

Analysis

Kenneth Domanik
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.

Analysis

Contents

Analysis	1
Contents	
Analysis: Instruction Set 1.....	3
Start Up Procedure	
1: Start the Beam.....	4
2: Move to the Sample and Focus	12
3: Turn On Backscattered Electron Imaging (If not already on).....	17
Next Step.....	25
Analysis: Instruction Set 2a.....	26
Analyzing Individual Points	
Analysis: Instruction Set 2b.....	35
Multi Point Analysis	
Analysis: Instruction Set 2c	51
Stage Line Scan Analysis	
Analysis: Instruction Set 3.....	66
Taking BSE Pictures	
1: Obtain the Image.....	67
2: Save the Image as a tif file.....	73
3: Print the Image on Paper.....	80
4: Turn Fast display Off.....	86
Analysis: Instruction Set 4.....	89
Taking EDS Spectra	
Appendix 1	101
Analysis Screen Disappears after pressing Continue	
Appendix 2	102
Reset the Probe Marker	
Appendix 3	105
Adjust BSE Image Brightness and Contrast	
1: Using Automatic Mode: (easier).....	106
2: Using Manual Mode: (more control).....	110

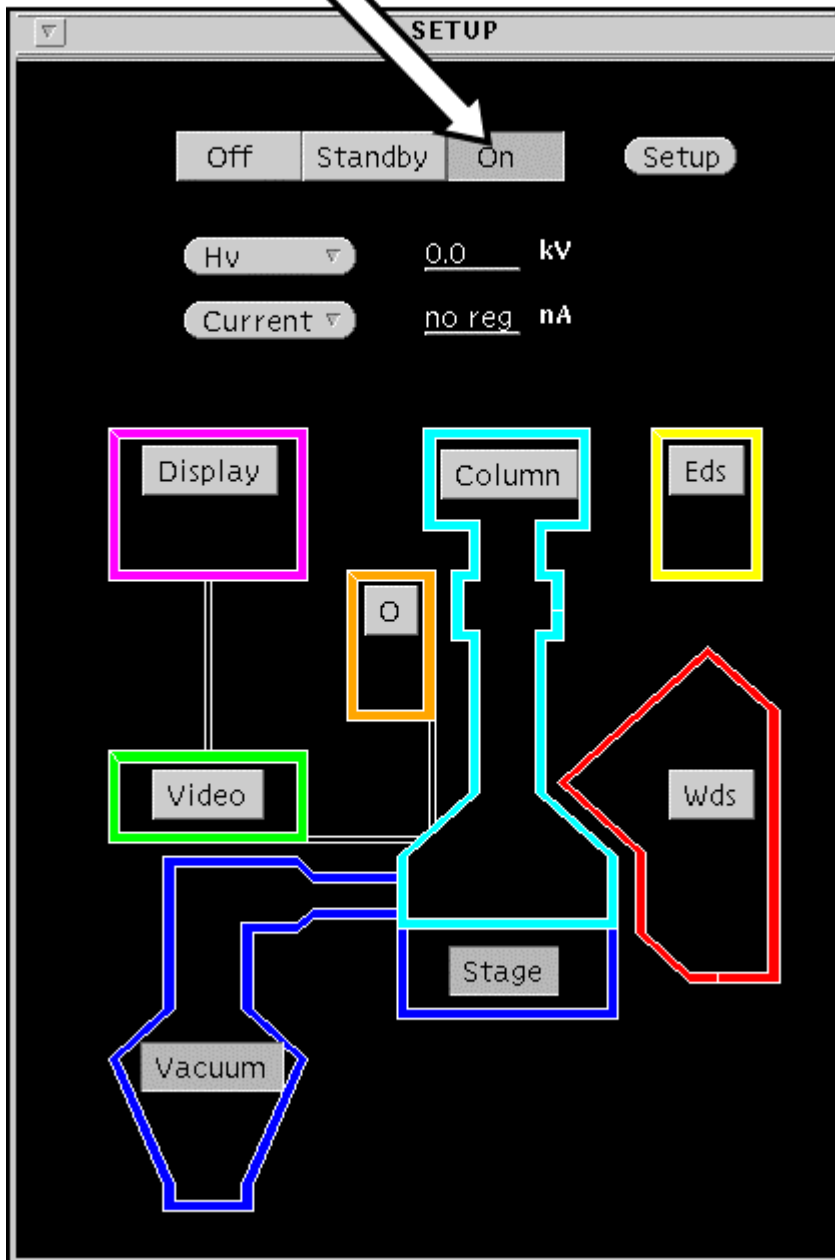
Analysis: Instruction Set 1

Start Up Procedure

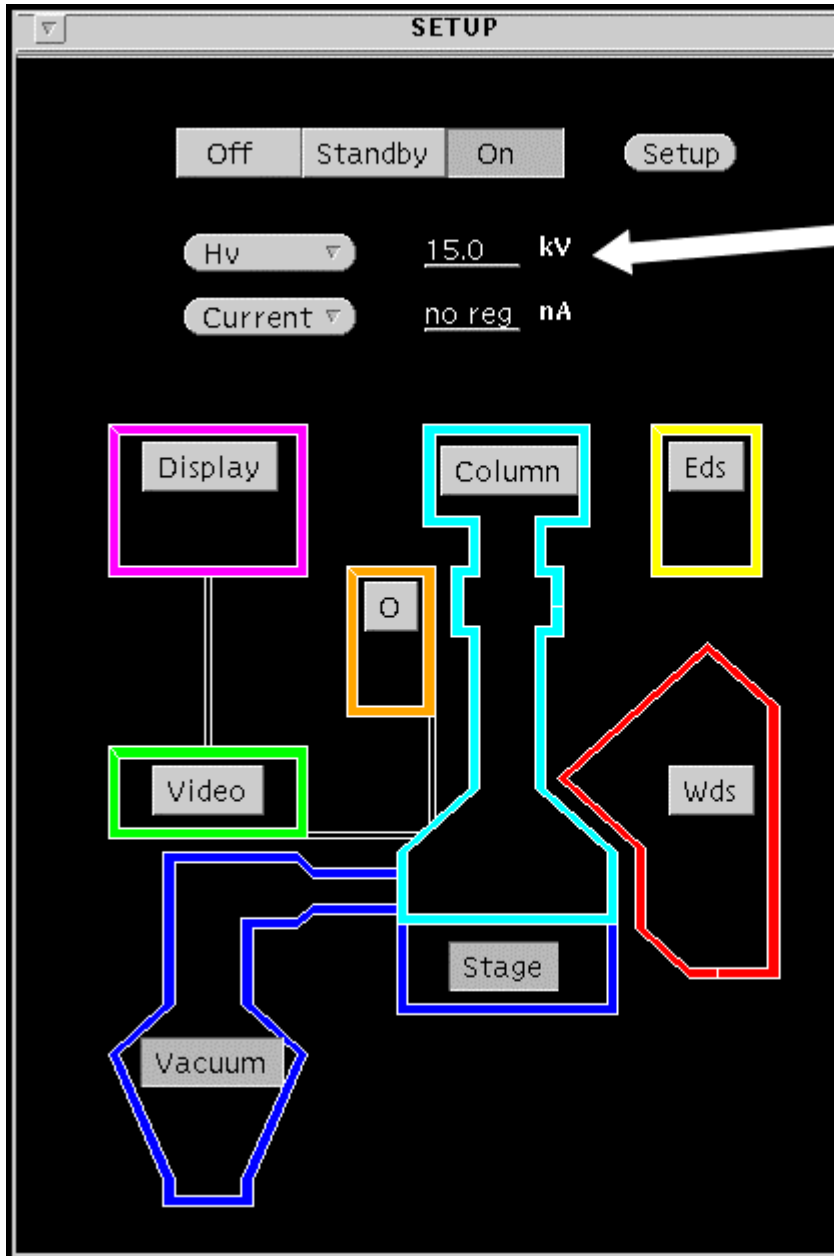
1: Start the Beam

Step 1

Press This Button



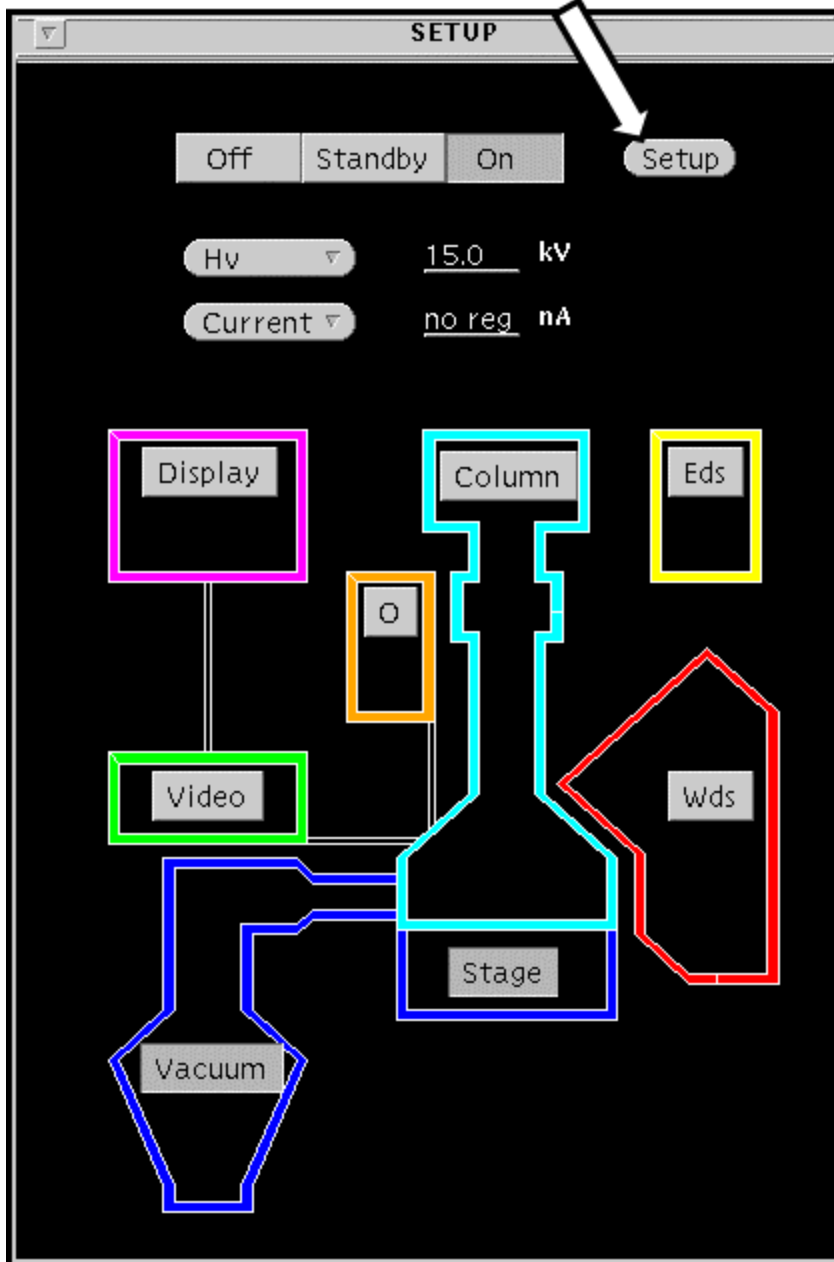
Step 2



Wait for the voltage to go to 15kV

Step 3

Press This Button



Step 4

Click on
HV15

SAVE/LOAD SETUP FILES

Sort by: Selected Setups:

Name	Comment	Hv	Current	Date
HV15		15.0	20.0	15/Sep/04 07:27:

Chosen Setup : _____

Comment : _____

Step 5


SAVE/LOAD SETUP FILES

Sort by: Selected Setups:

Name	Comment	Hv	Current	Date
HV15		15.0	20.0	15/Sep/04 07:27:

Chosen Setup:

Comment:



Press This Button

Step 6



Wait for this message to appear.

Step 7

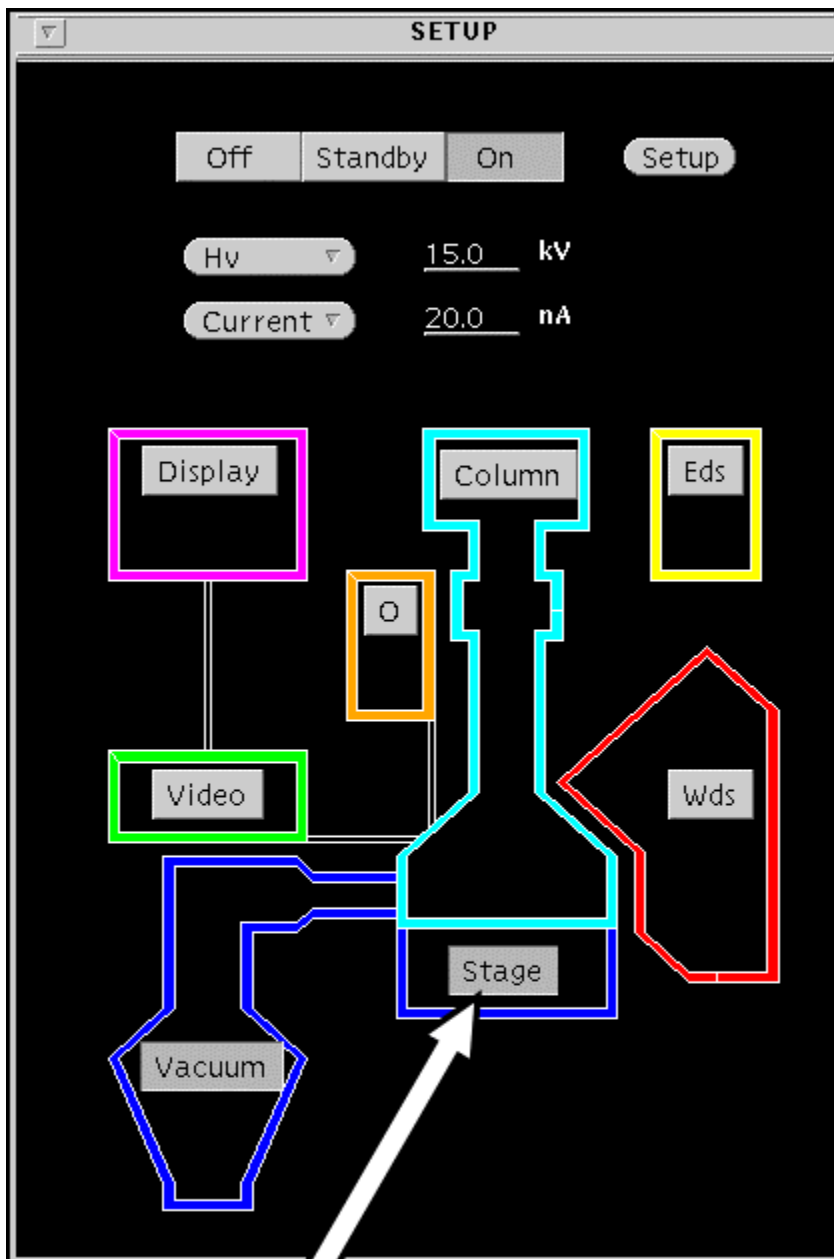
Click this pin
to close window



2: Move to the Sample and Focus

Step 1

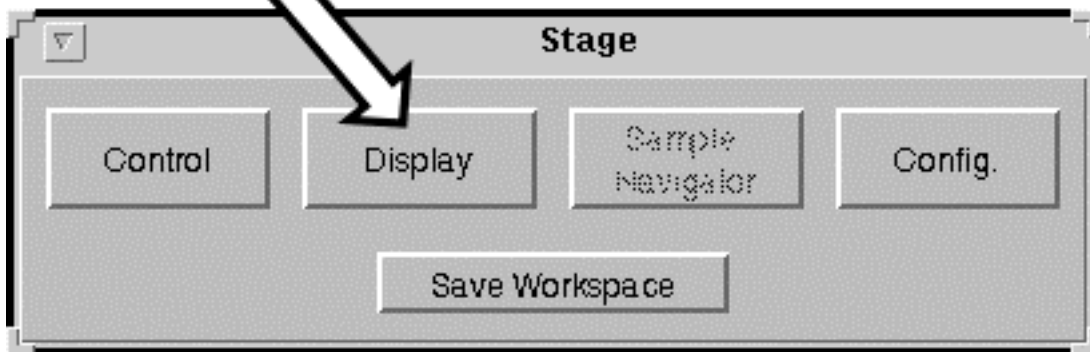
If the Stage window is not already open:



**Press This
Button**

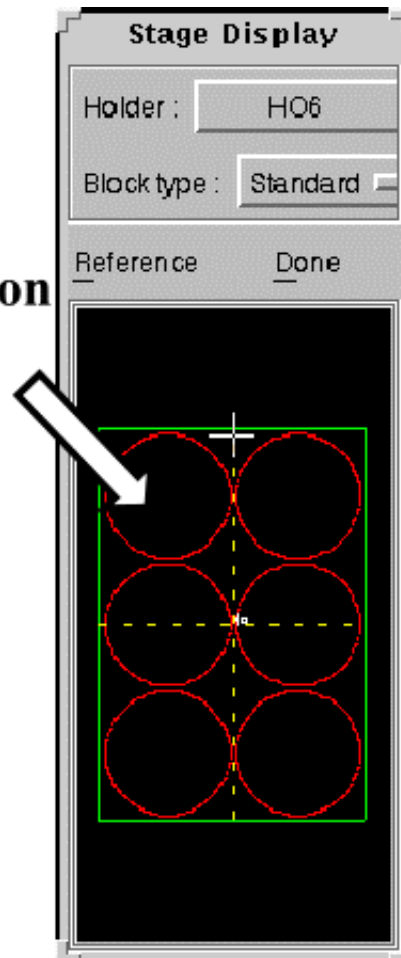
Step 2

**Press This
Button**



Step 3

**L-Click on the position
on the shuttle where
your sample is.**



The Stage will move to the chosen position.

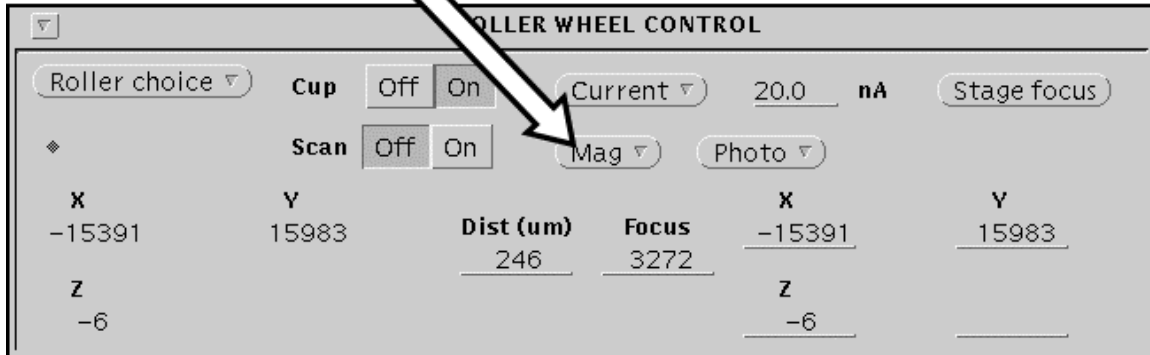
Step 4

Optically focus the reflected light image using the Z roller wheel.

3: Turn On Backscattered Electron Imaging (If not already on)

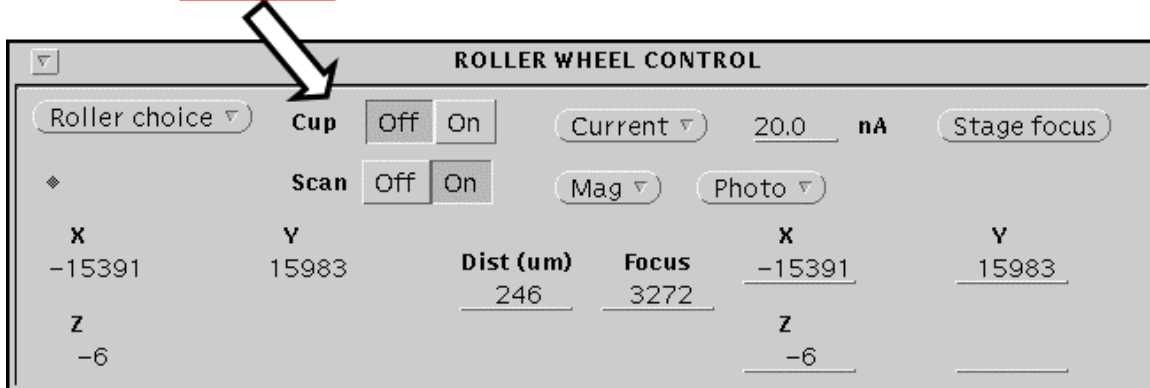
Step 1

R-Click and select MIN from menu.

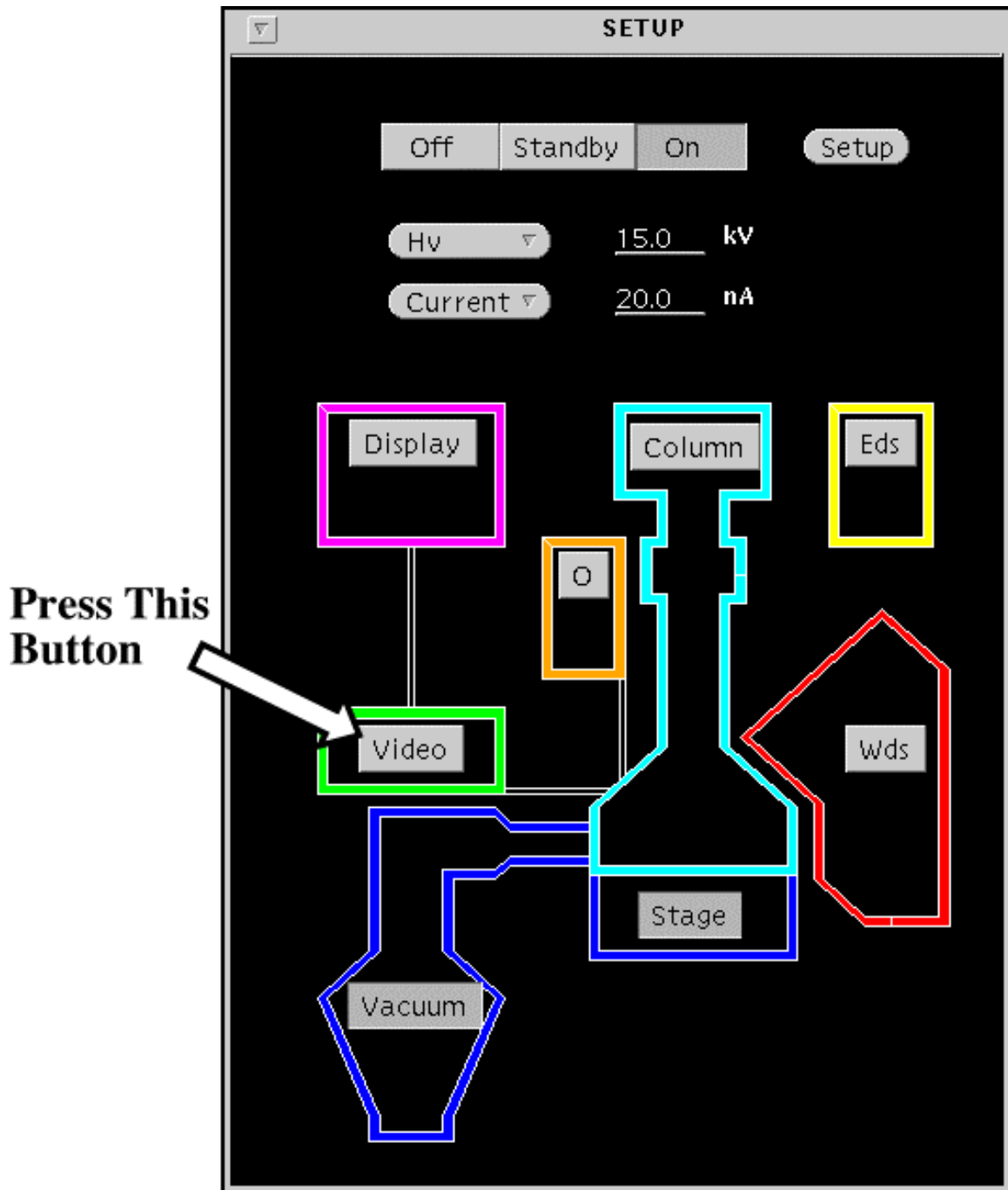


Then:

**Turn the Cup Off and
Turn the Scan On**

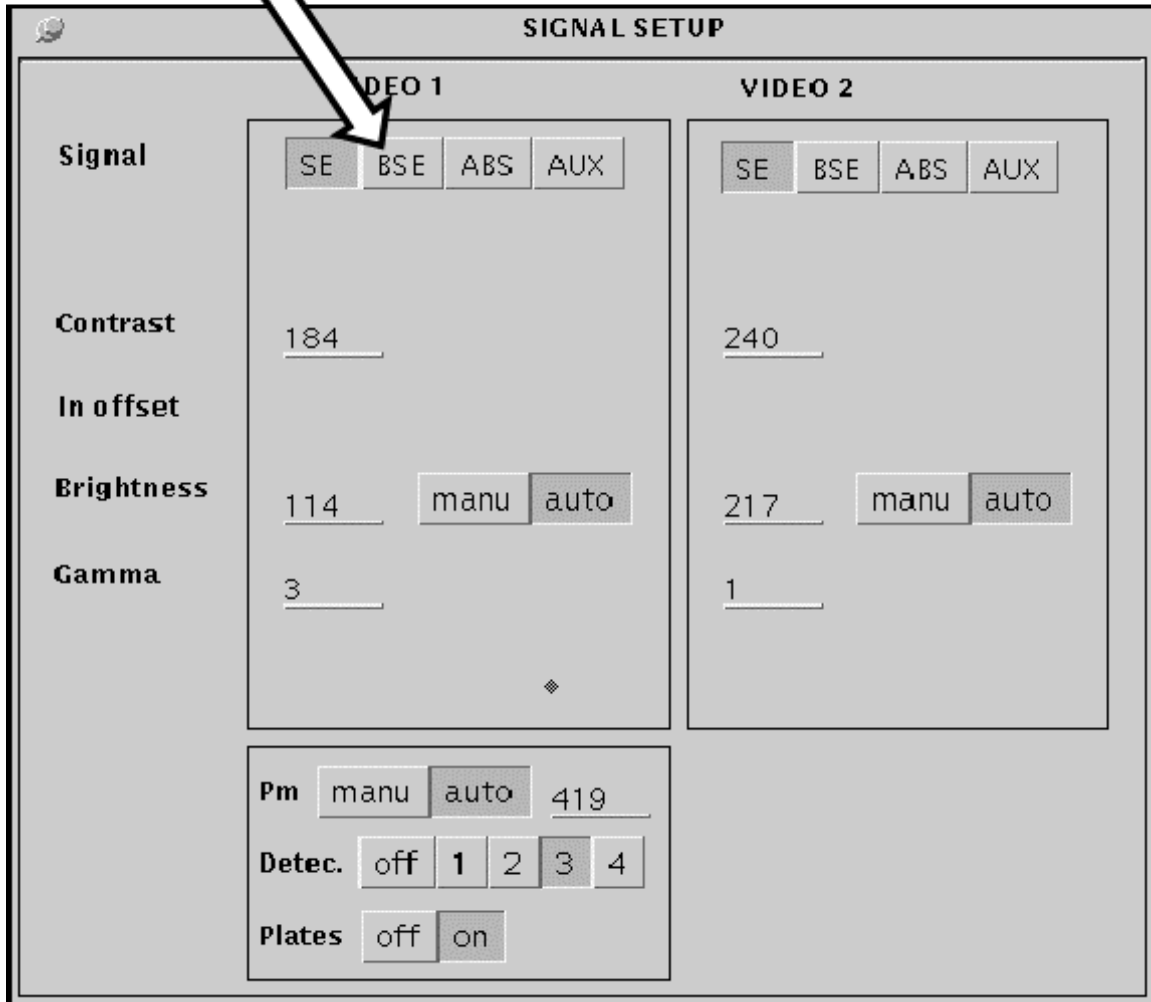


Step 2



Step 3

**Press This
Button**



The image shows a 'SIGNAL SETUP' menu with two columns for 'VIDEO 1' and 'VIDEO 2'. The 'Signal' row has buttons for 'SE', 'BSE', 'ABS', and 'AUX'. An arrow points to the 'SE' button for VIDEO 1. Other settings include Contrast, In offset, Brightness, Gamma, Pm, Detec., and Plates.

	VIDEO 1	VIDEO 2
Signal	<input checked="" type="button" value="SE"/> <input type="button" value="BSE"/> <input type="button" value="ABS"/> <input type="button" value="AUX"/>	<input checked="" type="button" value="SE"/> <input type="button" value="BSE"/> <input type="button" value="ABS"/> <input type="button" value="AUX"/>
Contrast	<input type="text" value="184"/>	<input type="text" value="240"/>
In offset		
Brightness	<input type="text" value="114"/> <input type="button" value="manu"/> <input type="button" value="auto"/>	<input type="text" value="217"/> <input type="button" value="manu"/> <input type="button" value="auto"/>
Gamma	<input type="text" value="3"/>	<input type="text" value="1"/>
◆		
Pm	<input type="button" value="manu"/> <input type="button" value="auto"/> <input type="text" value="419"/>	
Detec.	<input type="button" value="off"/> <input type="button" value="1"/> <input type="button" value="2"/> <input type="button" value="3"/> <input type="button" value="4"/>	
Plates	<input type="button" value="off"/> <input type="button" value="on"/>	

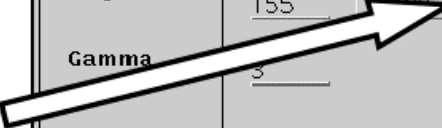
Step 4

If not already
on:

Press This
Button


SIGNAL SETUP

	VIDEO 1	VIDEO 2
Signal	<input type="button" value="SE"/> <input type="button" value="BSE"/> <input type="button" value="ABS"/> <input type="button" value="AUX"/>	<input type="button" value="SE"/> <input type="button" value="BSE"/> <input type="button" value="ABS"/> <input type="button" value="AUX"/>
Contrast	<input type="text" value="224"/> <input type="button" value="Atomic"/>	<input type="text" value="240"/>
In offset	<input type="text" value="-139"/> <input type="button" value="manu"/> <input type="button" value="auto"/>	
Brightness	<input type="text" value="155"/> <input type="button" value="manu"/> <input type="button" value="auto"/>	<input type="text" value="512"/> <input type="button" value="manu"/> <input type="button" value="auto"/>
Gamma	<input type="text" value="3"/>	<input type="text" value="1"/>
Gain	<input type="button" value="low"/> <input type="button" value="high"/>	
Coupling	<input type="button" value="ac"/> <input type="button" value="dc"/>	
Polar	<input type="button" value="ground"/> <input type="button" value="bias"/>	
Plates	<input type="button" value="off"/> <input type="button" value="1"/> <input type="button" value="2"/> <input type="button" value="3"/> <input type="button" value="4"/>	
Detector	<input type="button" value="off"/> <input type="button" value="on"/>	



Step 5

**L-Click this button
to set Roller wheels back to Stage**



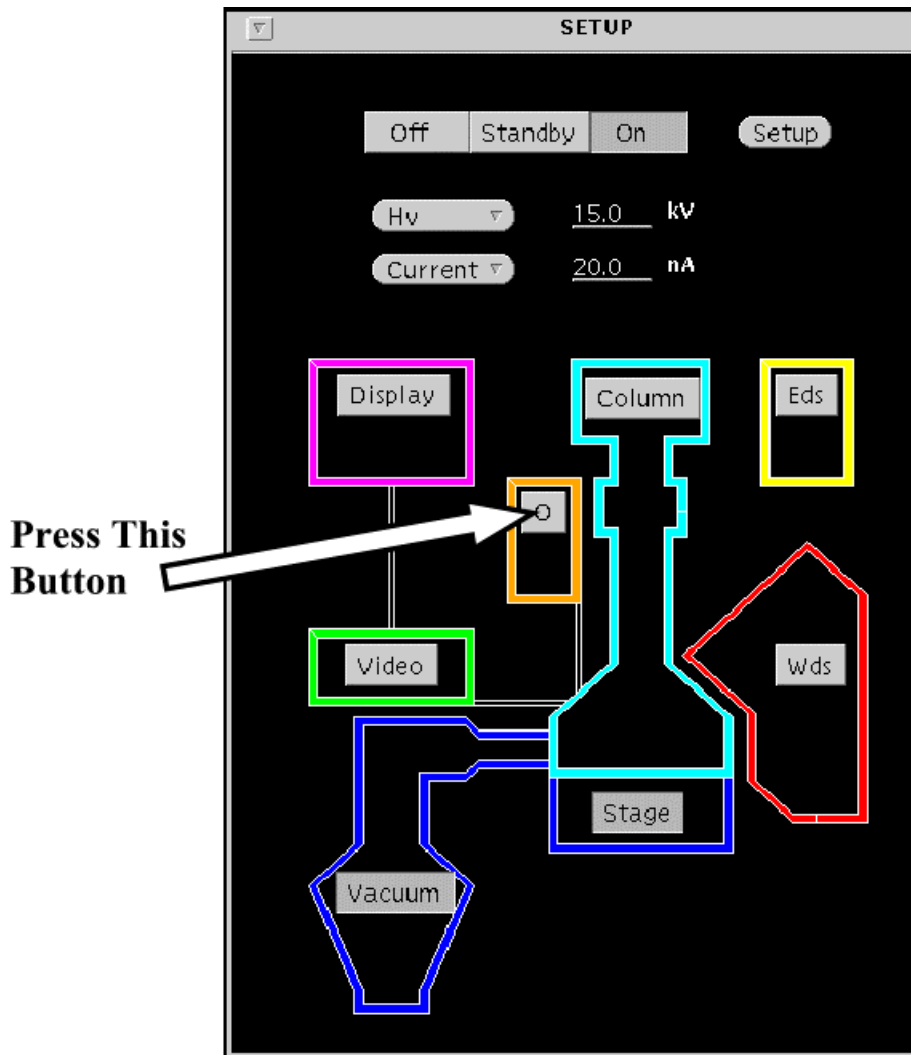
The screenshot shows a software window titled "ROLLER WHEEL CONTROL". The interface includes several control elements:

- A dropdown menu labeled "Roller choice" with a downward arrow, currently showing "Stage focus". A white arrow points to this menu.
- Buttons for "Cup" (Off/On) and "Scan" (Off/On).
- A "Current" dropdown menu set to "20.0 nA".
- "Mag" and "Photo" dropdown menus.
- A table of parameters:

Focus	Dist (um)	Dist (um)	Focus	X	Y
3272	615	615	3272	-642	4908
VS1 gamma	VS1 cont			Z	
3	184			-99	

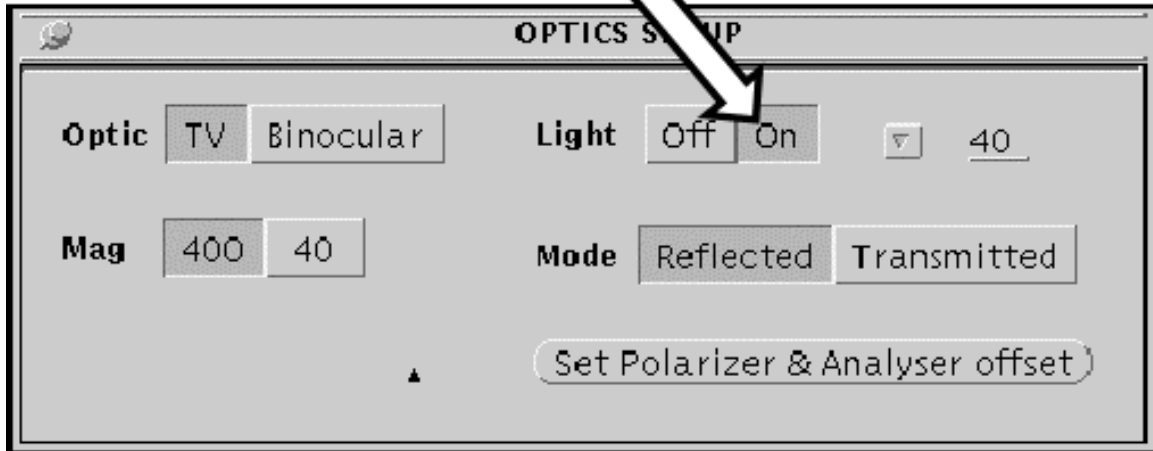
Step 6

The light on the reflected light image will probably go out. If it does and the Optics Setup window is not already open, then:



Step 7

**Press This
Button**



Next Step

Go to either:

**Analysis Instruction Set 2a – Individual Point Analysis
(p. 26)**

**Analysis Instruction Set 2b – Multi Point Analysis (p.
35)**

**Analysis Instruction Set 2c – Stage Line Scan Analysis
(p. 51)**

**Analysis Instruction Set 3 - Taking BSE Pictures (p. 66)
or**

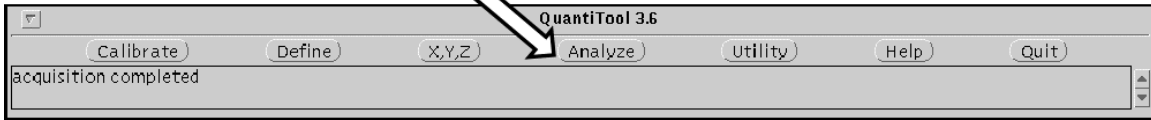
Analysis Instruction Set 4 - Taking EDS Spectra (p. 89)

Analysis: Instruction Set 2a

Analyzing Individual Points

Step 1

**Press This
Button**



Step 2

R-Click this button and select Single Point from the pop-up menu.



Step 3

R-Click this line and select your Define File name from the pop-up menu.



Step 4

Type in a comment on this line
that will help you identify this analysis later.

QuantiTool 3.6

Calibrate Done Acquire Analyze Utility Help Quit

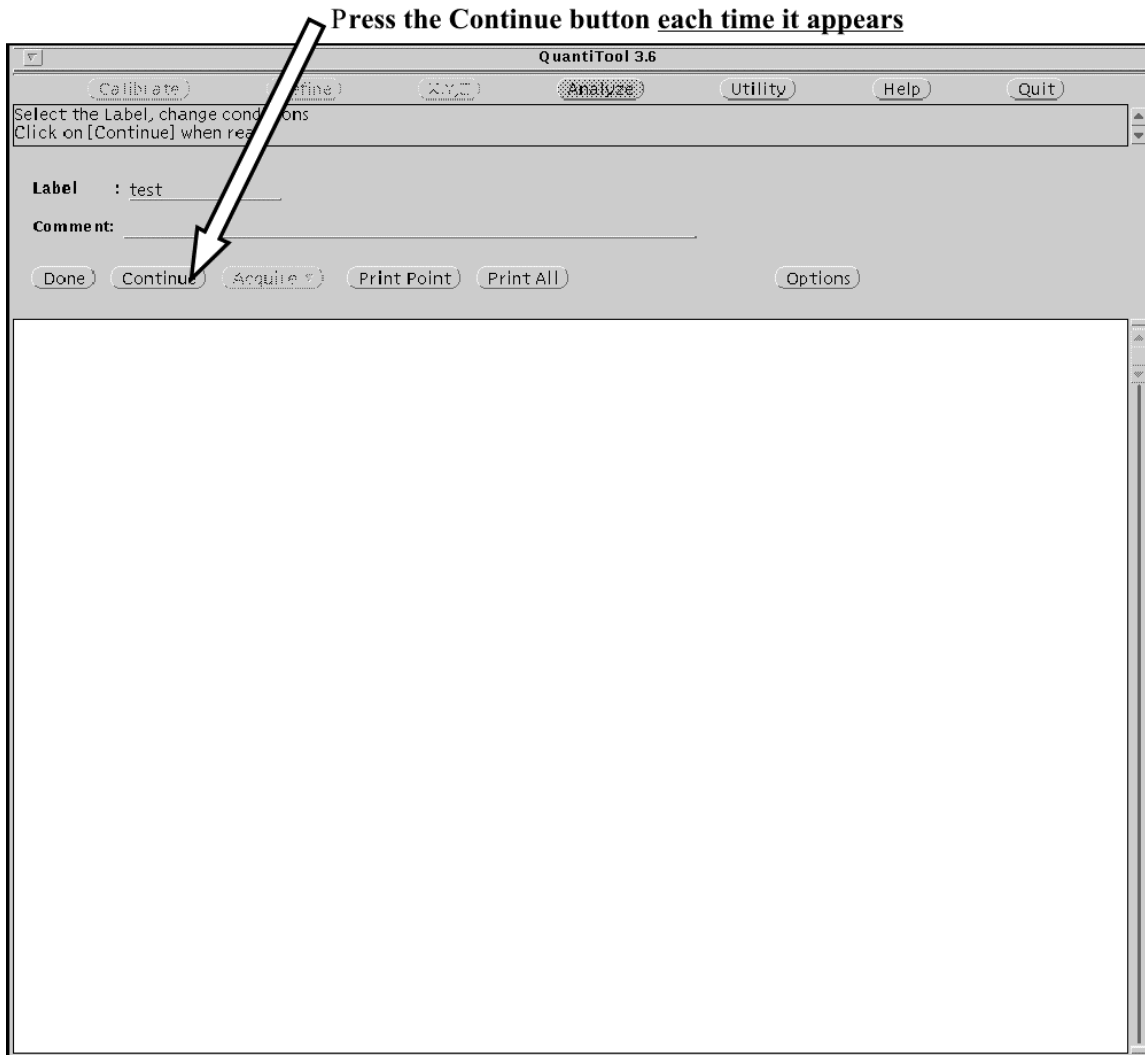
Select the Label, change conditions
Click on [Continue] when ready

Label : test

Comment: _____

Done Continue Acquire Print Point Print All Options

Step 5



Note the Continue button needs to be pressed at least twice (for all analyses after the first one).

The Continue button needs to be pressed three times for the first analysis with a define file containing one beam condition and four times for the first analysis with a define file containing two beam condition.

Step 6

The spectrometer motors will begin to move and the analysis will start.

Note: If the analysis screen disappears at this point , follow the directions given in Appendix 1 p. 101.

Step 7

When the analysis is done, the computer will beep and the results will appear on the screen.

The screenshot shows the QuantiTool 3.6 software interface. At the top, there is a menu bar with buttons for Calibrate, Define, Analyze (highlighted), Utility, Help, and Quit. Below the menu bar, a text box contains the instruction: "Select the Label, change conditions Click on [Continue] when ready".

The main area of the interface contains the following information:

Label : test
Comment: _____

Buttons: Done, Continue, Acquire, Print Point, Print All, Options

Analysis Results:

```
Ni      7.3  8.3   7.1   1.02  0.0009  8.3   0.0755
Elt.    k-ratio  Correc.
Na      0.0002  1.9513
K       0.0000  1.1313
Si      0.2062  1.2614
Mg      0.0723  1.4856
Al      0.0013  1.4552
Ca      0.1653  1.1204
Mn      0.0000  NaN
Ti      0.0001  1.2502
Fe      0.0004  1.2347
Cr      0.0002  1.2365
Ni      0.0000  1.2232

iteration : 3

Analysis no. 1 within test
```

Elt.	Conc. (wt%)	1sigma (wt%)	Norm Conc. (wt%)	Norm Conc. (at%)	Compound	Concen. (wt%)
Na	0.0408	0.010801	0.0408	0.0385	Na2O	0.055
K	0.0051	0.006179	0.0051	0.0028	K2O	0.006
Si	26.0110	0.072236	26.0333	20.0884	SiO2	55.647
Mg	10.7357	0.049611	10.7449	9.5809	MgO	17.803
Al	0.1838	0.007048	0.1839	0.1477	Al2O3	0.347
Ca	18.5205	0.076487	18.5364	10.0230	CaO	25.914
Mn	0.0000	0.000000	0.0000	0.0000	MnO	
Ti	0.0184	0.008384	0.0184	0.0083	TiO2	0.031
Fe	0.0541	0.021352	0.0541	0.0210	FeO	0.070
Cr	0.0245	0.016358	0.0245	0.0102	Cr2O3	0.036
Ni	0.0045	0.025665	0.0045	0.0017	NiO	0.006
O	44.3160		44.3540	60.0775	by stoichiometry	
total :	99.9144		100.0000	100.0000		99.914

be careful, strong correction for Mn (0.00)

Step 8

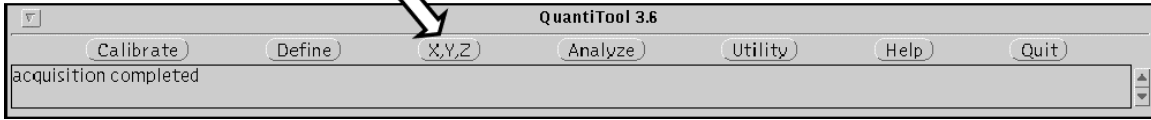
REPEAT STEPS 4 – 7 for each analysis.

Analysis: Instruction Set 2b

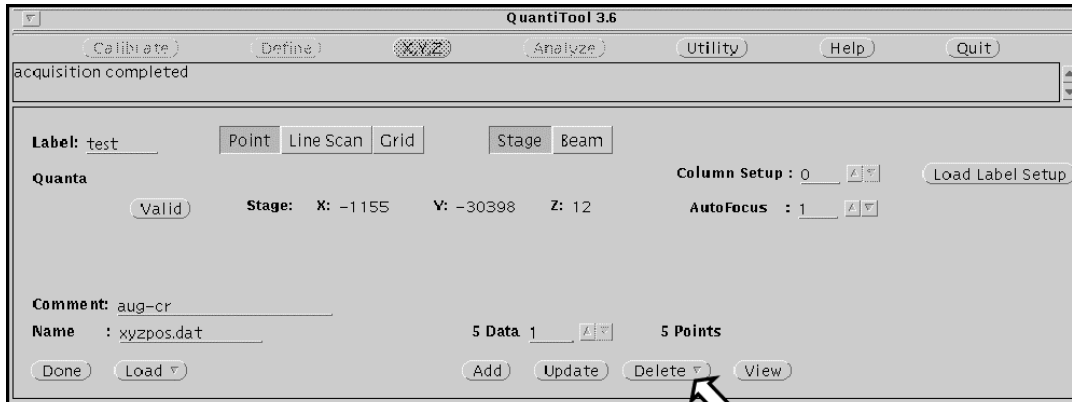
Multi Point Analysis

Step 1

**Press This
Button**

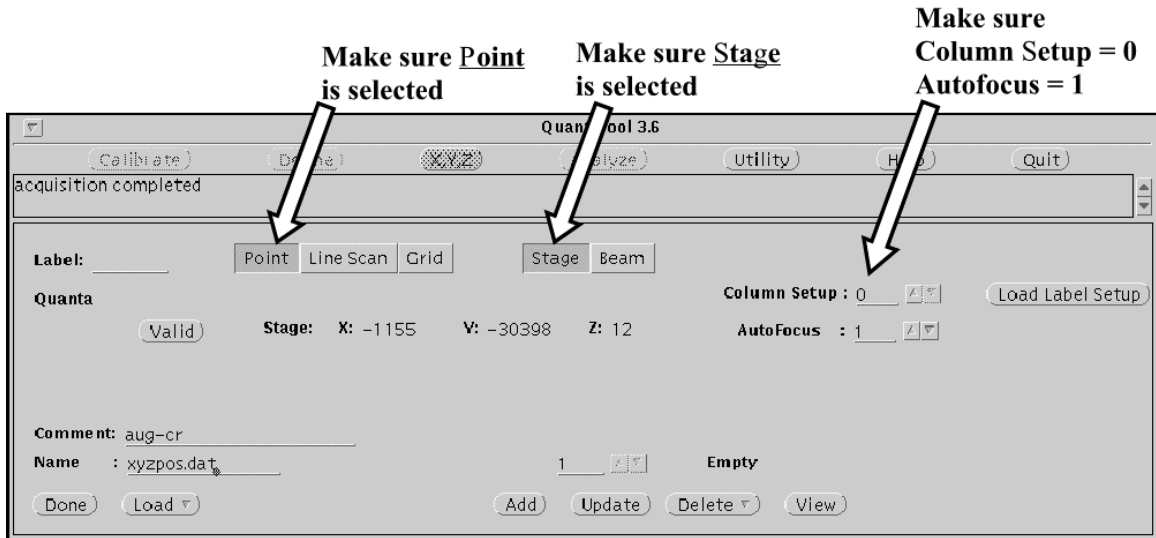


Step 2



R-Click this button and select Delete All from the pop-up menu.

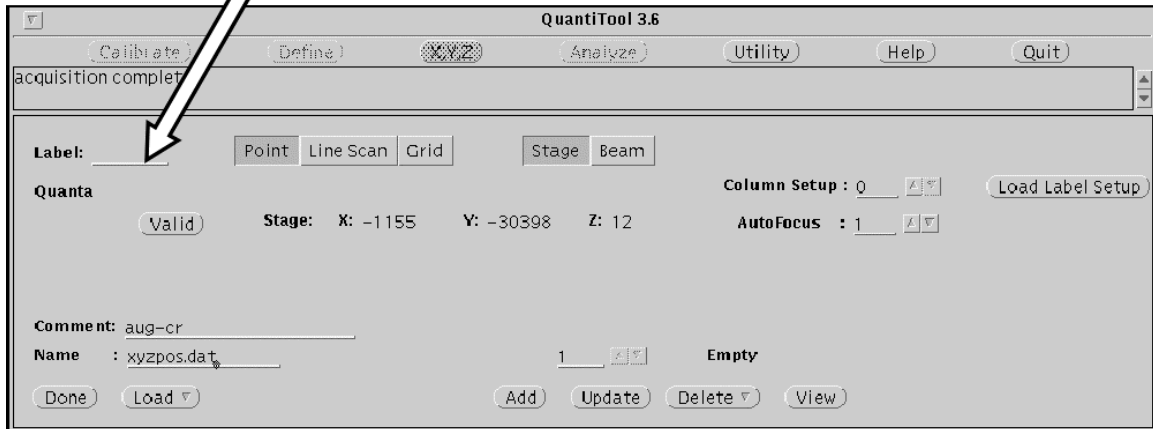
Step 3



Note: If you needed to change from Line Scan to Point you will need to reset the probe marker. See Appendix 2 on p. 102 for instructions.

Step 4

R-Click on this line and select your define file from the pop-up menus



Step 5

Turn the **Cup Off** and
Turn the **Scan On**

ROLLER WHEEL CONTROL

Roller choice ▾ Cup Off On Current ▾ 20.0 nA Stage focus

Scan Off On Mag ▾ Photo ▾

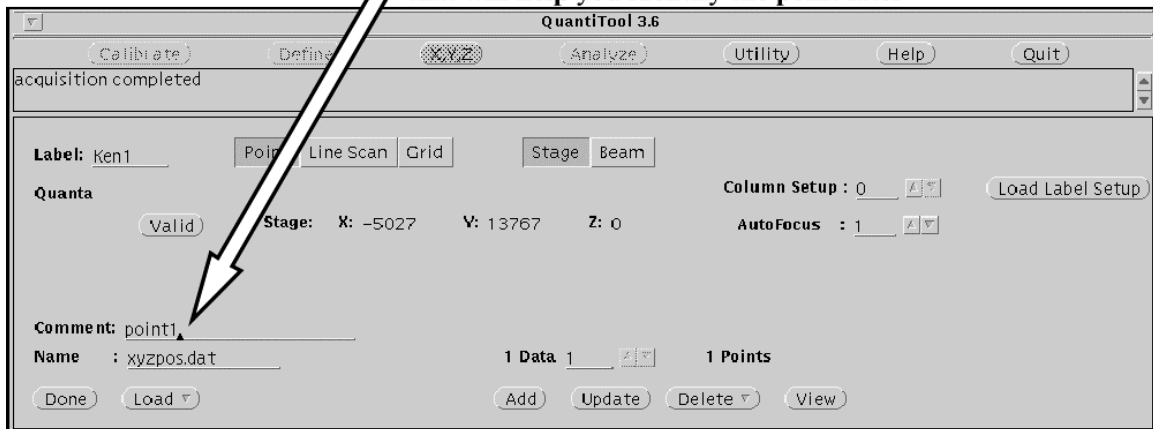
X	Y	Dist (um)	Focus	X	Y
-15391	15983	246	3272	-15391	15983
Z				Z	
-6				-6	

Step 6

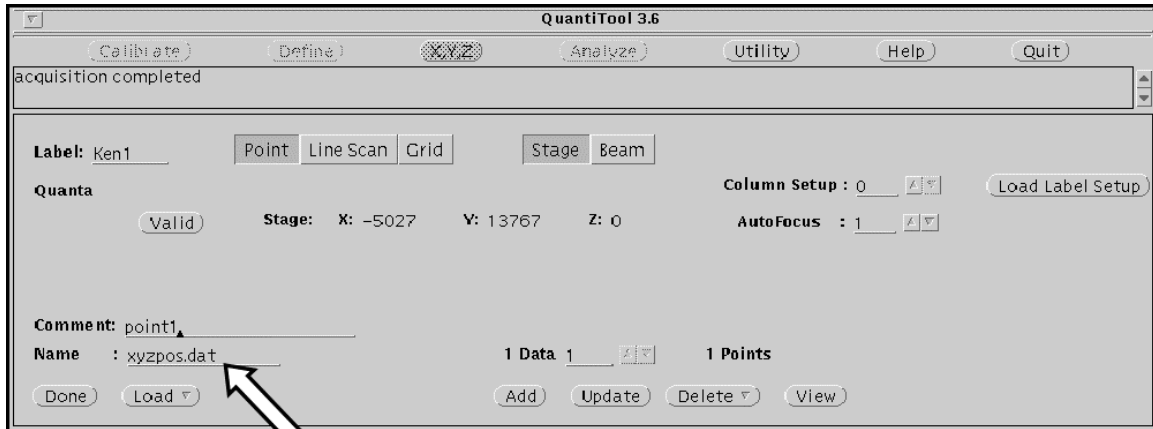
Move to the first spot that you want to analyze and focus the optical image.

Then:

**Type in a comment on this line
that will help you identify the point later**



Step 7



VERY IMPORTANT!
DO NOT change the entry on this line.

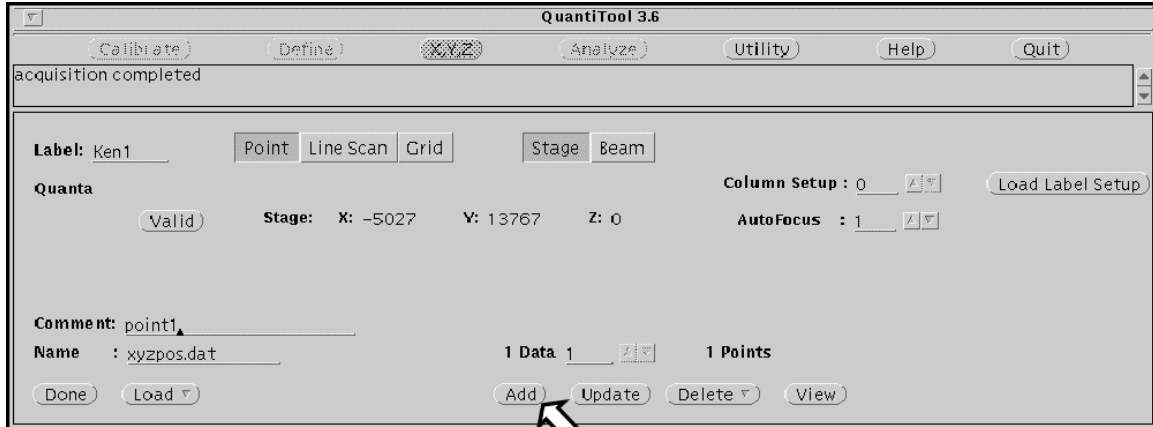
If you do be sure to change it back to **xyzpos.dat before doing anything else.**

Step 8

**Press This Button
to set the stage position**



Step 9



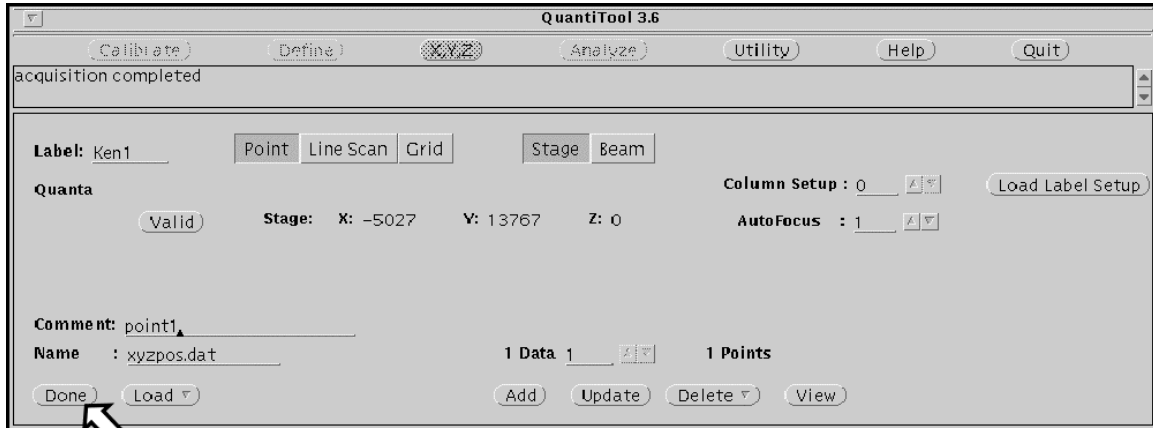
**Press This Button
to add the point to the analysis list**

Step 10

REPEAT STEPS 6 – 9 for each analysis point.

Step 11

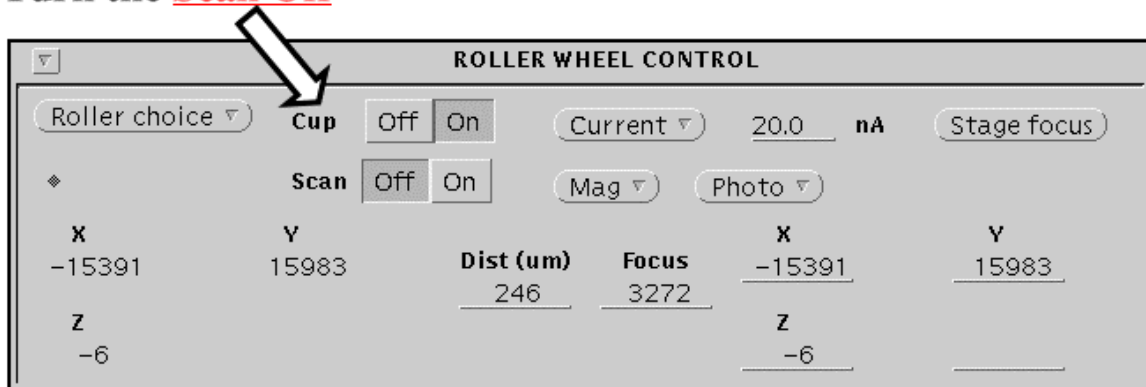
When you're finished setting up points:



Press This Button

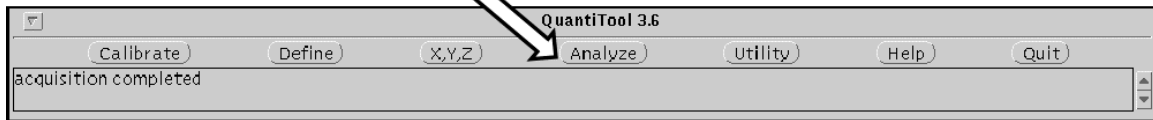
Step 12

Turn the **Cup On** and
Turn the **Scan Off**



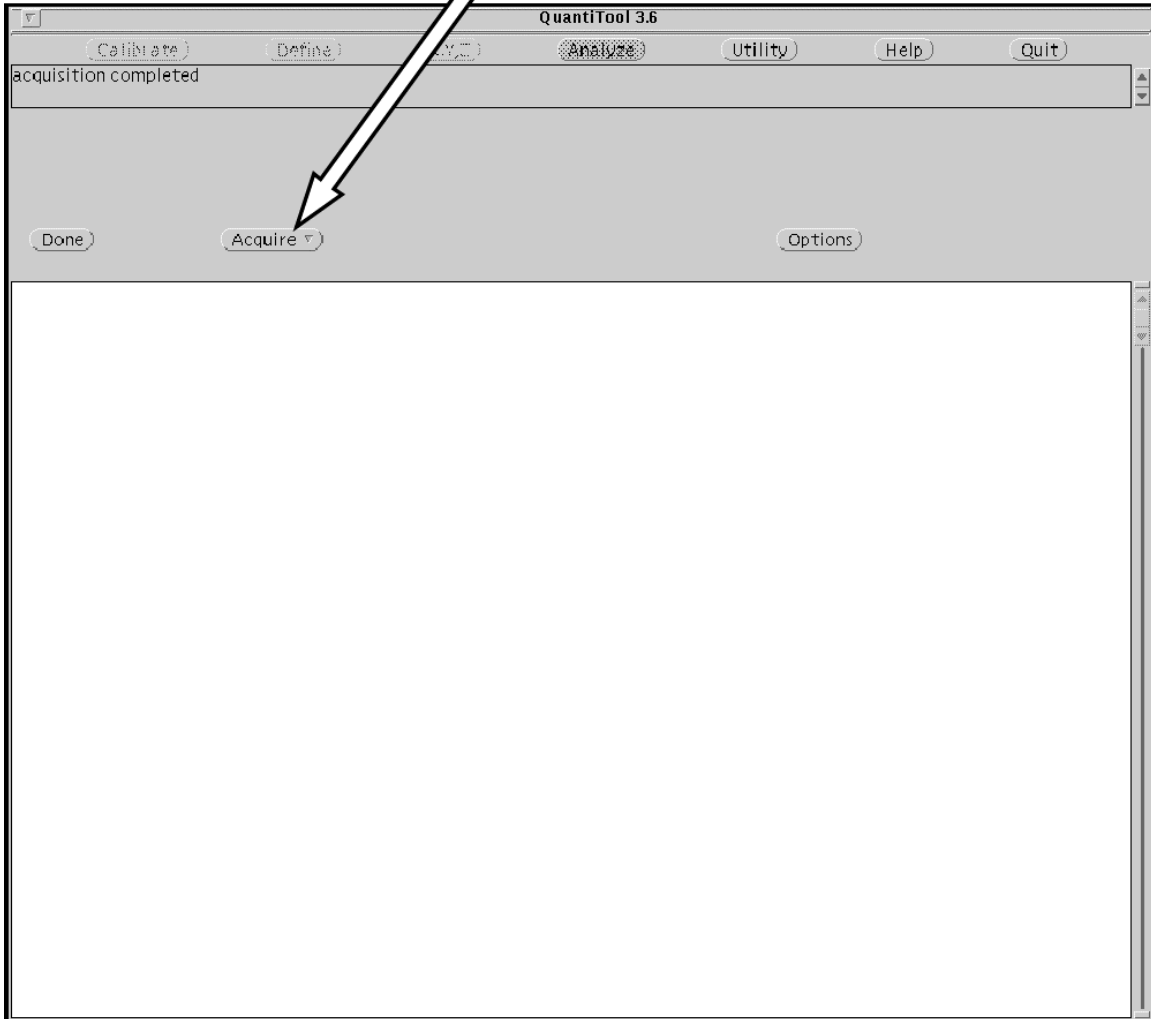
Step 13

**Press This
Button**



Step 14

R-Click this button and select Multi Point_ from the pop-up menu.



Step 15

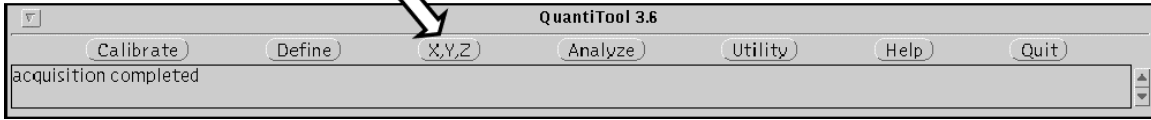
The analysis list will run. Watch the first point or two to make sure things are working the way you want.

Analysis: Instruction Set 2c

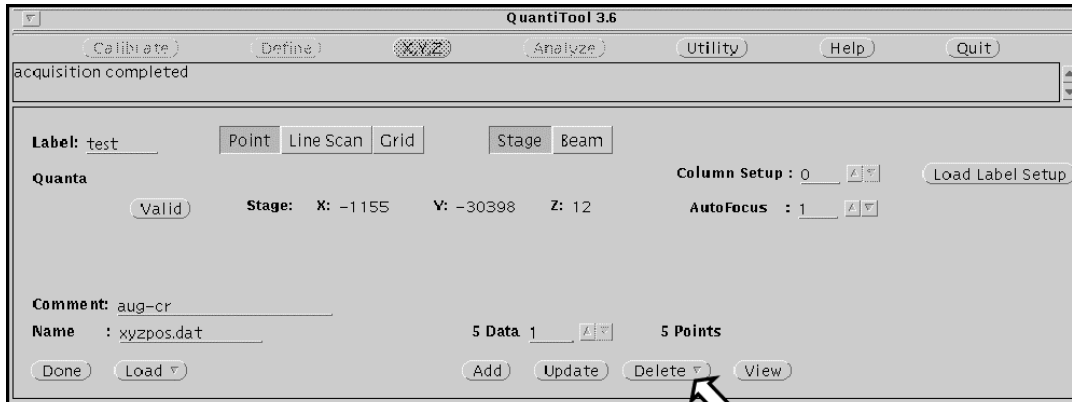
Stage Line Scan Analysis

Step 1

**Press This
Button**

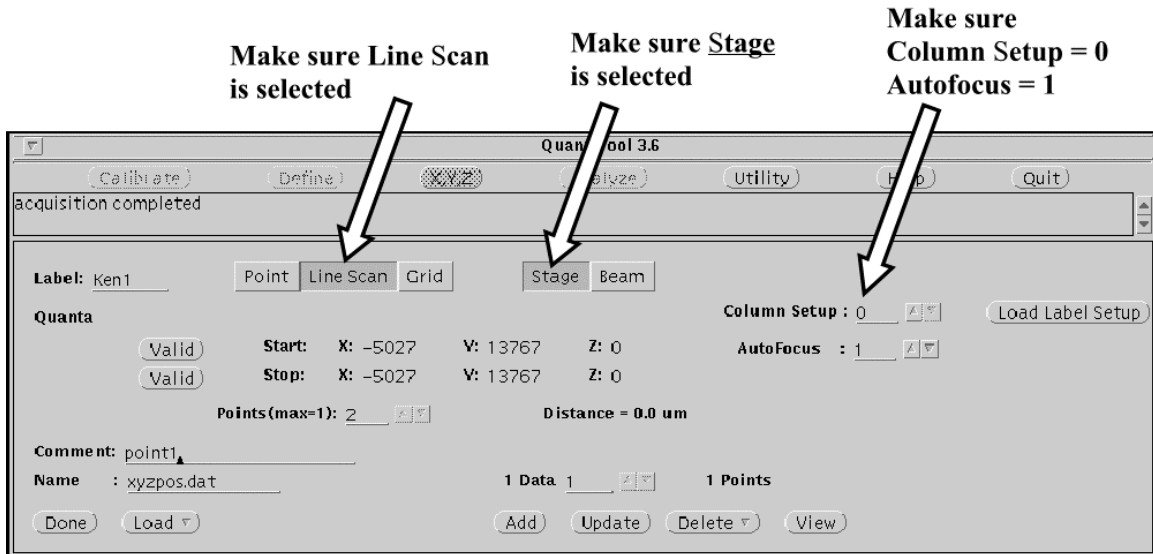


Step 2



R-Click this button and select Delete All from the pop-up menu.

Step 3



Note 1: If you needed to change from Point to Line Scan you will need to reset the probe marker. See Appendix 2 on p. 102 for instructions.

Note 2: If you see more than one number after the word Distance - Press Done and start again with Step 1.

Step 4

Turn the **Cup Off** and
Turn the **Scan On**

ROLLER WHEEL CONTROL

Roller choice ▾ Cup Off On Current ▾ 20.0 nA Stage focus

Scan Off On Mag ▾ Photo ▾

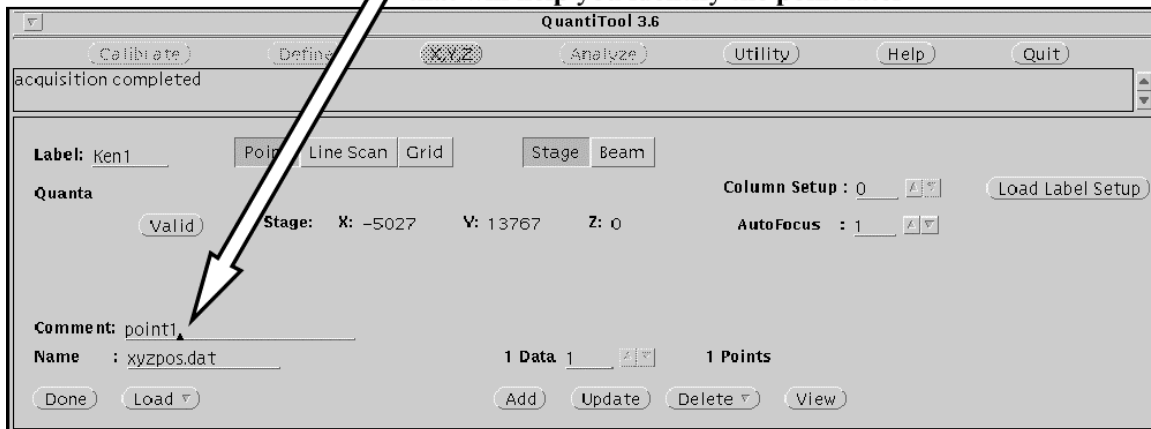
X	Y	Dist (um)	Focus	X	Y
-15391	15983	246	3272	-15391	15983
Z				Z	
-6				-6	

Step 5

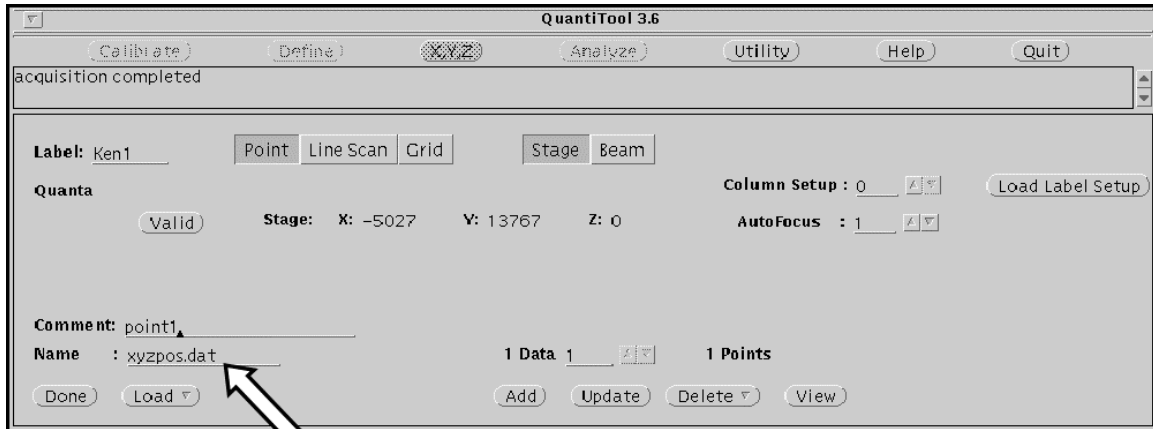
Move to the first spot that you want to analyze and focus the optical image.

Then:

**Type in a comment on this line
that will help you identify the point later**



Step 6



VERY IMPORTANT!
DO NOT change the entry on this line.

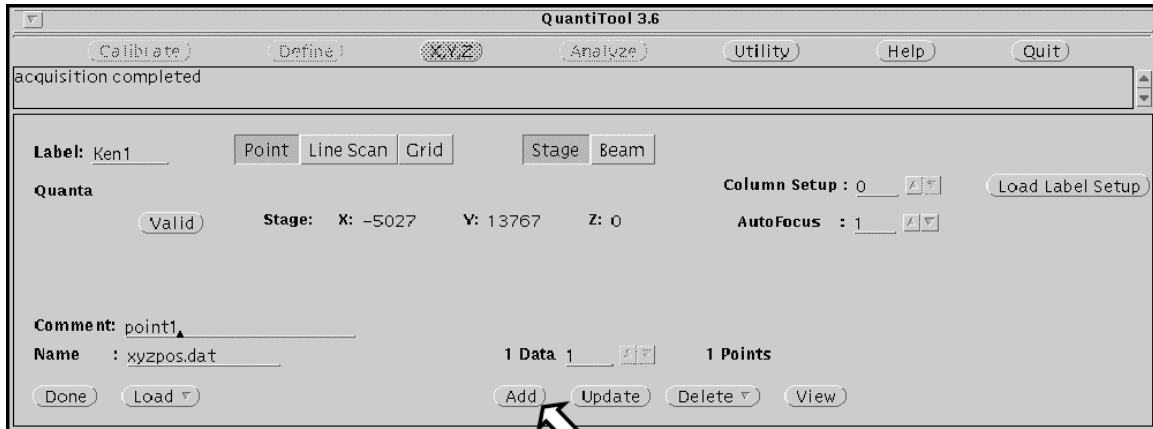
If you do be sure to change it back to **xyzpos.dat before doing anything else.**

Step 7

**Press This Button
to set the stage position**



Step 8



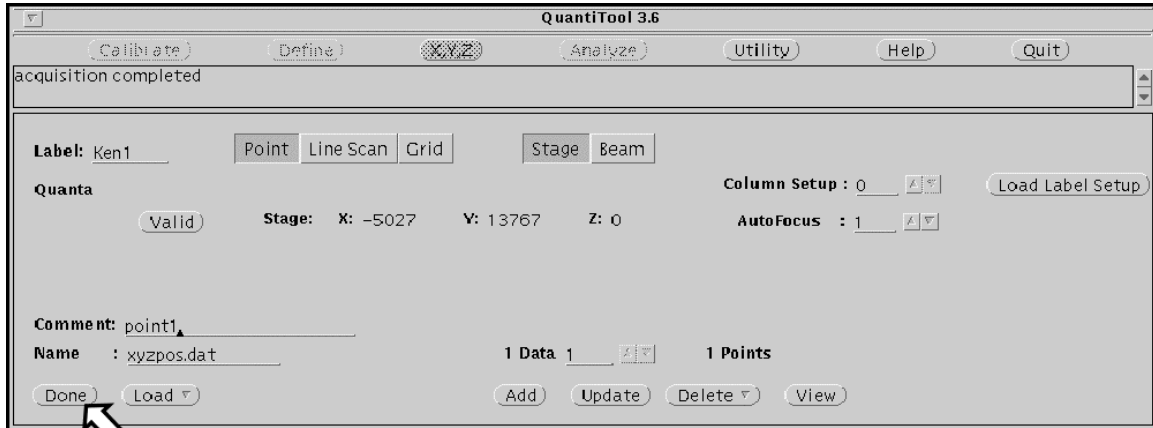
**Press This Button
to add the point to the analysis list**

Step 9

REPEAT STEPS 5 – 8 for each analysis point.

Step 11

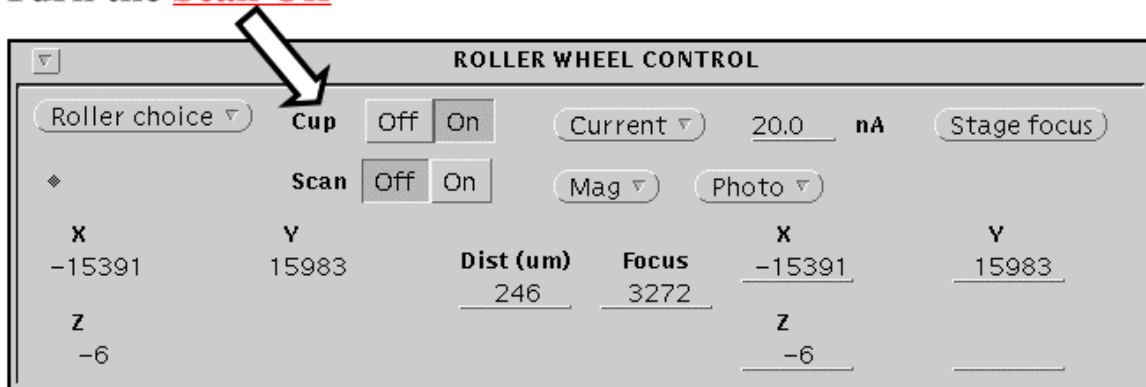
When you're finished setting up points:



Press This Button

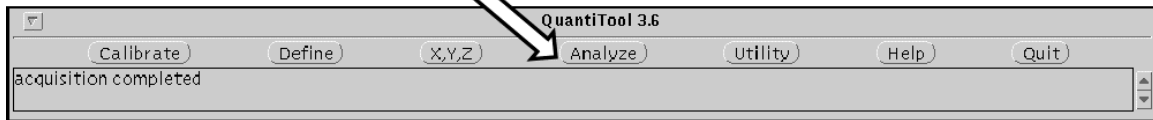
Step 12

Turn the **Cup On** and
Turn the **Scan Off**



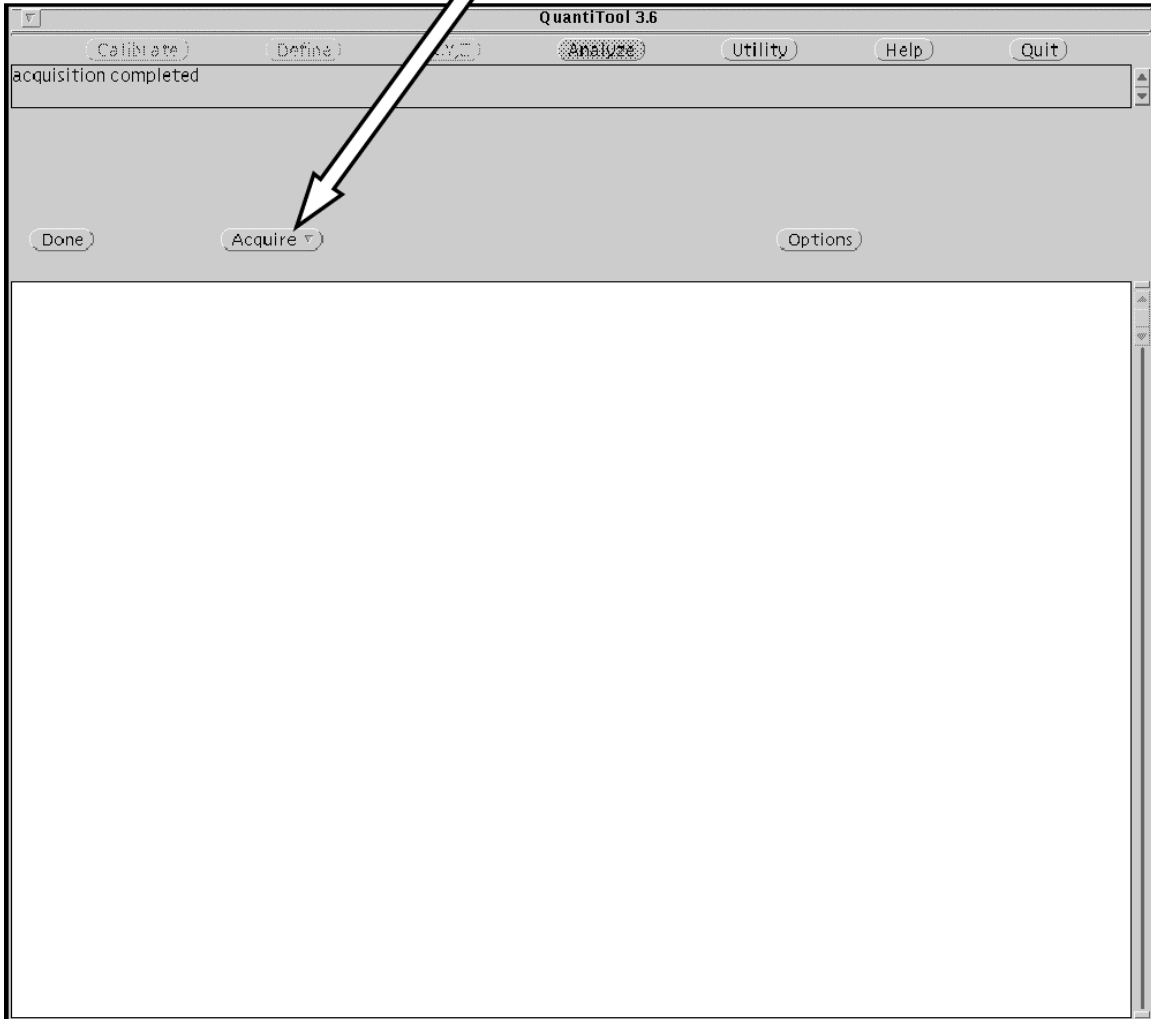
Step 13

**Press This
Button**



Step 14

R-Click this button and select Multi Point_ from the pop-up menu.



Step 15

The analysis list will run. Watch the first point or two to make sure things are working the way you want.

Analysis: Instruction Set 3

Taking BSE Pictures

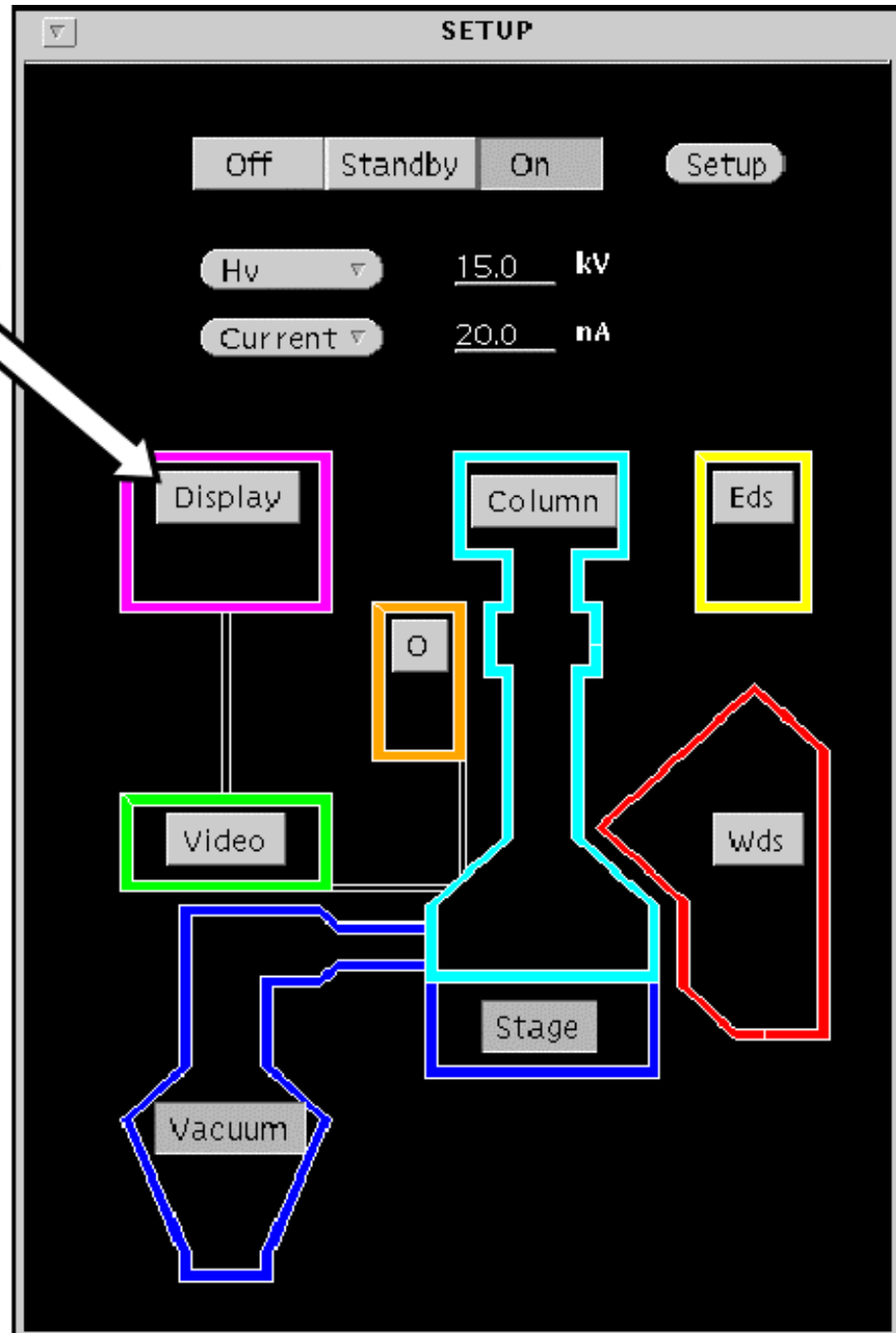
1: Obtain the Image

Step 1

Get the image you want to take a picture of on the BSE monitor screen.

Step 2

Press This Button



Step 3

IMAGE SETUP

ACQUISITION

Scanning mode Observation Fast display Off

Resolution 256 * 192 Frame time (s) 0.033

DISPLAY

Display Camera Marking Off Markers View

M1 VS1 Effects Off Check Center Split

M2 VS2

XR1 wds 1 2 3 4 Zoom Aver. time (s) 0.3

XR2 wds 1 2 3 4 Label 0

Monitoring Trace Distance meas.

SCANNING Off On

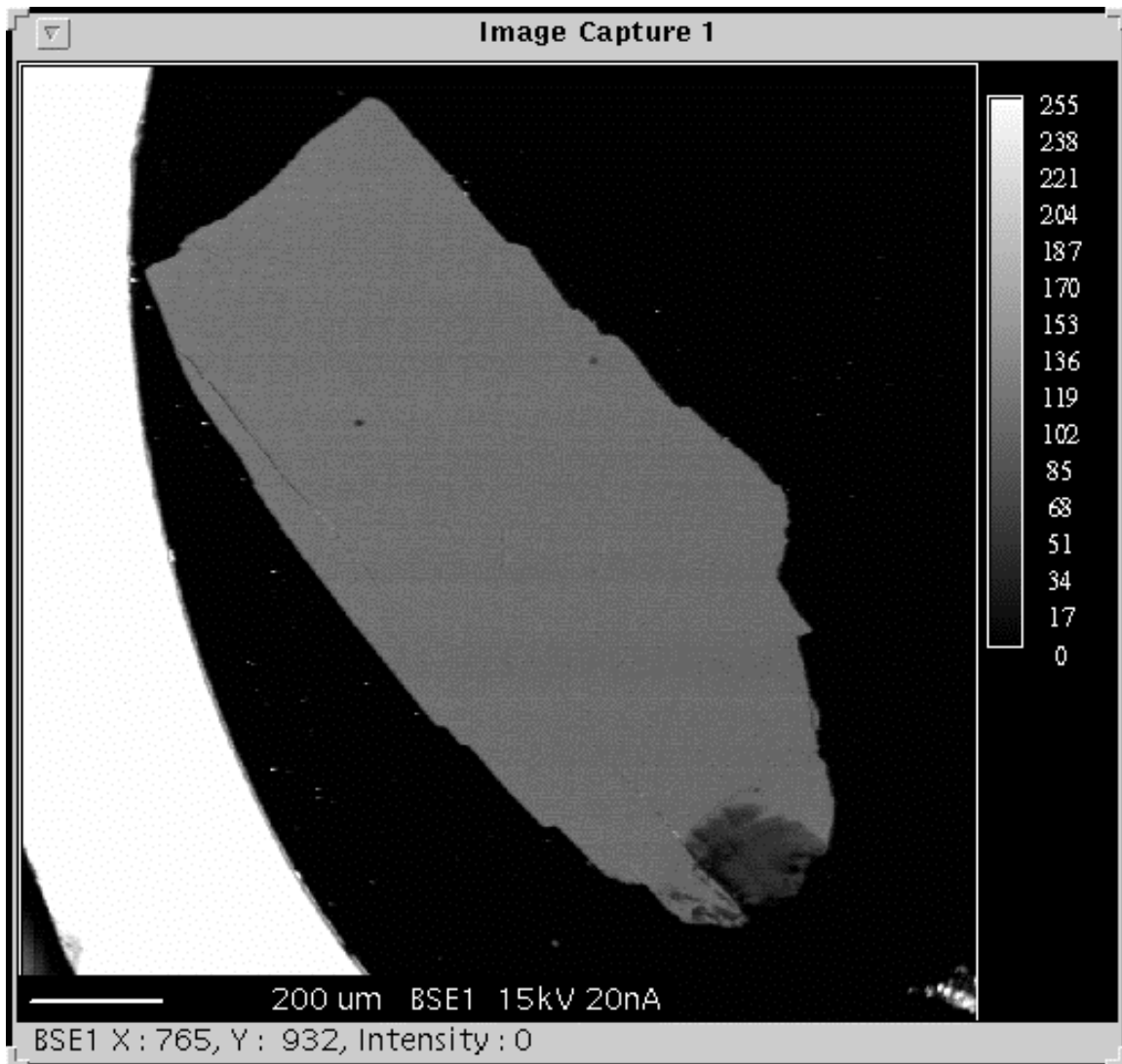
Probe marker (um) X 0.0 Y 0.0 Reset

Rotation (a) 0.0 Reset Off On

R-Click this button and select On from the pop-up menu.

Step 4

The picture will appear on the Sun computer screen.



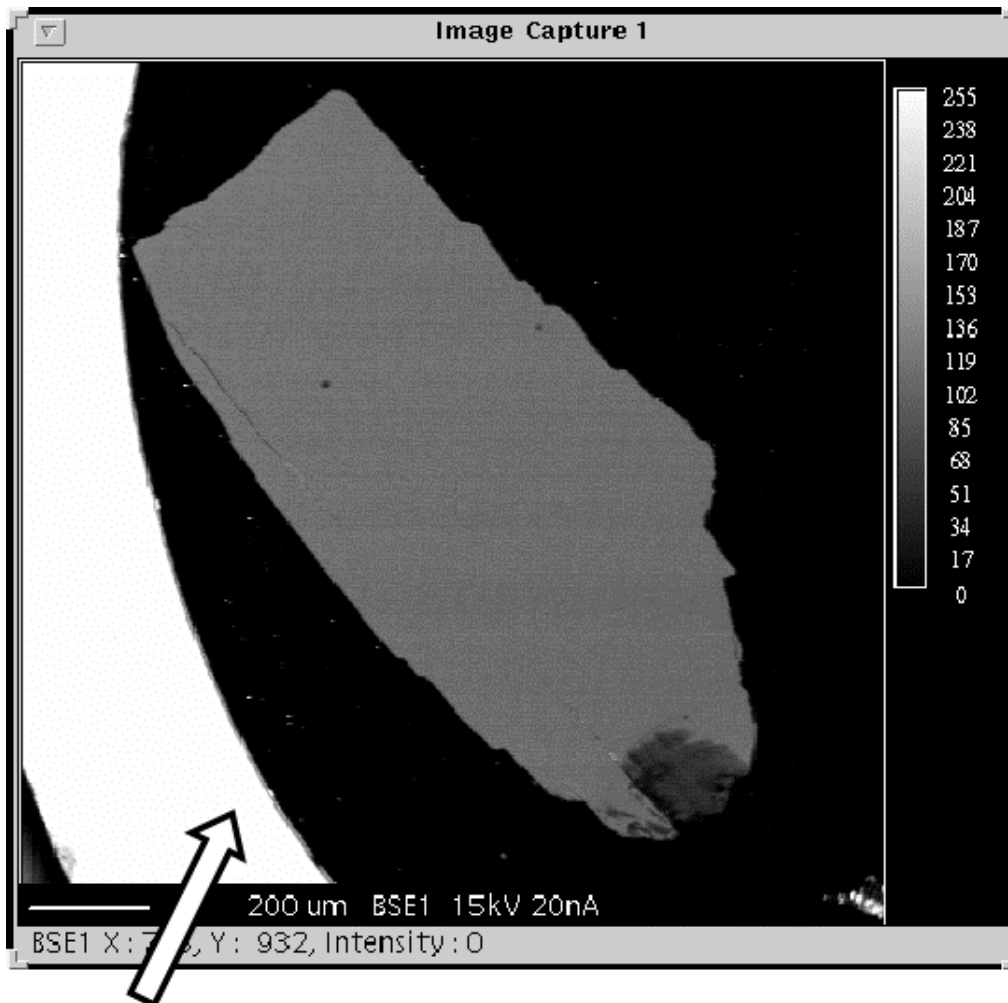
Step 5

If you need to adjust the brightness or contrast of the image follow the instructions in Appendix 3.

2: Save the Image as a tif file

Step 1

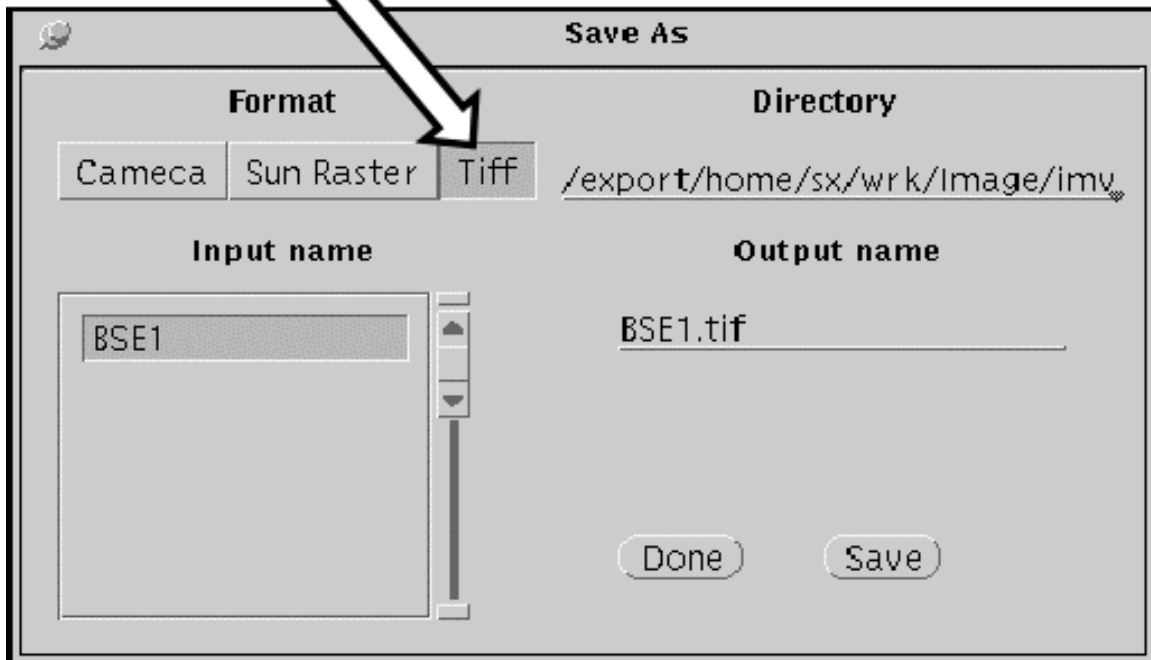
When you are ready to save the picture as a tiff file:



**R-Click on the picture
and select Save As
from the pop-up menu.**

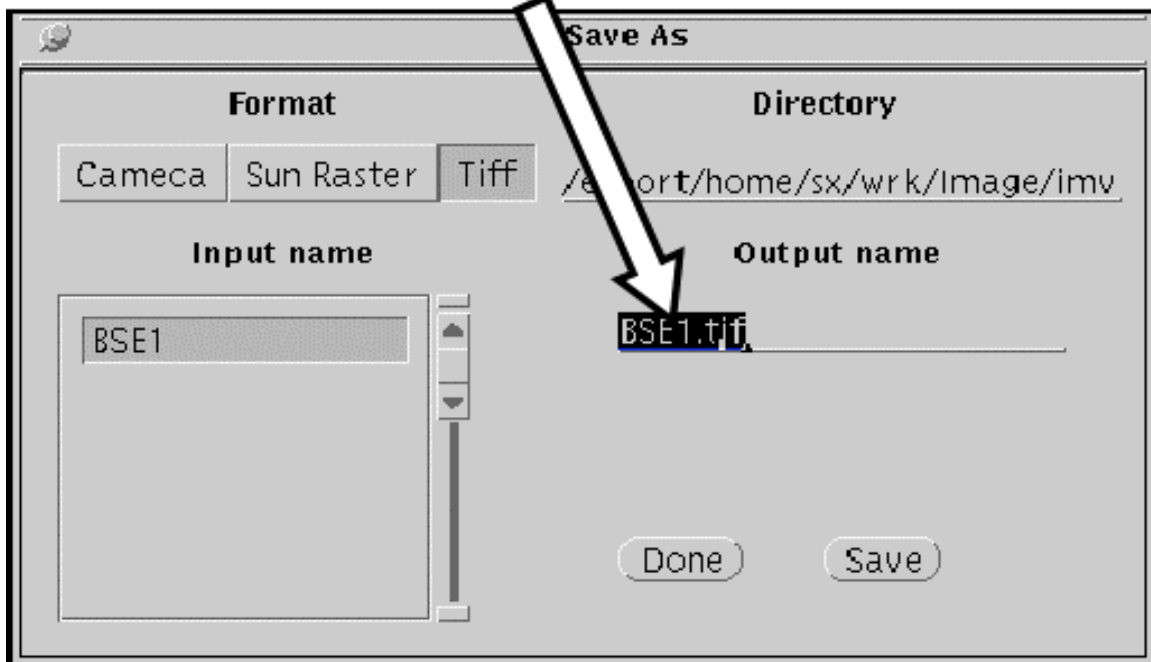
Step 2

Make sure this button is set to Tiff



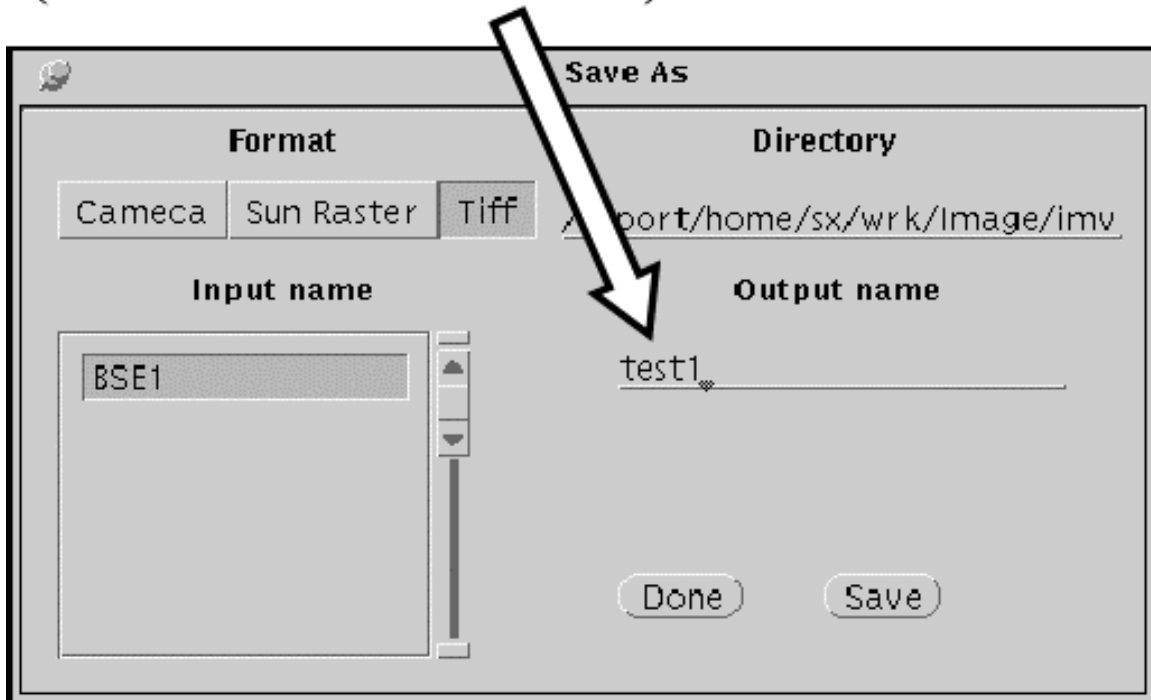
Step 3

Hold down the left mouse button and highlight this line. Then press the backspace key to delete it.

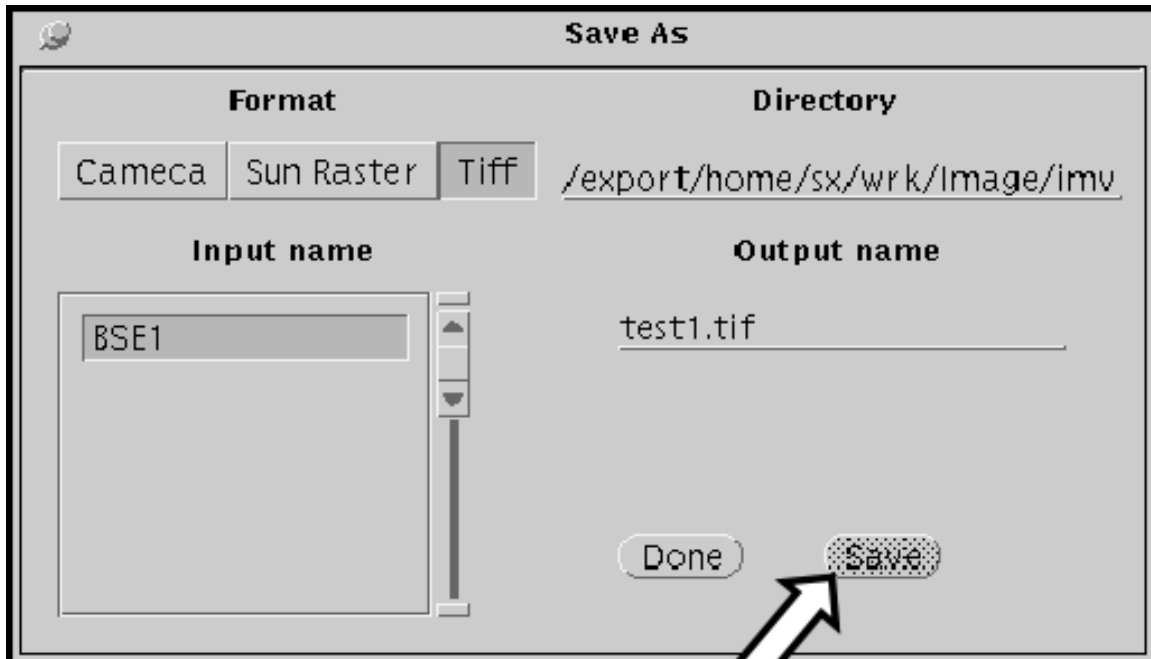


Step 4

**Type in a name to call the tiff file.
(Do not add .tif at the end).**



Step 5

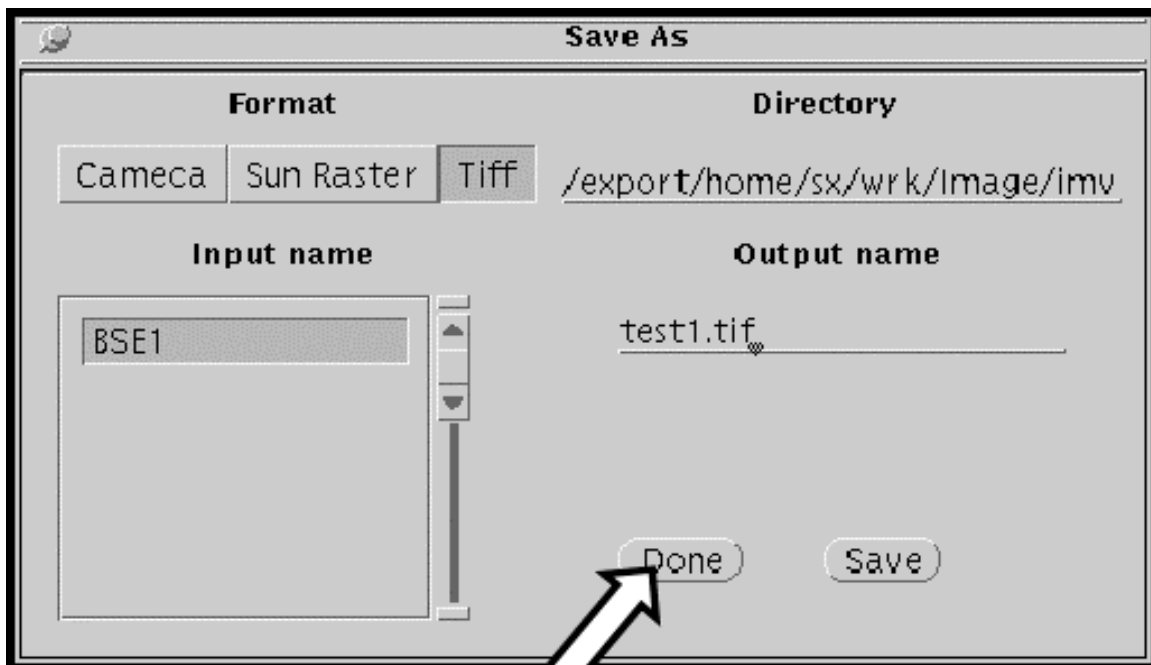


**Press This
Button**

Step 6

The save button will be grayed out until the picture is saved.

When it is done:



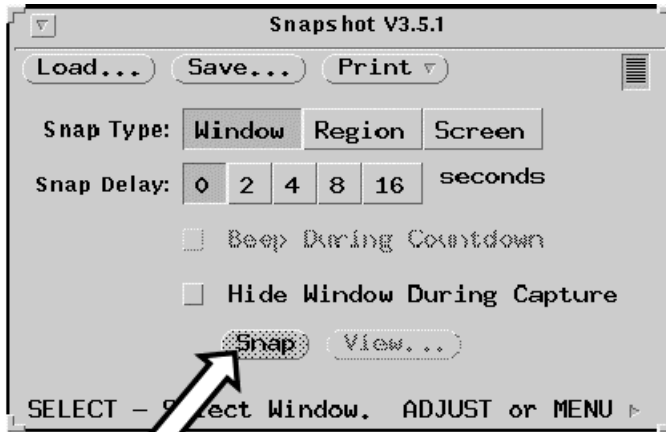
**Press This
Button**

3: Print the Image on Paper

Step 1

Double click on the camera icon (Snapshot) on the blue background of the Sun computer.

Step 2

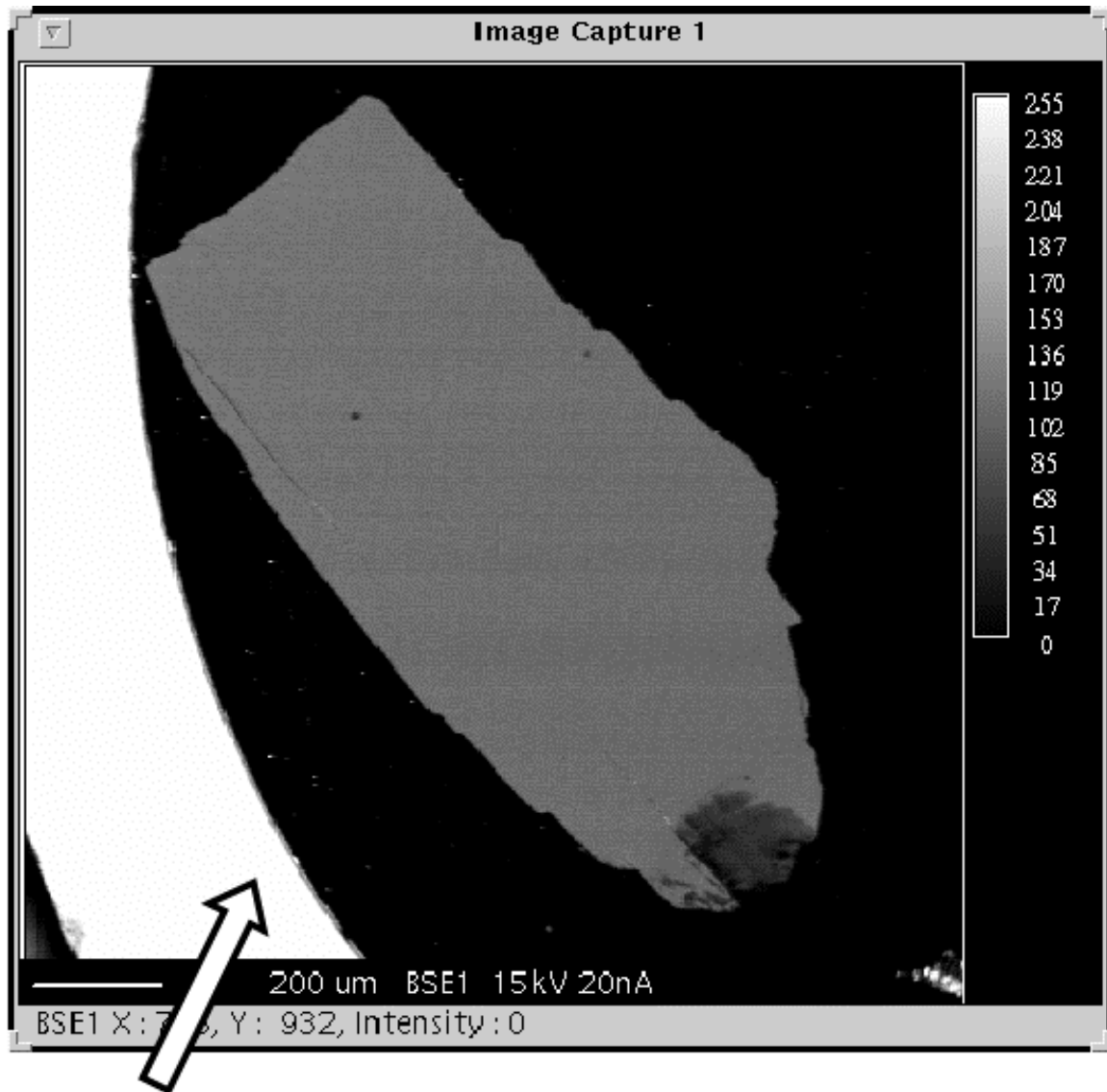


**Press This
Button**

Then:

L-Click on the button - Discard Snapshot.

Step 3



L-Click on the picture.

Step 4



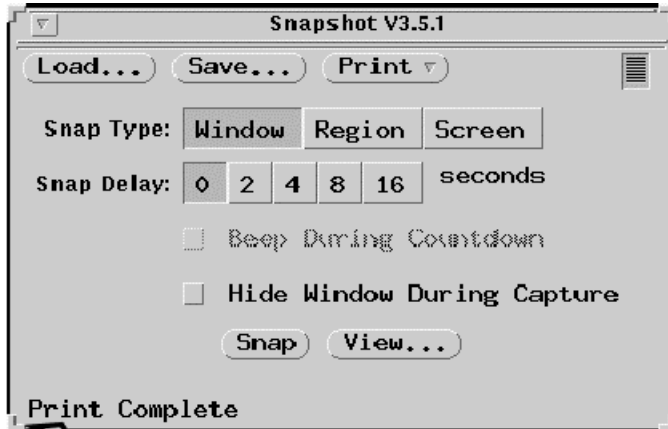
Wait for this message to appear

Then:

Press This
Button



Step 5



This message will appear when the picture has printed

4: Turn Fast display Off

Step 1

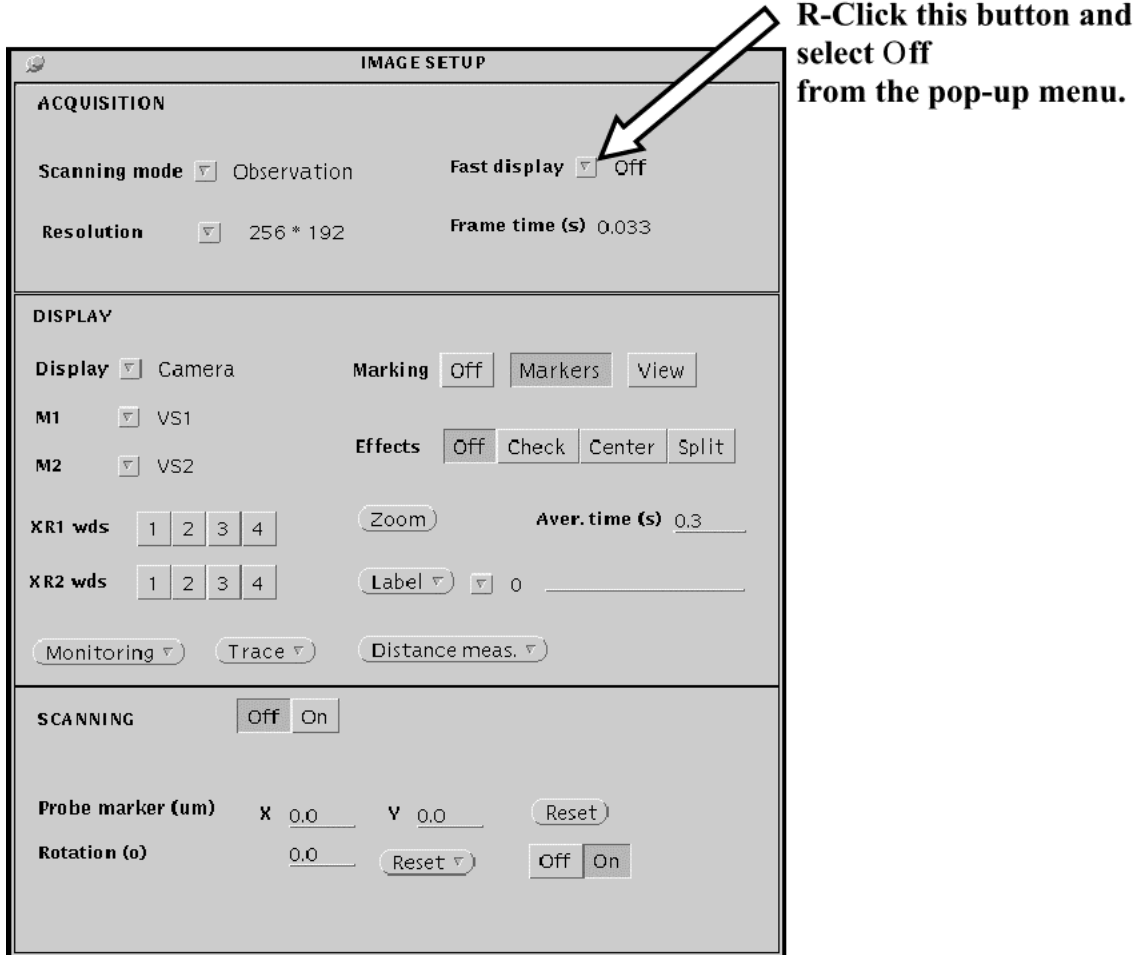


IMAGE SETUP

ACQUISITION

Scanning mode ▾ Observation Fast display ▾ Off

Resolution ▾ 256 * 192 Frame time (s) 0.033

DISPLAY

Display ▾ Camera Marking Off Markers View

M1 ▾ VS1 Effects Off Check Center Split

M2 ▾ VS2

XR1 wds 1 2 3 4 Zoom Aver. time (s) 0.3

XR2 wds 1 2 3 4 Label ▾ ▾ 0

Monitoring ▾ Trace ▾ Distance meas. ▾

SCANNING Off On

Probe marker (um) X 0.0 Y 0.0 Reset

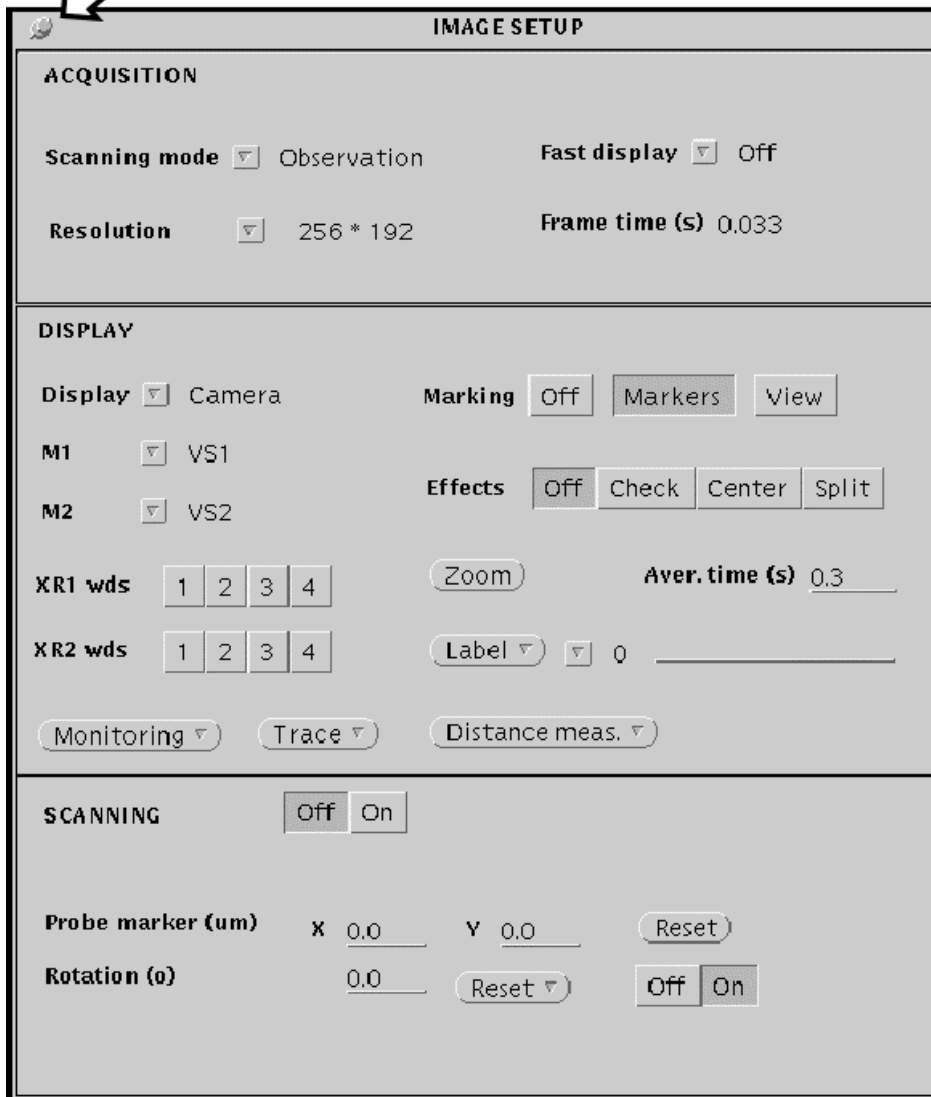
Rotation (o) 0.0 Reset ▾ Off On

R-Click this button and select Off from the pop-up menu.

Step 2

If you are done taking pictures:

L-Click this pin to close the Image Setup window.



The screenshot shows a software window titled "IMAGE SETUP" with a standard Windows-style title bar containing a pin icon on the left. An arrow points to this pin icon with the text "L-Click this pin to close the Image Setup window." The window is divided into three main sections: ACQUISITION, DISPLAY, and SCANNING.

ACQUISITION

Scanning mode	▼ Observation	Fast display	▼ Off
Resolution	▼ 256 * 192	Frame time (s)	0,033

DISPLAY

Display	▼ Camera	Marking	Off	Markers	View		
M1	▼ VS1	Effects	Off	Check	Center	Split	
M2	▼ VS2						
XR1 wds	1 2 3 4	Zoom		Aver. time (s)	0,3		
XR2 wds	1 2 3 4	Label	▼ 0				
		Monitoring	▼	Trace	▼	Distance meas.	▼

SCANNING

		Off	On			
Probe marker (um)	X	0,0	Y	0,0	Reset	
Rotation (o)		0,0	Reset	▼	Off	On

Analysis: Instruction Set 4

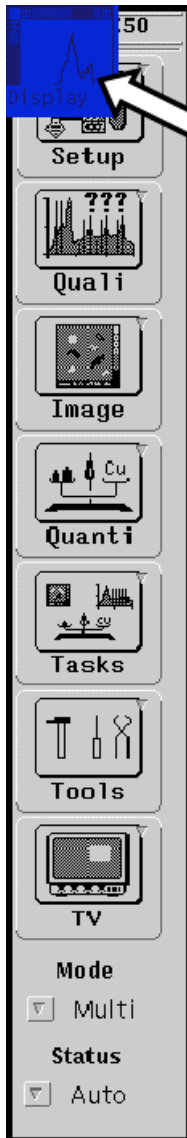
Taking EDS Spectra

Step 1

Move to the first spot that you want to analyze and focus the optical image.

Step 2

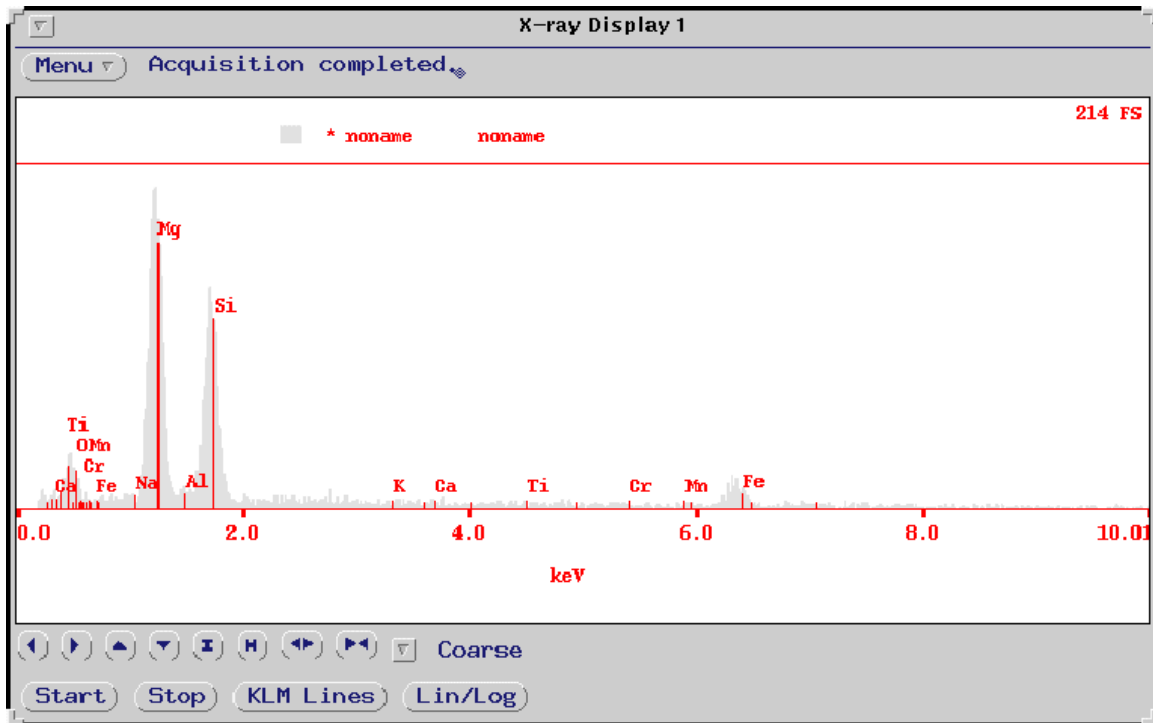
Open the EDS window (if not already open).



**Double Click
on this Icon**

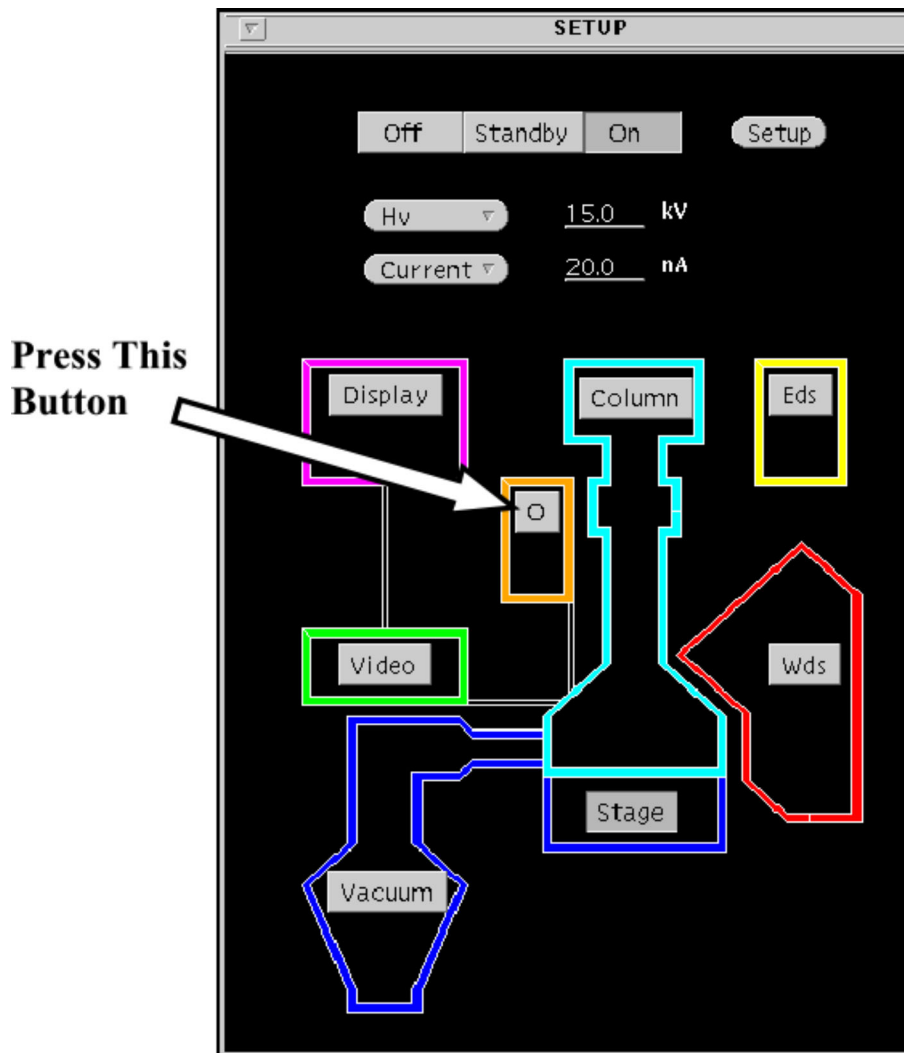
Step 3

The EDS window will appear.



Step 4

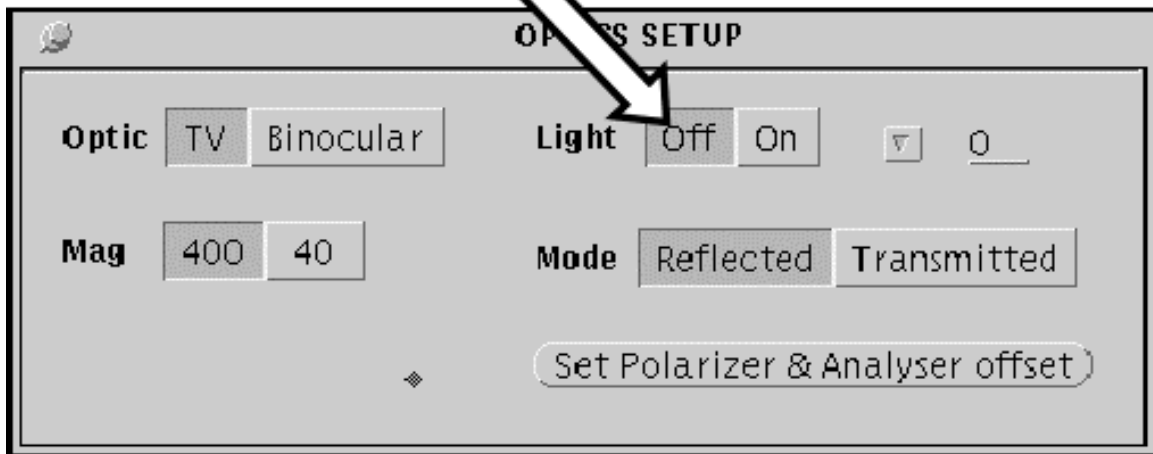
Open the Optics window (if not already open).



Step 5

Turn the light off.

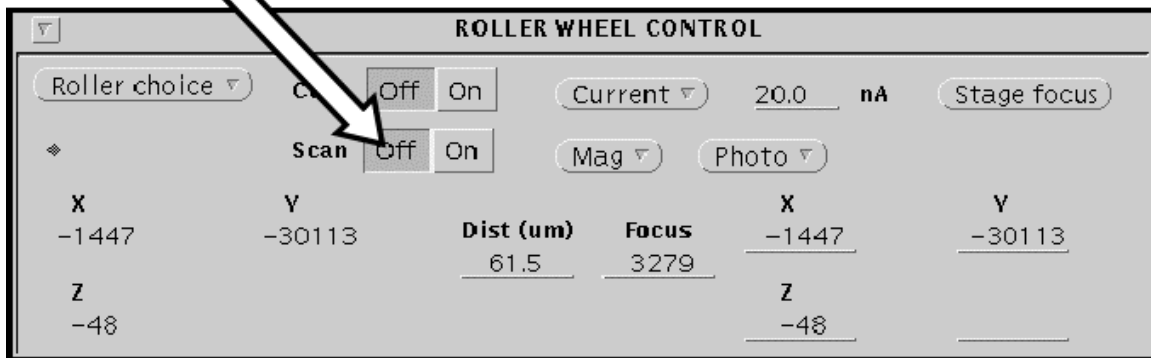
**Press This
Button**



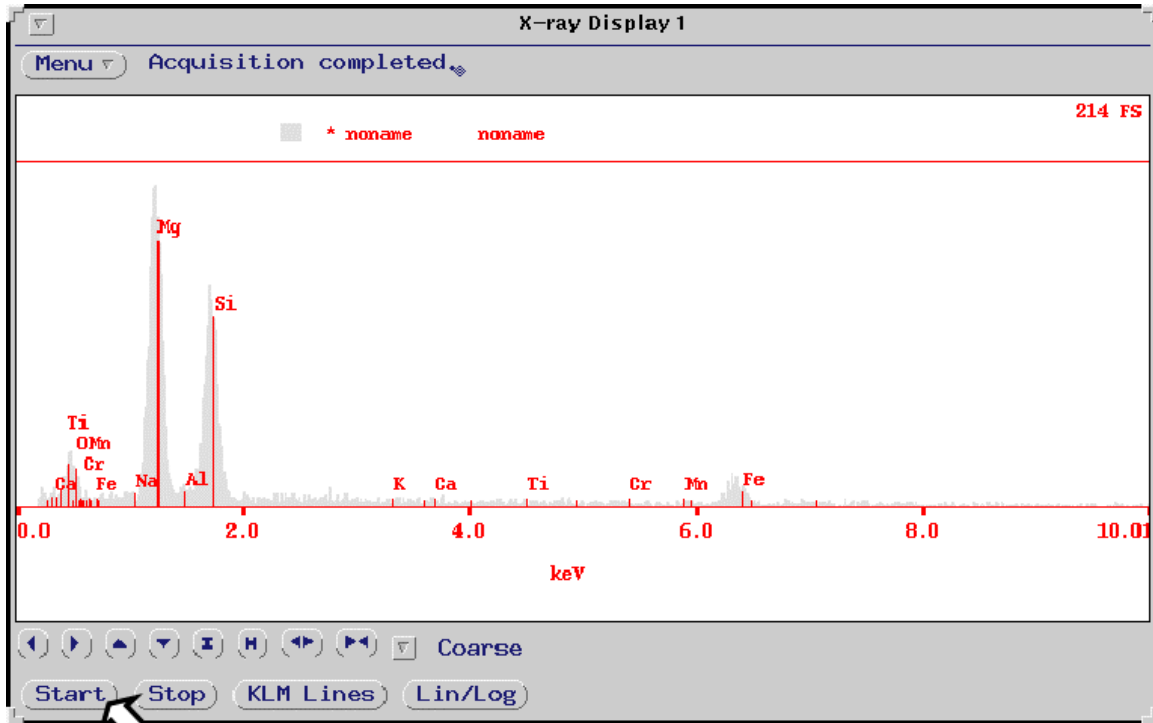
Step 6

Turn Scan Off.

**Press This
Button**



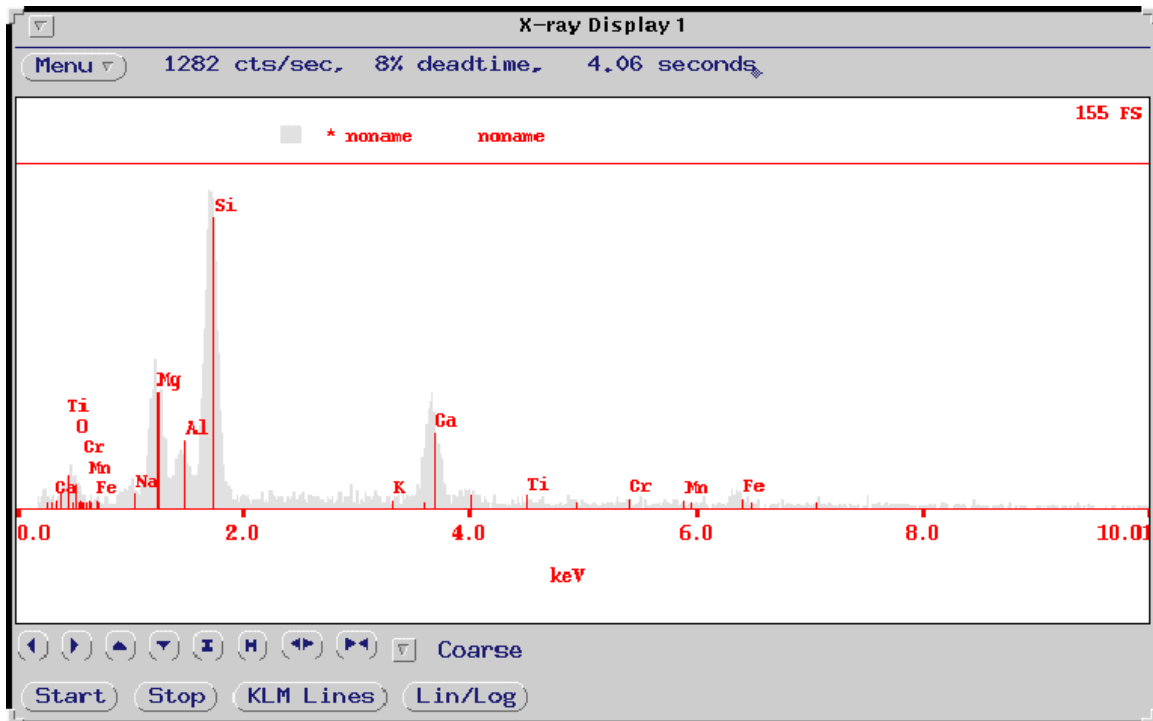
Step 7



**Press This
Button**

Step 8

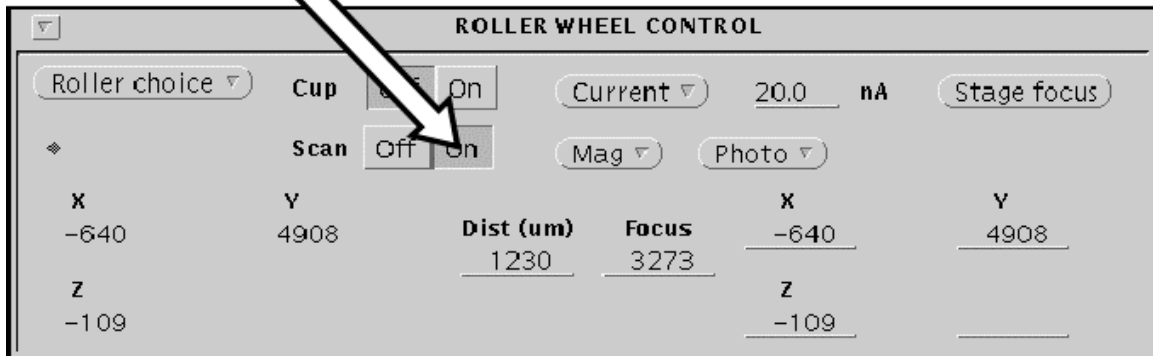
The EDS will accumulate an X-ray spectra.



Step 9

When the EDS spectrum is finished, turn the Scan on.

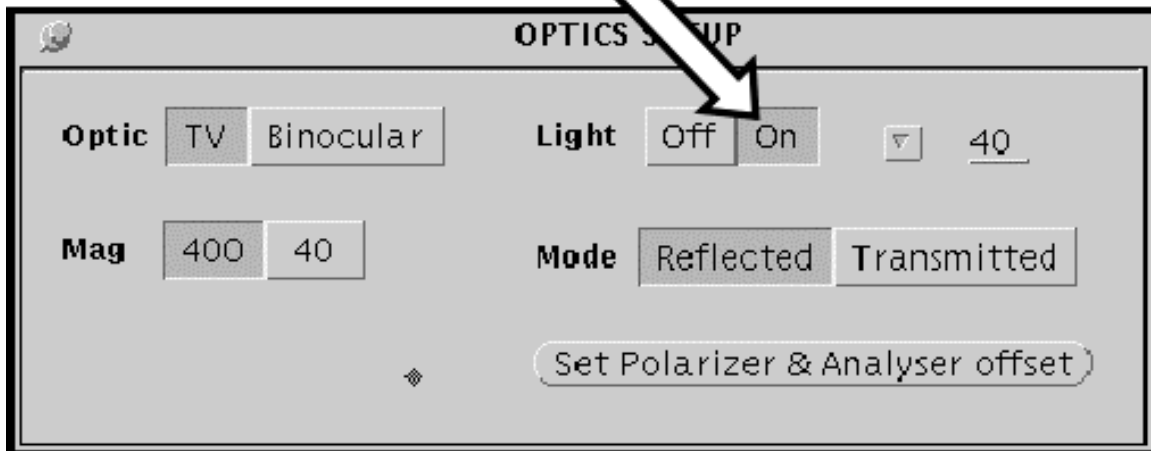
**Press This
Button**



Step 10

Turn the light on.

**Press This
Button**



Step 11

REPEAT STEPS 5 – 10 for each EDS analysis point you want to analyze.

Appendix 1

Analysis Screen Disappears after pressing Continue

If analysis screen disappears after you press the continue button for the final time you need to create another Define file that has a different name but the same elements and beam conditions to use for doing additional analyses.

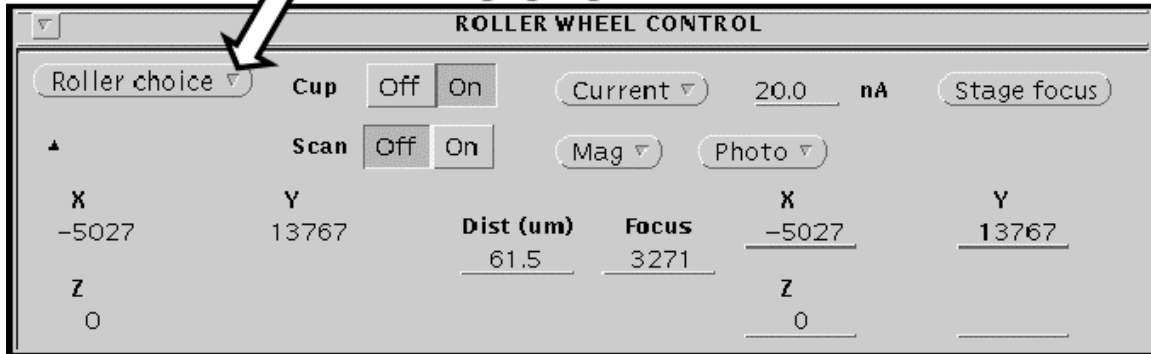
To do this follow steps 1 and 2 beginning on page 65 of the Calibration Instruction Set (Define an Analysis File: Instruction Set 4b Update an existing file). In Step 2, use the file name you were previously using. Modify this name (e.g. add a number after it), and skip to Step 7 and continue following the instructions to the end. After saving the new file, start with Step 1 of this instruction set (Analysis Instruction Set 2a). Be sure to use the new file name in Step 3

Appendix 2

Reset the Probe Marker

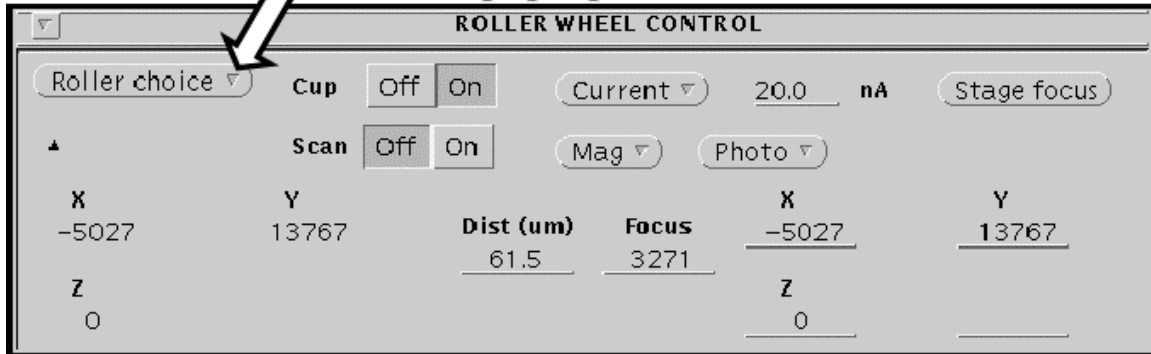
Step 1

R-Click this button and select Probe Marker from the pop-up menu.



Step 2

R-Click this button and select Stage from the pop-up menu.



Appendix 3

Adjust BSE Image Brightness and Contrast

1: Using Automatic Mode: (easier)

Step 1

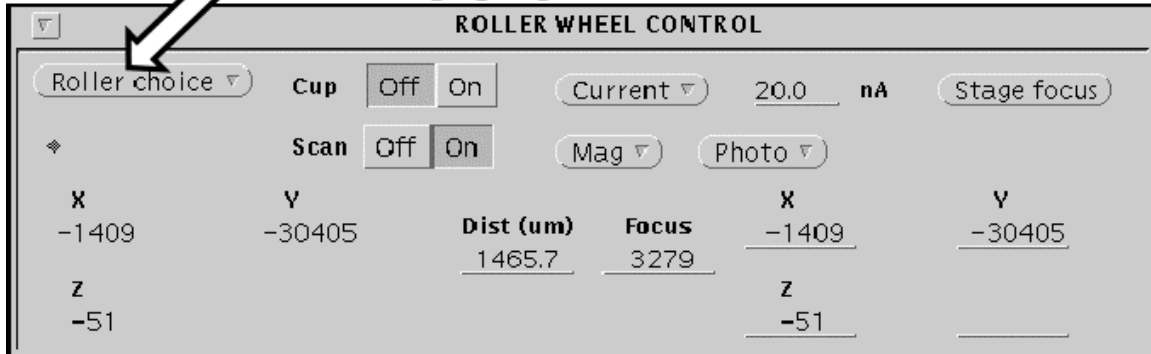
R-Click this Button and Select Video Mode 1 from the pop-up menu.

The screenshot shows the 'ROLLER WHEEL CONTROL' software interface. A white arrow points to the 'Roller choice' dropdown menu. The interface includes several control buttons and a data table.

X	Y	Dist (um)	Focus	X	Y
-1409	-30405	1465.7	3279	-1409	-30405
Z				Z	
-51				-51	

Step 2

R-Click this Button and Select Video Mode 1 from the pop-up menu.



Step 3

When you are finished adjusting the contrast:

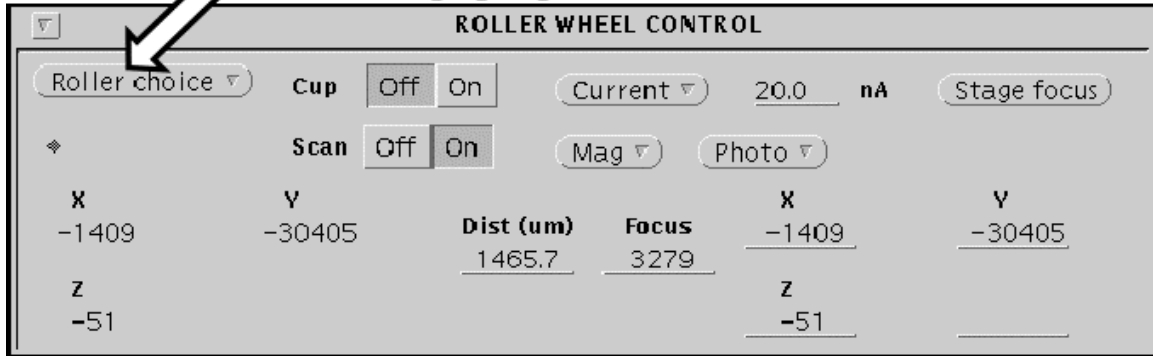
R-Click this button and select Stage from the pop-up menu.



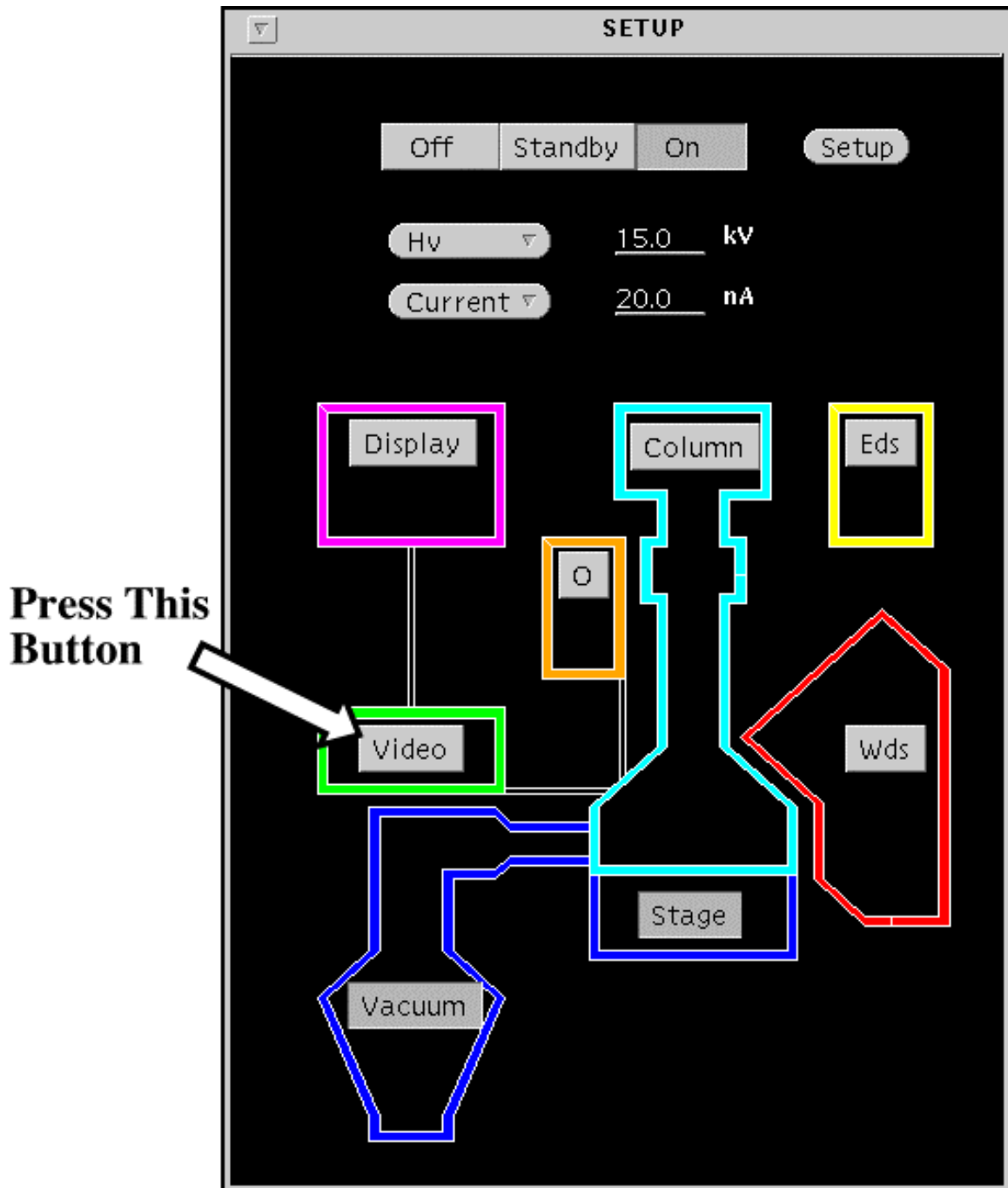
2: Using Manual Mode: (more control)

Step 1

R-Click this Button and Select Video Mode 1 from the pop-up menu.

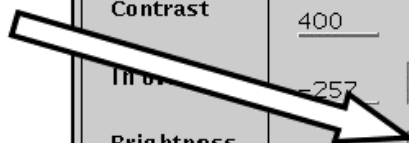


Step 2



Step 3

Press This Button



SIGNAL SETUP		
	VIDEO 1	VIDEO 2
Signal	<input type="button" value="SE"/> <input type="button" value="BSE"/> <input type="button" value="ABS"/> <input type="button" value="AUX"/>	<input type="button" value="SE"/> <input type="button" value="BSE"/> <input type="button" value="ABS"/> <input type="button" value="AUX"/>
Contrast	Contrast <input type="button" value="v"/> Atomic	
	<input type="text" value="400"/>	<input type="text" value="240"/>
Through	<input type="text" value="257"/> <input type="button" value="manu"/> <input type="button" value="auto"/>	
Brightness	<input type="text" value="31"/> <input type="button" value="manu"/> <input type="button" value="auto"/>	<input type="text" value="512"/> <input type="button" value="manu"/> <input type="button" value="auto"/>
Gamma	<input type="text" value="1"/>	<input type="text" value="1"/>
	▲	
Gain	<input type="button" value="low"/> <input type="button" value="high"/>	
Coupling	<input type="button" value="ac"/> <input type="button" value="dc"/>	
Polar	<input type="button" value="ground"/> <input type="button" value="bias"/>	
		Plates <input type="button" value="off"/> <input type="button" value="1"/> <input type="button" value="2"/> <input type="button" value="3"/> <input type="button" value="4"/>
		Detector <input type="button" value="off"/> <input type="button" value="on"/>

Step 4

Adjust Roller Wheel controls to obtain a good picture.

ROLLER WHEEL CONTROL						
Roller choice ▾	Cup	Off	On	Current ▾	20.0 nA	Stage focus
◆	Scan	Off	On	Mag ▾	Photo ▾	
VS1 offs	VS1 brigh			X		Y
-322	41	Dist (um)	Focus	13956		15363
		61.5	3279			
VS1 gamma	VS1 cont			Z		
1	412			-3		

VS1 Offset: Large changes in image brightness.

VS1 Brightness: Small changes in image brightness.

VS1 Gamma: Contrast for very bright objects (seldom used).

VS1 Contrast: Contrast adjustment.

Step 5

When you are finished with the picture:

Press This Button

The image shows a 'SIGNAL SETUP' menu with two columns for 'VIDEO 1' and 'VIDEO 2'. The 'VIDEO 1' column has settings for Signal (SE, BSE, ABS, AUX), Contrast (Atomic), Contrast value (296), In offset (155), Brightness (36), and Gamma (1). The 'VIDEO 2' column has settings for Signal (SE, BSE, ABS, AUX), Contrast (240), In offset (512), and Gamma (1). Below these are shared settings: Gain (low, high), Coupling (ac, dc), Polar (ground, bias), Plates (off, 1, 2, 3, 4), and Detector (off, on). An arrow points to the 'manu' button for VIDEO 1 Brightness.

	VIDEO 1	VIDEO 2
Signal	SE BSE ABS AUX	SE BSE ABS AUX
Contrast	Atomic	
Contrast	296	240
In offset	155	
Brightness	36	512
Gamma	1	1
Gain	low high	
Coupling	ac dc	
Polar	ground bias	
Plates		off 1 2 3 4
Detector		off on

Step 6

R-Click this button and select Stage from the pop-up menu.

