# Standard Operating Procedure I

## PHI 5000 *VersaProbe* II XPS/UPS

### (Basic XPS Measurement)

Yale West Campus
Materials Characterization Core
*ywcmatsci.yale.edu*

ESC II, Room E119E
810 West Campus Drive
West Haven, CT 06516

**Version 1.1, May 2017**

> 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 either uploaded to Yale Box, or copied to the Jump Drive provided by the Core.
> **NEVER** surf the web on the XPS computer to minimize the risk of the computer being hacked
> Yale West Campus MCC users should acknowledge MCC in their publications. Please check the following link for details: http://ywcmatsci.yale.edu/publications
> The core reserves the right to use the data for core promotion

# Table of Contents

# PHI 5000 *VersaProbe* II XPS/UP S Standard Operating Procedure (Basic XPS Measurement)

## 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
   810 West Campus Dr
   West Haven, CT 06516

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.

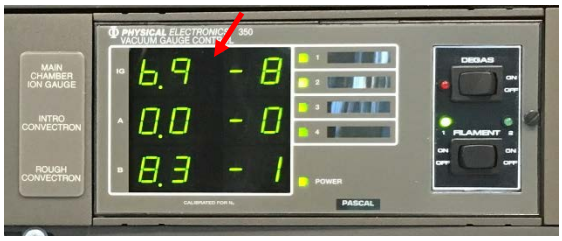
**Notice**: Please **follow** strictly the **SOP** to keep the facility under good condition. We **DO NOT** recommend user explorations on program unless endorsed by core staff.

## 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 $8\times10^{-6}$ 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

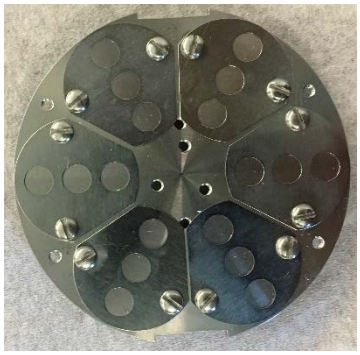
**Warnings:**

- Always wear **lab coat** and **gloves** **for vacuum sample preparation!**
- The sample for XPS and UPS needs to be **completely dried**! **If the pumping time in intro chamber exceeds 1 hour, the sample must be taken out for further treatment.**

### 3.1 Sample holders

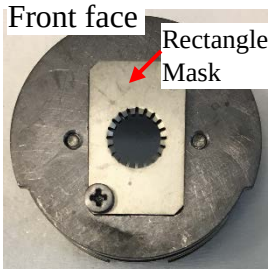
1) **1 inch** regular sample holder for **as-is** sample analysis. This is most used sample holder which can hold more **15** samples (3 x 3 $mm^2$)


2) **60 mm** large sample holder for **as-is** sample analysis. This holder is very useful to hold **large samples** and large number of small samples.

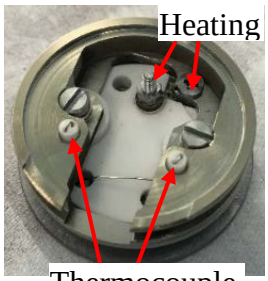


3) **Angle Resolved** (**AR) XPS/UPS** using **AR** sample holder.
**Note**: users should choose **AR** holder for **UPS** measurements

4) **1 inch Heating Sample Holder** with **heater** if *in situ* heating is required
**Temperature range**: **RT – 800 °C**



Rectangle Heater



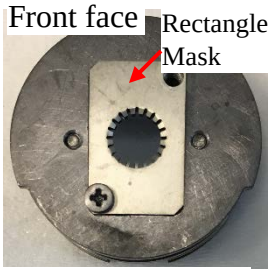
Thermocouple

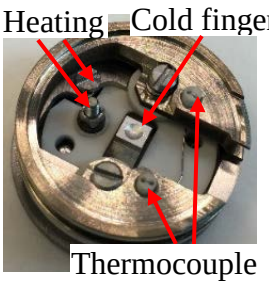
**Warning:**

> Use the **rectangle mask** only with **flat cross head** screws**.**
> Choose **the right size cross screwdriver** for sample mounting. The screws can be easily damaged with **wrong** size screwdrivers. Users are not allowed to fix sample holder, report to lab staff immediately**.**

> Choose the right type of holder in **HC Control** program and keep **Reading Heater Power** below **75%**. **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.

5) **1 inch Heating/Cooling Sample Holder** if *in situ* cooling and heating required
**Temperature range**: **-140 ºC – 600 ºC**




## 3.2 Sample Mounting

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


**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) (for **XPS/UPS**). (**sample cannot be heated in vacuum**)

   **Warning**: after sample mounting, **shake** and **tilt** sample holder to make sure no **loose** sample or particles on 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 (3x3 $mm^2$ ~ 5x5 $mm^2$)
      > **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

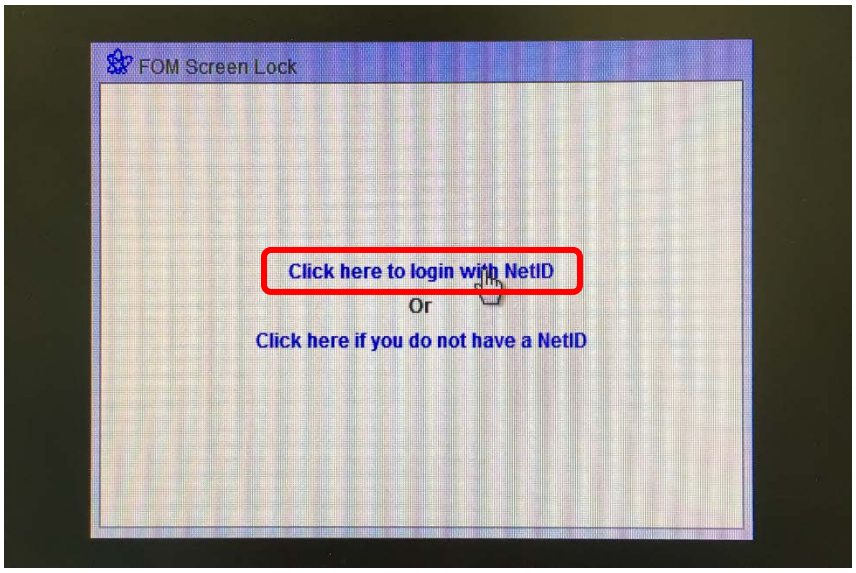
**Warning**: carefully remove all loose particles from the sample holder:

> **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 Activate FOM Program

> In the FOM Scree Lock window, click on **Click here to login with NetID** to unlock the screen.

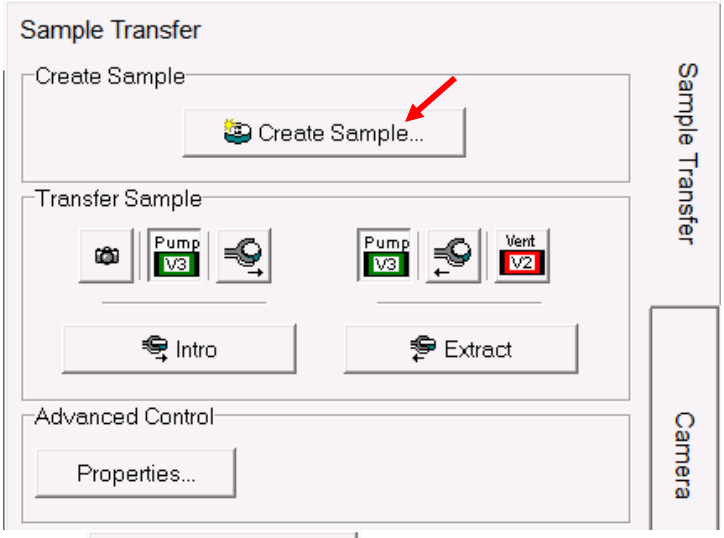



# 5 Create Sample in SmartSoft Program

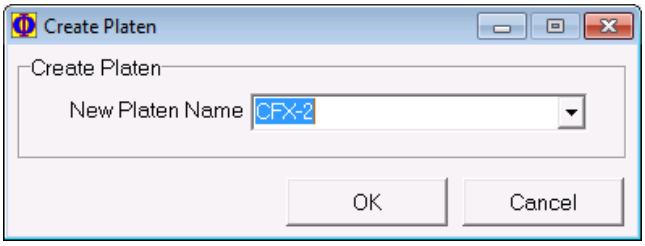
**Notice**: "**Create Sample"** is the first step of data analysis. **It must be done before taking picture of the sample in Intro Chamber**. The following steps should be taken to create sample (platen):

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

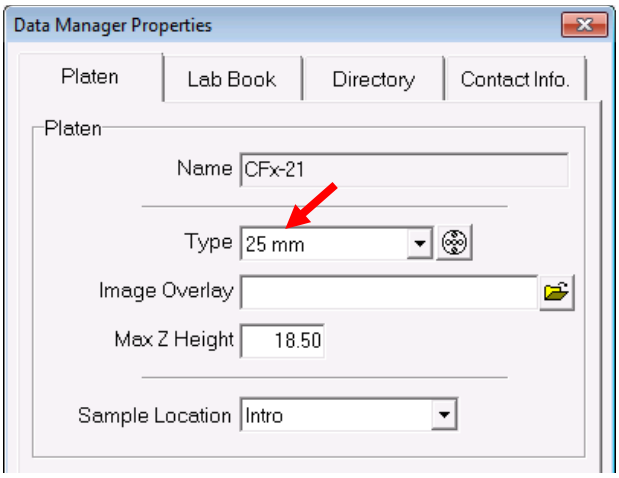
2) Click System to enter the **System** window, then click the **Sample Transfer** on the right side to enter **Sample Transfer** tab.



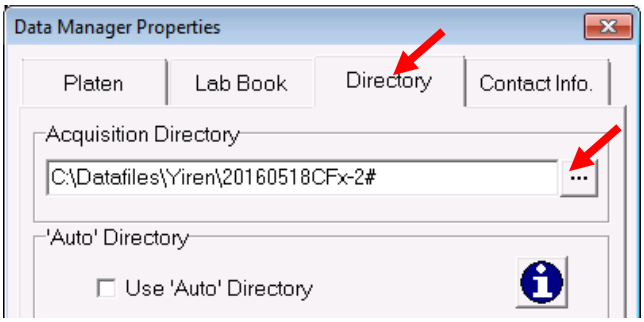

3) Click Create Sample... button to open **Create Platen** (sample) window
**Warning**: once a **New Platen Name** is created, **NEVER** change or **CREATE** a new platen name once the data collection has started. **Violations may lead to hardware damages** inside vacuum as all sampling positions are 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 if the **1 inch** regular sample holder is used. **Note**: choose the corresponding sample holder if **Angle Resolved (AR)** or **60 mm** sample holders is used.




5) Click **Directory** and specify the data **Acquisition Directory**

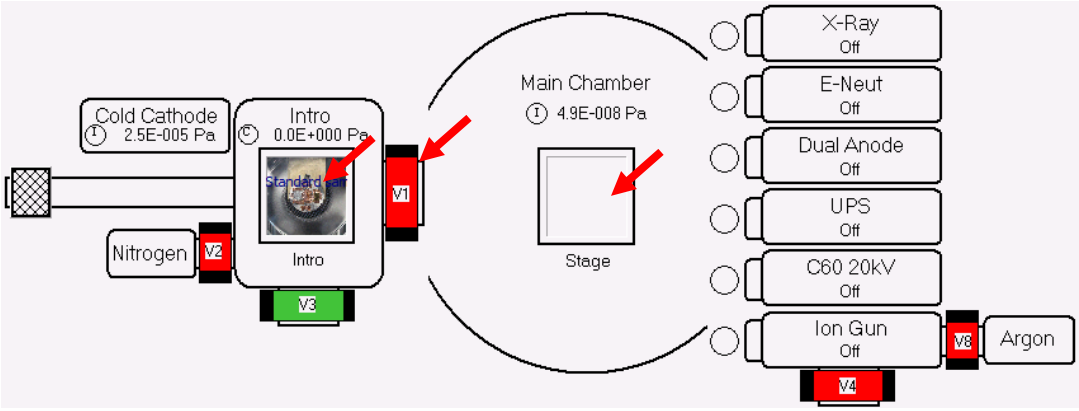



6) Hit **OK** and **Close** above window.

## 6 Sample Loading into Intro Chamber

1) The sample holder with either **solid** or **powder** sample must be **blown with nitrogen gas** to make sure **NO loose particles, hair or fibers on the holder**.
2) Check the system status before sample loading:
   a) 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 immediately if not.

b) In system diagram below, check if "**V1**" valve is **closed** (in **red** border) between **Intro** and **Main Chambers**. If not, **contact** lab manager immediately.




c) Turn **ON** the Main Chamber light.

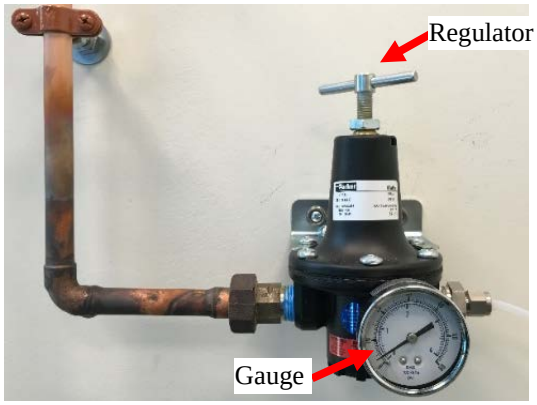

d) Confirm **no sample holder** inside **Main Chamber**:

> Look through the **view port** to confirm no samples in the **Main Chamber**,

> Check the **system diagram** in **SmartSoft** window above and make sure that the **blank square box** is shown in on the stage in **Main Chamber**. **Report** to lab manager immediately if not.

**Note**: User should remove and clean the previous sample holder left in the **Intro Chamber**. User can leave the sample inside the intro chamber after finish, which will be discarded by next user.

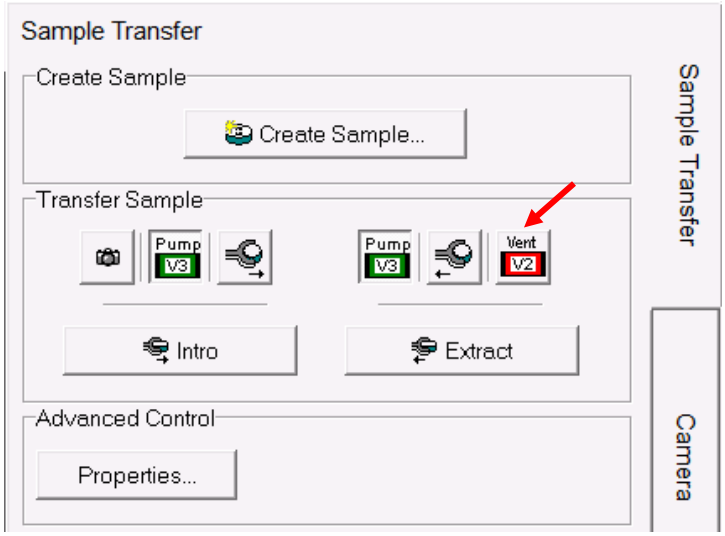
e) Check **building supply $N_2$ pressure** on the wall behind computer monitor and make sure it is set below ~ **3 psi**. If the pressure is above 3 psi, decrease the pressure setting to **3 psi** by turning the pressure regulator valve ***counterclockwise***. **Contact** MCC staff if assistance needed.




3) In the system diagram in Step 2), check if 

(V3) valve is in green indicating the **Intro Chamber** in vacuum, then click 

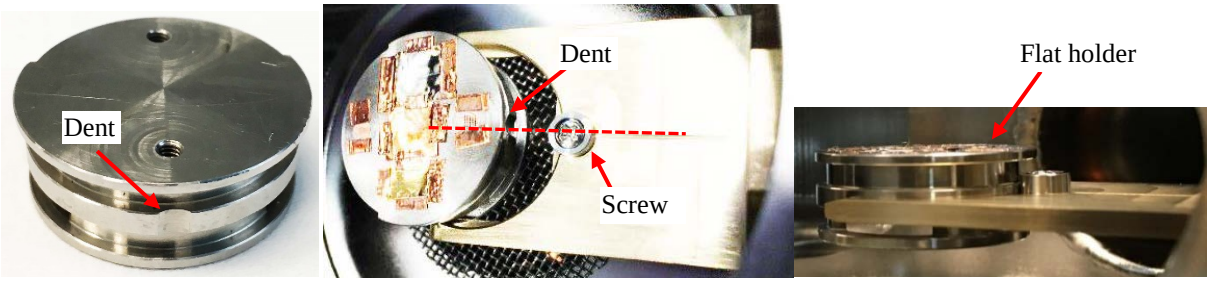
in the **Sample Transfer** window below:
**Warning**: the user must wear **gloves** at hold the glass cover during **venting**.



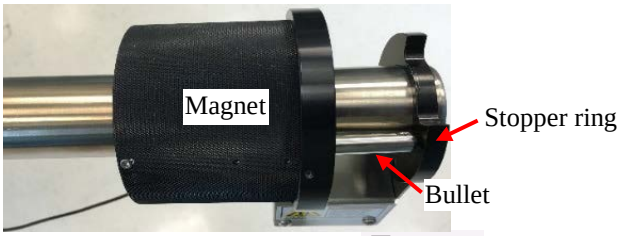

4) **Wear gloves**, wait until the **Intro Chamber** is vented, remove the **Viton cover** and **slide** the **nitrogen cleaned** sample holder along the round **dent** on the bottom of the sample holder onto the transfer arm fork
**Warning:**
> User **CAN NOT** touch the sample loading port with exposed skins
> **DO NOT** touch the camera atop the loading port
> The sample holder must have been cleaned with **nitrogen gas**

> **Violations** will lead to particles or fibers embedded into gate valve causing **chamber leaking**, or sucked into chamber causing severe **pump damage**.

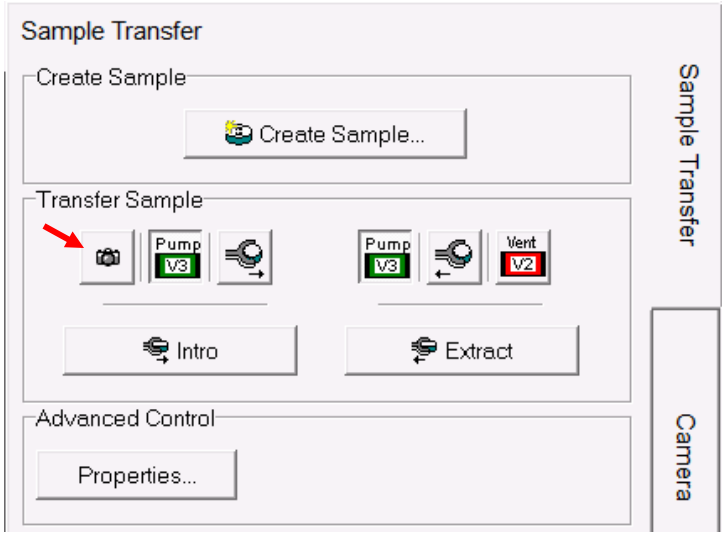
5) Make sure the **dent** on sample **holder aligns** with and **touch** the **screw** on the transfer arm fork and check from side window flange if the holder stays flat on the fork:
**Warning:** Users should follow **STRICTLY** the steps below to avoid transfer failure and parts damage.



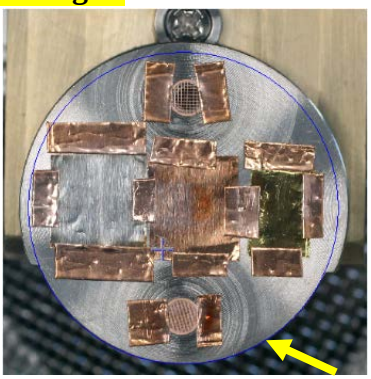

6) Leave the loading port **open**, **fully retract** the transfer arm so that the **bullet** on magnet **touches** the stopper ring on the transfer arm



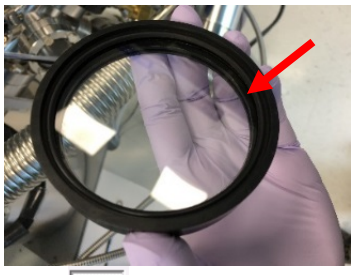

7) In the **SmartSoft** program System window, click button on the **Transfer Sample** window to take a picture of the sample holder:



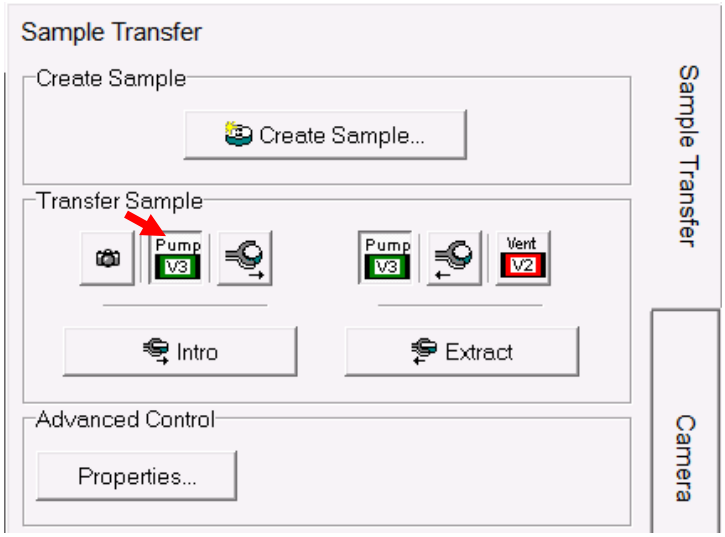
8) **Wait for several seconds** for picture loading into **SmartSoft**, click the Sample tab next to System to enter the **Sample** window. Check the sample holder photo and make sure the **blue circle** aligns well with the **bottom half** of the sample holder. **If not, contact lab manager**.



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



10) Click Pump button on the **Sample Transfer** window and **go back to Intro Chamber** quickly; **hold and press** the **Viton cover** into the **flange groove** during **initial Intro Chamber** pumping!
**Warning:** fail to follow above steps will cause the **Intro Chamber leaking** during pumping.
**Note**: **slight rotating** while **pressing** the **Viton cover** before pumping (before sucking air sound) may help to assure the **Viton cover** placed correctly on the flange.

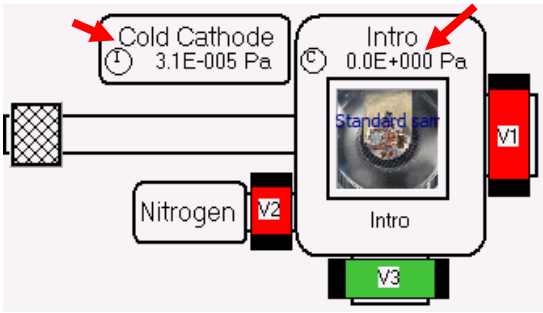



11) Check Intro Chamber **status right after pumping (after hearing sucking air sound)**

a) Click System to enter the **System** window and monitor the **Intro Chamber** pressure:

> The pressure reading from **Stage 1 pressure gauge** (Pirani gauge) should drop from **atmosphere pressure** to ~**3.0E+001 Pa** in ~**5 minutes;**

> Then, the reading from **Stage 2 pressure gauge** (Cold Cathode gauge) takes over and should drop from ~ **1E-002 Pa** to **4.0E-004 Pa** before sample transfer to **Main Chamber** typically in ~**15 minutes**;




**Warning**:

> The actual **Intro Chamber** pumping time will **vary on different samples**

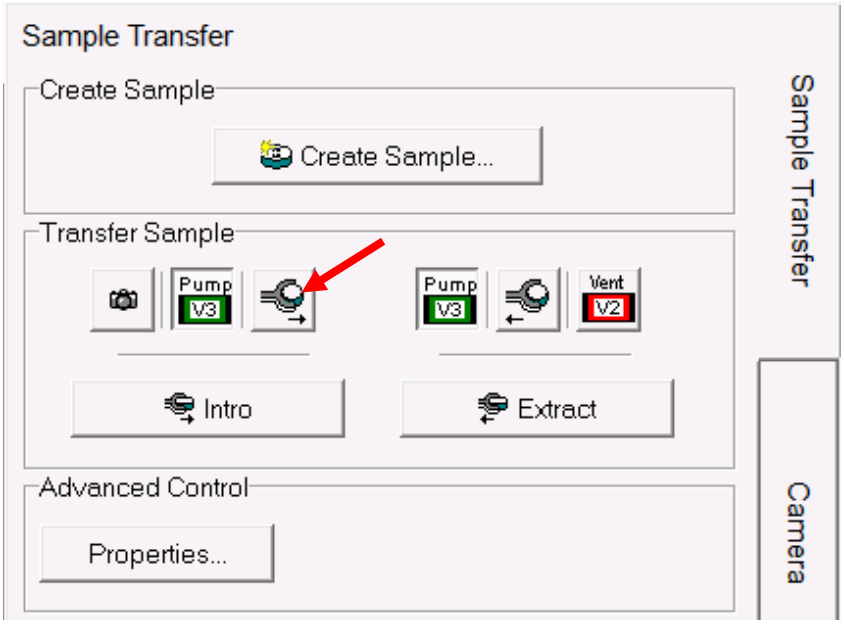
> It is strongly recommended to make sure user samples are **DRY and UHV (Ultra High Vacuum) compatible**. If pumping time **exceeds 60 minutes** to reach **4.0E-004 Pa**, the user must **remove** the samples from **Intro Chamber** for further treatment.

# 7 Sample Transfer into Main Chamber

**Warning:** Users should follow **STRICTLY** the steps below to avoid transfer failure and parts damage.

1) Before sample transfer to **Main Chamber**, the **Intro Chamber** pressure from Cold Cathode gauge **MUST** reach 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 stage**.
2) Make sure that the **Main Chamber** light is turned **ON**.

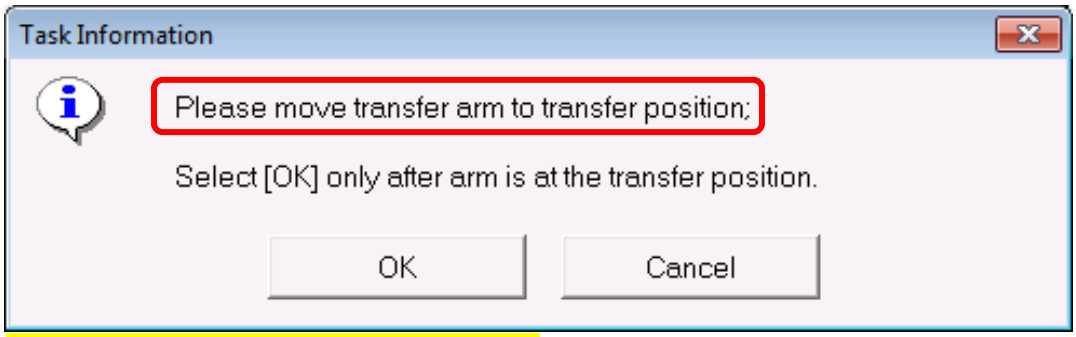

3) Click  in the **Transfer Sample** window to start sample transfer from **Intro Chamber** to **Main Chamber**



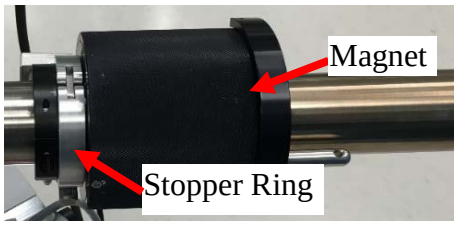
**Warning:**

> **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
> **MAKE SURE** that **magnet TOUCHES** the **stopper ring** on the transfer rod

4) Wait for the following **window** to appear:




5) **Follow the command in above window** to move the transfer arm **forward** and **MAKE SURE** the **magnet TOUCHES** the **stopper ring** and the **stopper ring** on the transfer arm


**Warnings**:

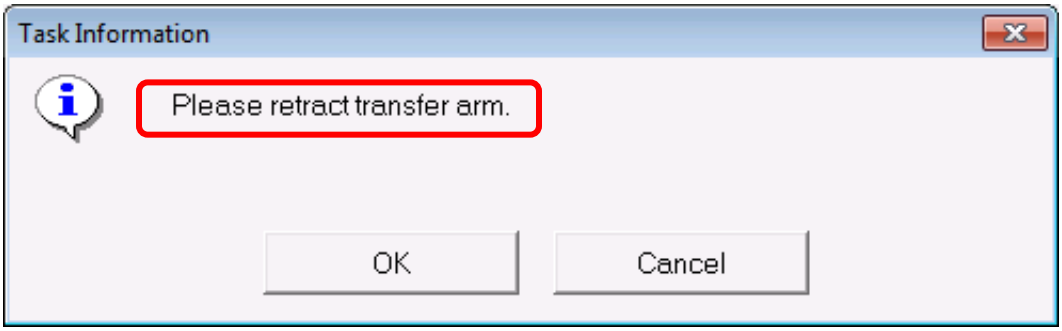
> User should **slow down** when approaching transfer position. The sample holder should be **SMOOTHLY** inserted into the sample stage in Main Chamber. **CONTACT** lab manager immediately if **strong force** felt at this step,
> **Slight adjustment** maybe needed to insert sample holder into the stage forks. The user should **CONTACT** lab manager for help,
> **NEVER force the sample into sample stage**. Expensive parts inside will be **damaged**. User account will be **suspended** and **repair cost** will be charged on PI's account.

6) **Leave** the transfer arm at the **transfer position**; **go back to the computer** and click **OK** on the popup window

**Warning:**

> **NEVER** click **OK** on the window before moving transfer arm forward. Or **serious mechanical damage** to both stage and sample holder will occur. User account will be **suspended** and **repair cost** will be charged on PI's account.

7) Wait the following **window** to appear. **DO NOT** click **OK**




**Warning**:

> Failed to follow above step will lead to **serious part damage to both sample stage and sample holder**. User account will be **suspended** and **repair cost** will be charged on PI's account.

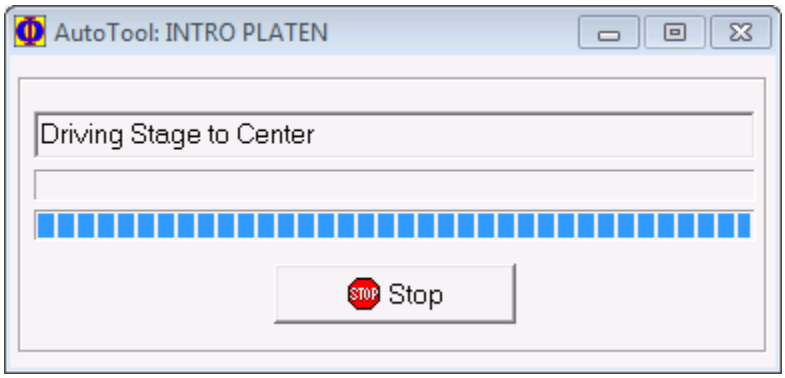
8) **Follows** the command in above window to **fully retract the transfer arm back** to the **Intro Chamber**

**Warning**:

> Retract the transfer arm **VERY SLOWLY** and watch the sample holder through **Main Chamber** view port. The sample holder should **NOT** move during transfer arm retraction.

9) **Go back to the computer** and click **OK** on above window

10) **Wait until** the stage is driven to the center.




11) **Watch closely** the Main Chamber pressure change

a) The pressure should drop continuously from **low E-006 Pa to E-007 Pa** range in a short time, **5 ~ 30 mins** depending on samples

**Warning**:

> To prevent sample **contamination** to Main Chamber:

> If the pressure **rises quickly/slowly above 5E-006 Pa**, **remove** the samples from **Main Chamber** immediately

> If the pressure stays **unchanged** at **low E-006 Pa** for more than **30 mins** also **remove** the samples from **Main Chamber**.

b) Wait until the pressure reaches **5E-007 Pa** before proceeding to **XPS Scan**.
**Warning**:

> To prevent sample **contamination** to Main Chamber:

> If the pressure drops **very slowly** and takes more than **60 mins** to reach **5E-007 Pa**, consider taking the samples out for further treatment.

# 8 XPS Scan

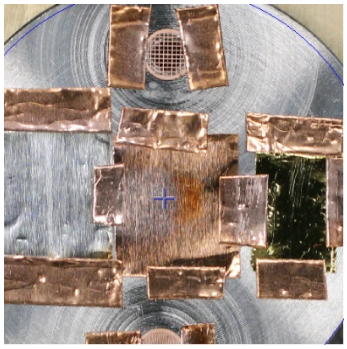
## 8.1 Initial analysis position definition and Z-Alignment

1) Wait until the **Main Chamber** pressure reaches **5E-007 Pa**
2) Define **initial** analysis **Position** with the sample in **Main Chamber**

> Click 

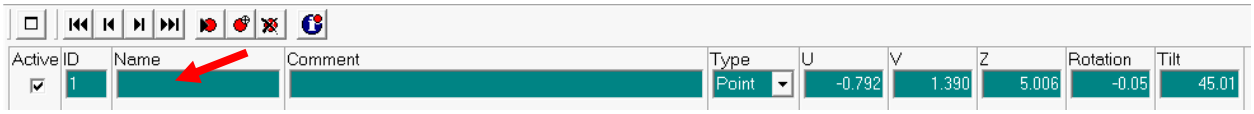
on top tab menu to enter **Sample** window

> **Hover** mouse cursor over the sample photo window on the left, **scroll** mouse wheel to enlarge photo and find the sample to be analyzed

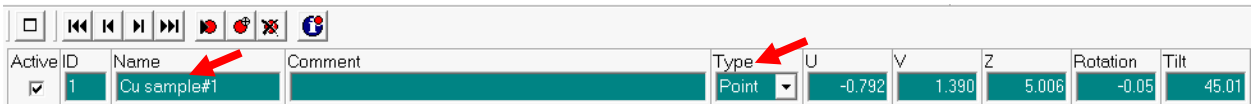


> **Right click** mouse on intended analysis position and select **Drive To Click** on the dropdown menu, the **Main Chamber Stage** will move to the selected position

> **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




> Type **Position Name** in the **Name** window above




> Select **Position Type** as **Point** in above table for regular point X-ray sources: **100 u25W15kV** and **200u50W15kV** in **XPS** window Source Setting in **Step 4**

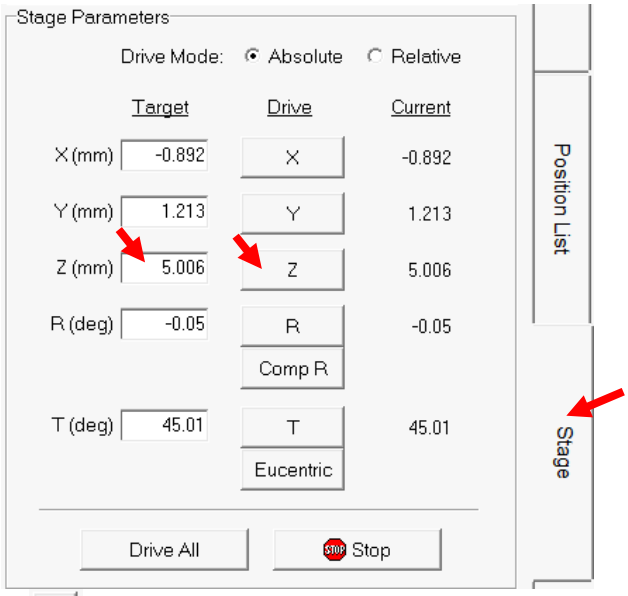
**Note**: Select Position Type as **HP** (High Power) in above table if the High Power X-ray source, **100u100W20kV_HP** is chosen in XPS window Source Setting in **Session 7.3**

3) Sample Z-Alignment

> Click **Stage** tab on the right side of **Sample** window to enter **Stage Parameters** window

> Input **12** in **Z(mm)** space and hit **Enter** on keyboard

> Hit Z button on the right side of input number (**12.000**) and wait for the sample stage to move to entered height (**12.000 mm**)




> Hit button on top of **SXI** image window to start **Z-Align**

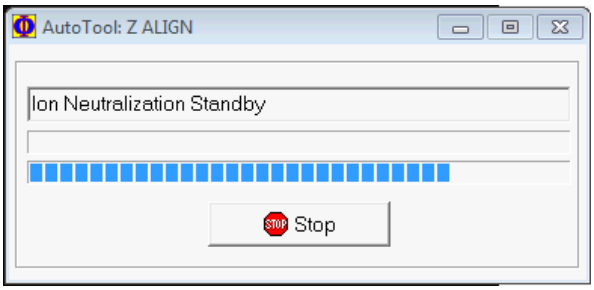

> The following windows will pop up sequentially, **wait until** all windows disappear

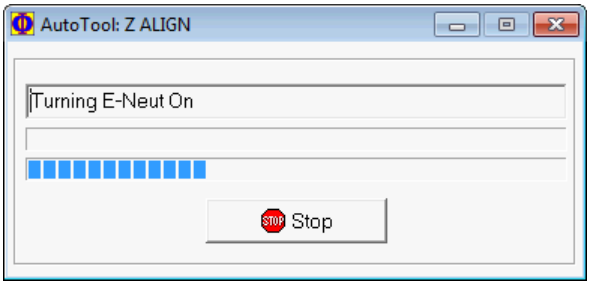
**Note**:

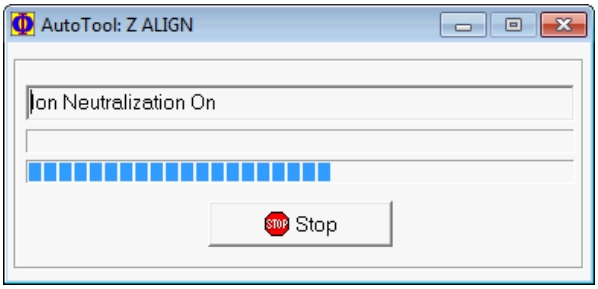
> The **Z-Align** optimizes the sample stage height at the maximal photoelectron counts, so **X-ray Source**, **ENEUT** (electron neutralizer) and **IGun** (Ion gun) will be turned **ON** during **Z-Align**.

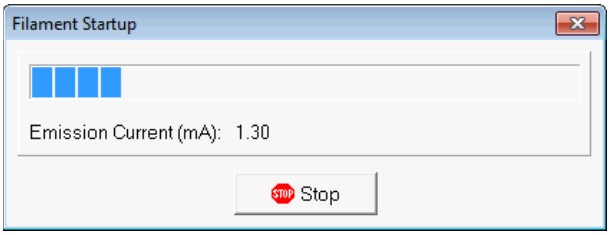
**Warning**:

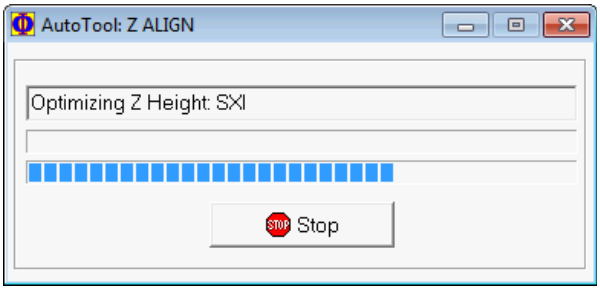
> Once **Z-Align** starts, the **Intro Chamber** pumping will be **stopped**. user needs to **pump** the **Intro Chamber** back to **4.0E-004 Pa** before sample transfer back to **Intro Chamber**



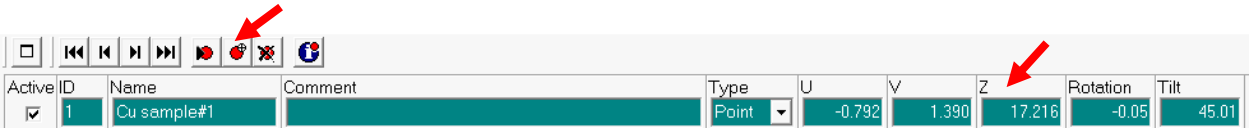




> **Click**  above **Position List Table** to **Update Position** after **Z-Align**
**Warning:** this step is **critical**, or the sample will stay at **~ 5 mm Z height** instead of aligned **Z height** at **~ 17 mm** depending on sample **thickness**
**Warning:**

> **Report lab manager** immediately if **Z-Align** fails. This may due to hardware failure or program bugs.




## 8.2 Create multiple positions on samples

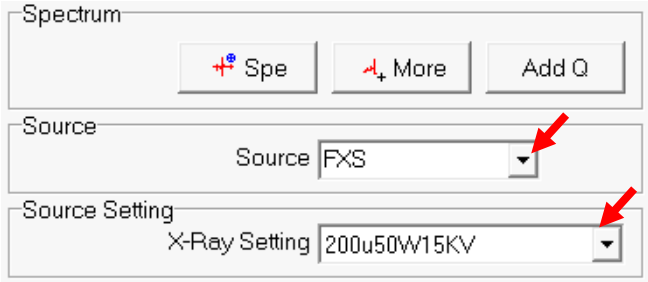
1) After **Z-Align**, **Right** Click on the interested areas on the **Sample Photo** and select **Create Point** on the dropdown list.
2) **Name** the positions and choose collection **Type** as either **Point** or **HP** for spectral analysis.
**Note**:

> If **Point** is chosen, typically the X-ray source of either **100 u25W15kV** or **200u50W15kV** should be chosen in **XPS** window Source Setting in **Session 7.3**
> If **HP** is chosen, the X-ray source of **100u100W20kV_HP** **must be chosen** in **XPS** window Source Setting in **Session 7.3**

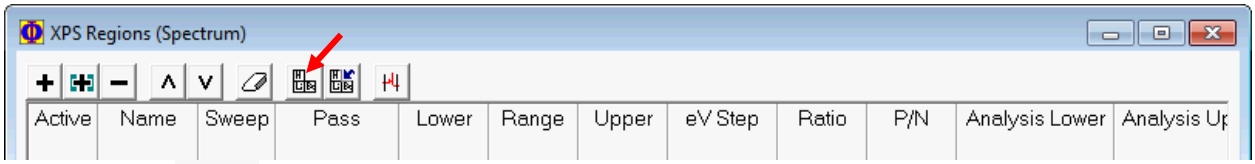
## 8.3 XPS Survey (Full Range) Scan

1) Click XPS tab on top tab menu to enter **XPS** analysis window and click **Spectrum** tab on the right side:
   a) In **Source** window, choose **FXS** (Focused X-ray Source)
   b) In **Source Setting** window choose typically **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**; choose **Point** as Position Type in the sample **Position List**
   > Choose **100u100W20kV_HP** to improve the signal/noise ration; choose **HP** as Position Type in the sample **Position List**
   > **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 Signal/Noise** is **very low** with **small X-ray power** even after hours of scans.




2) Click on top left of **Regions** window to enlarge the **XPS Regions (Spectrum)** window:

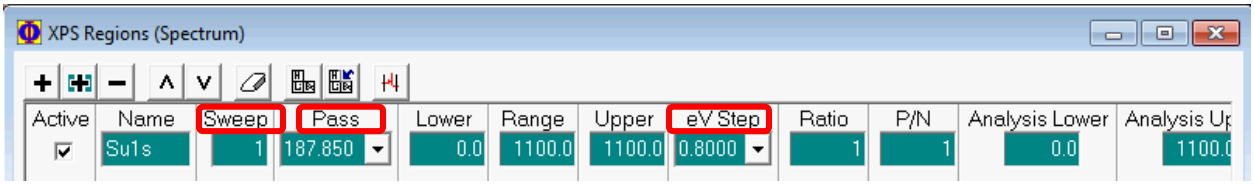



3) Click in above window to **erase** previous setup parameters
4) Click on button on the **XPS Regions** window to open the **Periodic Table**, and click Su to start a **Spectral Survey** (**Su**) Scan.

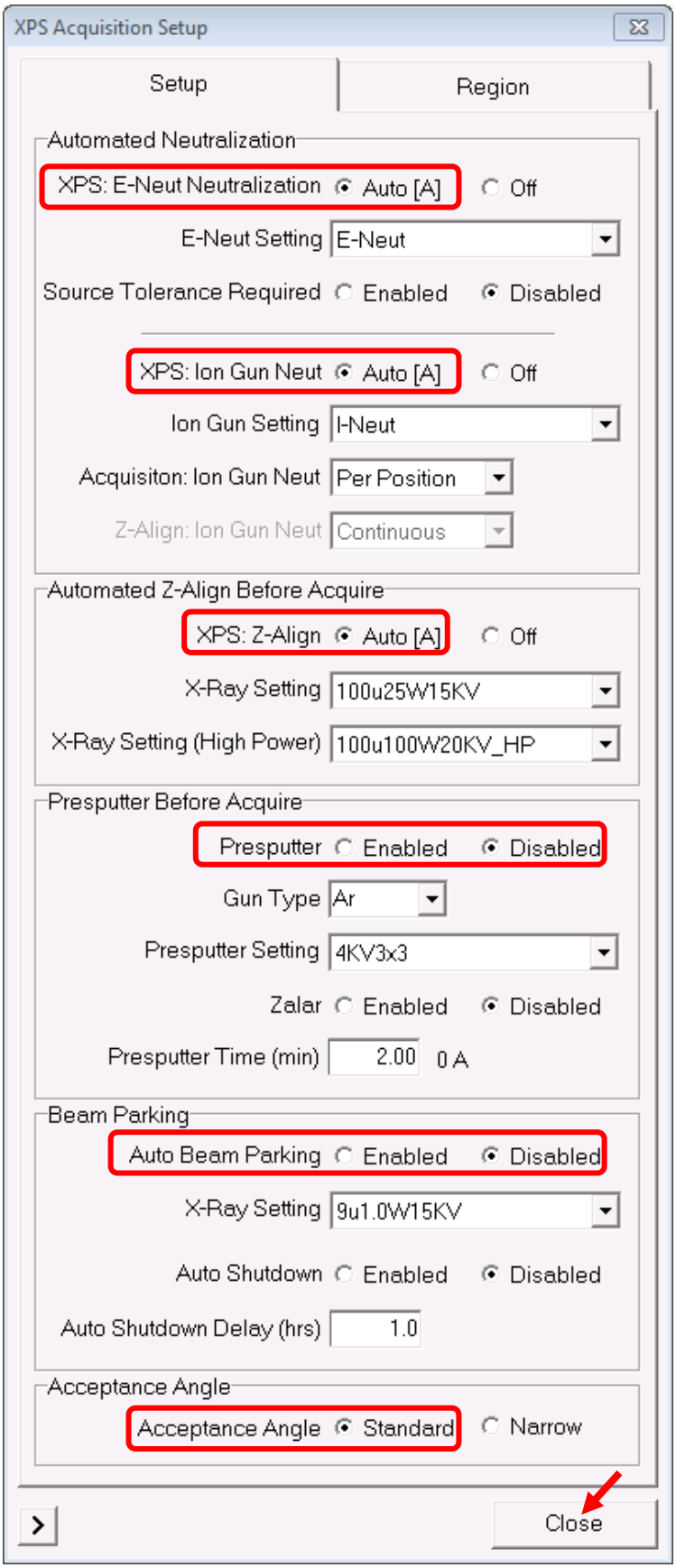

5) In **XPS Regions** window:
   a) Click Pass to change **Pass Energy** to typically **187.850** eV;
   b) Change eV Step to typically **0.8000** eV;
   c) Choose Sweep to typically **5** times;
   d) **Close** the enlarged **XPS Regions** window.

**Notice**:

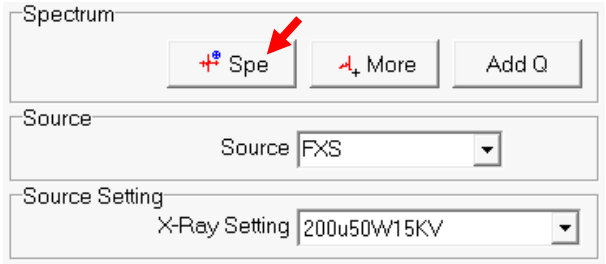
> For **Survey** scan, typically **highest Pass Energy** should be chosen to increase **signal sensitivity** and **larger Step** size for a quick scan.



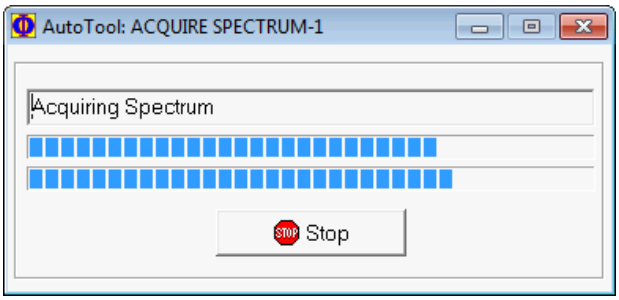

6) Click on XPS Setup... button on the bottom of the **Spectrum** window:
   a) Make sure **XPS: E-NEUT Neutralization** is checked on **Auto [A]**
   b) Make sure **XPS: Ion Gun Neut** is checked on **Auto [A]**
   c) **For Survey scan only**, check **Auto [A]** in **XPS: Z-Align**
   d) Make sure **Presputter** is **Disabled**
   e) Make sure **Auto Beam Parking** is **Disabled**
   f) Make sure **Acceptance Angle** is **Standard**
   g) Hit **Close** to quit **XPS Acquisition Setup**

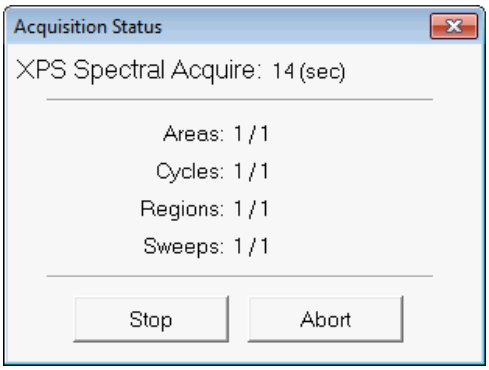

XPS Acquisition Setup
Setup
Region
Automated Neutralization
XPS: E-Neut Neutralization
Auto [A]
Off
E-Neut Setting
E-Neut
Source Tolerance Required
Enabled
Disabled
XPS: Ion Gun Neut
Auto [A]
Off
Ion Gun Setting
I-Neut
Acquisiton: Ion Gun Neut
Per Position
Z-Align: Ion Gun Neut
Continuous
Automated Z-Align Before Acquire
XPS: Z-Align
Auto [A]
Off
X-Ray Setting
100u25W15KV
X-Ray Setting (High Power)
100u100W20KV_HP
Presputter Before Acquire
Presputter
Enabled
Disabled
Gun Type
Ar
Presputter Setting
4KV3x3
Zalar
Enabled
Disabled
Presputter Time (min)
2.00
0 A
Beam Parking
Auto Beam Parking
Enabled
Disabled
X-Ray Setting
9u1.0W15KV
Auto Shutdown
Enabled
Disabled
Auto Shutdown Delay (hrs)
1.0
Acceptance Angle
Acceptance Angle
Standard
Narrow
Close

7) Hit Spe in Spectrum window to **start Survey** scan

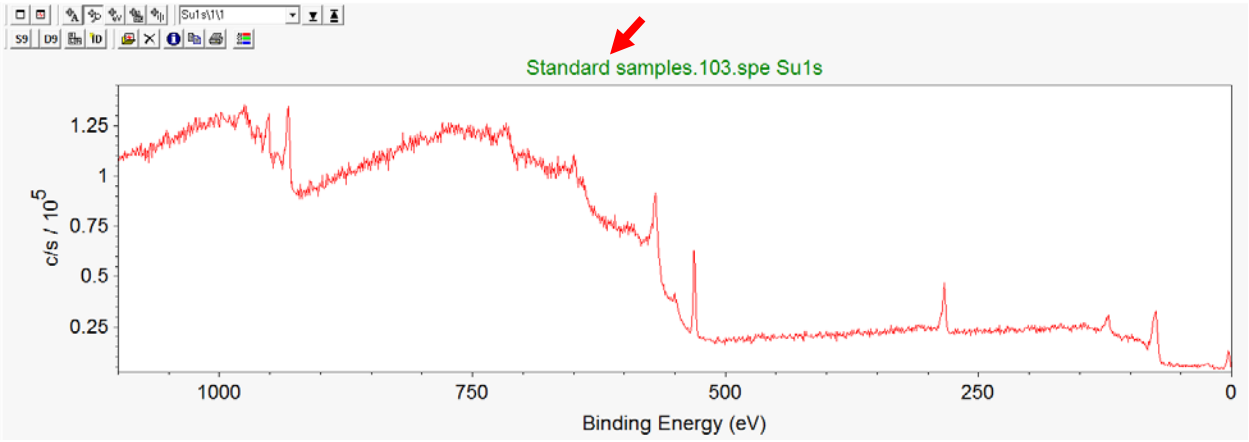



8) **Wait until** the following windows to appear:



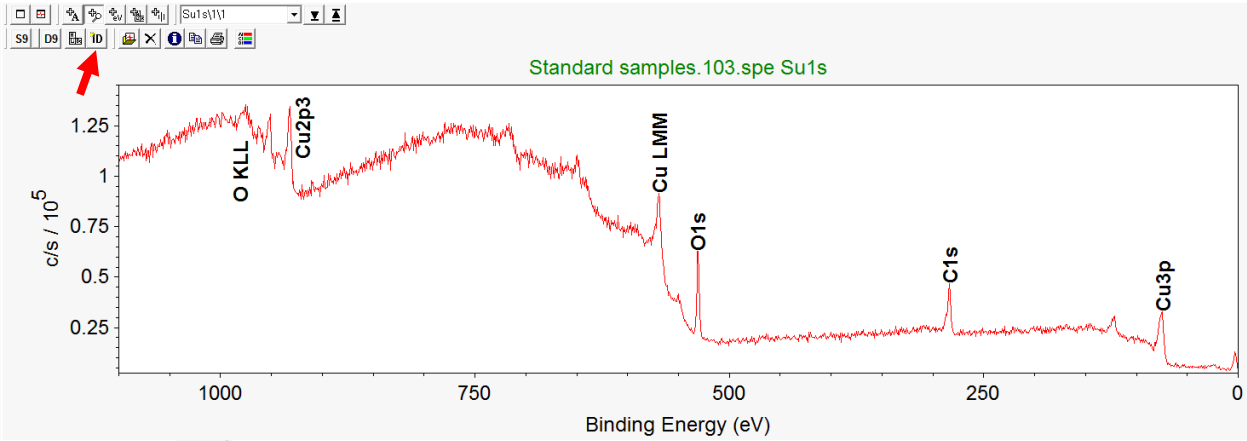




9) The Survey spectrum will appear in the **Spectral Window**.
**Note**:
   - The data **filename** starts with Platen Name "Standard samples", the serial number "103", spectra scan extension "**spe**", and scan type Survey "**Su1s**".
   - The data files have been automatically saved into specified folder which can also be opened in **MultiPak** program

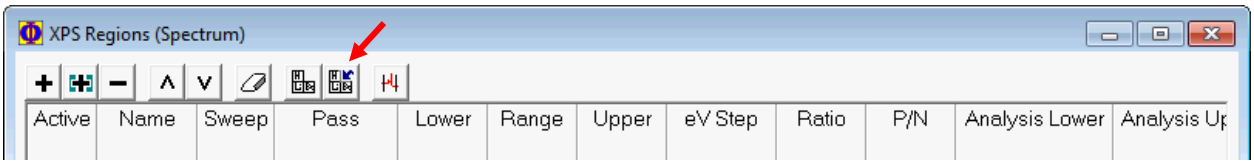


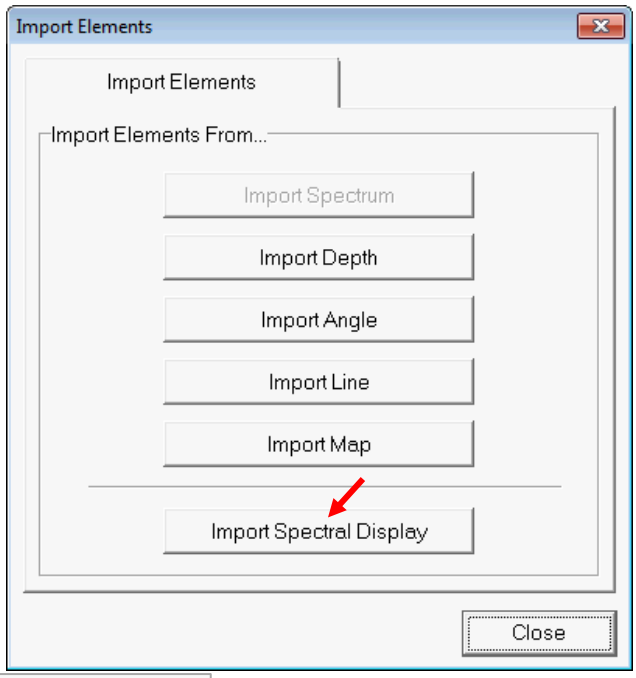
## 8.4 Regional (Elemental) Scan

1) Click  on the Survey spectrum just collected to start **Peak ID**

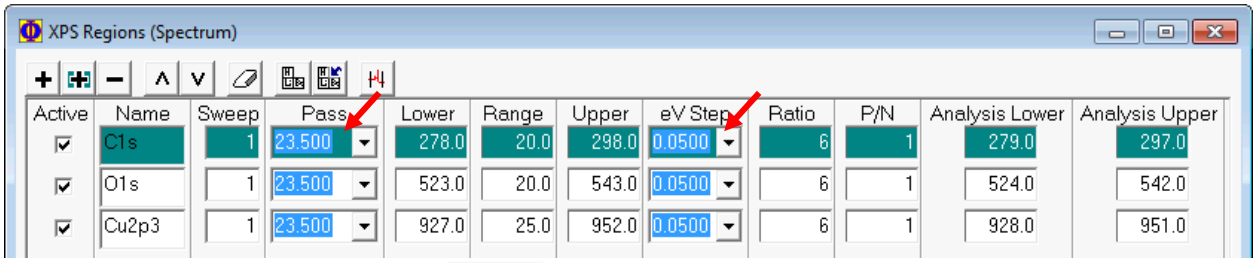



2) Click  on top left corner of **Regions** window to Show Table Details of the **XPS Regions (Spectrum)** window, and click  to open **Import Elements** window



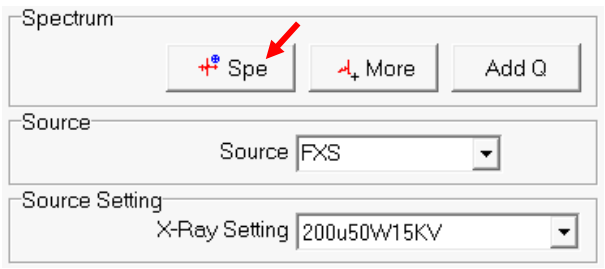




3) Click Import Spectral Display button in the **Import Elements** window above and hit **Close**.
4) The elements detected in **Survey Scan** are imported into the **XPS Regions** table as below.

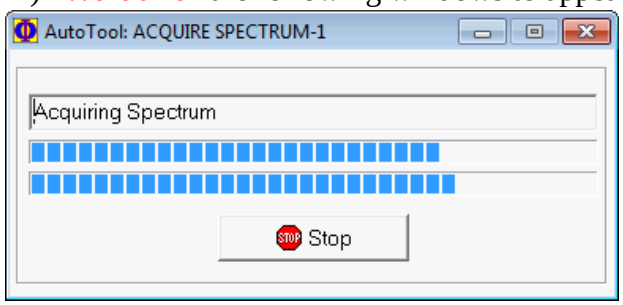


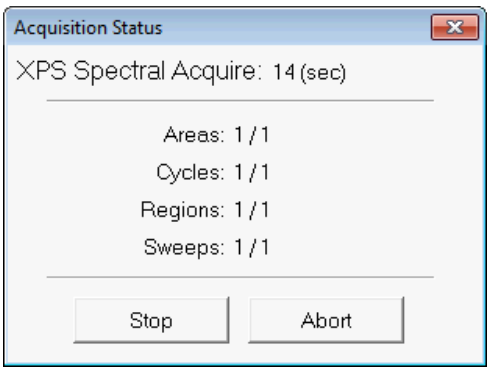

> Click space below Sweep to change **Number of Sweeps** to typically **5 ~ 20** depending on peak intensity collected in **Survey** spectra,
**Note**:
> > Consider choose **HP** source if samples are not sensitive to X-ray radiation. The signal will be doubled comparing to **200u50W15kV** source,
> > Remember to **change** the Position **Type** to **HP** in the Sample Position List in Sample window if **HP** source is chosen.
> Click space below Pass to change **Pass Energy** to typically **23.500** eV,
> Click space below eV Step to change **Step Size** to typically **0.1000** eV,
> **Close** the **XPS Regions (Spectrum)** window.
**Note**:
> For **Region** scan, typically **smaller Pass Energy** (**23.5 – 58.7 eV**), **smaller Step** size (**0.05 – 0.25 eV**), and **20 ms Time Per Step** should be chosen to increase **spectral energy resolution**.
**Warnings**:
> **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 spectral resolution.

5) Click on XPS Setup... button on the bottom of the **Spectrum** window:
   a) Make sure **XPS: E-NEUT Neutralization** is checked on **Auto [A]**
   b) Make sure **XPS: Ion Gun Neut** is checked on **Auto [A]**
   c) **Uncheck** **Auto [A]** in **XPS: Z-Align**
6) Hit Spe in Spectrum window to start **Region** scan



7) **Wait until** the following windows to appear




8) The **Region Scan** spectra will appear in the **Spectral Window** following the sequence in **Regions** table.

**Note**:

> The data files will be automatically saved into specified folder which can be opened in **MultiPak** program.

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

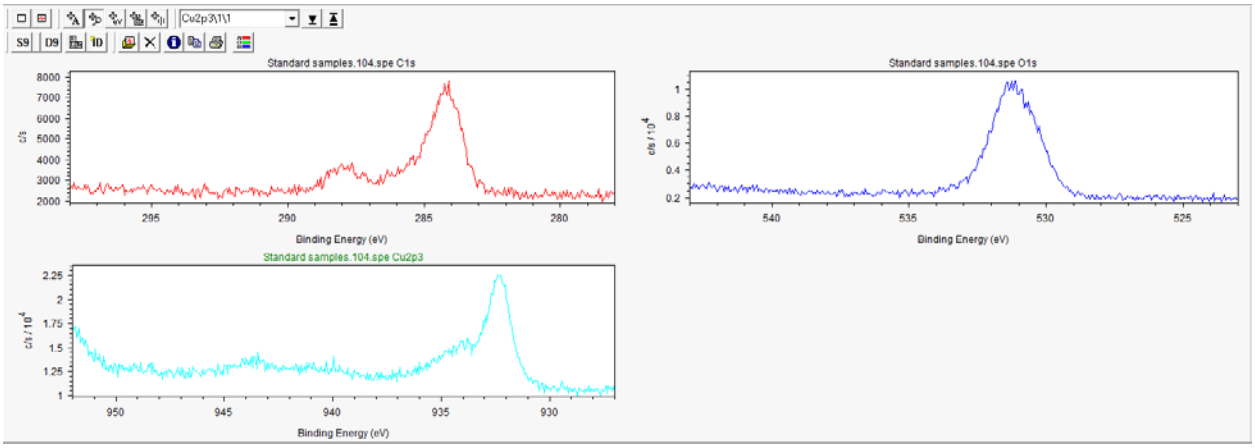
> The **Lower** energy and **Range** can be adjusted to scan the interested regions only.

> **Pass Energy** and **Step** size setup:

- Good resolution: **Pass Energy**: **23.5 - 58.7 eV**; **Step Size**: **0.125 - 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 individually created elements or regions
- **Cycle** to all created regions



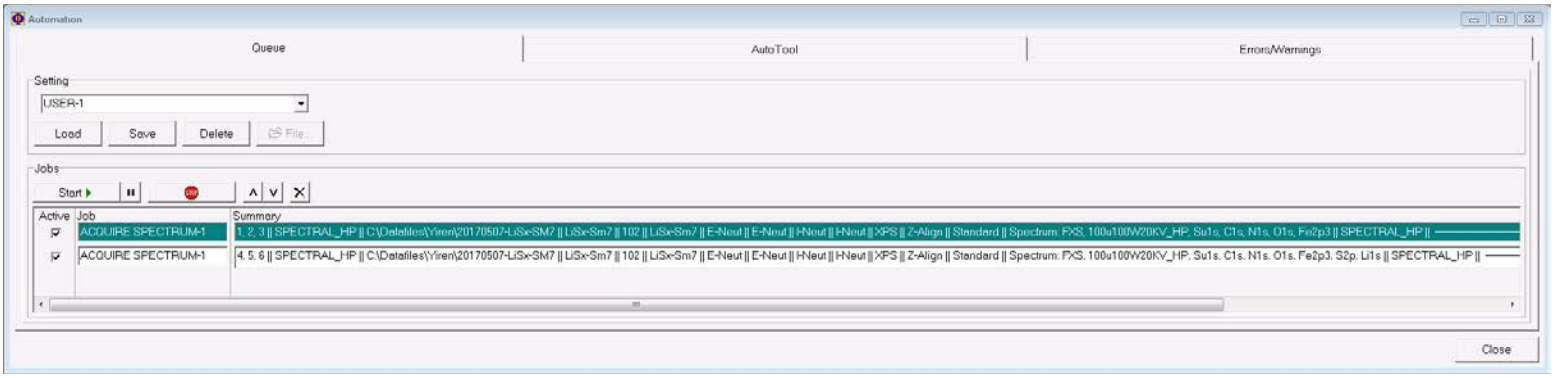

9) **Add Q** (Add to Queue mode) if interested elements are **different** **on different samples**:

a) **Make sure** **a complete list of elements** has been set up in the **Regions** window,

b) Go back to **Sample** window, **check** **ONLY** intended sample positions on the list,

c) Go to **XPS** window, **select** **ONLY** interested elements on intended sample positions,

d) Hit Add Q button in **Spectrum** window to add selected queue to the **Add Q** window below:




e) **Minimize** above window, go back to **Sample** window and repeat **Step b) – d)** until all sample positions are selected;

f) Maximize the **Add Q** window and hit Start to start spectral scan.

# 9 Closing XPS Measurement

1) Checklist before sample extraction:

   a) Go to **System** → **Transfer Sample** window, if the **V3 valve** is in green 

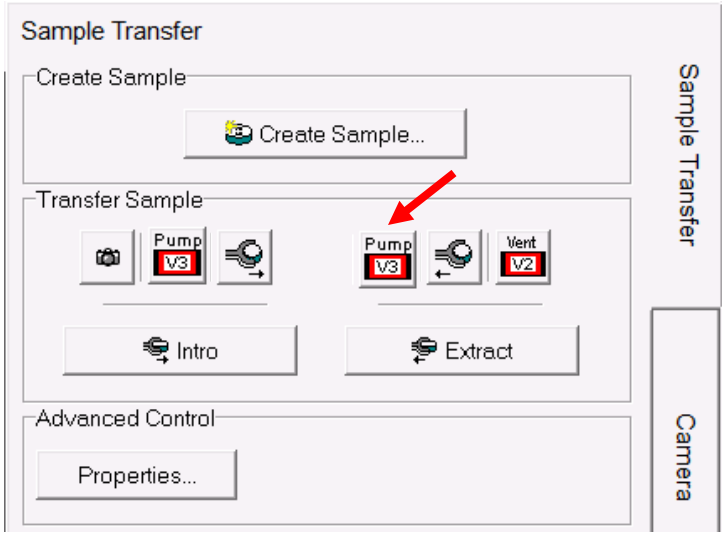
, indicating the **Intro Chamber** is in **good** vacuum, then **skip** **Step 2)** below;

   b) Else If **V3 valve** is in red 

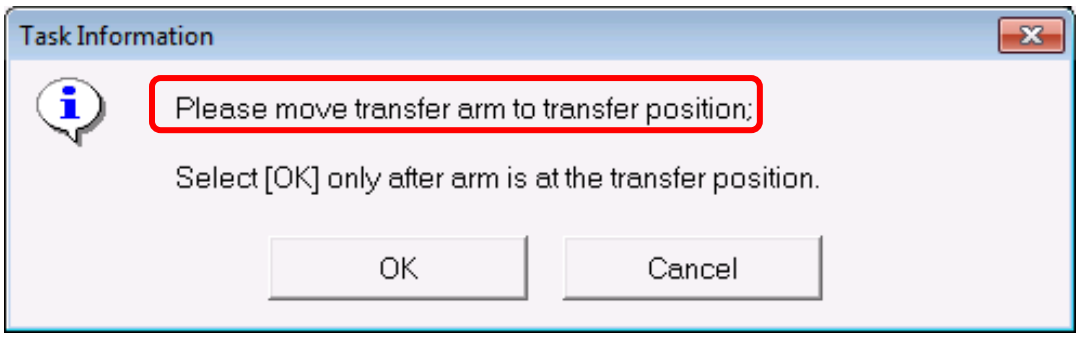
, **continue** **Step 2)** below

2) Click 

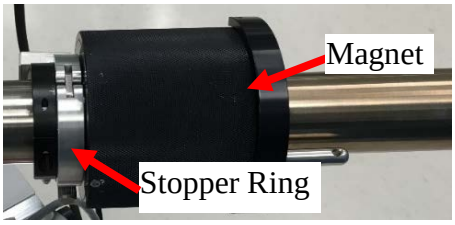
button on the **Transfer Sample** window, **wait until** the **Intro** pressure reaches below **4.0E-004 Pa** on Cold Cathode gauge.
**Warning**:
   > Sample transfer at high **Intro Chamber** pressure will introduce **serious contaminations** to **Electron Energy Analyzer**, **X-ray**, **UV sources** and sample stages.




3) **Wait until** the **Intro Chamber** pressure reaches below **4.0E-004 Pa** and then **click**  to start **Sample Extraction**
4) **Wait until** the following **window** appears:

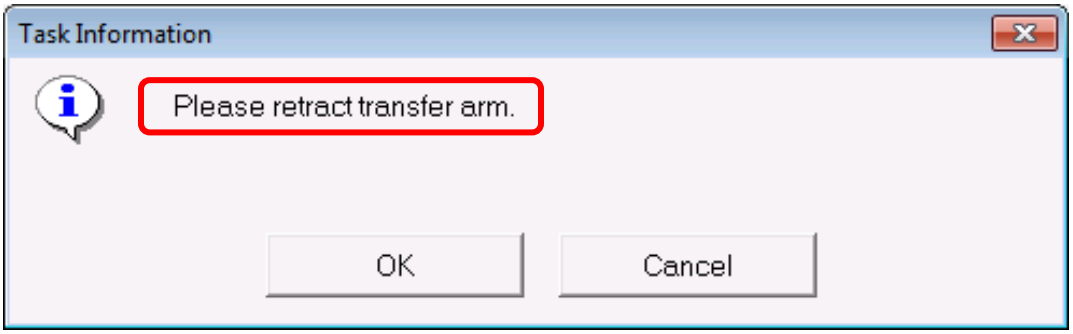


5) **Follow the command in above window** to move the transfer arm **forward** and **MAKE SURE** the **magnet TOUCHES** the **stopper ring** and the **stopper ring** on the transfer arm.
**Warnings**:
   a) User should **slow down** when approaching transfer position. The sample holder should be **SMOOTHLY** inserted into the sample stage in Main Chamber. **CONTACT** lab manager immediately if **strong force** felt at this step,
   b) **Slight adjustment** maybe needed to insert sample holder into the stage forks. The user should **CONTACT** lab manager for help,
   c) **NEVER force** the sample into sample stage. Expensive parts inside will be **damaged**.




6) **Leave** the transfer arm at the **transfer position**; **go back to the computer** and click **OK** on the popup window
**Warning:**
   a) **NEVER** click **OK** on the window before moving transfer arm forward. Or **serious mechanical damage** to both stage and sample holder will occur. User account will be **suspended** and **repair cost** will be charged on PI's account.

7) Wait the following **window** to appear. **DO NOT** click **OK**:



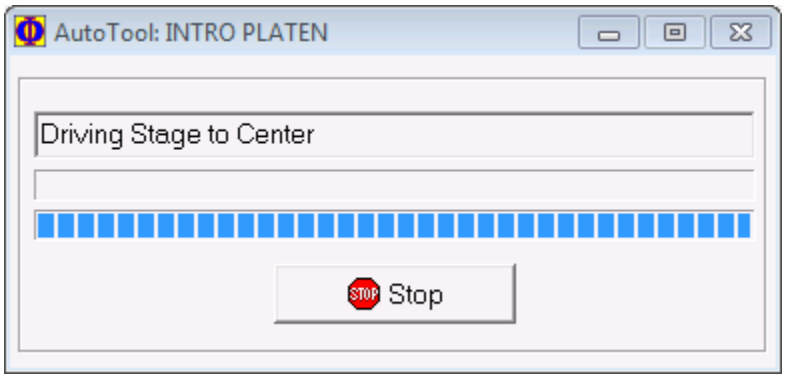

**Warning:**
   a) Failed to follow above step will lead to **serious part damage to both sample stage and sample holder**. User account will be suspended and repair cost will be charged on PI's account.

8) **Follows** the command in above window to **fully retract the transfer arm back** to the **Intro Chamber**
**Warning**:

a) Retract the transfer arm **VERY SLOWLY** and watch the sample holder through **Main Chamber** view port. The sample holder should **STAY** on the transfer arm during extraction.

9) **Go back to the computer** and click **OK** on above window

10) **Wait until** the stage is driven to the center.



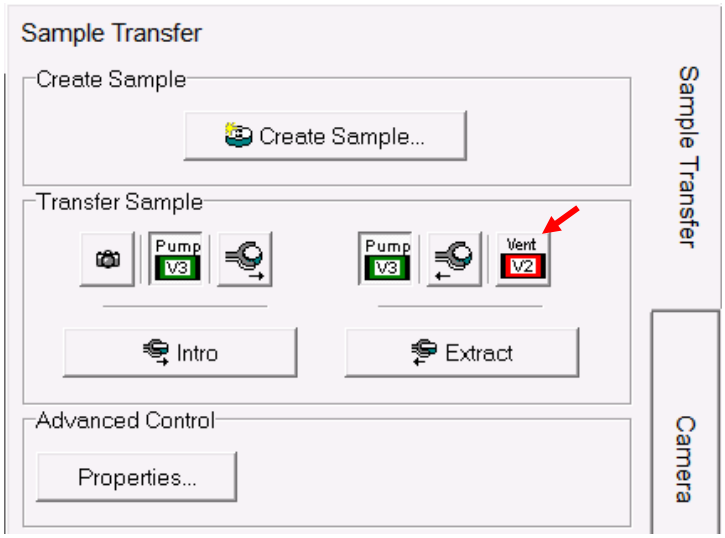

11) **Turn OFF** the **chamber light**.


12) **If your samples can be discarded, then Skip step 13) – 15)** and leave your samples inside Intro Chamber. The next user should clear your samples on the holder. **Otherwise** continue to steps below to **retrieve your samples from Intro Chamber**.

13) Click 

in **Sample Transfer** window below to **vent** the **Intro Chamber**

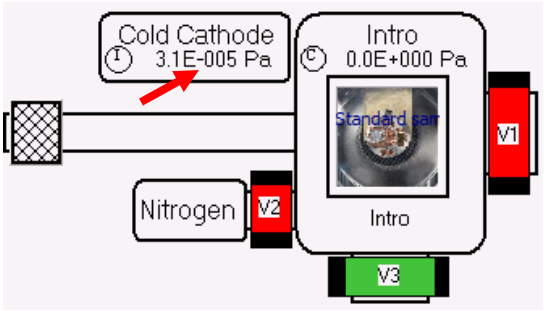
**Note:** the user must wear **gloves** at hold the glass cover during **venting**




14) **Wear gloves**, wait until the **Intro Chamber** is vented, remove the sample and put the **Viton cover** back.
**Warning:**
   a) User **CAN NOT** touch the sample loading port with exposed skins
   b) **DO NOT** touch the camera atop the loading port

**15) Pump the Intro Chamber back to vacuum:**
**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 **hold and press the Viton cover against the flange during Intro Chamber pumping!**
   **b)** Watch the **Cold Cathode** gauge reads **4.0E-004 Pa**
   **Warning: DO NOT logoff FOM if the pressure fails to reach 4.0E-004 Pa** in a short time ~ **5- 10 minutes**. It may indicate a chamber leak. **Report** to lab manager before leave**.**




16) Leave **SmartSoft** and **Watcher** programs **ON**.
17) **Back up your data** either through internet (**Yale Box Sync**) or using **Core USB drive**.
**Warning**:
   > **personal USB drive is prohibited on instrument computers**.

18) Logoff **FOM program**

a) Click the  icon at the task bar below to activate the **FOM** program, and hit **Logoff** button in the program.

**Note**: you may need to click the  twice to activate the **FOM** program

b) Clear the bench and put tools back into the box and then check the message box in FOM window

**c)** Hit **Logoff** button to activate **FOM Screen Locker**

19) **Sign out** on the **logbook** and report any problems on instrument.

20) **Remove** sample from the holder and **clean** it with clean wipes and **IPA**.

21) **Store** the sample holder, tongs and other tools in the tool box.

# 10 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**

# 11 Chamber Leaking Emergency Operation Procedure

1) **Intro Chamber** leaking:
   This could happen during **Intro Chamber** pumping after sample loading or sample removal.
   > The **Intro Chamber** pressure may sticks to ~ low **E+001 Pa** longer than ~ **15 minutes** if the chamber is empty or has very dry samples inside, and turbo pump fails to start;
   > Or after turbo pumping starts, it takes more than **60 minutes** to reach **4.0E-004 Pa**.

   **To retrieve good vacuum in Intro Chamber:**
   a) **While the Intro Chamber is still being pumped,** press and slight turn the **Viton cover**, and **check** if the pressure improves quickly, if **NOT**
   b) **Vent** the intro chamber through **SmartSoft** program. Take the cover off and **check** inner side of **Viton O-ring** for any **dents, particles**; clean with **Kimwipes** and **isopropanol**. Put the cover back, **make sure** the cover is **placed properly** and start pumping. **Hold the cover firmly into the groves on the port during initial pumping.** If the pressure is still not improved quickly, **contact Core manager immediately.**
2) **Main Chamber** leaking

   The Main Chamber could leak if the **V1 valve is not closed properly** due to either program failures or **V1** valve damage.

   > To check if the **Main Chamber** is leaking:
   > > Make sure the **V1** valve is closed (in **red** border).
   > > If the **Main Chamber pressure drops rapidly from low E-007 or high E-008 Pa to E-006 ~ E-003 Pa** when the **Intro Chamber is vented** for sample loading, indicating a series leak in the Main Chamber, **a serious leak** exists through **V1** valve from **Intro Chamber**.

3) **Report the leak to Core manager immediately**