

Calibration Contents

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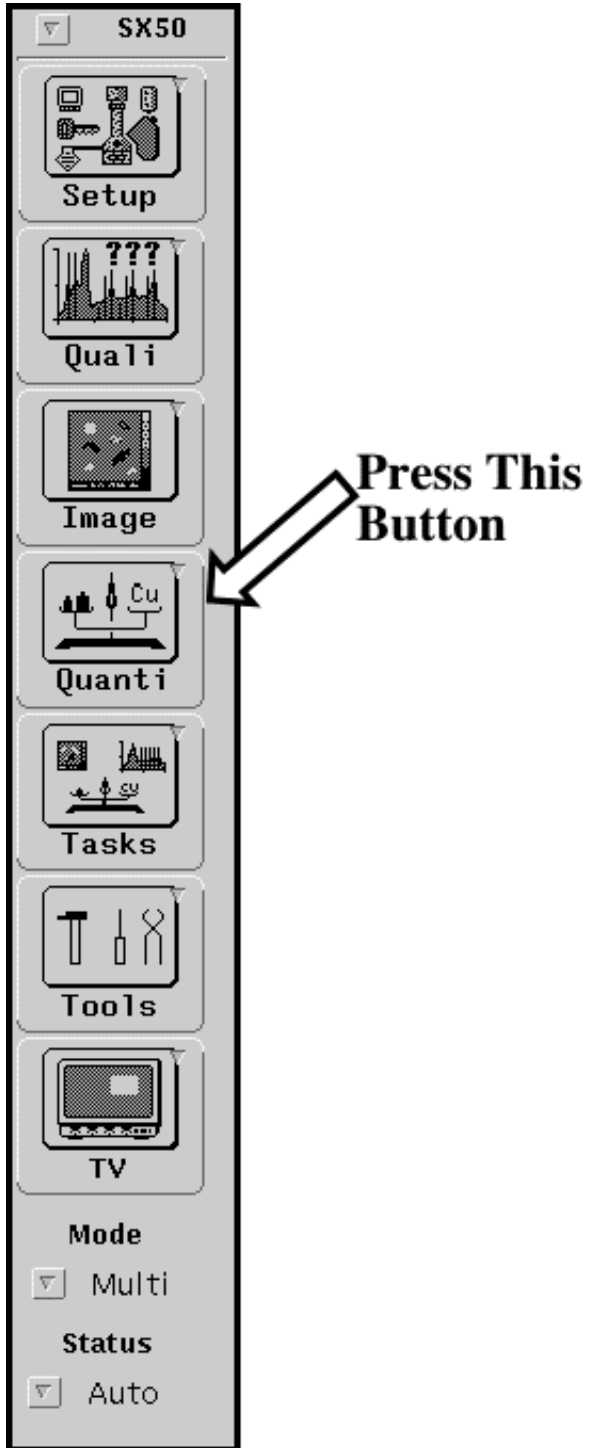
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Electron Microprobe Laboratory Manager
Lunar and Planetary Laboratory
University of Arizona
May 2005

Note: These instructions reflect current procedures in our lab on our Cameca SX50 only.

Calibration: Instruction Set 1

Starting Calibration: Manual and Multiple

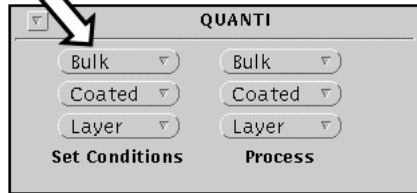
Step 1



Step 2

The following screen will appear at the lower left corner of the screen. When it does:

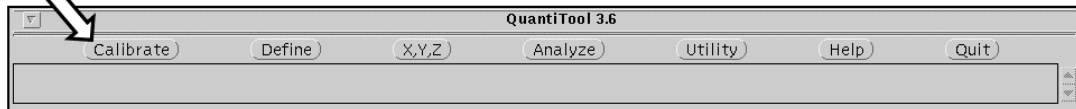
Press This Button



Step 3

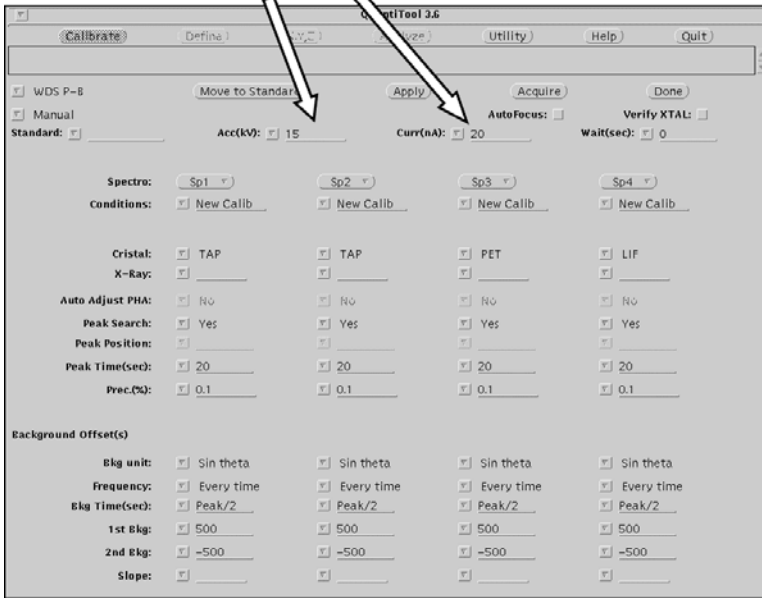
The following screen will appear at the top of the screen. When it does:

Press This Button



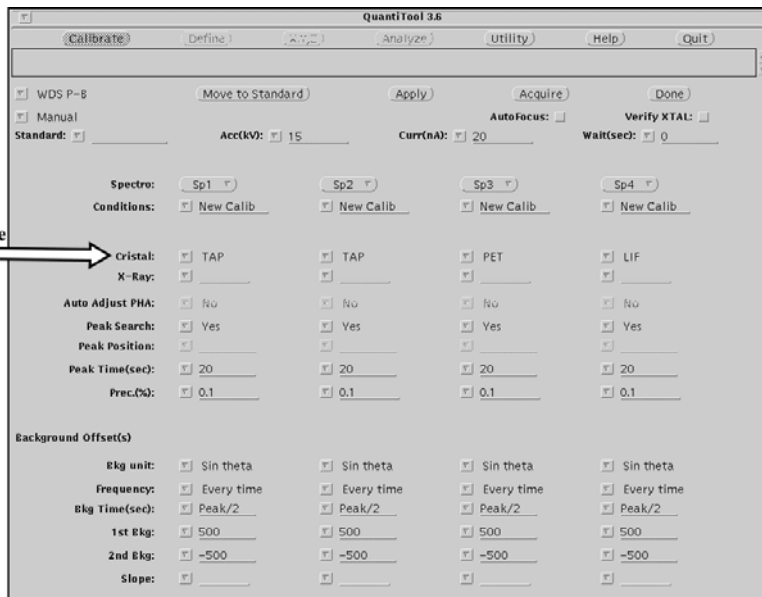
Step 4

Make sure the Voltage and Current are Correct

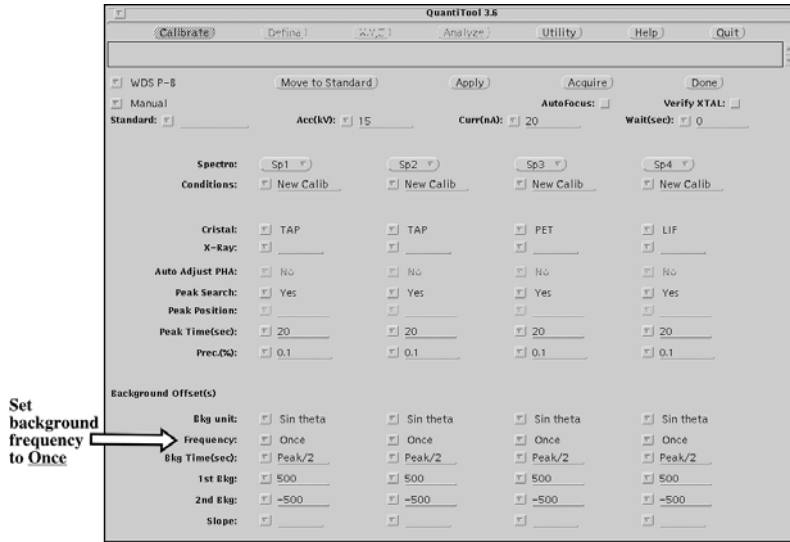


Step 5

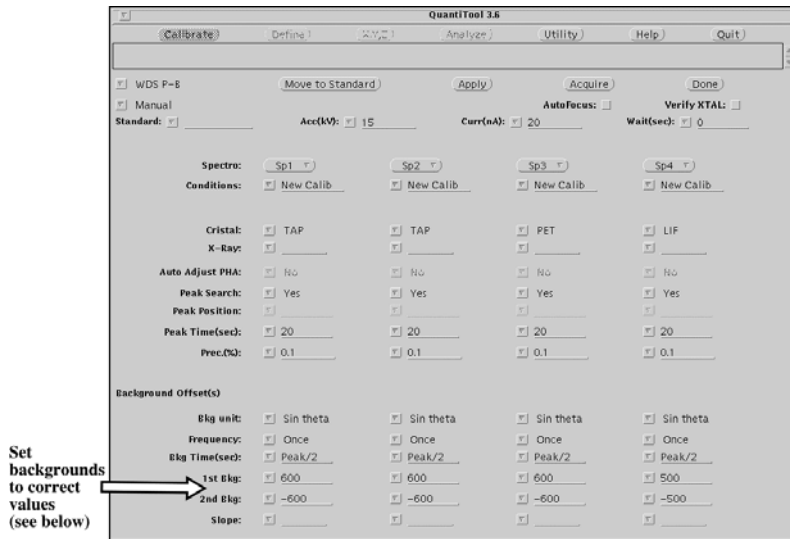
Make sure Crystals are correct



Step 6



Step 7



Default Backgrounds

TAP +600, - 600
PET +600, - 600
LIF +500, -500
PC1, 2, 3 +2000, -2000

Next Step

Go to either:

Manual Calibration Instruction Set 2a (p. 8)

or

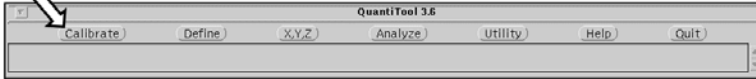
Multiple Calibration Instruction Set 2b (p. 22)

Calibration: Instruction Set 2a

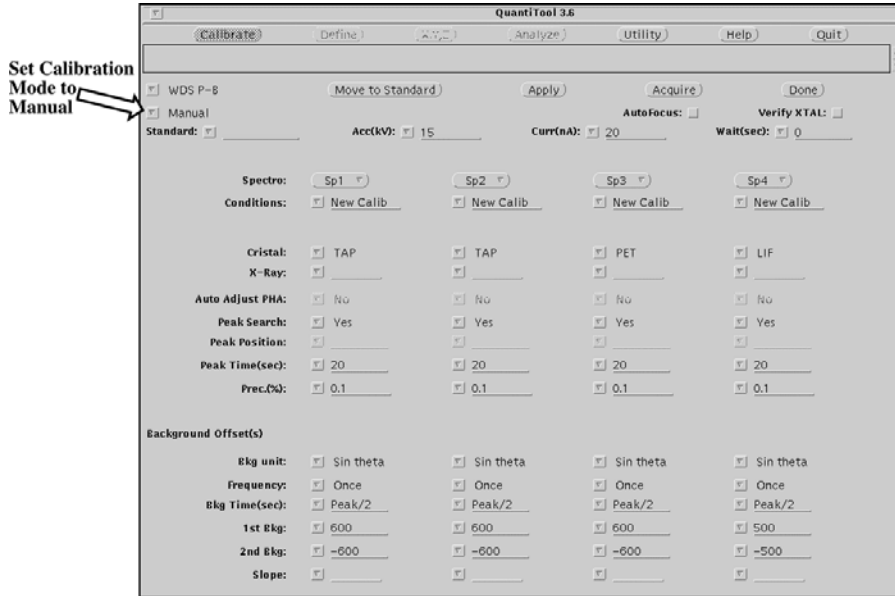
Manual Calibration

Step 1

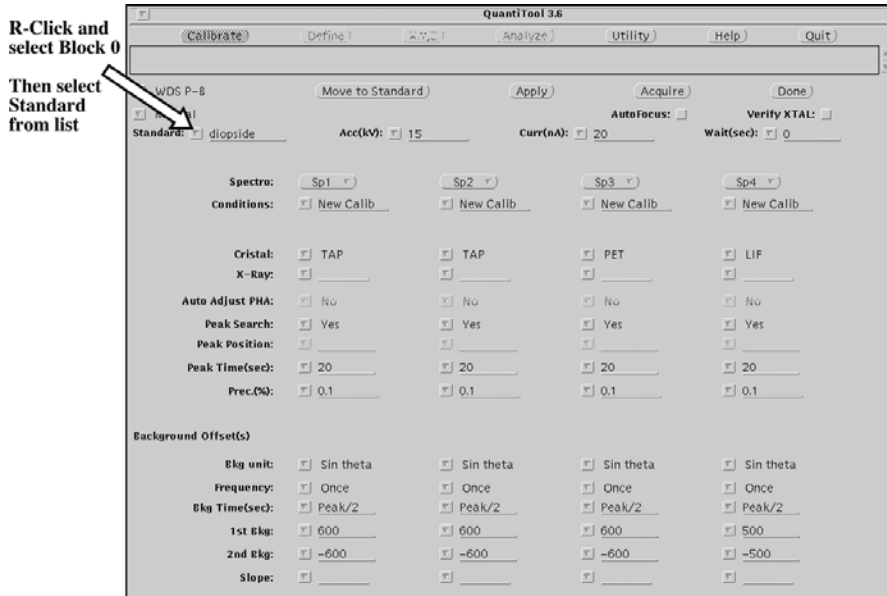
Press This Button



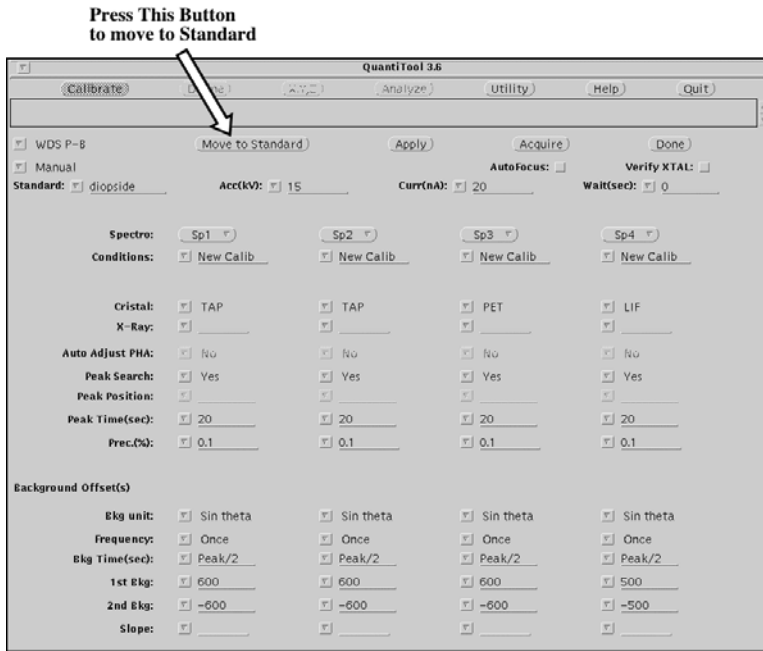
Step 2



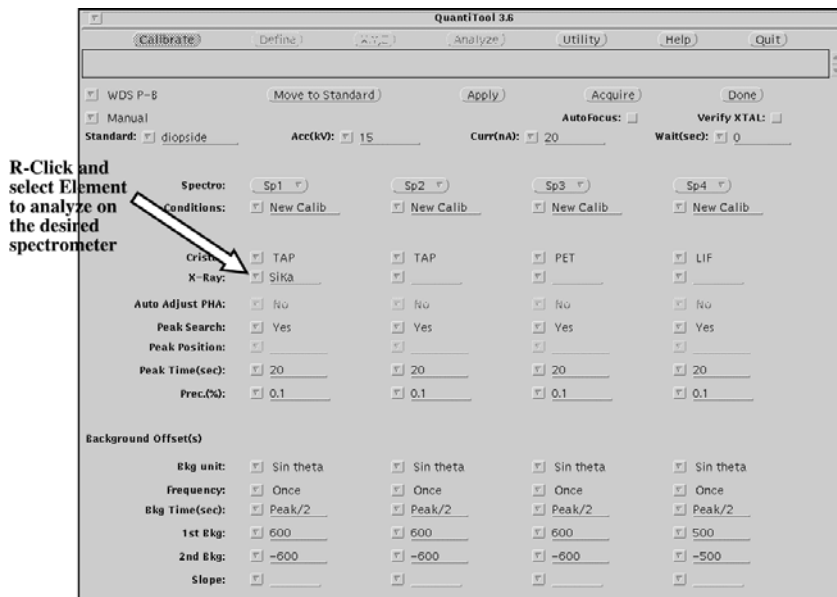
Step 3



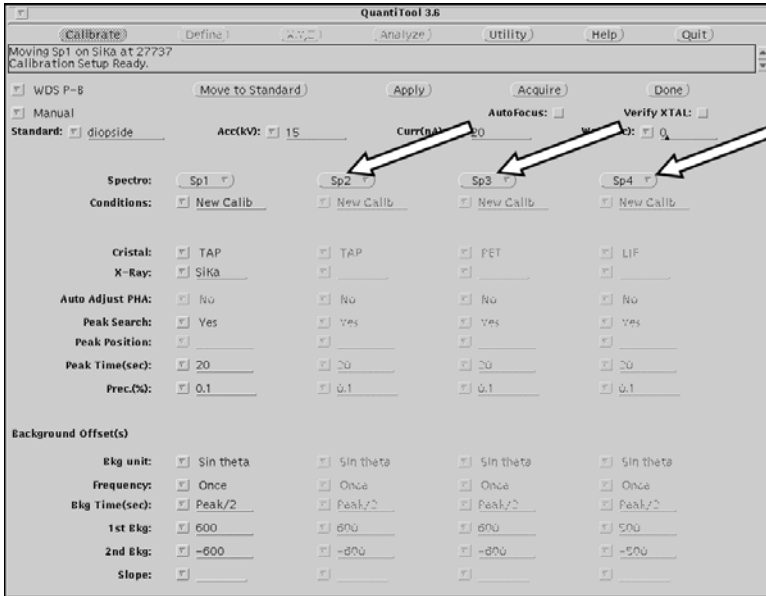
Step 4



Step 5

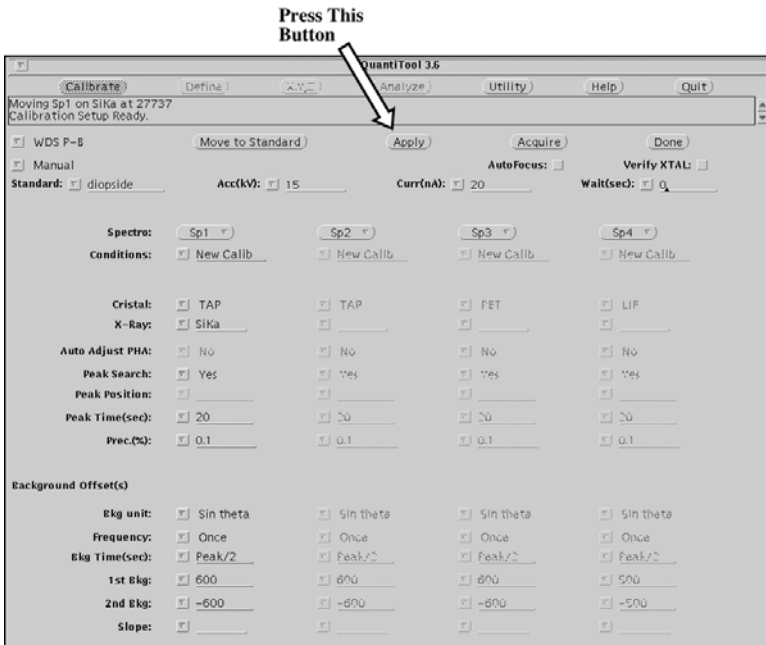


Step 6



Turn off unused spectrometers by toggling the Sp# buttons

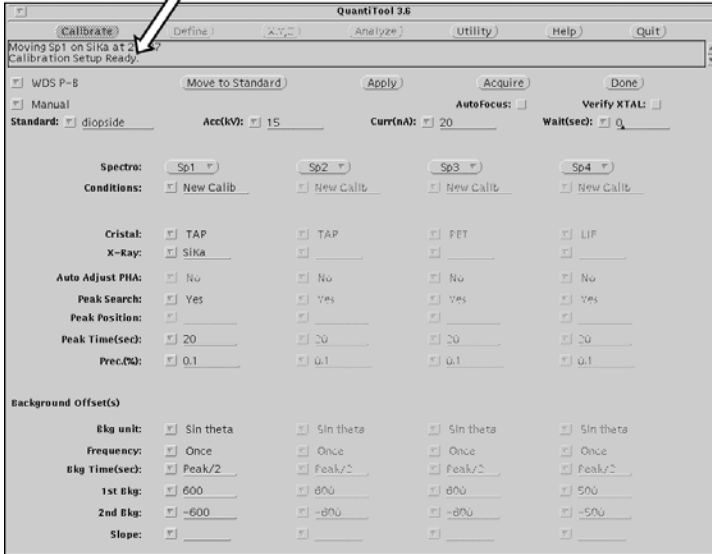
Step 7



Press This Button

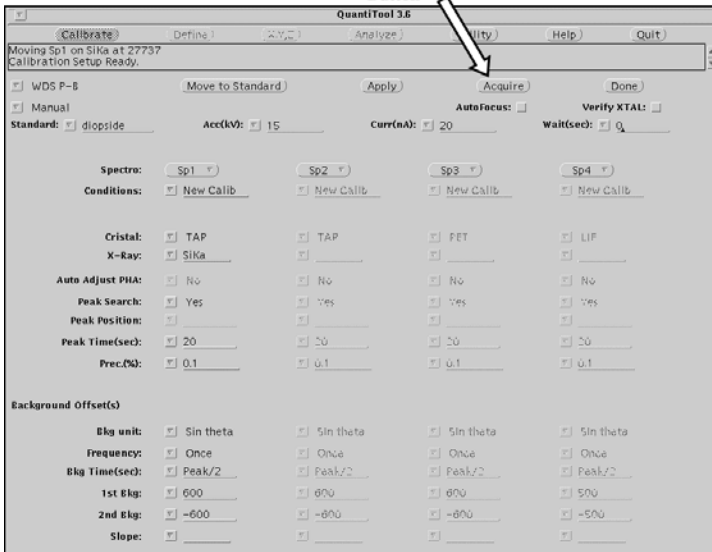
Step 8

Wait for the beep and these words to appear



Step 9

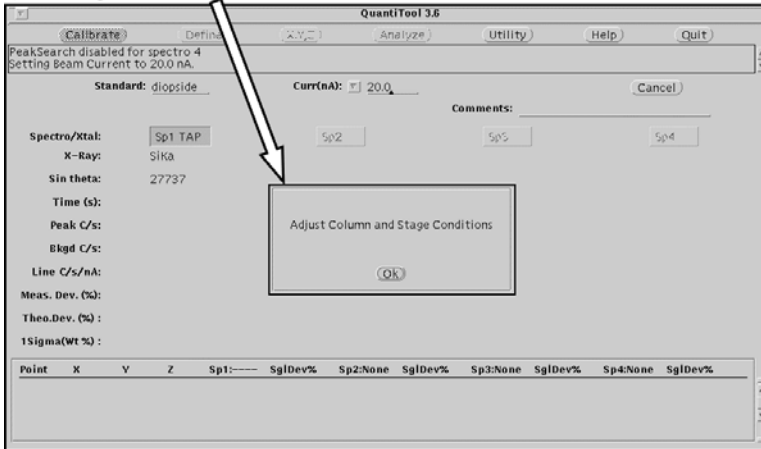
Press This Button



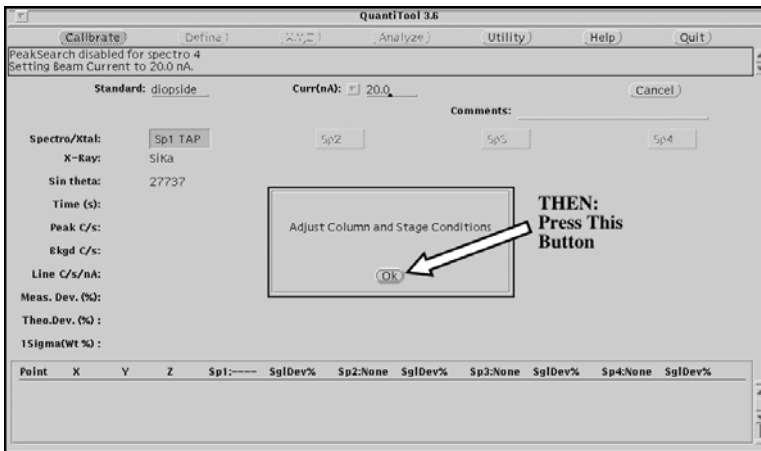
Step 10

When this screen appears:

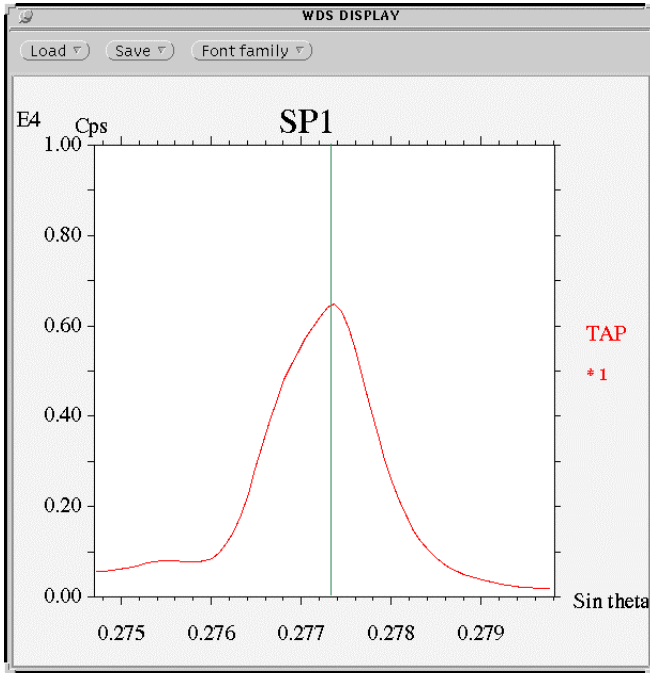
- 1) Focus
- 2) Turn Scan on - Cup Off
- 3) Find a good spot in SE video mode
- 4) Turn Cup on - Scan Off



Step 11

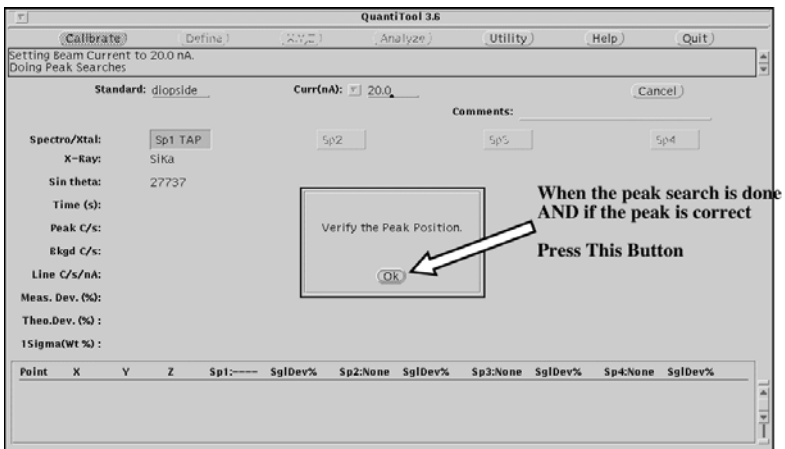


Step 12



The microprobe will do a peak search

Step 13

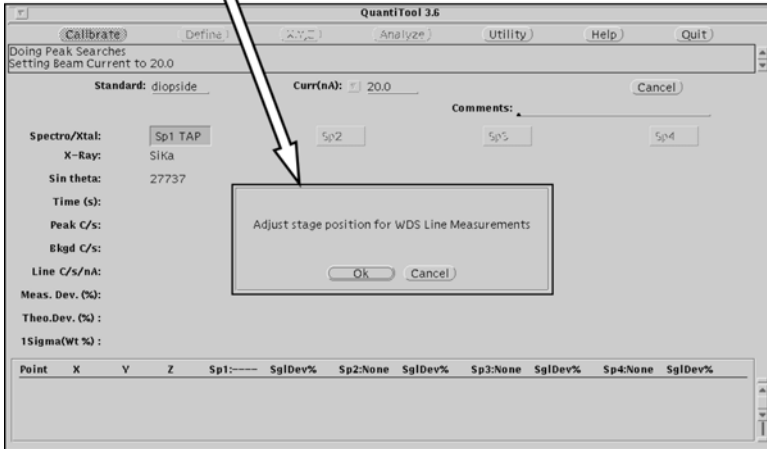


Step 14

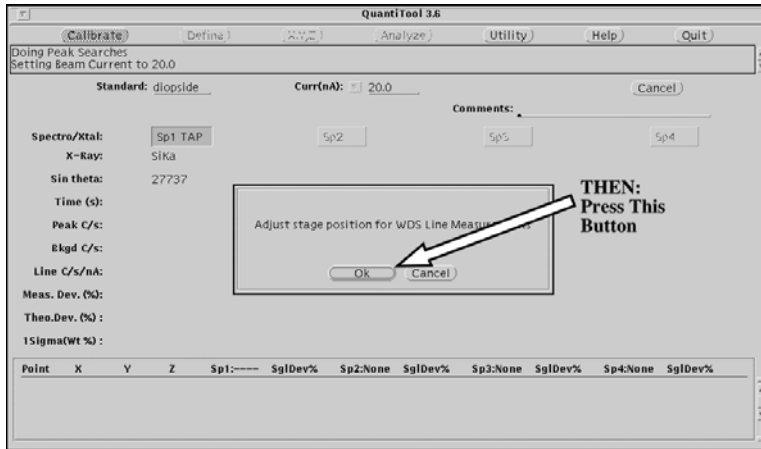


Step 15

- When this screen appears:
- 1) Turn Scan on - Cup Off
 - 2) Move to a new spot on the standard using the SE monitor
 - 3) Turn Cup on - Scan Off
 - 4) Make sure the optical focus is still good



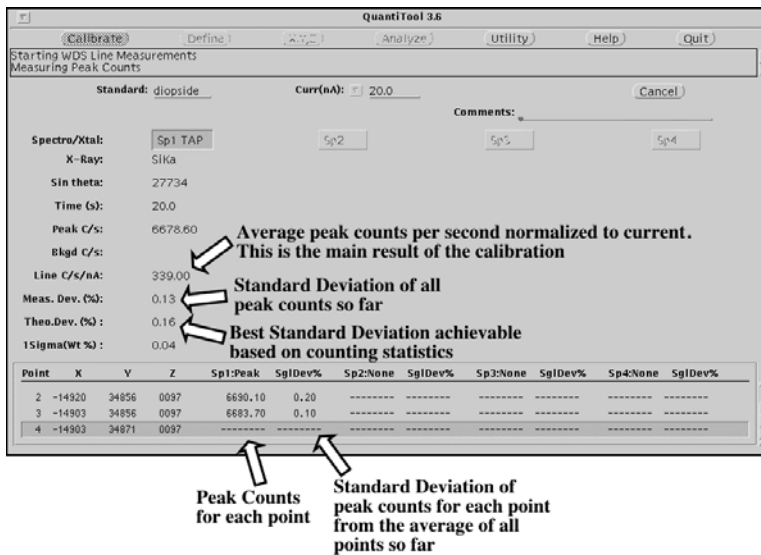
Step 16



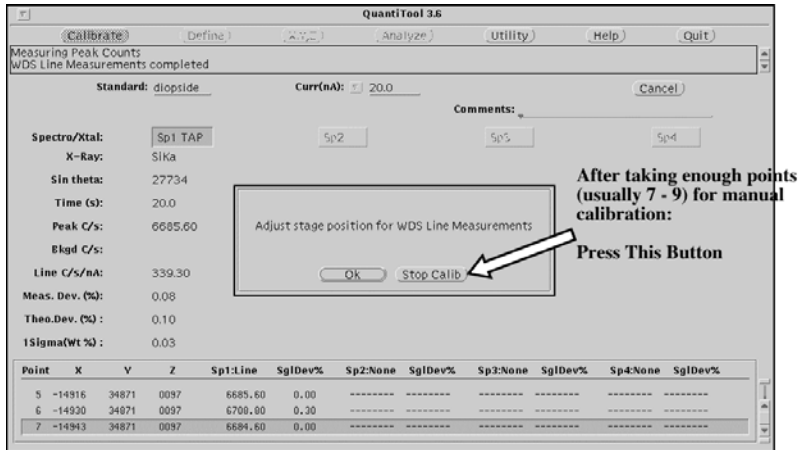
Step 17

REPEAT THE LAST TWO STEPS 7 — 9 times (for manual calibrations) so that you have 7 — 9 calibration points.

During calibration the screen will look approximately like this:

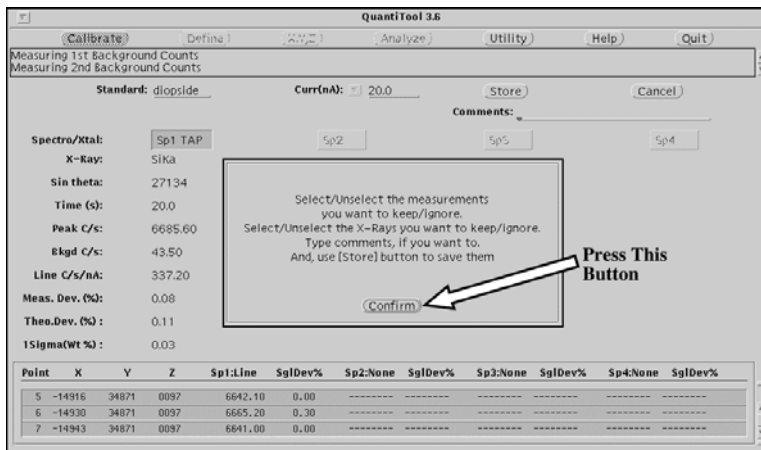


Step 18



Step 19

The microprobe will collect backgrounds and subtract them from the peak counts. When it is done:



Step 20

QuantTool 3.6

Measuring 1st Background Counts
Measuring 2nd Background Counts

Standard: diopside Curr(nA): 20.0

Spectro/Xtal: Sp1 TAP Sp2 Sp3 Sp4
X-Ray: SiKa
Sin theta: 271.34
Time (s): 20.0
Peak C/s: 6681.70
Bkgd C/s: 43.50
Line C/s/nA: 337.00
Meas. Dev. (%): 0.06
Theo. Dev. (%): 0.11
1Sigma(Wt %): 0.03

Point	X	Y	Z	Sp1:Line	SglDev%	Sp2:None	SglDev%	Sp3:None	SglDev%	Sp4:None	SglDev%
5	-14916	34871	0097	6642.10	0.10	-----	-----	-----	-----	-----	-----
6	-14930	34871	0097	6665.20	0.40	-----	-----	-----	-----	-----	-----
7	-14943	34871	0097	6641.00	0.00	-----	-----	-----	-----	-----	-----

Unselect any calibrations that appear to be obviously inaccurate. Don't unselect too many.

Note: If you have done 10 or more points; first make sure all the points are selected (dark) then unselect the ones you don't want.

Step 21

QuantTool 3.6

Measuring 1st Background Counts
Measuring 2nd Background Counts

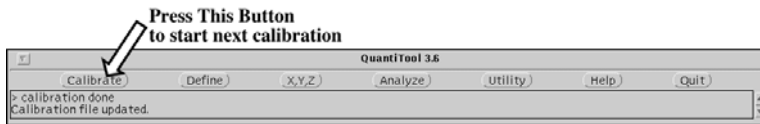
Standard: diopside Curr(nA): 20.0

Spectro/Xtal: Sp1 TAP Sp2 Sp3 Sp4
X-Ray: SiKa
Sin theta: 271.34
Time (s): 20.0
Peak C/s: 6681.70
Bkgd C/s: 43.50
Line C/s/nA: 337.00
Meas. Dev. (%): 0.06
Theo. Dev. (%): 0.11
1Sigma(Wt %): 0.03

Point	X	Y	Z	Sp1:Line	SglDev%	Sp2:None	SglDev%	Sp3:None	SglDev%	Sp4:None	SglDev%
5	-14916	34871	0097	6642.10	0.10	-----	-----	-----	-----	-----	-----
6	-14930	34871	0097	6665.20	0.40	-----	-----	-----	-----	-----	-----
7	-14943	34871	0097	6641.00	0.00	-----	-----	-----	-----	-----	-----

Press This Button

Step 22



Step 23

REPEAT STEPS 3 — 22 for each calibration.

Next Step

When finished with calibrations:

**Go to Utility
Instruction Set 3 (p. 42)**

Calibration: Instruction Set 2b

Multiple Calibration

Step 1

Set Calibration Mode to Multiple

The screenshot shows the 'Calibrate' window in QuantTool 3.6. The 'Calibrate' menu item is highlighted. The window contains several sections of controls:

- Buttons:** Calibrate, Define, Analyze, Utility, Help, Quit, Move to Standard, Apply, Acquire, Done, Point..., List..., Append, AutoFocus, Verify XTAL.
- Standard:** WDS P-B, Multiple, Standard: [dropdown], Acc(kV): 15, Curr(nA): 20, Wait(sec): 0.
- Spectro:** Sp1, Sp2, Sp3, Sp4 (dropdowns).
- Conditions:** New Calib (dropdowns).
- Cristal:** TAP, TAP, PET, LIF (dropdowns).
- X-Ray:** [dropdowns].
- Auto Adjust PHA:** No, No, No, No (dropdowns).
- Peak Search:** Yes, Yes, Yes, Yes (dropdowns).
- Peak Position:** [dropdowns].
- Peak Time(sec):** 20, 20, 20, 20 (dropdowns).
- Prec.(%):** 0.1, 0.1, 0.1, 0.1 (dropdowns).
- Background Offset(s):**
 - Bkg unit:** Sin theta, Sin theta, Sin theta, Sin theta (dropdowns).
 - Frequency:** Once, Once, Once, Once (dropdowns).
 - Bkg Time(sec):** Peak/2, Peak/2, Peak/2, Peak/2 (dropdowns).
 - 1st Bkg:** 600, 600, 600, 500 (dropdowns).
 - 2nd Bkg:** -600, -600, -600, -500 (dropdowns).
 - Slope:** [dropdowns].

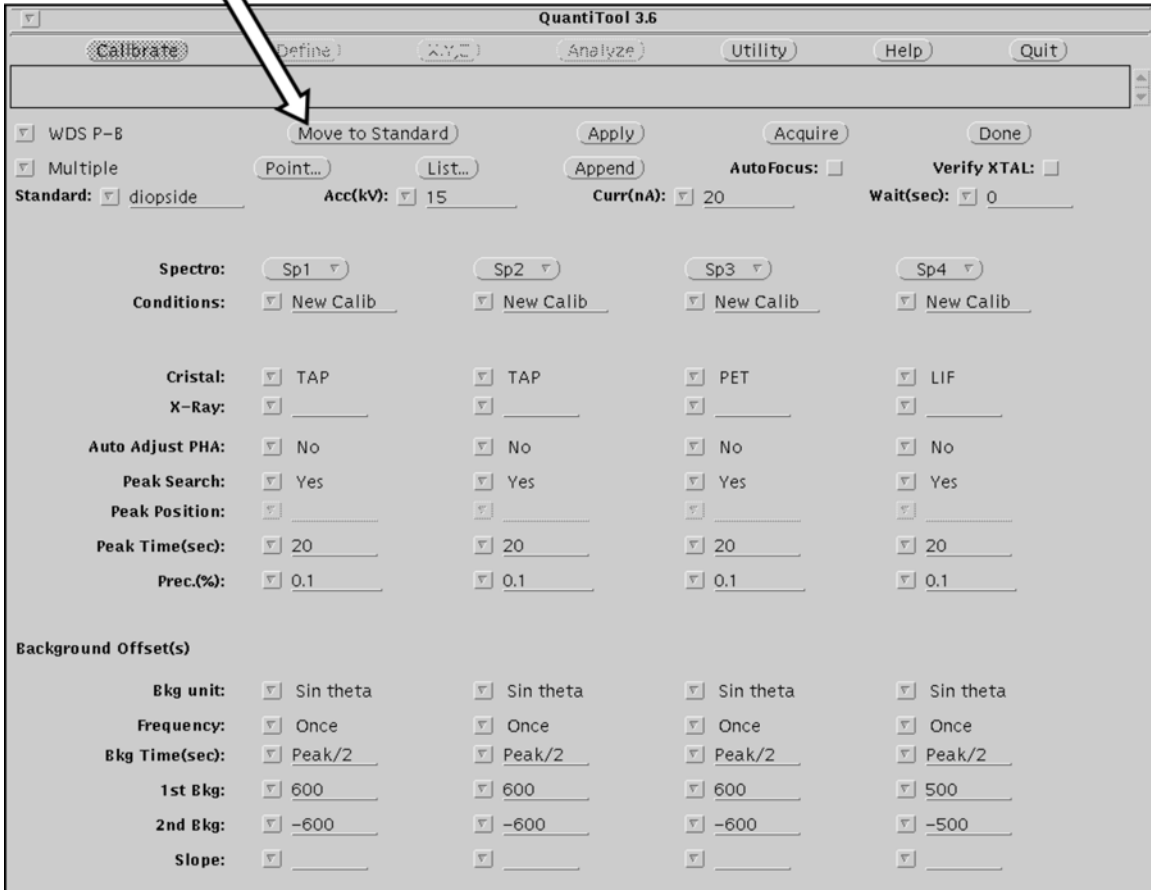
Step 2

R-Click and
Select Block 0
Then select
Standard
from List



Step 3

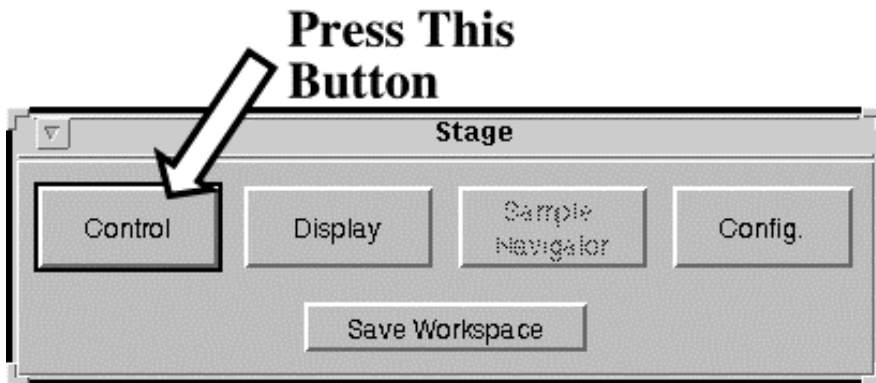
Press This Button
to move to standard



Step 4

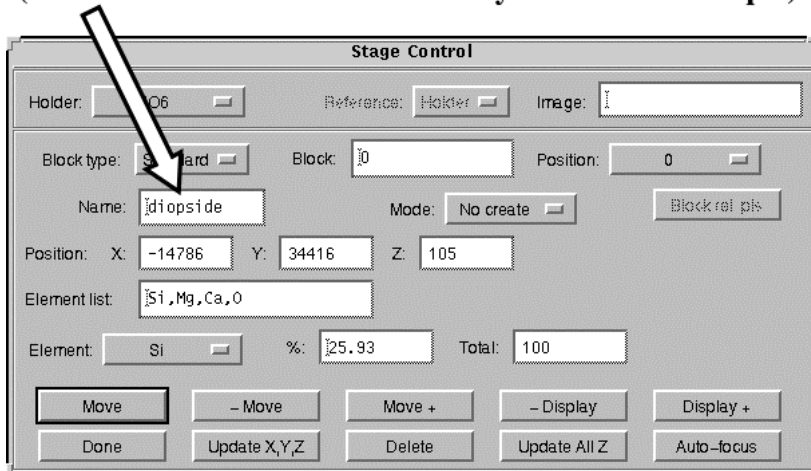
Go to the Stage window in the lower left hand corner of the computer screen. If it is not open, open it by pressing the Stage button in the Setup screen (i.e. the schematic picture of the microprobe).

THEN:



THEN:

**R-Click and select the correct Standard from the list.
(This should be the same standard you selected in Step 2)**



Step 5

- 1) Focus on Standard
- 2) Turn Scan On — Cup Off
- 3) Find a good spot in SE video mode
- 4) Turn Cup On — Scan Off

THEN:

VERY IMPORTANT! Make sure that the standard selected in the Stage Control window is the same as the standard selected in the Calibration window **AND** that you really are on that standard. **Only when you are sure - THEN:**

Holder: HO6 Reference: Hokler Image: 1

Block type: Standard Block: 10 Position: 0

Name: diopside Mode: No create Block rel pos

Position: X: -14786 Y: 34416 Z: 105

Element list: Si, Mg, Ca, O

Element: Si %: 25.93 Total: 100

Buttons: Move, - Move, Move +, - Display, Display +, Done, Update X,Y,Z, Delete, Update All Z, Auto-focus

**Press This
Button**

Step 6

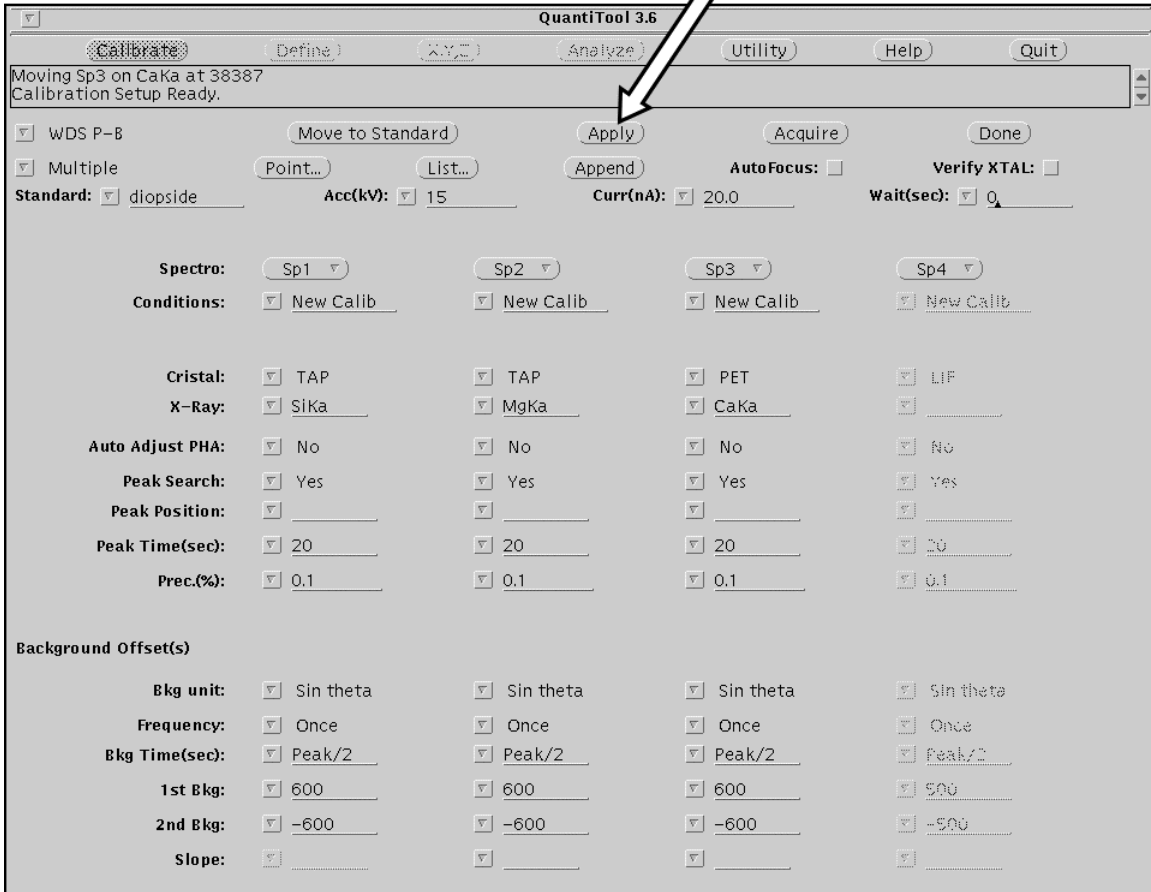
The screenshot shows the QuantiTool 3.6 software interface. At the top, there are menu options: Calibrate, Define, Analyze, Utility, Help, and Quit. A status bar at the top indicates "The selected file label already exists!". Below this, there are several control buttons: WDS P-B, Move to Standard, Apply, Acquire, and Done. A "Multiple" checkbox is checked, and there are buttons for "Point...", "List...", and "Append". The "AutoFocus" checkbox is unchecked, and "Verify XTAL" is checked. The "Standard" is set to "diopside", "Acc(kV)" is 15, "Curr(nA)" is 20.0, and "Wait(sec)" is 0. The interface is divided into four columns for spectrometers Sp1, Sp2, Sp3, and Sp4. Each column has a "Spectro:" dropdown menu and a "Conditions:" dropdown menu. Below these are various parameters for each spectrometer, including "Cristal:", "X-Ray:", "Auto Adjust PHA:", "Peak Search:", "Peak Position:", "Peak Time(sec):", and "Prec.(%)". At the bottom, there is a "Background Offset(s)" section with parameters for "Bkg unit:", "Frequency:", "Bkg Time(sec):", "1st Bkg:", "2nd Bkg:", and "Slope:". Two annotations with arrows are present: one pointing to the "Spectro:" dropdown for Sp1 with the text "R-Click and select Elements to analyze on the desired spectrometers", and another pointing to the "Spectro:" dropdown for Sp4 with the text "Turn off unused spectrometers by toggling the Sp# buttons".

R-Click and select Elements to analyze on the desired spectrometers

Turn off unused spectrometers by toggling the Sp# buttons

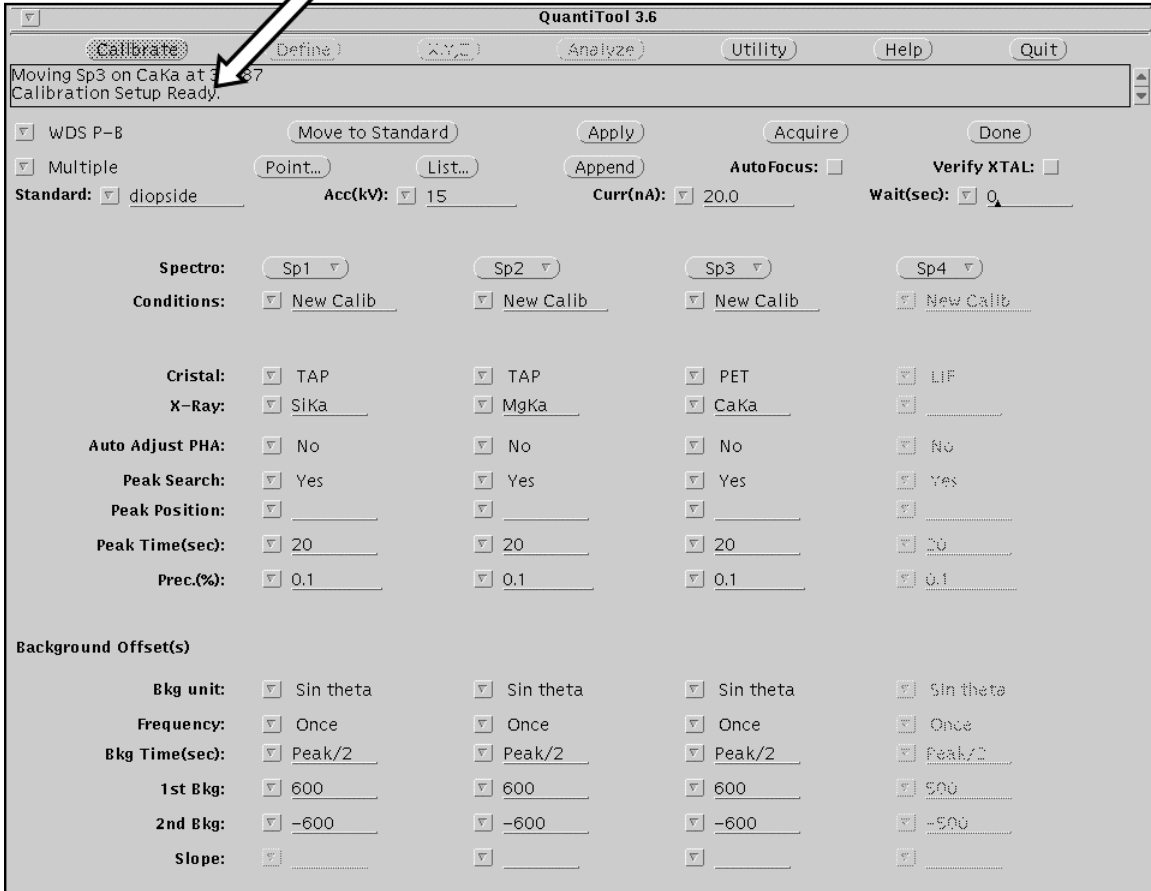
Step 7

Press This Button



Step 8

Wait for the beep and these words to appear



Step 9

Press This Button

QuantiTool 3.6

Calibrate Define X,Y,Z Analyze Utility Help Quit

Moving Sp3 on CaKa at 38387
Calibration Setup Ready.

WDS P-B Use to Standard Apply Acquire Done

Multiple Point... List... Append AutoFocus: Verify XTAL:

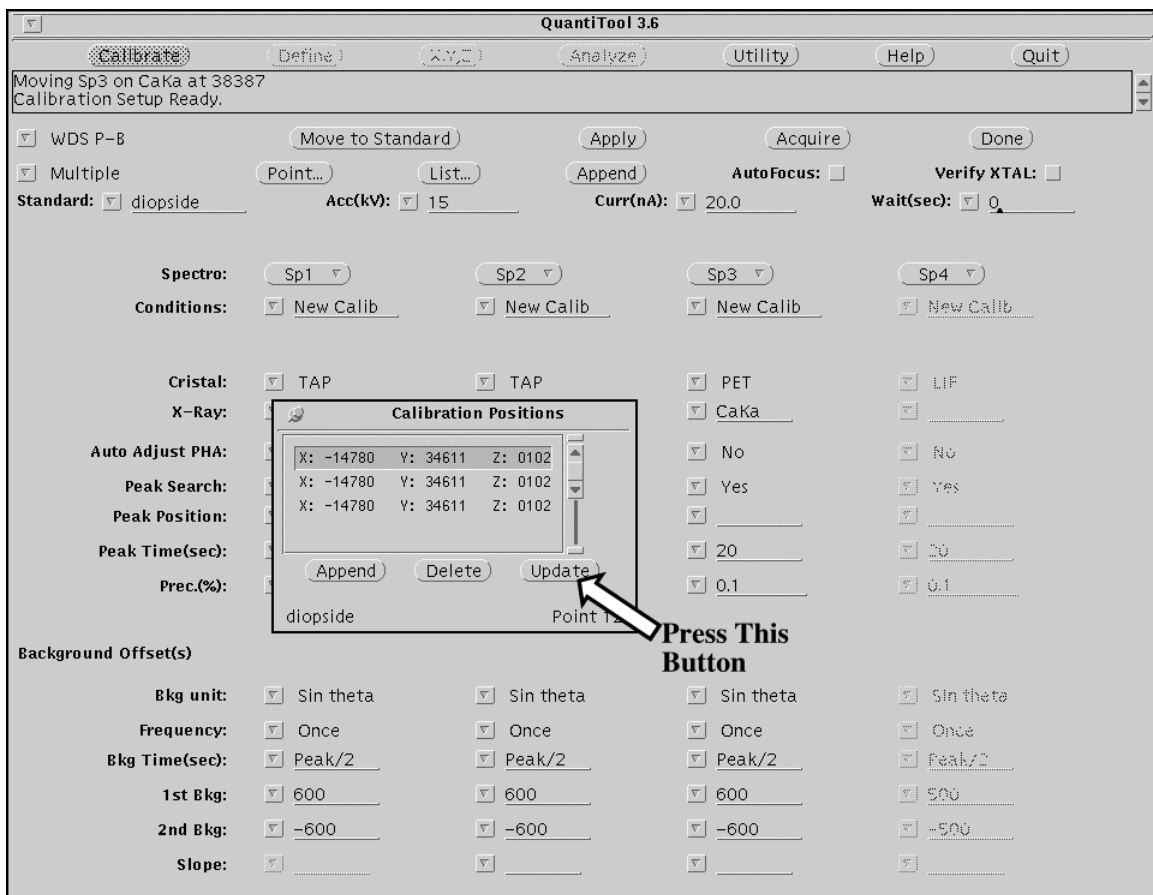
Standard: Acc(kV): Curr(nA): Wait(sec):

	Sp1	Sp2	Sp3	Sp4
Spectro:	<input type="text" value="Sp1"/>	<input type="text" value="Sp2"/>	<input type="text" value="Sp3"/>	<input type="text" value="Sp4"/>
Conditions:	<input type="text" value="New Calib"/>	<input type="text" value="New Calib"/>	<input type="text" value="New Calib"/>	<input type="text" value="New Calib"/>
Cristal:	<input type="text" value="TAP"/>	<input type="text" value="TAP"/>	<input type="text" value="PET"/>	<input type="text" value="LIF"/>
X-Ray:	<input type="text" value="SiKa"/>	<input type="text" value="MgKa"/>	<input type="text" value="CaKa"/>	<input type="text" value=""/>
Auto Adjust PHA:	<input type="text" value="No"/>	<input type="text" value="No"/>	<input type="text" value="No"/>	<input type="text" value="No"/>
Peak Search:	<input type="text" value="Yes"/>	<input type="text" value="Yes"/>	<input type="text" value="Yes"/>	<input type="text" value="Yes"/>
Peak Position:	<input type="text" value=""/>	<input type="text" value=""/>	<input type="text" value=""/>	<input type="text" value=""/>
Peak Time(Sec):	<input type="text" value="20"/>	<input type="text" value="20"/>	<input type="text" value="20"/>	<input type="text" value="20"/>
Prec.(%):	<input type="text" value="0.1"/>	<input type="text" value="0.1"/>	<input type="text" value="0.1"/>	<input type="text" value="0.1"/>
Background Offset(s)				
Bkg unit:	<input type="text" value="Sin theta"/>	<input type="text" value="Sin theta"/>	<input type="text" value="Sin theta"/>	<input type="text" value="Sin theta"/>
Frequency:	<input type="text" value="Once"/>	<input type="text" value="Once"/>	<input type="text" value="Once"/>	<input type="text" value="Once"/>
Bkg Time(Sec):	<input type="text" value="Peak/2"/>	<input type="text" value="Peak/2"/>	<input type="text" value="Peak/2"/>	<input type="text" value="Peak/2"/>
1st Bkg:	<input type="text" value="600"/>	<input type="text" value="600"/>	<input type="text" value="600"/>	<input type="text" value="500"/>
2nd Bkg:	<input type="text" value="-600"/>	<input type="text" value="-600"/>	<input type="text" value="-600"/>	<input type="text" value="-500"/>
Slope:	<input type="text" value=""/>	<input type="text" value=""/>	<input type="text" value=""/>	<input type="text" value=""/>

Step 10

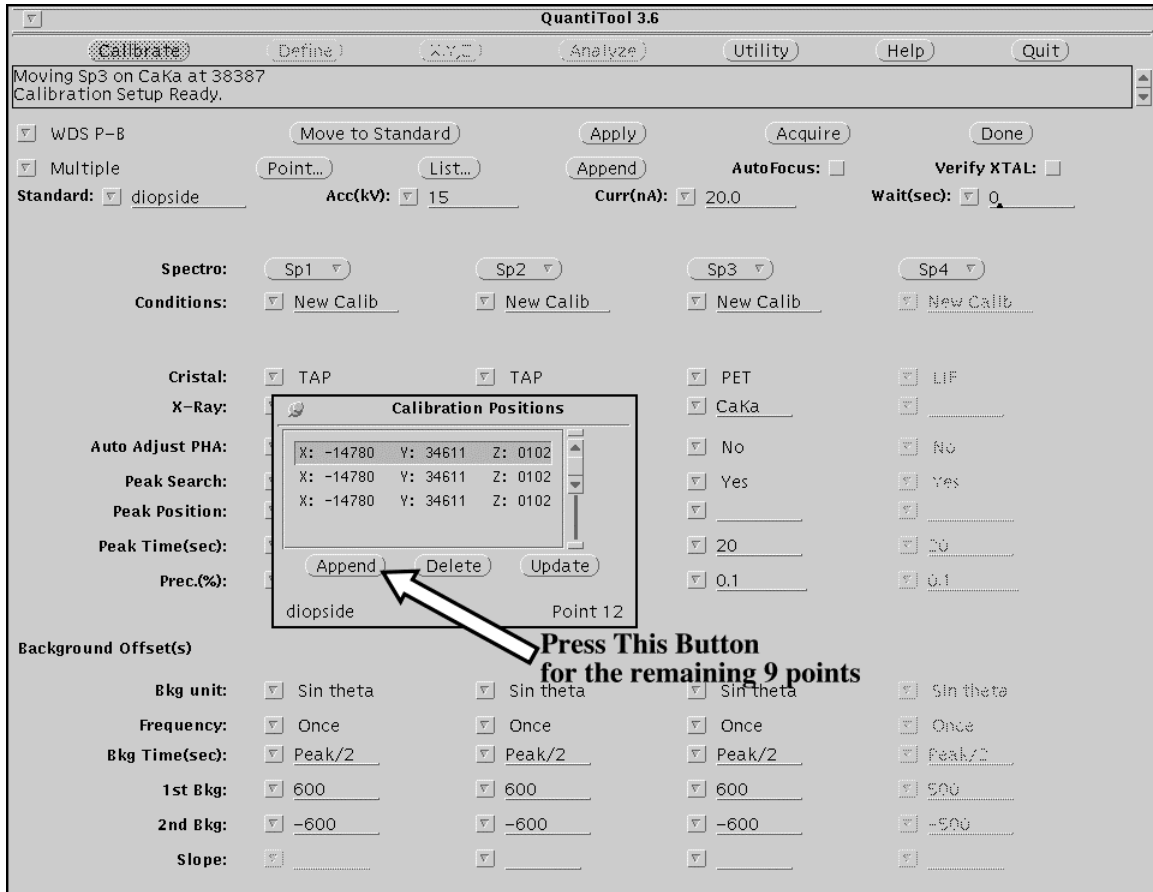
- 1) Turn Scan On — Cup Off
- 2) Move to another good spot on the standard
- 3) Make sure your still in good optical focus

THEN:



Step 11

After using the Update button to set the first three points, use the Append button to set the remaining 9 points (see below).



Step 12

After setting 12 points, turn Cup — On and Scan — Off

THEN:

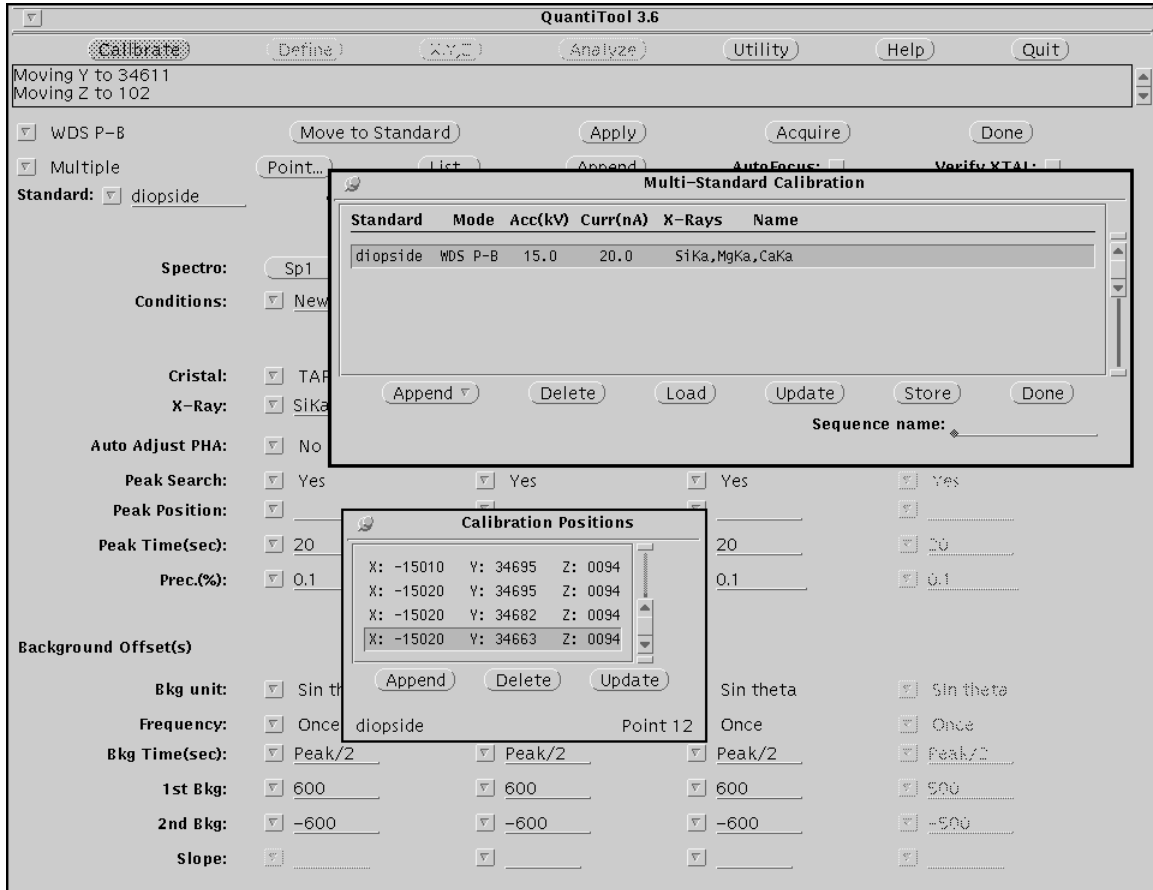
Press This Button

The screenshot shows the QuantiTool 3.6 software interface. The 'Apply' button is highlighted with a white arrow and the text 'Press This Button'. A 'Calibration Positions' dialog box is open, showing a table of X, Y, and Z coordinates for four points.

X	Y	Z
-15010	34695	0094
-15020	34695	0094
-15020	34682	0094
-15020	34663	0094

Step 13

The standard will go on the list of points to run later.



Step 14

Multi-Standard Calibration

Standard	Mode	Acc(kV)	Curr(nA)	X-Rays	Name
diopside	WDS P-B	15.0	20.0	SiKa,MgKa,CaKa	

Calibration Positions

X: -15010	Y: 34695	Z: 0094
X: -15020	Y: 34695	Z: 0094
X: -15020	Y: 34682	Z: 0094
X: -15020	Y: 34663	Z: 0094

L-Click these two pins to close the two windows

Step 15

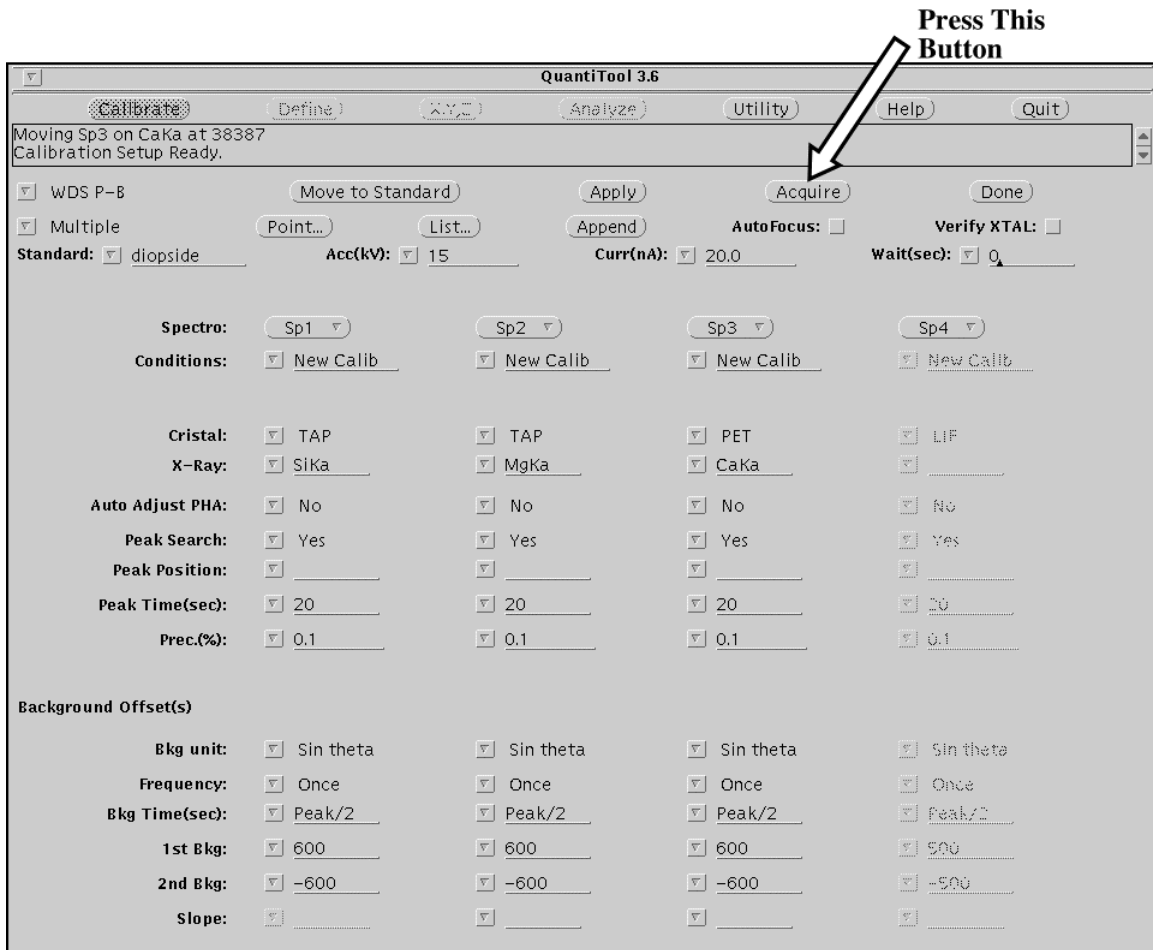
Repeat Steps 2 — 14 for each standard you are going to calibrate. When all calibrations are set up go to Step 16.

Step 16

When you are ready to start the calibrations:

VERY IMPORTANT! - Turn Cup On — Scan Off

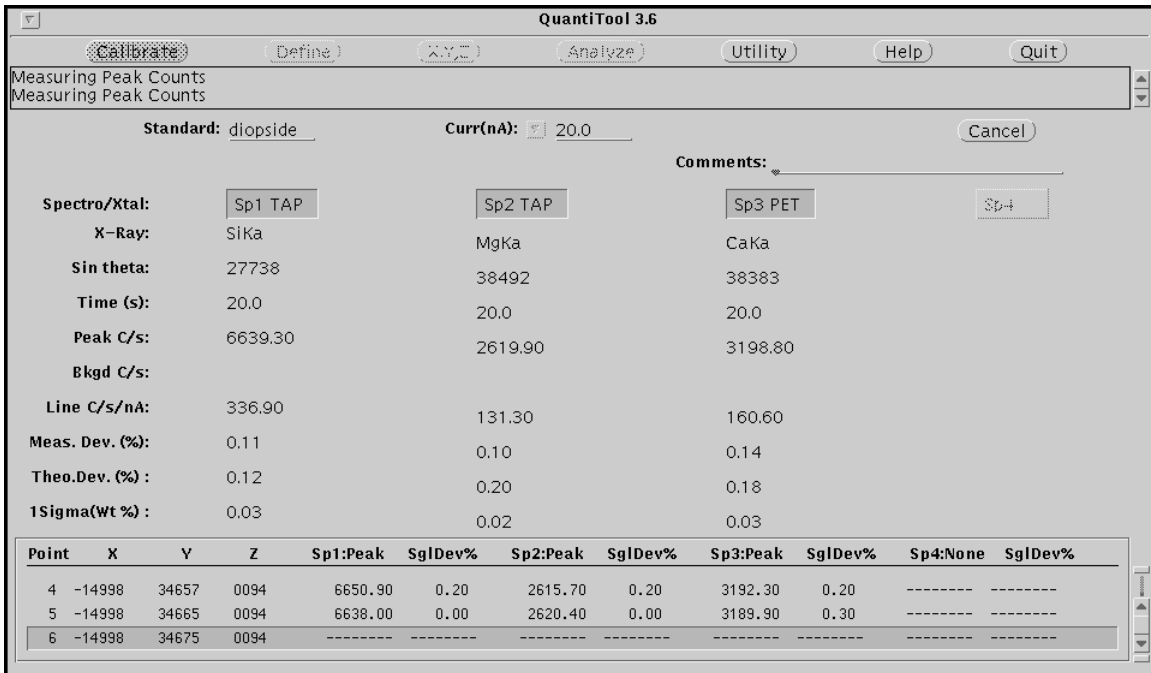
THEN:



Step 17

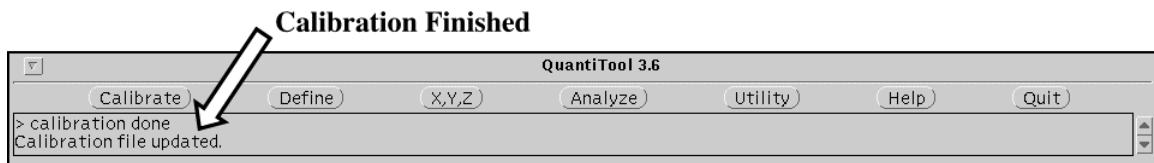
Calibration should take about 4 minutes per standard.

During calibration the screen will look like this:



Step 18

After calibration the screen will look like this:



Next Step

When finished with calibrations:

**Go to Utility
Instruction Set 3 (p. 42)**

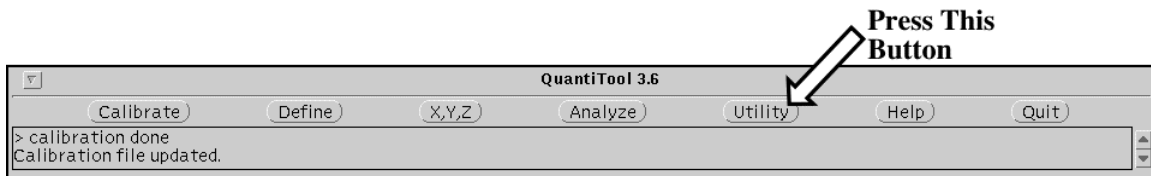
Calibration: Instruction Set 3

Utility: Erase a Calibration

Step 1

After calibrating it is necessary to erase one element calibration in order to cause the calibration database to re-write correctly. You should always include one extra calibration that you can throw away for this purpose.

In order to erase a calibration:



Step 2

The screenshot shows a 'Label Files' dialog box. The main area contains a list of files with their extensions. Below the list are buttons for 'Select All', 'Unselect', 'Delete', and 'Print List'. At the bottom, there is a 'Files' list with several options, and a 'Done' button. An annotation 'Press This Button' with an arrow points to the 'Calib WDS Peak-Bckgrd' option in the 'Files' list.

File Name	.dec	.exp	.phy	.kex	.cor	.qdf
0707Met1						
0707Sil1						
250-A						
25Aug04						
25Aug04-1						
517_1						
61604Sil1						
61604Sil2						
616o4Met1						

Buttons:

Files

- Analysis Labels
- Calib WDS Peak-Bckgrd
- Calib WDS Integral
- Calib EDS
- Calib WDS Bckgrd
- Calib Curves

Page: 1
Sort by: Z
Then by: Date

Press This Button →

Step 3

The screenshot shows a software window titled "Standards" with a table of calibration standards. Below the table are buttons for "Select All", "Unselect", "Delete", and "Print List". At the bottom, there is a "Files" list, a "Page:" dropdown set to "1", a "Change Bckgrd" button, a "Sort by:" dropdown set to "Date", a "Calib Curve" button, a "Then by:" dropdown set to "Z", and a "Done" button. Two white arrows point from the text below to the "Sort by:" and "Then by:" dropdowns.

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
Mg Ka	diopside	Sp2 TAP	15.0	20.1	38492	20	600	-600	0.00	128.799	27/Sep/04
Si Ka	diopside	Sp1 TAP	15.0	20.1	27738	20	600	-600	0.00	334.530	27/Sep/04
Ca Ka	diopside	Sp3 PET	15.0	20.1	38383	20	600	-600	0.00	159.616	27/Sep/04
Fe Ka	fayalite	Sp4 LIF	15.0	20.1	48083	20	500	-500	0.00	109.273	27/Sep/04

Files

- Analysis Labels
- Calib WDS Peak-Bckgrd
- Calib WDS Integral
- Calib EDS
- Calib WDS Bckgrd
- Calib Curves

Page: 1 Change Bckgrd

Sort by: Date Calib Curve

Then by: Z Done

Sort the calibration list first by date then by Z

Step 4

Press This Button

The screenshot shows a software window titled "Standards" with a table of data and several control buttons. An arrow points to the "Unselect" button.

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
Mg Ka	diopside	Sp2 TAP	15.0	20.1	38492	20	600	-600	0.00	128.799	27/Sep/04
Si Ka	diopside	Sp1 TAP	15.0	20.1	27738	20	600	-600	0.00	334.530	27/Sep/04
Ca Ka	diopside	Sp3 PET	15.0	20.1	38383	20	600	-600	0.00	159.616	27/Sep/04
Fe Ka	fayalite	Sp4 LIF	15.0	20.1	48083	20	500	-500	0.00	109.273	27/Sep/04

Buttons: Select All, Unselect, Delete, Print List

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
-------	----------	---------	----	----	-------	--------	------	------	-------	--------	------

Files

- Analysis Labels
- Calib WDS Peak-Bckgrd
- Calib WDS Integral
- Calib EDS
- Calib WDS Bckgrd
- Calib Curves

Page: 1 Change Bckgrd

Sort by: Date Calib Curve

Then by: Z Done

Step 5

Select the calibration
you want to erase

THEN:

Press This Button.

The screenshot shows a software window titled "Standards" with a table of calibration data. The table has columns: X-Ray, Std.Name, Spectro, kV, nA, SineT, PkTime, Bkg1, Bkg2, Slope, C/s/nA, and Date. The data rows are:

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
Mg Ka	diopside	Sp2 TAP	15.0	20.1	38492	20	600	-600	0.00	128.799	27/Sep/04
Si Ka	diopside	Sp1 TAP	15.0	20.1	27738	20	600	-600	0.00	334.530	27/Sep/04
Ca Ka	diopside	Sp3 PET	15.0	20.1	38383	20	600	-600	0.00	159.616	27/Sep/04
Fe Ka	fayalite	Sp4 LIF	15.0	20.1	48083	20	500	-500	0.00	109.273	27/Sep/04

Below the table are buttons: Select All, Unselect, Delete, and Print List. The "Delete" button is highlighted with an arrow. Below the buttons is a smaller table showing the selected row:

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
Ca Ka	diopside	Sp3 PET	15.0	20.1	38383	20	600	-600	0.00	159.616	27/Sep/04

At the bottom of the window, there is a "Files" section with a list of files: Analysis Labels, Calib WDS Peak-Bckgrd, Calib WDS Integral, Calib EDS, Calib WDS Bckgrd, and Calib Curves. To the right of the files are controls for Page (1), Sort by (Date), Then by (Z), and buttons for Change Bckgrd, Calib Curve, and Done.

Step 6

The screenshot shows the 'Standards' software interface. At the top, there is a table with columns: X-Ray, Std.Name, Spectro, kV, nA, SineT, PKTime, Bkg1, Bkg2, Slope, C/s/nA, and Date. The table contains four rows of data. Below the table are buttons for 'Select All', 'Unselect', 'Delete', and 'Print List'. A second table below shows a single row of data for 'Ca Ka diopside Sp3 PET 15.0 20.1 38383 20 600 -600 0.00 159.616 27/Sep/04'. A 'Confirm deletion' dialog box is overlaid on this row, with 'Delete' and 'Cancel' buttons. An arrow points to the 'Delete' button with the text 'Press This Button'. At the bottom, there is a 'Files' list, 'Page' and 'Sort by' dropdowns, and 'Change Bckgrd', 'Calib Curve', and 'Done' buttons.

X-Ray	Std.Name	Spectro	kV	nA	SineT	PKTime	Bkg1	Bkg2	Slope	C/s/nA	Date
Mg Ka	diopside	Sp2 TAP	15.0	20.1	38492	20	600	-600	0.00	128.799	27/Sep/04
Si Ka	diopside	Sp1 TAP	15.0	20.1	27738	20	600	-600	0.00	334.530	27/Sep/04
Ca Ka	diopside	Sp3 PET	15.0	20.1	38383	20	600	-600	0.00	159.616	27/Sep/04
Fe Ka	fayalite	Sp4 LIF	15.0	20.1	48083	20	500	-500	0.00	109.273	27/Sep/04

Buttons: Select All, Unselect, Delete, Print List

X-Ray	Std.Name	Spectro	kV	nA	SineT	PKTime	Bkg1	Bkg2	Slope	C/s/nA	Date
Ca Ka	diopside	Sp3 PET	15.0	20.1	38383	20	600	-600	0.00	159.616	27/Sep/04

Dialog Box: Confirm deletion
Buttons: Delete, Cancel

Press This Button

Files:
Analysis Labels
Calib WDS Peak-Bckgrd
Calib WDS Integral
Calib EDS
Calib WDS Bckgrd
Calib Curves

Page: 1
Sort by: Date
Then by: Z
Buttons: Change Bckgrd, Calib Curve, Done

Step 7

The screenshot shows the 'Standards' software interface. It features two tables of standards data. The top table lists four standards: Mg Ka, Si Ka, Ca Ka, and Fe Ka. The bottom table lists one standard: Ca Ka. Below the tables are several control buttons: 'Select All', 'Unselect', 'Delete', and 'Print List'. At the bottom of the interface, there is a 'Files' list, a 'Page' dropdown set to 1, a 'Sort by' dropdown set to Date, and a 'Then by' dropdown set to Z. A 'Done' button is highlighted with a white arrow pointing to it from the text 'Press This Button'.

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
Mg Ka	diopside	Sp2 TAP	15.0	20.1	38492	20	600	-600	0.00	128.799	27/Sep/04
Si Ka	diopside	Sp1 TAP	15.0	20.1	27738	20	600	-600	0.00	334.530	27/Sep/04
Ca Ka	diopside	Sp3 PET	15.0	20.1	38383	20	600	-600	0.00	159.616	27/Sep/04
Fe Ka	fayalite	Sp4 LIF	15.0	20.1	48083	20	500	-500	0.00	109.273	27/Sep/04

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
Ca Ka	diopside	Sp3 PET	15.0	20.1	38383	20	600	-600	0.00	159.616	27/Sep/04

Files

- Analysis Labels
- Calib WDS Peak-Bckgrd
- Calib WDS Integral
- Calib EDS
- Calib WDS Bckgrd
- Calib Curves

Page: 1 Change Bckgrd

Sort by: Date Calib Curve

Then by: Z Done

Press This Button

Next Step

Go to either:

**Define an Analysis File Instruction Set 4a: Create a
New File (p. 51)**

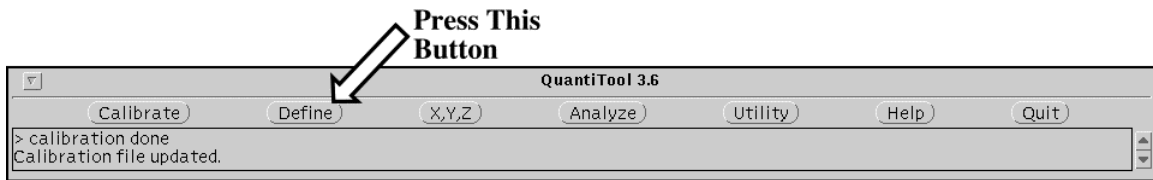
or

**Define an Analysis File Instruction Set 4b: Update an
Existing File (p. 65)**

Define an Analysis File: Instruction Set 4a

Create a New File

Step 1



Step 2

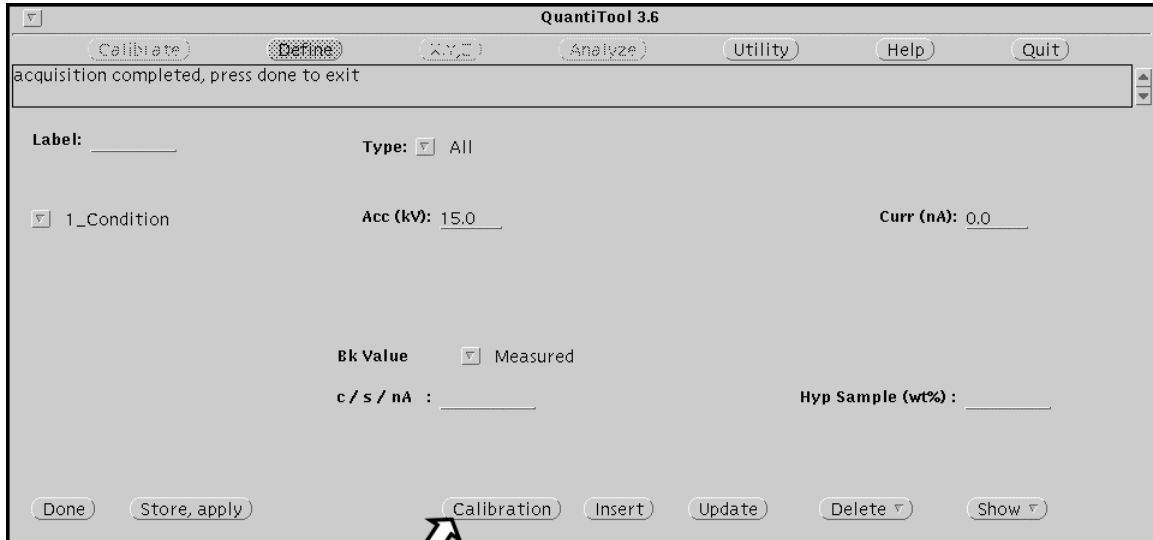
Make sure the voltage is correct

The screenshot shows a software window titled "Quantity of 3.6". The window has a menu bar with "Calibrate", "Define", "X,Y,Z", "Analyze", "Utility", "Help", and "Quit". Below the menu bar, a status bar reads "acquisition completed, press done to exit". The main area contains several input fields and dropdown menus:

- Label:** _____
- Type:** All
- 1_Condition:** 1_Condition
- Acc (kV):** 15.0
- Curr (nA):** 0.0
- Bk Value:** Measured
- c / s / nA :** _____
- Hyp Sample (wt%) :** _____

At the bottom of the window, there is a row of buttons: "Done", "Store, apply", "Calibration", "Insert", "Update", "Delete", and "Show".

Step 3



Press This
Button

Step 4

Append Elements

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
F Ka	MgF2	Sp2 TAP	15.0	20.1	71210	20	600	-600	0.00	57.926	04/Oct/04
F Ka	MgF2	Sp2 TAP	15.0	20.1	71199	20	0	-800	1.00	56.891	28/Sep/04
Na Ka	albite-Cr	Sp1 TAP	15.0	20.1	46339	20	600	-600	0.00	47.611	01/Oct/04
Na Ka	albite-Cr	Sp1 TAP	15.0	20.1	46350	20	600	-600	0.00	48.225	27/Sep/04
Mg Ka	diopside	Sp2 TAP	15.0	20.1	38495	20	600	-600	0.00	130.282	11/Oct/04
Mg Ka	diopside	Sp2 TAP	15.0	20.1	38492	20	600	-600	0.00	129.191	11/Oct/04
Al Ka	chrom-s	Sp2 TAP	15.0	20.1	32467	20	600	-600	0.00	67.196	06/Oct/04
Al Ka	anor-s	Sp1 TAP	15.0	20.1	32460	20	600	-600	0.00	249.556	06/Oct/04
Al Ka	anor-s	Sp2 TAP	15.0	20.1	32464	20	600	-600	0.00	306.051	06/Oct/04

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
-------	----------	---------	----	----	-------	--------	------	------	-------	--------	------

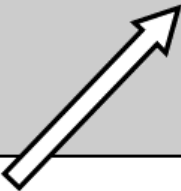
Files

- Calib WDS Peak-Bckgrd
- Calib WDS Integral
- Calib EDS

Page:

Sort by:

Then by:



Sort the calibration list in the most convenient way (usually either by Date then Z or by Z then Date).

Step 5

Select the calibrations you want in the order you want them to run (see below)

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
F Ka	MgF2	Sp2 TAP	15.0	20.1	71210	20	600	-600	0.00	57.926	04/Oct/04
F Ka	MgF2	Sp2 TAP	15.0	20.1	71199	20	0	-800	1.00	56.891	28/Sep/04
Na Ka	albite-Cr	Sp1 TAP	15.0	20.1	46339	20	600	-600	0.00	47.611	01/Oct/04
Na Ka	albite-Cr	Sp1 TAP	15.0	20.1	46350	20	600	-600	0.00	48.225	27/Sep/04
Mg Ka	diopside	Sp2 TAP	15.0	20.1	38495	20	600	-600	0.00	130.282	11/Oct/04
Mg Ka	diopside	Sp2 TAP	15.0	20.1	38492	20	600	-600	0.00	129.191	11/Oct/04
Al Ka	chrom-s	Sp2 TAP	15.0	20.1	32467	20	600	-600	0.00	67.196	06/Oct/04
Al Ka	anor-s	Sp1 TAP	15.0	20.1	32460	20	600	-600	0.00	249.556	06/Oct/04
Al Ka	anor-s	Sp2 TAP	15.0	20.1	32464	20	600	-600	0.00	306.051	06/Oct/04

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
Na Ka	albite-Cr	Sp1 TAP	15.0	20.1	46339	20	600	-600	0.00	47.611	01/Oct/04

Files

Calib WDS Peak-Bckgrd
Calib WDS Integral
Calib EDS

Page: 1
Sort by: Z
Then by: Date Done

During analysis, the elements will be analyzed (**FOR EACH SPECTROMETER**) in the order they appear from left to right in the Define window (Step 2). One way to pick calibrations is to start with Spec. 1 and pick all the elements on it in the order you want them to run and then proceed and do the same on Spec. 2, Spec 3, Spec. 4. However, other ways of picking calibrations are possible that will give the same result.

Step 6

Append Elements

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
Ti Ka	rutile1	Sp4 LIF	15.0	20.1	68279	20	500	-500	0.00	72.204	27/Sep/04
Ti Ka	rutile1	Sp3 PET	15.0	20.1	31412	20	600	-600	0.00	590.810	27/Sep/04
V Kb	v	Sp4 LIF	15.0	20.1	56739	20	500	-500	0.00	27.620	06/Oct/04
Cr Ka	chrom-s	Sp3 PET	15.0	20.1	26168	20	600	-600	0.00	371.096	06/Oct/04
Cr Ka	cr	Sp2 LIF	15.0	20.1	56868	20	500	-500	0.00	124.392	02/Oct/04
Cr Ka	cr	Sp3 PET	15.0	20.1	26165	20	600	-600	0.00	960.048	02/Oct/04
Cr Ka	cr	Sp4 LIF	15.0	20.1	56875	20	500	-500	0.00	202.508	02/Oct/04
Cr Ka	chrom-s	Sp3 PET	15.0	20.1	26167	20	600	-600	0.00	372.038	01/Oct/04
Cr Ka	chrom-s	Sp4 LIF	15.0	20.1	56872	20	500	-500	0.00	75.749	27/Sep/04

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
Na Ka	albite-Cr	Sp1 TAP	15.0	20.1	46339	20	600	-600	0.00	47.611	01/Oct/04
Mg Ka	diopside	Sp2 TAP	15.0	20.1	38495	20	600	-600	0.00	130.282	11/Oct/04
Al Ka	anor-s	Sp1 TAP	15.0	20.1	32460	20	600	-600	0.00	249.556	06/Oct/04
Si Ka	diopside	Sp1 TAP	15.0	20.1	27733	20	600	-600	0.00	335.674	11/Oct/04
K Ka	kspar-OR1	Sp3 PET	15.0	20.1	42768	20	600	-600	0.00	92.869	04/Oct/04
Ca Ka	wollast	Sp3 PET	15.0	20.1	38390	20	600	-600	0.00	303.729	07/Oct/04
Ti Ka	rutile1	Sp4 LIF	15.0	20.1	68279	20	500	-500	0.00	72.204	27/Sep/04
Cr Ka	chrom-s	Sp3 PET	15.0	20.1	26167	20	600	-600	0.00	372.038	01/Oct/04
Mn Ka	rhod-791	Sp3 PET	15.0	20.1	24020	20	600	-600	0.00	257.149	07/Oct/04

Files

- Calib WDS Peak-Bckgrd
- Calib WDS Integral
- Calib EDS

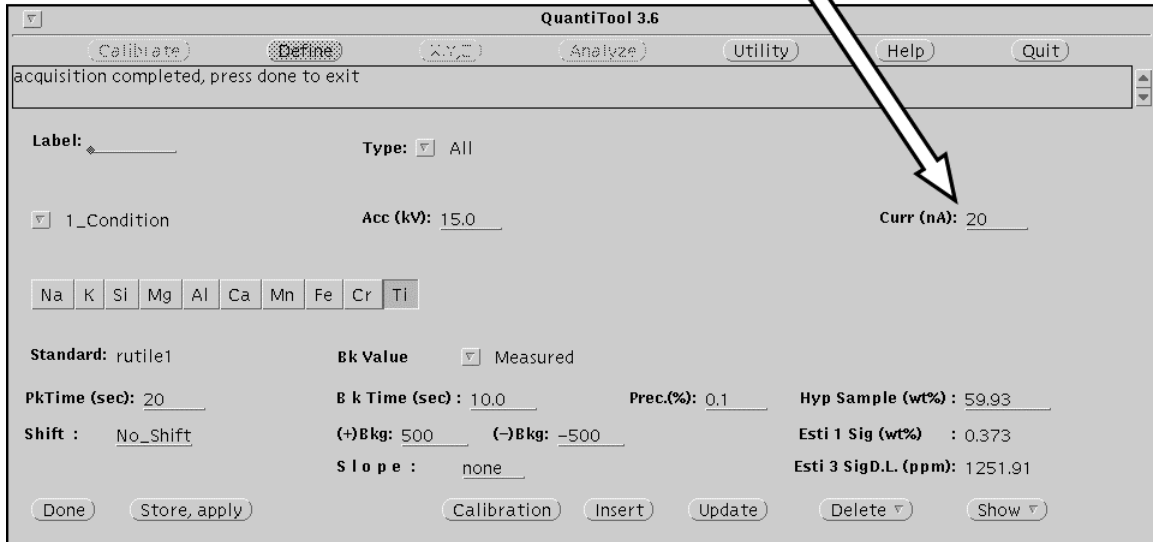
Page: 1
Sort by: Z
Then by: Date

Done

After selecting all the required calibrations: - Press This Button

Step 7

Set the Current to the desired value (usually 20 nA)



Step 8

**Set the Analysis Type:
Stoichiometric for Oxides
All for Elements**

QuantiTool 3.6

Calibrate Define Analyze Utility Help Quit

acquisition completed, press done to exit

Label: _____ Type: Stoichio. Anion: O _____

Valence: -2 _____

1_Condition Acc (kV): 15.0 _____ Curr (nA): 20 _____

Na K Si Mg Al Ca Mn Fe Cr Ti

Standard: rutile1 Bk Value Measured

PkTime (sec): 20 _____ B k Time (sec) : 10.0 _____ Prec.(%) : 0.1 _____ Hyp Sample (wt%) : 59.93 _____

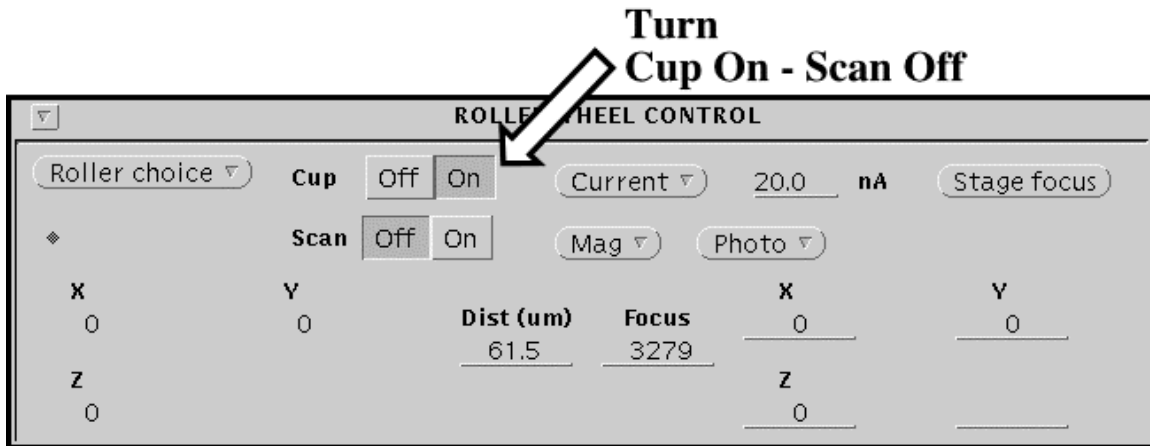
Shift : No_Shift (+)Bkg: 500 _____ (-)Bkg: -500 _____ Esti 1 Sig (wt%) : 0.373 _____

Valence: 4 _____ S l o p e : none _____ Esti 3 SigD.L. (ppm): 1251.91 _____

Done Store, apply Calibration Insert Update Delete Show

Step 9

VERY IMPORTANT! Make sure to turn Cup On — Scan Off



Step 10

acquisition completed, press done to exit

Label: test5 Type: Stoichio. Anion: O
Valence: -2
1_Condition Acc (kV): 15.0 Curr (nA): 20

Na K Si Mg Al Ca Mn Fe Cr Ti

Standard: rutile1 Bk Value: Measured

PkTime (sec): 20 B k Time (sec): 10.0 Prec.(%): 0.1 Hyp Sample (wt%): 59.93
Shift: No_Shift (+)Bkg: 500 (-)Bkg: -500 Esti 1 Sig (wt%): 0.373
Valence: 4 S l o p e : none Esti 3 SigD.L. (ppm): 1251.91

Done Store, apply Calibration Insert Update Delete Show

Press This Button

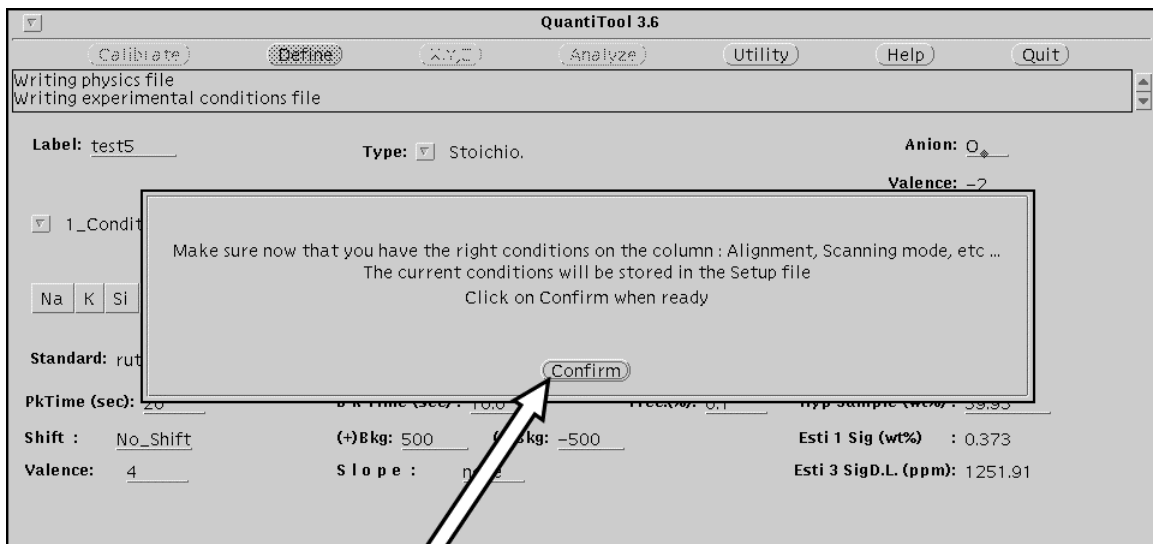
Step 11

VERY IMPORTANT — WAIT!

DO NOT try to change any settings until the following screen appears.

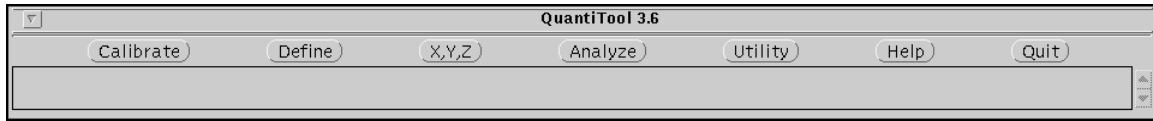
THEN: Adjust the beam size if necessary.

THEN:



**Press This
Button**

Step 12



The Define file has been stored.

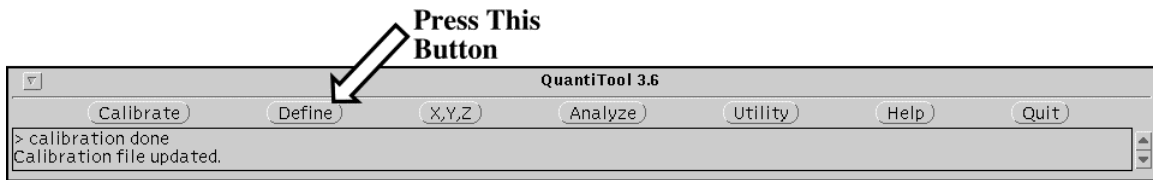
Next Step

Go to Analysis Instruction Set

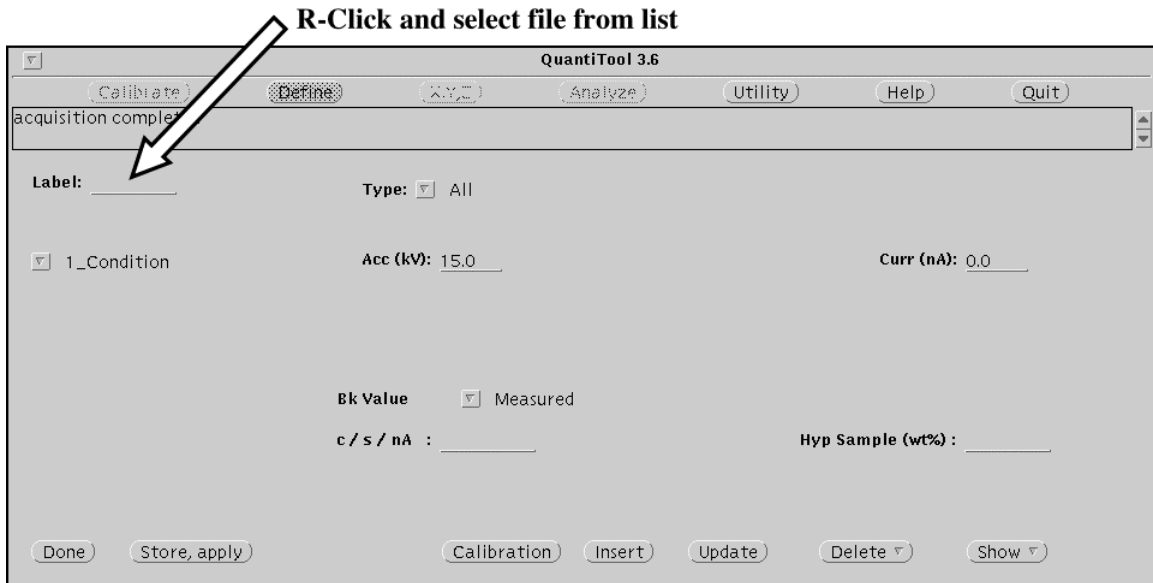
Define an Analysis File: Instruction Set 4b

Update an Existing File

Step 1



Step 2



Step 3

L-Click on the calibration you want to change

The screenshot shows the QuantiTool 3.6 software interface. At the top, there is a menu bar with buttons for Calibrate, Define, X.Y.Z, Analyze, Utility, Help, and Quit. Below the menu bar, a message box displays the text "The selected file label already exists!". The main interface contains several input fields and buttons for configuring calibration parameters. An arrow points to the 'Na' button in the element selection row.

Label: test	Type: Stoichio.	Anion: 0
<input checked="" type="checkbox"/> 1_Condition	Acc (kV): 15.0	Valence: 2
Na	K	Si
Mg	Al	Ca
Mn	Fe	Cr
Ti		
Standard: albite-Cr	Bk Value: Measured	
PkTime (sec): 20	Bk Time (sec): 10.0	Prec.(%): 0,1
Shift: No_Shift	(+)Bkg: 600	(-)Bkg: -600
Valence: 1	Slope: none	Hyp Sample (wt%): 8.77
		Esti 1 Sig (wt%): 0.068
		Esti 3 SigD.L. (ppm): 424.50

Buttons at the bottom: Done, Store, apply, Calibration, Insert, Update, Delete, Show

Step 4

QuantiTool 3.6

Calibrate Define X,Y,Z Analyze Utility Help Quit

The selected file label already exists!

Label: test Type: Stoichio. Anion: 0
Valence: 2
1_Condition Acc (kV): 15.0 Curr (nA): 20

Na K Si Mg Al Ca Mn Fe Cr Ti

Standard: albite-Cr Bk Value Measured
PkTime (sec): 20 B k Time (sec): 10.0 Prec.(%): 0.1 Hyp Sample (wt%): 8.77
Shift : No_Shift (+)Bkg: 600 (-)Bkg: -600 Esti 1 Sig (wt%) : 0.068
Valence: 1 Slope : none Esti 3 SigD.L. (ppm): 424.50

Done Store, apply Calibration Insert Update Delete Show

Press This Button

Step 5

Select a new Calibration from the list

The screenshot shows a software window titled "Update Element". It contains two identical tables, one above the other. Each table has the following columns: X-Ray, Std.Name, Spectro, kV, nA, SineT, PkTime, Bkg1, Bkg2, Slope, C/s/nA, and Date. The data in both tables is as follows:

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
Na Ka	albite-Cr	Sp1 TAP	15.0	20.1	46350	20	600	-600	0.00	48.225	27/Sep/04

Below the tables, there is a "Files" section with a list of three files: "Calib WDS Peak-Bckgrd", "Calib WDS Integral", and "Calib EDS". To the right of the files, there are controls for "Page: 1", "Sort by: Date", and "Then by: Z". A "Done" button is located at the bottom right of the window.

Note: Only Calibrations for the same element, spectrometer, crystal, and voltage are shown on the Update Element list.

Step 6

Update Element

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
Na Ka	albite-Cr	Sp1 TAP	15.0	20.1	46350	20	600	-600	0.00	48.225	27/Sep/04

X-Ray	Std.Name	Spectro	kV	nA	SineT	PkTime	Bkg1	Bkg2	Slope	C/s/nA	Date
Na Ka	albite-Cr	Sp1 TAP	15.0	20.1	46350	20	600	-600	0.00	48.225	27/Sep/04

Files

- Calib WDS Peak-Bckgrd
- Calib WDS Integral
- Calib EDS

Page:

Sort by:

Then by:

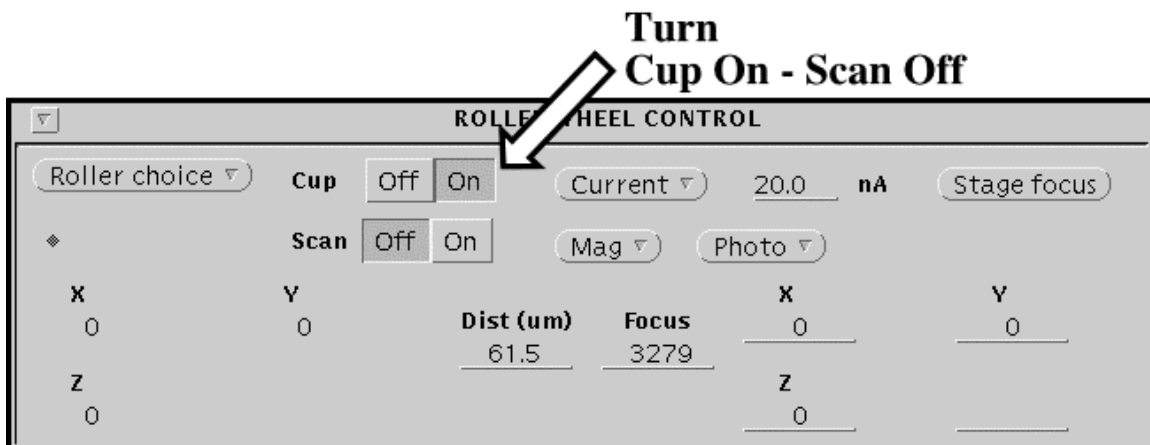
**Press This
Button**

Step 7

Repeat Steps 3 — 6 for each calibration to be changed.

THEN:

VERY IMPORTANT! Make sure the Cup is On — Scan is Off



Step 8

QuantTool 3.6

Calibrate Define Analyze Utility Help Quit

The selected file label already exists!

Label: test Type: Stoichio. Anion: 0

1_Condition Acc (kV): 15.0 Valence: 2

1 2 3 4 5 6 7 8 9 10

Standard: albite-Cr Bk Value Measured

PkTime (sec): 20 B k Time (sec): 10.0 Prec.(%): 0.1 Hyp Sample (wt%): 8.77

Shift : No_Shift (+)Bkg: 600 (-)Bkg: -600 Esti 1 Sig (wt%) : 0.068

Valence: 1 Slope : none Esti 3 SigD.L. (ppm): 424.50

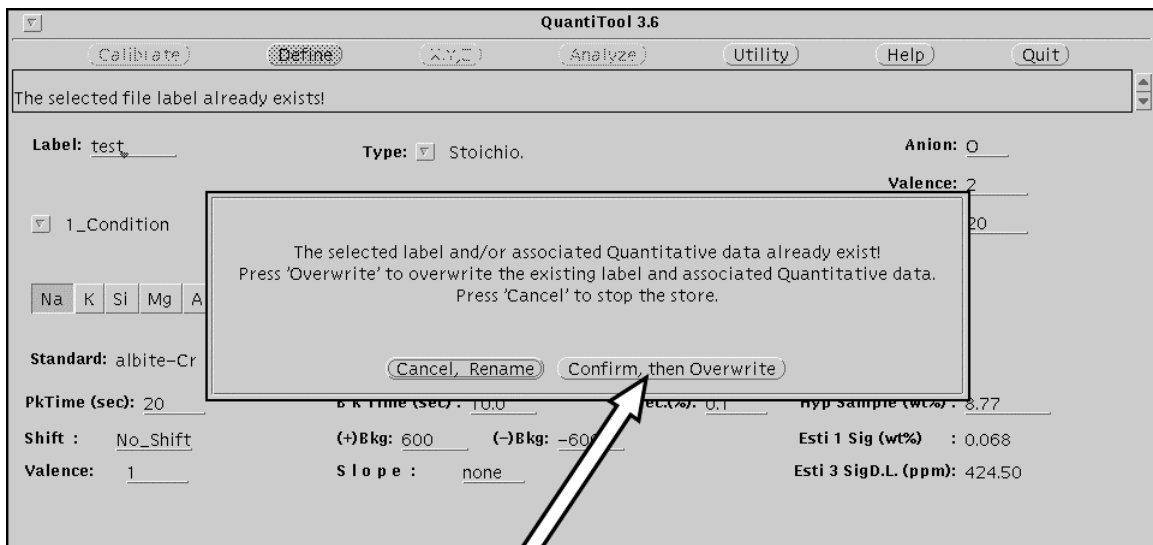
Done Store, apply Calibration Insert Update Delete Show

Press This
Button

Step 9

VERY IMPORTANT! If you are **CERTAIN** that the previous analysis data for this file has been saved or is no longer needed:

THEN:



**Press This
Button**

Note: This screen will not appear if there is no previous analysis data for this file.

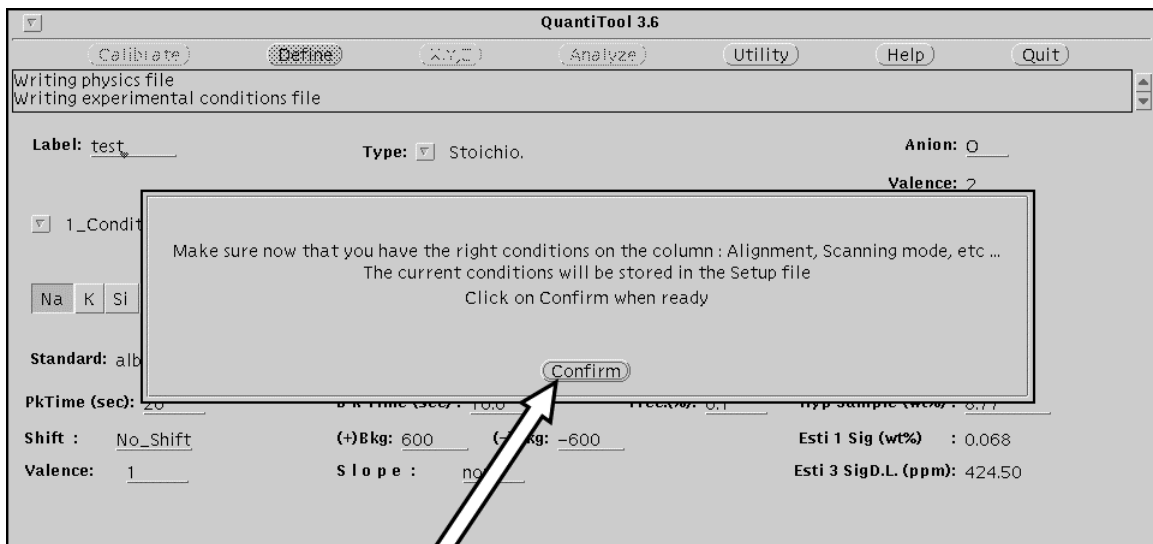
Step 10

VERY IMPORTANT — WAIT!

DO NOT try to change any settings until the following screen appears.

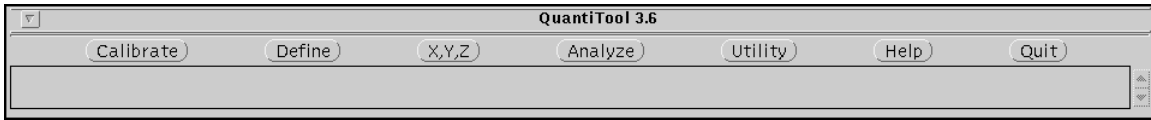
THEN: Adjust the beam size if necessary.

THEN:



**Press This
Button**

Step 11



Define file stored.

Next Step

Go to Analysis Instruction Set