Standard Operating Procedure I
PHI 5000 *VersaProbe* II XPS/UPS
(Basic XPS Measurement)

> *Please **FOLLOW the SOP strictly** to keep the facility in good condition. Any **explorations are strongly prohibited** unless permitted by lab manager*
> ***NEVER*** *use your own USB drive on the* XPS computer. *Data can be retrieved from Yale data server*
> ***NEVER*** *surf the web on the* XPS computer *in order to minimize the risk of the computer being hacked*

> *Yale West Campus MCC facility users must acknowledge MCC in their publications that rely significantly on MCC resources. The general acknowledgement for SEM should read:*
> "The spectra were taken using the PHI VersaProbe II at Yale West Campus Materials Characterization Core (MCC)."
> *The core reserves the right to use the data for core promotion*

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PHI 5000 *VersaProbe* II XPS/UPS Standard Operating Procedure (Basic XPS Meansurment)

1   Introduction

1) Features of this Instrument:
> Micro-area element composition and chemical state determination on material surfaces
> Analysis of insulating samples with dual beam charge neutralization method
> Depth profile analysis of structures and interfaces

2) Location

Materials Characterization Core
Room E119
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3) Primary Staff Contact

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The Yale West Campus MCC Facilities are operated for the benefit of all researchers. If you encounter any problems with this facility, please contact the staff member listed above immediately. There is never a penalty for asking questions. If the equipment is not behaving exactly the way it should, contact a staff member.

Notice: Please **follow** strictly the **SOP** to keep the facility under good condition. We **DO NOT** recommend user explorations on program unless endorsed by core staff.

2   System Initial Status Check

Notice: Before sample preparation and loading, the users are required to check the XPS system status following the steps listed below:

1) Check to see if the XPS instrument is **ON**, i.e., the **green light** highlighted by an arrow on the right side control rank is illuminated and there are pressure displays on the vacuum gauge reading:
   Warning: If you find that the XPS instrument is **OFF**, contact a MCC staff member for help.

2) Check if the XPS computer is **ON**. Start the computer if it is **OFF**.
3) Check the pressure reading of the Main Chamber Ion Gauge on the XPS control rack:
   It should be in the range of **low 10^{-7} Pa** or **high 10^{-8} Pa** when the system is idle.
   Warning: If the pressure reading is above 8×10^{-6} Pa, contact a MCC staff member for help.

4) **Sign in** on the **logbook** and put down **date**, **usage time**, **sample materials**, Main Chamber and Intro Chamber initial **pressure** before sample loading, **data collection modes** used, and report any issues during measurement.

3   Sample Preparation

Warnings:

> **Always wear gloves for vacuum sample preparation!**
> **The sample for XPS and UPS needs to be completely dried! If the pumping time exceeds 1 hour, the sample has to be taken out for further treatment.**

3.1   Sample holders

1) Select a **1 inch** regular sample holder for **as-is** sample analysis

Front face   Back face

2) Choose **1 inch Heating Sample Holder** with **heater** if *in situ* heating is required
Temperature range: **RT – 800 ºC**

Front face   Heating

Heating area   Thermocouple
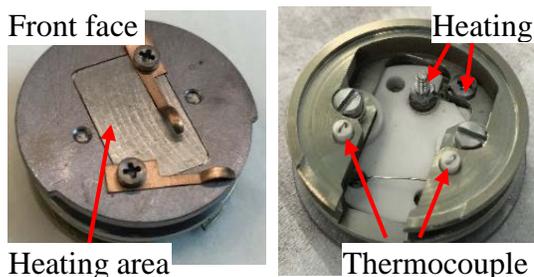
Warning:

> **Always use the vacuum only screwdrivers** to adjust screws on the holder. **The screws can be easily damaged by using wrong size screwdrivers. Users are not allowed to fix sample holder, report** to lab staff immediately.

> Keep **Reading Heater Power below 75%** when tune the **PID** control in **HCControl** program. **Violation** will lead to **damage** to the **stage** and **sample holder**. User's account will be suspended and repair cost will be charged on PI's account.

3) Choose **1 inch Heating/Cooling Sample Holder** if *in situ* cooling and heating required
Temperature range: **-140 ºC – 600 ºC**

Front face   Heating   Cold finger

Heating area   Thermocouple
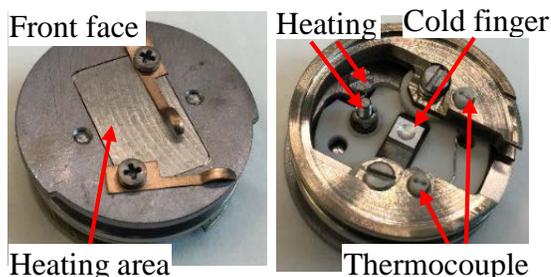
4) Contact MCC staff member to run **angle resolved** (**AR) XPS/UPS** using **AR** sample holder

3.2 Sample Mounting

Warning: the **maximum** sample thickness is **5 mm** above sample holder top surface

5 mm

Notice: The **minimal** sample size should be **3 mm x 3 mm for XPS** and **5 mm x 5 mm for UPS** (UV beam size at stage is ~ 4 mm x 4 mm)**.**

1) **Non-powder samples**:
   a) Fix sample with **copper clips** or **Mo cover** on the sample holder (the sample can be **heated** in vacuum):
   b) Fix sample using **double sided Scotch tape** (for **XPS** only) or **carbon tape** (for **XPS/UPS**). (**sample cannot be heated in vacuum**)

   Warning: after sample mounting, **shake** the sample holder and **turn it around** to make sure the sample will not come off the holder.

2) **Powder Samples:**
   a) Choose **Si wafer** as support (the sample can be **heated** in vacuum):
      > **Add powder in solvents** such as ethanol, isopropanol (IPA) or Dichloromethane (DCM) and mix the solution using ultrasonicator.
      > **Drip** small amount of suspension onto a piece of Si wafer (3×3 mm^2 ~ 5×5 mm^2)
      > **Dry** sample about 10 minutes under infrared lamp
      > Repeat above steps to have enough deposition on Si substrate
      > **Mount** sample on the holder using copper clips or Mo cover if heating is needed in vacuum;
   b) **Choose double sided tapes** (**sample cannot be heated in vacuum**)
      > **Sprinkle** powder on **Scotch** tapes (**XPS only**) or **carbon conductive** tapes (**XPS/UPS**), then cover the surface with weighing paper and firmly press the surface with spatula
   **Warning**: carefully remove all loose particles from the sample holder:
      > **Shake** and tilt down the sample holder to make sure no additional particles comes loose
   > **Blow** the sample surface with dry N$_2$ inside fume hood

4   Create Sample

Notice: "**Create Sample"** is the first step of data analysis. It **SHOULD ONLY** be performed **before** loading the sample into **Intro Chamber**. The following steps should be taken to create sample (platen):

1) Open **SmartSoft-VersaProbe** program on desktop (if the program was closed) and click **OK** (no password required).

2) Click ![System icon] **System** to enter the **System** window, then click the **Sample Transfer** on the right side to enter **Sample Transfer** tab.
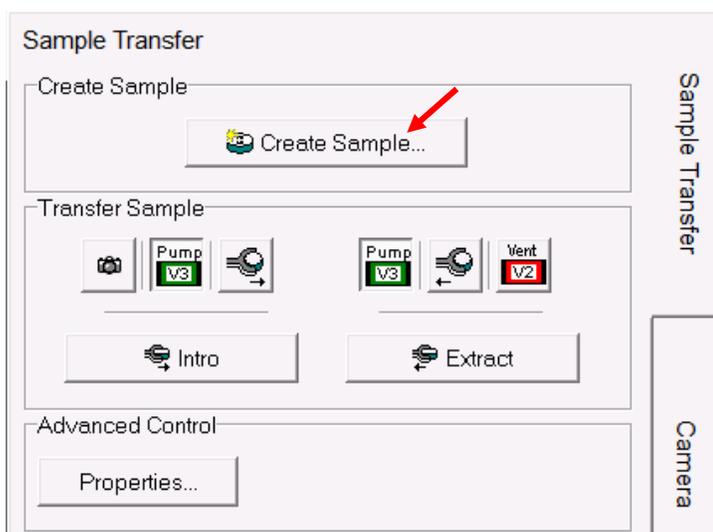

3) Click ![Create Sample button] button to open Create Platen (sample) window
   Warning: once a **New Platen Name** is created, **NEVER** change or **CREATE** new platen names in the middle of data collection. <mark>Violations may lead to hardware damages</mark> inside vacuum as all sampling positions are affiliated with the initial Platen Name.
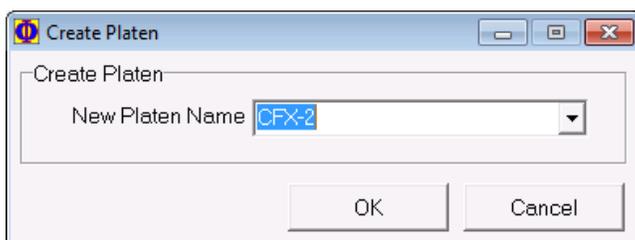
4) Create a new platen name and hit **OK**, the **Data Manager Properties** window appears. **Make sure** sample holder of 25 mm is chosen.
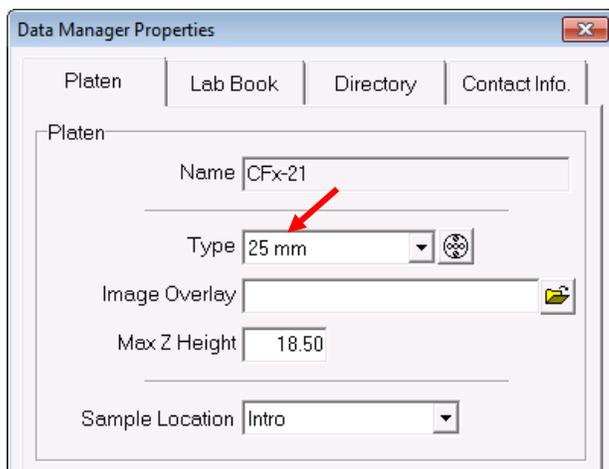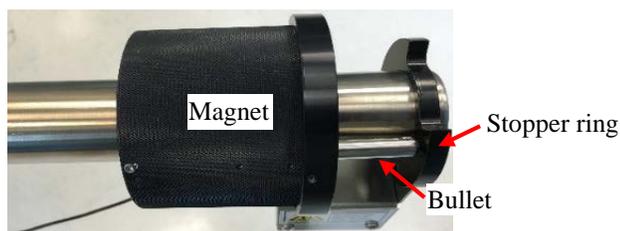
5) Click **Directory** and specify the data **Acquisition Directory**
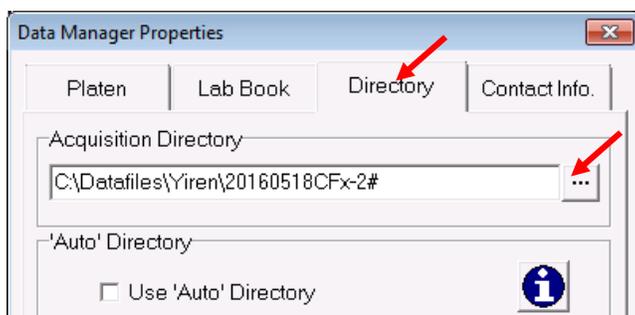
5   Transfer Sample

1) **Check the system status before sample loading**:

> Check to ensure the **transfer arm** is fully **retracted** that the **bullet** on magnet **touches** the stopper ring on the transfer arm


> At the bottom right of the **SmartSoft** program window, check to **confirm** X-ray source is **OFF** `XRay: Off`, Electron Neutralizer **OFF** `ENeut: Off [A]` and Ion gun **OFF** `IGun: Off [A]`. Contact Core manager if not **OFF**.

> Confirm **no sample holder** inside the Intro Chamber or Main Chamber: only blank square boxes are shown in both chambers.
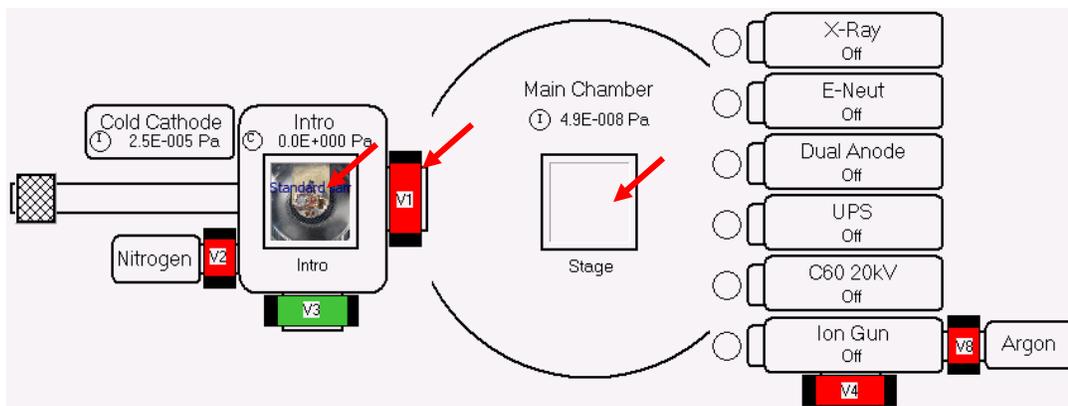
> In above system diagram, check if "**V1**" valve is **closed** (in **red** border) between **Intro** and **Main Chambers**. If not, contact MCC staff immediately.
> Check [LM1] **building supply N₂ pressure** on the wall behind computer monitor and make sure it is set below ~ **3 psi**. If the pressure is above 3 psi, decrease the pressure setting to **3 psi** by turning the pressure regulator valve *counterclockwise*. Contact MCC staff if need assistance.


Regulator

Gauge
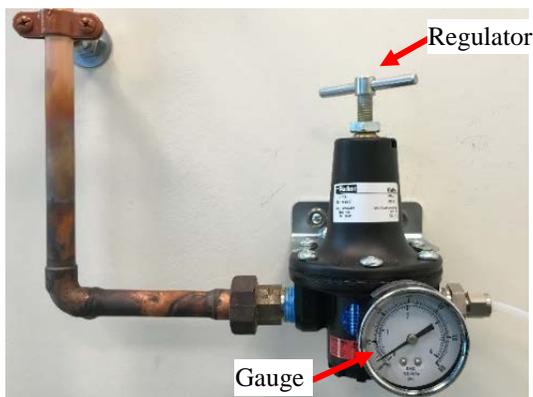
2) If the Intro Chamber is in vacuum, click in the **Sample Transfer** window
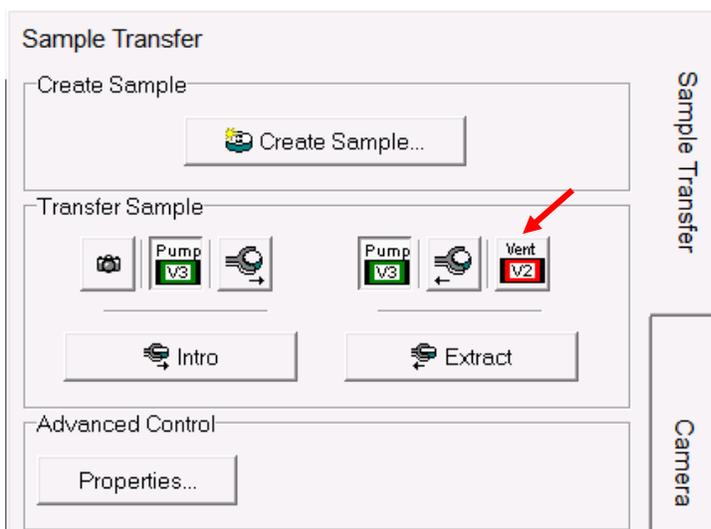
3) Wait until the Intro Chamber is vented, remove the Viton cover and slide the sample holder along the round groove on the bottom of the sample holder onto the transfer arm fork

4) Make sure the **dent** on sample **holder aligns** with and **touch** the **screw** on the transfer arm fork and check from side window flange if the holder stays flat on the fork:
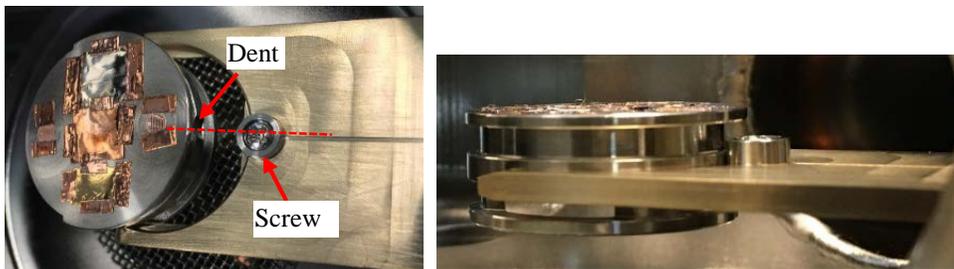

5) Leave the top flange **open**, **fully retract** the transfer arm so that the **bullet** on magnet **touches** the stopper ring on the rod
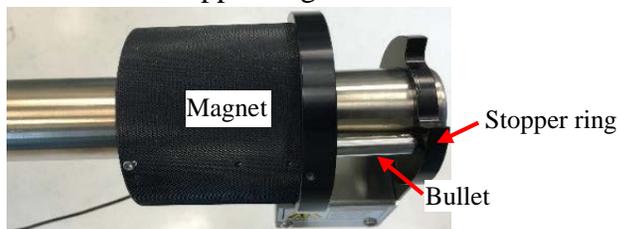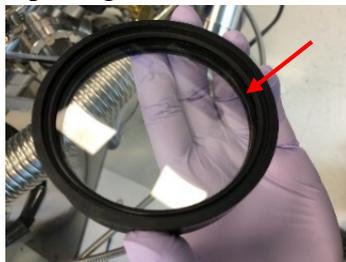

6) Click button on the **Transfer Sample** window to take a picture on the samples:
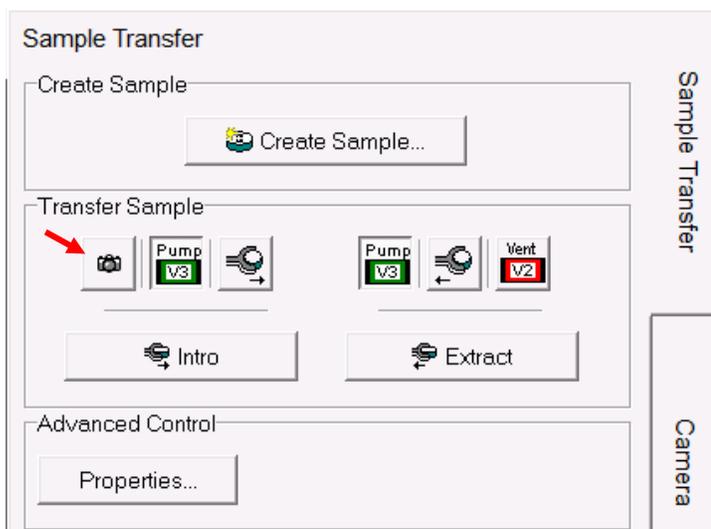
7) **Wait for several seconds**, click the [Sample] tab next to [System] enter the Sample window. Check the sample holder photo and make sure the **blue circle** aligns well with the **bottom rim** of the sample holder. **If not, contact lab manager**.
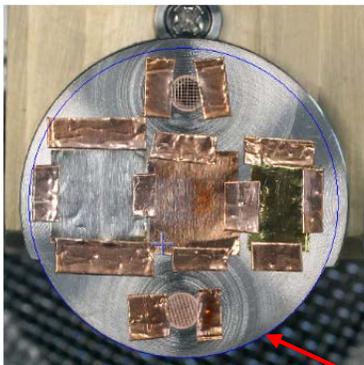

8) **Check carefully the sealing surface of** the **Viton cover** and the groove and **clean** using Kimwipes with isopropanol if dusts/particles can be seen and then put the cover back onto the top flange on the Intro Chamber


9) Click [Pump V3] button on the **Transfer Sample** window and go back to Intro Chamber quickly to **hold and press the Viton cover against the flange during Intro Chamber pumping!** **Warning: fail to follow above steps will cause the Intro Chamber leaking during pumping**.

Note: **slight rotating** adjustments on the **Viton cover** may help to assure the **Viton cover** placed correctly on the flange.
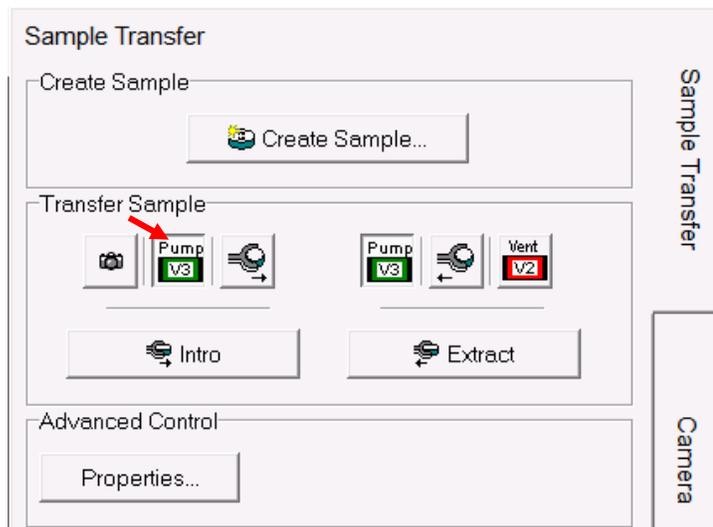
10) Check Intro Chamber **status right after pumping**

> Click to enter the **System** window, monitor the pressure drop from Stage 1 pressure gauge (Pirani gauge) from atmosphere pressure to ~3.0E+000 Pa in 5 minutes and then Stage 2 pressure gauge (Cold Cathode gauge) starts at ~ 1E-002 Pa and drops continuously to **4.0E-004 Pa** required for sample transfer to Main Chamber within ~15 minutes.
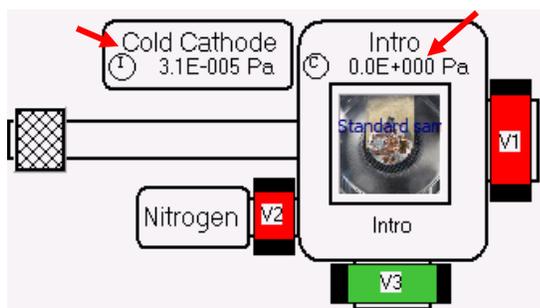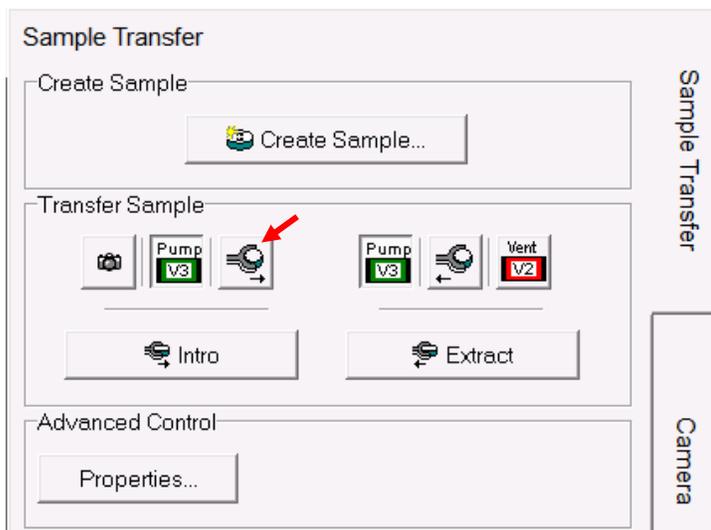

Warning:
> The actual Intro Chamber pumping time will vary on different samples
> It is strongly recommended to make sure user samples are **DRY and UHV (Ultra High Vacuum) compatible**. If pumping time **exceeds 60 minutes**, the samples are required to be **removed** from **Intro Chamber** for further processing

11) Before sample transfer to **Main Chamber**, check the Cold Cathode pressure reading to make sure the **Intro Chamber** pressure reaches below **4.0E-004 Pa**
Warning: Sample transfer at high Intro Chamber pressure will introduce **serious contaminations** to **Electron Energy Analyzer**, **X-ray**, **UV sources** and sample stages.

12) Click in the **Transfer Sample** window to transfer sample from **Intro Chamber** to **Main Chamber**

Warning: NEVER stop the transfer process in the middle and **force to close** the **SmartSoft** program in **Task Manager**. **Contact** Core manager immediately if did by accident

13) Wait for the following **three windows** popped up in sequence:

14) **DO NOT** click **OK** when the last **Task Information** window above pops up.
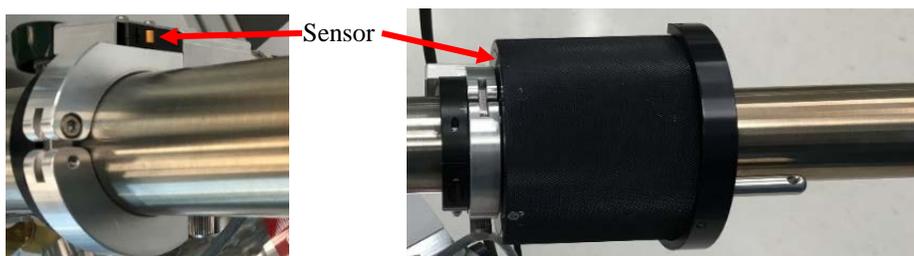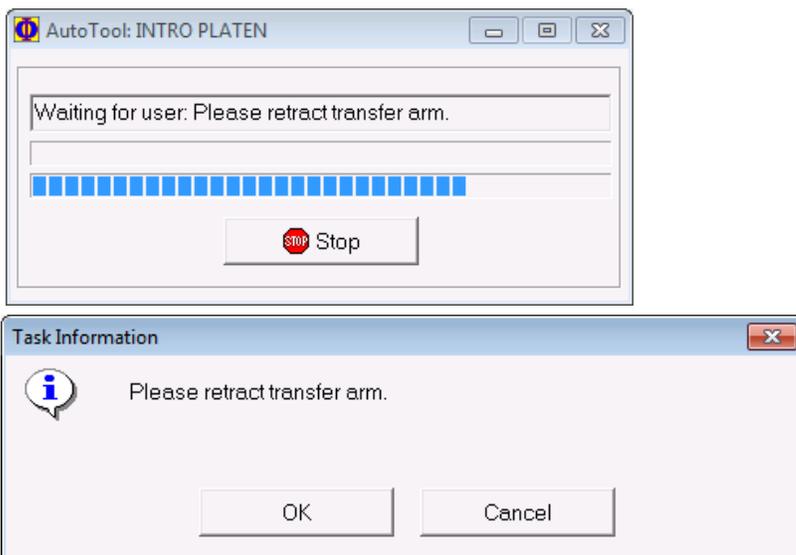   a) <mark>Follows the instruction</mark> in **Task Information** window to move **FIRST** the transfer arm into the transfer position in **Main Chamber** and **THEN** click **OK.**
   b) The transfer positon means that the transfer arm forks slide smoothly into the round groves on sample holder and the transfer arm magnet touches the **sensor** on the front stopper ring on transfer arm.

Notice: slight adjustment maybe needed to insert sample holder into the stage forks.

Warning: Clicking **OK** first on above window before the transfer arm is moved into transfer position in Main Chamber will lead to <mark>serious mechanical damage</mark> to both stage <mark>and sample holder.</mark> User account will be suspended and repair cost will be charged on PI's account.

Sensor

15) Wait the following **two windows** popped up in sequence:

16) **DO NOT** click **OK** when the last **Task Information** window pops up. Follows the instruction on **Task Information** window above to fully retract the transfer arm back to the **Intro Chamber FIRST** and **THEN** click **OK**

Warning: Clicking **OK** first on above window before the transfer arm is retracted will lead to <mark>serious mechanical damage</mark> to both stage and sample holder. User account will be suspended and repair cost will be charged on PI's account.
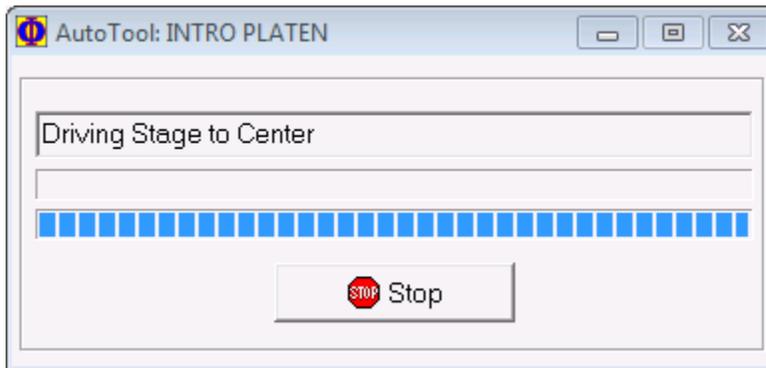
17) After clicking **OK** Wait in the **Task Information** window, **wait until** the stage is driven to the center.

6   XPS Scan

1) Define analysis **Position** with the sample in **Main Chamber**

   a) Click ⊙ Sample on top tab menu to enter **Sample** window

   b) Hover mouse cursor over the sample photo window on the left, scroll mouse wheel to enlarge photo and find the sample to be analyzed
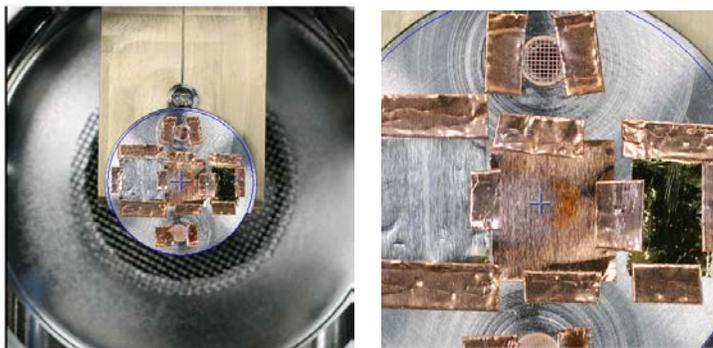
   c) Right click mouse on intended analysis position and select **Drive To Click** on the dropdown menu, the **Main Chamber Stage** will move to selected position

   d) Right click mouse on blue cross cursor select **Create Point at Stage** on the dropdown menu, an **Active Position** (ID: 1) will be created in the **Position List Table** below sample photo window

   e) Type **Position Name** in the **Name** window above

f) Click **Stage** tab on the right side of **Sample** window to enter **Stage Parameters** window and chane **Z(mm)** height from **5.0** to **12.0** (hit **Enter** and then hit [z] tab on the right to move stage to entered height)
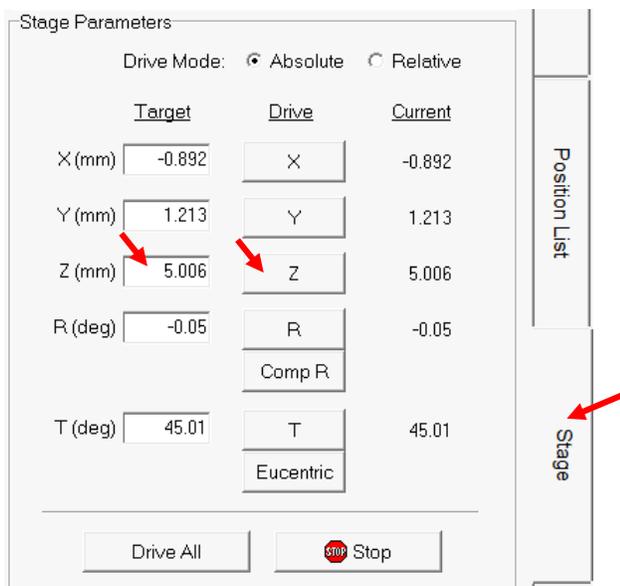
g) Hit [Z↓] button on top of **SXI** image window to start **Z-Align**

h) The following windows will pop up sequentially, **wait until** all windows disappear

i) **Click** above **Position List Table** to **Update Position** after **Z-Align**
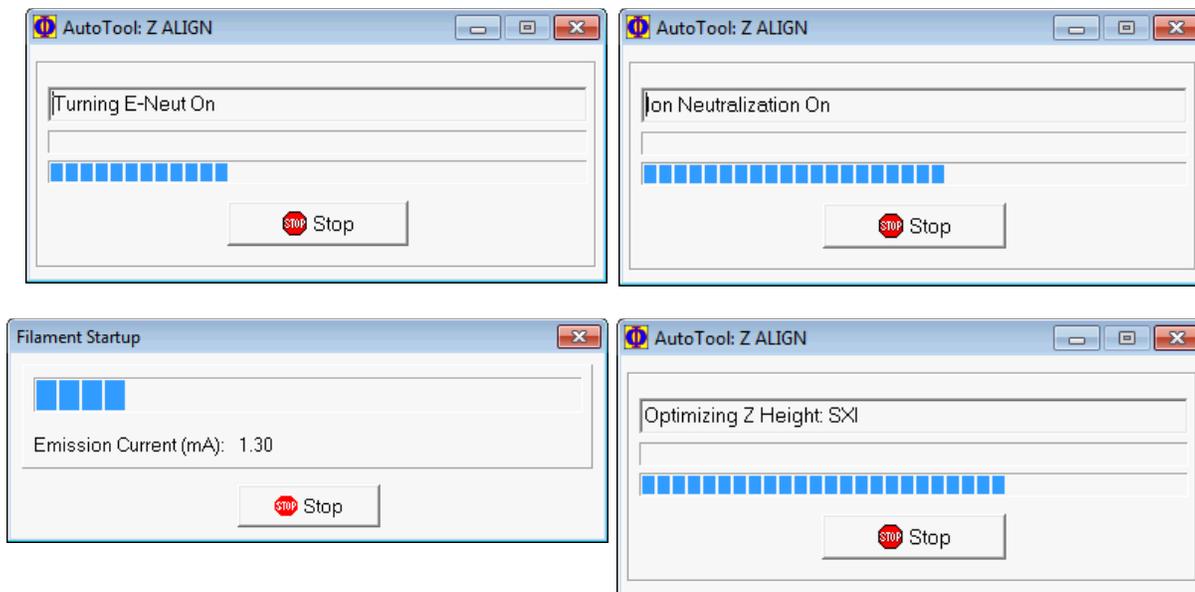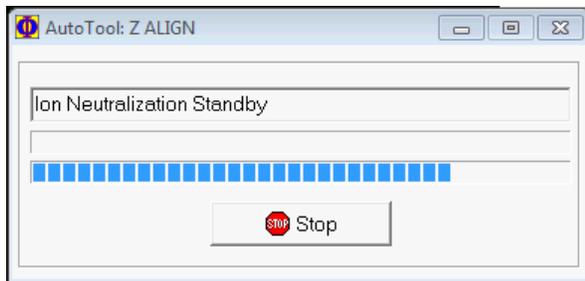**Warning:** this step is **critical**, or the sample will stay at **~ 5 mm Z height** instead of aligned **Z height** at **~ 17 mm**
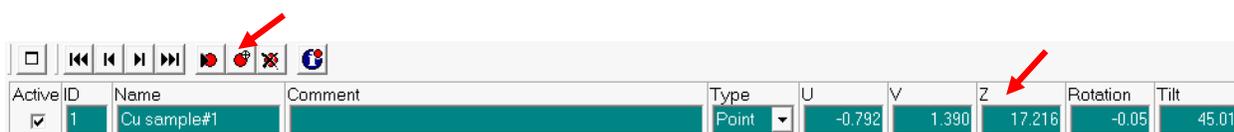
2) Click **XPS** tab on top tab menu to enter **XPS** analysis window and click **Spectrum** tab on the right side. In Source window, choose **FXS** (Focused X-ray Source) and in S**ource Setting** window choose **200u50W15kV** (X-ray beam size: 200 µm, power: 50 W, and e-beam energy: 15 kV)
Note: typically choose **200u50W15kV** or smaller power X-ray beam **100u25W15kV** if X-ray induced surface damage is a concern. **DO NOT** choose smaller power starting with smaller beam size of **50um** or less which are for **X-ray imaging** or **micro analysis** only. The **spectral S/N** is **very low** with **small X-ray power** even after hours of scans.
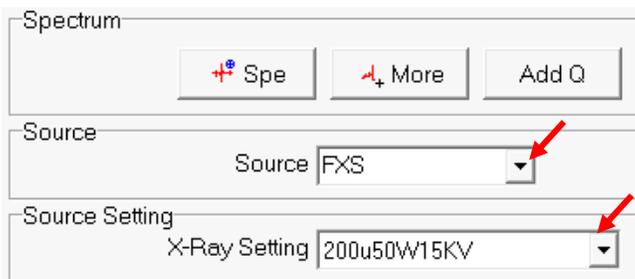
3) Click on top left of **Regions** window to enlarge the **XPS Regions (Spectrum)** window:

4) Click on button on the XPS Regions window to open the Periodic Table, and click on on the table to start a Spectral Survey (**Su**) Scan, then close the table.

5) In **XPS Regions** window, click `Pass` to change **Pass Energy** to **typically 187.850** eV, and `eV Step` to **typically 0.8000** eV. Close the window.

Notice: for **Su**rvey scan, typically **highest Pass Energy** should be chosen to increase **signal sensitivity** and **larger Step** size for a quick scan.

6) Hit `Spe` in Spectrum window to **start Survey** scan
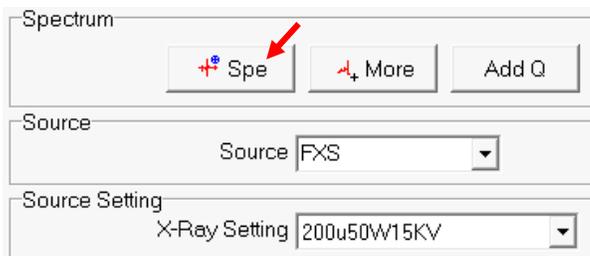
7) **Wait until** the following windows to appear:
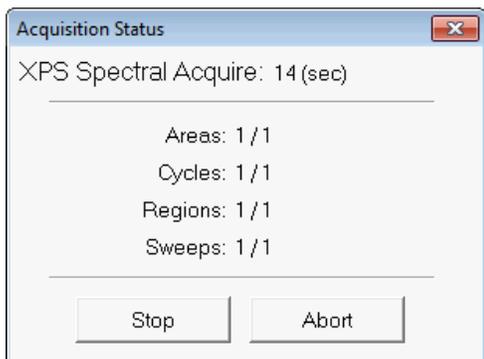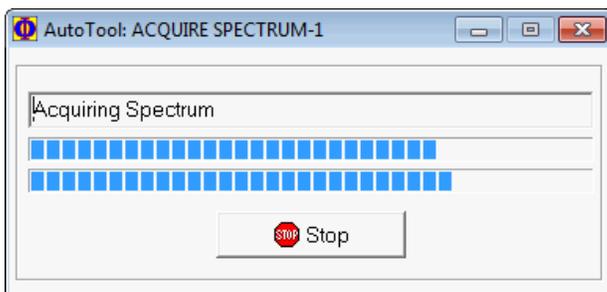
8) The Survey spectrum will appear in the **Spectral Window**.
   Notice:
   > the data **filename** starts with Platen Name "**Standard samples**", the serial number, spectra scan extension "**spe**", and scan type Survey "**Su1s**".
   > the data files have been automatically saved into specified folder which can be opened in **MultiPak** program
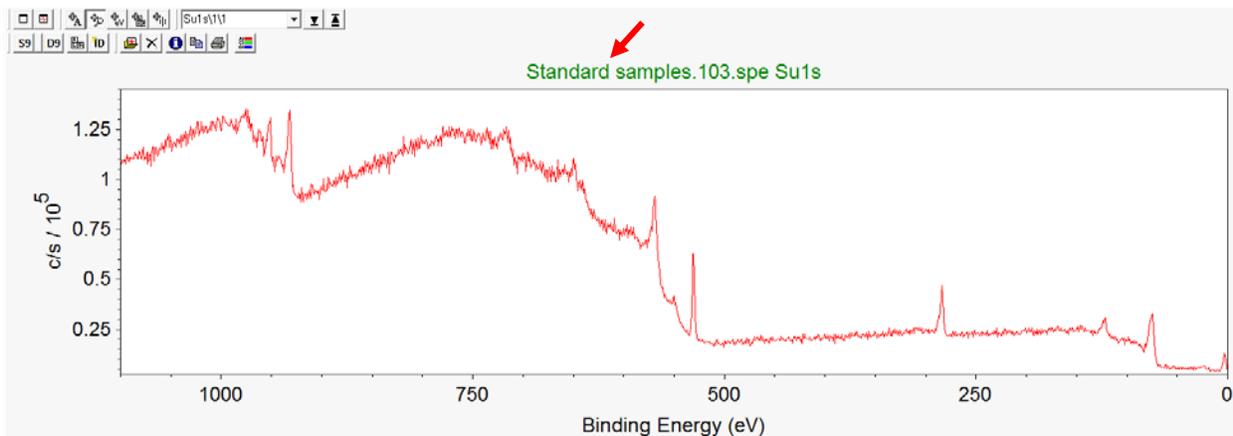
9) Click [ID] above spectrum to start Peak ID
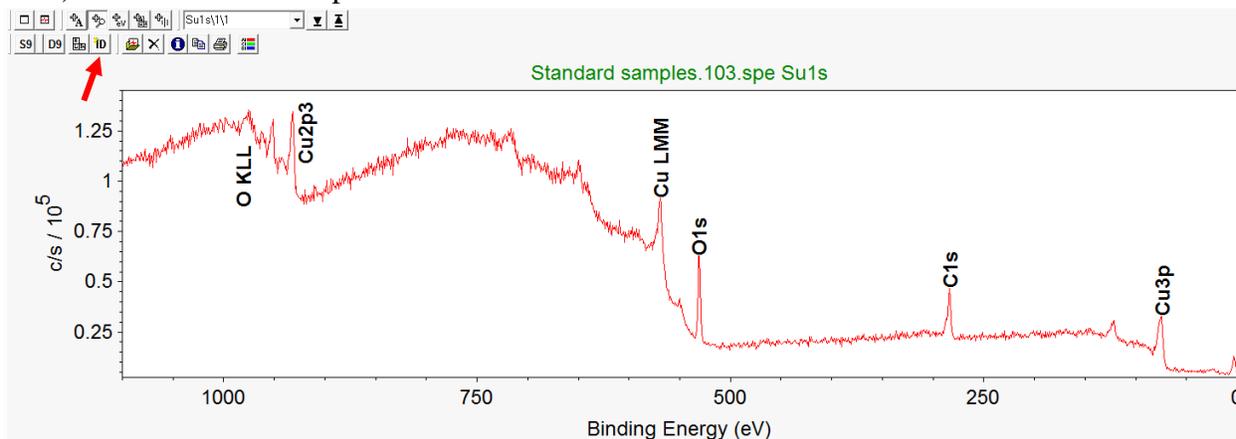
10) Click [🔍] on top left of **Regions** window to enlarge the **XPS Regions (Spectrum)** window, and click [🖼] to open **Import Elements** window

11) Click Import Spectral Display button in the **Import Elements** window and hit **Close**.
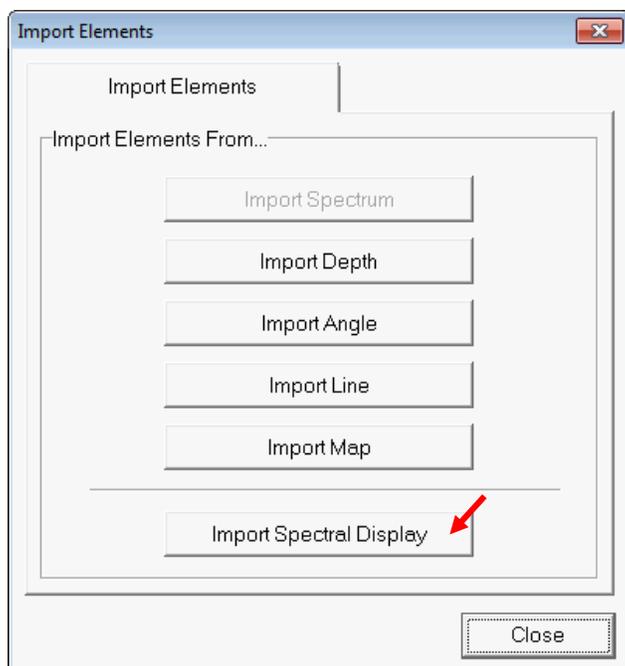
12) The elements detected in **Survey Scan** are imported into the **XPS Regions** table as below. Click `Pass` to change **Pass Energy** to **typically 23.500** eV, and `eV Step` to **typically 0.0500** eV. Close the window.

Notice: for **Region** scan, typically **smaller Pass Energy** (**23.5 – 58.7 eV**) and **smaller Step** size (**0.05 – 0.25 eV**), and **20 ms Time Per Step** should be chosen to increase **spectral energy resolution**.

Warning:

> **Always keep in mind** that both **Signal Sensitivity** and **Energy Resolution** should be considered for **Region Scan**.

> Set **Pass Energy too small** (< **23.5 eV**) will **only lower X-ray intensity/sensitivity** and make spectrum noisy;

> Set **Step** size **too small** (<**0.05 eV**) will **only increase scan time** without too much improvement in energy resolution.

13) Hit `+ Spe` in Spectrum window to **start Region** scan
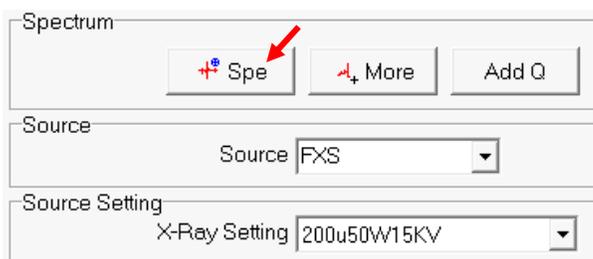
14) **Wait until** the following windows to appear
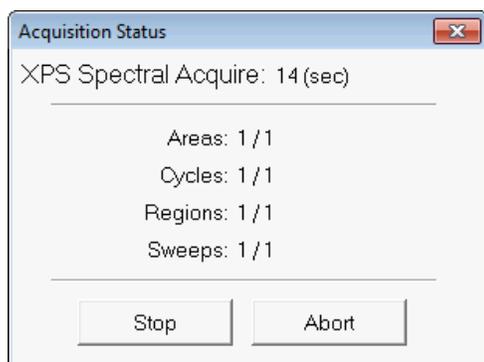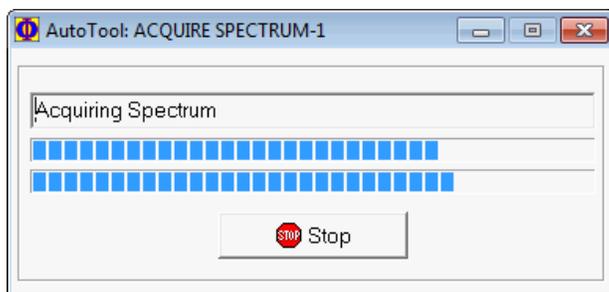
15) The **Region Scan** spectra will appear in the **Spectral Window** following the sequence in **Regions** table.

Notice:

> the data files have been automatically saved into specified folder which can be opened in **MultiPak** program.

> The **Sweep** times can be increased if the spectrum is noisy.

> The **Lower** energy and **Range** can be adjusted to scan the interested regions only.

> **Pass Energy** and **Step** size setup:
- Good resolution: **Pass Energy**: **23.5** - **58.7 eV**; **Step Size: 0.125** to **0.25 eV/step; 20 ms Time Per Step**
- Superior resolution: **Pass Energy 11.75 eV** or lower. **Step Size: 0.05 eV/step** or lower; **20 ms Time Per Step**

> **Sweep** & **Cycle** suggestions:
- Sweep only to individual created elements or regions
- Cycle to all created regions
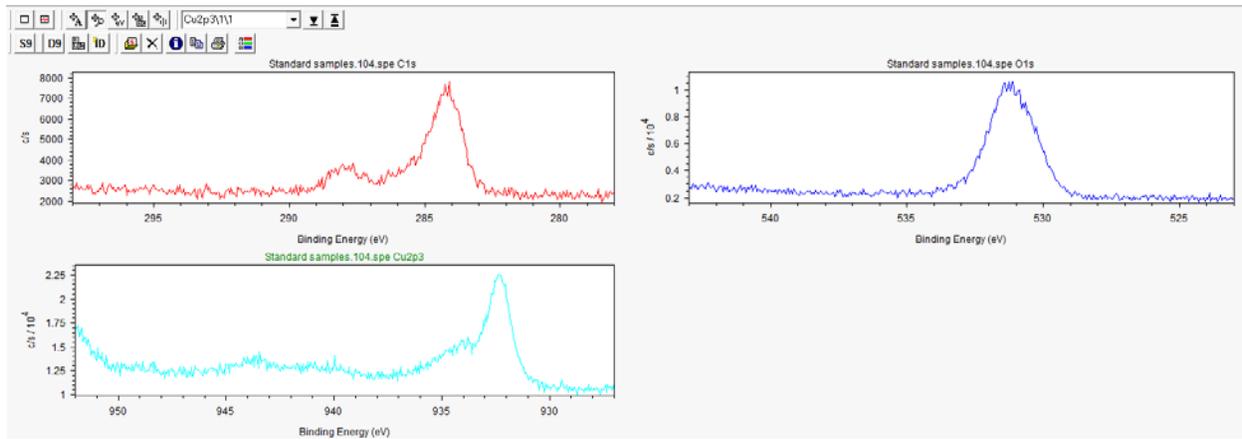
7   Closing XPS Measurement

1) Checklist before Sample Extraction:
    >   Make sure **UV source** if used is turned off correctly

2) Click <mark>Pump V3</mark> button on the **Transfer Sample** window, **wait until** the **Intro** pressure reaches below **4.0E-004 Pa** on Cold Cathode gauge.
    Warning: Sample transfer at high Intro Chamber pressure will introduce **serious contaminations** to **Electron Energy Analyzer**, **X-ray**, **UV sources** and sample stages.
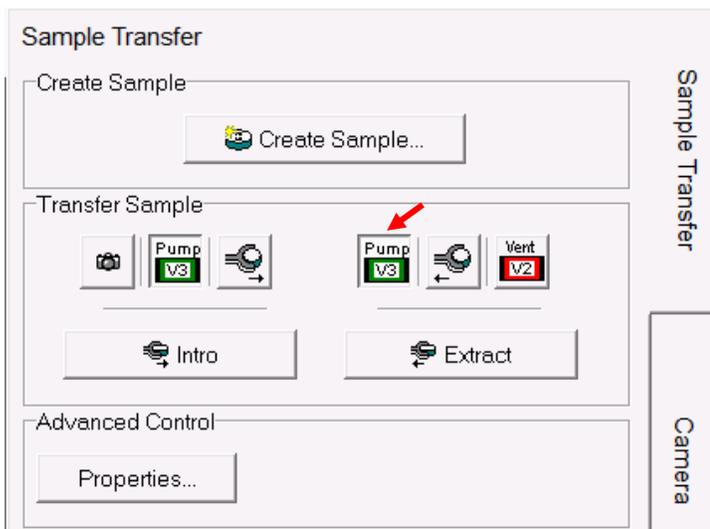
3) **Make sure** that the Intro Chamber pressure reaches below **4.0E-004 Pa** and then click   to start **Sample Extraction**

4) Wait for the following **three windows** popped up in sequence:

5) **DO NOT** click **OK** when the last **Task Information** window above pops up.
    a) Follows the instruction in **Task Information** window to move **FIRST** the transfer arm into the transfer position in **Main Chamber** and **THEN** click **OK.**
    b) The transfer positon means that the transfer arm forks slide smoothly into the round groves on sample holder and the transfer arm magnet touches the **sensor** on the front stopper ring on transfer arm.

Notice: slight adjustment maybe needed to insert sample holder into the stage forks.

Warning: Clicking **OK** first on above window before the transfer arm is moved into transfer position in Main Chamber will lead to serious mechanical damage to both stage and sample holder. User account will be suspended and repair cost will be charged on PI's account

6) Wait the following **two windows** popped up in sequence:

7) **DO NOT** click **OK** when the last **Task Information** window pops up.
   a) <mark>Follows</mark> the instruction on **Task Information** window above to fully retract the transfer arm back to the **Intro Chamber FIRST** and **THEN** click **OK** to close **Task Information** window.

   Warning: Clicking **OK** first on above window before the transfer arm is retracted will lead to <mark>serious mechanical damage to both stage and sample holder.</mark> User account will be suspended and repair cost will be charged on PI's account.

8) The stage will automatically be moved to the transfer position, the **Intro Gate Valve V1** will automatically be **closed** and the following prompt will appear.

9) Click ![Vent V2] in Sample Transfer window to vent the **Intro Chamber**
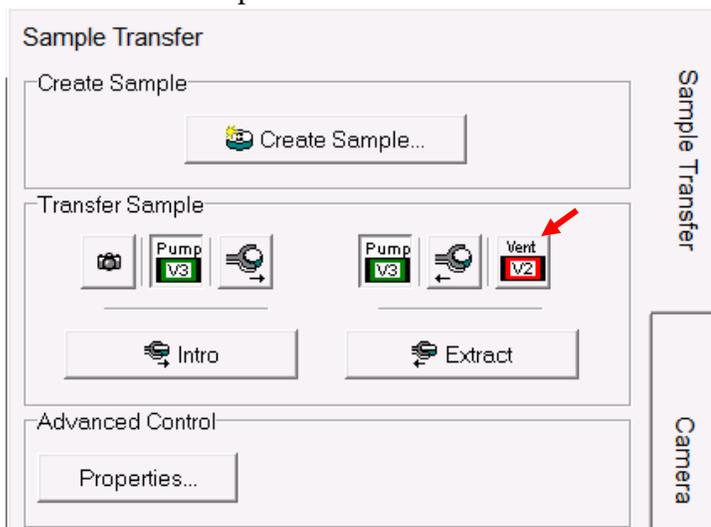
10) **Hold** the Intro Chamber **Viton cover** during venting, remove the sample and put the **Viton cover** back.

11) <mark>If there is no immediate user next, the user needs to **pump** the **Intro Chamber** back to vacuum:</mark>

   Warning: DO NOT leave the **Intro Chamber** in **air,** violation to this step will lead to **user account suspension!**

   a) Click ![Pump V3] button on the **Transfer Sample** window and go back to Intro Chamber quickly to <mark>hold and press the Viton cover against the flange during Intro Chamber pumping</mark>!

   b) **Warning: fail to follow above steps will cause** <mark>the Intro Chamber leaking during pumping</mark>.

   c) Check closely to see the **Intro** pressure drops from ~ **1.6E+003 Pa** to **0.0E+000 Pa** and **Cold Cathode** gauge reads from ~ **2.1E-002 Pa** to **3.0E-002 Pa**

   Warning: DO NOT leave the **XPS** room until **Cold Cathode** gauge reads low **E-002 Pa,** which indicates **no obvious leak** in the **Intro Chamber.**
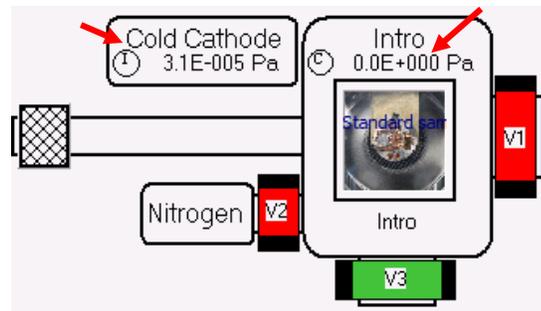
8   XPS Data Reduction

8.1   **Survey** Data Reduction in MultiPak

After collecting the **survey** spectra, open the **MultiPak** software to reduce the data. The **MultiPak** Spectrum window and **Periodic Table** will appear.
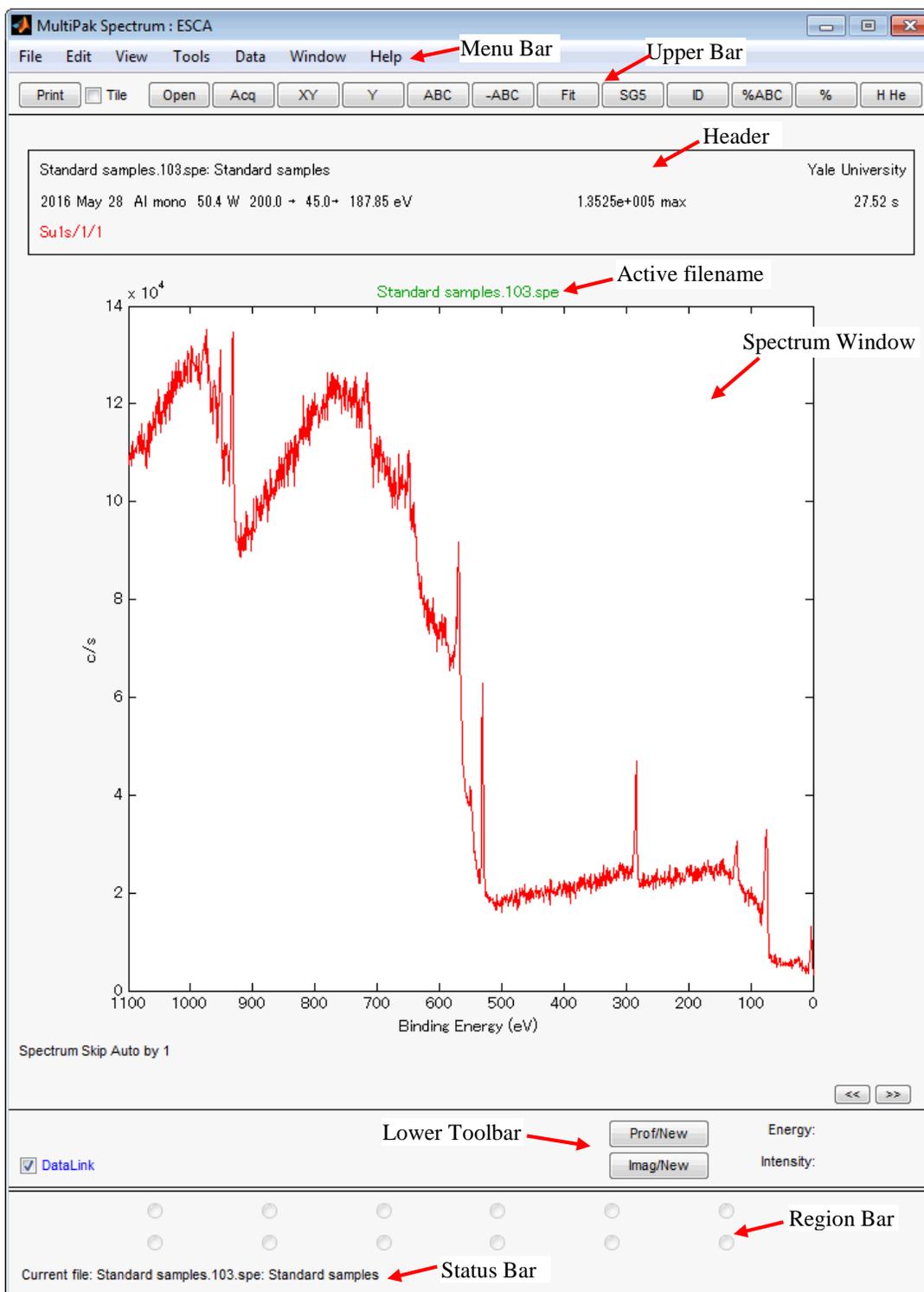
1) Click the Last [Acq] button to open most recently acquired **Survey spectrum** or click the **File** button on the upper tool bar and choose the file from a dropdown menu.

   > The filename is displayed in **green**, indicating that this data is "selected." Any **MultiPak** functions activated will be performed on this data.
   > The header (displayed only when **Header** in the **View** menu is checked) lists the file's data acquisition parameters (date of acquisition, beam voltage of acquisition, etc.).
   > The third line of the header will be updated with a processing history as data reduction routines are applied to the data.

2) Click the Peak **ID** button to identify and label the peaks in the spectrum. This will:

   > Perform an automatic charge correction based on the position of the **C1s** peak.
   > Search its database to identify and label the peaks.
   > Create a **push button** for each element in the **Region** bar at the bottom of the **Spectrum** window.
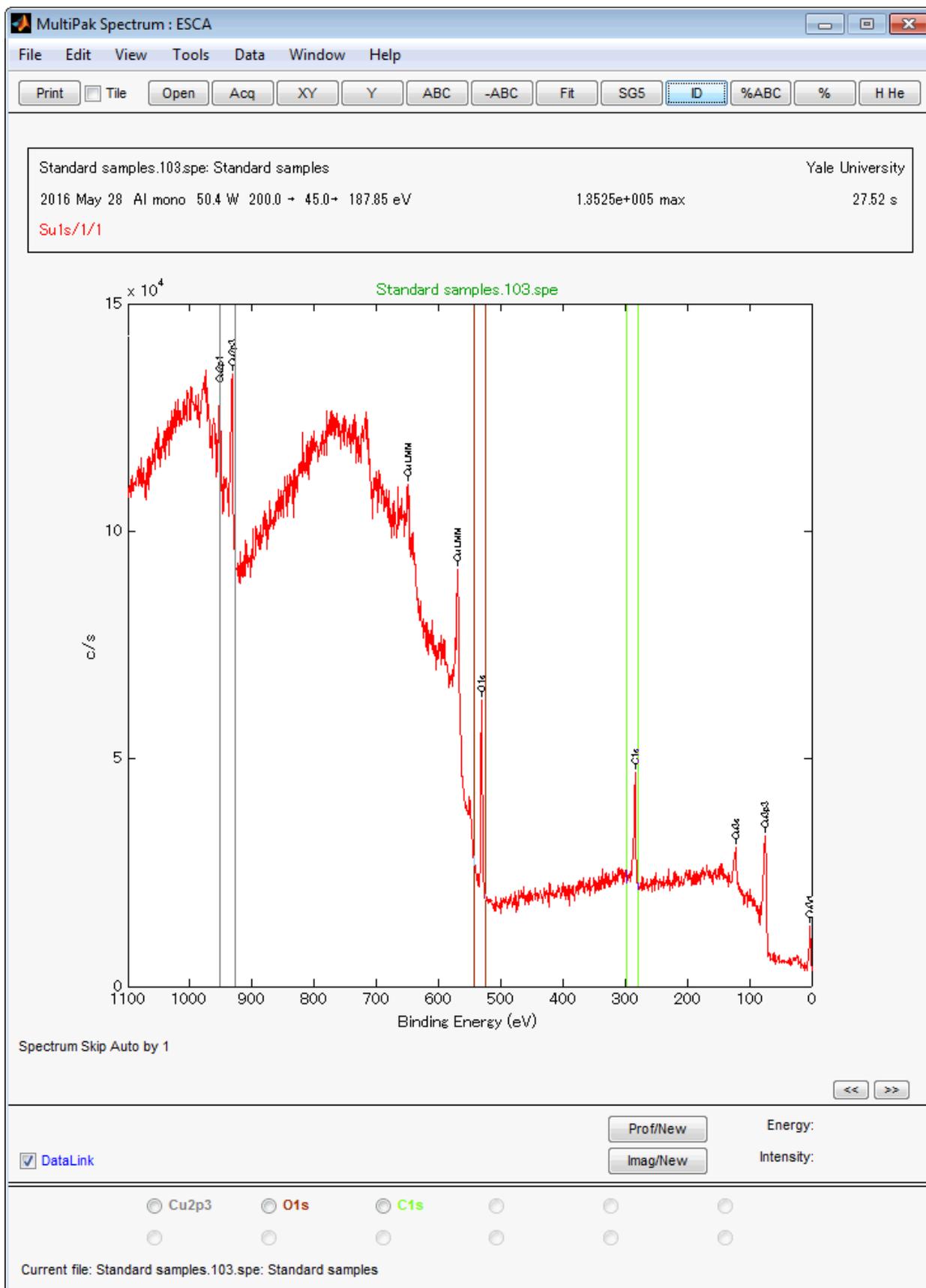
3) **Verify the accuracy** of the labels using your experience and the ***Handbook of X-ray Photoelectron Spectroscopy***. If mistakes were made, you can correct them with one or more of the software tools available.

> To remove a label, click on it and drag it outside the window using the left mouse button or turn **OFF** the element in the **MultiPak Periodic Table** by toggling it off with the left mouse button.

> To label a peak or element that was missed by the **Auto PeakID** routine, you can turn that element **ON** in the **Periodic Table** by clicking the button for it. This method will also create a pushbutton at the bottom of the Spectrum window for that new element.

> If you do not know the identity of an unlabeled peak, you can manually search through the database of labels.

> > Using the middle or right mouse button, click on an open area of the spectrum. This will display a cursor and list ESCA transitions that occur at the current cursor location.

> > While still holding down the mouse button, you can drag the cursor and search around the area of the unknown peak to see what elements occur in that energy range.
> > **Note**: If you use the right mouse button for this operation, releasing the mouse button places the current label on the display at the location of the cursor.
> > **Note**: If using the middle mouse button, when you release the mouse button, no labeling will take place.
> > **Note**: When labeling peaks in this manner, no buttons for the associated elements will be created at the bottom of the window. If you want to add buttons for elements that were not identified by PeakID, you must create them by selecting the element from the Periodic Table.

4) Quantify Data in MultiPak.
Quantification of ESCA spectra is most accurate when **Peak Area** measurements are used. In most situations, it is recommended that you use a **Shirley Background** Subtraction for making the area measurement.

> To view or change the end points that will be used to define the background subtraction for a specific elemental peak, click the button in the **Region Bar** at the bottom of the **Spectrum** window for that element and the software will automatically expand the region for that element's default peak and display the cursors that are used for background subtraction. If it is necessary to adjust the cursors, click and drag the cursor using the left mouse button. While the cursor is being moved, a line depicting the background will be displayed to assist you in positioning the cursor.

## 8.2   **Multiplex (Region Scan)** Data Reduction in MultiPak

After collecting the **Multiplex** spectra, open the **MultiPak** software to reduce the data. The **MultiPak** Spectrum window and **Periodic Table** will appear. (For detailed information about the use of MultiPak, refer to the MultiPak Software Manual.)

1) Click the ⬚Acq⬚ button (which will open the file most recently acquired) or click the **File** button on the upper tool bar to open the file of choice from a **File** selection menu.

   > Initially, all the individual spectra will be displayed on one energy axis, and there will be a button for each element at the bottom of the **Spectrum** window (in the **Region** bar).
   > To view the spectrum of an individual element, click the button that represents the element.
   > The filename is displayed in green, indicating that this data is "selected." Any MultiPak functions activated will be performed on this data.
   > The header (displayed only when Header in the View menu is checked) lists the file's data acquisition parameters (date of acquisition, beam voltage of acquisition, etc.).
   > The third line of the header will be updated with a processing history as data reduction routines are applied to the data.

2) **Quantify Data in MultiPak**.
   Quantification of ESCA spectra is most accurate when Peak Area measurements are used. In most situations, it is recommended that you use a Shirley Background Subtraction for making the area measurement.

   > To view or change the end points that will be used to define the background subtraction for a specific elemental peak, click the button in **Region** bar at the bottom of the Spectrum window for that element and the software will automatically expand the region for that element's default peak and display the cursors that are used for background subtraction. If it is necessary to adjust the cursors, click and drag the cursor using the left mouse button. While the cursor is being moved, a line depicting the background will be displayed to assist you in positioning the cursor.
   > Click the ⬚%⬚ button to view the results of the atomic concentration calculation.
   > To print the **AC Table**, open the **File** pull-down list on the **AC Summary** window and click **Print**

3) **Chemical Information in MultiPak**.
   If the focus of the spectrum is to examine the peak shape and position of the spectra to obtain chemical shift information, there are several software tools in **MultiPak** to help you with that task. Before reducing the data in a spectrum, a smoothing routine may be applied to the data set to improve the accuracy of peak position and peak area measurements. A **Savitzky-Golay Smooth** routine is recommended because it retains the original shape of the data curve during the smoothing process. Depending on the size of the data steps in your data and the width of the peaks, a 5-, 7-, or 9- point smoothing

operation is typically used. To set up the preprogrammed **Smooth** button, which is displayed on the upper tool bar, click **Data>Smooth/DerivativeSetup**. The **Smooth/Derivative** setup options will be displayed below the spectrum. Using the left mouse button, select the **Savitzky-Golay Smooth** and the desired number of points to be used for smoothing. If you selected nine points, the Smooth button in the upper tool bar will read "SG9." The smoothing functions operate on the displayed data. If all of the data regions are displayed, all of the regions will be smoothed. If you click the Smooth button a second time, the data will be smoothed twice.

4) **Energy Scale Calibration in MultiPak**.
The first step in extracting chemical information from a peak is to correct the energy scale for any shifts that are due to charging. Typically, this is done by looking at the position of the **C1s** peak from the hydrocarbon that is present on the surface of most samples. If the **C1s** peak is not at approximately **284.8 eV**, it should be adjusted using the **Shift** routine.

> To open the **Shift** routine, select **Shift Setup** from the **Data** pull-down list.
> When a **green** cursor appears on the screen, position it with the left mouse button on the center of the hydrocarbon portion of the **C1s** peak.
Note: When you release the mouse button, the C1s will shift to 284.8eV (or the position defined for C1s in your **MultiPak Periodic Table** database).
Note: If no carbon is present on the sample, a peak from a matrix element with a known chemistry and binding energy may be used in place of the C1s to correct the energy scale for charge-induced shifts.
Note: The energy can be read by positioning the cursor on the peak and reading the value on the right side of the lower Spectral Shift tool bar.

5) **Binding Energy Identification in MultiPak**.
The next step in extracting chemical information is to determine the binding energy of the peaks for each element. This can be done in several ways.

> With the peak displayed in the **Spectrum** window, position the cursor at the center of the peak, using the middle or right mouse button. The location of the cursor is displayed in terms of the binding energy and intensity at the right side of the lower tool bar in the Spectrum window.
> The **FWHM/Area Tool** may also be used to determine the binding energy of a peak.
>> With the peak displayed in the **Spectrum** window, select the **FWHM/Area** function from the **Tool** pull-down list.
>> The **FWHM/Area Tool** routine will automatically search for the most intense peak in the displayed data and display a list of information that describes the peak including its binding energy. A **blue** cursor is displayed where the software has determined the location of the peak to be. If you want to change this position, grab the cursor using the left mouse button and drag it to the desired location. When you have finished moving the cursor, the displayed information will be updated.

6) **Curve Fit in MultiPak**.

If the peak of interest contains more than one chemical state, it may be necessary to use the **Curve Fit** routine to determine the binding energy of each of the chemical states. To perform a curve fit, use the following steps:
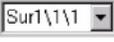
> Make any necessary corrections to the energy scale using the **Spectral Shift** routine.
> Click the Curve **Fit** button in Upper Toolbar.
> Deselect the **Setup** mode and adjust the energy cursors for background subtraction.
> Select a **Gaussian-Lorentzian** peak shape from pop-up **Curve Fit Setup** window. (This peak shape is appropriate for most ESCA peaks.)
> Define initial bands by pointing and clicking at the desired band position with the **right** mouse button. Repeat to add more bands at separate peak positions.
> Readjust the initial band estimates by entering numerical values in the **Curve Fit Setup** menu, or drag the flags attached to the currently selected band, using the left mouse button.
> Click **Fit** to start the fitting routine.
> Evaluate the resulting peak positions as shapes using your experience, reference spectra, and database information.
> Make any necessary adjustments to the band parameters and click **Fit** again.
> When you are satisfied with the results, hit **ViewSumm** button on the **MultiPak Spectrum** window to see the **Summary** information. The fitted curve data can also be exported in **ASCII** by hitting **File** menu in the **Curve Fit Setup** window.
> Click **Exit** to leave the Curve Fit routine.
   Note: The error (in **red**) follows the baseline evenly, indicating a good fit.
   Note: The Curve Fit Summary table contains peak position information that can be used to determine the identity of the chemical state and peak area information to quantify those chemical states. For a more detailed discussion of the Curve Fit software, refer to the **MultiPak Software Manual**.

8.3   **Multiplex** Data Reduction in **SmartSoft**

1) Spectral Display
   > Use the [image] **Zoom** to enlarge a region of interest and the Right-click Context menu; **Fullscreen** to revert back.
   > Select the [image] Show One. Use the [Sur1\1\1] Current Curve to view a single **Region**.
   > Click the [image] **Fullscreen** button for a full screen view of the **Spectral Viewer**.
   > **Note**: With **Profile** or **Line** or **Map** data displayed; use the [image] **Fullscreen Data/Tile** button to enlarge the highlighted file's **Spectral** or **Profile** Viewer.

2) Annotate the Spectral Viewer
   > Double-click within the viewer and type in text to annotate. Click outside the text box to set the text.
   > To move the text, click and drag the text to the desired location.
   > To edit text, double-click over the text box, then edit. Right-click over the text for more options (cut, copy, paste, delete, etc.).
   > Right-click over the viewer for additional options, including delete all annotation. Click the **Properties** option to open the **Graph Annotation Properties** box. Use this box to select the text font, size, color, etc.

3) Data Reduction
   > Use the [S9] **9 Point Savitsky-Golay Smooth** to smooth the data.
   > Use the [ID] Peak Identification to identify peaks
   > Use the [image] Energy Cursor to identify peaks
   > Use the [image] Element Cursor to identify peaks

4) Output the Data
   > Click the [image] button to **Copy** the selected viewer to the clipboard. This allows the data to be imported as a graphic into any Microsoft Office application (Word, PowerPoint, etc.).
   > Click the button to [image] **Print** the selected viewer to the default printer. **NOTE**: Annotation changes made to the viewer are not saved as part of the data file.

9   Some Analysis Concepts

- **High X-ray Power ➔ High sensitivity** but **poor spatial resolution** (beam size is **bad**)
- **Low X-ray Power ➔ Low sensitivity** but **good spatial resolution** (beam size is **good**)
- **High Pass energy ➔ High sensitivity** but **poor energy resolution** (peak is **FAT**)
- **Low Pass energy ➔ Low sensitivity** but **good energy resolution** (peak is **THIN**)
- **Big Step size ➔ Acquire time** is **short** but **peak shape** (resolution) is **bad**
- **Small Step size ➔ Acquire time** is **long** but **peak shape** (resolution) is **good**
- **High sensitivity ➔ Signal-to-Noise** is **better** so total **acquire time** can be **shorter**
- **Low sensitivity ➔ Signal-to-Noise** is **worst** so total acquire time needs to be **longer**
- **Sensitivity = Signal Intensity** or **counts**

10 Checklist after Experiment

1) **Sign out** on the logbook and report any problems on instrument.
2) Leave SmartSoft and Watcher programs **ON**.
3) Do **NOT plug personal USB flash drive** into the instrument computer for data transfer. Copy the data to the **Core USB flash drive** and use the workstation outside to transfer data.
4) **Turn OFF computer monitor** and the **chamber light**.
5) **Remove** sample from holder and **clean** it with clean wipes and **IPA**.
6) **Store** the specimen holder, tongs and other tools in the tool box.

11 Vacuum Loss Emergency Operation Procedure

Vacuum Loss:

Case 1: In **Main Chamber**, opened the UPS leak valve **too quickly** leading to pressure jump to low **10^{-3} Pa**.

Steps to follow:

1) **Close** the UPS leak valve quickly and watch the pressure to drop back to **5×10^{-7} Pa**
2) **Contact Core stuff** to initialize the stage if error message appears during stage movement
3) **Leave note** on the **logbook**

Case 2: In **Main Chamber**, when finishing UPS scan, closed **UPS 1** and **2** valves **first** and **left UPS leak valve open**, leading to main chamber pressure drop to low **10^{-3} Pa**.

Steps to follow:

1) **Close** the UPS leak valve quickly and watch the pressure to drop back to **5×10^{-7} Pa**
2) **Contact Core stuff** to initialize the stage if error message appears during stage movement
3) **Leave note** on the **logbook**

Case 3: In **Intro Chamber**, the **cover** was **NOT** placed properly, leading to extra pumping time (> 10 minutes) for first stage rotary pump and failed for second stage pumping by turbo pump pressure staying ~**10^{+1} Pa** longer than 10 minutes.

which then either turns off the ion gauge and/or the ion pumps, but the Main Chamber pressure is still likely to be less than 1×10^{-2} Pa.

Steps to follow:

1) **Readjust** the cover by pressing and slight turning and **check** if the pressure drops quickly, if **NOT**
2) **Vent** the intro chamber in **SmartSoft** and take the cover off. **Check** inner side gasket for any dents, particles, clean with wipes and IPO. Put the cover back, **make sure** the cover is **placed properly** and start pumping. If the pressure is still not dropping normally, **contact Core stuff**
3) **Leave note** on the **logbook**