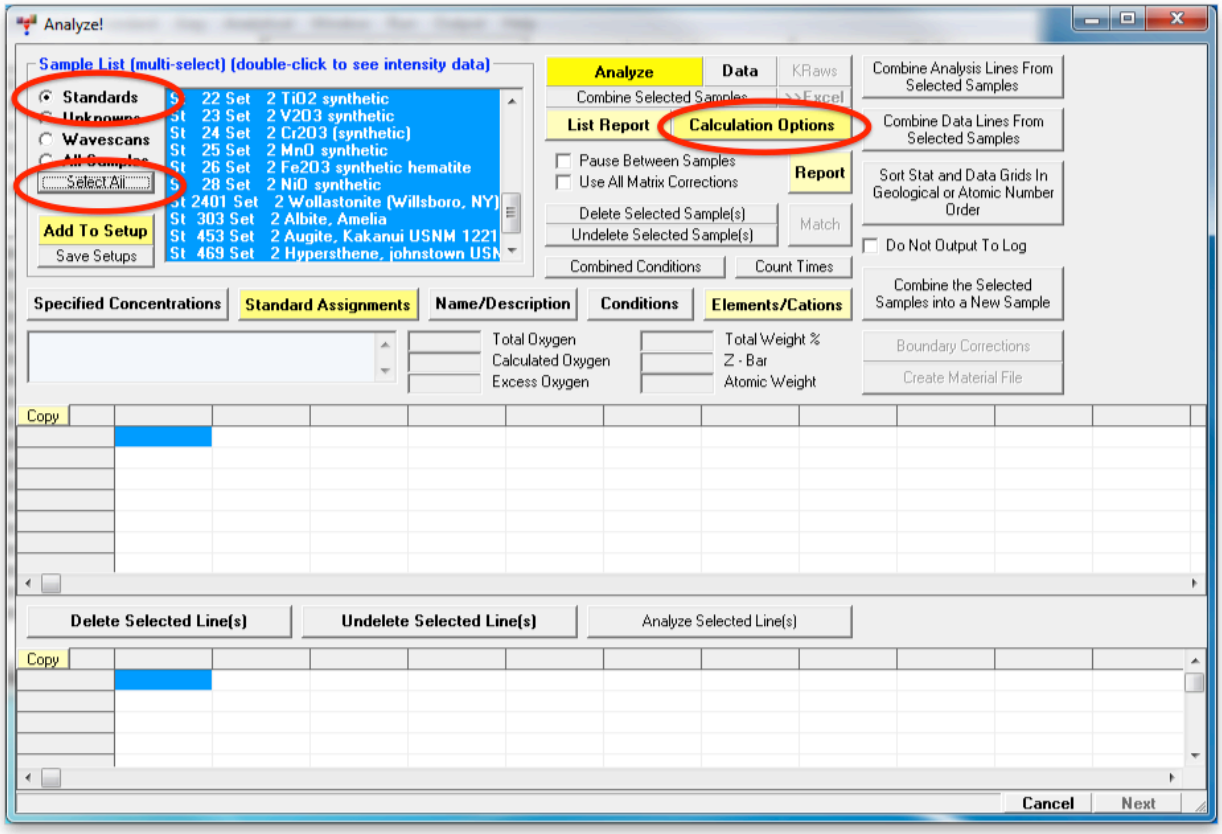


When done adjusting individual elements, click the **OK** button to store the updated MAN background corrections.

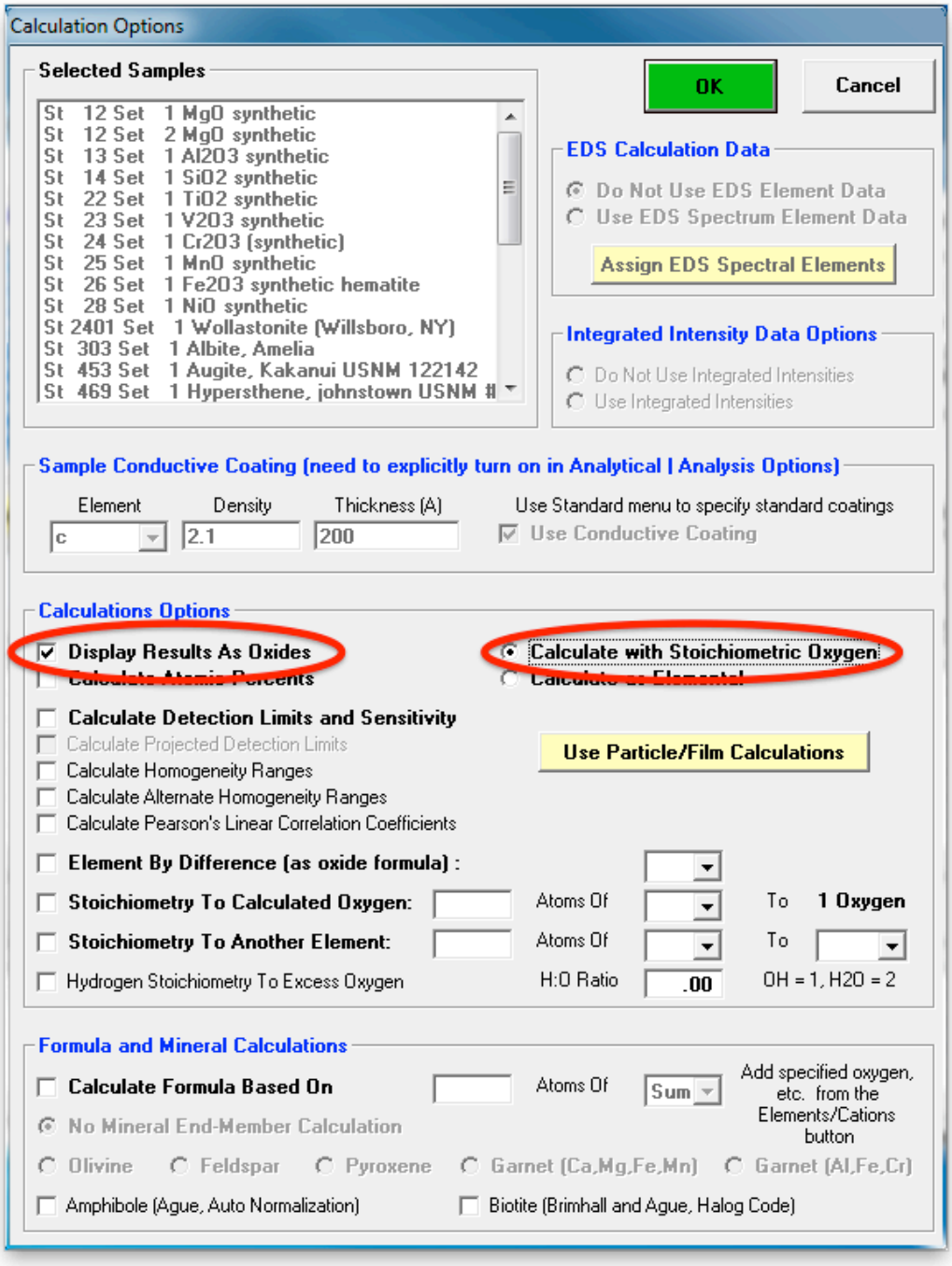
Analyze Standard Samples

The user will now analyze all of the raw standard data to calculate quantitative compositions. The results of the standards provide a valuable check on the quality of the analysis.

Click the **Analyze** button in the main PROBE FOR EPMA log window to re-open or bring forward the **Analyze!** window. Under *Sample List* select the *Standards* button. Click the **Select All** button to highlight all standards. Click the **Calculation Options** button.



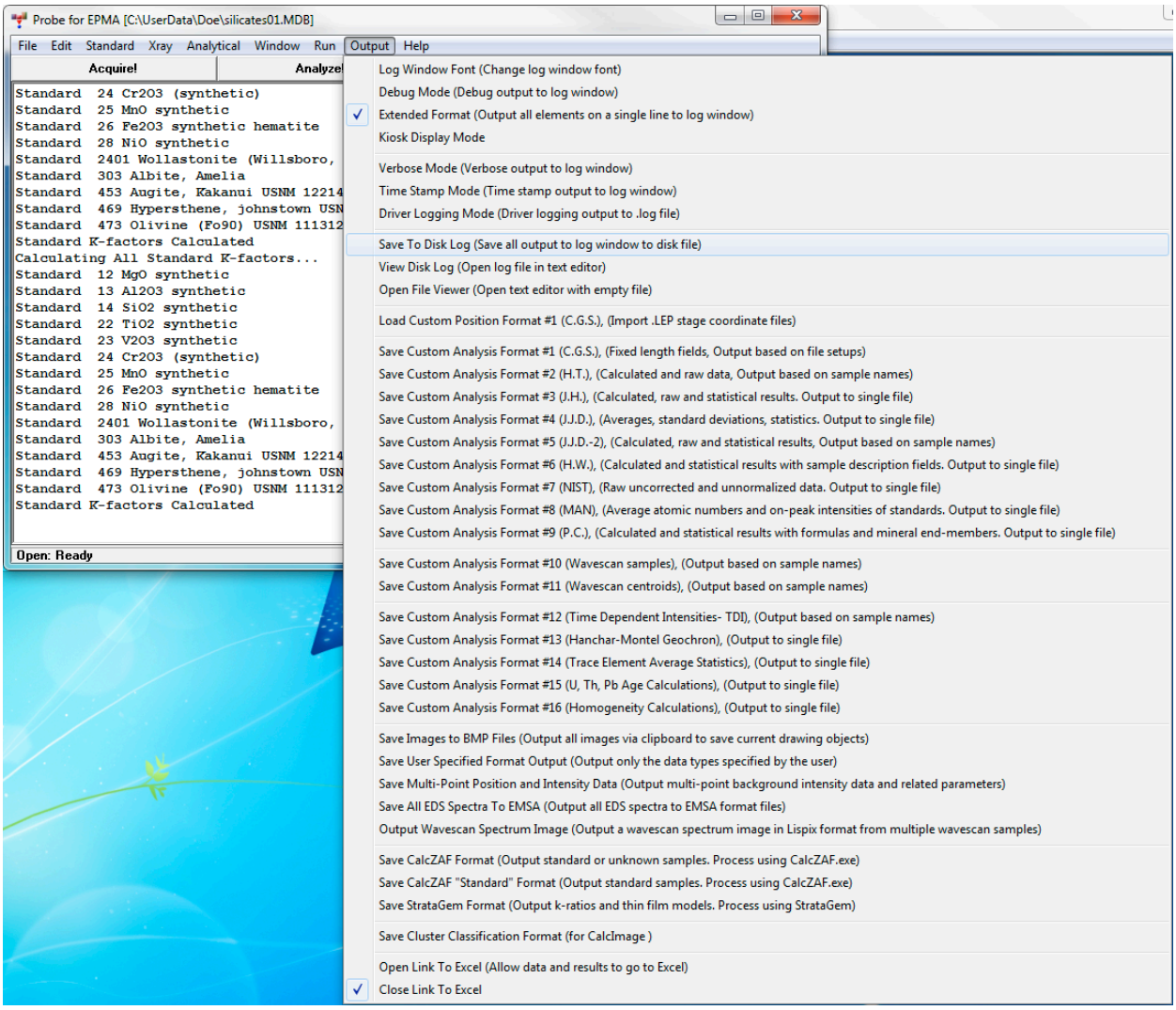
This action opens the **Calculation Options** dialog box. As all standards and unknowns are oxide compounds and oxygen is not measured in this method, click the tick box *Display Results As Oxides* and the radio button *Calculate with Stoichiometric Oxygen* under *Calculations Options*. Elemental results are always calculated and written to the log window.



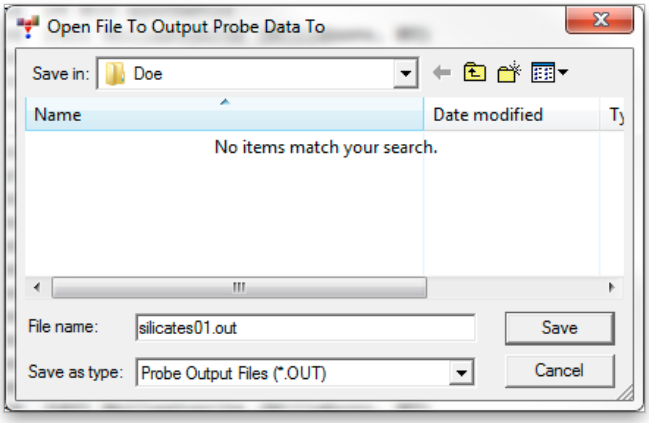
Click the **OK** button to output data in oxide form.

Analyzing all of the data on the standards will create a large amount of output, possibly overflowing the log window buffer, depending on the value specified in the LogWindowBufferSize parameter in the PROBEWIN.INI file. The size of the log window buffer is limited only by the amount of memory available. Setting this parameter to 512000 bytes is roughly equivalent to 300 pages of average density text. In some cases saving all log window output to a user specified text file for viewing with a text editor or printing to a printer may be best.

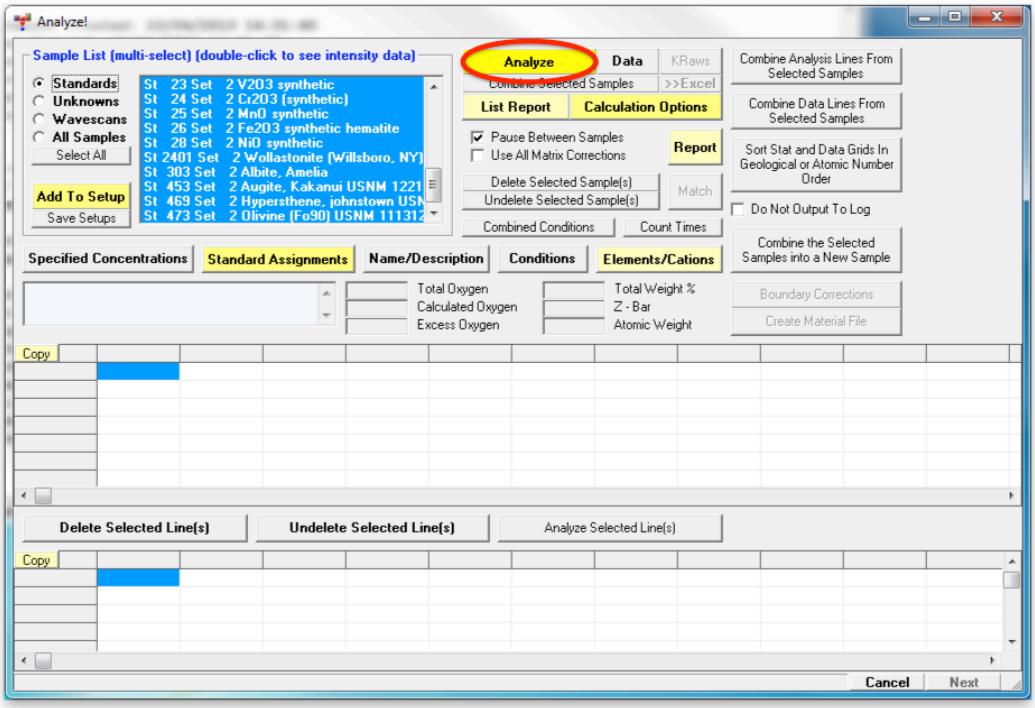
Select **Output** from the menu bar in the main log window and click **Save to Disk Log**.



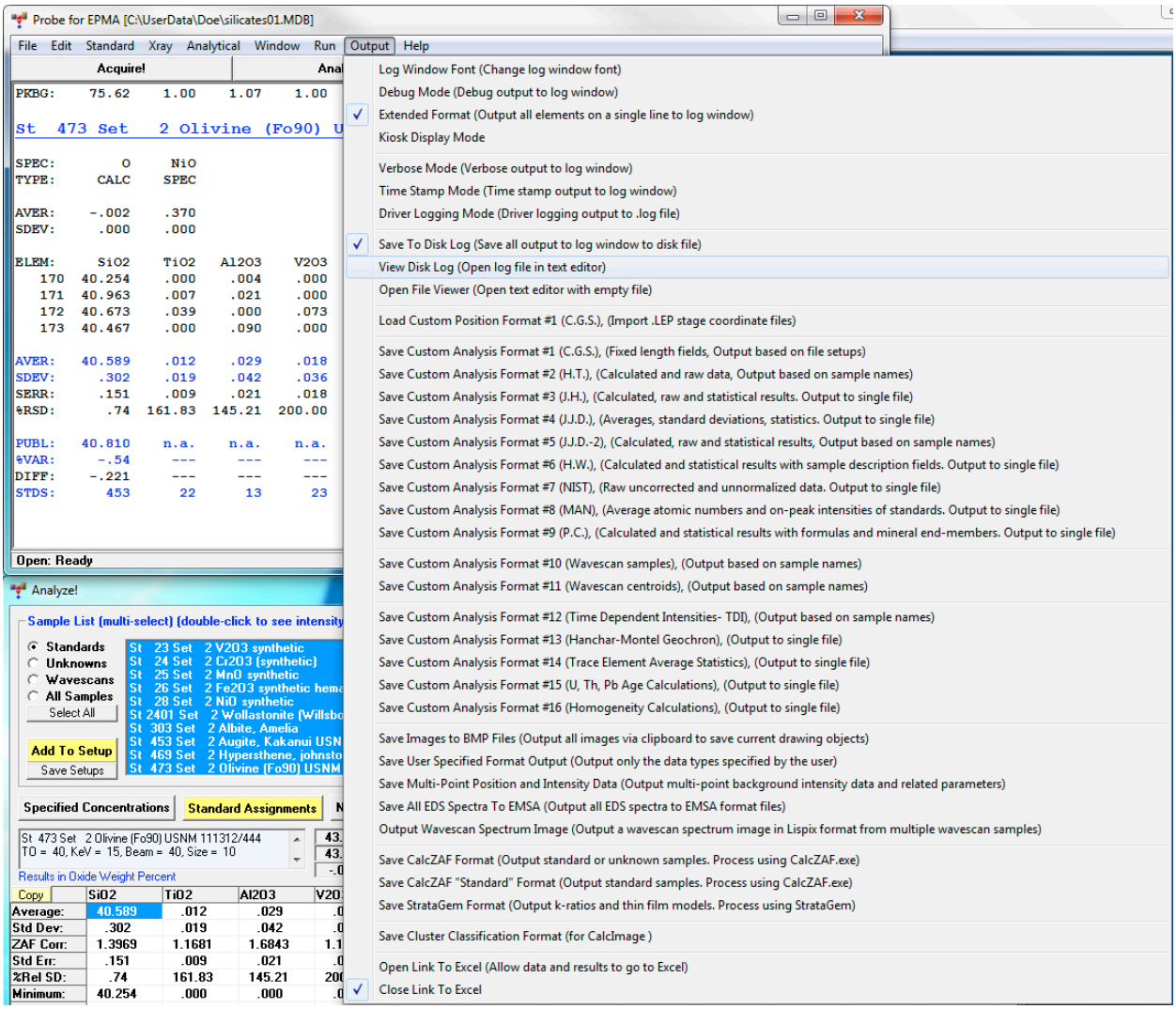
This opens the **Open File To Output Probe Data To** dialog box. The *Save in:* location will be the directory specified for the original file name (SILICATES01.MDB). All subsequent files created by the user will use this location. Edit the *File name* if desired. The default output file has the extension .OUT. Note that the raw data is always saved in the .MDB run file for future re-calculation and/or output. Click **Save** when finished.



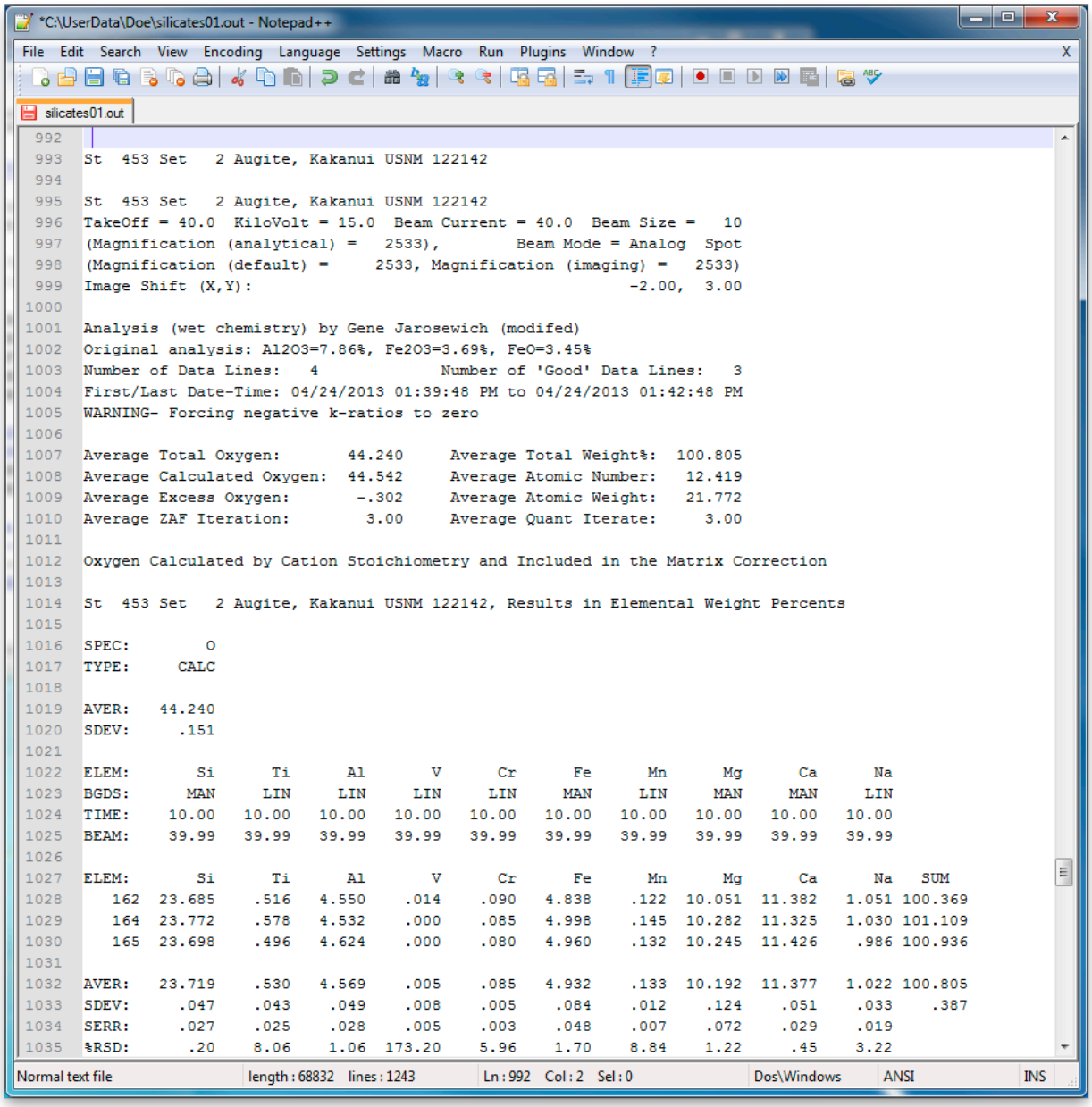
Select the **Analyze!** button in the main PROBE FOR EPMA log window to bring forward the **Analyze!** dialog box. Click the **Select All** button highlighting all standards again. Then click the **Analyze** button. This will analyze all selected standard data into the specified text file. If the *Pause Between Samples* tick box is selected, the program will pause after each standard to allow the user to view the results directly in the **Analyze!** window.



To view this data in full, return to the main PROBE FOR EPMA log window and select **Output** from the menu bar again and click **View Disk Log** from the menu.

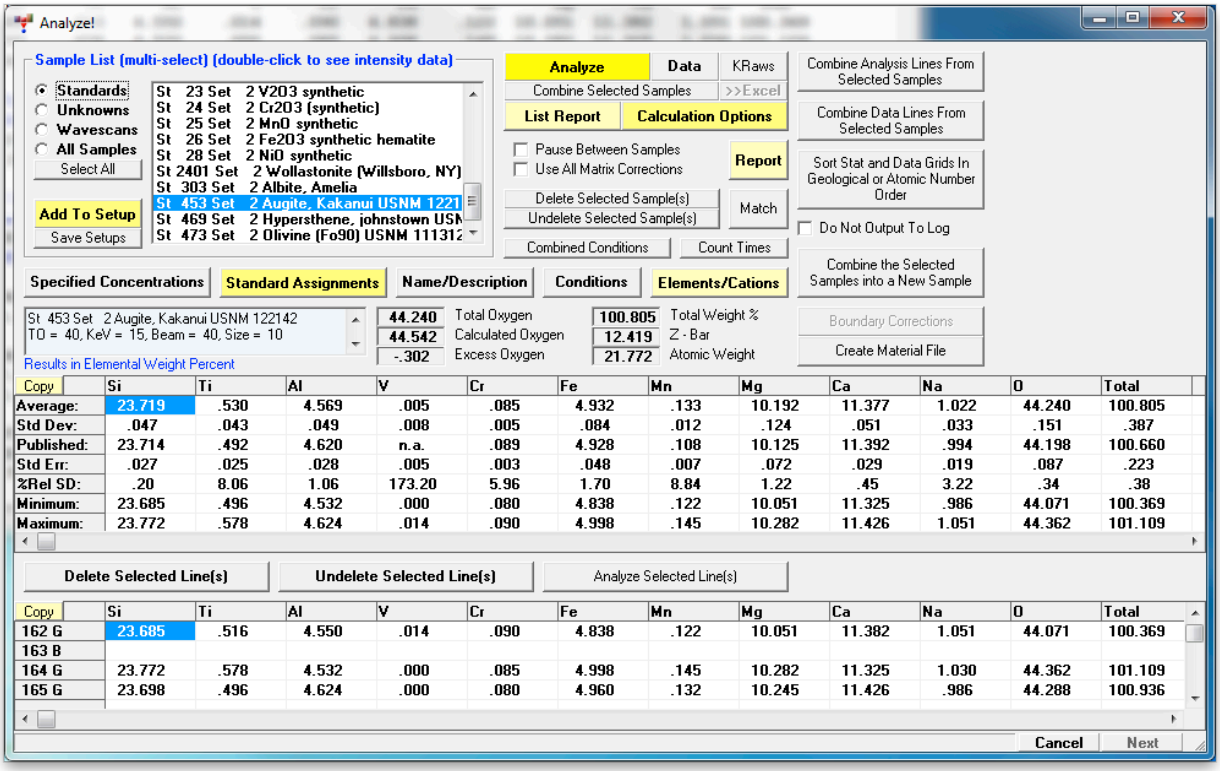


This opens the file editor. This example utilizes the editor **Notepad++**, seen below. A number of text file viewers may be used. To utilize a specific editor such as Notepad, Textpad or Word, edit the FileViewer keyword in the PROBEWIN.INI file.

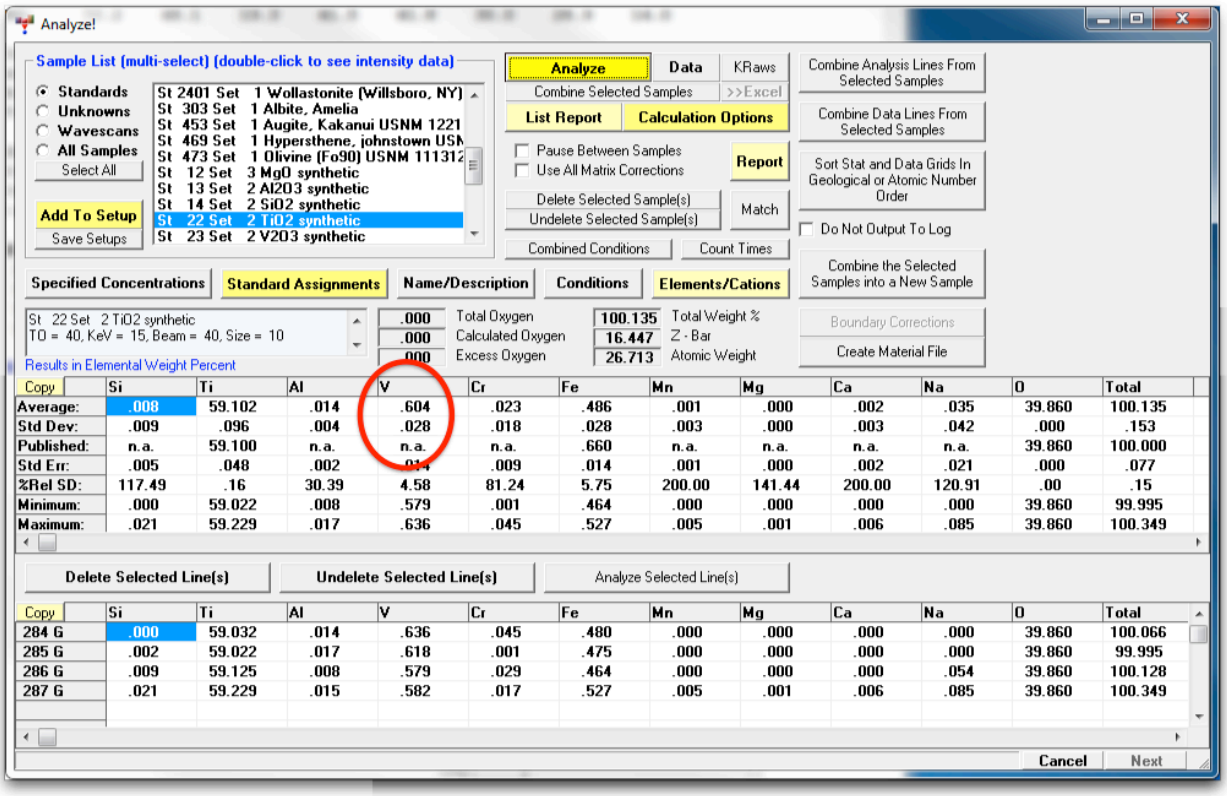


The user may now scroll through the analyzed standards using the text editor or may direct the file data to a printer by selecting **File** from the **Notepad++** menu bar and clicking on **Print** in the drop-down menu.

Since all elements were acquired on all standards, examination of the results will serve as quality control for the calibration. Several of the standard compositions will be displayed in the **Analyze!** window. The first example is the Augite Kakanui standard displayed below, which is currently assigned as primary standard for silicon. To be able to directly compare the results with the published values, the tick box *Display Results As Oxides* in the **Calculation Options** window is deselected and elemental wt% are displayed. An excellent agreement between measured and published values is observed. No composition is calculated for line 163 B, which was disabled before by the user.



The analysis of the TiO_2 standard reveals apart from titanium 0.5 wt% iron which is a known contamination, and 0.6 wt% vanadium. This sample has no vanadium; here the user sees the notorious Ti-V spectral interference. This will be corrected for (shortly) using the automatic interference correction routine.



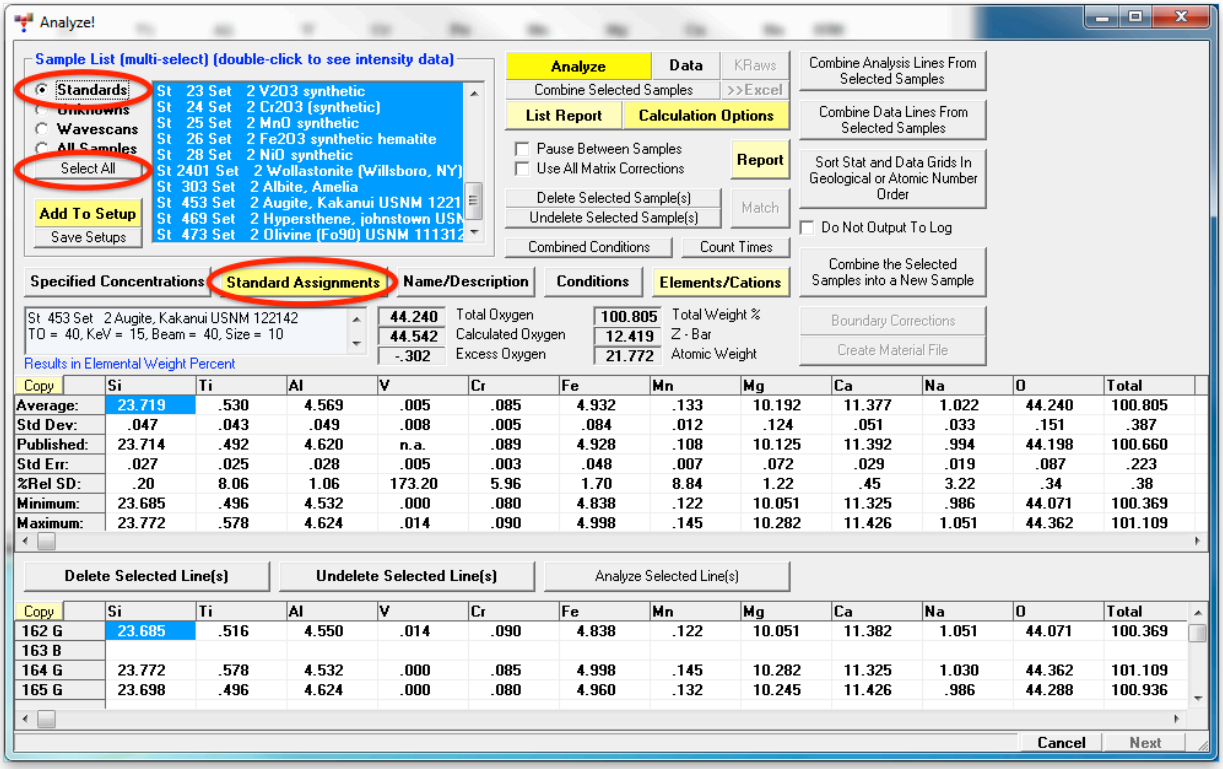
All of the data lines gathered on the standards are examined in the same way and appear close to their standard database values. To save space they will not be reproduced here.

Spectral Interference Assignments

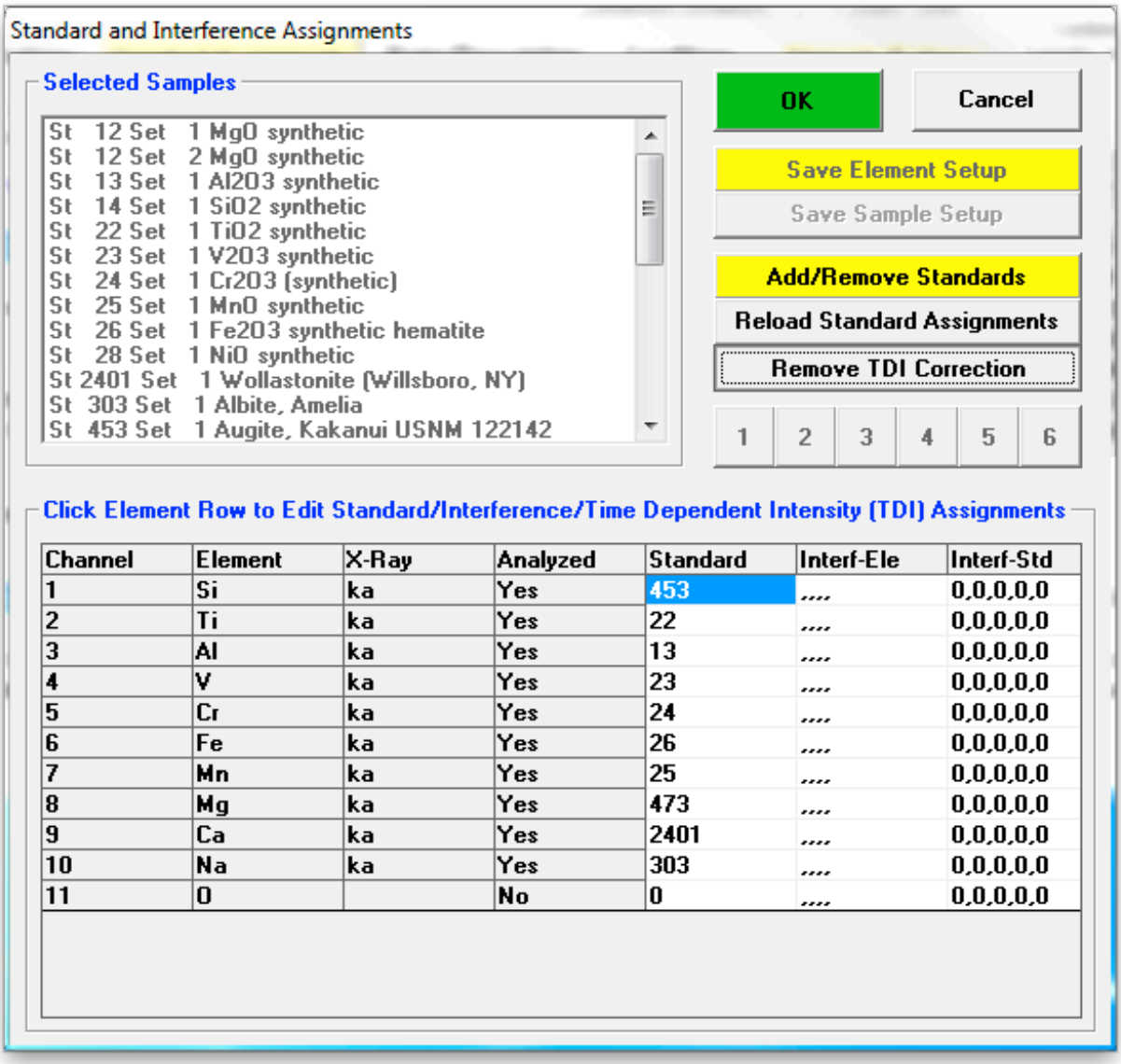
PROBE FOR EPMA allows the user to select a fully quantitative correction for spectral interferences. The program can only correct for interferences if both the interfered and interfering elements are measured. Further, data for an interference calibration standard must be acquired that contains a major concentration of the interfering element and none of the interfered element or any other elements that interfere with the interfered element.

The review of the analysis results for the standards in the previous section has identified interferences between various transition metals which require correction. Many transition metal Kα x-ray lines are interfered by the element to the left in the periodic table of elements, e.g. Ti interferes with V, V with Cr, and so forth.

Select the *Standards* button in the *Sample List* and click the **Select All** button in the **Analyze!** window. Next, click the **Standard Assignments** button.

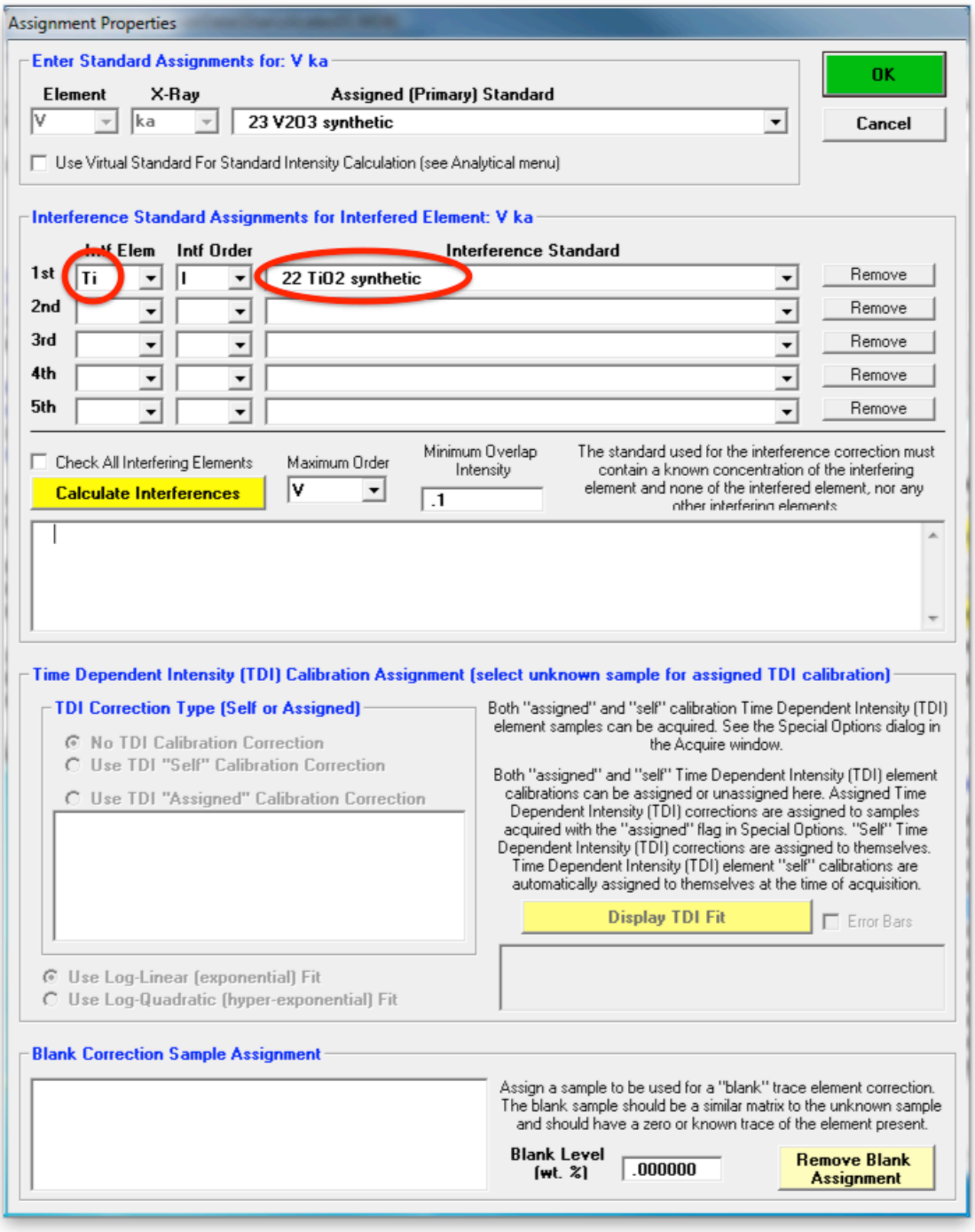


Clicking this button opens the **Standard and Interference Assignments** dialog box.



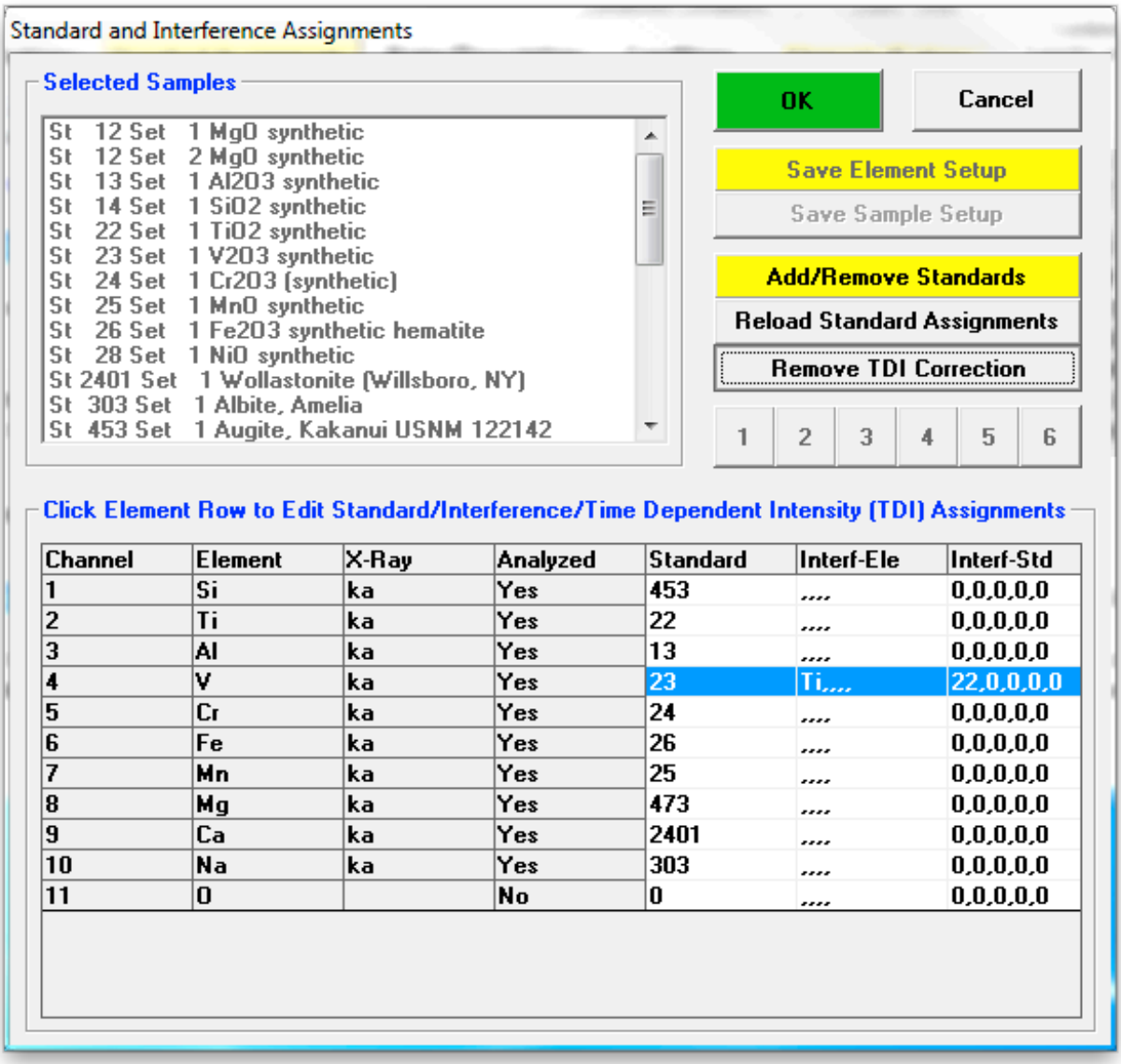
Click on the element row for vanadium to edit the *Interference Assignments*.

The **Assignment Properties** dialog box for vanadium opens. Select the first *interference element* for this element and the corresponding *standard* from the respective drop down menus. Select a standard that contains a known amount of the interfering element but none of the interfered element.

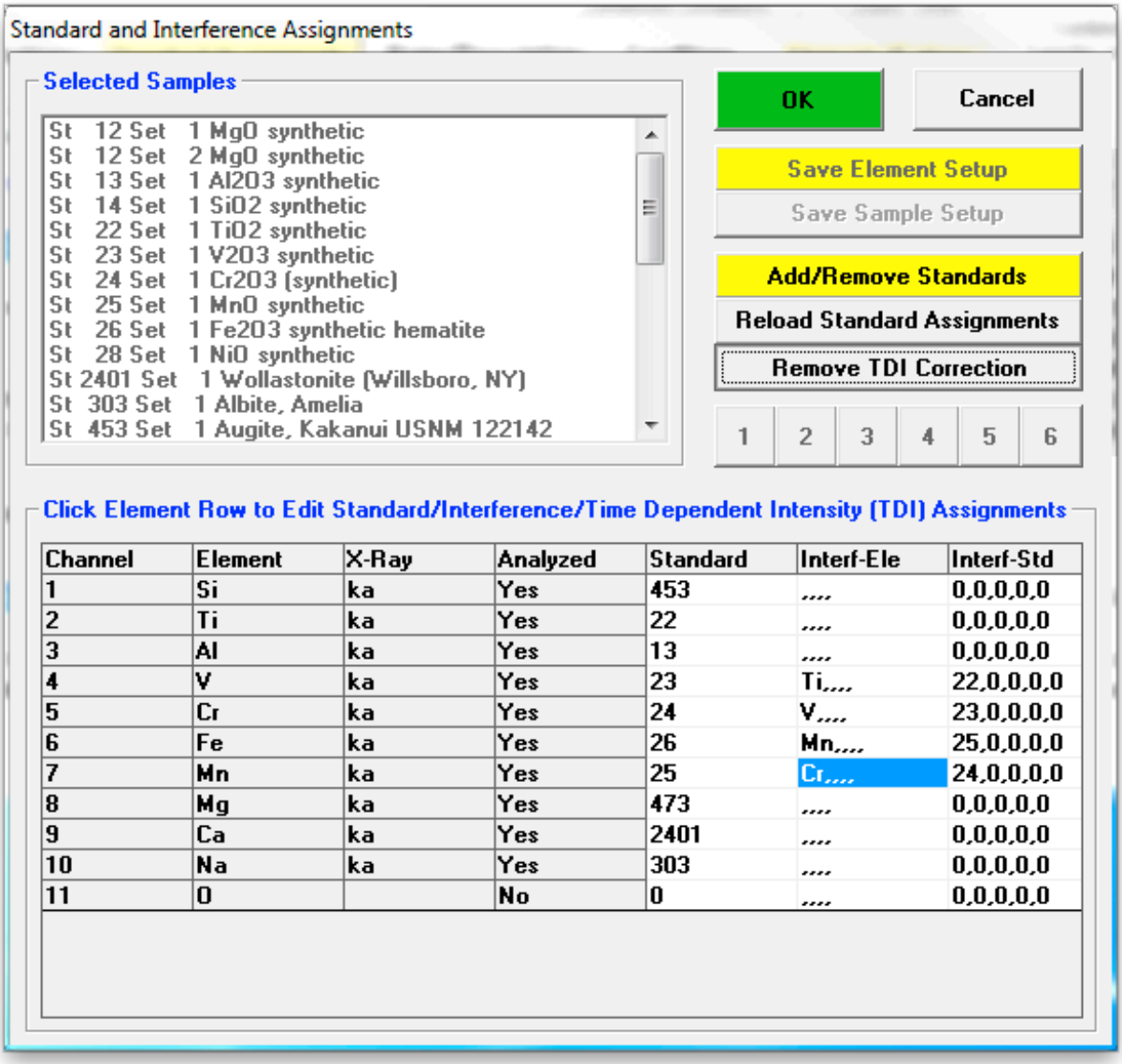


Click the **OK** button when finished.

The Standard and Interference Assignments window will appear as below.

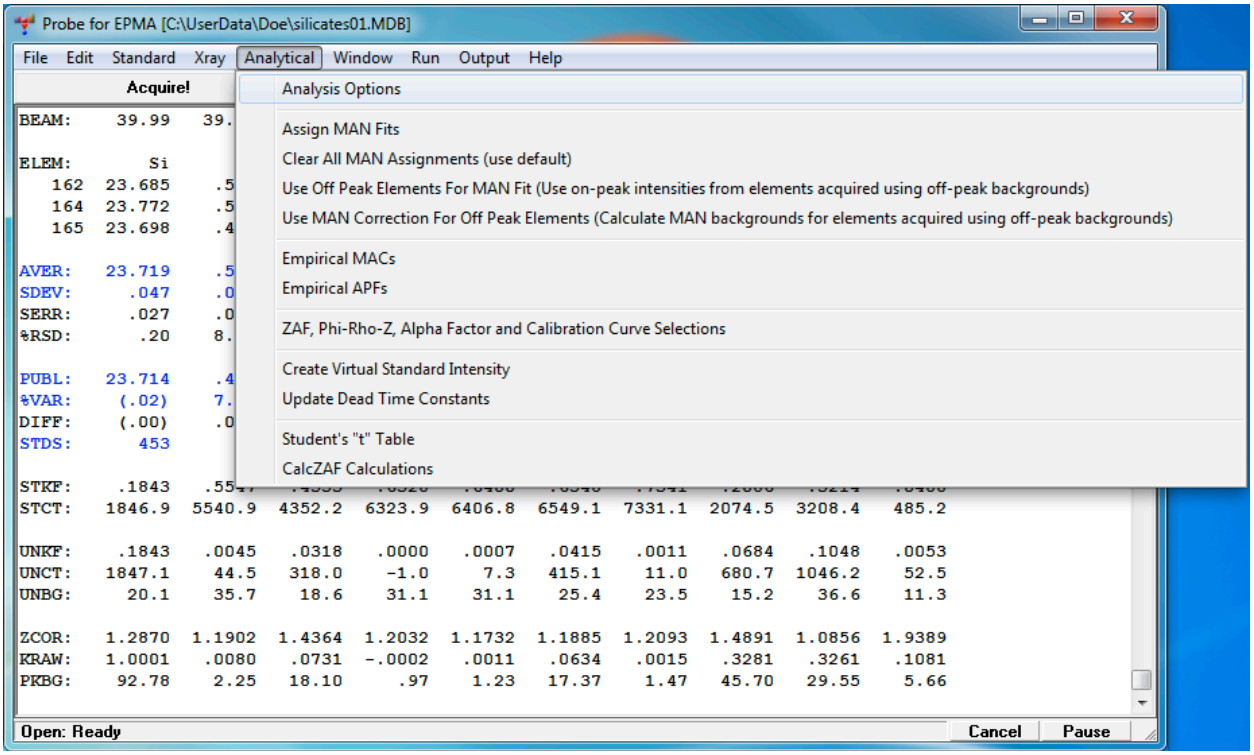


Repeat these editing steps for all of the other element interferences, resulting in the following **Standard and Interference Assignments** window.

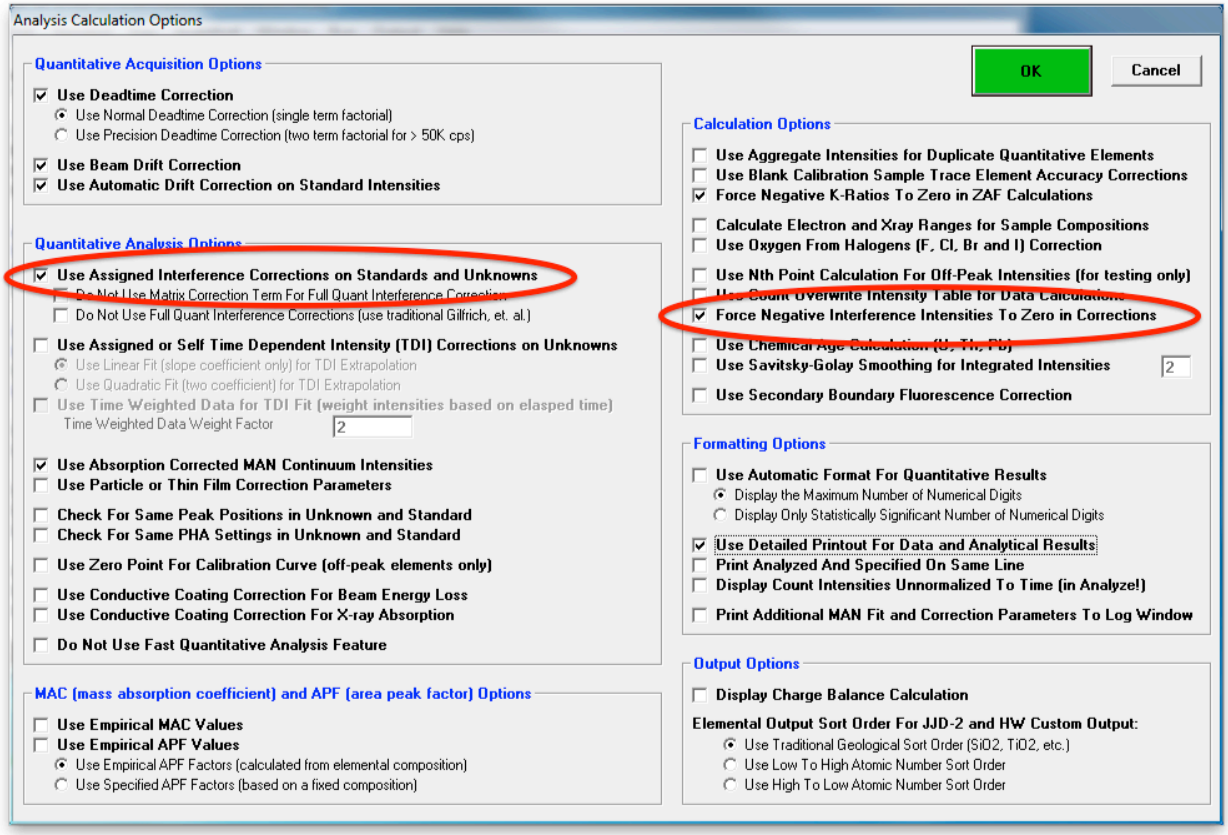


Click the **OK** button when finished returning to the **Analyze!** window.

Next, check the analysis options that are currently assigned. From the main PROBE FOR EPMA log window, select **Analytical** from the menu bar and click **Analysis Options** from the menu choices.



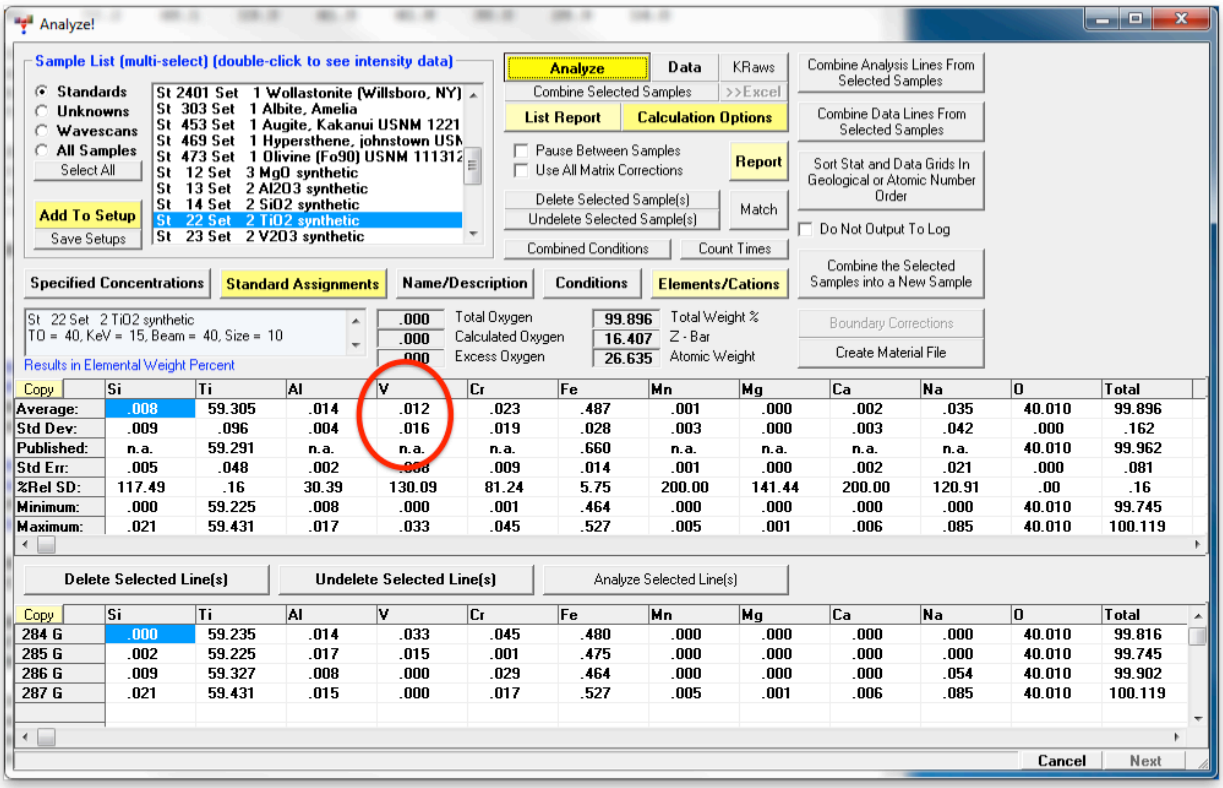
This opens the **Analysis Calculation Options** window. Check that the tick boxes for *Use Assigned Interference Corrections on Standards* and *Unknowns and Force Negative Interference Intensities To Zero in Corrections* are marked.



A range of other options can be switched on an off in this window which affect the calculation and output of the quantitative data. See the PROBE FOR EPMA User's Guide and Reference manual for details.

Click the **OK** button returning to the main log window.

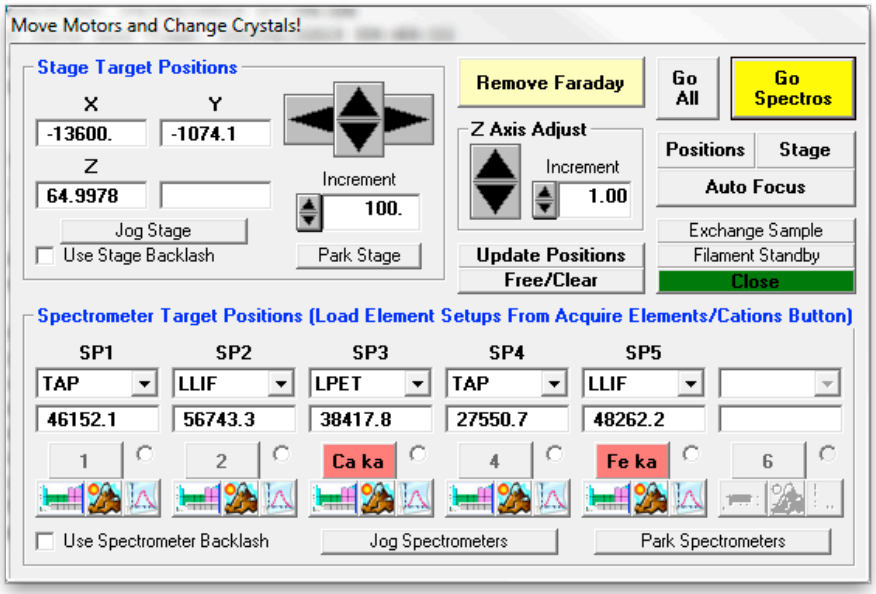
The user then reanalyzes the standards (**Analyze** button in the **Analyze!** window), utilizing the spectral interference correction routine. The results for the TiO_2 standard are dramatic; the apparent 0.6 wt% vanadium concentration has been replaced with an average 0.01 wt% content (which is below the detection limit).



The user is ready to move on to unknown samples.

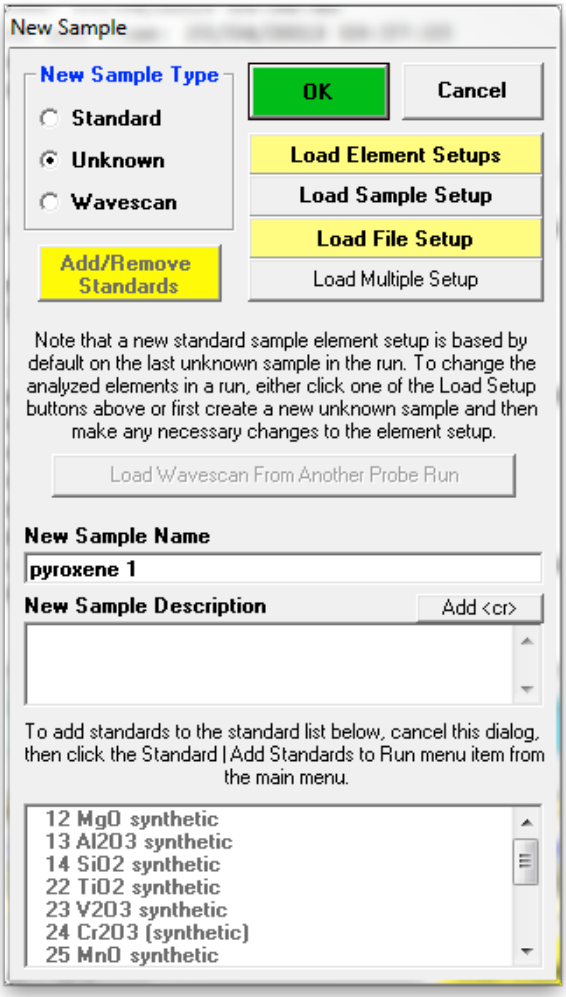
Manual Unknown Sample Data Collection and Analysis

To collect x-ray data on an unknown sample, bring forward the **Acquire!** dialog box and click the **Move** button. Enter the stage coordinates of the first unknown sample and click **Go All,** or use the cursor buttons in the *Stage Target Positions* section, or use the joystick to drive the stage, if available. Adjust the Z focus.



Click the **New Sample** button in the **Acquire!** window to activate the **New Sample** dialog box.

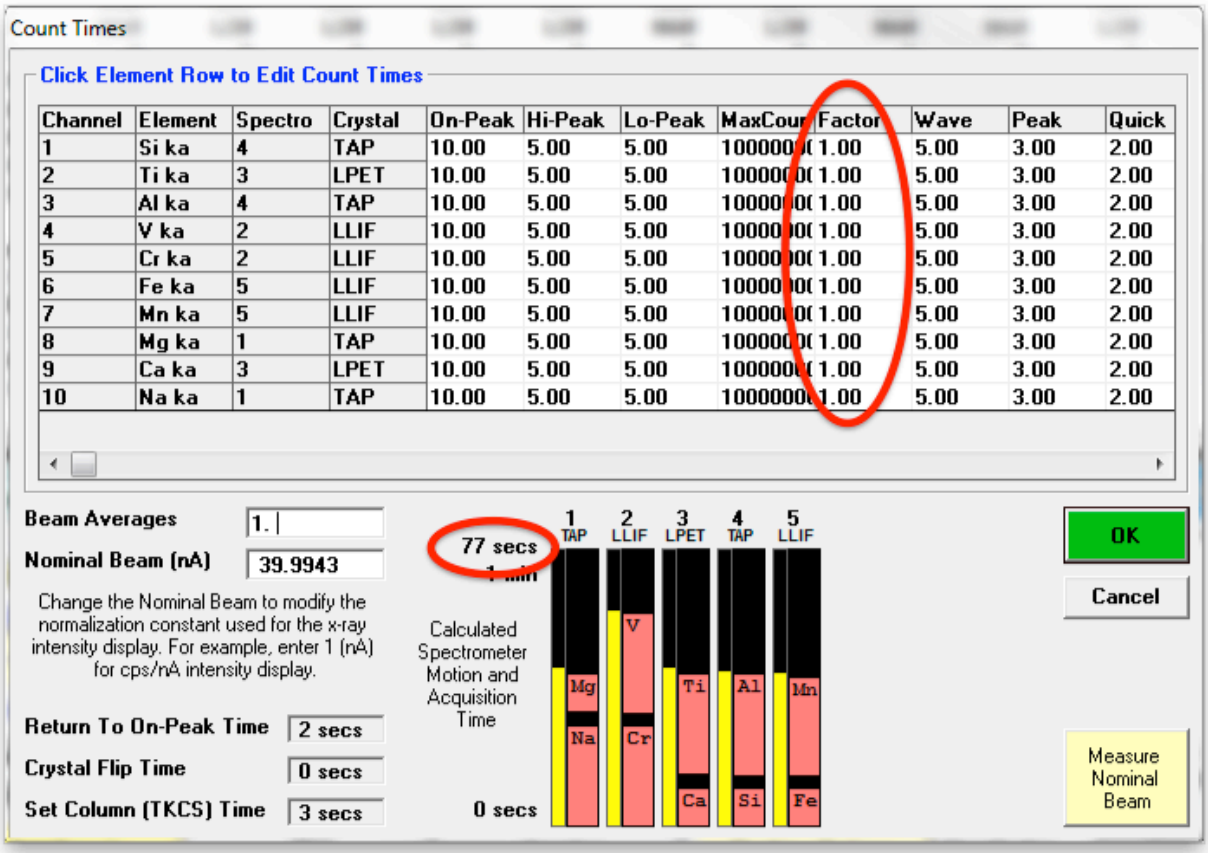
Check the *Unknown* button under *New Sample Type*. Enter an appropriate sample name and description into the *New Sample Name* and *New Sample Description* text boxes.



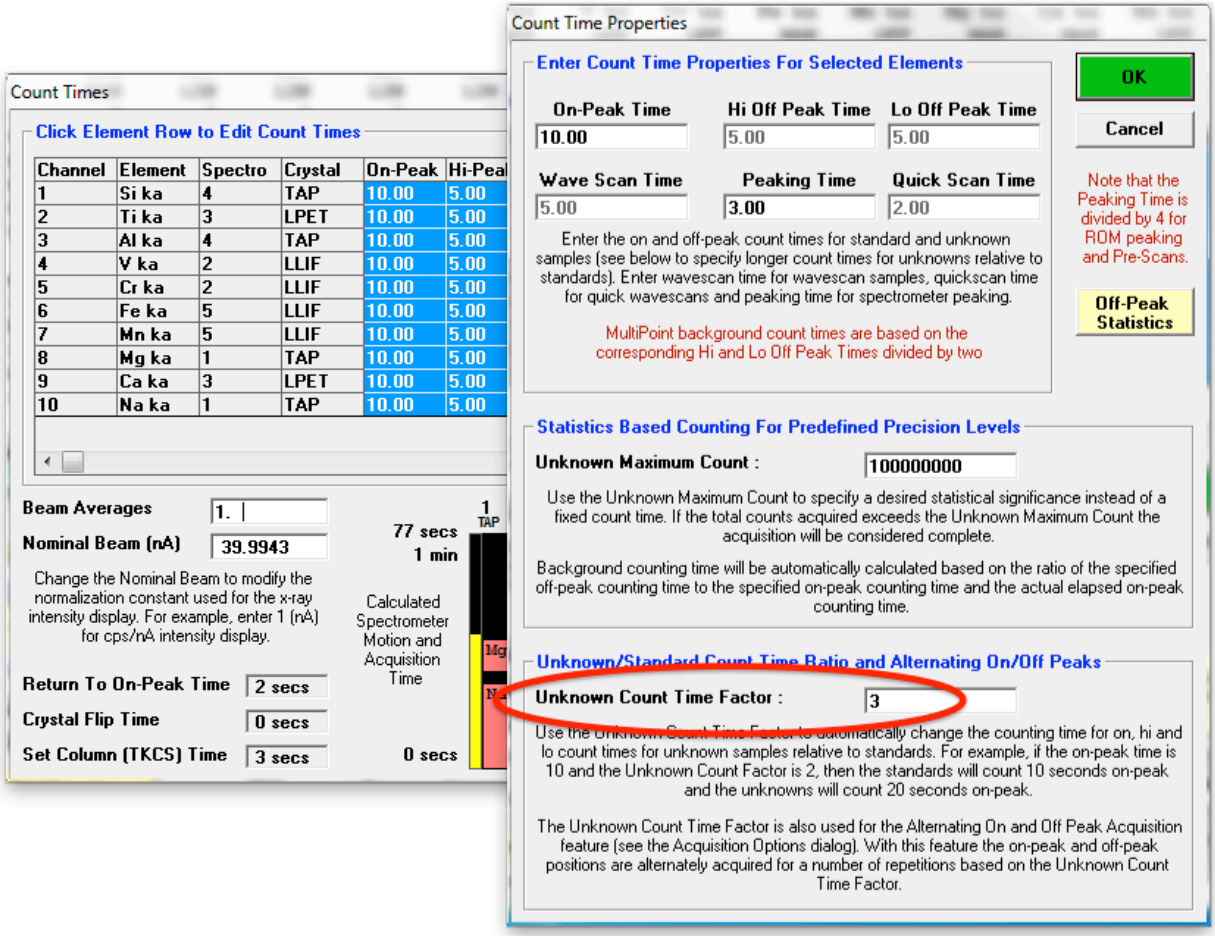
Click the **OK** button.

By default, all settings from the last measurement before will be loaded, in this case the standards acquisition using the pyroxene setup. Before starting the acquisition, any measurement conditions which don't require the acquisition of new standards can be changed, such as beam current and size in the **Analytical Conditions** window or count times, which can be increased to improve precision and detection limits for minor and trace elements.

Click on the **Count Times** button in the **Acquire!** window to launch the **Count Times** window. In this example, the *Unknown Count Time Factor (Factor)* in the **Count Times** window will be modified. This factor is a simple multiplication of the default count times (peak and backgrounds) on the standards. Currently this value, which is shown in the Factor column, is set to 1, leading to an estimated acquisition time of 77 seconds per point.

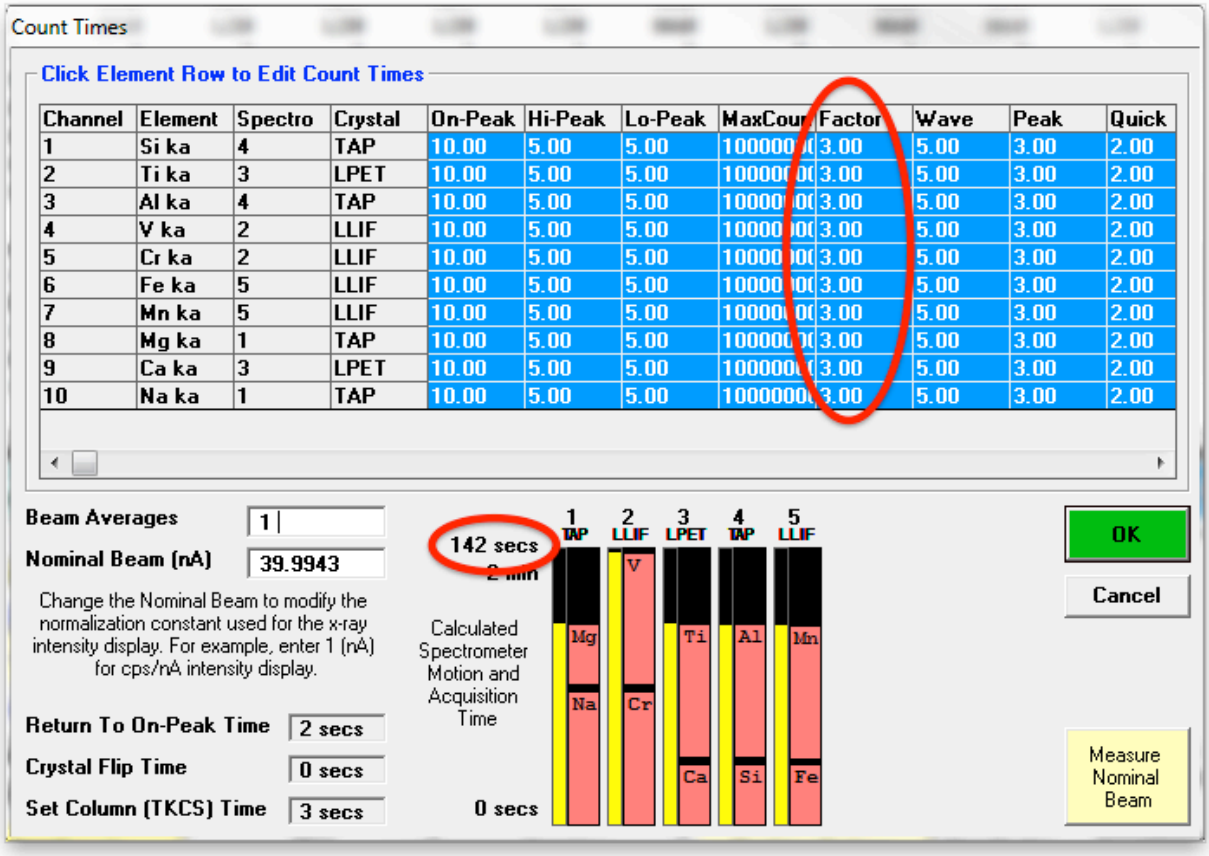


Left-click and drag the mouse across the rows in the table to select all elements. This opens the **Count Time Properties** window. In there, change the *Unknown Count Time Factor* from 1 to 3.



Click **OK** to return to the **Count Times** window.

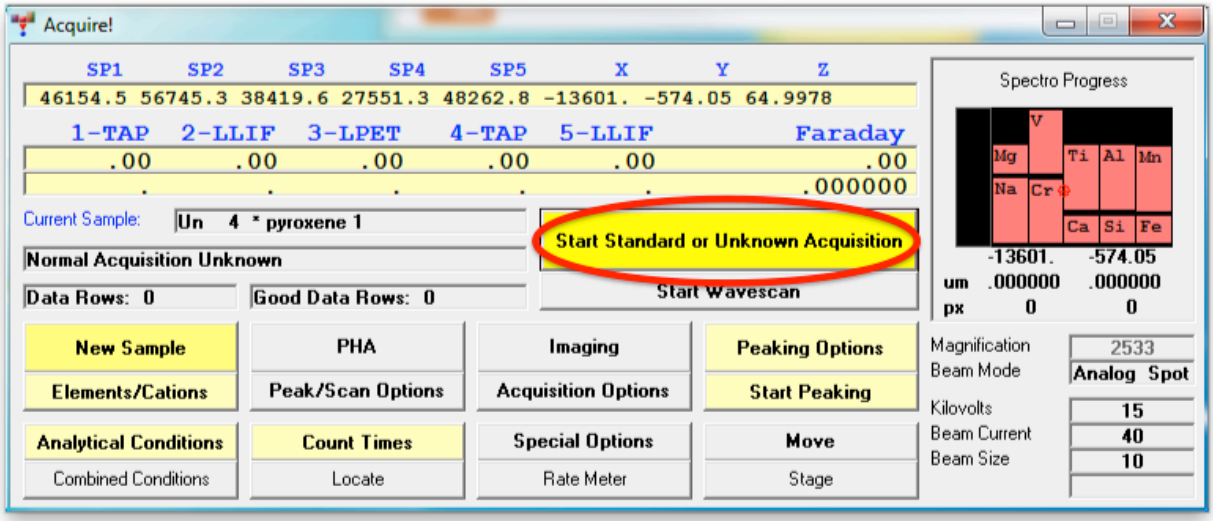
The **Count Times** window now shows a new estimate of 142 seconds for a measurement of unknown.



Further, the graphical spectrometer motion bars next to the time estimate indicate how efficient the usage of the spectrometers is. Spectrometer 2 needs more time compared to the other four spectrometers as off-peak backgrounds are acquired for both Cr and V. It would therefore be more efficient to either reduce counting times for Cr and/or V, or, as both are expected to be present only at minor to trace level in the unknown samples, extend counting times on the other spectrometers.

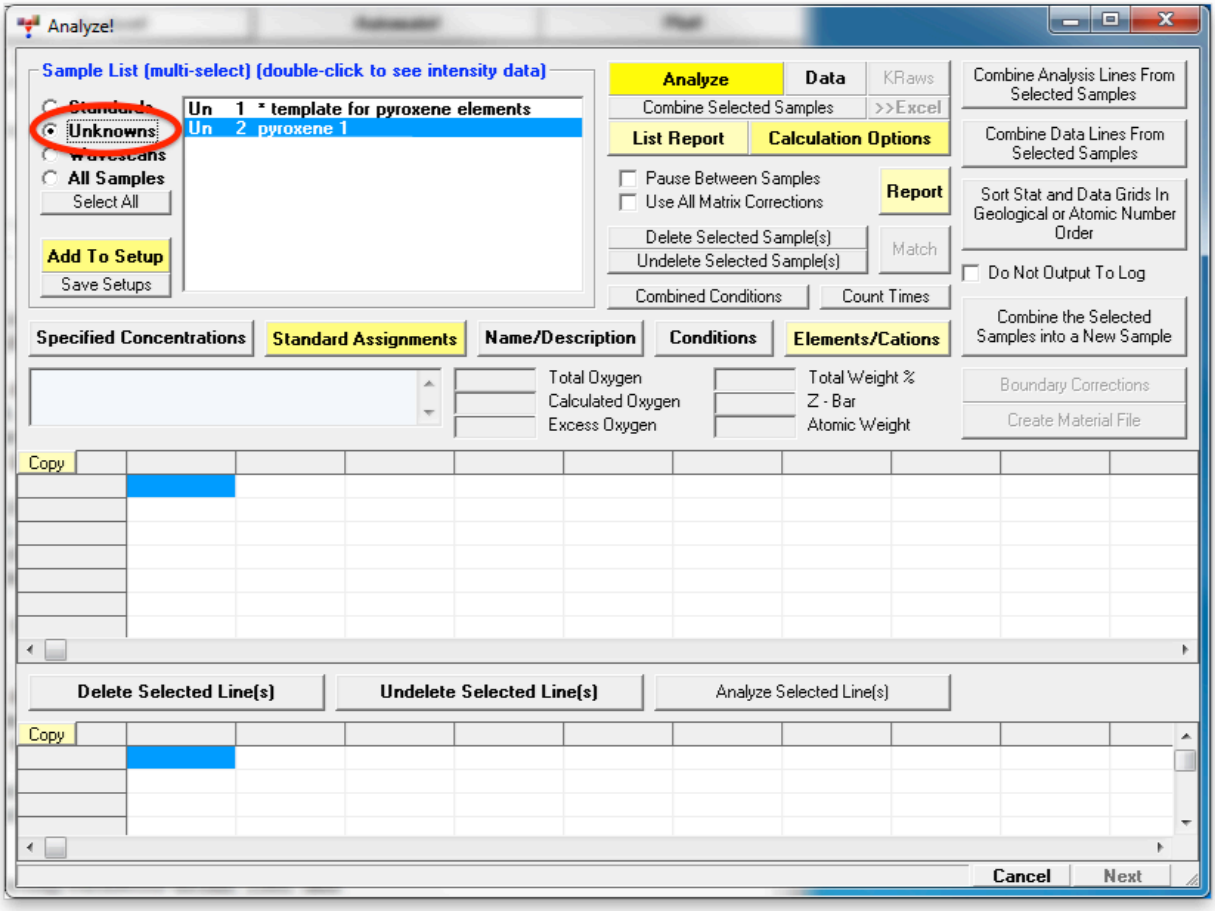
Adjust all count times as desired and click **OK** to return to the **Acquire!** window.

Click the **Start Standard or Unknown Acquisition** button in the **Acquire!** window to start the acquisition of the unknown.

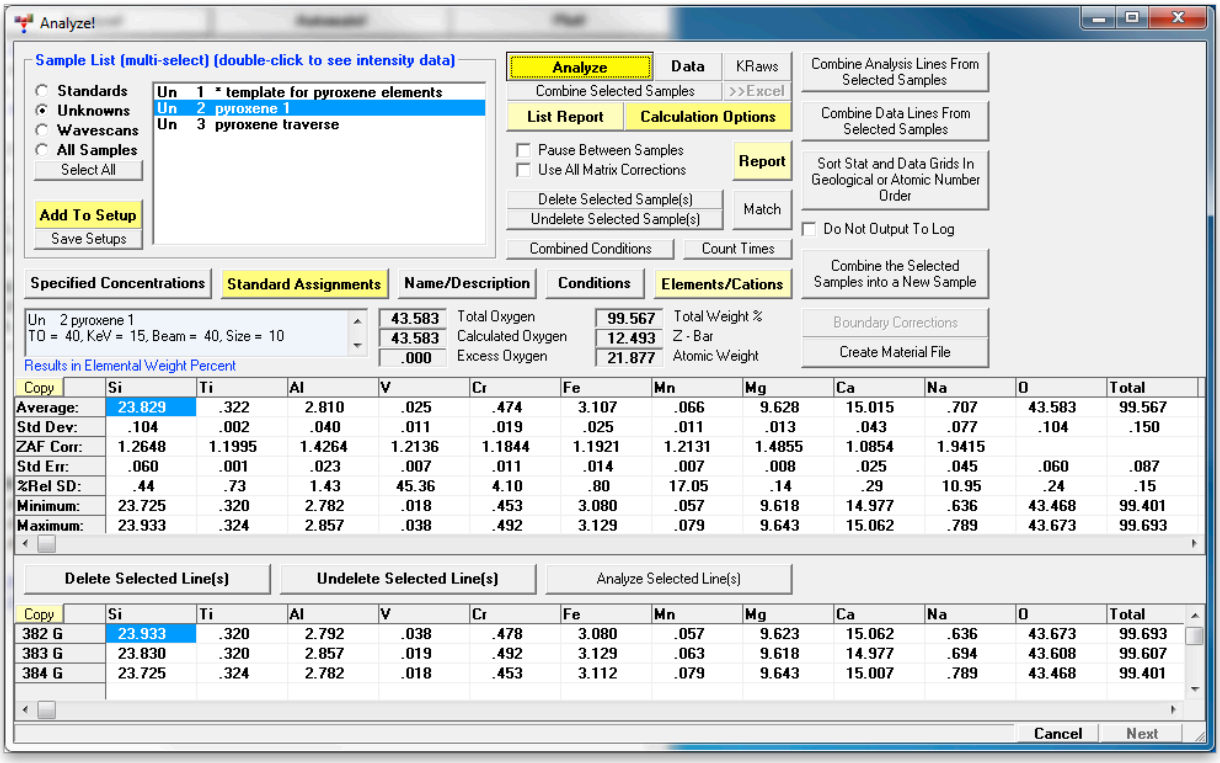


Again, progress can be monitored in the **Acquire!** window. After completion of the measurement, move the stage to a different position and click the **Start Standard or Unknown Acquisition** button again to start another measurement. Repeat this procedure a third time so that three random spots are acquired on the same pyroxene sample.

Next, reopen or bring forward the **Analyze!** window. Click the *Unknowns* button under the *Sample List* buttons and highlight (select) *Un 2 pyroxene 1*.

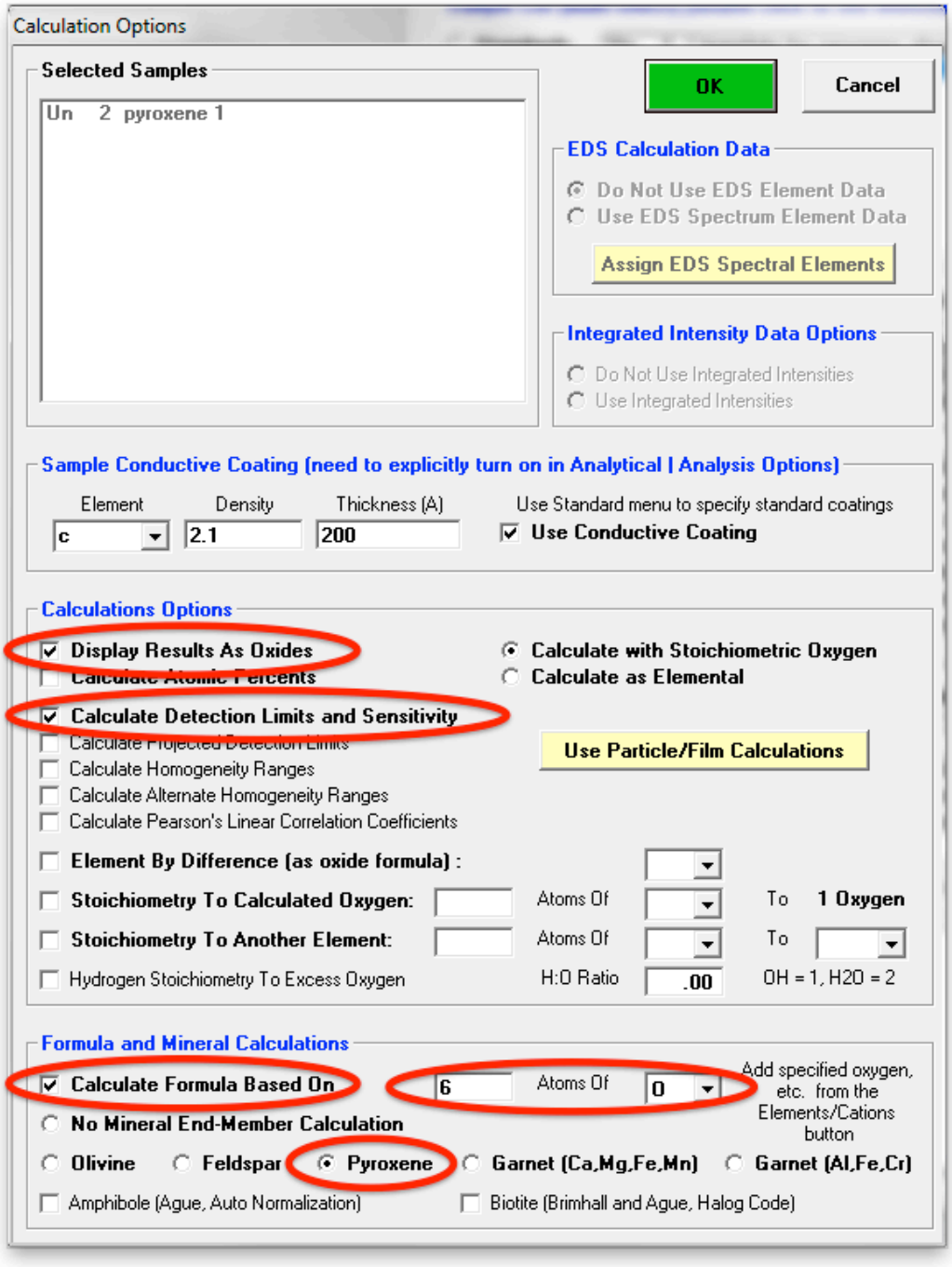


Click the **Analyze** button to calculate the results for these three points. The values are viewed below.



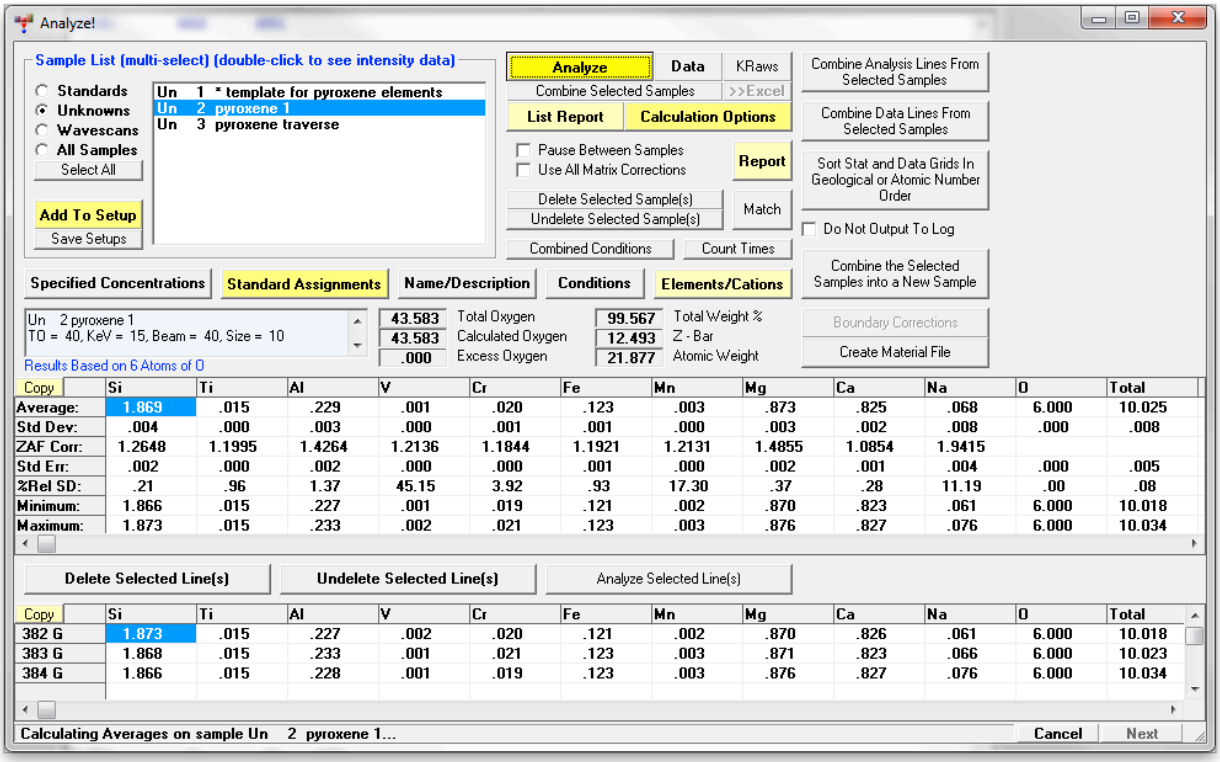
To set the software up to perform additional calculations, click the **Calculation Options** button.

This opens the **Calculation Options** dialog box. Make the following changes: under *Calculations Options* check the *Display Results as Oxides* and *Calculate Detection Limits and Sensitivity* boxes. Under *Formula and Mineral Calculations* check the *Calculate Formula Based On* box. Select *Pyroxene*, enter *6 Atoms of* in the text box, and select O (oxygen) from the drop down menu.



Click the **OK** button to return to the **Analyze!** window. Click the **Analyze** button again.

The table in the Analyze! window now shows the formula units for the pyroxene formula calculation as below.



Additional data is written to the log window, which can be stored to a text file and viewed as described before, or simply be highlighted with the mouse and copied to the system clipboard with the key combination <Crtl>+C. The contents of the log window are shown on the following pages.

The user may obtain a large amount of information besides elemental and oxide weight percent data. These expanded capabilities include formula and mineral end member calculations, an extended set of detection limit and statistics including homogeneity and analytical sensitivity. See the User's Guide and Reference documentation for calculation details.

Un   2 pyroxene 1
TakeOff = 40.0 KiloVolt = 15.0 Beam Current = 40.0 Beam Size =   10
(Magnification (analytical) =   2533),   Beam Mode = Analog Spot
(Magnification (default) =   2533, Magnification (imaging) =   2533)
Image Shift (X,Y):   -.50,   .00
Number of Data Lines: 3   Number of 'Good' Data Lines: 3
First/Last Date-Time: 04/25/2013 02:11:23 PM to 04/25/2013 02:20:08 PM

Average Total Oxygen: 43.583   Average Total Weight%:   99.567
Average Calculated Oxygen: 43.583   Average Atomic Number: 12.493
Average Excess Oxygen: .000   Average Atomic Weight: 21.877
Average ZAF Iteration: 3.00   Average Quant Iterate: 3.00

Oxygen Calculated by Cation Stoichiometry and Included in the Matrix Correction

Un   2 pyroxene 1, Results in Elemental Weight Percents

SPEC:	O
TYPE:	CALC
AVER:	43.583
SDEV:	.104

ELEM:	Si	Ti	Al	V	Cr	Fe	Mn	Mg	Ca	Na
BGDS:	MAN	LIN	LIN	LIN	LIN	MAN	LIN	MAN	MAN	LIN
TIME:	30.00	30.00	30.00	30.00	30.00	30.00	30.00	30.00	30.00	30.00
BEAM:	40.11	40.11	40.11	40.11	40.11	40.11	40.11	40.11	40.11	40.11

ELEM:	Si	Ti	Al	V	Cr	Fe	Mn	Mg	Ca	Na	SUM
382	23.933	.320	2.792	.038	.478	3.080	.057	9.623	15.062	.636	99.693
383	23.830	.320	2.857	.019	.492	3.129	.063	9.618	14.977	.694	99.607
384	23.725	.324	2.782	.018	.453	3.112	.079	9.643	15.007	.789	99.401
AVER:	23.829	.322	2.810	.025	.474	3.107	.066	9.628	15.015	.707	99.567
SDEV:	.104	.002	.040	.011	.019	.025	.011	.013	.043	.077	.150
SERR:	.060	.001	.023	.007	.011	.014	.007	.008	.025	.045	
%RSD:	.44	.73	1.43	45.36	4.10	.80	17.05	.14	.29	10.95	
STDS:	602	539	609	584	631	524	509	607	602	651	
STKF:	.1884	.5489	.1277	1.0000	.3856	.6539	.1612	.2085	.1386	.0392	
STCT:	387.65	1658.01	272.03	358.23	169.81	460.67	107.33	336.16	336.12	43.93	
UNKF:	.1884	.0027	.0197	.0002	.0040	.0261	.0005	.0648	.1383	.0036	
UNCT:	387.70	8.10	41.98	.07	1.76	18.36	.36	104.50	335.38	4.08	
UNBG:	1.92	3.15	1.08	.30	.42	.86	.64	1.12	2.39	.47	
ZCOR:	1.2648	1.1995	1.4264	1.2136	1.1844	1.1921	1.2131	1.4855	1.0854	1.9415	
KRAW:	1.0001	.0049	.1543	.0002	.0104	.0399	.0034	.3108	.9978	.0928	
PKBG:	202.96	3.57	39.87	1.25	5.21	22.36	1.57	94.93	141.17	9.63	
INT%:	----	----	----	-6.53	----	----	-1.31	----	----	----	

Un   2 pyroxene 1, Results in Oxide Weight Percents

SPEC:	O
TYPE:	CALC
AVER:	.000
SDEV:	.000

ELEM:	SiO2	TiO2	Al2O3	V2O3	Cr2O3	FeO	MnO	MgO	CaO	Na2O	SUM
382	51.201	.534	5.276	.056	.699	3.963	.073	15.958	21.075	.857	99.693
383	50.980	.534	5.398	.028	.719	4.026	.081	15.949	20.956	.936	99.607
384	50.756	.541	5.257	.026	.663	4.003	.102	15.992	20.998	1.064	99.401
AVER:	50.979	.536	5.310	.037	.693	3.997	.085	15.966	21.009	.953	99.567
SDEV:	.222	.004	.076	.017	.028	.032	.015	.022	.060	.104	.150
SERR:	.128	.002	.044	.010	.016	.018	.008	.013	.035	.060	
%RSD:	.44	.73	1.43	45.36	4.10	.80	17.05	.14	.29	10.95	
STDS:	602	539	609	584	631	524	509	607	602	651	

Un   2 pyroxene 1, Results Based on 6 Atoms of O

SPEC: O
TYPE: CALC

AVER: 6.000
SDEV: .000

ELEM:	Si	Ti	Al	V	Cr	Fe	Mn	Mg	Ca	Na	SUM
382	1.873	.015	.227	.002	.020	.121	.002	.870	.826	.061	10.018
383	1.868	.015	.233	.001	.021	.123	.003	.871	.823	.066	10.023
384	1.866	.015	.228	.001	.019	.123	.003	.876	.827	.076	10.034
AVER:	1.869	.015	.229	.001	.020	.123	.003	.873	.825	.068	10.025
SDEV:	.004	.000	.003	.000	.001	.001	.000	.003	.002	.008	.008
SERR:	.002	.000	.002	.000	.000	.001	.000	.002	.001	.004	
%RSD:	.21	.96	1.37	45.15	3.92	.93	17.30	.37	.28	11.19	

Pyroxene Mineral End-Member Calculations

	Wo	En	Fs
382	45.4	47.9	6.7
383	45.3	47.9	6.8
384	45.3	48.0	6.7
AVER:	45.3	47.9	6.7
SDEV:	.1	.1	.1

Detection limit at 99 % Confidence in Elemental Weight Percent (Single Line):

ELEM:	Si	Ti	Al	V	Cr	Fe	Mn	Mg	Ca	Na
382	.009	.008	.007	.019	.018	.017	.016	.010	.007	.013
383	.009	.007	.008	.021	.019	.017	.015	.010	.007	.012
384	.009	.007	.007	.019	.018	.017	.015	.011	.007	.013
AVER:	.009	.007	.007	.020	.018	.017	.015	.010	.007	.013
SDEV:	.000	.000	.000	.001	.000	.000	.000	.000	.000	.001
SERR:	.000	.000	.000	.001	.000	.000	.000	.000	.000	.000

Percent Analytical Relative Error (One Sigma, Single Line):

ELEM:	Si	Ti	Al	V	Cr	Fe	Mn	Mg	Ca	Na
382	.2	1.7	.6	25.9	3.2	.9	14.6	.3	.2	2.1
383	.2	1.7	.6	53.1	3.2	.9	12.9	.3	.2	1.9
384	.2	1.6	.6	52.6	3.3	.9	10.7	.3	.2	1.8
AVER:	.2	1.7	.6	43.9	3.2	.9	12.7	.3	.2	1.9
SDEV:	.0	.0	.0	15.6	.1	.0	2.0	.0	.0	.1
SERR:	.0	.0	.0	9.0	.0	.0	1.1	.0	.0	.1

Detection Limit (t-test) in Elemental Weight Percent (Average of Sample):

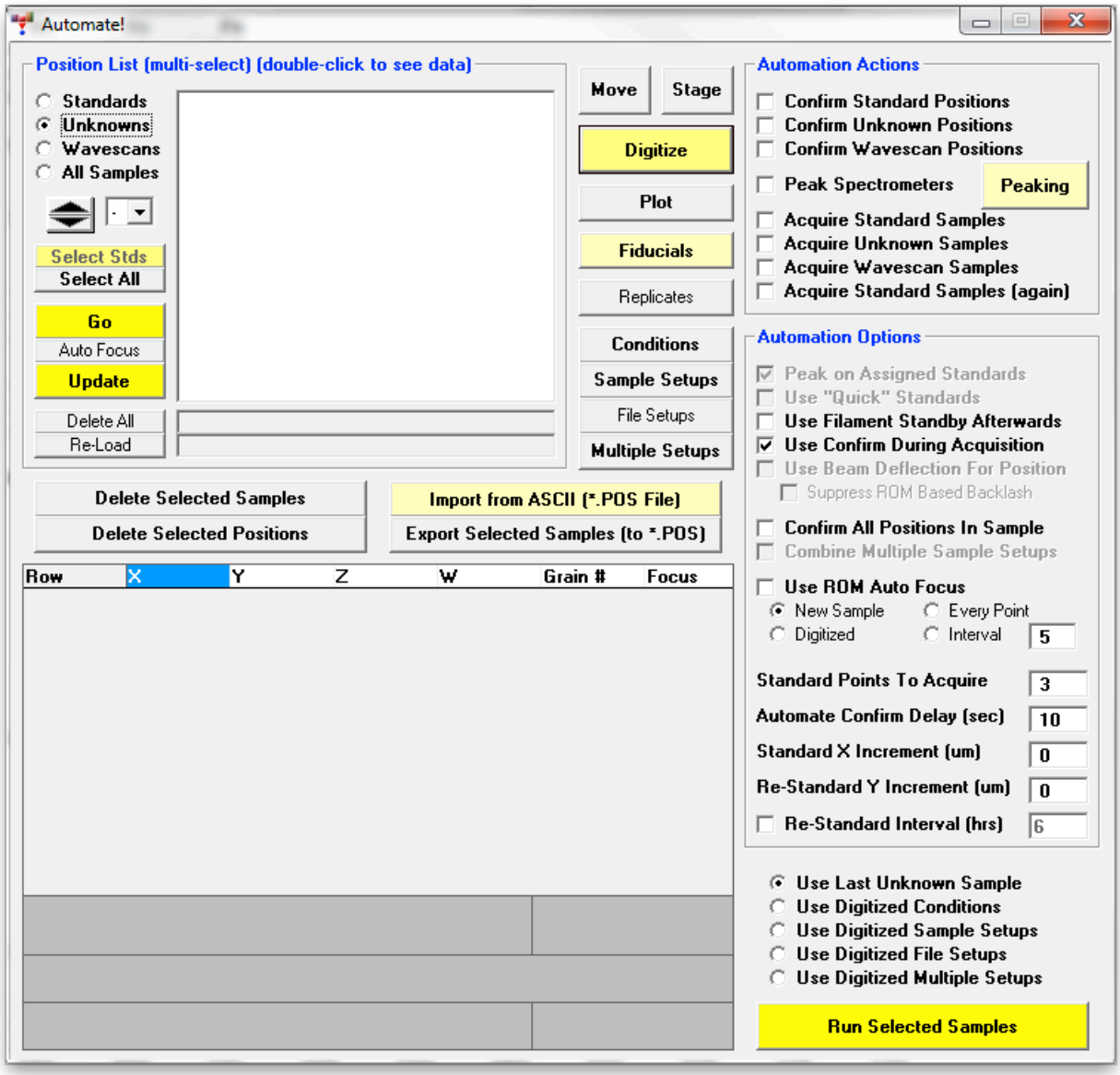
ELEM:	Si	Ti	Al	V	Cr	Fe	Mn	Mg	Ca	Na
60ci	---	.001	---	.008	.016	---	.009	---	---	.062
80ci	---	.002	---	.014	.028	---	.015	---	---	.111
90ci	---	.004	---	.022	.044	---	.024	---	---	.171
95ci	---	.005	---	.033	.065	---	.035	---	---	.252
99ci	---	.012	---	.076	.149	---	.080	---	---	.582

Analytical Sensitivity (t-test) in Elemental Weight Percent (Average of Sample):

ELEM:	Si	Ti	Al	V	Cr	Fe	Mn	Mg	Ca	Na
60ci	.099	.001	.037	.002	.014	.022	.003	.010	.038	.061
80ci	.177	.002	.065	.003	.025	.038	.006	.018	.068	.109
90ci	.274	.003	.101	.005	.039	.059	.009	.028	.105	.169
95ci	.403	.004	.149	.008	.058	.088	.013	.042	.155	.249
99ci	.931	.010	.343	.018	.133	.202	.030	.096	.357	.575

Digitized Sample Data Collection and Analysis

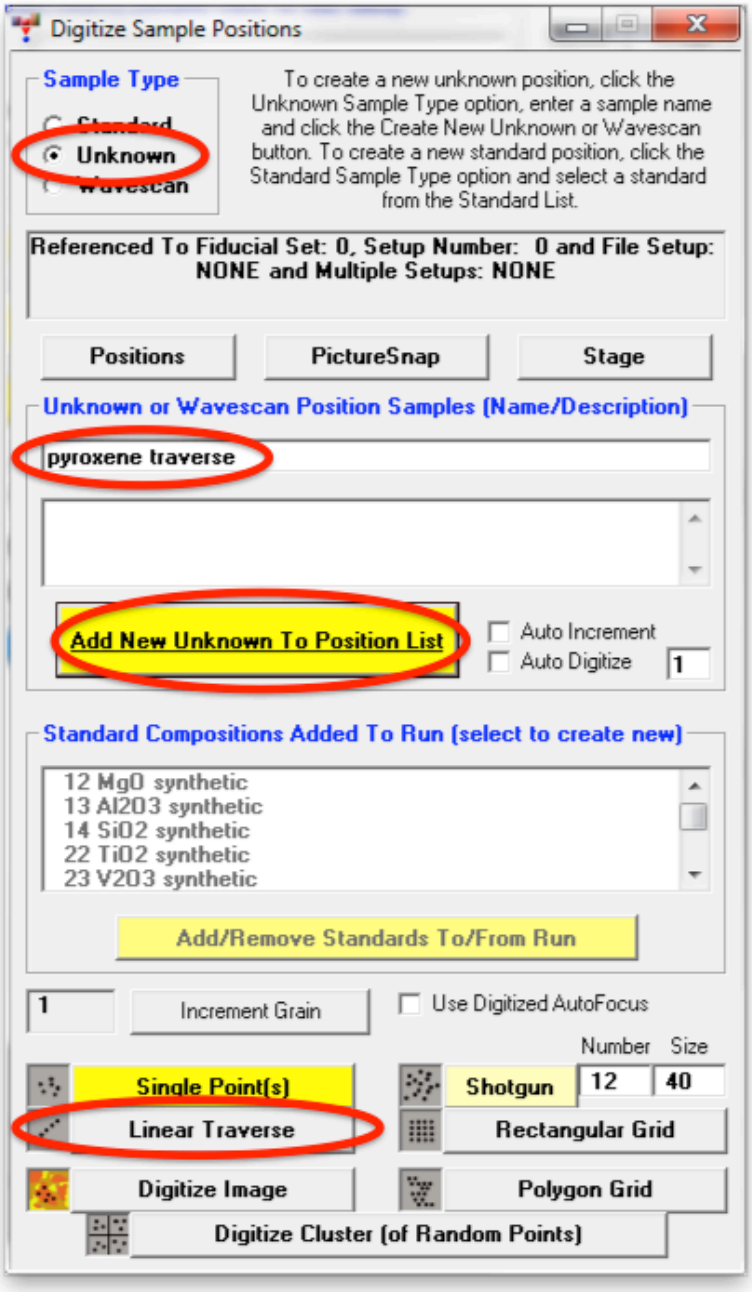
Acquisition of data on unknown samples can also be automated. As an example the user will perform a digitized traverse across an unknown pyroxene grain. The user can digitize standards, unknowns or wavescan positions based on random points, linear traverse or rectangular or polygon gridded areas. Check that the *Unknowns* button is clicked in the **Automate!** window.



Click the **Digitize** button in the **Automate!** window.

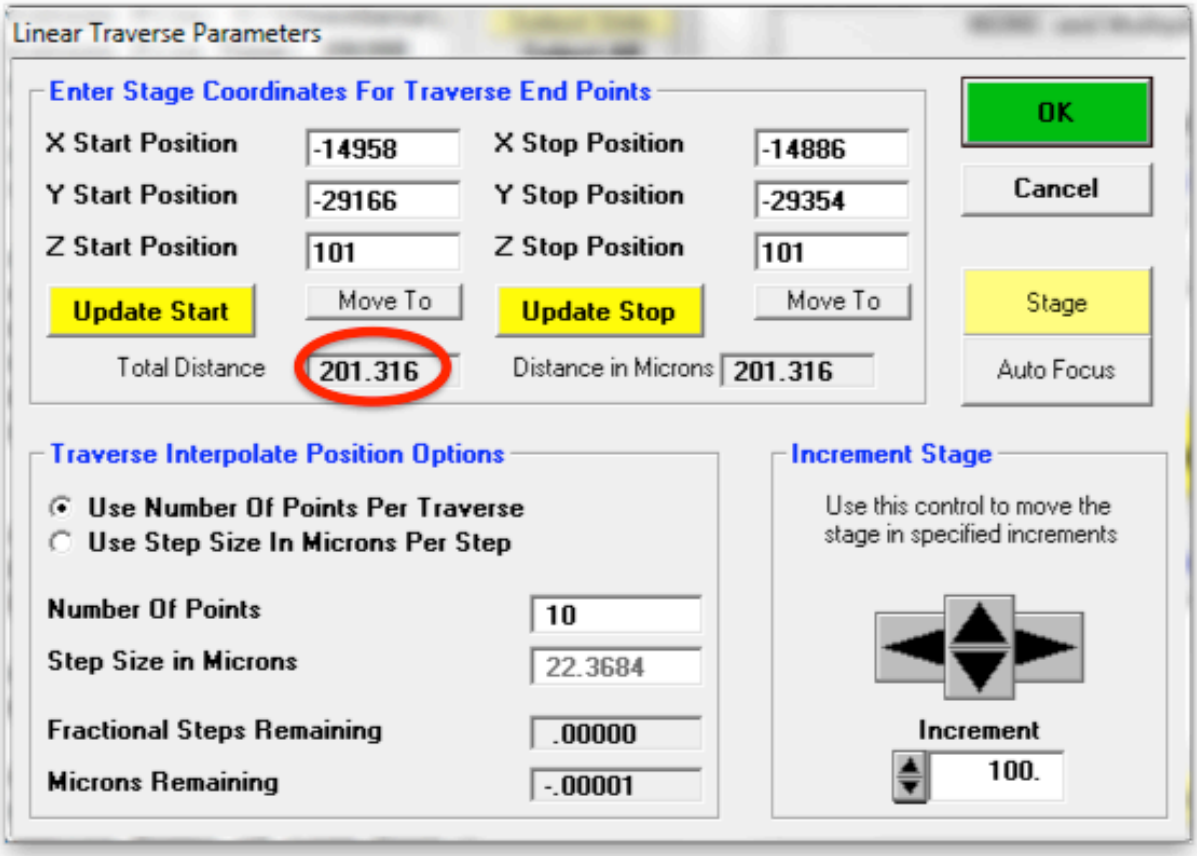
This opens the **Digitize Sample Positions** dialog box.

To create an unknown digitized sample, click *Unknown* under *Sample Type* and enter a sample name in the *Unknown or Wavescan Position Samples* text box. Next, click the **Create New Unknown or Wavescan** button. The unknown sample will now appear in the *Position List* list box of the **Automate!** window. Finally, click the **Linear Traverse** button to create a traverse of digitized points.

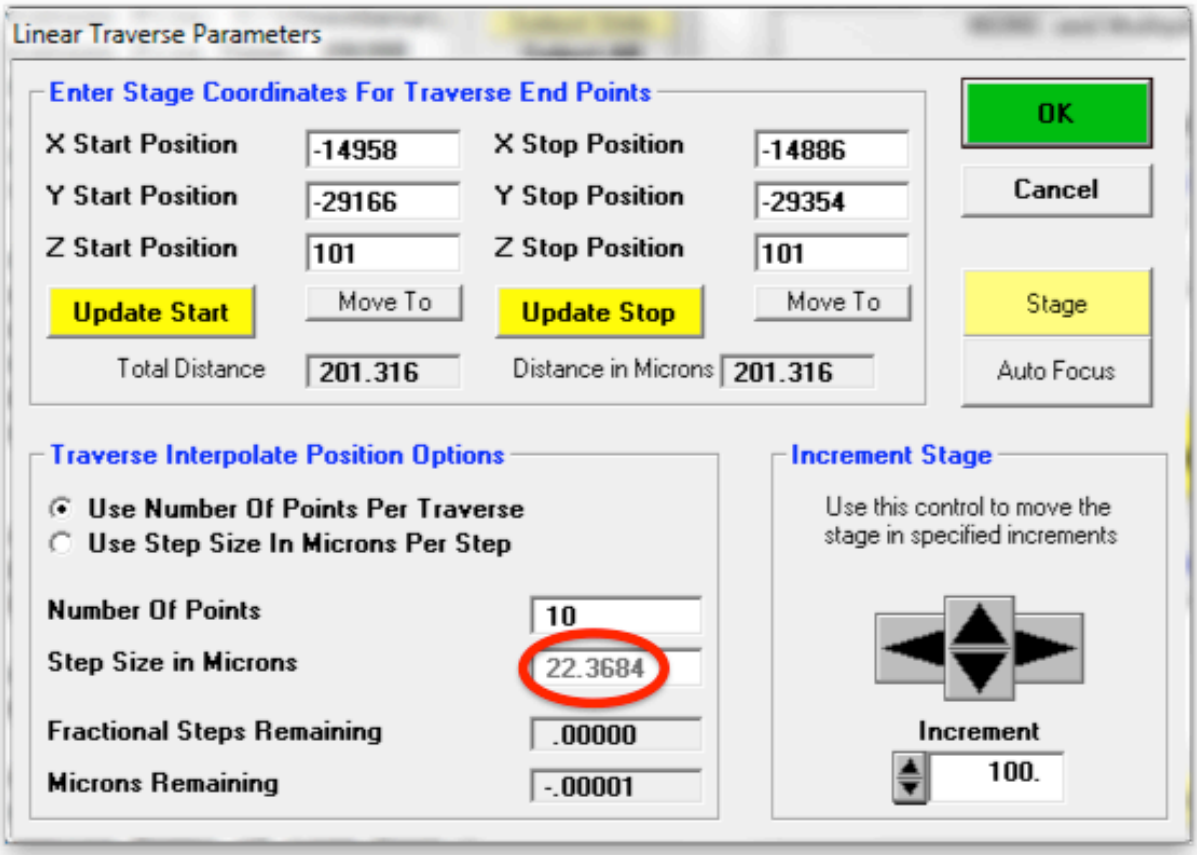


Note: Other available digitization options include rectangular and polygon grids as well as clusters of random points. The user can also digitize positions from an image acquired on the microprobe, or load a previously scanned image file for stage position calibration and then digitize samples on that image via the **PictureSnap!** menu.

The **Linear Traverse Parameters** dialog box opens. Move to the start position of the linear traverse, and click the **Update Start** button. Move to the stop position and click the **Update Stop** button. The *Total Distance* is displayed.

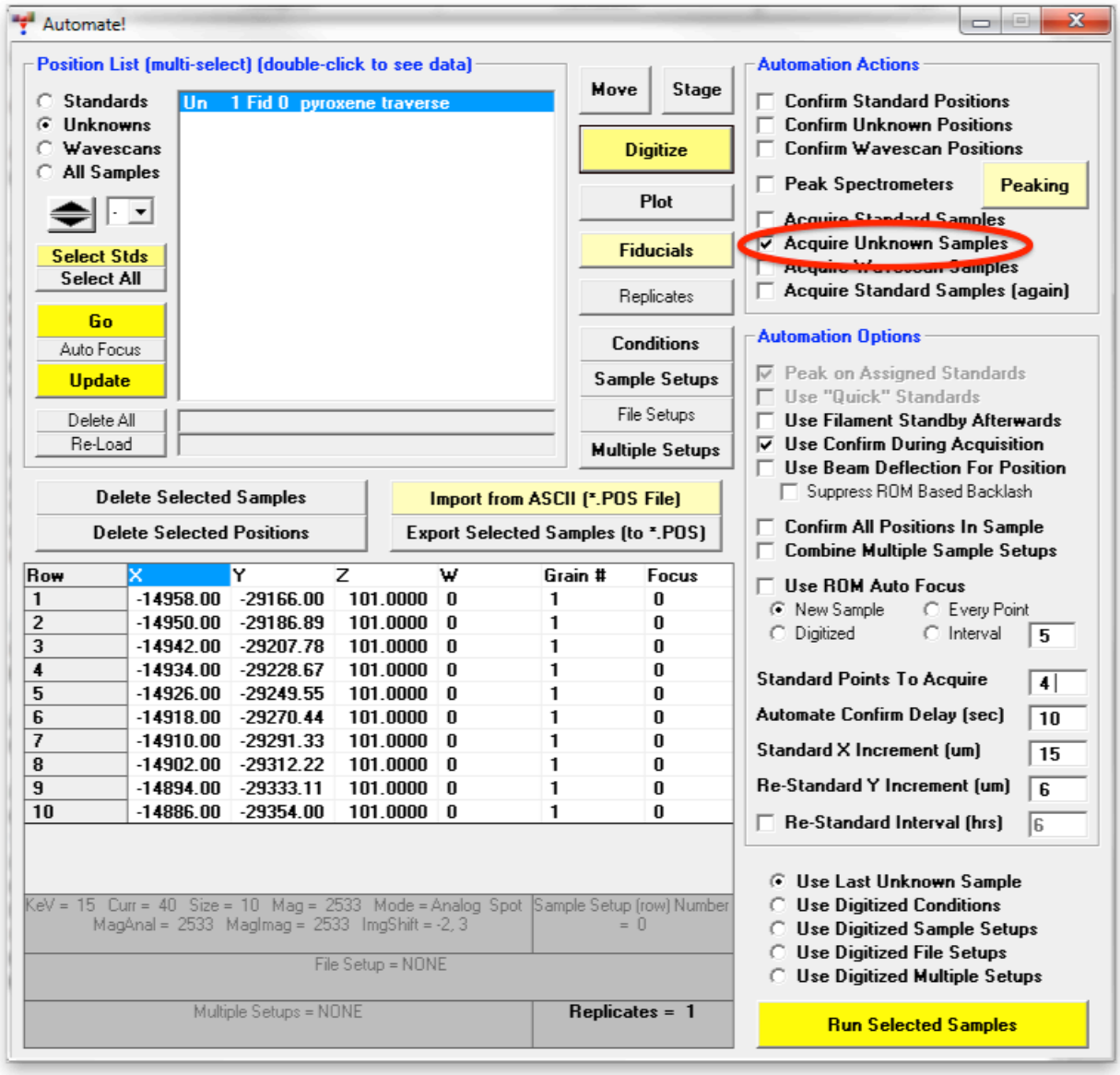


Select *Use Number of Points Per Traverse* or and type 10 into the *Number of Points* text box. The *Step Size in Microns* is calculated.



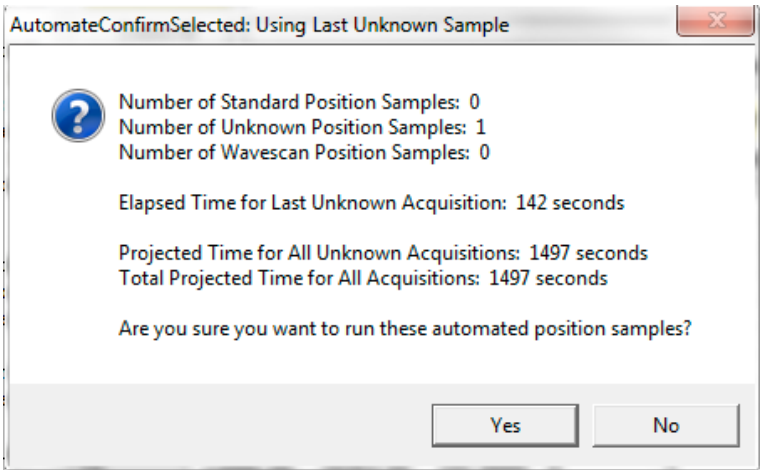
Click the **OK** button to add the 10-point traverse to the position list and return to the **Automate!** window.

Now all of the calculated analysis positions have been digitized and listed. Under *Automation Actions* click the *Acquire Unknown Samples* button.

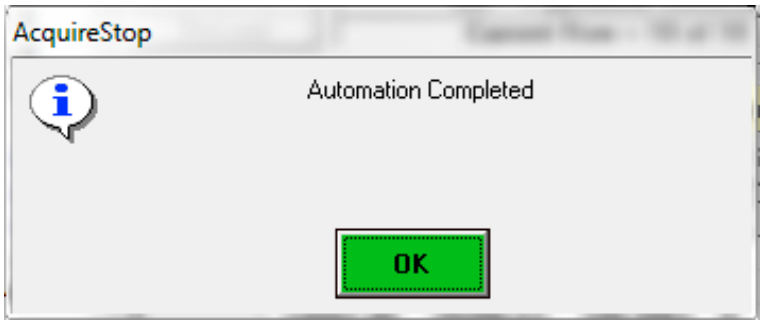


Click **Run Selected Samples** button to initiate the traverse.

The **AutomateConfirmSelected** window opens. Click **Yes**.

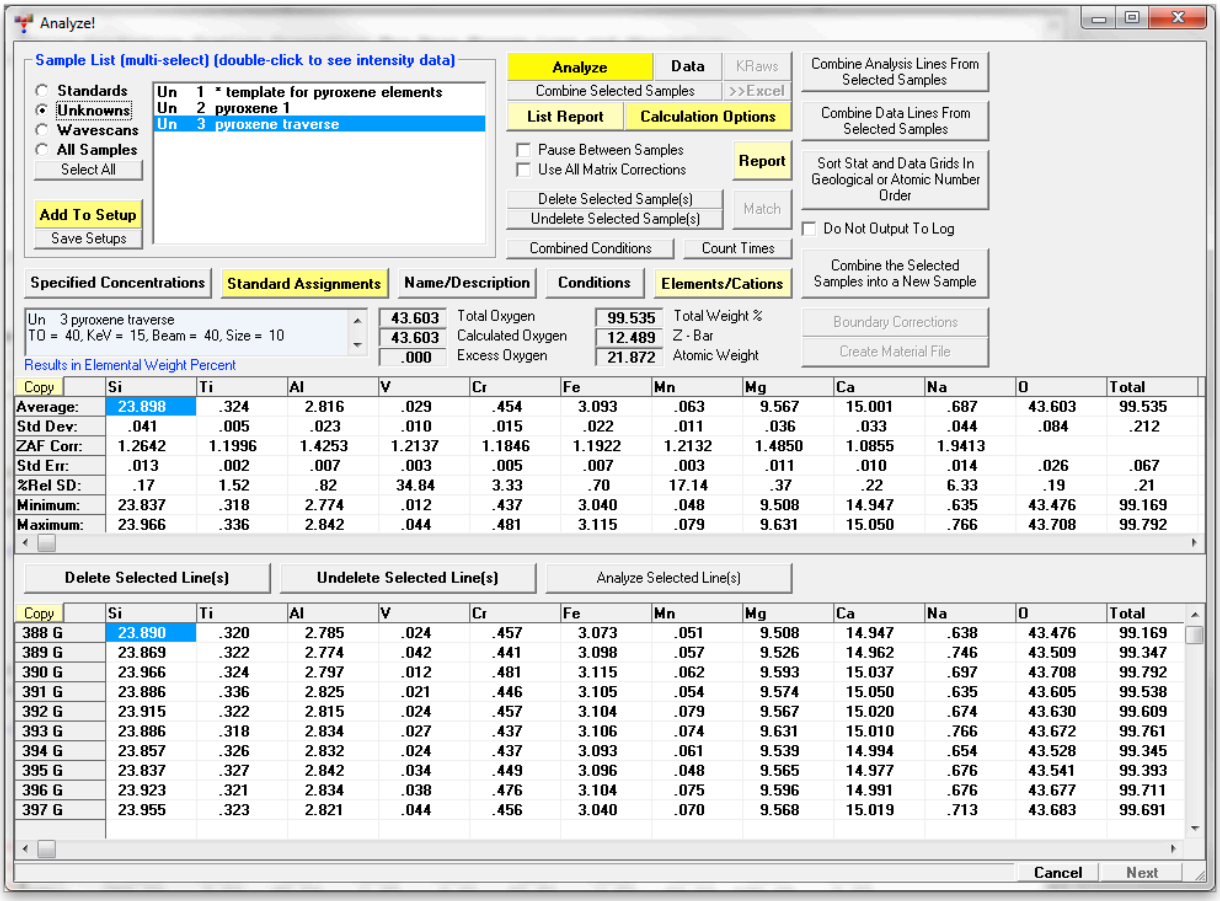


When the traverse is completed the familiar **AcquireStop** window appears.



Click the **OK** button returning the user to the **Automate!** dialog box.

To analyze the data obtained from the traverse, again open the **Analyze!** window, select the *Un 3 pyroxene traverse* sample in the *Sample List*, and process the data in a similar way as before.

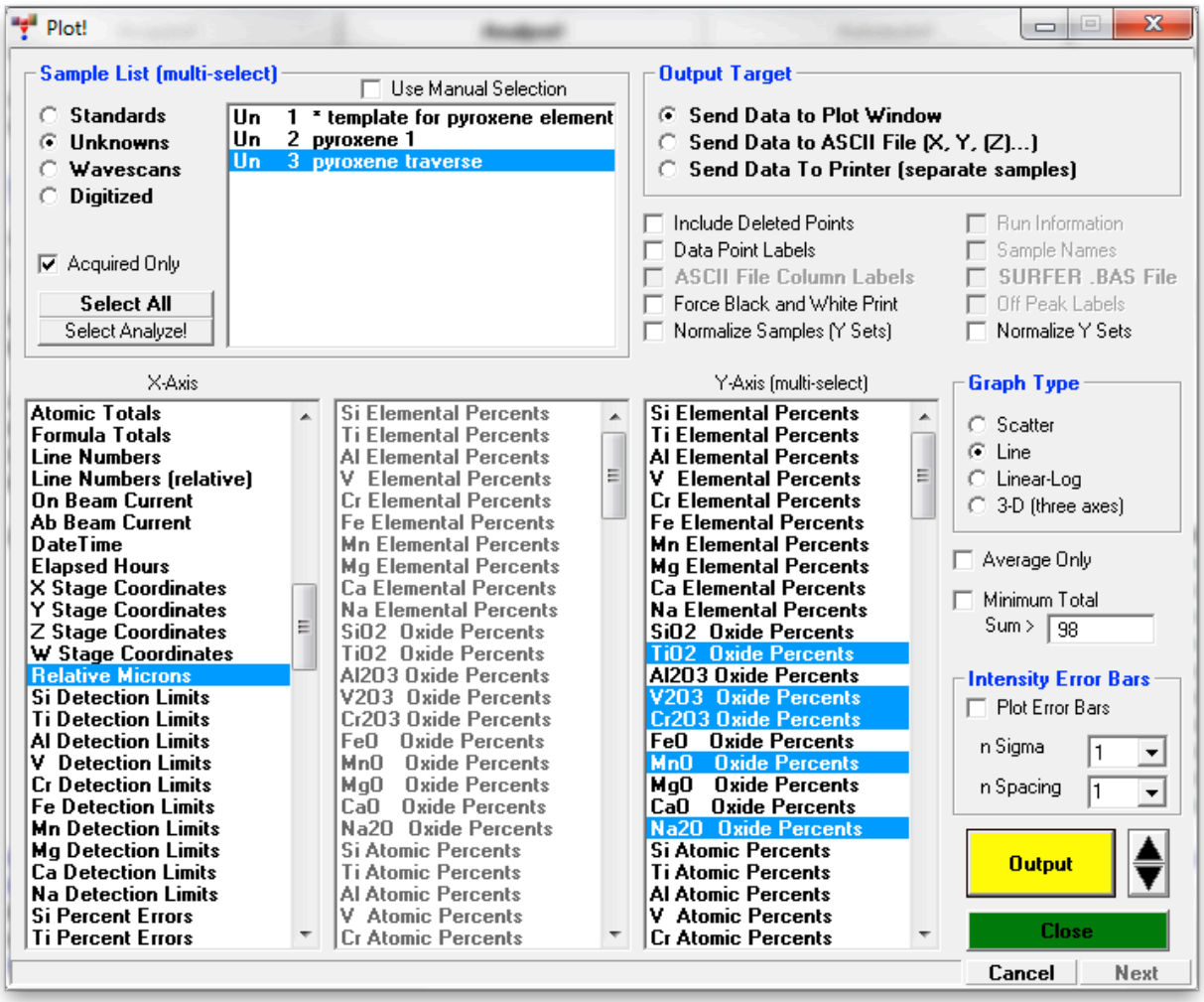


The next two sections will illustrate more powerful options to plot and export analysis data.

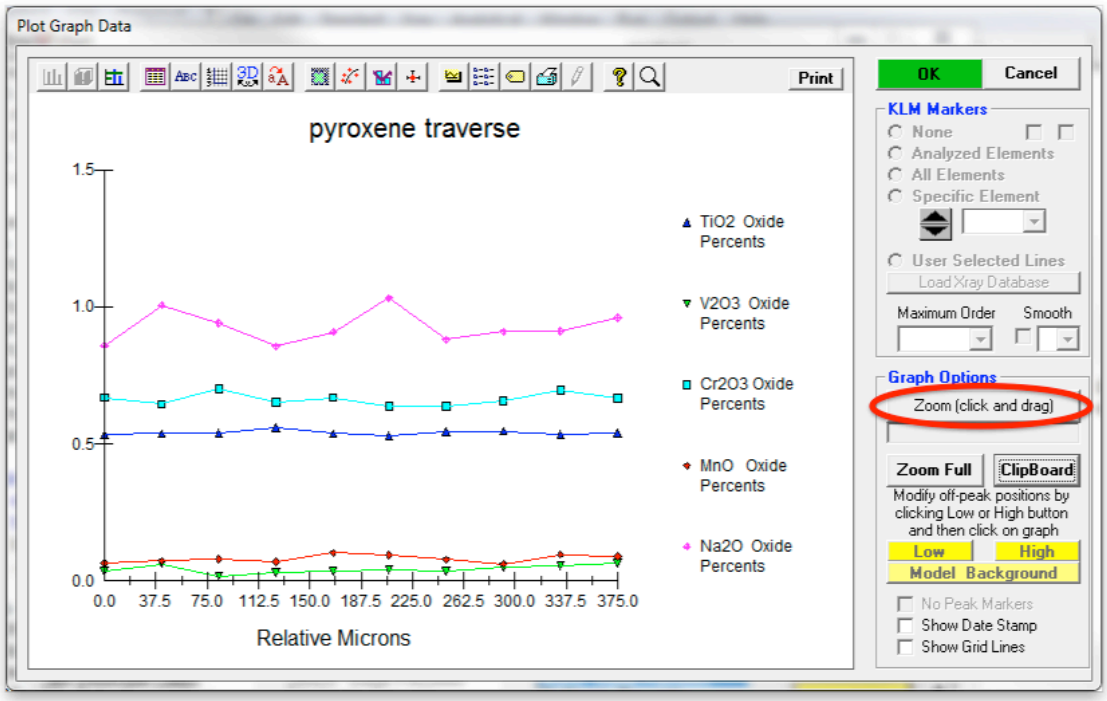
Plotting Analysis Data

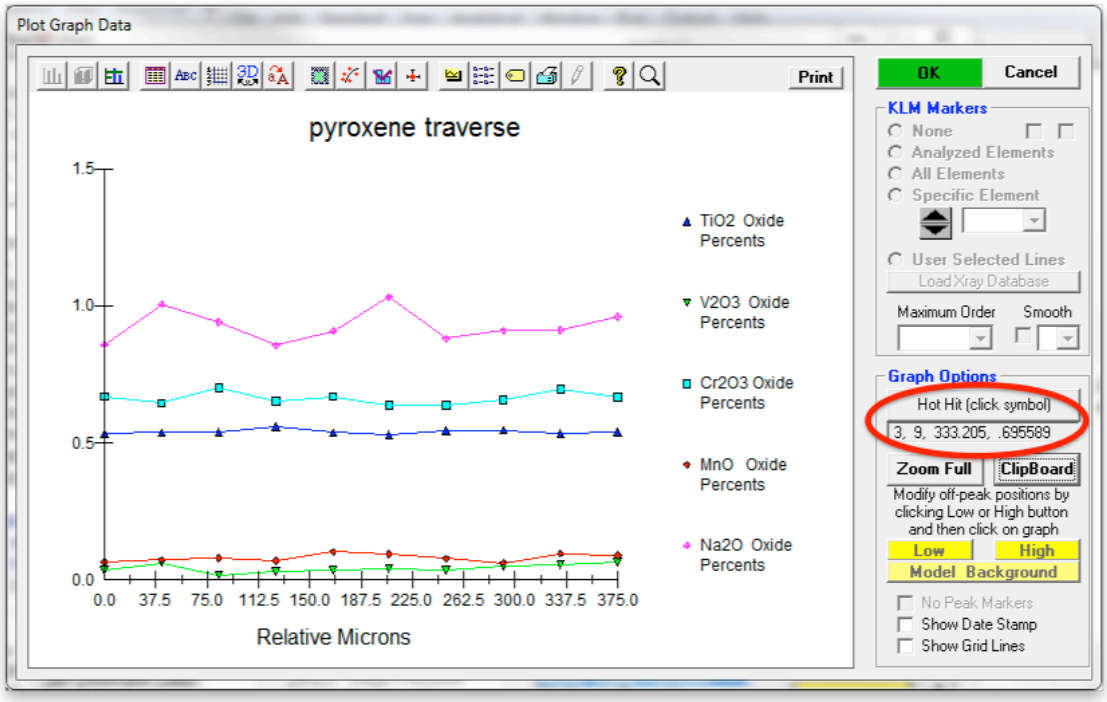
The user may wish to examine the traverse data in a graphical presentation. Click the **Plot!** button in the main PROBE FOR EPMA log window. This reopens the **Plot!** dialog box that was already used for plotting wavescans.

First, choose the pyroxene traverse from the *Sample List* list box. Select *Relative Microns* for the *X-Axis*. To plot for example the data for all minor elements in the pyroxene sample, ctrl-click to multi-select *TiO2*, *V2O3*, *Cr2O3*, *MnO*, and *Na2O* oxide percents in the *Y-Axis* list. Select a *Graph Type* and the button *Send Data to Plot Window*. Finally, click the **Output** button.



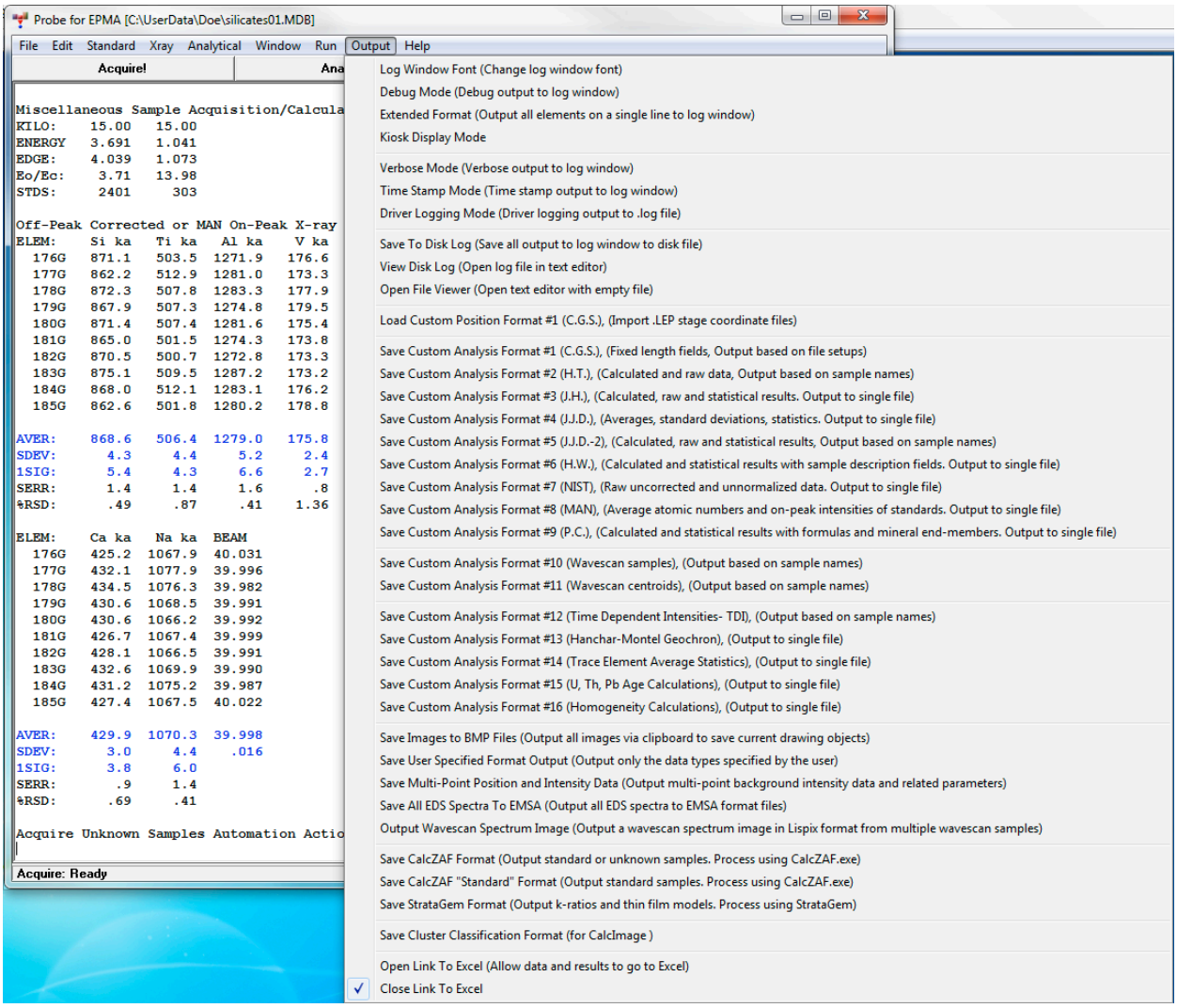
This opens the **Plot Graph Data** window with the graph of the selected data. Clicking the **Zoom** button toggles its function with the **Hot Hit** mode, in which the weight percent concentration may be read directly by clicking on a point and reading the corresponding values in the box below the **Hot Hit** button. Any graph may be directly output using the **Print** button.



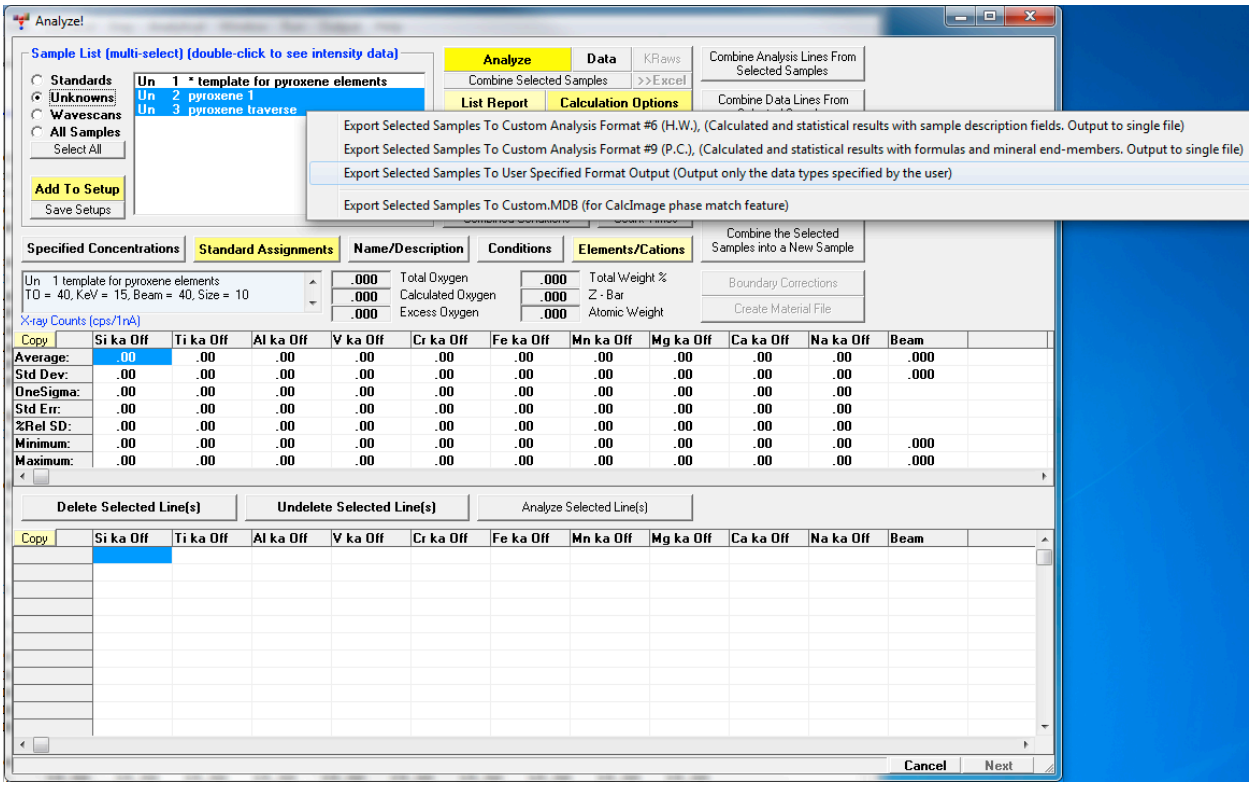


Output of Analyzed Data

In addition to saving and viewing the log window output, which has already been discussed, a wide variety of other output options are available to the user and can be accessed through the **Output** menu of the main window of PROBE FOR EPMA. These include customized analysis output, saving to an ASCII file and sending data to Excel directly. Note that all raw data is always automatically saved in the .MDB run file for future re-calculation and /or output.

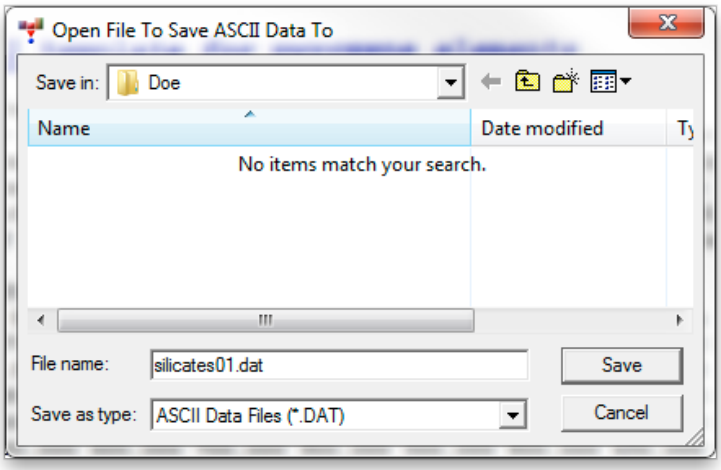


The most flexible output procedure is the *User Specified Format Output*, which is also accessible directly in the **Analyze!** window. Select the range of samples from the *Sample List* of the **Analyze!** window to be output to file. Then right click in the *Sample List* and choose *Export Selected Samples To User Specified Format Output*.

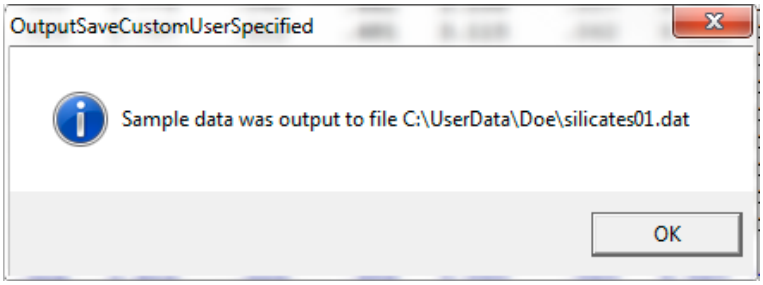


This opens the **User Specified Custom Output** window. Select the data types to export.

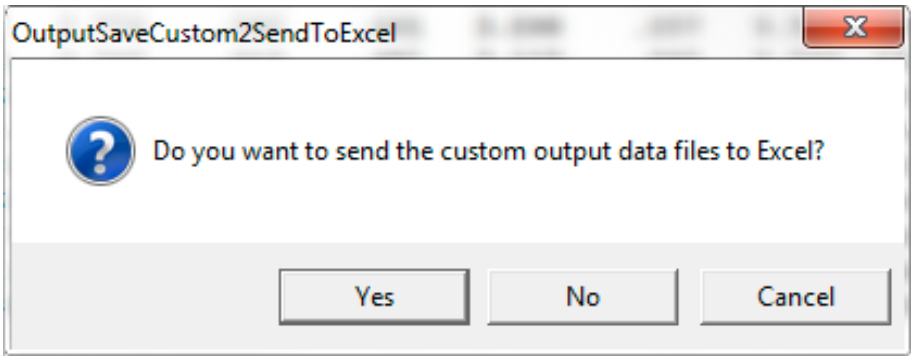
Click **OK**. This opens the **Open File To Save ASCII Data To** dialog box. Choose a file name and click **Save**.



The software will then reanalyze the selected samples and write the results to the specified file. On completion, the following message box is displayed.



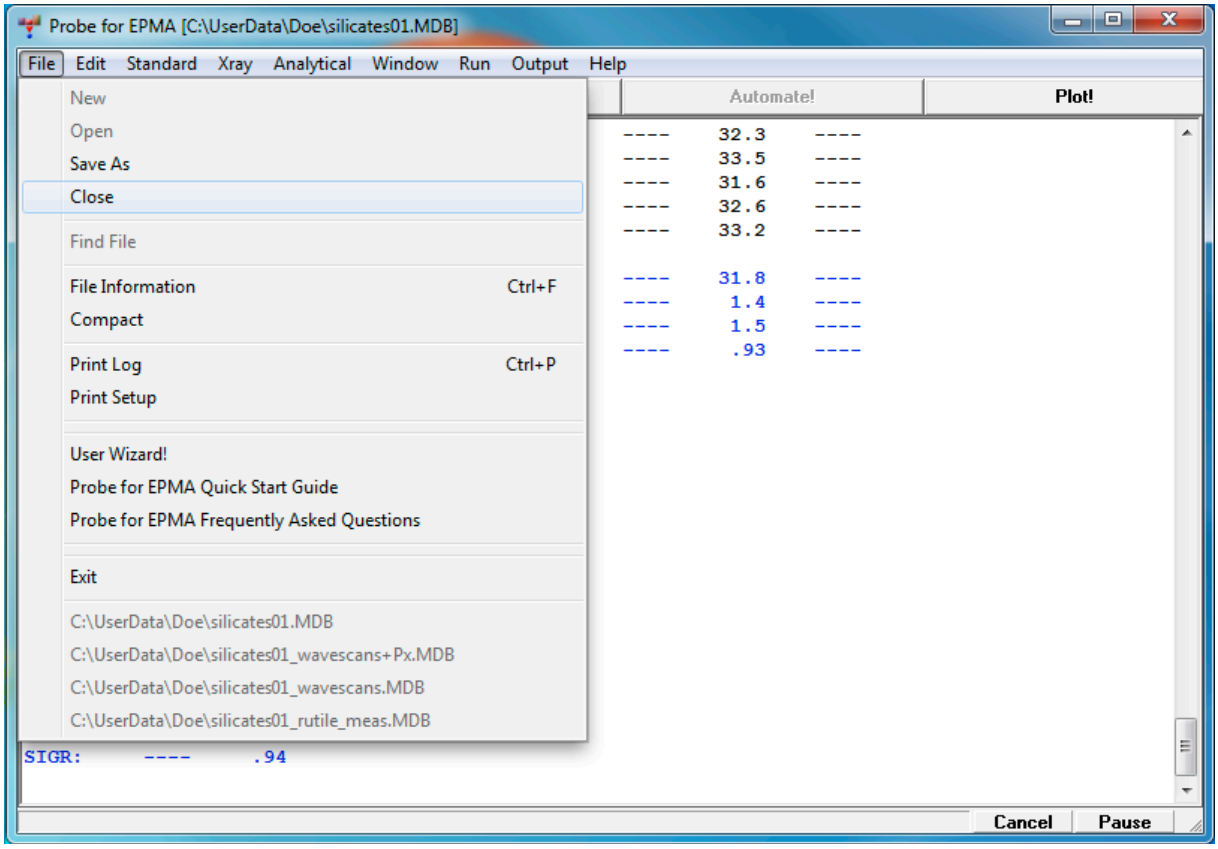
Click **OK**. Next, the following dialog box is displayed, asking the user if the data should be sent directly to Excel.



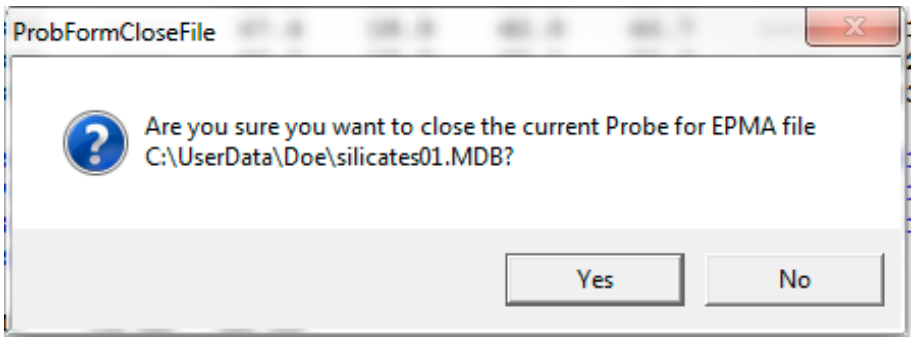
If Excel is installed, click **Yes**. The data will then directly be opened in a new Excel spreadsheet for saving and/ or further processing.

Closing the Current Run and Probe for EPMA

To end the analysis session from the main PROBE FOR EPMA log window, select **File** from the menu bar and click **Close** from the menu selections.



This opens the **ProbFormCloseFile** window. Click **Yes** to close this file.



Finally, close PROBE FOR EPMA by selecting **File** from the menu bar and clicking **Exit**.