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ALIGNMENT PROCEDURE

The alignment procedure involves (5) steps. It is Important that these steps are carried out in the following order.

A. X-ray Controller must be set up with Spot Size and Power Setting correct for ALL spot sizes.

B. The crystal must be adjusted to place the x-ray spot on the sample at the center of the field of view of the lens.

C. Lens focus adjustment must track B.E. so lens is focused to "best focus" for all B.E. Values. This involves establishing slope and intercept for the relation between (Vr) Retardation Voltage and (V1) Focus Voltage.

D. DAC Constant for the Retardation Supply must be calibrated.

E. Detection Width Constant and Pass Energy calibration must be performed to obtain accurate Spectra.

Some of these adjustments are difficult and usually require someone who has had training in person. It is hard to describe some of the observations that need to be made. It is usually easier to demonstrate the desired results.

A. X-RAY GUN CONTROLLER ALIGNMENT

1. We recommend the use of a P-31 Phosphor to visualize the X-Ray spot. A useful target can be assembled from the following.

    a. Piece of silicon wafer approximately 1.5 cm square.
    b. Piece of tape, approximately the same size as the silicon, with sticky on both sides. 3M, Part Number 665, is a linerless tape that works well in the vacuum. Place tape on polished side of silicon wafer.
    c. Prepare a square of 200 lpi, chem-etched, screen. Place screen on the tape. Gently pass the back of a razor blade over the screen to attach the screen evenly to the tape. DO NOT cause the screen to get buried.
    d. Sprinkle a small amount of P-31 powder over the tape-screen sandwitch. Spread evenly with razor blade. Tape off excess P-31 powder.

This target provides an easy way to measure spot sizes. The grid spacing is 127 microns per grid.

2. **During spot power and size adjustment, be aware of three (3) very important issues.**

    a. If a spot is too small for the power setting the anode can be damaged. The vacuum system will be flooded with water. The water will do extensive damage to the electron gun, lens, detector, flood gun and pumping system. This is critical for the three larger spots. The smallest spot can not be made small enough to do damage if the power is set at 15 watts (1.5 mA).
        i. To avoid damage on the three larger spots you must look at the spot size immediately after a change of the Power or Focus setting. If the spot appears too small immediately turn the "Manual Spot Control" switch to OFF and then turn the spot focus pot two turns counter-clockwise. Turn the "Manual Spot Control" switch back to the size you are adjusting and again look at the spot size. Repeat if necessary.
    b. If the phosphor is out of focus the spot will look bigger than it is at the anode.
        i. The apparent size of the spot on the phosphor target is an X-Ray image, formed by the crystal, of the anode. The Vf focus voltage adjust the size of the electron beam that creates the X-Rays at the anode. The image of this spot may be out of focus if the distance from the crystal to the phosphor target is incorrect.

ii. **Before starting Spot Size and Power adjustments make the following test.** Use the smallest spot. Adjust the power to 15 Watts (1.5 mA). Bring the phosphor target into optical focus of the microscope. This is done by adjusting the height of the sample with the Z adjustment of the XYZR stage. Center the phosphor target in the field of view of the microscope using the XY stage adjustments. Use the non-Bragg adjustments on the crystal gimbals to move the X-Ray spot in the left-right direction on the phosphor. Move the spot in both directions until you find the position that produces the smallest spot. Now adjust the Vf voltage for the smallest spot to see if it can be made smaller. Recheck the non-Bragg adjustment.

iii. Leave the stage Z axis adjustment and the Crystal non-Bragg adjustment in this position for the Spot Size and Power adjustments.

c. After setting the Power to the correct values ALWAYS be sure you have made the spot too big by turning the Focus Pot counter-clockwise. Starting with the spot too large, make ½ turn clockwise adjustments checking that the spot becomes smaller until the correct spot size is reached. If turning the spot size pot clockwise makes the spot fuzzy or larger, you have gone past the correct spot condition. You must turn the spot size switch to off and turn the focus pot 3-4 turns counter-clockwise. Check the spot and repeat until the spot becomes large and well defined.

FOR 8702 X-RAY CONTROLLERS

The power for the largest spot is set by adjusting the I2KV pot. The Pierce Voltage for the larges spot is always set to 110 +/-10 Volts with the Glassman OFF.

The power for the three smaller spots is set by adjusting the Pierce Voltages. The I2KV is not changed from the value used for the larges spot.

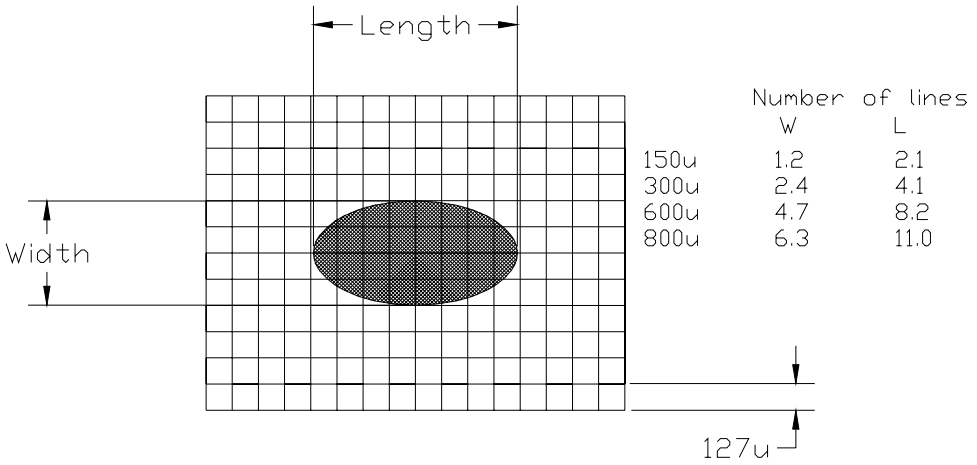
The settings for the spots and lines are listed below.

FOR 9600 SERIES X-RAY GUN CONTROLLERS

The power settings are set at the factory and do not need adjustments. The I2KV automatically adjusts to keep the correct power setting. The V Pierce is at about 750 Volts for “Spot Off” and about 100 Volts for all spot sizes.

SETTINGS FOR SPOTS AND LINES

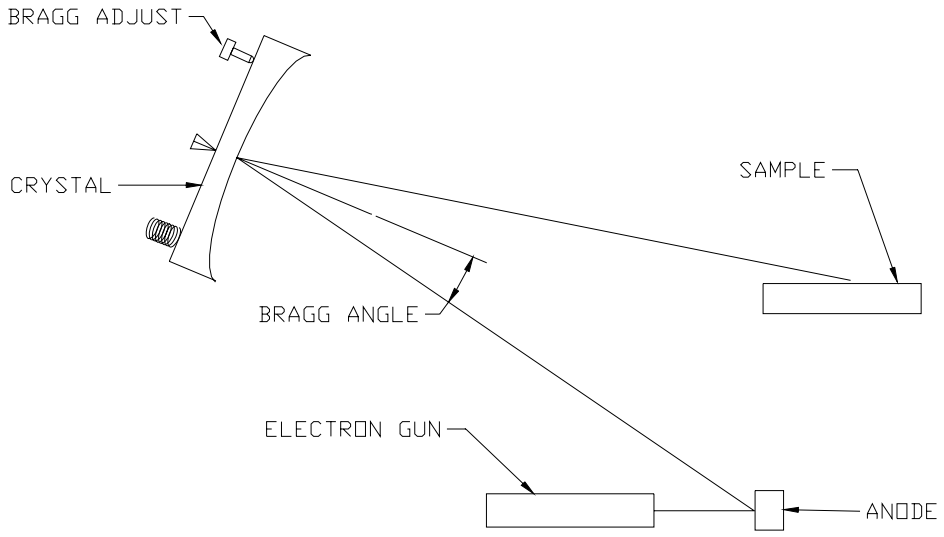
SPOT #	SPOT SIZE		POWER	GLASSMAN	
	Min Safe	Typical		(8702)	(9600)
1	100	150	15W	4 ma	1.5
2	250	300	50W	7 ma	5
3	500	600	100W	12 ma	10
4	800	900-1000	200W	22 ma	20
L1	100x700	150x700	50W	7 ma	5
L2	250x1000	300x1200	100W	12 ma	10
L3	450x1400	500x1400	200W	22 ma	20



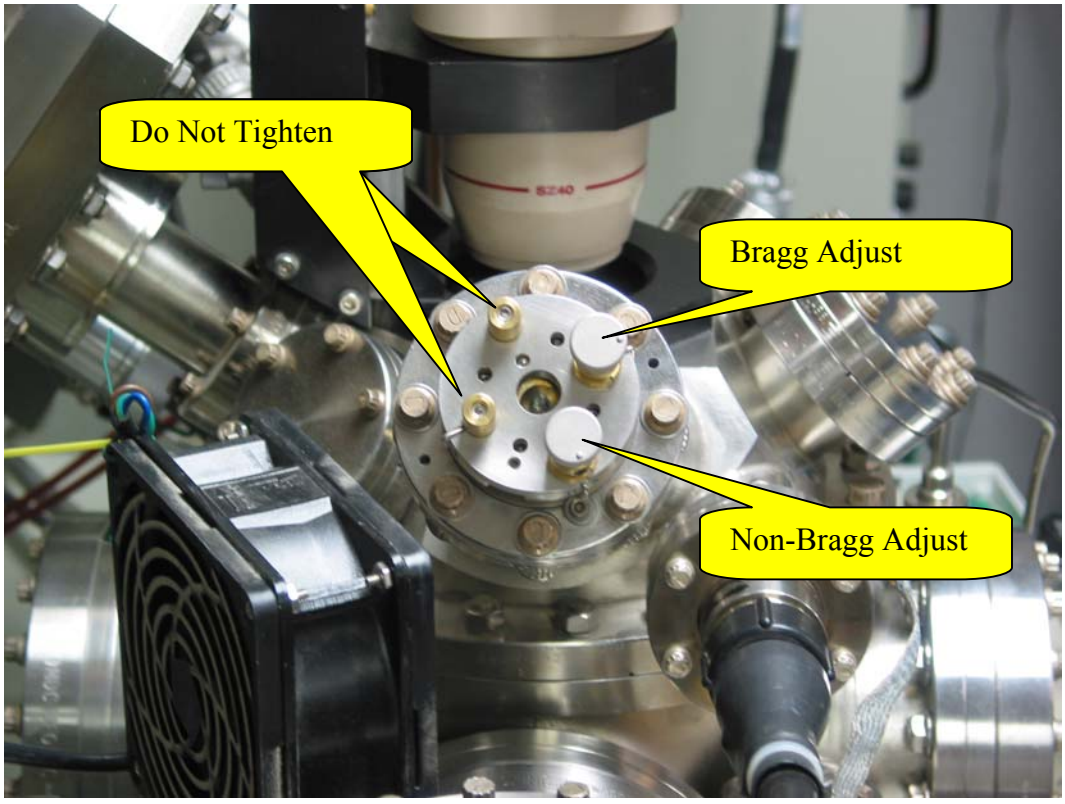
Using Fluorescent Screen
with 200 lines/inch grid

B. CRYSTAL ALIGNMENT

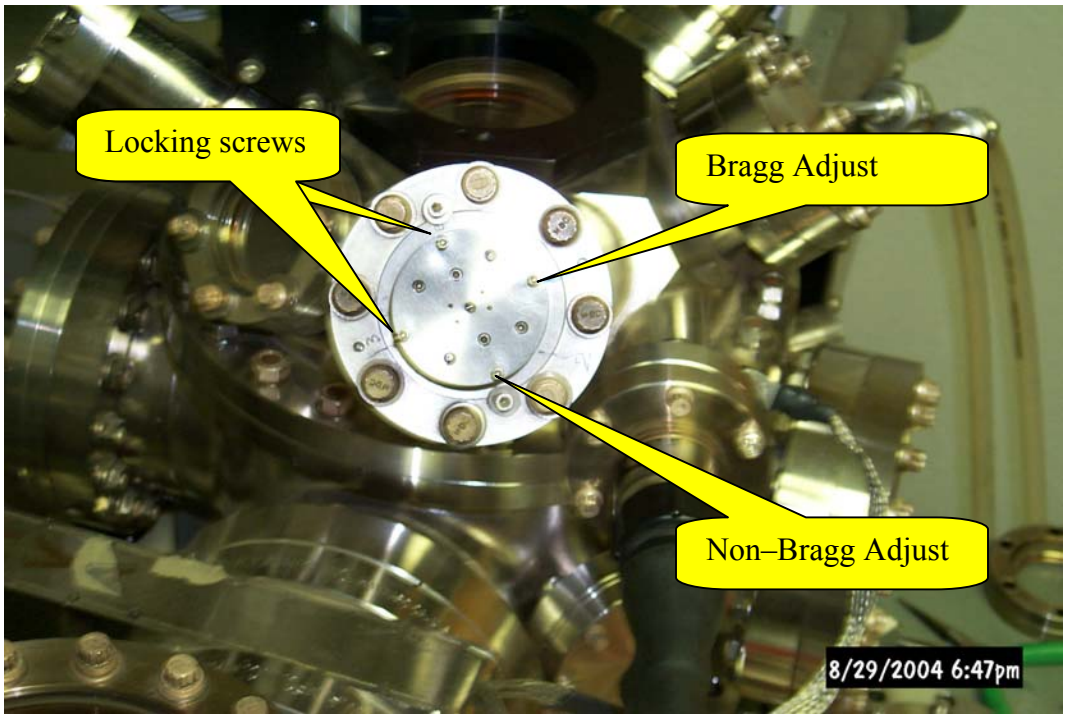
The Goal of the crystal alignment is to image the X-Rays produced at the Anode to the Sample. The Plane containing the point of X-Ray Production, the center of the Monochromator Crystal and the point of intersection of the X-Rays with the sample surface is call the Bragg Plane. The angle between the line from the point of X-Ray production and the center of the crystal and the line perpendicular the to surface of the crystal at the center is the Bragg angle. This angle must be adjusted to maximize the intensity of the reflected X-Rays. The crystal normal and the line from the center of the crystal to the focal point of the reflected X-Rays form an angle that is equal to the Bragg angle.



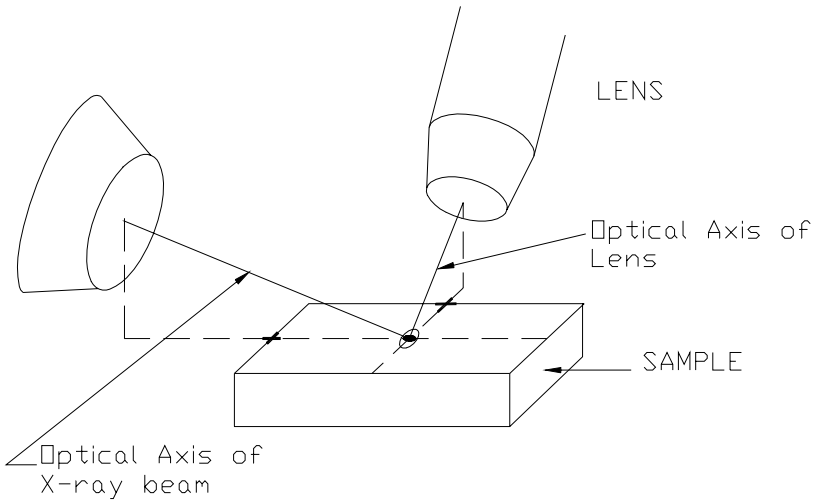
The Bragg angle is adjusted using the “Bragg” adjustment on the crystal gimbals.



New crystal gimbals



Old crystal gimbals



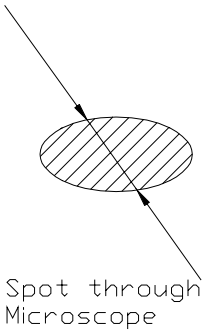
Steps:

1. Obtain a uniform, flat gold sample approximately 1 cm x 1 cm square. Sputter deposited gold on a silicon wafer is good. Metal foils are not flat and cause problems.

2. Place the gold and fluorescent screen side by side on an electrically isolated sample holder. Arrange to monitor photo current from the gold with an electrometer.

3. Turn x-rays on. Set x-rays to 600u (Spot #3). Put spot on fluorescent screen. Make Bragg adjustment on the crystal to obtain brightest spot. Be sure edges have the same brightness.

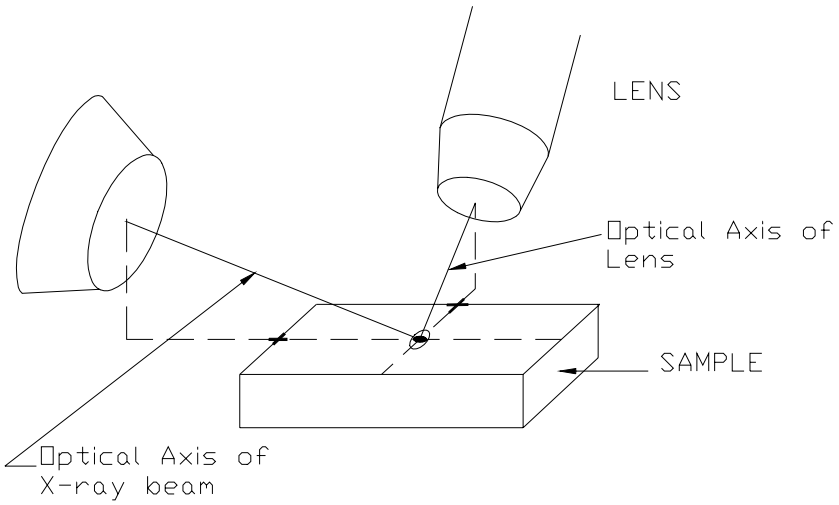
   Spot will lose intensity at arrow heads due to Bragg reflection loss. Two points marked need to be same brightness.



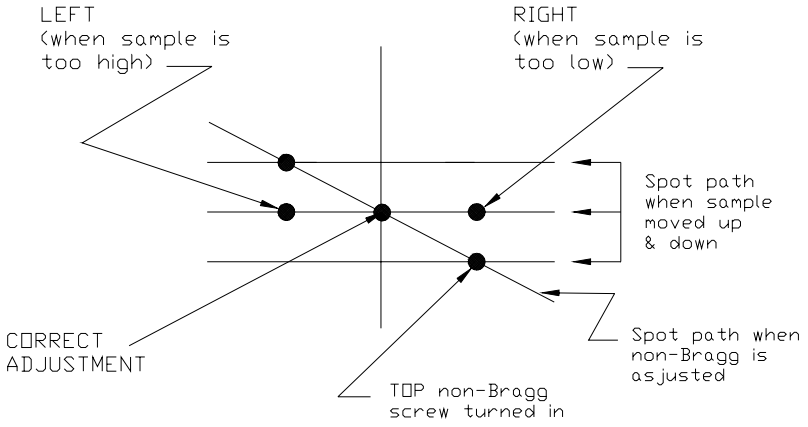
NOTE FOR OLD STYLE GIMBALS: **NEVER TIGHTEN SCREWS WHEN THE OPPOSING SCREW IS IN CONTACT WITH THE TILT PLATE. A SMALL SPRING IS USED TO PROVIDE TENSION. THE SCREW IN THE LOCATION OF THE SPRING IS USED TO LOCK THE ADJUSTMENT. THE LOCATION WITHOUT THE SPRING IS THE ADJUSTMENT. THE ADJUSTMENT PUSHDS AGAINST THE OPPOSITE SPRING. NEVER LOCK WITH HIGH FORCE. USE VERY LIGHT "TOUCH" TO LOCK.**

4. Move to gold. Made fine adjustment of Bragg to maximize electrometer reading. Record the electrometer reading in note book.

5. The brightest spot may not be where the lens is "looking". The sample height and the non-Bragg screws are used to move the spot to the center of the lens field of view.



Isometric view of problem



Problem as viewed looking through microscope

The most accurate alignment is obtained with the second lens turned off. This requires a special "TEST" printed circuit board. The spectrometer supply is turned off. The top cover is moved back about 10 cm. The cable connector that attaches to the top of the first board is removed. The "TEST" board is connected to the top of the first board. Then the connector is attached to the "TEST" board. The "TEST" board is then configured to turn off the second lens. Turn on spectrometer supply.

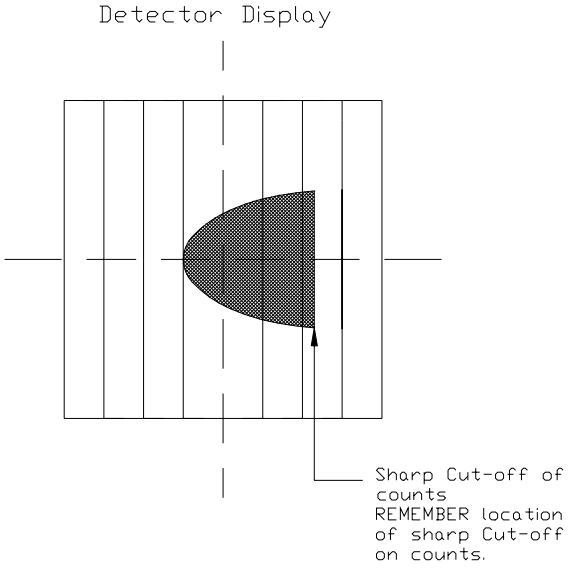
WARNING: DANGEROUS HIGH VOLTAGE IS PRESENT ON THESE BOARDS AND CABLES.

If this board is not available, proceed without it.

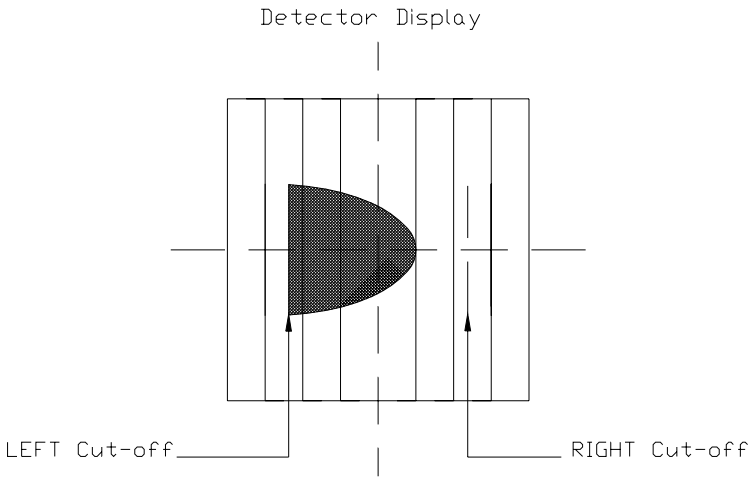
a. Set the computer to acquire an unscanned spectrum. Res 3, Spot size 600u, 999 seconds.

b. Adjust the sample height for maximum counts using the counter on the computer or an external counter.

c. For NEW STYLE GIMBALS just use the Brag Micro adjusting knob. For the OLD STYLE Gimbals loosen both non-Bragg screws. Tip non-Bragg until counts drop by at least 30%. Move sample height to get maximum counts. Look at the detector display. Repeat moving the spot **OUT** of adjustment until detector display looks as follows:



d. Now turn non-Bragg adjustment screw in opposite direction and move stage in opposite direction to obtain cut-off on other side of detector display.

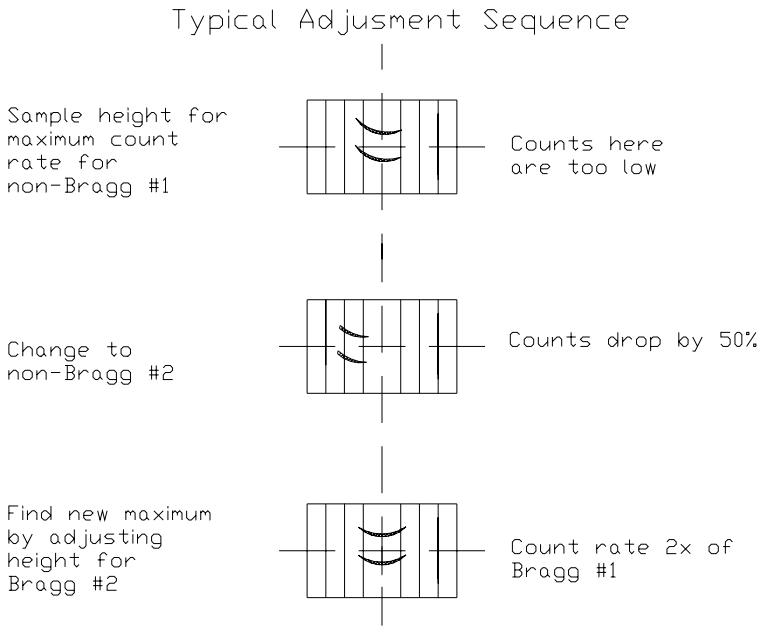


e. Use <> control on scope to place the left and right cut-off equal distance from the center. Repeat to check. The centering of the scope is very important.

f. Check that electrometer reading has not changed. Readjust Bragg to reestablish the maximum photo current if necessary. Continue to maintain the maximum photo current throughout the rest of this procedure. Start making small adjustments (0.1 turn) of the non-Bragg adjustment screw. After changing the screw, move sample height to obtain maximum counts. Measure carefully and make table of values. Keep change adjustment screw, going in one direction, and then obtain maximum counts moving sample height.

g. You will determine the adjustment direction that results in each maximum being higher than the last maximum.

h. As you precede the detector pattern will move toward the center after you find the best height for the sample. It is usually true (but not always) that the non-Bragg adjustment moves the pattern away from the center and sample height then moves it back to the center past the last position. Typical adjustment sequence:



Spend considerable time to find the highest possible count rate. The dot pattern must be well centered when finished. Carefully make final adjustment of Bragg to maximize photo current then recheck non-Bragg.

Finally, if you are using the old style gimbals, very lightly tighten the set screw. Be sure not to let the count rate drop or the dot pattern move. With the new Gimbals there is no need to secure or lock the final adjustment.

i. To verify the adjustment is okay, carefully adjust sample height to obtain maximum counts. The dot pattern should be exactly centered and appear symmetric from side to side. If you can not obtain this there maybe a magnetic problems in the chamber or the lens may be damaged. You will need to describe to us any change from the ideal condition you find.

C. V1 ADJUSTMENT

1. If you have 8701B or 8724, the software will provide a V1 Curve Calibration. Please follow software instructions. With the 8701 Spectrometer power supply there is no valid V1 adjustment.

D. THE CALIBRATION OF THE DAC CONSTANT

1. The calibration of the DAC, Detector Width and Pass Energy is covered in the software manual. Please refer to these instructions.

2. Finally you should make a table of the Spectrometer Power Supply settings for future use. The table below can be used for this data.

    a. Also included is a Table for you to fill out. To fill this out pull the spectrometer supply out. Slide the top cover back about 10 cm. In the center front a cable connector is attached to the first card. Along the top are labels:

V+, V-, Img, Gnd, Vtrim, V1, Vq, V0, V2, DET

Set the computer to record an unscanned spectrum with lower B.E. at 82eV for HP work station software. For PC Vectra, ESCAVB and ESCA 2000 software the center B.E. is set at 84eV. Collect for Res 4. Record all voltages in the Table with a 1000:1 high voltage probe. Make measurements for Res 3, Res 2 and Res 1. Read the magnet current with the front panel meter. Fax us the completed Table.