# Calibration
Contents

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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

SX50

Setup

Quali

Image

Press This Button

Quanti

Tasks

Tools

TV

Mode
Multi

Status
Auto
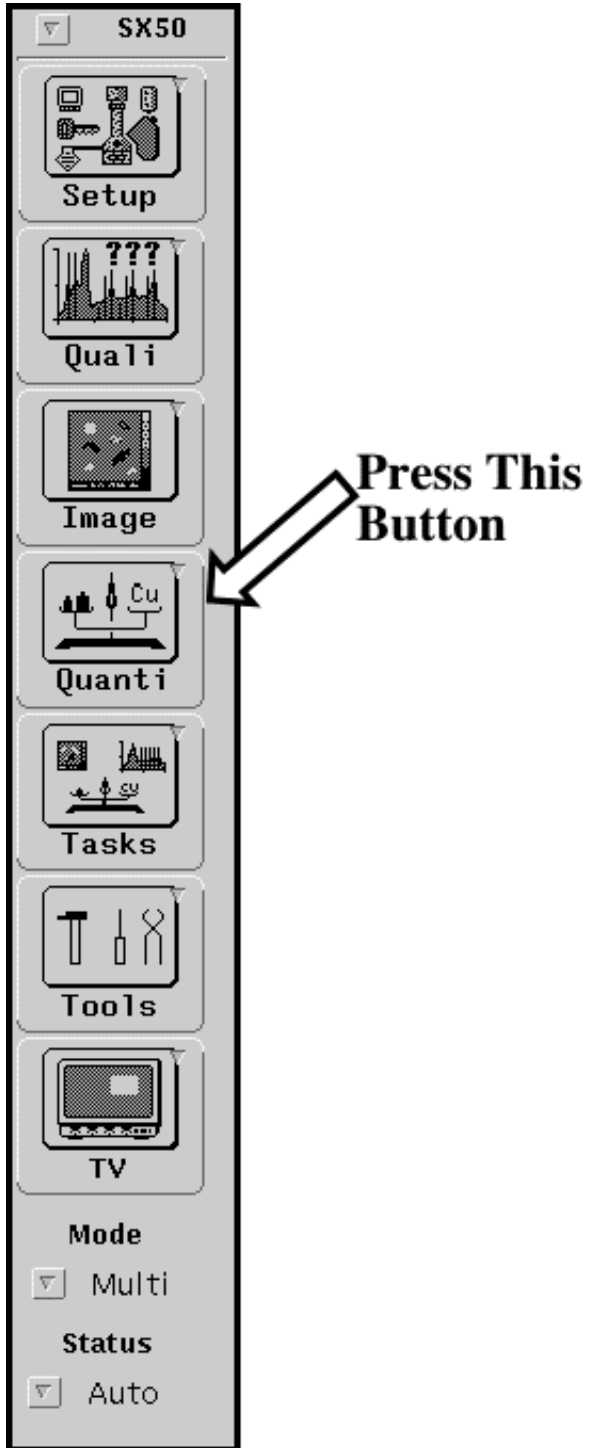
Step 2

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

Press This
Button

QUANTI

Bulk	Bulk
Coated	Coated
Layer	Layer

Set Conditions   **Process**
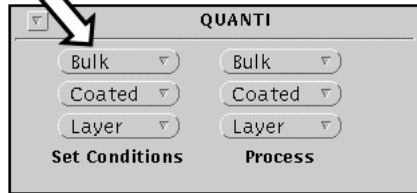
Step 3

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

Press This
Button

QuantiTool 3.6

Calibrate   Define   X,Y,Z   Analyze   Utility   Help   Quit
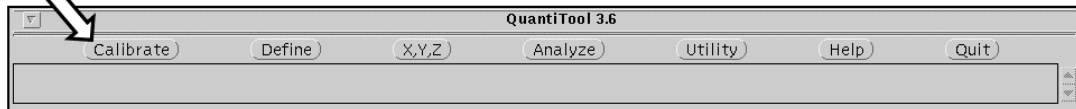
Step 4

Make sure the Voltage and Current are Correct
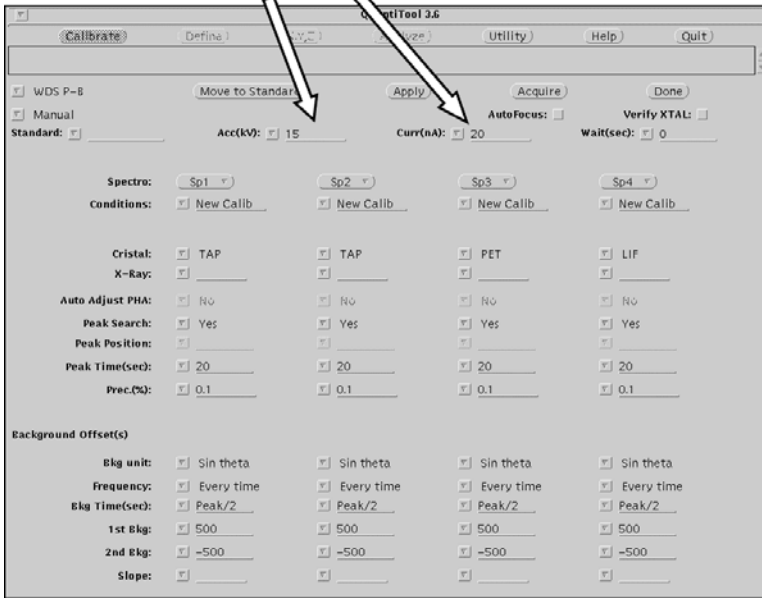
Step 5

Make sure Crystals are correct

Step 6

QuantiTool 3.6

Calibrate Define W.V. Analyze Utility Help Quit

WDS P-B   Move to Standard   Apply   Acquire   Done
Manual   AutoFocus: Verify XTAL:
Standard: Acc(kV): 15   Curr(nA): 20   Wait(sec): 0

Spectro:	Sp1	Sp2	Sp3	Sp4
Conditions:	New Calib	New Calib	New Calib	New Calib
Cristal:	TAP	TAP	PET	LIF
X-Ray:				
Auto Adjust PHA:	No	No	No	No
Peak Search:	Yes	Yes	Yes	Yes
Peak Position:				
Peak Time(sec):	20	20	20	20
Prec.(%):	0.1	0.1	0.1	0.1

Background Offset(s)

Bkg unit:	Sin theta	Sin theta	Sin theta	Sin theta
Frequency:	Once	Once	Once	Once
Bkg Time(sec):	Peak/2	Peak/2	Peak/2	Peak/2
1st Bkg:	500	500	500	500
2nd Bkg:	-500	-500	-500	-500
Slope:				

Set background frequency to Once →

Step 7

QuantiTool 3.6

Calibrate Define W.V. Analyze Utility Help Quit

WDS P-B   Move to Standard   Apply   Acquire   Done
Manual   AutoFocus: Verify XTAL:
Standard: Acc(kV): 15   Curr(nA): 20   Wait(sec): 0

Spectro:	Sp1	Sp2	Sp3	Sp4
Conditions:	New Calib	New Calib	New Calib	New Calib
Cristal:	TAP	TAP	PET	LIF
X-Ray:				
Auto Adjust PHA:	No	No	No	No
Peak Search:	Yes	Yes	Yes	Yes
Peak Position:				
Peak Time(sec):	20	20	20	20
Prec.(%):	0.1	0.1	0.1	0.1

Background Offset(s)

Bkg unit:	Sin theta	Sin theta	Sin theta	Sin theta
Frequency:	Once	Once	Once	Once
Bkg Time(sec):	Peak/2	Peak/2	Peak/2	Peak/2
1st Bkg:	600	600	600	500
2nd Bkg:	-600	-600	-600	-500
Slope:				

Set backgrounds to correct values (see below) →

Default Backrounds

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

Next Step

Go to either:
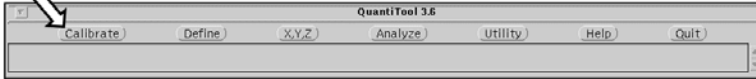**Manual Calibration Instruction Set 2a (p. 8)**
or
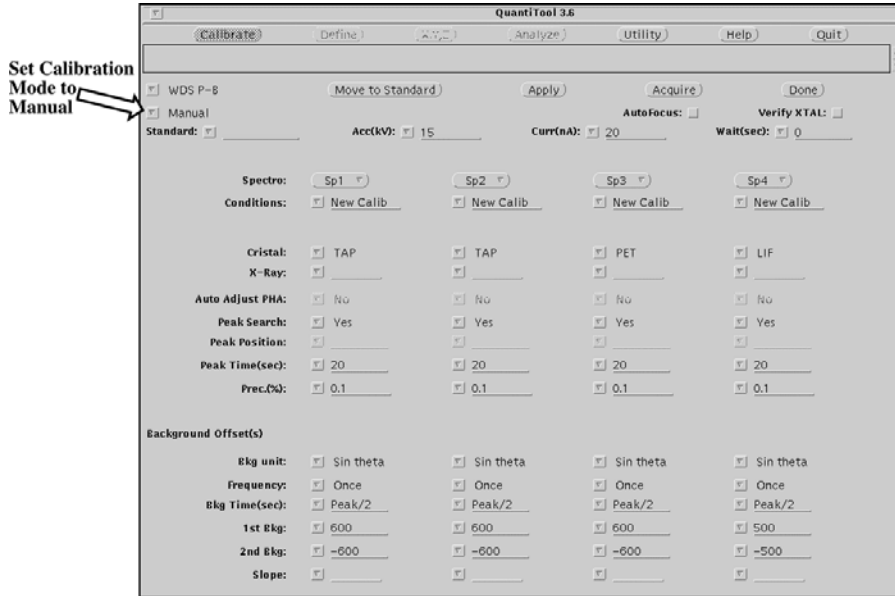**Multiple Calibration Instruction Set 2b (p. 22)**

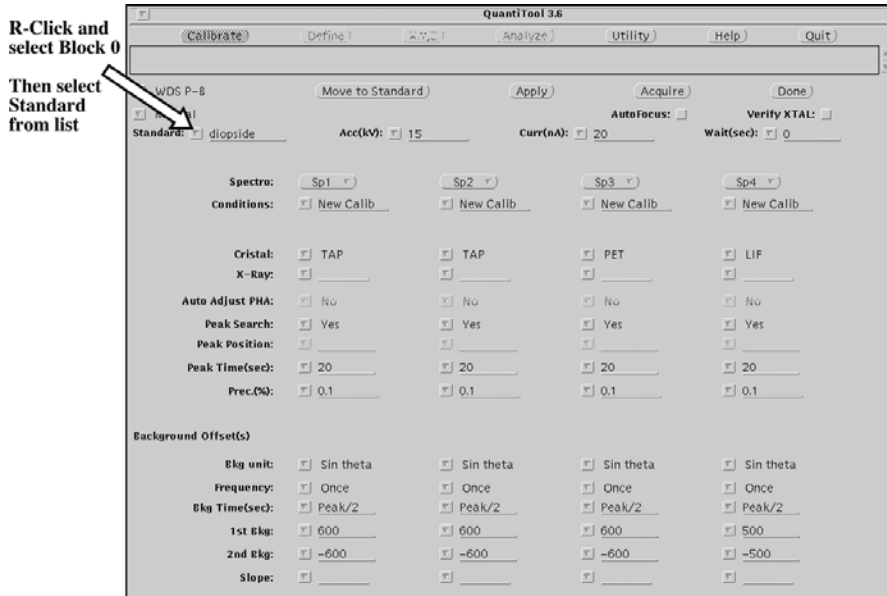# Calibration: Instruction Set 2a
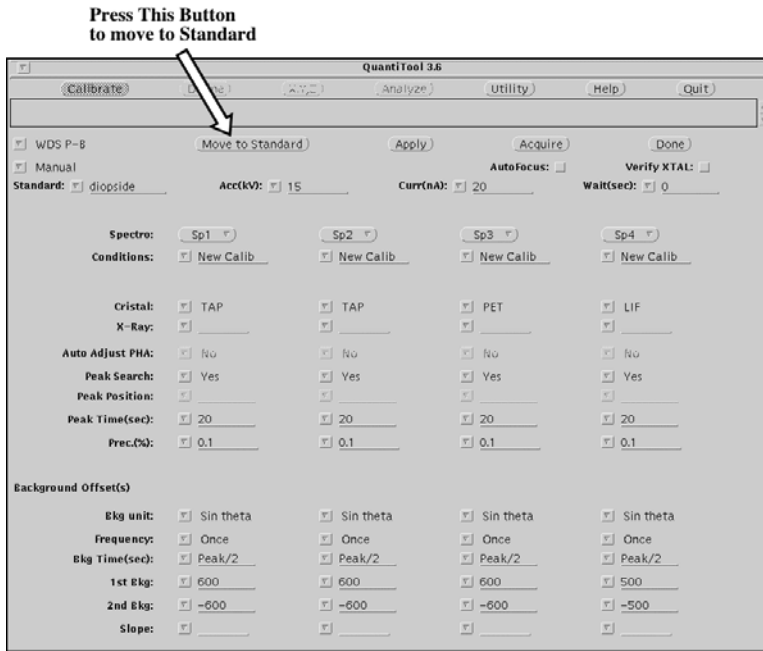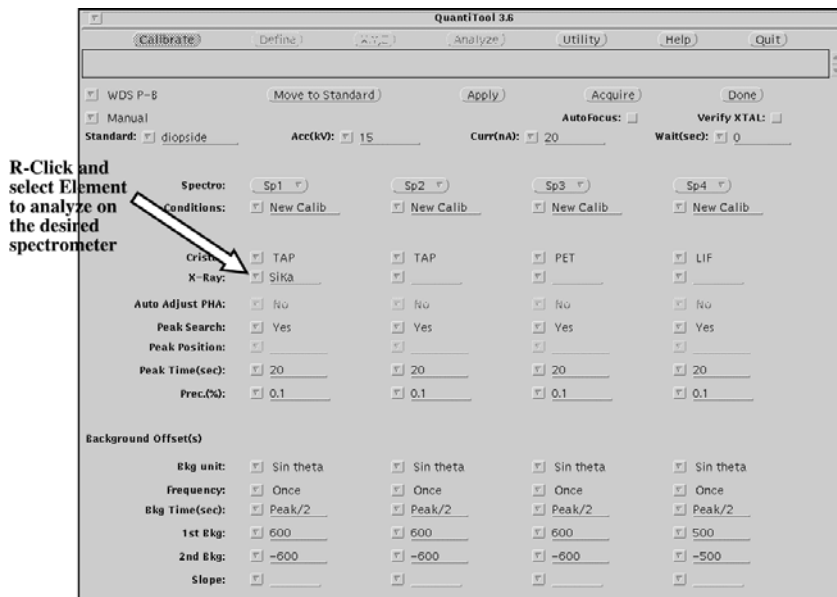
Manual Calibration

Step 1

Press This
Button
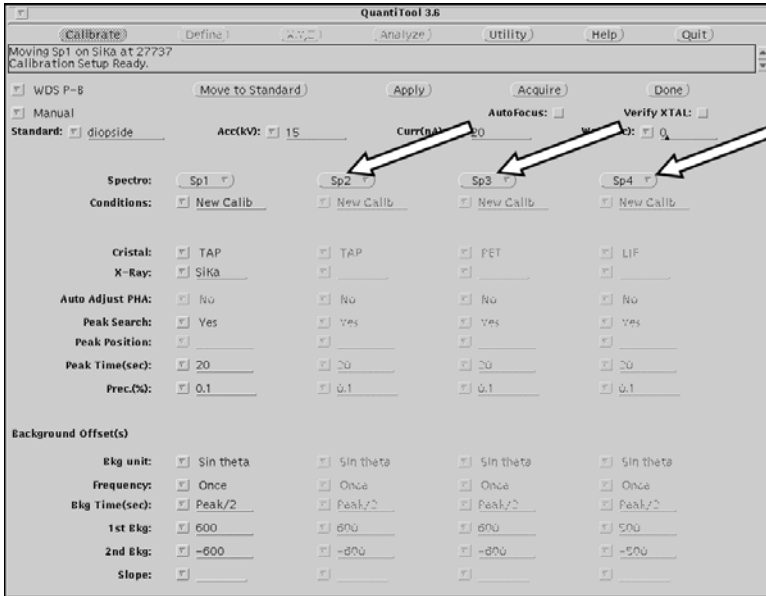
QuantiTool 3.6

Calibrate   Define   X,Y,Z   Analyze   Utility   Help   Quit

Step 2

Set Calibration
Mode to
Manual

Step 3

R-Click and
select Block 0

Then select
Standard
from list

Step 4

Press This Button
to move to Standard

Step 5

R-Click and
select Element
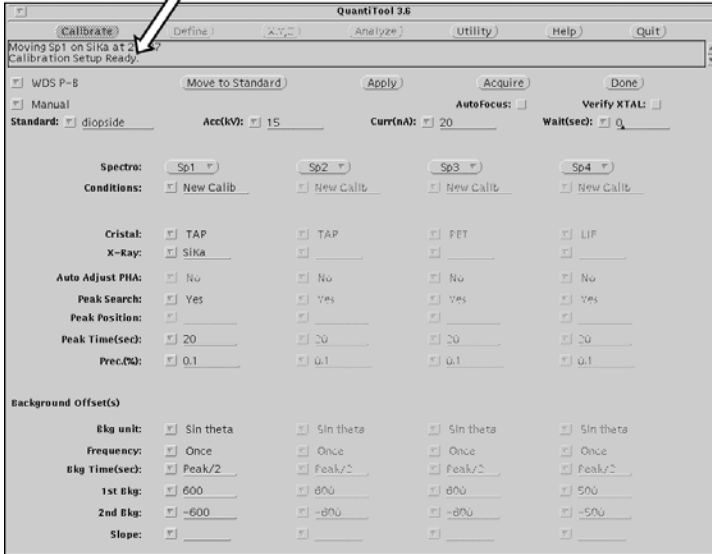to analyze on
the desired
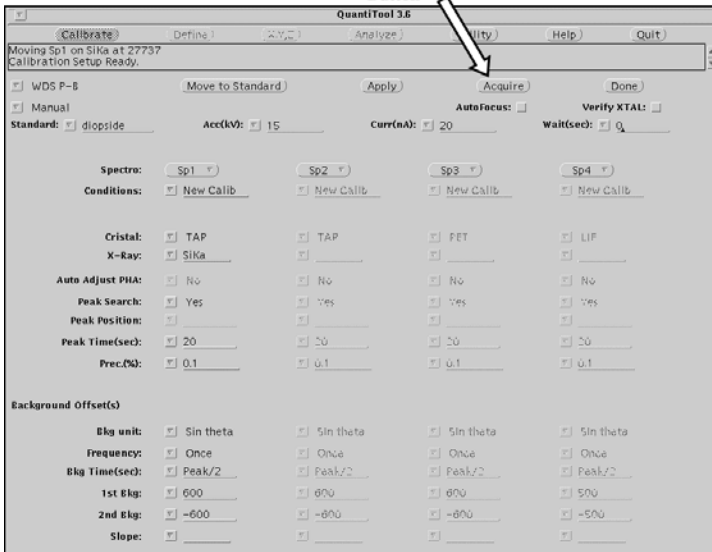spectrometer

Step 6

Step 7
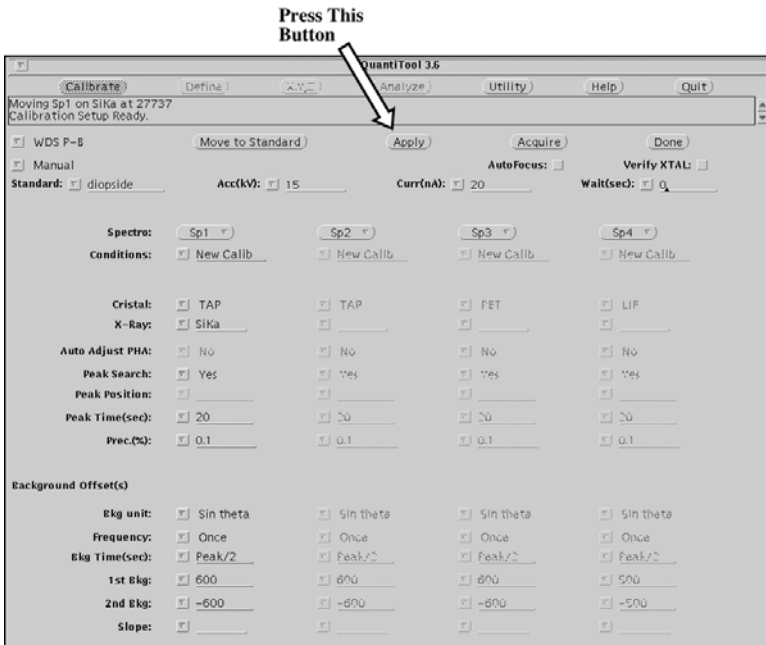
Step 8
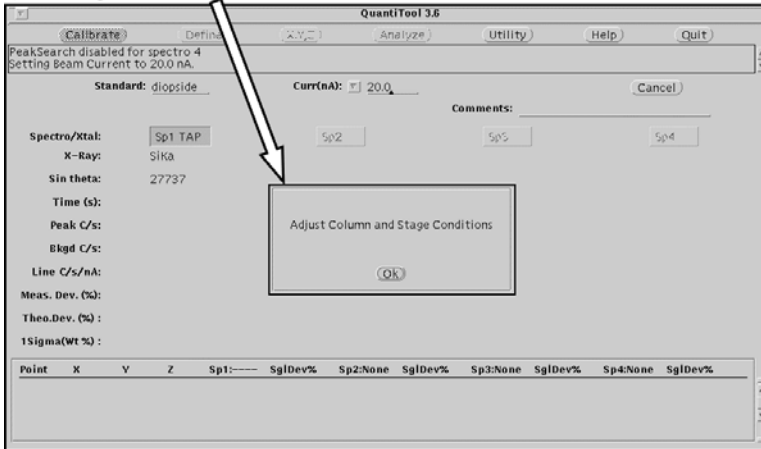
Wait for the beep and these words to appear
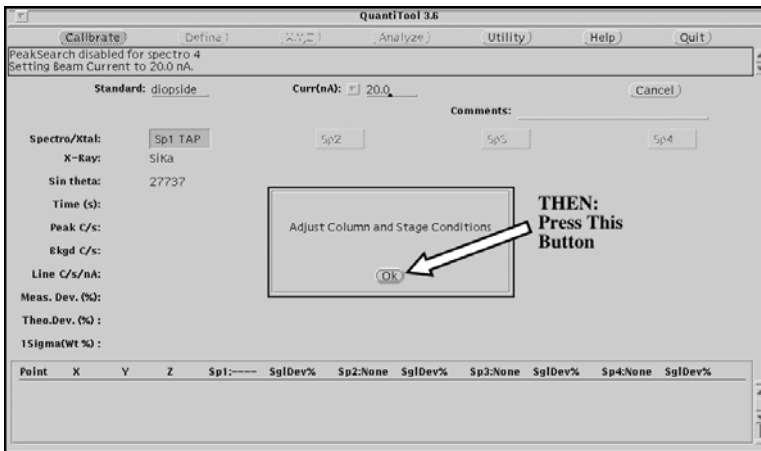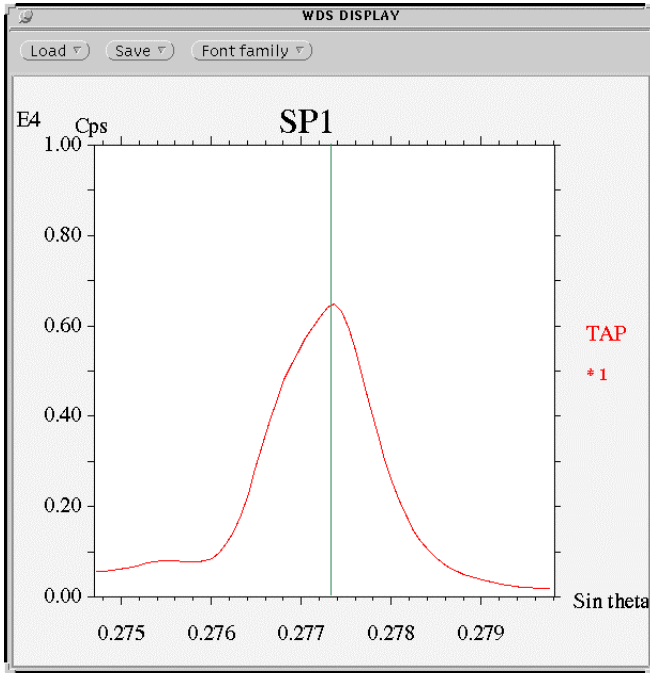

Step 9

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

WDS DISPLAY

Load ▽   Save ▽   Font family ▽

E4   Cps   SP1

The microprobe will do a peak search

Step 13

QuantiTool 3.6

Calibrate   Define   X,Y,Z   Analyze   Utility   Help   Quit

Setting Beam Current to 20.0 nA.
Doing Peak Searches

Standard: diopside   Curr(nA): ▽ 20.0   Cancel
                                              Comments:

Spectro/Xtal: Sp1 TAP   Sp2   Sp3   Sp4
X-Ray: SiKa
Sin theta: 27737
Time (s):   Verify the Peak Position.
Peak C/s:
Bkgd C/s:
Line C/s/nA: Ok
Meas. Dev. (%):
Theo.Dev. (%) :
1Sigma(Wt %) :

Point   X   Y   Z   Sp1:---- SglDev% Sp2:None SglDev% Sp3:None SglDev% Sp4:None SglDev%

**When the peak search is done
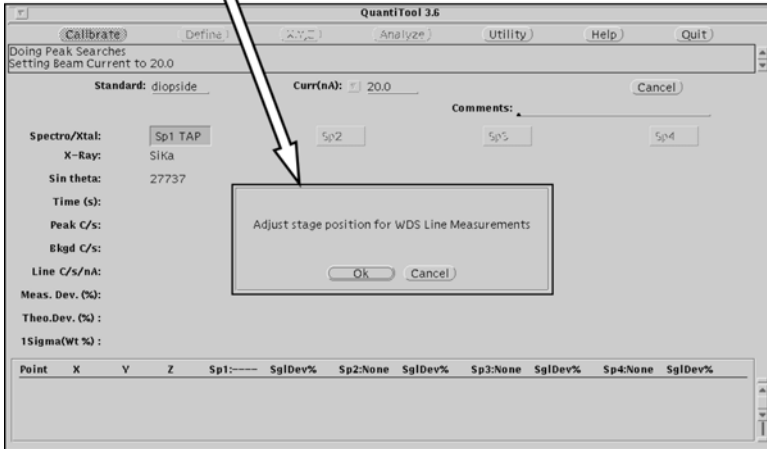AND if the peak is correct**

Press This Button

15

Step 14

Step 15

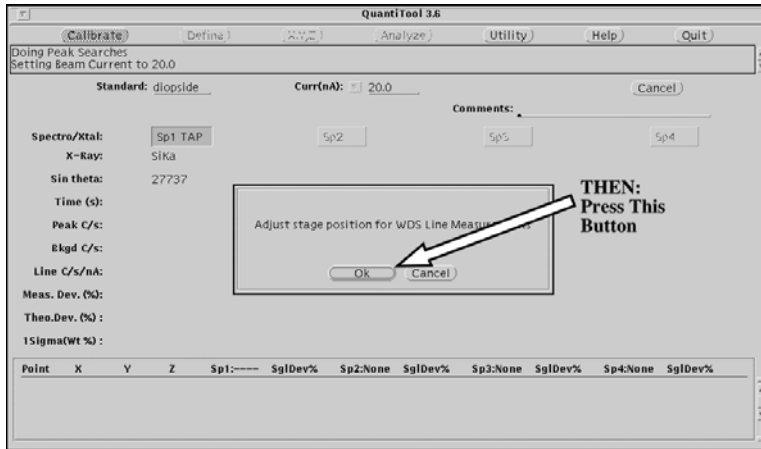When this screen appears:
1) Turn Scan on - Cup Off
2) Move to a new spot on the standard
   using the SE monitor
3) Turn Cup on - Scan Off
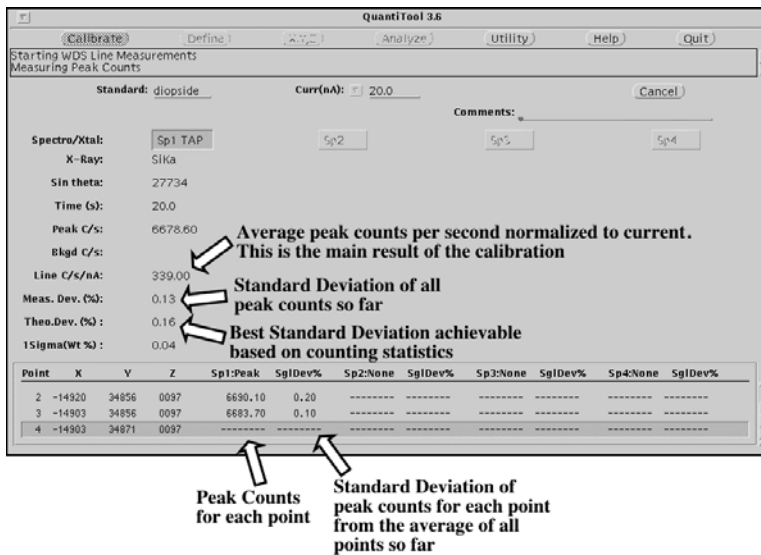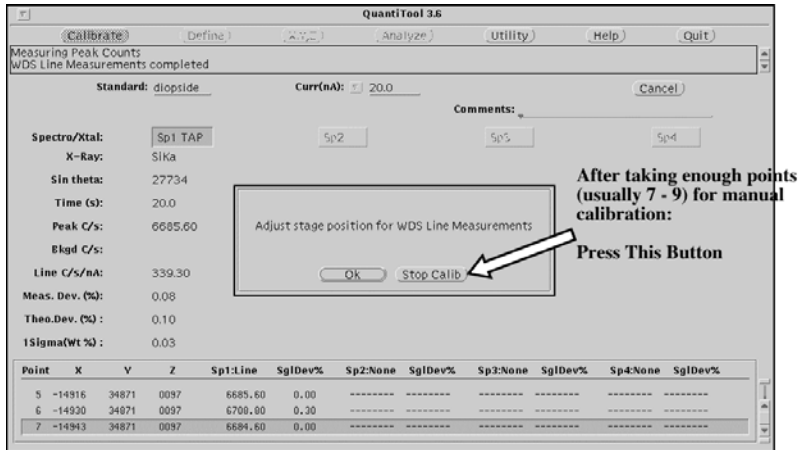4) Make sure the optical focus is still
   good

Step 16

Step 17

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

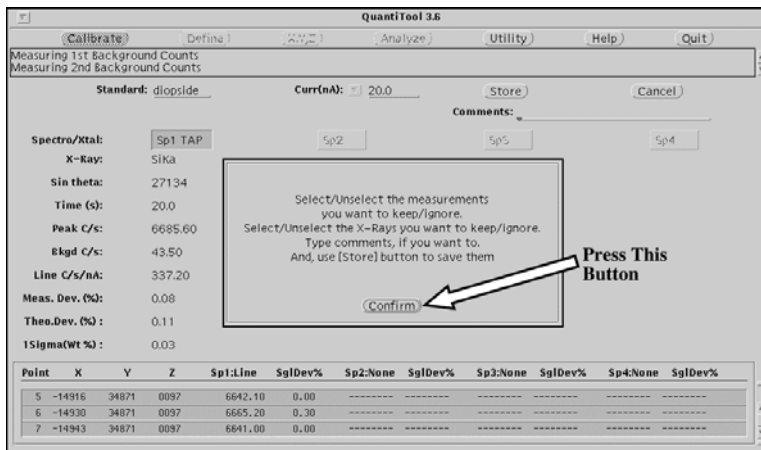**During calibration the screen will look approximately like this:**

Step 18

Step 19

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

Step 20

QuantiTool 3.6

Calibrate | Define | X,Y,Z | Analyze | Utility | Help | Quit

Measuring 1st Background Counts
Measuring 2nd Background Counts

Standard: diopside   Curr(nA): 20.0   Store   Cancel

Comments:

Spectro/Xtal:	Sp1 TAP Sp2 Sp3 Sp4
X-Ray:	SiKa
Sin theta:	27134
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

Press This Button

QuantiTool 3.6

Calibrate | Define | X,Y,Z | Analyze | Utility | Help | Quit

Measuring 1st Background Counts
Measuring 2nd Background Counts

Standard: diopside   Curr(nA): 20.0   Store   Cancel

Comments:

Spectro/Xtal:	Sp1 TAP Sp2 Sp3 Sp4
X-Ray:	SiKa
Sin theta:	27134
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	--------	--------	--------	--------	--------	--------

Step 22

Press This Button
to start next calibration

QuantiTool 3.6

Calibrate   Define   X,Y,Z   Analyze   Utility   Help   Quit

> calibration done
Calibration file updated.

Step 23

REPEAT STEPS 3 — 22 for each calibration.

Next Step

When finished with calibrations:

Go to Utility

Calibration: Instruction Set 2b

Multiple Calibration

Step 1

Set Calibration Mode to Multiple

QuantiTool 3.6

Calibrate   Define   X,Y,Z   Analyze   Utility   Help   Quit

WDS P-B   Move to Standard   Apply   Acquire   Done

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

Standard: ▽ _____   Acc(kV): ▽ 15   Curr(nA): ▽ 20   Wait(sec): ▽ 0

	Sp1 ▽	Sp2 ▽	Sp3 ▽	Sp4 ▽
Spectro:				
Conditions:	▽ New Calib	▽ New Calib	▽ New Calib	▽ New Calib
Cristal:	▽ TAP	▽ TAP	▽ PET	▽ LIF
X-Ray:	▽ _____	▽ _____	▽ _____	▽ _____
Auto Adjust PHA:	▽ No	▽ No	▽ No	▽ No
Peak Search:	▽ Yes	▽ Yes	▽ Yes	▽ Yes
Peak Position:	▽ _____	▽ _____	▽ _____	▽ _____
Peak Time(sec):	▽ 20	▽ 20	▽ 20	▽ 20
Prec.(%):	▽ 0.1	▽ 0.1	▽ 0.1	▽ 0.1

Background Offset(s)

Bkg unit:	▽ Sin theta	▽ Sin theta	▽ Sin theta	▽ Sin theta
Frequency:	▽ Once	▽ Once	▽ Once	▽ Once
Bkg Time(sec):	▽ Peak/2	▽ Peak/2	▽ Peak/2	▽ Peak/2
1st Bkg:	▽ 600	▽ 600	▽ 600	▽ 500
2nd Bkg:	▽ -600	▽ -600	▽ -600	▽ -500
Slope:	▽ _____	▽ _____	▽ _____	▽ _____

Step 2

**R-Click and
Select Block 0**

**Then select
Standard
from List**

QuantiTool 3.6

Calibrate | Define | X,Y,□ | Analyze | Utility | Help | Quit

WDS P-B   Move to Standard   Apply   Acquire   Done

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

Standard: diopside   Acc(kV): 15   Curr(nA): 20   Wait(sec): 0

	Sp1	Sp2	Sp3	Sp4
Spectro:	Sp1	Sp2	Sp3	Sp4
Conditions:	New Calib	New Calib	New Calib	New Calib
Cristal:	TAP	TAP	PET	LIF
X-Ray:				
Auto Adjust PHA:	No	No	No	No
Peak Search:	Yes	Yes	Yes	Yes
Peak Position:				
Peak Time(sec):	20	20	20	20
Prec.(%):	0.1	0.1	0.1	0.1

Background Offset(s)

Bkg unit:	Sin theta	Sin theta	Sin theta	Sin theta
Frequency:	Once	Once	Once	Once
Bkg Time(sec):	Peak/2	Peak/2	Peak/2	Peak/2
1st Bkg:	600	600	600	500
2nd Bkg:	-600	-600	-600	-500
Slope:				

Step 3

Press This Button
to move to standard

	QuantiTool 3.6					
	Calibrate	Define	X,Y,Σ	Analyze	Utility	Help	Quit

WDS P–B   Move to Standard   Apply   Acquire   Done

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

Standard: diopside   Acc(kV): 15   Curr(nA): 20   Wait(sec): 0

Spectro:	Sp1	Sp2	Sp3	Sp4
Conditions:	New Calib	New Calib	New Calib	New Calib
Cristal:	TAP	TAP	PET	LIF
X–Ray:				
Auto Adjust PHA:	No	No	No	No
Peak Search:	Yes	Yes	Yes	Yes
Peak Position:				
Peak Time(sec):	20	20	20	20
Prec.(%):	0.1	0.1	0.1	0.1

Background Offset(s)

Bkg unit:	Sin theta	Sin theta	Sin theta	Sin theta
Frequency:	Once	Once	Once	Once
Bkg Time(sec):	Peak/2	Peak/2	Peak/2	Peak/2
1st Bkg:	600	600	600	500
2nd Bkg:	–600	–600	–600	–500
Slope:				

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:

Press This Button
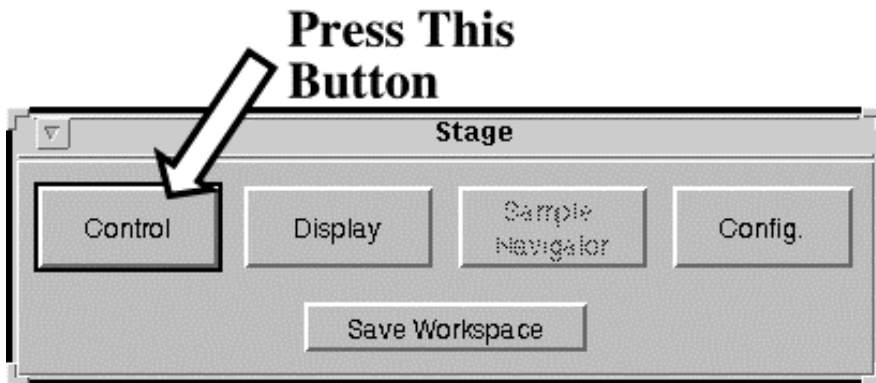
THEN:

R-Click and select the correct Standard from the list. (This should be the same standard you selected in Step 2)
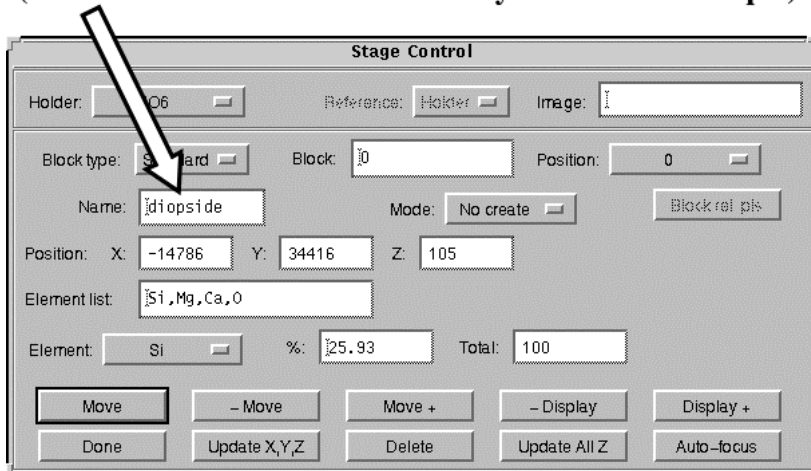
Step 5

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:**

Press This Button

Step 6

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

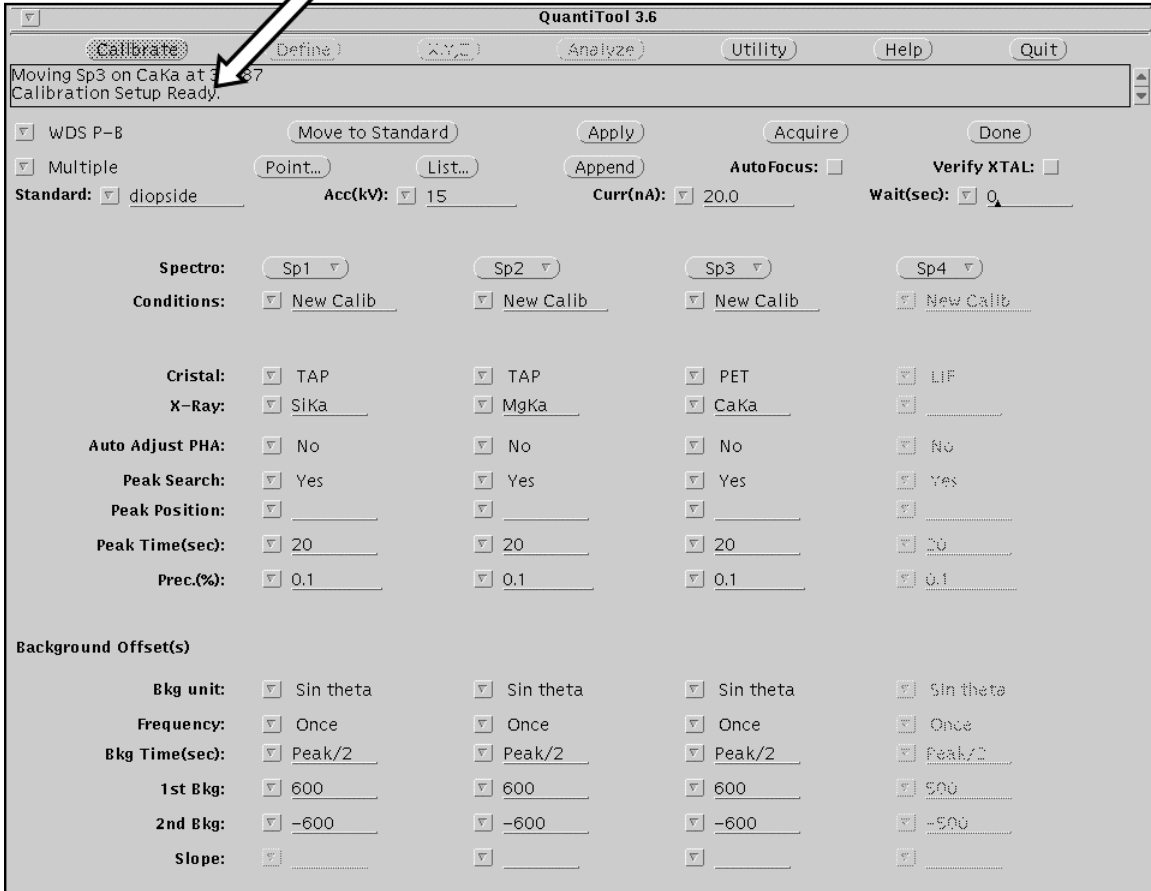**Turn off unused spectrometers by toggling the Sp# buttons**

Step 7

Press This
Button

QuantiTool 3.6

Calibrate   Define   X,Y,Z   Analyze   Utility   Help   Quit

Moving Sp3 on CaKa at 38387
Calibration Setup Ready.

WDS P−B   Move to Standard   Apply   Acquire   Done

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

Standard: diopside   Acc(kV): 15   Curr(nA): 20.0   Wait(sec): 0

Spectro:	Sp1	Sp2	Sp3	Sp4
Conditions:	New Calib	New Calib	New Calib	New Calib
Cristal:	TAP	TAP	PET	LIF
X−Ray:	SiKa	MgKa	CaKa	
Auto Adjust PHA:	No	No	No	No
Peak Search:	Yes	Yes	Yes	Yes
Peak Position:				
Peak Time(sec):	20	20	20	20
Prec.(%):	0.1	0.1	0.1	0.1

Background Offset(s)

Bkg unit:	Sin theta	Sin theta	Sin theta	Sin theta
Frequency:	Once	Once	Once	Once
Bkg Time(sec):	Peak/2	Peak/2	Peak/2	Peak/2
1st Bkg:	600	600	600	600
2nd Bkg:	−600	−600	−600	−600
Slope:				

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Step 8

Wait for the beep and these words to appear

QuantiTool 3.6

| Calibrate | Define | X,Y,Z | Analyze | Utility | Help | Quit |

Moving Sp3 on CaKa at 3..67
Calibration Setup Ready.

WDS P-B   Move to Standard   Apply   Acquire   Done

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

Standard: diopside   Acc(kV): 15   Curr(nA): 20.0   Wait(sec): 0

	Sp1	Sp2	Sp3	Sp4
Spectro:	Sp1	Sp2	Sp3	Sp4
Conditions:	New Calib	New Calib	New Calib	New Calib
Cristal:	TAP	TAP	PET	LIF
X-Ray:	SiKa	MgKa	CaKa	
Auto Adjust PHA:	No	No	No	No
Peak Search:	Yes	Yes	Yes	Yes
Peak Position:				
Peak Time(sec):	20	20	20	20
Prec.(%):	0.1	0.1	0.1	0.1

Background Offset(s)

Bkg unit:	Sin theta	Sin theta	Sin theta	Sin theta
Frequency:	Once	Once	Once	Once
Bkg Time(sec):	Peak/2	Peak/2	Peak/2	Peak/2
1st Bkg:	600	600	600	600
2nd Bkg:	-600	-600	-600	-600
Slope:				

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   (...e to Standard)   (Apply)   (Acquire)   (Done)
▽ Multiple   (Point...)   (List...)   (Append)   **AutoFocus:** ☐   **Verify XTAL:** ☐

Standard: ▽ diopside   **Acc(kV):** ▽ 15   **Curr(nA):** ▽ 20.0   **Wait(sec):** ▽ 0

Spectro:	(Sp1 ▽)	(Sp2 ▽)	(Sp3 ▽)	(Sp4 ▽)
Conditions:	▽ New Calib	▽ New Calib	▽ New Calib	▽ New Calib
Cristal:	▽ TAP	▽ TAP	▽ PET	▽ LIF
X–Ray:	▽ SiKa	▽ MgKa	▽ CaKa	▽
Auto Adjust PHA:	▽ No	▽ No	▽ No	▽ No
Peak Search:	▽ Yes	▽ Yes	▽ Yes	▽ Yes
Peak Position:	▽	▽	▽	▽
Peak Time(sec):	▽ 20	▽ 20	▽ 20	▽ 20
Prec.(%):	▽ 0.1	▽ 0.1	▽ 0.1	▽ 0.1

Background Offset(s)

|				
Bkg unit:	▽ Sin theta	▽ Sin theta	▽ Sin theta	▽ Sin theta
Frequency:	▽ Once	▽ Once	▽ Once	▽ Once
Bkg Time(sec):	▽ Peak/2	▽ Peak/2	▽ Peak/2	▽ Peak/2
1st Bkg:	▽ 600	▽ 600	▽ 600	▽ 600
2nd Bkg:	▽ –600	▽ –600	▽ –600	▽ –600
Slope:	▽	▽	▽	▽

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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:
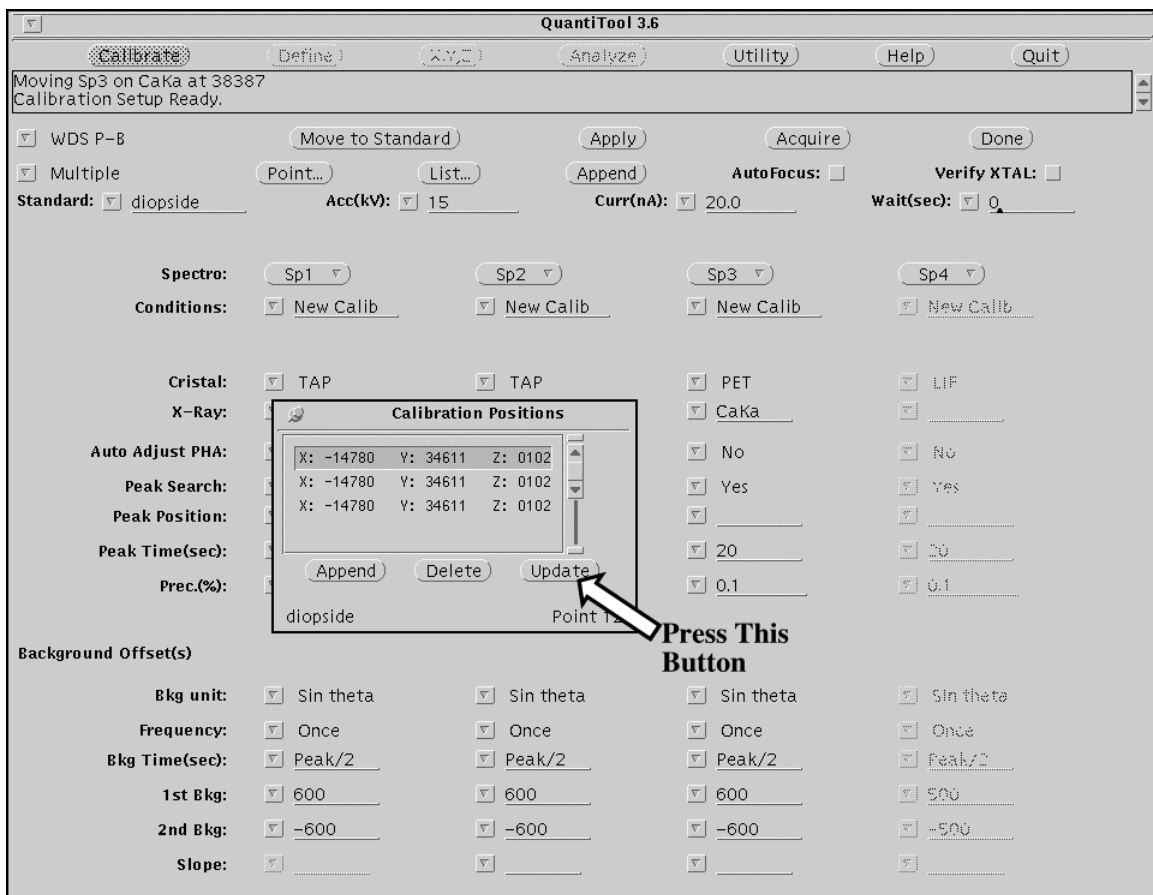
Step 11

After using the Update button to set the first three points, use the Append button to set the remaining 9 points (see below).
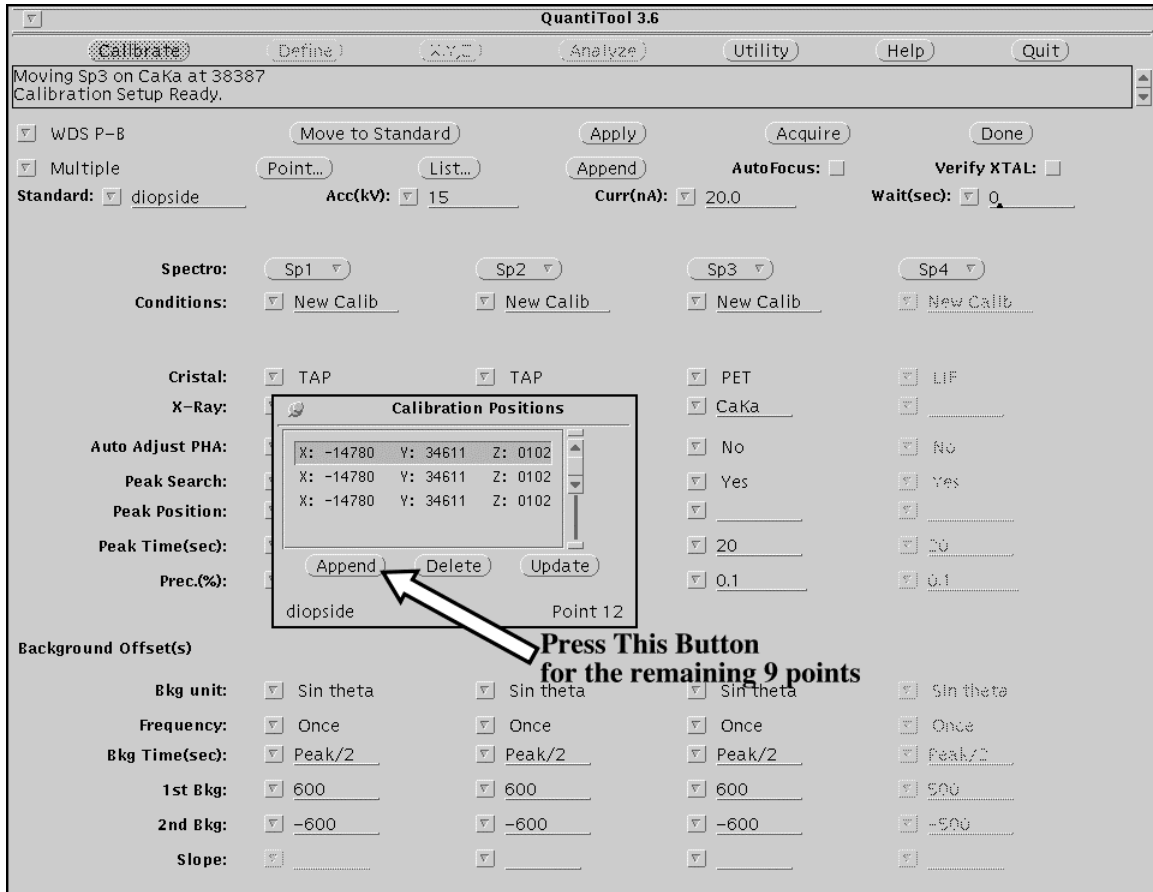
Step 12

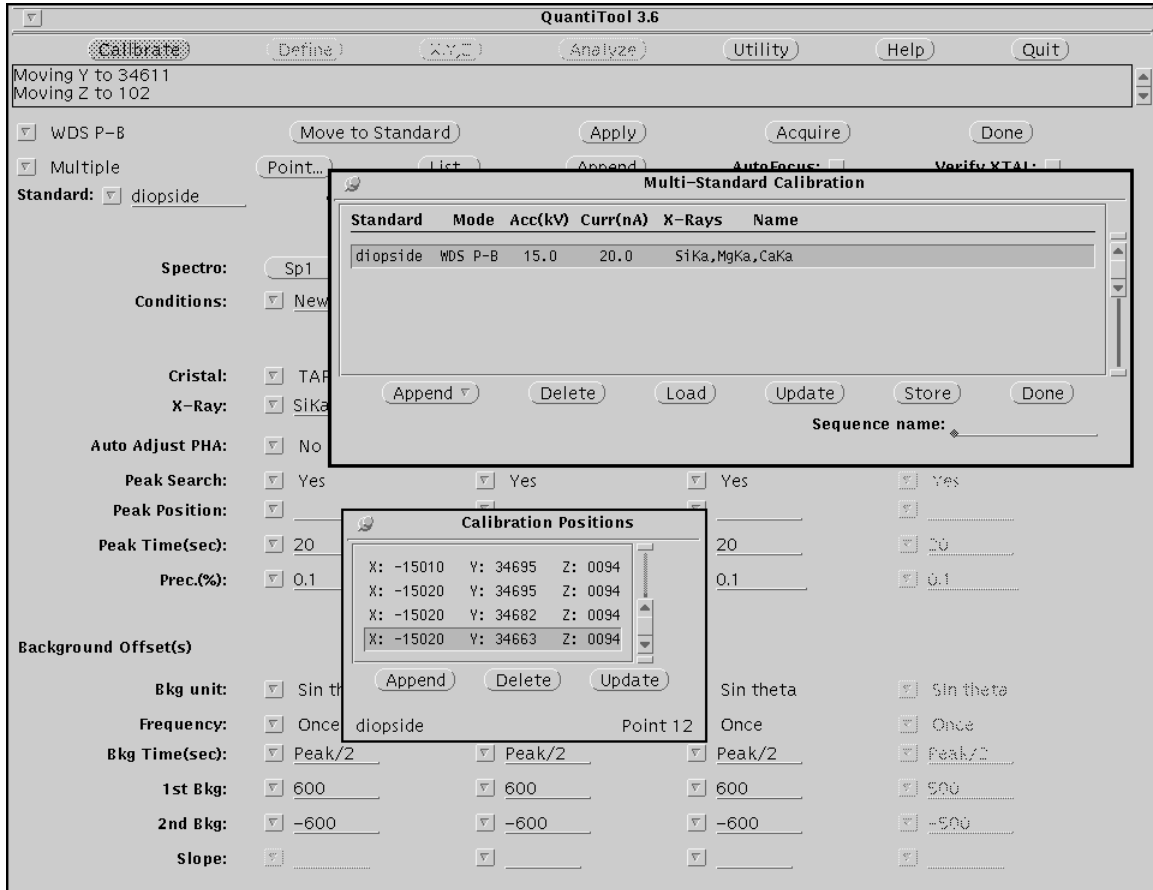**After setting 12 points, turn Cup — On and Scan — Off**

THEN:

Press This Button

Step 13

The standard will go on the list of points to run later.
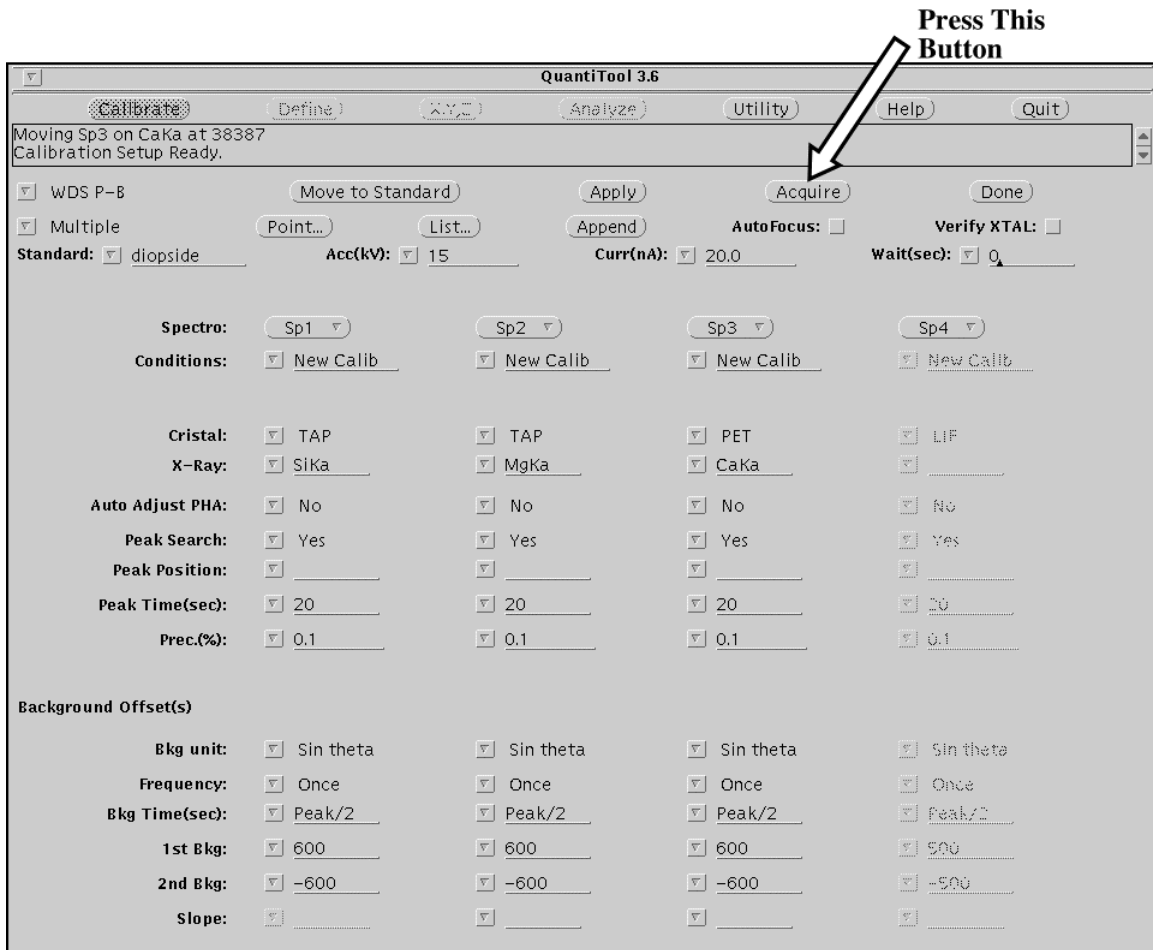
Step 14

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:

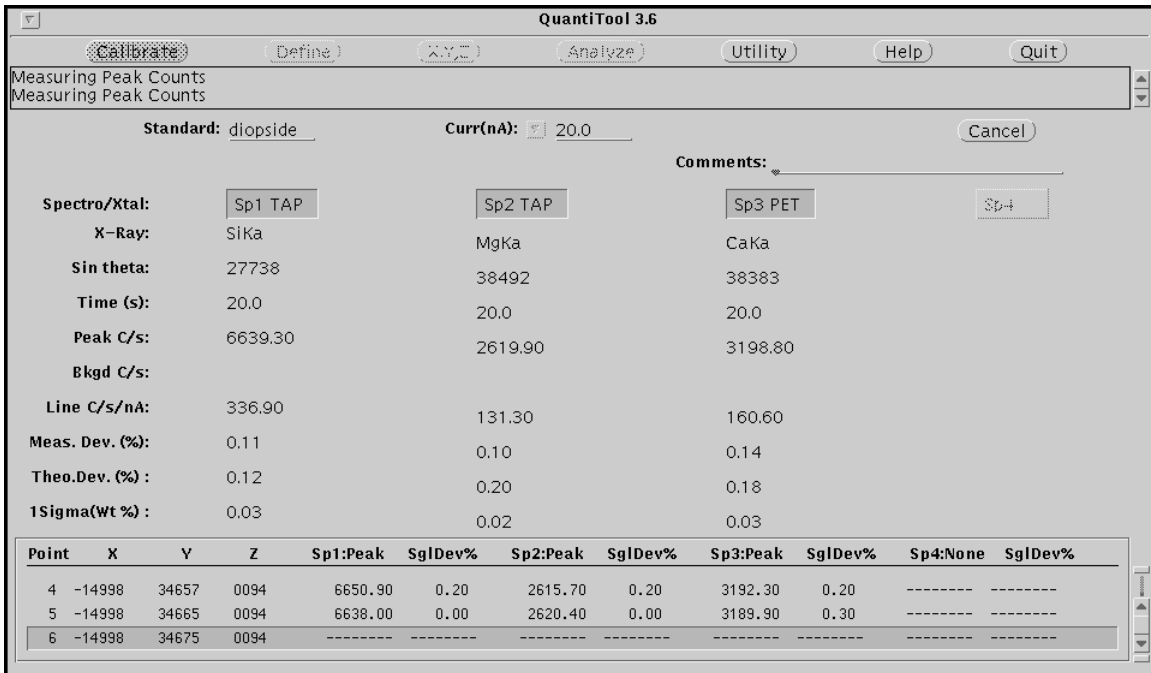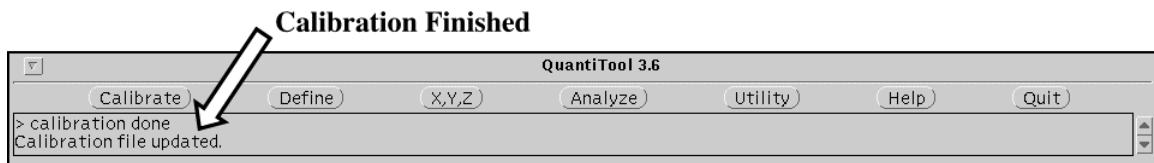## VERY IMPORTANT! - Turn Cup On — Scan Off

THEN:

Press This Button

Step 17

Calibration should take about 4 minutes per standard.

During calibration the screen will look like this:

QuantiTool 3.6						
Calibrate	Define	X,Y,Z	Analyze	Utility	Help	Quit

Measuring Peak Counts
Measuring Peak Counts

Standard: diopside   Curr(nA): 20.0   Comments: Cancel

Spectro/Xtal:	Sp1 TAP	Sp2 TAP	Sp3 PET	Sp4
X–Ray:	SiKa	MgKa	CaKa	
Sin theta:	27738	38492	38383	
Time (s):	20.0	20.0	20.0	
Peak C/s:	6639.30	2619.90	3198.80	
Bkgd C/s:				
Line C/s/nA:	336.90	131.30	160.60	
Meas. Dev. (%):	0.11	0.10	0.14	
Theo.Dev. (%) :	0.12	0.20	0.18	
1Sigma(Wt %) :	0.03	0.02	0.03	

Point	X	Y	Z	Sp1:Peak	SglDev%	Sp2:Peak	SglDev%	Sp3:Peak	SglDev%	Sp4:None	SglDev%
4	-14998	34657	0094	6650.90	0.20	2615.70	0.20	3192.30	0.20	--------	--------
5	-14998	34665	0094	6638.00	0.00	2620.40	0.00	3189.90	0.30	--------	--------
6	-14998	34675	0094	--------	--------	--------	--------	--------	--------	--------	--------

Step 18

After calibration the screen will look like this:

Calibration Finished

QuantiTool 3.6						
(Calibrate)	(Define)	(X,Y,Z)	(Analyze)	(Utility)	(Help)	(Quit)

> calibration done
Calibration file updated.

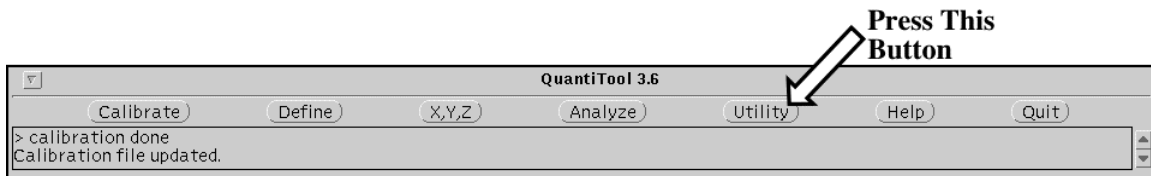# Next Step

When finished with calibrations:

Go to Utility

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

Label Files

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

(Select All)   (Unselect)   (Delete)   (Print List)

Files

Press This Button

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

Page: ▽ 1

Sort by: ▽ Z

Then by: ▽ Date   (Done)

Step 3

Standards

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

(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

Sort by: ▼ Date

Then by: ▼ Z

(Change Bckgrd)

(Calib Curve)

(Done)

Sort the calibration list first by date then by Z

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Step 4

Press This Button

Standards

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

(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.

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

(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

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

(Change Bckgrd)

(Calib Curve)

(Done)

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Step 6

Step 7

Next Step

Go to either:
Define an Analysis File Instruction Set 4a: Create a
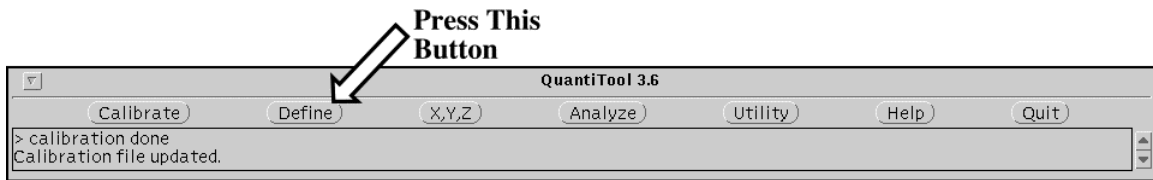**New File (p. 51)**
or
Define an Analysis File Instruction Set 4b: Update an
Existing File (p. 65)

Define an Analysis File: Instruction Set 4a
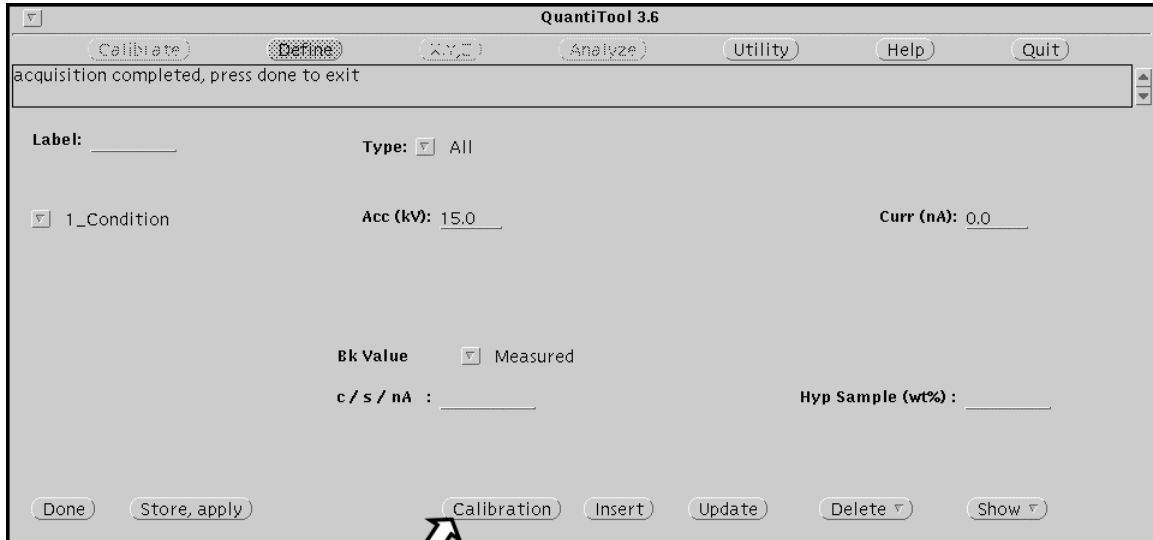
Create a New File

Step 1

Press This Button

	QuantiTool 3.6	
Calibrate	Define	X,Y,Z	Analyze	Utility	Help	Quit

> calibration done
Calibration file updated.

Step 2

Make sure the voltage is correct

```
┌─────────────────────────────────────────────────────────────────────────────┐
│ ▽                              Quanti   ol 3.6                                 │
│  (Calibrate)    (Define)    (X,Y,□)    (  lyze)    (Utility)  (Help)  (Quit)  │
│ ┌─────────────────────────────────────────────────────────────────────────┐▲│
│ │acquisition completed, press done to exit                                  │▼│
│ └─────────────────────────────────────────────────────────────────────────┘ │
│                                                                               │
│   Label: _____         Type: ▽  All                                        │
│                                                                               │
│   ▽  1_Condition           Acc (kV): 15.0          Curr (nA): 0.0            │
│                                                                               │
│                                                                               │
│                        Bk Value    ▽  Measured                                │
│                        c / s / nA : _____    Hyp Sample (wt%) : _____   │
│                                                                               │
│                                                                               │
│  (Done)  (Store, apply)     (Calibration) (Insert) (Update) (Delete ▽) (Show ▽)│
└─────────────────────────────────────────────────────────────────────────────┘
```

Step 3

QuantiTool 3.6

| Calibrate | Define | X,Y,Σ | Analyze | Utility | Help | Quit |

acquisition completed, press done to exit

Label: _____

Type: ▽ All

▽ 1_Condition

Acc (kV): 15.0

Curr (nA): 0.0

Bk Value   ▽ Measured

c / s / nA : _____

Hyp Sample (wt%) : _____

| Done | Store, apply | Calibration | Insert | Update | Delete ▽ | Show ▽ |

Press This Button

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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: ▽ 1

Sort by: ▽ Z

Then by: ▽ Date   (Done)

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)

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
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 (<u>FOR EACH SPECTROMETER</u>) 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

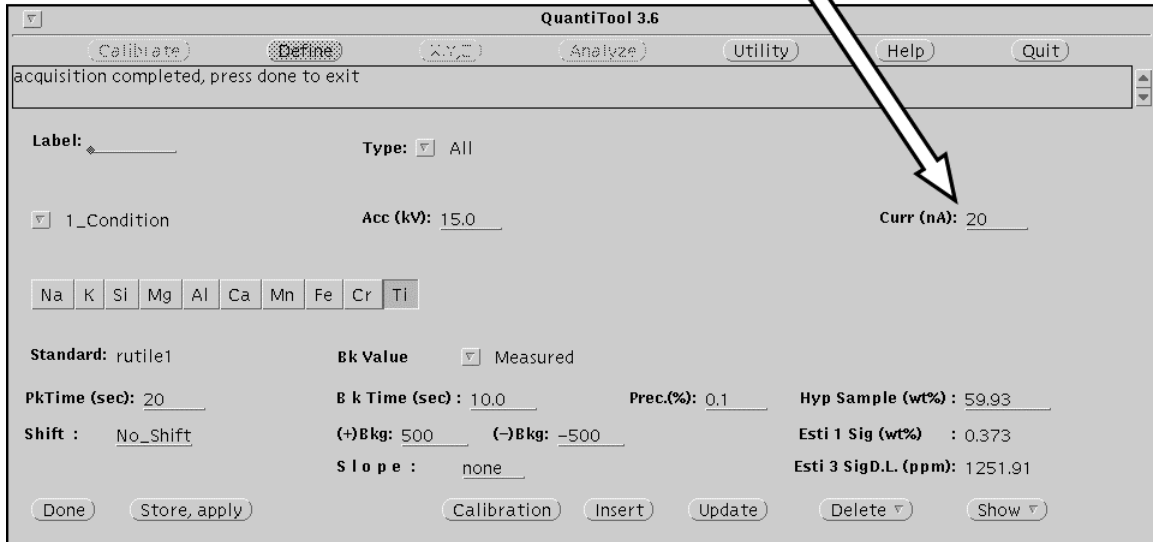**Then by:** Date

(Done)

After selecting all the required calibrations: - Press This Button

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Step 7

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

Step 8

Set the Analysis Type:
Stoichiometric for Oxides
All for Elements

Step 9

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

Step 10

QuantiTool 3.6

| Calibrate | Define | X,Y,Z | Analyze | Utility | Help | Quit |
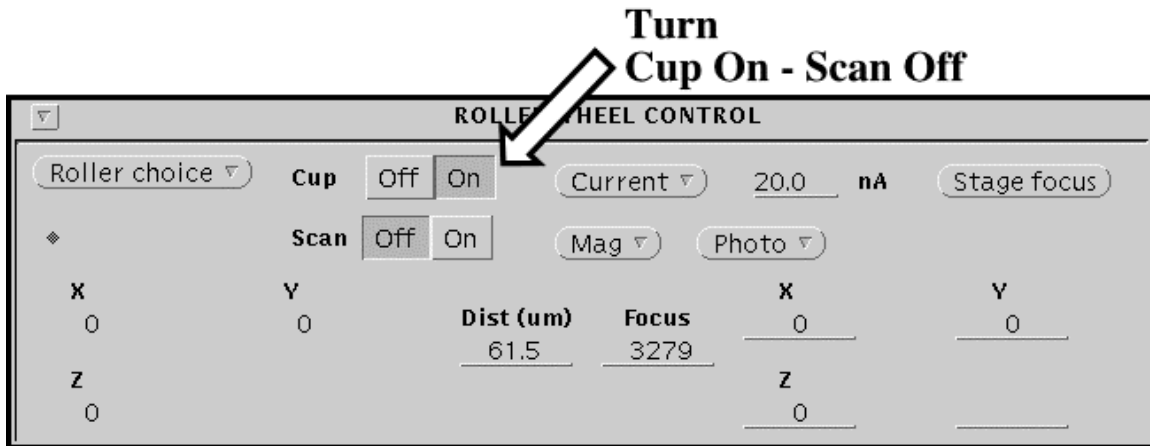
acquisition completed, press done to exit

Label: test5   **Type:** ▽ Stoichio.   **Anion:** O₀

                                                               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.
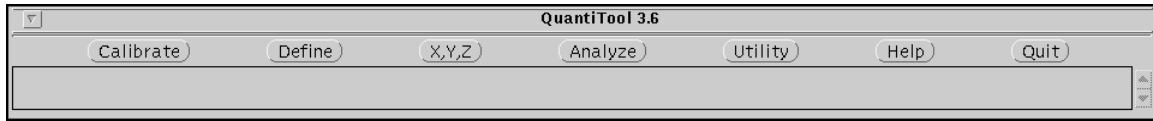
THEN: Adjust the beam size if necessary.

THEN:
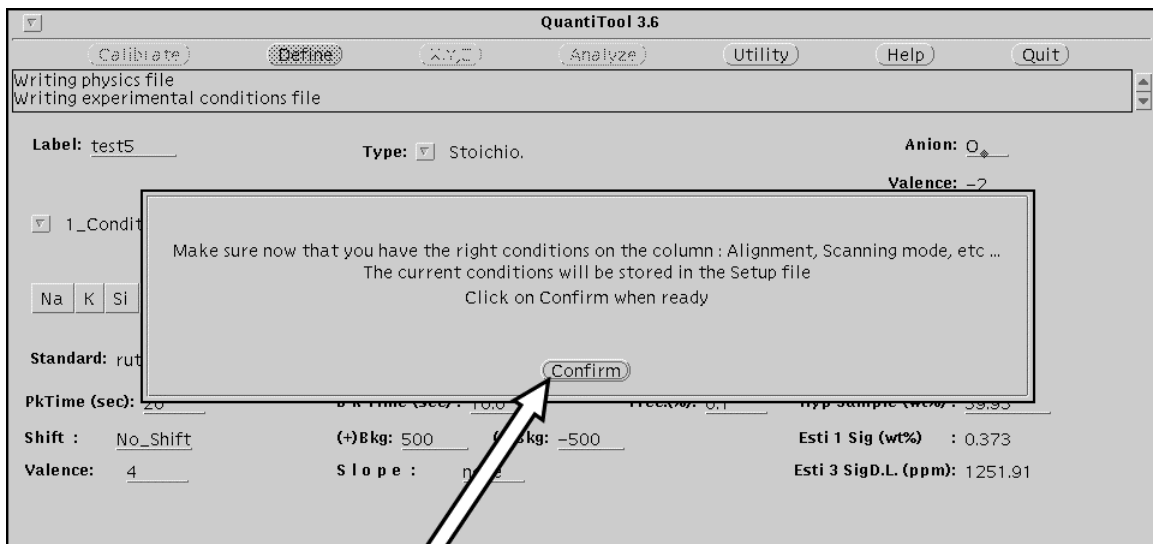
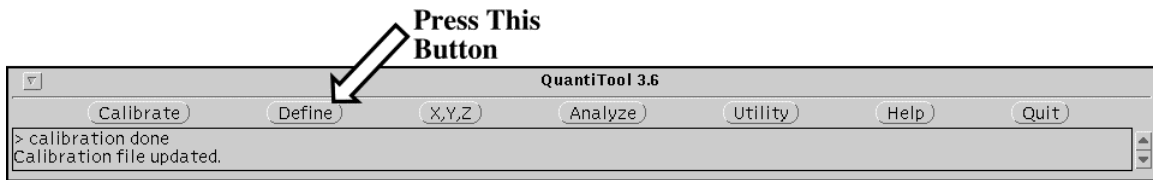**Press This Button**

Step 12

The Define file has been stored.
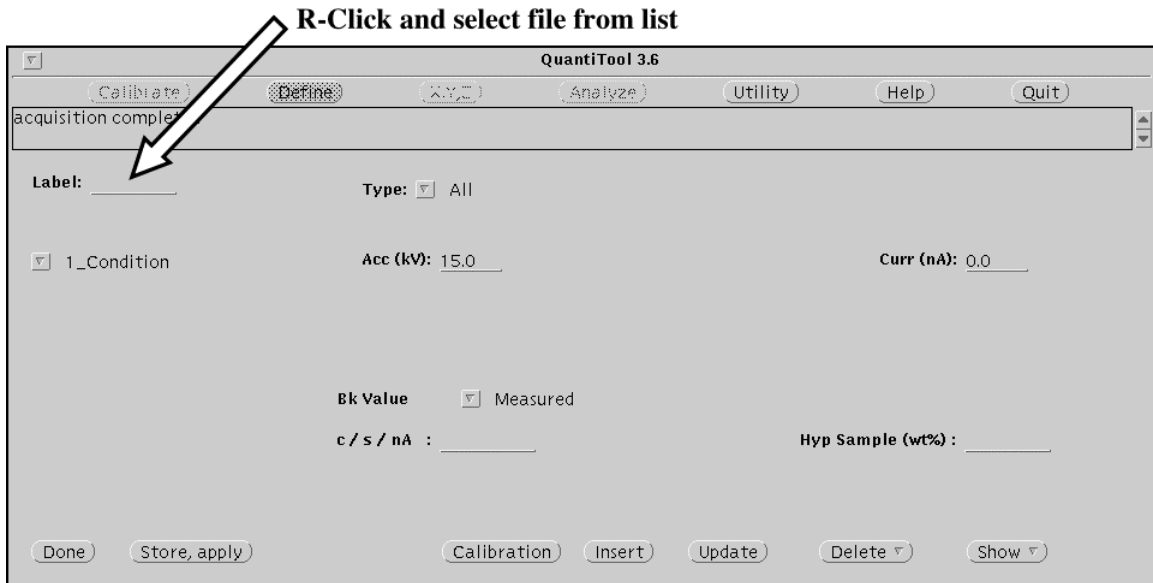
Next Step

Go to Analysis Instruction Set

Define an Analysis File: Instruction Set 4b

Update an Existing File

Step 1

Press This Button

	QuantiTool 3.6				
Calibrate	Define	X,Y,Z	Analyze	Utility	Help	Quit

> calibration done
Calibration file updated.

Step 2

R-Click and select file from list

QuantiTool 3.6

| Calibrate | Define | X,Y,Z | Analyze | Utility | Help | Quit |

acquisition complete

Label: _____

Type: ▽ All

▽ 1_Condition

Acc (kV): 15.0

Curr (nA): 0.0

Bk Value   ▽ Measured

c / s / nA : _____

Hyp Sample (wt%) : _____

(Done)   (Store, apply)   (Calibration) (Insert) (Update)   (Delete ▽)   (Show ▽)

Step 3

L-Click on the calibration you want to change

QuantiTool 3.6

Calibrate | Define | X.Y. | Analyze | Utility | Help | Quit

The selected file label already exists!

Label: test   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: 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   S l o p e :   none   Esti 3 SigD.L. (ppm): 424.50

Done | Store, apply   Calibration | Insert | Update   Delete ▽ | Show ▽

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Step 4

Step 5

Select a new Calibration from the list

```
                                    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          Page:  ▽  1
              Calib WDS Integral
              Calib EDS                      Sort by: ▽  Date


                                           Then by: ▽  Z        ( Done )
```
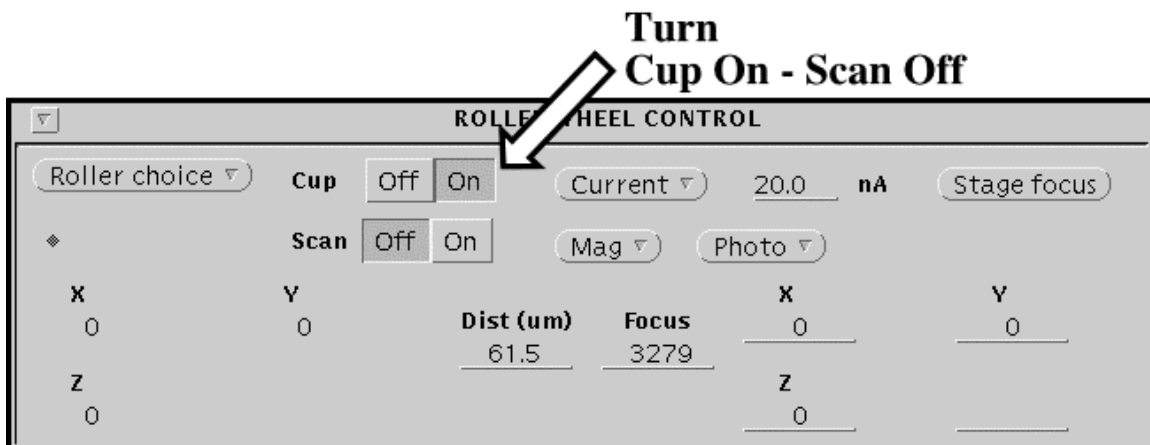
Note: Only Calibrations for the same element, spectrometer, crystal, and voltage are shown on the Update Element list.

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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: ▽ 1

Sort by: ▽ Date

Then by: ▽ Z   (Done)

Press This Button

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Step 7

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

THEN:

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

Turn
Cup On - Scan Off

Step 8

Press This Button

Step 9

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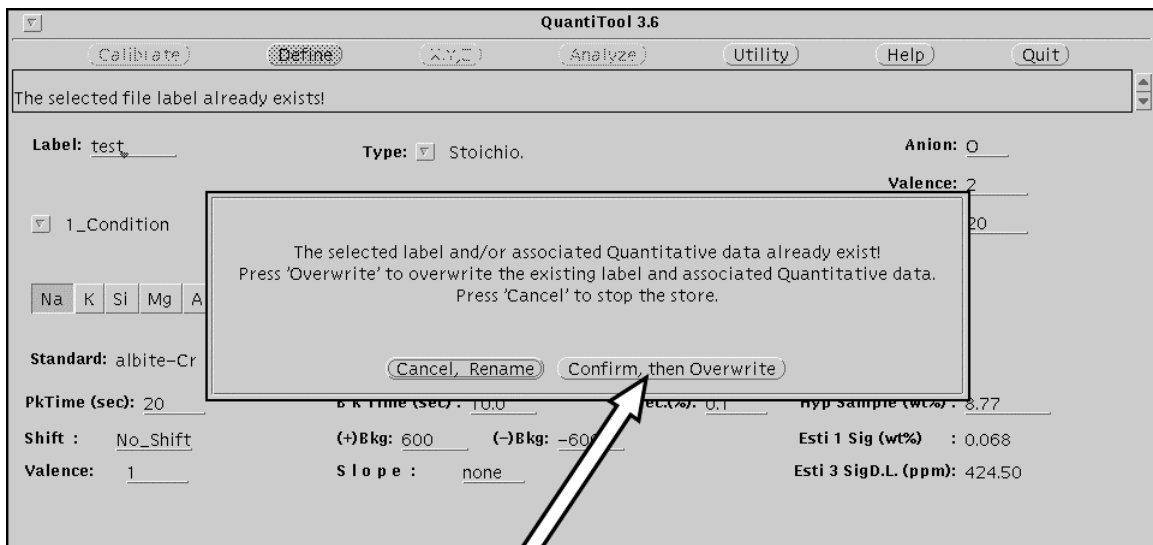
THEN:

Note: This screen will not appear if there is no previous analysis data for this file.
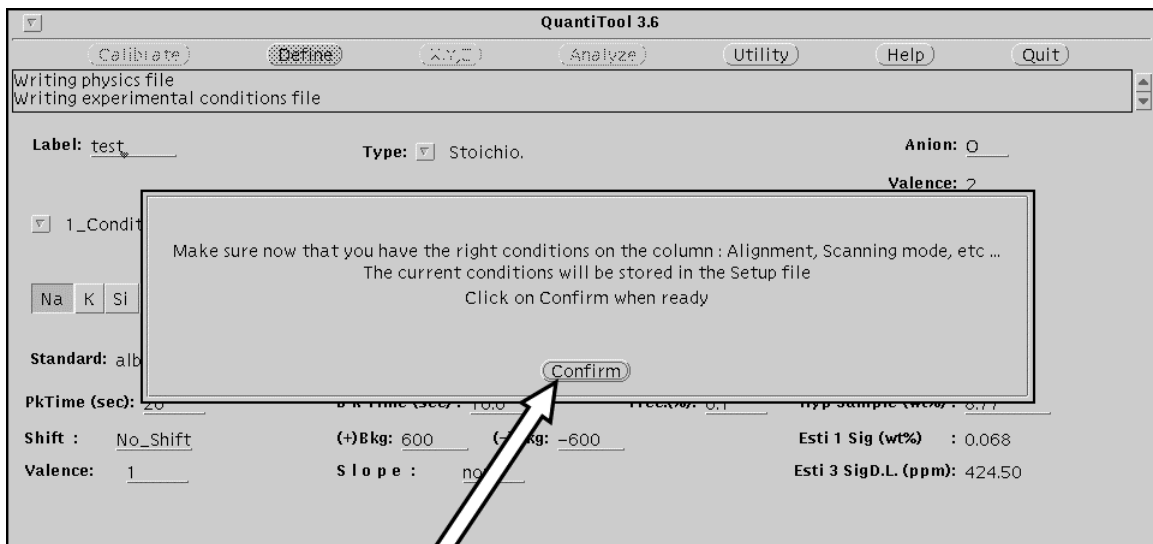
Step 10

VERY IMPORTANT — WAIT!

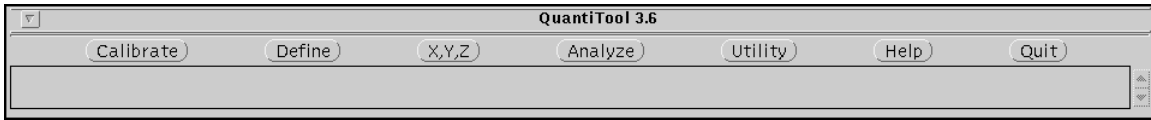# <u>DO NOT</u> try to change any settings until the following screen appears.

THEN: Adjust the beam size if necessary.

THEN:

Press This
Button

Step 11

Define file stored.

Next Step

Go to Analysis Instruction Set