Perkin-Elmer PHI 5100 XPS SOP

This SOP is intended as a Memory Aid Only, not as a training document. It is assumed you understand the principals involved in the operation and safe use of the instrument. If this is your personal copy of the SOP you can annotate these procedures as you see fit and are encouraged to do so.

By using this equipment you are accepting full responsibility for its safe operation and any damages and/or necessary repairs that occur as a result of your use of the equipment. "I didn't know." is not an excuse. If you don't know or have any doubts about what you are doing please stop and ask someone.

1. Mount the sample on a 25mm puck. All samples must be mounted on one of the 25mm pucks provided and cannot extend over the edge of the puck. The analysis area is a 10mm x 4mm region in the center of the puck and the long axis is parallel to the front edge of the counter top. An ideal sample size is 15mm x 10mm, allowing mechanical mounting with the two screws and washers on the puck without interfering with the analysis area. All samples are to be mounted mechanically; no paint of any kind is to be used.

2. Place the sample puck on the transfer fork in the intro system using the sample transfer tool provided. Place the cover on the intro system and start the pumping unit. Make sure the "up to air" valve is closed and open the valve to the intro unit on top of the pump and the valve under the intro. 15 minutes of pumping is needed for most samples, 30 minutes is required for samples with a high surface area such as powders or fibers.

3. To insert the puck into the analysis chamber open the gate valve and using the magnet on the control arm slide the fork into the chamber. Raise the stage to transfer the sample, retract the transfer arm and close the gate valve. Move the stage to the analysis position indicated by the black marks on the X and Y micrometers.

4. To start the x-rays first make sure that the rheostat on the power supply is fully counter clockwise to the zero position. Turn on the power switch on the x-ray source control and then turn on the High Voltage with the large red button on the far right. Using the large rheostat control knob on the power supply, slowly raise the voltage to 15kV. Chose the desired anode using the buttons on the left side of the central LED display. On the right side of the display

push the corresponding button to display the power to that anode. Using the up and down arrow buttons beneath the display, raise the power to 300W. Using the buttons on the right side of the LED display change it to HV and raise the voltage to 15kV again using the rheostat.

5. To obtain a survey spectrum use the “Acquisition” pull down menu at the top of the page in the AugerScan program and click on “New survey”. A standard survey starts at 1000eV and ends at 0eV, using 0.5eV/step, 89.45 pass energy, 10 sweeps and 30msec/step.

6. To turn off the system toggle the INT/EXT switch up and down, turn the rheostat on the power supply to zero and turn the power to the x-ray source control off.

7. To remove the sample from the system move the X and Y stage controls to the red marks on the micrometer scale. The intro chamber should still be under vacuum, check to see that the vacuum gauge, in the panel below the intro, is still pinned below zero. If not it will have to be pumped down again as if a new sample were being introduced. If the vacuum is still good, open the gate valve, slide in the transfer fork onto the bottom set of grooves on the sample puck. Lower the stage until it is free of the sample puck. Slide the transfer fork all the way out of the system and close the gate valve. Close the butterfly valve immediately below the intro and open the up to air valve to its right. Open the main valve on the high purity nitrogen tank and close it again right away. Lift the cover from the intro, slide the fork forward so the sample is centered in the opening and grab the sample with the transfer tool. Slide the fork back, lift the sample out and replace the cover.