# Probe for EPMA Quick Start Instructions

Setting up a New Run for Quantitative Acquisition

This guide is intended as a quick start and just covers the basic steps for creating a new Probe for EPMA run. Please see the Getting Started and Advanced Topics manuals for more details and step by step instructions. On-line context sensitive help is available by simply hitting the F1 key. For menu help, select the menu and then hit the F1 key.

1. Open Probewin.exe (Probe for EPMA) by double-clicking on the Probewin.exe icon shortcut.

2. When prompted "Do you want to interface to the microprobe" click "Yes". For off-line data processing click "No".
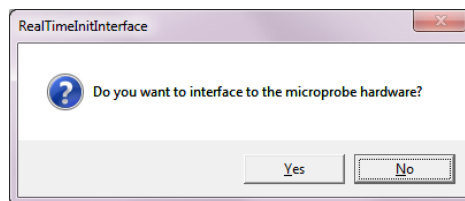

3. Create a new probe run by selecting the File | New menu.

4. In the C:\UserData folder enter a filename that describes your EPMA activity for this session and browse/create a folder that will allow you to locate your data subsequently.
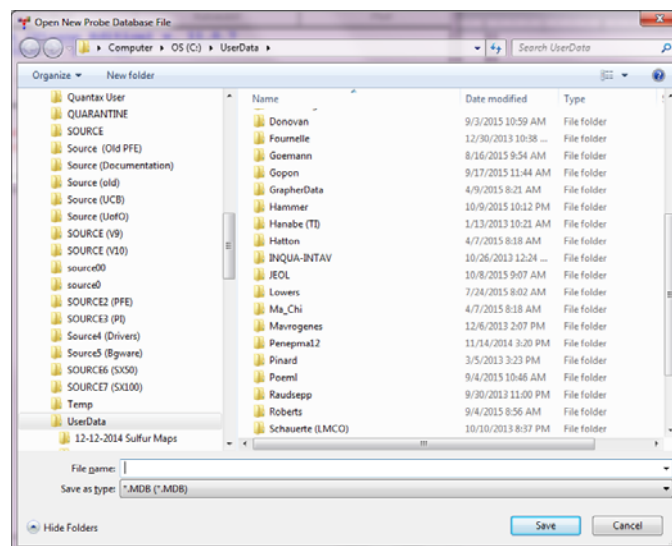

For example if your name is Joe Smith and you are characterizing Al-Ti alloys and the date is 12-08-2009 you might create a probe run file with the following path: C:\UserData\Smith\12-2009\Al-Ti_12-08-2009.MDB.

Once you enter a filename the program will prompt you with a user information dialog. You should edit the user name and sample identification and if necessary the account number and other information fields which are automatically saved to the USER.MDB user database.
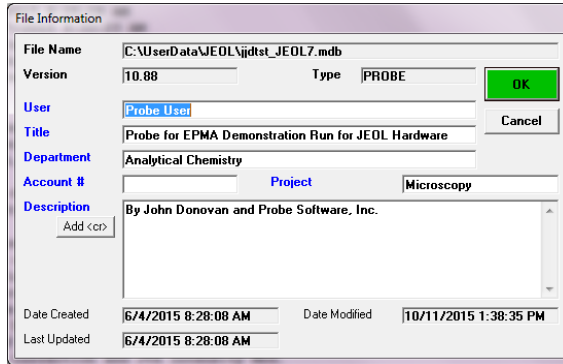

Note: the User database can be subsequently browsed using the Userwin.exe application for reporting, re-charge and/or accounting purposes.

5. Next, open the 'Acquire!' window by clicking the 'Acquire!' button in the Probewin Log window.
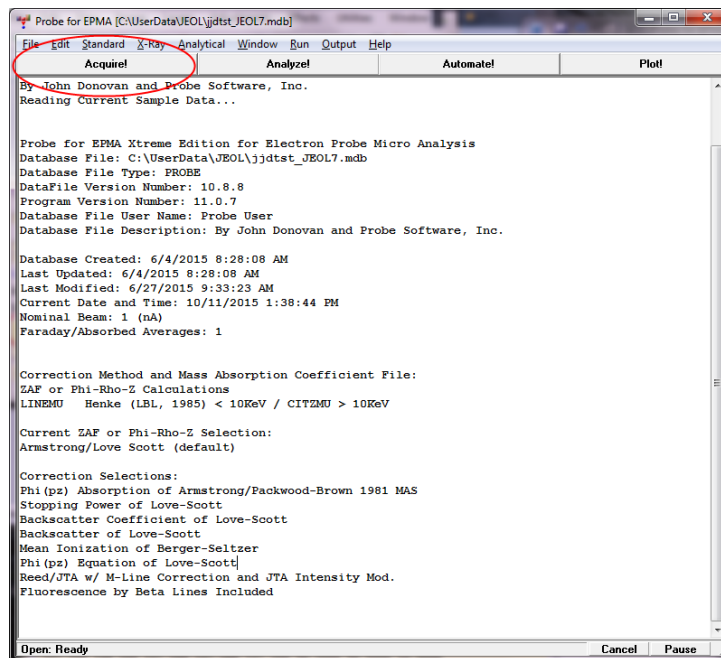

6. In the 'Acquire!' window click the 'New Sample' button to create a new sample template for your analyses. See "Creating a New Run" in the Getting Started Manual for more details.
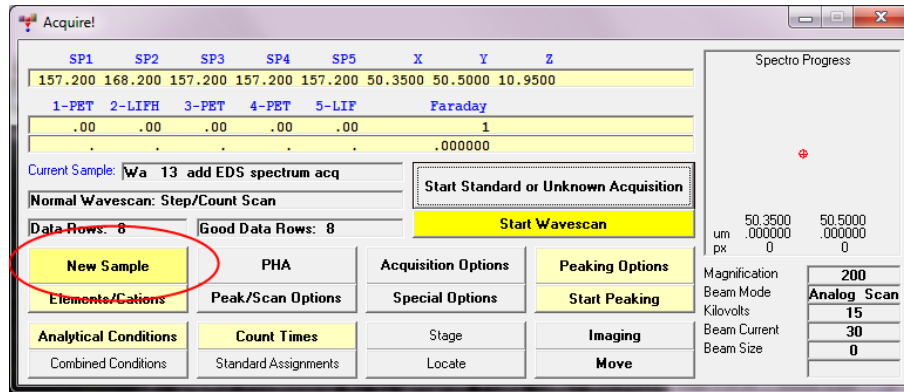
7. From this 'New Sample' dialog you can:
   - Create a blank sample (just click OK).
   - Or select previously saved individual element setups from the SETUP.MDB element setup database (previously saved from the 'Analyze!' window, 'Add To Setups' button) using the 'Load Element Setups' button.

   - Or select a previously saved sample setup from within the current probe run (not available until a sample setup has been saved from the 'Analyze!' window, 'Save Setups' button) using the 'Load Sample Setup' button.

   - Or load a sample setup from a previous probe run using the 'Load File Setup' button.
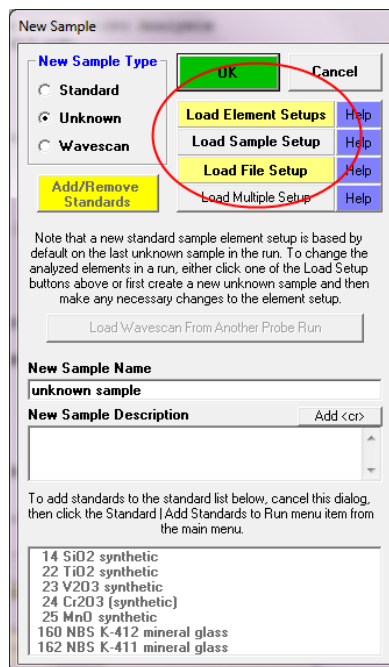
8. If creating a blank sample, one may then use the 'Move' window to select appropriate spectrometers, crystals, and also elements and x-ray lines to move to

using the drop down crystal lists and also the colored periodic table buttons. This would be the case when creating a new run "from scratch".

Once the first N elements are selected, click the 'Go Spectros' button to move the spectrometers to the nominal peak positions for the selected elements. The program will move spectrometers, flipping any crystals that are necessary.
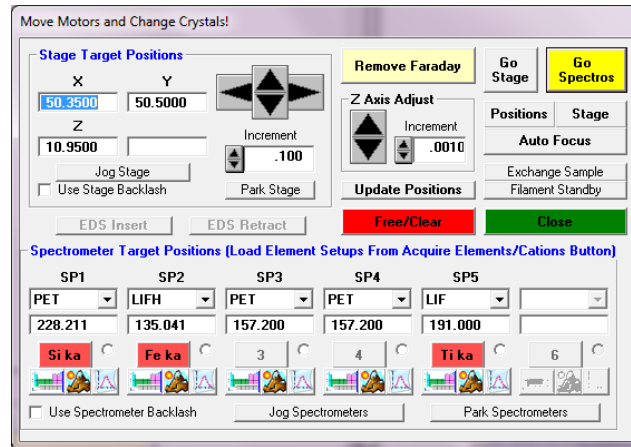

At this point one could use the 'Peaking' and 'PHA' buttons (see graphic buttons next to the tiny periodic table buttons) in the 'Move' window to manually peak the spectrometers (on appropriate standards), but it is probably better to immediately click the 'Acquire!' window, 'Elements/Cations' button and read in the nominal spectrometer element setups from the Move window using the colored element/x-ray line buttons as shown below.
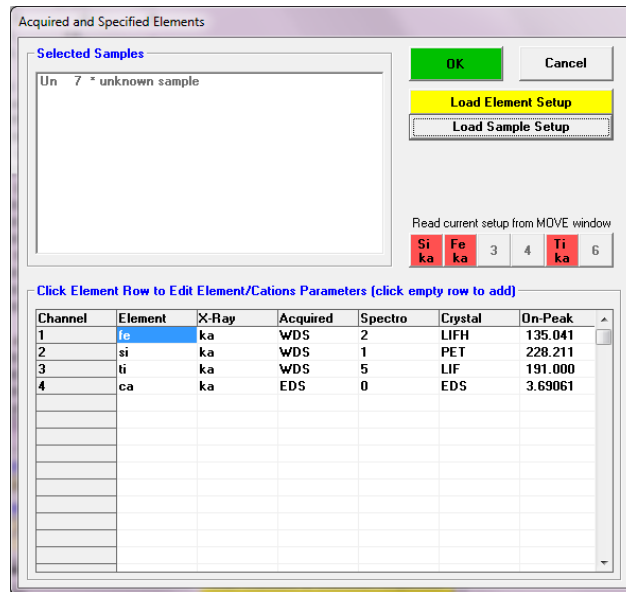
Repeat step 8 for additional elements on the various spectrometers. Later one can use the manual or automated peaking and PHA acquisition from the 'Acquire!' or 'Automate!' windows to optimize the spectrometer Bragg angle and PHA settings.

9. However, if the 'Acquire!' window, 'New Sample' button, 'Load Element Setups' or 'Load File Setup' buttons were utilized in step 7 above, then the user has already loaded previously saved element or sample setups to obtain optimized spectrometer settings (though it might still be a good idea to re-peak the spectrometers and check the PHA settings as described in the following steps).
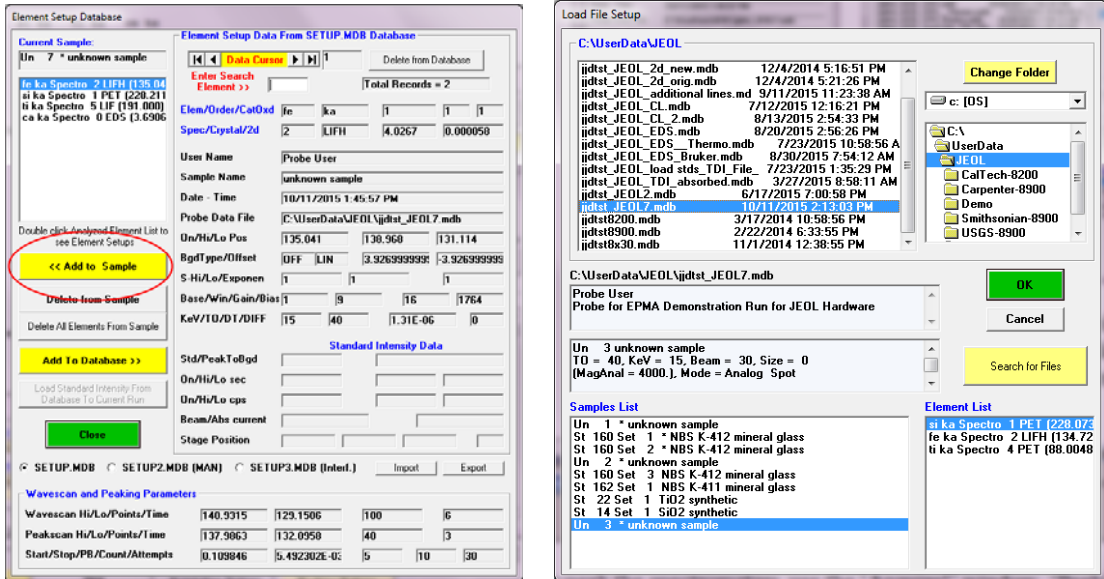
Note that if you load a file setup from a previous probe run, the program will ask you if you want to load the standard intensities from the previous run. If the standardization was recently acquired it might be ok to use, though usually one should re-acquire the standards to ensure maximum accuracy.
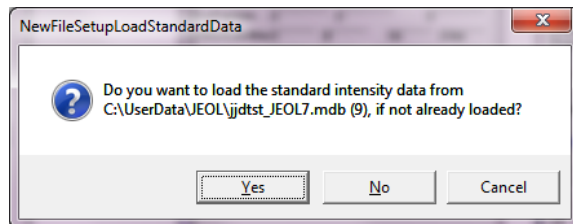

10. To manually peak the spectrometers, use the 'Acquire!' window, 'Peaking Options' and 'Start Peaking' buttons (after manually locating an appropriate standard for peaking for each element or set of elements). See "Manually Peaking Using the Acquire! Window" in the Getting Started manual for more details.
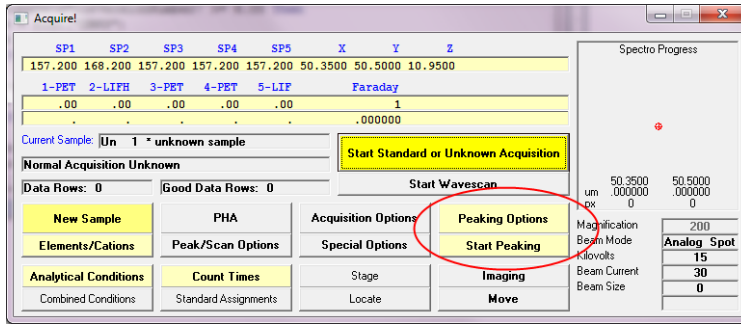
For automated peaking use the 'Automate!' window, 'Peaking' button to select the elements for peaking (be sure the check the 'Peak Spectrometers' checkbox), and then select the standards to peak in the 'Automate!' window, 'Position List' and click the 'Run Selected Samples' button to start the automated peaking.

But before starting the automated peaking, this might be a good time to check that the instrument conditions and beam are properly set. You can use the 'Acquire!' window, 'Analytical Conditions' button to set those parameters.
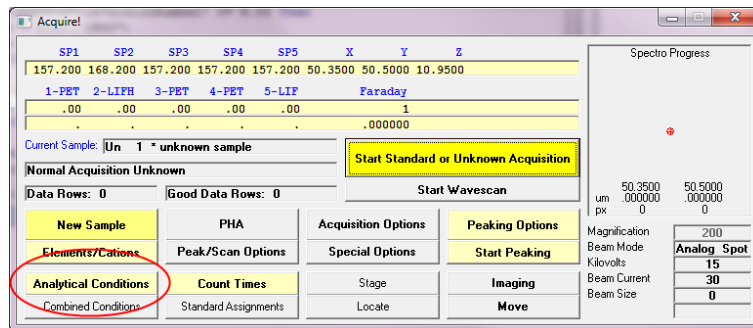
11. It might be also be a good idea to check the 'Automate!' window, 'Confirm Standard Positions' checkbox.
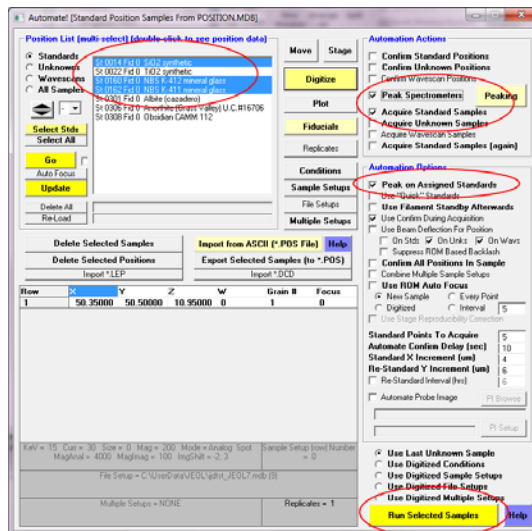
12. If the 'Peak On Assigned Standards' checkbox is checked, be sure to assign the elements to specific standards using the 'Analyze!' window, 'Standard Assignments' button so that the program only attempts a peak center on the specified primary standard.
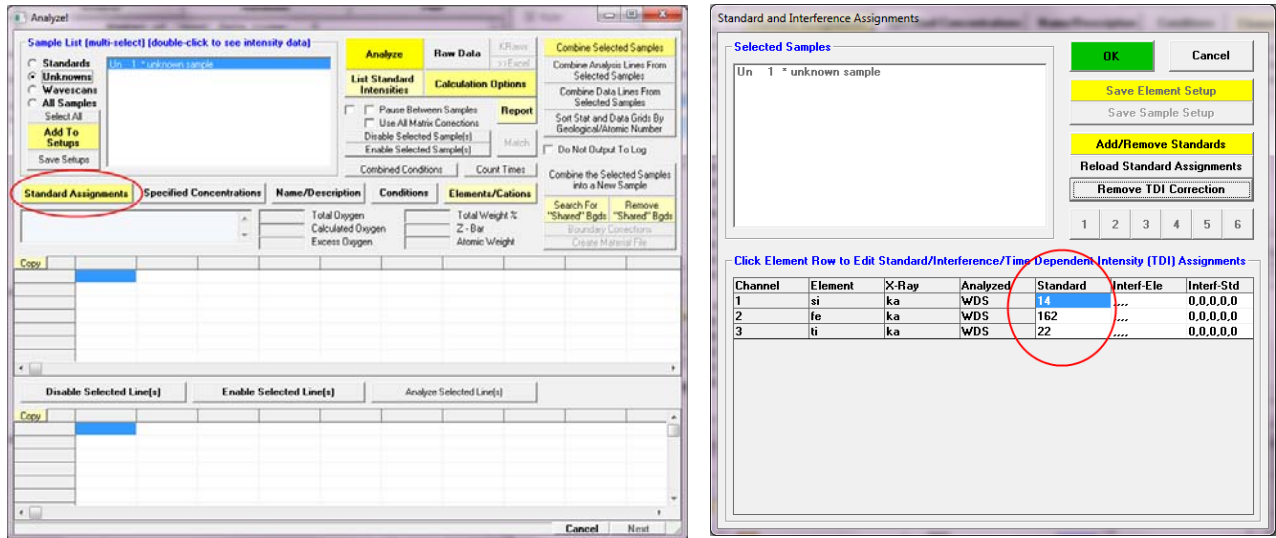

13. Note that a PHA acquisition can be manually or automatically acquired before or after the peaking procedure (normally the PHA acquisition is performed after the peak center).

14. When the 'Automate!' window, 'Run Selected Samples' button is clicked, the program will move automatically to each selected standard and perform the specified

peaking and PHA acquisitions. During this time the program will allow you to pause to confirm (and refocus) the location of each standard on the standard block (the position adjustments will be automatically remembered for next time).
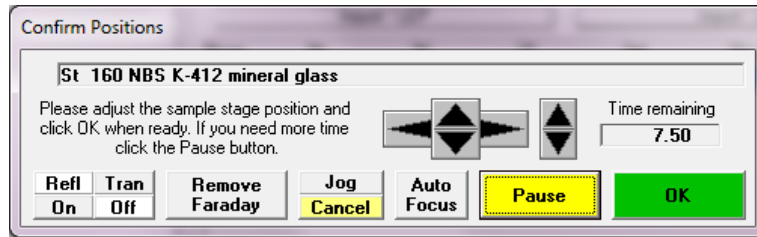

Information about the spectrometer peak intensities will be located in the Probewin Log window. If the peak operation is successful the program will indicate "Yes" under the "peaked" column. A "No" without an asterisk means the spectrometer was not specified or did not peak properly (the asterisk means no peak attempt data yet).

```
ROM Parabolic Peak Center Results:
 Element  Spectr  Peaked   StartPk    StopPk    Std  Offset    StartI     StopI
   ti ka   1 PET    Yes    88.4240   88.4940    522   -.49    1605.5    1572.4
    v ka   2 PETH   Yes    79.6966   79.6581    523   .508    2766.4    2737.4
   cr ka   3 LIFH   *No    .000000   .000000    524   .071       .0        .0
   al ka   4 TAP    Yes    90.9685   91.3303    513   -.43    1717.0    1712.3
   fe ka   5 LiF    *No    .000000   .000000    526   .445       .0        .0
```

The "Offset" is the offset from the theoritical peak position in spectrometer units. Generally the found peak should be within a few % of the calculated peak position or something may be wrong.

Once the spectrometers are all peaked properly and the PHA scans look good as shown here, one may proceed to specify the acquisition times and acquisition order for each element.
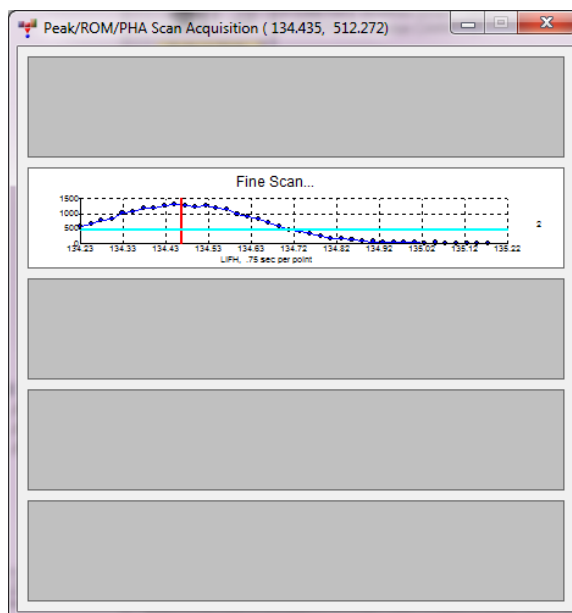
15. Note that once the peaking and PHA acquisitions are completed the results can be reviewed from the 'Run' | 'Display, Fit and Export Spectrometer Peaking and PHA Scans' menu.
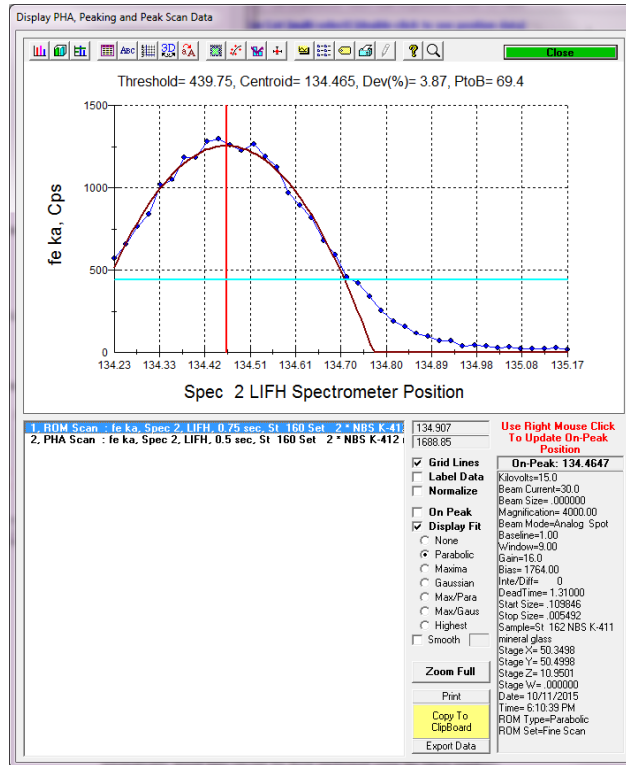
Note that all spectrometer peaking and PHA acquisitions (PHA, Bias and Gain scans) are automatically stored here (except for those performed using the Move window).

You are now ready to acquire wavescan and/or standard intensities.

Wavescan Acquisition To Check For Off-Peak Interferences

1. At this time it might be appropriate to acquire a wavescan sample to determine if there are any off-peak interferences that need to be dealt with. Usually it is sufficient to acquire a manual wavescan on a sample or standard with a composition similar to your unknown although that is not strictly necessary, since any composition will serve to simply display the KLM markers.

2. First go to an appropriate sample for the wavescan acquisition, then click the 'Acquire!' window, 'New Sample' button and select the 'Wavescan' option. Enter a name such as "check bgds" and click OK.


3. Then check the 'Acquire!' window, 'Count Times' dialog to make sure that the count times for the wavescan elements are reasonable, then click OK.
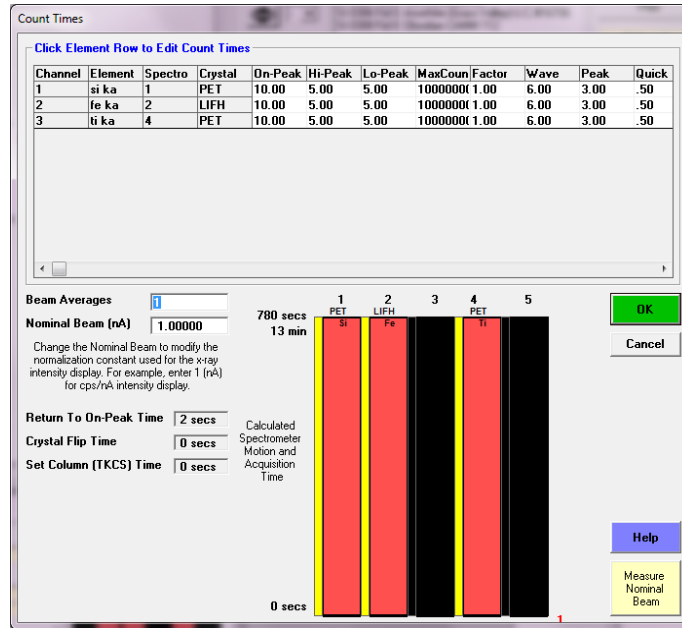
4. When you are ready to acquire the wavescan sample, simply click the 'Acquire!' window, 'Start Wavescan' button and the program will automatically acquire an intensity scan for each element automatically.
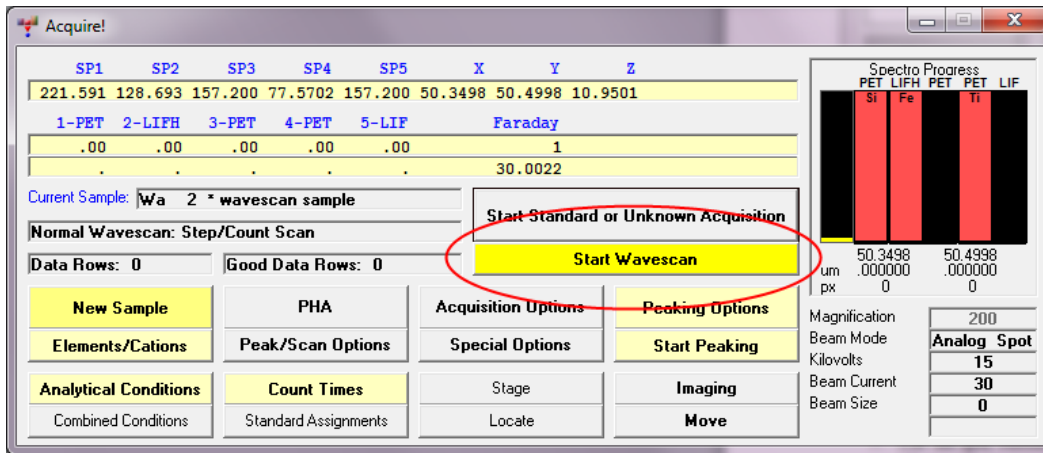

5. Once the wavescan acquisition has been completed, use the 'Plot!' window to display the wavescan intensities. See "Off-Peak Adjustments From the Plot! Window" in the Getting Started manual for more details.
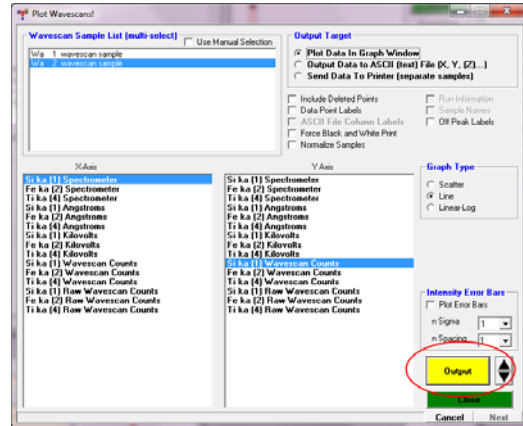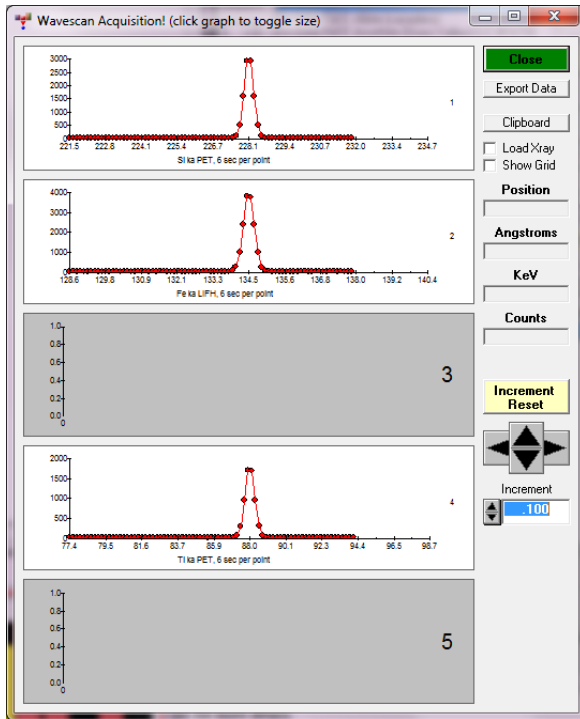
7. Use the spin button to select the next spectrometer and element pair and click the 'Output' button to display the wavescan and the current off-peak positions (in green). The x-ray KLM markers can be displayed by selecting the 'Analyzed Elements' option.
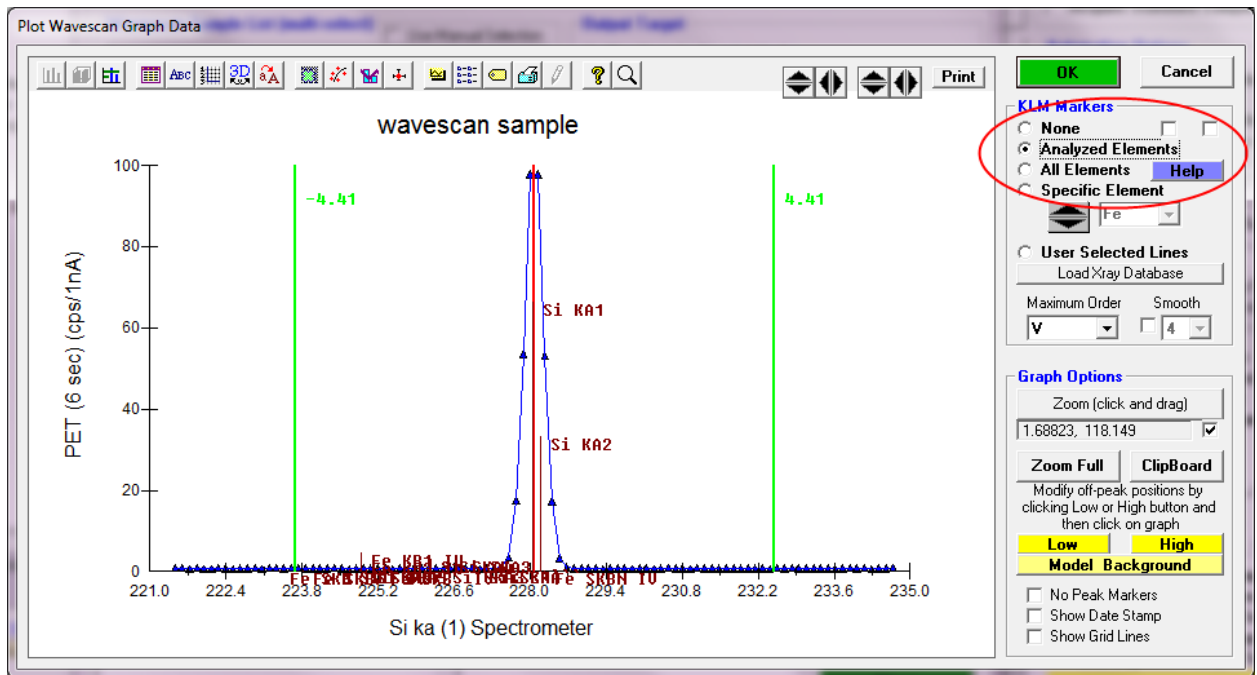
Then use the mouse to click and drag the preferred zoom area to see the background better.
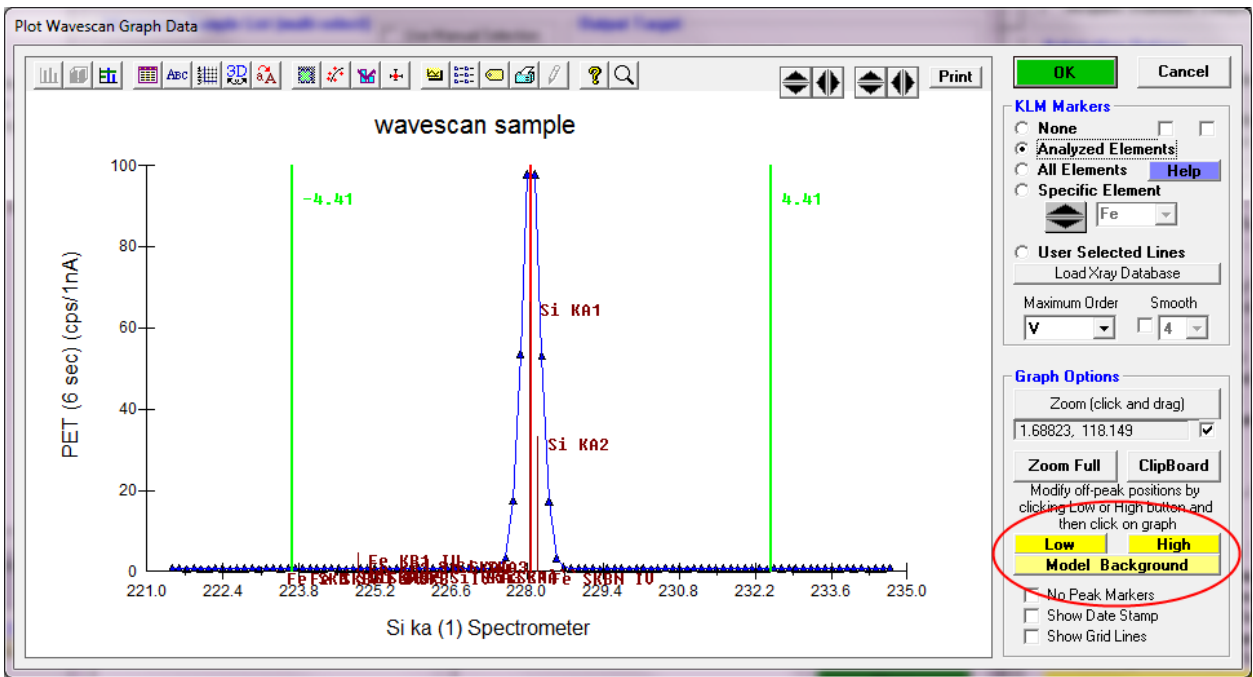
8. Then simply adjust the off-peak positions by clicking the 'Low' and/or 'High' buttons and clicking on the graph where they should be re-located.
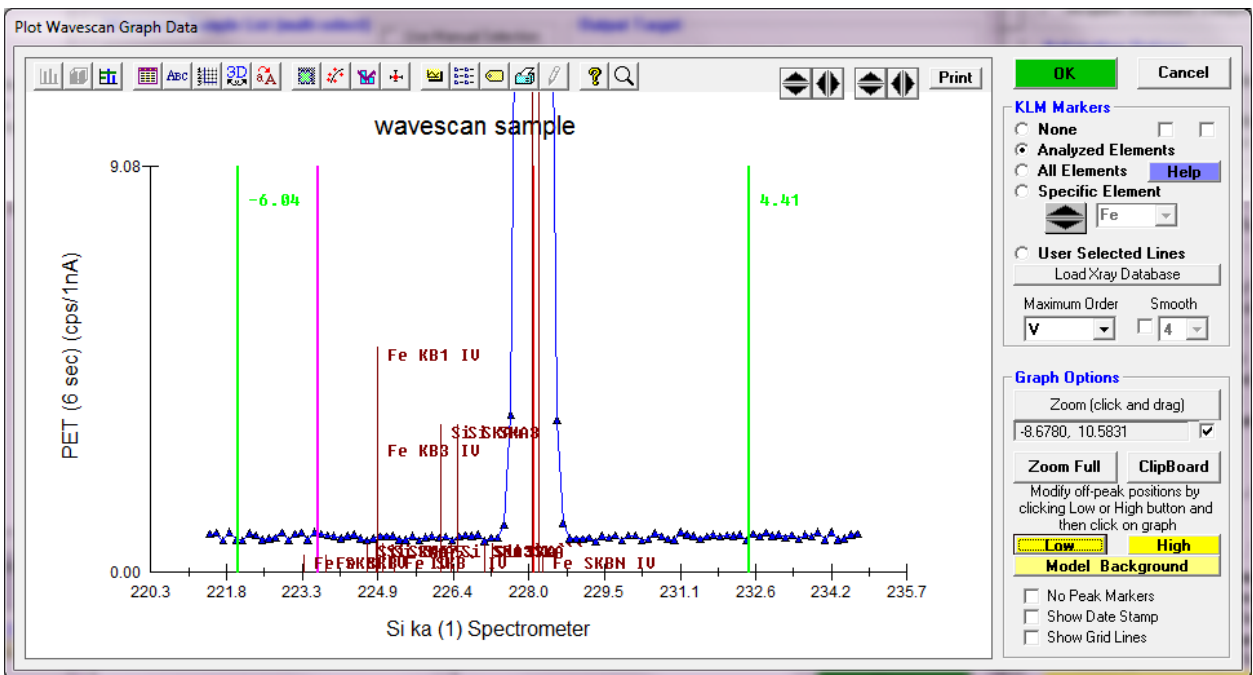
9. When you click OK, the new off-peak positions will be utilized automatically in the next unknown or standard acquisition. Repeat steps 7 to 9 for each element in the wavescan.

Acquisition of Standard Intensities

1. In the 'Automate!' window, click on the 'Standards' option and click the 'Select Stds' button to select the current standards in the run. You have previously confirmed standard positions during peaking, but you can check that option again if you would like.

2. Check the 'Acquire Standard Samples' checkbox and then click on the 'Run Selected Samples' button. See "Loading Standard Position Files" in the Getting Started manual for more details.

3. Once primary standards have been run, you can check that they agree with each other by running secondary standard and checking that it analyzes correctly from the 'Analyze!' window by selecting the secondary standard and clicking the 'Analyze' button.
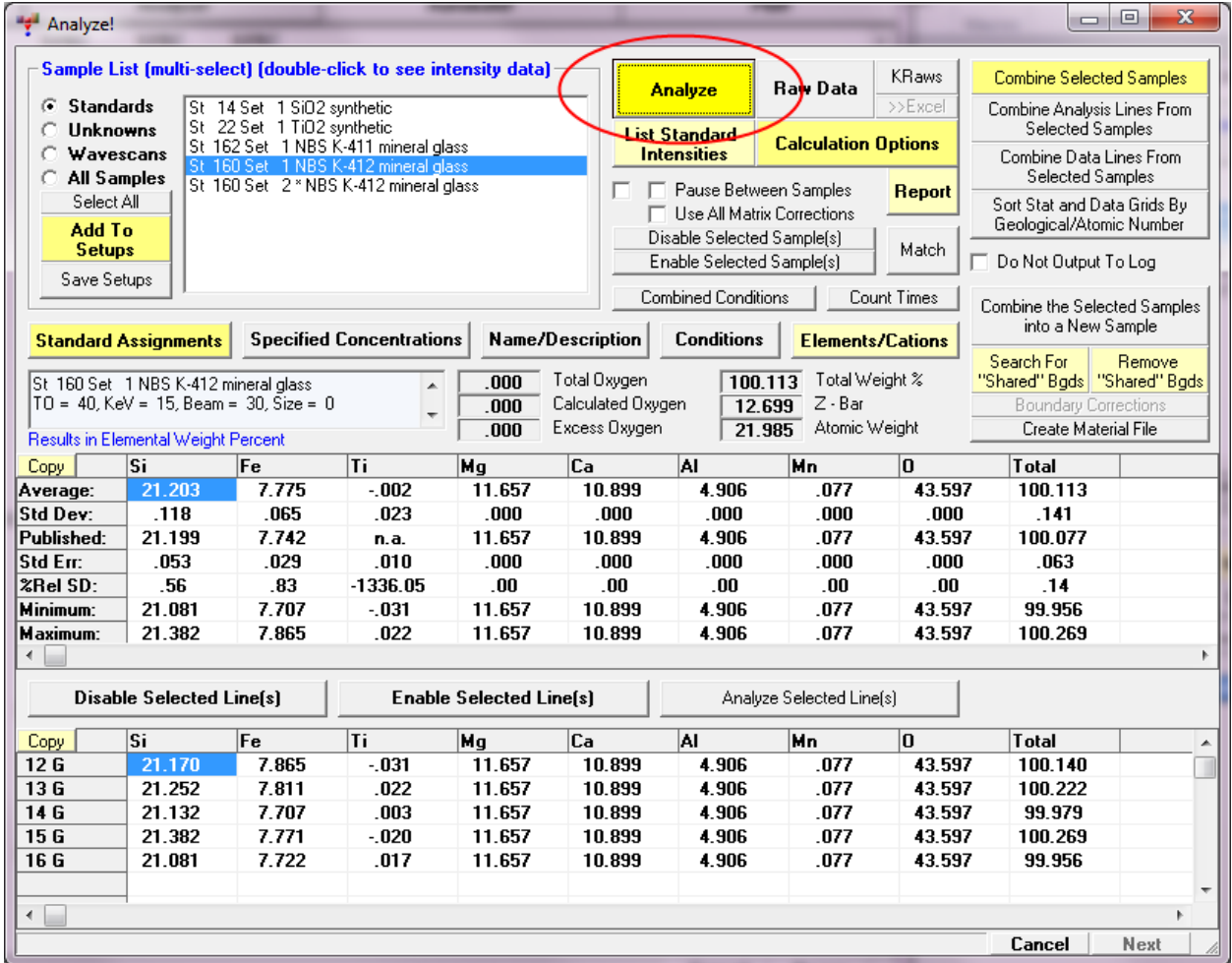
See "Analyze Standard Data" in the Getting Started manual for more details.

In this case a good secondary standard for an Al-Ti alloy might be a NIST standard in which the user can compare the measured 'Average' results with the 'Published' results from the Standard.MDB standard database.

In the Log window the analysis is printed out with the Published values and the relative percent error ("%VAR") so the user can quickly compare the expected value witrh the actual measured value. Note that if the standard being analyzed is the assigned primary standard for that element, then the "%VAR" will be close to zero (but not exactly due to the presence of other elements not assigned as the primary standard for that element).

However elements that are not using the standard as the assigned primary standard (that is a secondary standard) will provide a meaurement of the relative accuracy of the instrument assuming that the secondary is close to the composition of the unknown samples to be analyzed.
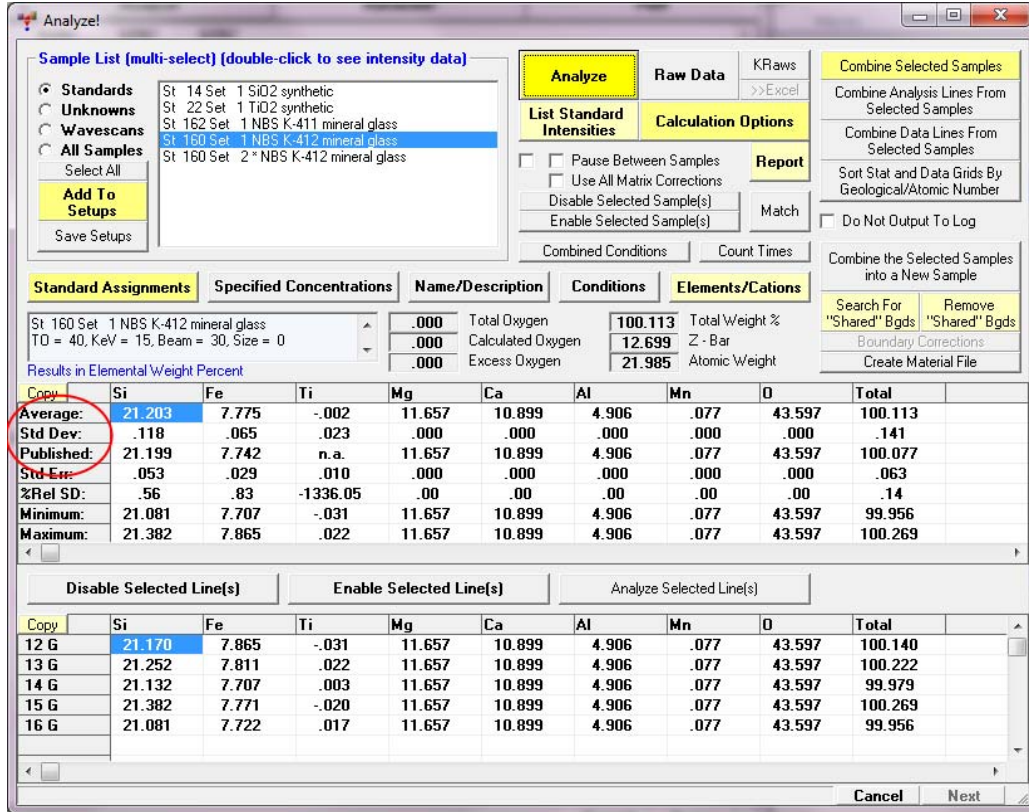
In the log window the output is similar but more detailed. Here is a NIST Al alloy analyzed using pure metals as elements as primary standards for all elements:

```
ELEM:       Ti        V        Cr       Al       Fe    SUM
29        .055     .021     .171   89.932     .182 100.061
30        .043    -.030     .206   89.425     .235  99.579
31        .025    -.002     .246   89.452     .190  99.611
32        .058     .004     .198   89.445     .233  99.638

AVER:     .045    -.002     .205   89.563     .210  99.722
SDEV:     .015     .021     .031     .246     .028    .227
SERR:     .007     .011     .015     .123     .014
%RSD:    33.0  -1153.6     15.0       .3    13.4

PUBL:     .040     n.a.     .173   89.716     .205    .000
%VAR:    13.09      .00    18.64     -.17    2.41
DIFF:     .005     .000     .032    -.153    .005
STDS:      522      523      524      513      526
```

Note the "%VAR" and "DIFF" lines which show the relative % error from the published composition and the algebraic difference in absolute weight %.

Using the 'Analyze!' window, 'Data' button it is also useful to check that the "SDEV" or measured standard deviation of the intensities are within 3 sigma of the predicted variance as shown in the line labled "1SIG".

```
On-Peak (off-peak corrected) or MAN On-Peak X-ray Counts (cps/1nA) (and Faraday
Current):
ELEM:    ti ka    v ka    cr ka    al ka    fe ka    BEAM
BGD:       OFF      OFF      OFF      OFF      OFF
SPEC:        1        2        3        4        5
CRYST:     PET     PETH     LIFH      TAP      LiF
ORDER:       1        1        1        1        1
   29G      .16      .06      .51   267.70      .54   29.982
   30G      .13     -.09      .61   265.97      .70   29.991
   31G      .07     -.01      .73   266.07      .57   30.015
   32G      .17      .01      .59   266.01      .70   30.016

AVER:      .13     -.01      .61   266.44      .63   30.001
SDEV:      .04      .06      .09      .84      .08     .017
1SIG:      .06      .06      .08      .94      .07
SIGR:      .78      .98     1.13      .90     1.19
SERR:      .02      .03      .05      .42      .04
%RSD:     33.0  -1153.5     15.0       .3     13.4
```

Variances larger than 3 sigma may indicate sample inhomogeneity or instrument instability.

Digitizing Position Samples

1. From the 'Automate!' window click on the 'Digitize' button. You will now be able to digitize stage positions of samples you wish to acquire intensity data for.
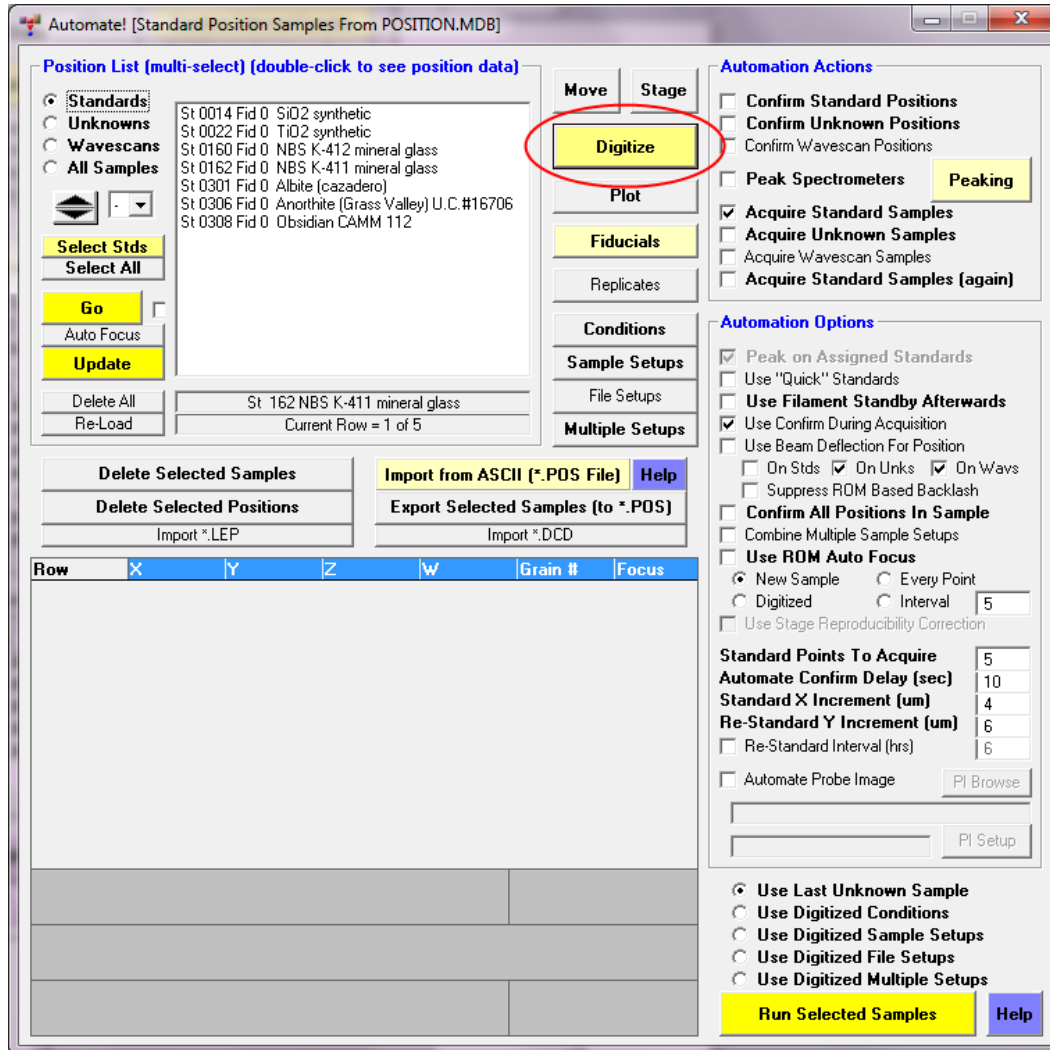
2. In the 'Digitize Sample Positions' window choose the 'Unknown' option and enter a sample name in the text field (a sample description is optional) and click the 'Add New Unknown or Wavescan To Position List' to add an empty position sample to the 'Automate!' window position sample list.
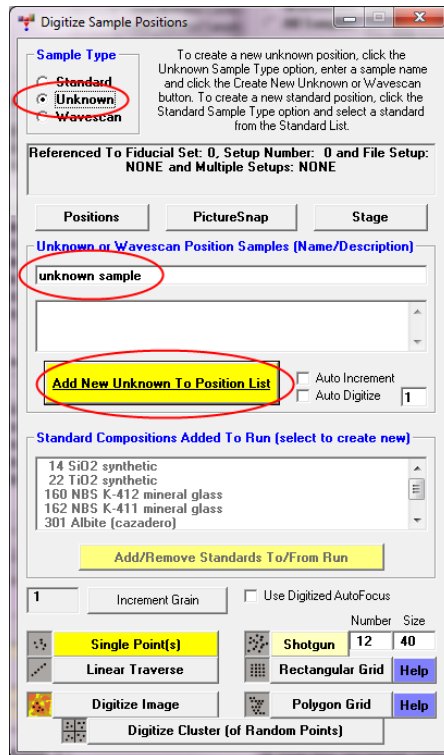
3. Then click on the 'Stage' button to bring up the 'Stage Map' window to navigate to the sample coordinates. Select the appropriate stage map image or drawing from the 'Holder Selection' drop down list.
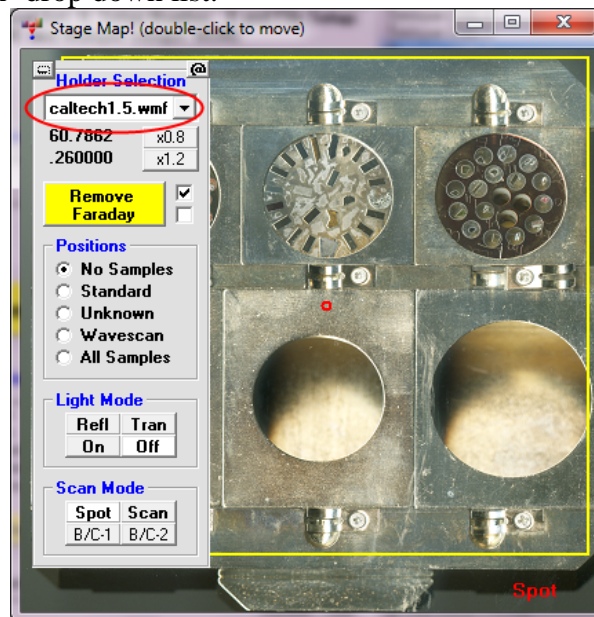

4. In the 'Stage Map' window, double-click on where you think your sample is. Use the instrument joystick or stage fine adjustment controls and locate your first acquisition position. Once you find a good spot, focus on the sample surface using reflected light, using the instrument Z stage position adjustment.

5. In the 'Digitize Sample Positions' window, click on 'Single Point(s)' or 'Linear Traverse' or whatever mode you want to digitize coordinate data and click OK once you have established the coordinates for that acquisition. You may create any number of position samples, with any sample name and digitize any number of points in each position sample.
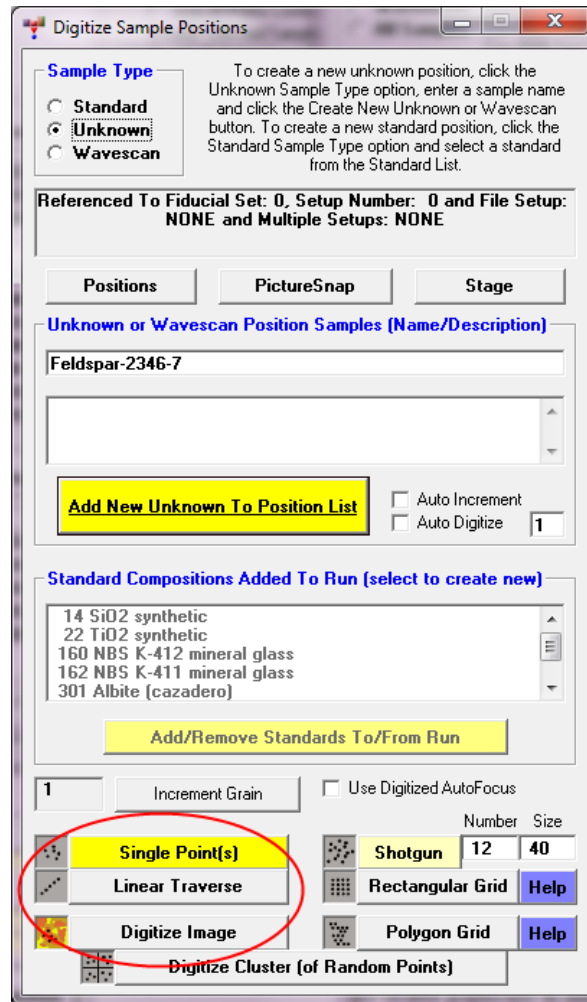

6. Repeat steps 4 and 5 until all of your unknown samples have been digitized.

7. In the 'Stage Map' window, choose to show locations of the sample by selecting from the 'Positions' options, for example 'Unknowns' to double check where your coordinate positions are located graphically in instrument stage coordinates.

Tip: **Picture Snap!** is a great tool included in Probe for EPMA. It simply requires an image file (jpeg, bmp, or gif) of your sample mount from any source (slide scanner, flat bed scanner or electron image from any instrument). Access Picture Snap! from the Probewin log window, 'Run' menu by selecting the 'Display Picture Snap!' menu.
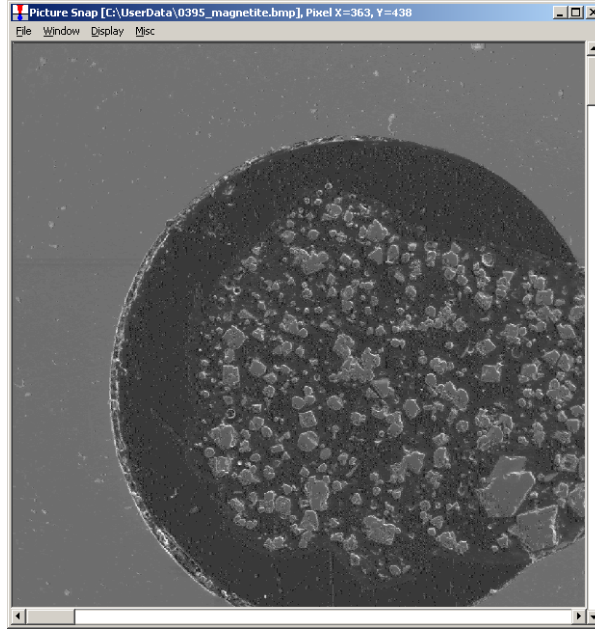
Load your image file and then choose the 'Window' | 'Calibrate' menu. In the 'Picture Snap Calibration' window, select either the 'Two Points' option (for orthogonal rectangular samples) or the 'Three Points' option (for round samples subject to rotation).
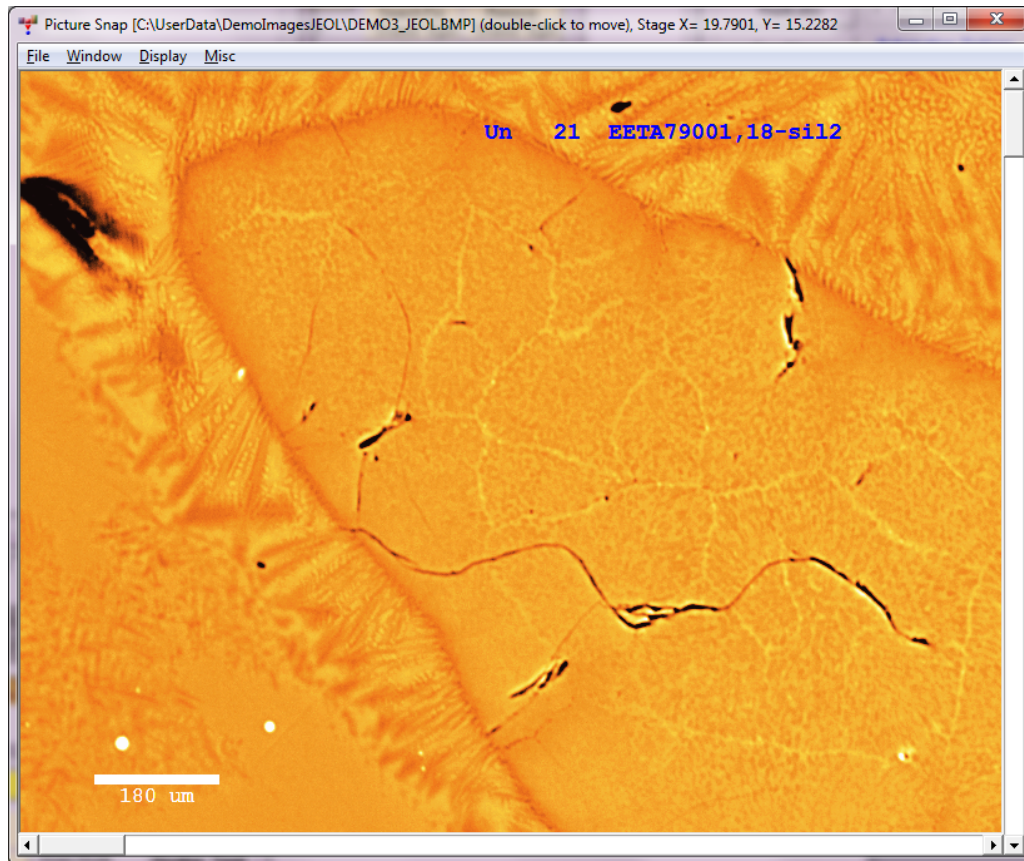
Use the stage controls and navigate to 2 or 3 points that you can easily recognize on the image and also find the stage coordinates of in the instrument imaging system (visible light or electron imaging). Click 'Read Current Stage Coordinate' to read the stage position of the feature. Generally choose 2 points that are diagonally opposed on the sample or if using three points, choose points that describe a triangle over most of the image area for best accuracy. Then also click the 'Pick Pixel Coordinate On Picture' and click the corresponding spot on the picture for the image coordinates for each calibration position.

When finished choosing reference points click the 'Calibrate Picture' button. Now you should be able to navigate anywhere on your image by double-clicking the feature in the image. Other menus allow you to display the position sample coordinates as they are digitized.

Automated Sample Acquisition

1. In the 'Automate!' window choose the 'All Samples' option. Then click the 'Select Stds' button and establish that you picked the correct standards for your samples. Then holding down the <ctrl> button select the unknowns you also wish to acquire intensities for.
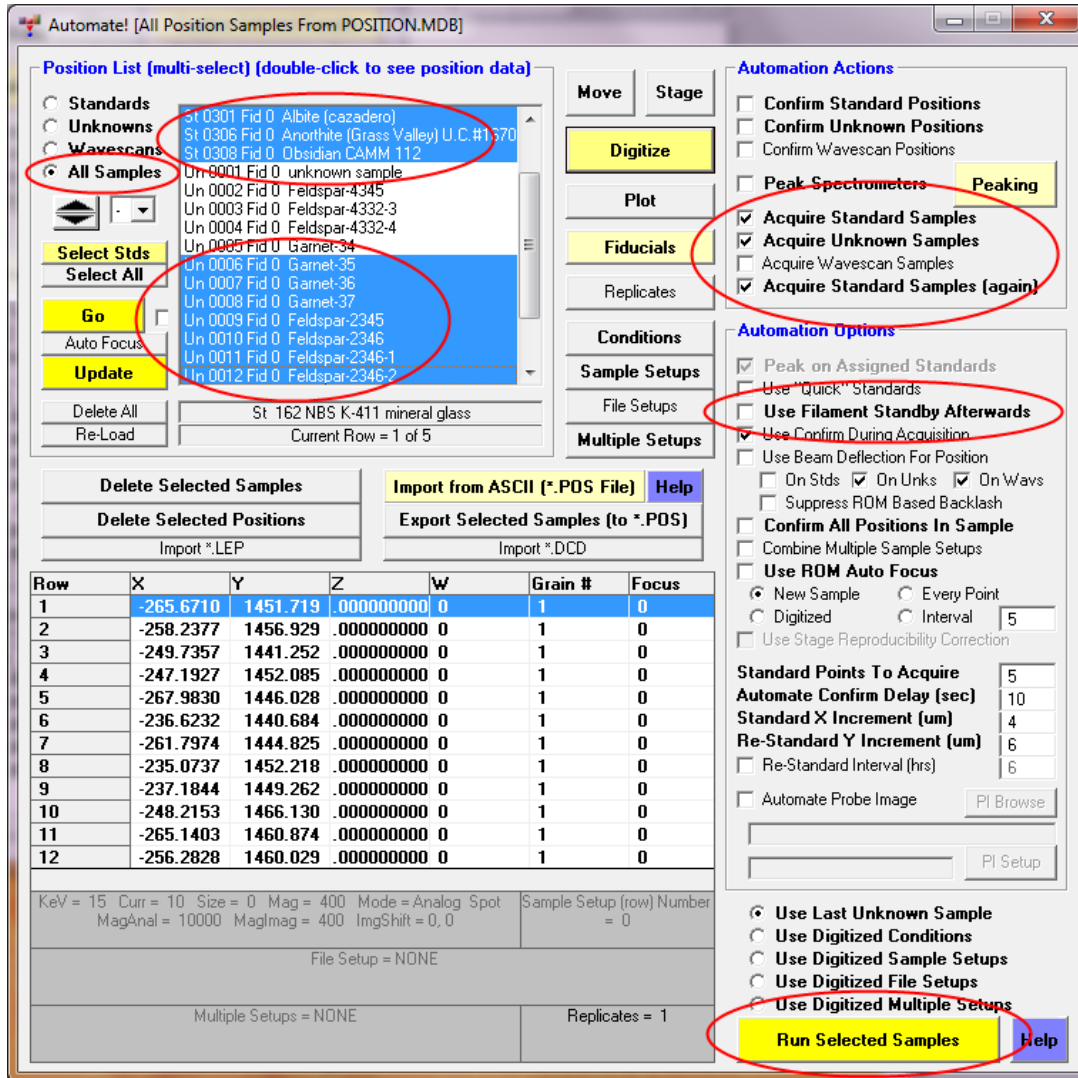

2. Now click the following checkboxes: 'Confirm Standard Positions', 'Acquire Standard Samples' (optional since you may have just done this), 'Acquire Unknown Samples', 'Acquire Standards Again', and optionally the 'Use Filament Standby Mode Afterwards' and 'Use Confirm During Acquisition'.

3. Double-check everything and click the 'Run Selected Samples' button. This will bring up a confirmation dialog with the total time estimate. Check carefully and click "Yes" and the automated run will start. The standard positions will be confirmed again. Make sure they are still in focus.

AutomateConfirmSelected: Using Last Unknown Sample

Number of Standard Position Samples: 7
Number of Unknown Position Samples: 7
Number of Wavescan Position Samples: 0

Elapsed Time for Last Standard Acquisition: 27 seconds
Elapsed Time for Last Unknown Acquisition: 27 seconds

Projected Time for All Standard Acquisitions: 44 mins or 2622 secs
Projected Time for All Unknown Acquisitions: 48 mins or 2892 secs
Total Projected Time for All Acquisitions: 1.53 hours

Are you sure you want to run these automated position samples?

Yes   No

Manual Acquisition

1. You may also simply acquire sample data manually using the 'Acquire!' window. Simply click the 'Acquire!' window, 'New Sample' button, click the 'Unknown' option (or click the 'Standard' option and select a standard from the list for running a standard manually) and enter a sample name. Note that wavescan samples can be acquired from this same window and should be used to check for off-peak interferences.
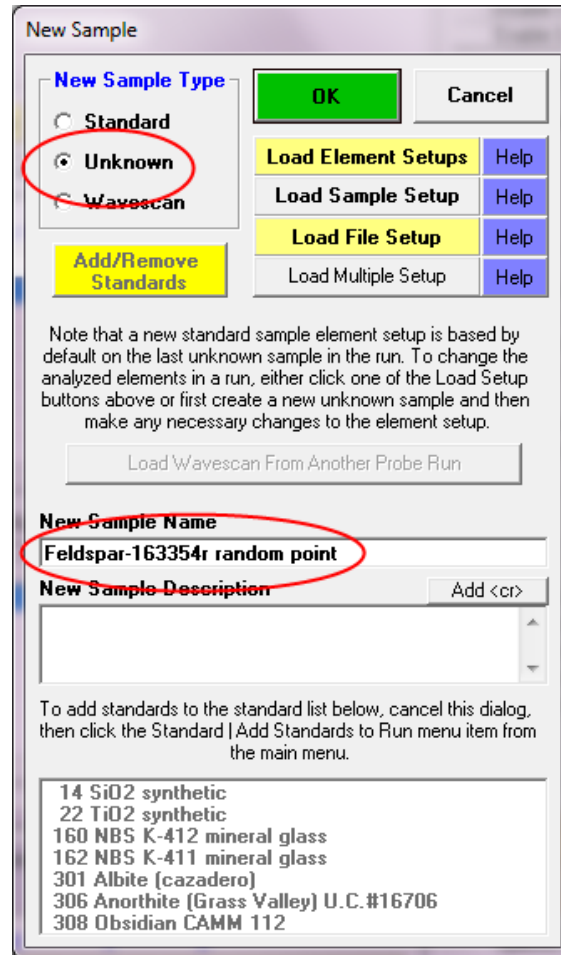
2. Then click the 'Acquire!' window, 'Start Standard Or Unknown Acquisition' button to start the manual data acquisition. The elapsed time meter will display the acquisition progress. When finished you should acquire additional replicate points until the average and standard deviations look reasonable. See "Manual Count Acquisition Using the Acquire! Window" in the Getting Started manual for more details.

3. Click the 'New Sample' button again to name a different unknown sample for manual acquisition. Note that your samples may be specified any way (up to 100 points per sample) that you prefer, although usually all data points in a single samples should be related spatially or compositionally since an average and standard deviation will be calculated for each sample.

Analyzing Your Data

To obtain your composition results simply use the 'Analyze!' window and select the samples to analyze and click the 'Analyze' button.

Use the 'Calculation Options' to add oxygen by stoichiometry, atomic percents, detection limits, etc. Use the 'Elements/Cations' button to add unanalyzed elements for calculation purposes (leave the x-ray line blank).

Many more output options are available from the Probewin Log window, 'Output' menu and the 'Plot!' window. See "Output of Analyzed Data" in the Getting Started manual for more details.

Tips & Common Problems

1. Note that by acquiring intensity data for all the elements in each standard you can examine the complete analysis of each and see that you should be able to get close to zero concentrations on standards that do not contain the element of interest. This is very important for trace elements- that is: can you measure zero? If the concentration is significantly negative then it is likely that one or more off-peak positions is being interfered by a secondary x-ray peak.

2. Establish which spectrometers should be used for which elements. You will want to avoid crystal flipping within a single sample. Put in proper count times for what you are doing. Check that the count times are approximately balanced for all spectrometers to avoid a spectrometer sitting idle (you might as well be counting photons).

3. Use the 'Stage Map' window displaying the unknowns to show where all of your random points or linear traverses are. Figure out which numbers correspond to the unknowns you want.

4. If you made a mistake in the digitizing go to the 'Automate!' window and double click on the sample you need to alter. The sample positions you have digitized will come up in the box below the sample position list. Choose the duplicate or mis-digitized points and click on 'Delete Selected Positions' then OK.

5. Consider acquiring images on all your sample positions. These positions can be plotted on the images once the acquisition has completed.

John Donovan, Probe Software
10-11-2015