Sample Insertion:

a. Mount the sample on insertion rod. This is done by removing the sample mount segment (hex screw fastener), mounting the sample and replacing the mount on the rod or by directly placing the sample on the sample rod with the mount in place. The sample should be firmly held in place and its dimensions should not extend beyond the diameter of the insertion rod.

b. Open the differential pump valve as shown in figure 1. This reduces the leak rate while the rod is moving. The rod is inserted midway in the sample chamber. This must be done manually by running the drive screw mechanism (IN BUTTON) until the sample holder is visible in the middle of the sample chamber. The STOP button must be pressed to stop the insertion process. If this is not done, the sample insertion will open the gate valve to air and vent the main chamber. At this point, the responsible staff member must be contacted and you will be charged for 8 hours of machine time required to re-pump the system to its ultimate vacuum pressure.

c. At this point, the roughing pump is actuated (the pump that backs the turbo pump is used for this purpose). The pressure is monitored using the Convectron gauge attached to this chamber. At a pressure of around 300 millitorr, the turbo pump is turned on.

d. The pressure in the introduction chamber (sample chamber) is pumped to the mid-10^-4 torr region. This is a function of the sample and will require more time when the sample is porous or contains significant amounts of weakly bound species such as water vapor. The sample should be held at this low pressure for a period of no less than 10 minutes to ensure that the main chamber pressure is within the operating range, <1.5 x 10^-7 torr.

e. When the pressure in the sample chamber is low enough for a sufficient time to permit sample degassing (at least 10 minutes), the sample can be introduced into the main chamber by pressing the proper button on the drive screw mechanism (IN). The gate valve will automatically open and the sample rod will run into the main chamber until it is turned to STOP. This is done
manually by pressing the STOP button when the sample is in or near the appropriate operating position. This marked on the insertion rod manipulator by a large black arrow (figure 1)! DO NOT PROCEED BEYOND THIS MARK as it may cause damage to the analyzer. The sample is now in the analysis position. By rotating the external control on the lead screw, fine adjustments can be made to the sample position.

**Turning the Instrument ON**

The main chamber pressure must be below $1.5 \times 10^{-7}$ torr prior to turning the x-ray source on.

**X-Rays:**

a. Turn the water chiller on the main (top) control panel in the rack ON. You will hear the pump start. A light near the middle of the rack should be glowing neon to indicate that the water is sufficiently de-ionized to start the x-ray source. **IF THIS IS INDICATOR NOT ON, DO NOT TURN THE X-RAYS ON.** Check with a responsible staff person.

b. Press the choice of x-ray source (Mg or Al) on the top control panel (2 buttons must be activated) as shown in figure 2. Then press the RED HV ON button on the right side.

c. The HV button will flash red showing that the high voltage power supply is activated. Using the large Variac control (at the center of the power supply below the control panel), increase the high voltage until the flashing stops. This indicates that the supply is within the operating range. Set the high voltage to around 10 KV.

d. Press the control button marked with an up arrow (power adjustment – figure 2). This will turn the x-ray filament on and increase the power to the chosen x-ray source. The power may be increased to 250 watts. For anode lifetime considerations, this should not be exceeded. The up arrow will scroll the power at two rates depending on the amount of time the button is depressed. Therefore, fine power adjustment is possible. After adjusting the power, re-adjust the high voltage using the Variac to 10-12KV. **DO NOT EXCEED 12KV!!!!** When this is complete, the remainder of the instrument may be turned on.
Analyzer Controls (figure 3):

The electron multiplier, system control and analyzer power supply must be switched on (figure 3 far left side of the panels). The controls are located in the sloping relay rack panels. Turn the power switches (not shown) **ON** and allow about 5 minutes for proper warm up and stabilization.

The electron multiplier voltage is set manually by selecting the voltage prior to turning the supply to operate. Generally the high voltage is pre-set to ~2.7 KV to reduce internal charge-up and provide sufficient count rates and should not need adjustment. Turn the HV **ON** (OFF/ON Below).

No adjustments are required on the system control. At rest, the DVM should read ~300 (decimal point display is not operative).

The analyzer power supply (lower panel) adjusts the **analyzer pass energy** (figure 3). This determines the instrumental resolution (200 eV pass for survey spectra and either 25 eV or 50 eV for high resolution spectra). No other adjustments should be made.

Computer Control:

The computer monitor is located on the set of racks on the left hand side of the vacuum chamber for the xps system (see figure 4). Typically, this is turned off. Turn it on. The computer is not networked **at present**; therefore, there is no sign-on. The operational program is RBD Enterprise **Auger Scan Version 3.1**. The program has been pre-set and calibrated by the staff and should not be adjusted. The main computer screen after opening the program is shown in figure 4.

Select **Acquisition** from the menu or by its appropriate button on the Auger Scan menu bar. The menu allows the operator to select a number of sub-menus. For XPS spectra, the only sub-menu used is the **“survey settings”** or **“settings”** selection. Figure 4 shows the program with the **“survey settings”** sub-menu activated.

For Survey Spectra, generally a 1000 to 0 eV range is chosen using a 1 eV increment. A larger range is available so long as the x-ray source energy is avoided (stay at least -50 eV away from
1253.6 or 1486.6 eV, for Mg or Al K-alpha, respectively). Smaller increments in binding energy can be used keeping in mind that the acquisition time will be increased.

High resolution spectra are obtained using a 10 to 20 eV region around the core-level peak binding energy being studied (e.g., C 1s at 285 eV should have a window extending from ~300 to 280 eV). The increment depends on the binding energy accuracy needed for the data. Typically, this is adjusted to 0.05 eV per step. The minimum increment is 0.025 eV per step and must be reset after each measurement. The program automatically rounds 0.025 off to 0.03 eV and the display increments become difficult to read.

The Pass Energy (set manually on the analyzer control) should be entered as a logging parameter. Pass energy is set manually on the power supply as discussed. The logging parameter box does not control the instrument in this case.

The time per step or dwell time is adjustable. For most XPS measurements, this should be set ≥50 ms/step. The lower limit is limited by the maximum count rate obtainable in the system. The maximum time per step is limited to 500ms by the program. The total number of scans determines the noise level of the resulting spectral data. Obviously the more scans that are obtained, the better the detection limit of the method is. Typically, a well defined sample will require from 2 to 5 scans for a survey. High resolution spectra will generally take more time to acquire and require as much time as needed to produce adequate data for subsequent analysis. Scans can be added to the spectral data after acquiring the data by activating the small green button (arrow shown in the figure) and typing the number of additional scans required. After each scan set remember to save the data. Not doing this can result in data loss. Small signals can be better detected by spending a longer dwell time but data acquisition can be very slow. The operator should optimize the dwell time versus the number of scans obtained to produce an optimum signal to noise level for a given problem.

Repeated analyses can be performed by re-displaying saved data. This provides the “settings” parameters. Simply click start. If the data is not saved, you may loose it. This will produce a response in the program: “Acquiring will delete all previous data. Acquire new data?” Answer
“YES” and the program will begin to acquire similar data for another sample. Remember to save your spectral data.

Data are stored in the XPS data file on the C-drive of the computer. The data can be copied to a standard floppy disk for transfer and further processing in *.txt format. This format is chosen from the file menu by EXPORTING the data.

Instrument Shut-down

a. Turn the x-ray source OFF by turning the HV on/off button on the control panel (top of the rack) OFF. Manually turn the Variac control to ZERO. Allow the water to circulate for 5 minutes then turn the main power off on the control panel (left most switch).

b. Turn the electron multiplier HV switch to OFF. Then sequentially turn the ac power to the multiplier, system control and analyzer power supply OFF.

c. Remove the sample from the main vacuum chamber by pushing the OUT button of the sample rod drive motor. This will stop mid-way in the sample chamber after the gate valve closes.

d. Turn the turbo pump OFF followed by the mechanical backing pump. The valve to the roughing pump used to differentially pump the o-rings should be closed.

e. At this time, the sample chamber can be vented using the vent valve near the Convectron vacuum gauge. It should be opened slowly. The turbo pump should slow down and stop rotating as air is vented into the system. When venting is complete, the sample rod can be removed from the sample chamber and your sample de-mounted. Close the Vent Valve for further use.

f. To store the system when you are finished using it, the sample rod is re-inserted mid-way into the sample chamber as described earlier. Remove the tubing from the small roughing pump (differential pump line to the o-rings) and reconnect it to the vent valve using the supplied connector. This should be tightened so that it is not apparently leaking and the pressure monitored until it is in the 10 torr or less on the Convectron gauge. Make sure that the gate valve to the turbo-molecular pump is closed to prevent oil from being pumped from the turbo backing pump.

At this time the system shut-down is complete.

Problems: see John Thomas x69657, Greg Haugstad x51352 or John Kadlec x44136

This system is presently being modified to improve its operation. If things don’t look correct, please check with one of the above people.