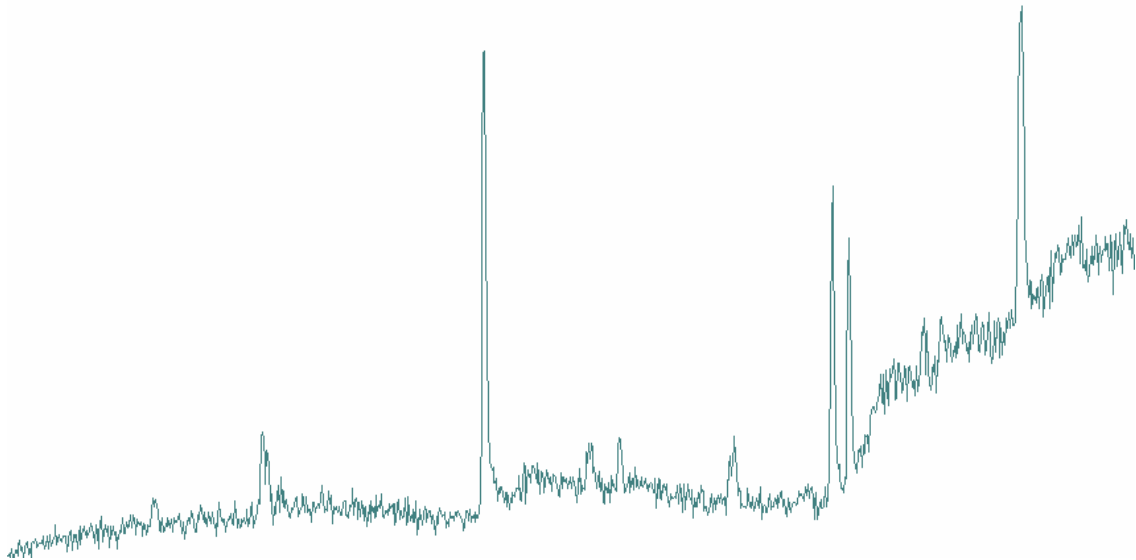


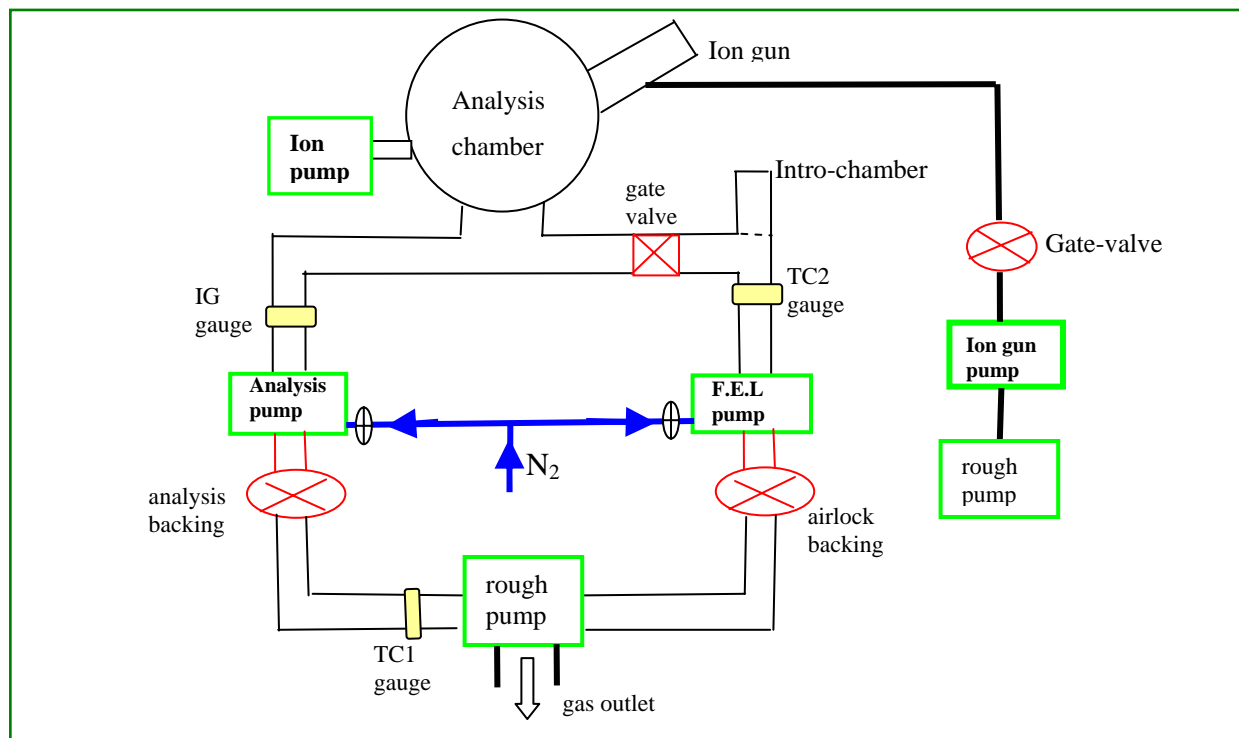
XPS

Brief Operation Manual



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XPS Operation Manual



A. Login

Log in the computer system and also write down the date, your name, & time on the log sheet.


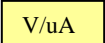

B. Load your samples

- 1) Make sure that:
 - a) all power on the control rack is off
 - b) the gate valve between the intro-chamber and the analysis chamber is closed.
- 2) **FULLY** close the “airlock backing” and the “analysis backing”.
- 3) Open the valve of the nitrogen tank.
- 4) Turn off the “F.E.L.” pump.
- 5) Wait for the “TC2” gauge to reach $>1.7 \times 10^1$ mbar (~10min).
- 6) Open the lid of the entry lock and load specimen; then close the lid of the entry lock.
- 7) Close the valve of the nitrogen tank.
- 8) Turn on the “F.E.L.” pump.

- 9) Make sure the “analysis backing” is closed tightly.
- 10) Open the “airlock backing”.
- 11) Wait for the “TC2” gauge to go down to $<1.3 \times 10^{-3}$ mbar and the “F.E.L.” pump green light to go “on”.
- 12) Wait ~10 min and then open the “analysis backing”.
- 13) Make sure the pressure of the “IG” gauge is $<1 \times 10^{-8}$ mbar; then open the gate valve between the intro-chamber and the analysis chamber. (“IG” = 03 E is good.)
- 14) Turn the illuminator on.
- 15) Slide the magnet to transfer the samples to the analysis chamber from the intro-chamber. After moving the samples from the sample-rod to the sample holder, slide magnet back to the original position.
- 16) Make sure the sample-rod is returned to the original position; then gently close gate valve between the intro-chamber and the analysis chamber.
- 17) Remove the magnet. (The magnetic field will affect the flying routine of the emitted photoelectrons.)
- 18) Turn the illuminator off.

C. Prepare to operate the ion gun (skip this if you don't need surface cleaning or depth profiling)

- 1) Make sure your sample is grounded.
- 2) Make sure the X-ray source and the e-gun are off. (You CANNOT run the X-ray source while you operate the ion gun.)
- 3) Turn off the ion pump.
- 4) Shut down the gate valve underneath the operation table.
- 5) Flush the Ar gas tube four times.
- 6) After flushing
 - a) close “Valve A” & “Valve B”
 - b) close the valve of argon tank
 - c) open the gate valve underneath the operation table.
- 7) Close the shutter for the X-ray source.
- 8) Turn on the power of ion gun.
- 9) Open the pin valve on the ion gun to adjust the pressure of the “IG” gauge of around 5×10^{-8} mbar.
- 10) Press the “OPERATE” button; then press the “HV ON” button on the panel.
- 11) Confirm that the focus voltage is set up as specified:






- a) Energy =2000V (first step!)
 - b) Extractor =1763 V; Focus 1 =1507V; Focus 2 =1507V
- 12) Press the  button to increase the “ELECTRON EMISSION” up to 4mA.
 - 13) Turn on the power of the scan unit.
 - 14) Press the  button to switch the voltage/current display.
 - a) Then adjust the pin valve to optimize the “energy” current as follows:
 - i) For metals or ceramics, “ELECTRON EMISSION”=3mA, “energy” current=10μA
 - ii) For polymers, “ELECTRON EMISSION”=1mA, “energy” current=2-3μA
 - 15) Shut down the ion gun after finishing your sputtering.
 - 16) Press  to reduce the “ELECTRON EMISSION” to zero.
 - 17) Close the pin valve on the ion gun.
 - 18) Press the “STANDBY” button, and subsequently turn off the power of the ion gun.
 - 19) Turn off the power of the scan unit.
 - 20) Turn on the ion pump.
 - 21) Open the shutter for the X-ray source.








D. Run the electron gun *(skip this if your specimen is conductive)*

- 1) Make sure the pressure of the “IG” gauge is $<8 \times 10^{-9}$ mbar.
- 2) Turn—
 - a) the “FILAMENT” potentiometer to zero.
 - b) the “EMISSION” potentiometer to 10.
 - c) the “BEAM ENERGY” knob to zero (fully counter-clockwise).
- 3) Turn on the power of the electron gun.
- 4) Switch the digital panel meter to “Wehnelt” and set the “Wehnelt” = 0V.
- 5) Switch the digital panel meter to “I FIL” (the filament current).
- 6) **Slowly** (*it takes at least 90 seconds*) increase the filament current to **1.70~1.73A** by tuning the “FILAMENT” potentiometer.
- 7) Switch the digital panel meter to “IE”(the EMISSION current), then wait for the “IE”=0.08~0.20mA.
 - a) If the emission current “IE” cannot reach to 0.08mA after waiting 2 minutes, slowly increase the filament current “I FIL” to **1.73-1.77A** (*no more than 1.77A for this old filament*).
 - b) Wait until the emission current “IE” goes up to 0.08~ 0.20mA.
 - c) Then adjust the “FILAMENT” potentiometer to get “IE” = 0.0020 to 0.0080mA.

- 8) Set the “**BEAM ENERGY**”=100eV, then check the following settings: “**Wehnelt**” = 0V, “**L2**” = 68-70, and “**L1/3**” =250-253 (by turning the “gain” knob).
- 9) Switch the digital panel meter to “**E**” (**BEAM ENERGY**) and reduce the “**BEAM ENERGY**” knob to 5~15eV.
- 10) **Shut down** the electron gun after your session:
 - a) Gradually reduce the “**BEAM ENERGY**” to zero.
 - b) Switch the digital panel meter to “**I FIL**”, and slowly reduce the filament current to zero.
 - c) Turn off the power.

E. Acquire data

- 1) Make sure the “**IG**” gauge pressure is $<3 \times 10^{-9}$ mbar and the ion-pump pressure is $<8 \times 10^{-9}$ mbar.
- 2) Turn on the power of the chiller.
- 3) Switch on the X-ray source main power, and subsequently press the “**Auto-start**” button. Then wait for the “**emission stabilized**” light to turn on. The default power of X-ray is 300W.
- 4) Turn on the power of the “**multiplier supplies**” and the “**EAC2000-125**”.
- 5) Open the “**EIS**” software and load file based on nature of your sample:
 - a) If your sample is a conductor, load the file of “**C:\users\templet.xps**”, and save as another file with the desired name in your folder.
 - b) If your sample is an insulator, load the file of “**C:\users\insulate-templet.xps**”, and save as another file with the desired name in your folder.
- 6) After you set up the table, click the  button to start the measurement. The instrument will automatically conduct the scanning from Region 1 to Region 2, Region 3.....
During the scanning, you can click the  button to stop the scanning. You can use  the button to automatically adjust the Y-axis scale.
- 7) Align the sample position
In order to get the maximum counts and the best resolution, align the sample height:
 - a) Check to make sure the sample is in the original position.
 - b) Click the  button to open the “**EAC125**” window and enter the value of binding energy of the predominant element in your sample.
 - c) After you check the box of “**multiplier**”, then a dynamic count bar will display on the lower right. In this case, you can adjust the sample Z-handle to make the count number reach the maximum.
 - d) After adjustment, close this window and click the  button to start the scanning.
- 8) **Attention:** During data acquisition, keep watching:
 - a) Watch the “**IE**”(emission current) of electron gun. Adjust the “**FILAMENT**” potentiometer to keep the emission current stable and constant during operation of the

- electron gun for neutralization. The value of emission current has a *significant* effect on the position and the shape of peak.
- b) Watch the “IG” gauge. If the “IG” gauge becomes $>8.5 \times 10^{-9}$ mbar, turn off the X-ray source, electron gun, the “multiplier supplies” and the “EAC2000-125”.
 - c) The spot size of X-ray beam is about 1.5mm in diameter. You have to collect signal from two or three different locations on the same sample.
- 9) Save and export data. (*Always save your data after scanning!*)
- a) Use “Save as” for your modified data, and name the new file “xxxx-offset.xps”.
 - b) *Before* exporting your data, polish the data:
 - i) Click the  button to calibrate (offset) the spectra. (Use C1s as the reference peak.)
 - ii) Click the  button
 - iii) Two cursors will display on the spectrum; move the two cursors to the two ends of a curve, and click the  button to smooth the curve; repeat by clicking the  button.
 - iv) Click the  button to remove the background of the curve.
- 10) After you have calibrated and smoothed the spectrum, click “export” in the “File” menu, and select “X,Y DATA” to export the file with text format. You can use EXCEL to open the exported file.
- 11) Peak intensity (peak area): If you click the  button, two cursors will display on the spectrum.
- a) Move the two cursors to “hug” a peak
 - b) Click the  button, and a “peak information” page will appear and help you to obtain the information of a peak (e.g. peak area).
- 12) Quantification: After the value of peak intensity (the peak area after background removal) is obtained, the atomic concentration of an element, C_i , can be expressed as shown in the formula: (where I_i is the peak intensity for element i , and S_i is the sensitivity factor for the peak i .)

$$C_i = \frac{I_i / S_i}{\sum_i I_i / S_i}$$

F. End Your Data Acquisition

Follow these procedures to make the instrument stay under the “OFF” status.

- 1) Save and back up your data.
- 2) Shut down the “EIS” software.
- 3) Turn off the “multiplier supplies” power and the “EAC2000-125” power.
- 4) Press the “Auto Start” button to make the X-ray source stay under the “stand by” status, and subsequently switch off the power supply of X-ray source.
- 5) Follow the following procedure described in “Section D” to turn off the electron gun.

- 6) Wait at least 20 minutes after you turn off the X-ray source to allow the X-ray source to cool down, and then turn off the power of the chiller.
- 7) Do NOT shut down the vacuum pumps. The vacuum system is running all the time.

G. Unload Your Samples

- 1) If you have operated the X-ray source, the ion gun, or the electron gun, you must wait for *at least 10 minutes* before you unload the sample. (This allows the filaments to cool down.) During waiting, keep the chiller running.
- 2) Open the gate valve between the intro-chamber and the analysis chamber.
- 3) Turn the illuminator on.
- 4) Slide the magnet to transfer the samples from the analysis chamber to the intro-chamber.
- 5) **Make sure the sample-rod is already returned to the original position.**
- 6) **FULLY** close the gate valve between the intro-chamber and the analysis chamber.
- 7) Switch the illuminator off.
- 8) **FULLY** close the “airlock backing” and the “analysis backing”.
- 9) Open the valve of the nitrogen tank.
- 10) Turn off the “F.E.L.” pump.
- 11) Wait for the “TC2” gauge to reach $>1.7 \times 10^1$ mbar.
- 12) Open the lid of the entry lock, and take out your samples. Then close the lid of the entry lock.
- 13) Close the valve of the nitrogen tank.
- 14) Turn on the “F.E.L.” pump.
- 15) **Make sure the** “analysis backing” is closed tightly.
- 16) Open the “airlock backing”.
- 17) Wait for the “TC2” gauge to go down to $<1.3 \times 10^{-3}$ mbar and the “F.E.L.” pump green light to light.
- 18) Wait ~10 min. Then open the “analysis backing”.
- 19) Ultrasonically clean up the sample stubs and the tweezers with acetone.
- 20) Switch off the power of the chiller (20 min after tuning off the X-ray source).

H. Logoff

Log off the computer system and write your log-off time on the log sheet.