

PHI VersaProbe II Standard Operating Procedure

els 171116 SmartSoft 2.8.0.30

BEFORE YOU BEGIN

Wake up the IMac. Log in to CORAL, select the Versaprobe XPS, and click on **Engage**. **Save** the initial XPS Run Time Data. Log out of CORAL. Record your appointment in BusyCal with an estimated end time.

In the **XPS Performance Log**, record the Versaprobe base pressures and the N2 tank pressure along with the time the record is made, plus your MUMMS username. *Do not use the XPS unless base pressures are normal and the N2 tank is reading 40 psi or better.*

Make sure the two rough pumps on the floor are running and there's nothing abnormal – for instance pooled water or oil – in that area behind the instrument.

Turn on the main chamber lamp. Confirm that there is no platen (sample holder) on the stage or on the transfer arm. If there is a platen on the stage, move it into the intro chamber before you begin mounting your samples.

INITIAL SOFTWARE SET-UP

Complete the following setup procedure before setting up acquisition parameters or loading a sample.

Click on the **System** tab at the top of the SmartSoft window, then click on the **Sample Transfer** tab on the right side of the window.

Click on the  button at the top right of the window. Type in a new Platen Name (include your MUMMS username and the date, for example: elshaw171116). Click on **OK**. A tabbed window will open.

Select the platen type you will be using (25 mm or 60 mm). Make sure the Max Z Height is **18.5** mm (or lower if your sample is more than 3 mm tall).

Click on the **Lab Book** tab. Delete the text in the Filename field and in the Comment field. Leave the Position List-ID, Position List-Name, and Position List-Comment boxes checked.

Click on the **Directory** tab. Click on the  button beside the Acquisition Directory pathname, browse to your directory, and create a new subdirectory with today's date (example: 171116 for November 16, 2017).

Click on your new subdirectory folder to select it. Click on the  button.

Click on the **Contact** tab. Put your name in the Operator Name field.

Click on the  button to close the Data Manager Properties window.

SAMPLE MOUNTING

Wear clean gloves to handle your samples and the platen, and save the gloves (if still clean) for use at the end of your session. Mount your sample onto a platen. Use tape only if it's absolutely necessary and only enough to affix the sample. Avoid covering sample holder holes with tape. Mount samples on insulating tape if there's a possibility of differential charging. If you have multiple samples, make a drawing of the platen identifying each sample. Ensure that all samples are securely mounted and that there are no loose particles anywhere on the platen.

It is advisable to ensure that all samples on the platen are approximately the same height. If samples of varying heights are mounted together, special care must be taken to avoid accidentally running a tall sample into the analyzer when a shorter sample is being moved into position for analysis.

Keep in mind that pump-down time is charged in full. If your samples are porous or hygroscopic, use as small a sample quantity as can easily be analyzed, and keep samples in a vacuum desiccator until just before you mount them.

SAMPLE PUMPDOWN

In SmartSoft, go to the **System/Sample Transfer** menu.



Click on the  button at the top right of the Sample Transfer menu to backfill the intro chamber. Put on your gloves. Wait for the intro chamber Convector gauge to reach atmospheric pressure. *If the main chamber pressure degrades into the e-8 range or worse, record the maximum degraded pressure in the Performance Log and notify Libby.* Remove the intro chamber cap and set it down upside-down so that its flat sealing surface stays clean.

Before mounting your sample on the transfer arm, move the screen at the bottom of the intro chamber to cover the turbopump port.

Slide your platen onto the transfer arm fork with its big hole facing the main chamber. Check that the platen is level, then make sure the platen's alignment groove is precisely aligned against the transfer arm screw by using one gloved finger to attempt rotate the platen a bit. Confirm that the transfer arm magnet pin is touching the back stop plate. Don't put the intro chamber cap back on yet.



Click on the  button. SmartSoft will tell you to leave the intro chamber cap off and take a photo of your platen. Click **OK** to take the photo. Visually confirm that the flat sealing surface of the intro chamber cap is clean, then replace the cap securely on the intro chamber port.

Click on the **Sample** tab to display the intro chamber image. Wait patiently for the photo to display, then check that the bottom half of the platen is aligned with the blue circle and that the image is in focus.



If you need to re-take the photo, go back to the **System/Sample Transfer** menu, and click on the  button. Use the gooseneck lamp if needed to side-illuminate your samples for the photo. Again wait patiently for the photo to store and check the photo before starting the intro chamber pump-down.

When you have an acceptable photo image, click **OK** to start the pump-down. Watch the Intro Convector meter to confirm that the intro chamber pressure is dropping at a normal rate. Turn off the chamber lamp.

When the turbo pump valve (V3) opens, watch for the SmartSoft cold cathode gauge meter to turn on. An initial reading in the low e-4 Torr range means pump-down is starting normally and may finish in 20-30 minutes.

Wait for SmartSoft to pop up a window to tell you the intro chamber pressure has reached 1.4×10^{-6} Torr. This is **almost** the platen transfer pressure.

During pump-down, click on the **Sample** tab and create some initial analysis positions in the **Position List**.

After the analysis positions are created, check that the Position List is unlocked , then type a sample name in the **Name** field for each position.

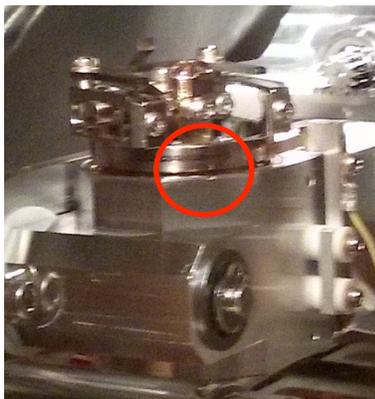
Change the initial Z value of the first sample position to **17.6mm** and hit Enter. Click on that Z value and use the **Set All** function (right mouse-button) to set all the rest of the sample positions to the same Z value. (Set a lower Z value for any sample that is unusually tall.)

STAGE INITIALIZATION

Perform this once per week and any time the platen transfer is not perfectly smooth.

If you are the first user of the week (starting from Monday), go to the **Sample/ Stage** tab and click on the **Initialize** button to initialize the stage and anode motors. Initialization will take about five minutes. When the initialization routine instructs you to, inspect the stage visually through the chamber window to make sure the rotation alignment marks are precisely aligned with each other (see picture below). If the marks are out of alignment, use the **Rotation** button to rotate the stage to the aligned position. Click **OK** when the rotation alignment is correct.

In the **XPS Performance Log** record that you have initialized the stage and whether the stage rotation required realignment.



SAMPLE TRANFER INTO THE MAIN CHAMBER

When SmartSoft displays a message that "Pump intro task is complete", **wait** for the cold cathode reading to drop **below** 1.4×10^{-6} Torr, then click on **OK**. The gate valve (V1) will open, and SmartSoft will tell you to introduce the platen into the main chamber.

When the gate valve (V1) opens, confirm that the main chamber pressure quickly settles in the $e-8$ Torr range or better.

If the main chamber pressure with the gate valve open is worse than $e-8$ Torr, do not introduce the platen into the main chamber. Instead, slide the magnet forward about 3 inches, then fully retract it to close the gate valve. Click "Cancel", then "Stop all" to end the Intro sequence.

Pump the sample in the intro chamber for another ten minutes, then click the Transfer Sample (Intro) button. 

If the main chamber pressure with the gate valve open is adequately low, slowly insert the transfer arm until the magnet contacts the front stop plate. Watch carefully to confirm the platen doesn't bump the stage. Then click **OK**. The stage will rise 1.5 mm to engage the platen. Watch to make sure the stage engages the platen smoothly.

When SmartSoft tells you to, slowly disengage the transfer arm from the platen. Watch to make sure the transfer arm disengages from the platen smoothly.

Retract the transfer arm all the way until the magnet pin hits the back stop plate. The gate valve will automatically close. Check that the main chamber pressure starts to recover.

Click on **OK** to confirm that the transfer arm is retracted. The stage will then move to the CENTER position.

*Confirm that the main chamber pressure is **better than 2.0e-8** after the gate valve is closed. If it is not, or is rising, do not turn on any sources – instead, wait for the main chamber to reach 2.0e-8 or better, or extract the platen from the main chamber to the intro chamber to pump longer.*

Turn off the main chamber lamp.

INITIAL Z ALIGN AND SURVEY ANALYSIS

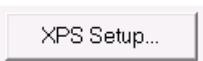
The following analysis routine will automatically move the stage to the first active sample position, adjust the stage height to the correct Z value to put the analysis region at the focal point of the energy analyzer, update the stage Z value for the sample position, then acquire a survey spectrum, then do the same for all other active sample positions.

In the **Position Table**, activate every sample position for which you want to run an initial survey.

Go to the **XPS /Spectrum** menu.

In the **Spectrum Settings** pull-down menu, select **Survey 1100eV standard**. Click on the **Load** button.

Use the default X-ray Setting **200um50W15kV** (for strongest signal in point mode) or select another setting of your choice.

Click on the  button. The **XPS Acquisition Setup** window will open.

If your sample may be insulating, set the electron gun and ion gun options under Automatic Neutralization to **Auto[A]**. If your sample is conductive to ground, set both options to **Off**. Confirm the **E-Neut Setting** is **E-Neut** and the **Ion gun Setting** is **I-Neut**. Leave Source Tolerance Required **Enabled**.

For the first analysis on each sample position, set **Automated Z-Align** to **Auto[A]**. *If you have already Z-aligned the sample position and updated its Z parameter, set **Automated Z-Align** to **Off**.*

Leave **SXI Photo**, **Presputter Before Acquire** and **Beam Parking** set to **Disabled**. Leave **Auto Shutdown** set to **Enabled**. Leave **Acceptance Angle** set to **Standard**.

Close the **XPS Acquisition Setup** window.

Click on the  button to start your analysis.

Additional note: STAND-ALONE Z ALIGN

Stand-alone Z Align can be useful before taking an SXI image of a new sample. The following explains how to use the stand-alone Z Align routine to adjust a sample analysis position to the focal point of the energy analyzer.

Preliminary checks:

- Set SXI **Image Size X** to the minimum scan size (175 um).
- If the sample is insulating, confirm that **XPS/SXI/Properties** shows both neutralizers set to **Auto[A]**.
- If you'd like to see a live graph of the signal intensity versus stage position while the Z Align routine is in process, right-click on the **Z Align** button and select **Z Align....** *Do not change any parameter values in the Z-Align window.* **Close** the Z-Align window when Z alignment is finished.

Main steps:

1. Highlight a sample position in the **Position Table**, then click on the  (Drive to Position) button to move the stage to that position.
2. Left-click on the  (Z Align) button above the SXI image window.
3. Wait for the Z-Align routine to finish (i.e. for the stage to stop moving), then click on the  (Update Position to Current Stage Position) button to store the corrected Z value.

SHUTDOWN PROCEDURE

Go to the **System/Sample Transfer** menu. Click on **Extract**. If you've been using the dual neutralizer, turn the E-Neut and Ar ion gun sources Off (rightclick on the source icon, then select **State: Off**). Wait for the intro chamber to reach 1.3e-6 Torr or better, then extract your platen back out to the intro chamber with the same care that you used to insert it. Never trust an automated stage!

When you are **EXTRACTING** the sample, make sure that when you first open the gate valve, the main chamber pressure settles to no worse than the e-8 Torr range. When the gate valve closes again, make sure the main chamber pressure drops quickly back down into the e-9 Torr range.

When SmartSoft indicates the automated **Extract** procedure has completely finished, click on the  button to backfill the intro chamber.

Wearing your clean gloves, remove the intro chamber cap and slide your platen off the transfer arm.

Move the screen at the bottom of the intro chamber to cover the turbo pump port.

Inspect the intro cap seal to make sure it's clean, then immediately replace cap and click on the  button.

When pumping begins, watch the intro chamber Convectron gauge for a few seconds to confirm that the chamber is pumping down at normal speed.

Make sure the main chamber lamp is turned off.

Remove your samples from the platen, clean any sample or tape residue off the platen, tighten down any loose screws, and put the platen away. Tidy up the sample preparation area.

Copy all of today's data onto your USB device.

LAST THINGS BEFORE YOU LEAVE THE ROOM

Don't forget to log out of CORAL when you're finished: Log back into **CORAL** (remembering to wait first for the IMac internet connection to be established.) Select the Versaprobe XPS and select **Disengage**. Check the Run Time Data for accuracy, update if needed. Click on the **Save and Disengage** button.

If the time period of your analysis session is different than what you initially recorded in the **BusyCal** calendar, update your **BusyCal** appointment record.

If you've had any nonstandard interactions with the XPS or the SmartSoft software, please record them in the **XPS Performance Log** (including any error messages) and tell Libby about it as well.

Turn out the light and close the door. Bye! See you next time!

ADDENDUM

If you find the C60 in Standby mode when you come in,

Leave the C60 in Standby. It is isolated from the main chamber by a mechanical valve, so unlike the other sources, it can safely stay on during sample transfer.

Record in the XPS Performance Log that you found the C60 in Standby.

The C60 source needs a minimum of 4 hours to stabilize before use. **If SmartSoft is restarted, the C60 is automatically turned off.** This means that if the C60 is on when you come in to use the XPS, it's important that if you must restart SmartSoft you also restart the C60 right away.

C60 RESTART PROTOCOL

If you must restart SmartSoft while the C60 is in Standby,

After you restart SmartSoft, immediately go to the **Hardware/Ion Gun/C60 10kV Ion Gun** menu. The C60 Settings Ion Gun name should be "PREVIOUS".

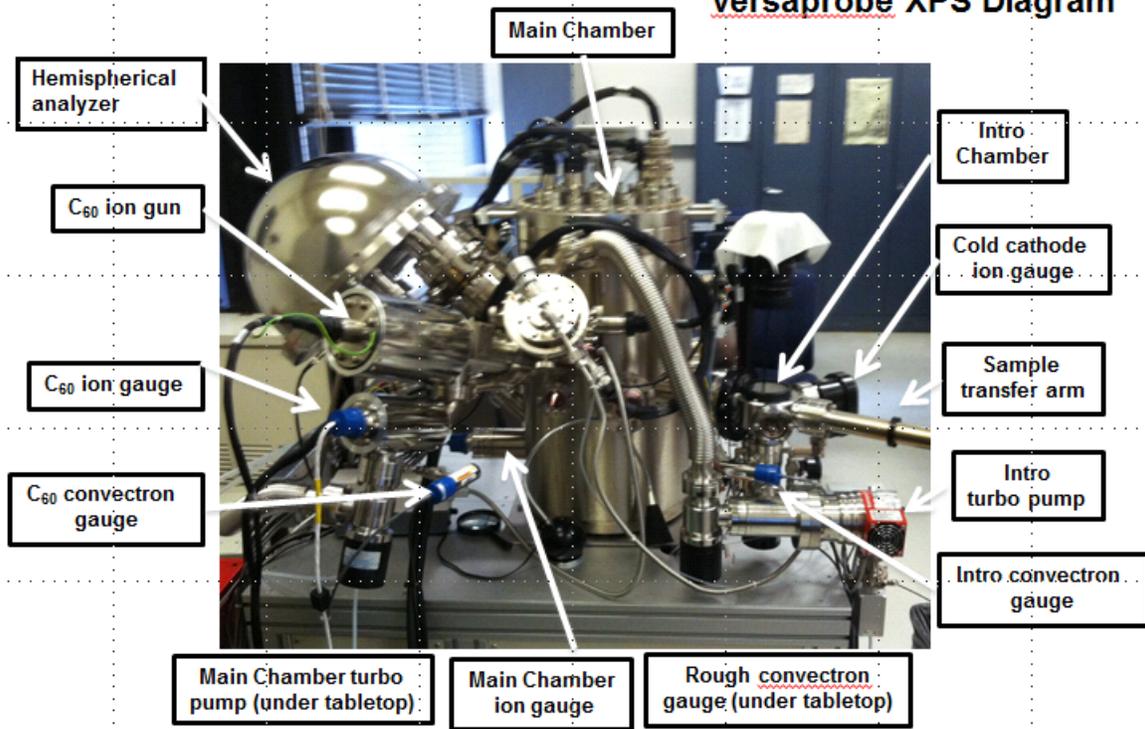
In the C60 menu, click on the **Standby** button. Wait for the cursor icon to change back to an arrow. This will take a few seconds.

After the cursor changes back to an arrow, increase the source control temperature by 5 degrees (don't forget to hit "Enter"). Then decrease it back to its former value (and hit "Enter"). (This step is a workaround for a stupid software bug.)

Observe the C60 ion gauge pressure. A minute or two after the C60 heater turns on, you should see a slight rise in pressure.

Record in the Performance Log that you restarted the C60 and whether you observed a pressure rise.

Versaprobe XPS Diagram



XPS Gauge	Base pressure (torr)
C ₆₀ ion gun	low 10 ⁻⁸
Main Chamber	low to mid 10 ⁻¹⁰
Intro Chamber (cold cathode)	10 ⁻⁸ to low 10 ⁻⁷

Gauge Type	Range (torr)
convection	10 ⁻⁴ to 760
cold cathode	10 ⁻⁹ to 10 ⁻²
hot cathode	10 ⁻¹¹ to 10 ⁻³

What if Intro or Main Chamber pressure is above normal base range?

The instrument may take a few hours to return to base pressure after use.

If the XPS has not been used recently, and this pressure is out of base range, then cease work and call Libby.

What if C₆₀ ion gun pressure is above normal base range?

This is normal if the C₆₀ gun is in standby or has just been turned off. Otherwise cease work and call Libby.

Always record the reason for a higher-than-normal pressure reading in the XPS Performance Log.