MProbe: X-Ray Gun Degassing, Spot Size Adjustment, Alignment and Calibration

Revised 07 2019

BSB

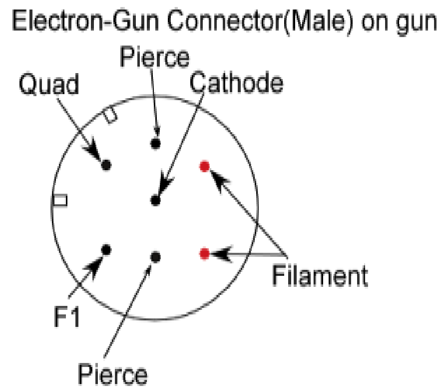
Gun replacement:

A. The gun has a cutout at the bottom end that should be orientated at about 11:00 o'clock. A both hole will than be at the 12:00 o'clock position.

B. The cutouts in the electrical connector to the gun should be at about 9 and 11 o'clock.

C. Restore system vacuum. After analyzer pressure is in the 10^{-8} T range, the gun start-up and alignment can be performed.

D. Reconnect gun connector.



Degassing the -Gun

Figure 1 9600 X-ray gun power supply and controller

1. Turn **on** the 9600 X-Ray gun controller, Fig 1. The Interlock OK LED should come on. If not insure that all necessary interlock conditions are met as indicated on the manual vacuum controller front panel. Turn **on** the Glassman High Voltage power supply, and press the **HV ON** button. The 2KV supply is referenced to the output of the Glassman so it must on even if its output voltage is at zero.
   a. Set the front panel toggle switch labeled "**SERVICE**" to the **Down** (slow)position
   b. If the system has been up to atmosphere, rotate the INCREASE TIME potentiometer fully **clockwise** (8 h ramp).
   c. If the system has been at pressures of <2 x 10^{-6} Torr, rotate the Time potentiometer to fully **counter clockwise** (2-3 h ramp).

Figure 2 X-ray gun power supply with cover open and spot size control box sitting on top

d. Pull the box about 6 inches out of the rack and slide the cover back, Fig 2. The red LEDs on the vertical board mounted to the inner bulkhead indicate the status of the ramps, Fig 3. LEDs 5 & 6, 4 & 7, and 3 & 8 indicate the ramp status of the filament, the 2KV, and the 10 KV, respectively. The top lights indicate that the function is enabled. The lower LED indicates the control loop is regulating I_{Fil}, I_{2KV} or $V_{10\ KV}$.

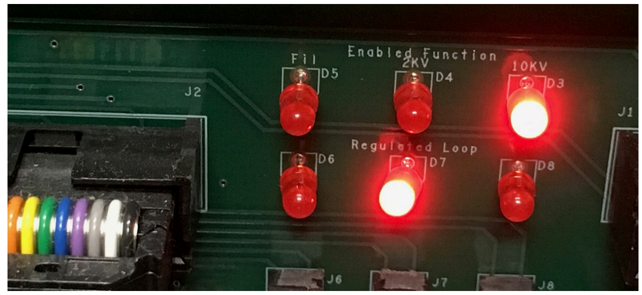


Figure 3 LED indicator licks for I_{fill}, I_{2KV}, I_{10KV}

Filament Ramp

e. Press the **Start Filament** on the 9600. The Filament On LED 5 & 6 should turn on. If the Sample Transfer is homed and gate #5 is closed then the Xfer OK LED should be on. On the inside board o LEDs 5 & 6 should be on.
f. The front panel meter defaults to I FIL and should read ~0.65Amps.
g. The supply will ramp the filament to 1.38 A. At the end of this period the ramp will switch to I_{2KV}, LED 4 on.
h. *I_{2KV} Ramp:* The 2KV will increase over 30 seconds to 2.3 KV. This can be monitored on the front panel meter. At ~1.9 KV the 2KV OK light on the front panel will turn on. The LED 7 in the second column will come on.
i. *Stand-By:* When the I_{2KV} ramp completes the auto start process will halt in Stand-By. The I_{2KV} will be at ~6 mA. You can return to Stand-by from the operate mode at any time.

2. When the vacuum has stabilized at <4 x 10^{-8} Torr, the high voltage ramp may be started. Be sure the Hawk circulator is operational.
   a. Set the "**SERVICE**" switch to **UP.** Select **OPERATE** on the 9600 X-Ray Gun controller front panel.
   b. Select the **100-micron Spot** size.
   c. The Hawk circulator will turn on. The Hawk circulator On LED will indicate this. The Flow OK LED will come on when the flow stabilizes.
   d. The Glassman voltage will jump to about 2.5 KV. The Glassman voltage will slowly ramp to 10 KV.
   e. If the gun has been in service and is well conditioned
      i. switch the service switch down and let the 10KV ramp quickly to about 5 KV. Then go back to service and check all meter settings.
      ii. The values in the table at right are typical at this point.

	Value	6/2019
I_{out}, mA	0.5 – 2.5	0.09
I_{fil}, mA	1.2±.05	1.1
V_{2KV}, KV	2.3	2.3
I_{2kV}, ,A	5.8 – 6.5	5.07
V_{Focus}. KV	3.8 – 4.2	8.44
V_{Quad}	0	0.02

iii. Next go to about 7 KV then return to Service.
iv. Hold for about 5 minutes. Then go to about 8KV.
v. Let it ramp in Service mode the rest of the way the first time.

f. When the Glassman voltage reaches about 9.2KV the 10 KV OK LED and the Spot On LED's will turn on. This indicates that the supply is now actively controlling the gun output current. The I_{Out} should read 1.5 mA

Preliminary Checks after 10 KV is attained

Range of typical values for the 9600 X-Ray Gun parameters									
	OFF		100 15W	250 (300) 50W	500 100 W	800 200 W	L1 50 W	L2 100 W	L3 200 W
I_{OUT}	0.05- 0.25		1.45(5)	5.0(1)	10.0(1)	20.0(5)	5.0(1)	9.7(7)	20.0(5)
I_{Fil}	1.2(5)		1.2(5)	1.2(5)	1.2(5)	1.2(5)			
V_{2KV}	2.30(2)		2.30(2)	2.30(2)	2.30(2)	2.30(2)			
I_{2KV}	5.8(4)		5.9(4)	6.5(3)	7.3(5)	8.1(6)			
V_Q	0		0	0	0	0	175(25)	240(40)	400(50)
V_F	8.2(1)		8.2(1)	8.25(.1)	8.3(.1)	8.4(.1)			

Table 1 X-Ray Gun Parameters

Notes

1. The "250" µm spot size is really a 300 µm spot (it is mismarked on the X-ray controller) and is referred to as 300, 250, or 200.
2. The front meter can read the major operating parameters. During start up these parameters help provide an indication of how well the gun is cleaning up. After the Glassman reaches 10KV all meter reading should be taken in both the Spot OFF and 100 Micron settings.
3. Compare the readings to the table above. If any reading is out of range do not select any other

Table 2 Spot Size Setting

Spot Size Settings

Spot	Size mm	Appears µm	Lines (360 µm grid)
100	100	100×174	0.3 x .5
250	300	300×522	0.8 × 1.4
500	500	500×870	1.4 × 2.4
800	800	800×1400	2.2 × 3.8
L1 (250)	100×400	174×400	0.5×1.1
L2 (500)	250×1000	435×1000	1.2 ×2.8
L3 (800)	500×1200	870×1200	2.4× 3.3

operating conditions. Let the gun operate in the 100 Micron Spot condition for an hour and see if the out of bounds reading are improving. If not contact Service Physics Inc for troubleshooting help.

Spot Size Adjustment

1. Note on measuring size.
    a. The gird lines on the phosphor are 363 μm apart.
    b. The spots will appear elliptical since they x-ray beam is at 35º to the surface (55º to the normal). The observed size in the horizontal distance (nonBragg) needs to be corrected by sin(35)=0.57. Thus, the observed spot needs to appear larger by 1/0.57 = 1.74, Table 2, Fig 4.
    c. If a spot is too small for the power setting the anode can be damaged resulting in catastrophic damage of the X-ray gun, vacuum system, etc. This is critical for the larger spots. The smallest spot cannot be made small enough to do damage if the power is set at 15 watts (1.5 mA).
    d. If the phosphor is out of focus (i.e. stage height not correct) the spot appears larger than it really is.

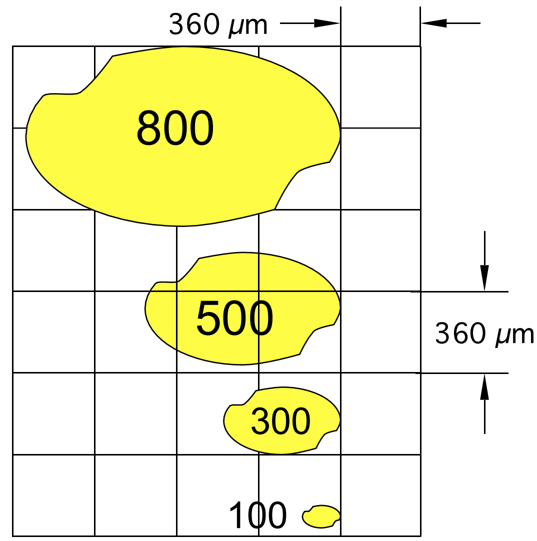


Figure 4 Spot sizes on Grid

    e. The power of each spot, $P(W) = 10000\ (V) \times I_{out}(A)$, is adjusted by the pots shown in Fig 6 below. When a spot size is selected an LED lights beside the pot to indicate which one to adjust to change I_{out}.
    f. The spots sizes are adjusted using the Hand Held Spot Size Control Box, Fig 5. There is one pot for each of the regular spots and two pots for each line spot. For the line spots one pot is used to adjust the width and the other pot the length, Fig 5. The LEDs indicate the pot to adjust for the spot in use.

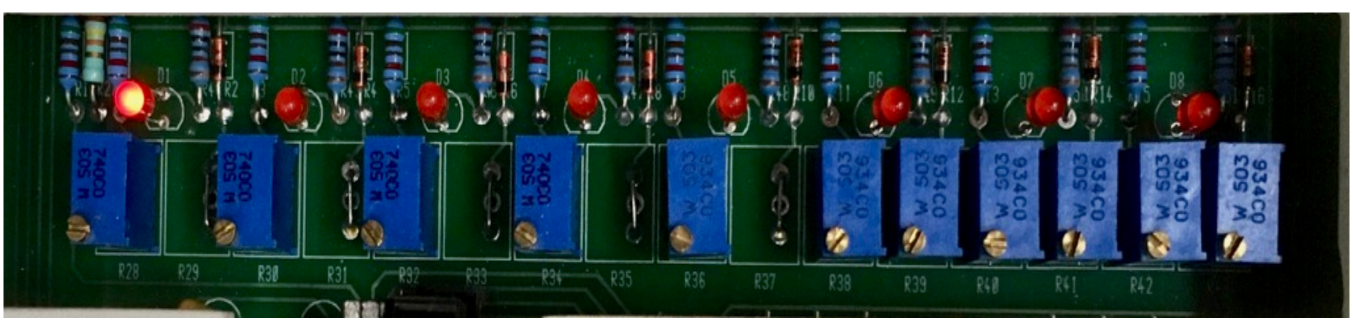


Figure 5 Spot size control pots for all ranges

2. If the 100 Micron Spot parameters are in range (Table above) and the vacuum is < 5 x 10^{-9} Torr then:
    a. Adjust the 100 Micron spot for a power to 15 Watts (1.5 mA I_{out})

b. Adjust the Z height of the stage to focus the spot.,
c. Center the spot on the phosphor using the x-y adjusts of the stage.
d. Use the non-Bragg angle adjustment knob on the crystal gimbal (Fig 6) to make the spot as small as possible. (This is to get the spot in focus.)
e. Adjust the spot size using the hand held spot size control box to be as small as possible. It should appear as ~100 x 174 µm

3. If the vacuum is < 5 x 10^{-9} select the 250 (300) Micron spot. Immediately check the spot size on the phosphor target. Adjust the spot to ensure that it is no smaller then 300x520 Microns. Check the gun parameters against the final test values shipped with the gun or Table 1 if document not available.
4. Let the vacuum return below 5 x 10^{-9}. Turn the R129 pot **10 turns clockwise** to reduce the power of the 500 µm spot to its minimum value.
5. Select the 500 Micron spot. Turn the pot ccw till you can see the spot and I_{out} is < 5 mA. It will take a few turns. Use the spot size control box to set the spot size. Increase the power of the 500-micron spot to 75 watts (I_{Out} = 7.5 mA) , wait 15 minutes to see how the pressure reacts. Turn the power up to full power (I_{out}=10 mA) check the pressure it needs to be < 2× 10^{-8}. Adjust the spot size. The voltage required to maintain the designated spot size increases as the power is increased.
6. For the 800-micron spot use an intermediate power of ~150 Watts (I_{out} =15 mA) (Pot R133) and set the size. Then go to full power of 200 watts.
7. If a Line/Spot gun is installed then the L1, L2 and L3 line may be selected. The L1, L2, and L3 lines have the same power as the 250 (300), 500 and 800 spots. The power density must be kept constant. A 500×500 µm spot has an area of 250,000 µm^2. Any line sizes with the same area may be used. A standard size of ~250×1000 µm is suggested. This can be changed for specific applications as long as the area is 250,000 µm^2 is maintained. The area for Ll is 90,000 and for L3 is 640,000. Practica1line dimensions are Ll ~ 100×700 and L3 ~ 500×1200. Note when you set the spot sizes you need to correct the horizontal measurement (first number) by the 35º angle of the source? First adjust the Quad pot (right pot) to the line length and then adjust the focus (left pot). You need to repeat each a few times. Be sure not to over focus the spot.

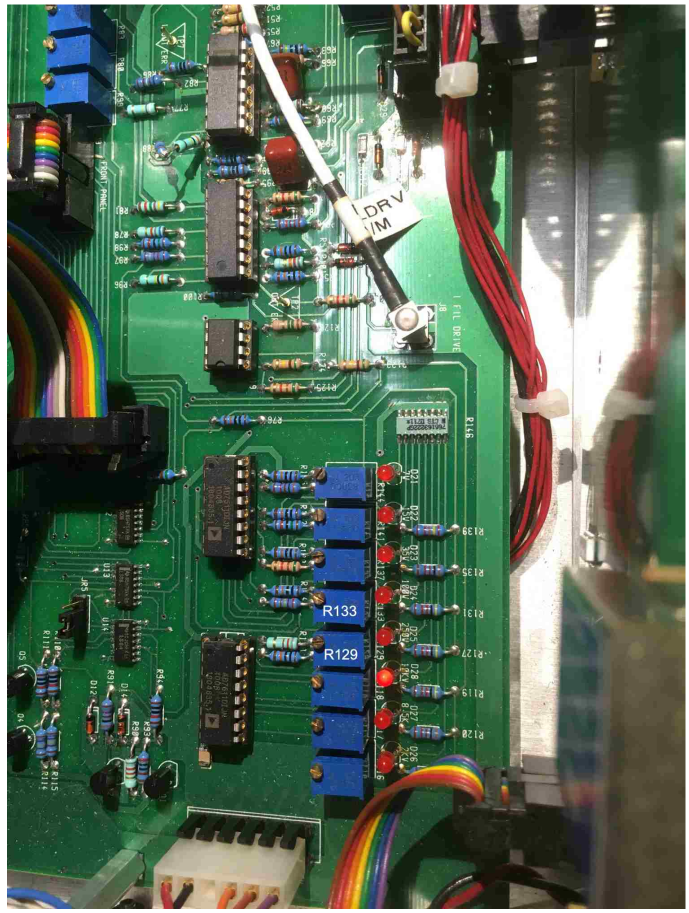


Figure 6 I_{out} control pots on board inside of 9600 power supply

8. Make a table of all the actual values shown in table 1 and put in the MProbe binder. The values should be monitored on a weekly basis.
9. After the gun is set up and has run for a day, return the Service/Operate toggle switch to the **down** or operate position. This will allow the supplies to ramp up to full power in about 3 to 4.5 minutes.
10. The controller can be set in the Computer Mode by pressing the front panel control. In this mode the computer can set the supply in Stand-by or in operate. The computer can also select any spot size and spot off.

Aligning the Monochromator and Microscopy.

1. Load a phosphor, Cu and Au sample into X-ray analyzer on the special stage that can measure the total sample current. **Make sure that the height of all the samples are the same.**
2. Place the Au sample into the x-ray beam position.
3. Choose a spot size of 500 µm and use the software to set up a scan for Au and stop it near the Au peak.
4. Adjust the Z motor control of the stage to maximize the counts for Au. Remove the cover of the controls for the monochromator crystal (black plastic cap to the left of the microscope). There are two controls for the monochromator the Bragg and nonBragg control. The Bragg control is at 8 and nonBragg is a 5 o'clock, Fig 7.

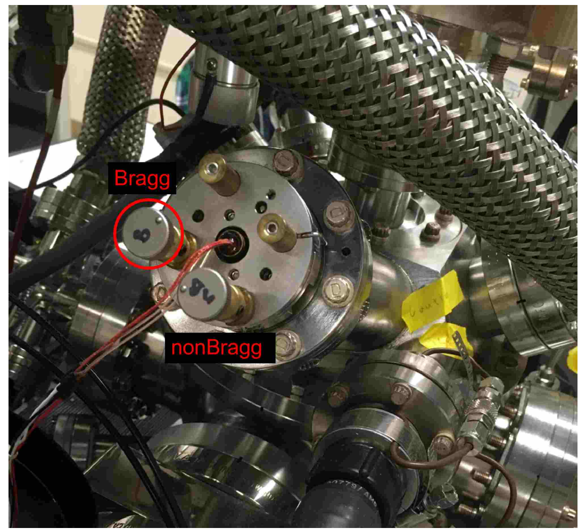


Figure 7 Quartz Monochromator adjustments. Bragg control marked B

5. Adjust the **Bragg** control to maximize the electrometer current or the count rate on Au. (best done with the electrometer)
6. Optimizing Z and nonBragg using the electrometer:
   a. Move the stage up and down in small steps (best to turn off the auto motor control and even computer control of the stepper motors and move the stage by hand) at each point maximizing the current. Using the **nonBragg** control
   b. After finding the position that yields the most counts tweak the **Bragg** control to optimize (this should be a very small adjustment).
   c. GO to step 8.
7. Optimizing Z and nonBragg with Au sample
   a. Maximize the count rate using the Z control of the stage (if you go to the motor control panel and turn off the computer control of the stepper motors, this is easier) . Write down the number of counts that you have.
   b. Move the **nonBragg** control in one direction to reduce the counts by ½.
   c. Adjust the Z control to bring back the counts.
   d. If the maximum in the counts is more that it was in step a above continue in the same direction and redo step 7 a-d If the maximum in the counts is less than it was in step E above adjust the **nonBragg** in the opposite direction. If it was more adjust the **nonBragg** again in the same direction.
   e. Continue doing steps a-e until no more improvement can be found.
   f. Adjust the **Bragg** to maximize the count rate.
   g. Redo steps a–f until both the Bragg and nonBragg are optimized.

8. Put the phosphor in the x-ray beam.
9. Maximize the counts by using the Z stepper motor control . Note that the Au may have a slightly different height then the phosphor.
10. Adjust the crosshair position so it is at the center of the spot.

Calibration of Detector

1. Load a Cu and Au sample into the XPS and focus on the Au sample. Note that the Au does not need to be sputter clean but the Cu will most certainly need sputtering. The Cu adjustment is less important and can be skipped most of the time.
2. Set the X-ray Controller to computer control and start the data collection program.
3. Go to: "settings: set up ESCA: Detector width".
4. Use 500 µm spot size.
5. Running the detector width calibration takes about ~15 minutes.
6. Go to V1 curves and run the procedure. If it completes, click calculation and then update register.
7. Put the Cu sample into the XPS and go to DAC
8. Run the procedure
9. Put the Au sample into the beam
10. Go back to the collection main program
11. Open performance test (top left)
12. Choose Gold diagonal
13. Run the test
14. For each curve go to peak and analyze it for area and peak width.

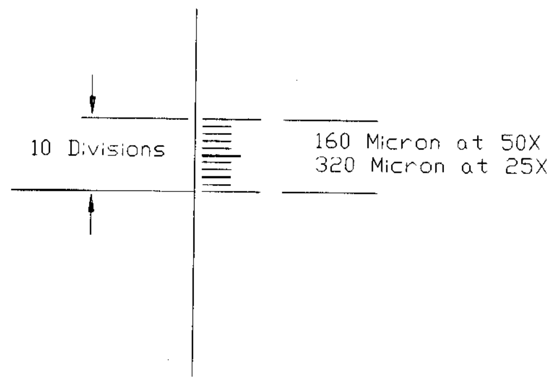


FIGURE 2

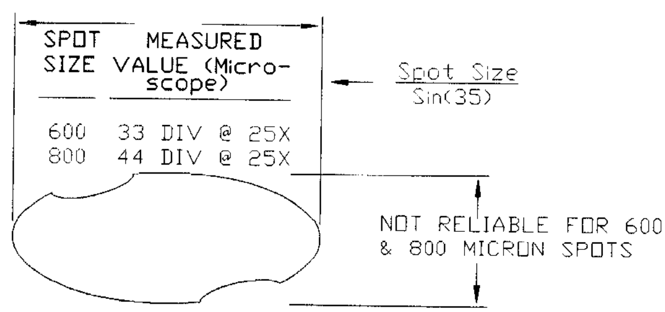
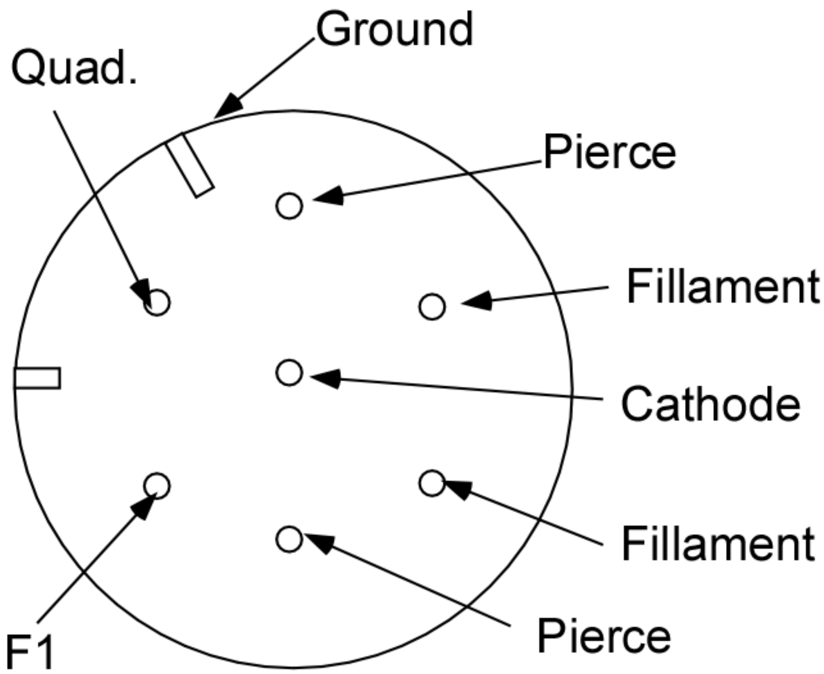


FIGURE 3

SERVICE PHYSICS, INC.



Resistance Between Pins

Pierce Ground P-G ________________Ω

Pierce Cathode P-C _______________Ω

Pierce Filament P-F _______________Ω

Cathode Filament C-F _____________Ω