

XPS Standard Operating Procedure 1: Sample Introduction

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These notes are meant to serve as an aid to assist users who have been trained and certified by MCC Staff. If ever you are unsure about the correct operation of the instrument or any of its components, please consult a MCC staff member before continuing. Never use equipment that you are not trained and approved to use.

Before using the MCC, please review the MCC User Handbook available through our website.

Sample Position and Mounting

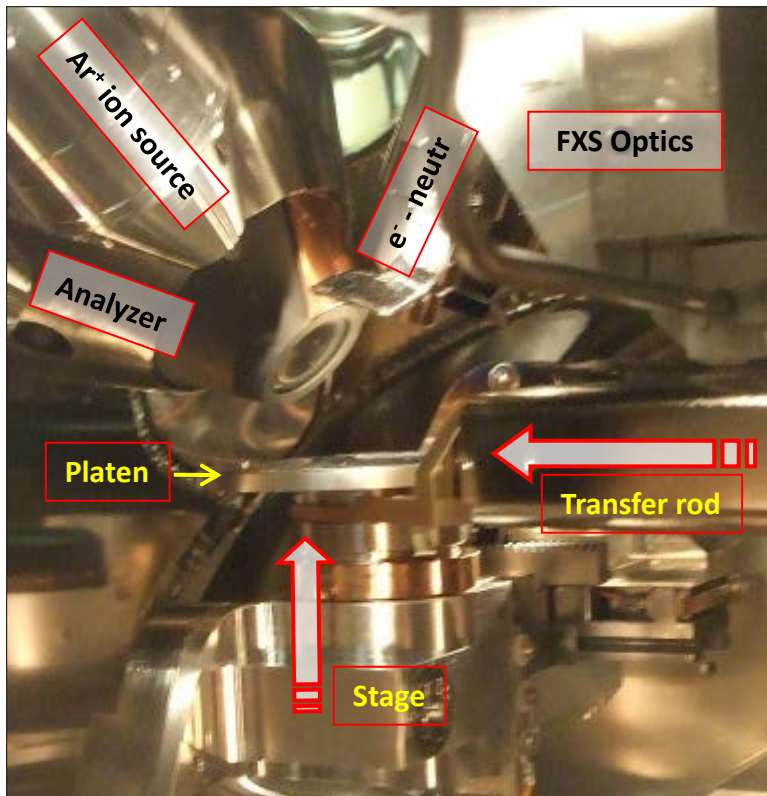


Fig. 1. View of sample location in main chamber

The sample is centrally positioned in the main chamber with all beam sources and the analyzer looking at it at pre-calibrated angles (Fig.1)

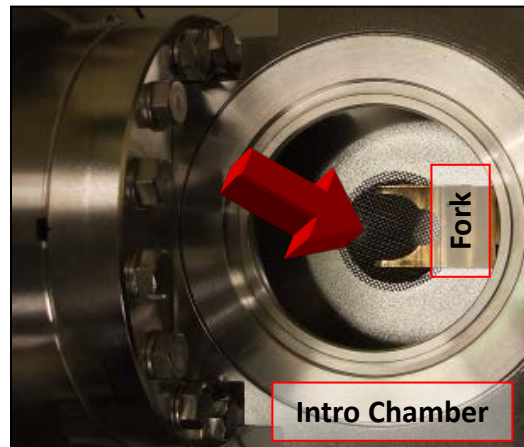
Before a sample can be placed into the analysis chamber, you must secure it to a sample mount. To avoid contaminating the sample surface, **do not handle the sample and mount with bare hands. Wear gloves!**

Caution

Samples must be ultrahigh vacuum compatible; handled with clean, nonmagnetic tools and gloved hands. Liquid and magnetic samples must not be introduced into the system.

Sample Introduction

The following sequence assumes that there are no samples in the system, the introduction chamber is pumped and the Transfer rod is fully withdrawn. The **Summitt** and **Vacuum Watcher** software must be open



- Open the main valve of dry N2 purge cylinder. The outlet pressure is fixed to 4 -5 psi
- Click the **Backfill Intro** button, wait for cover cap of **Intro** to be released! Close main valve on cylinder
- Remove cover cap from **Intro chamber**
- Mount **sample holder** on **Transfer rod** using the special tool. Make sure the small dent of the holder faces the fork and the sample holder is securely mounted
- Place cover on the **Intro chamber**

Optional Backfill Intro (if Sample platen is inside the Intro)

The screenshot shows the Watcher control interface. The central diagram illustrates the system components: V4 (Air Gun), V2 (Intro Chamber), V3 (Intro Turbo), V6 (Main Chamber), V7 (Rough Pump), and V1 (Ion Pump). The task control panel on the right shows the task 'Backfill Intro' selected, with a 'Process Task' button and a task status of 'Transfer Sample - Task Complete'. The bottom left panel displays binary sensors and sensors.

Binary Sensors	Sensors
Bake Inlet: Bake Fault	Intro Chamber Conv: 0.000E+000
Probe Status: Retracted	Main Chamber Ion Gauge: 4.300E-007
V1 closed status: closed	Intro Chamber Cold Cathode: 2.304E-004
V1 open status: not open	
V4 Gun Turbo Iso: V4 Closed	

The screenshot shows the Watcher control interface. The central diagram is identical to the first screenshot. The task control panel on the right shows the task 'Backfill Intro' selected, with a 'Process Task' button and a task status of 'Backfill Intro - Task Complete'. The bottom left panel displays binary sensors and sensors.

Binary Sensors	Sensors
Bake Inlet: Bake Fault	Intro Chamber Conv: 9.900E+004
Probe Status: Retracted	Main Chamber Ion Gauge: 2.800E-007
V1 closed status: closed	Intro Chamber Cold Cathode: 2.371E-004
V1 open status: not open	
V4 Gun Turbo Iso: V4 Closed	

Watcher

File Sessions Tools User Level Help

Control

Tasks

Task Control

Task: Pump Intro

Process Task

Task Status

Abort Task

Actuators

Binary Sensors

- Bake Intlk: **Bake Fault**
- Probe Status: **Retracted**
- V1 closed status: **closed**
- V1 open status: **not open**
- V4 Gun Turbo Iso: **V4 Closed**

Conversion Stage
Ion Gauge
Cold Cathode Gauge

Watcher

File Sessions Tools User Level Help

Control

Tasks

Task Control

Task: Transfer Sample

Process Task

Task Status

Abort Task

Actuators

User Choice

Pump Intro Complete. Transfer Sample?

User Choice

Introduce the sample now

OK

Sample Introduction

The screenshot shows the Watcher software interface. The 'Task Control' dropdown menu is set to 'Task: Pump Intro'. The 'Task Status' field is empty. The 'Control' panel displays a schematic diagram of the chamber system with components like V4, Ar Gun, Main Chamber, Ion Pump, Intro Turbo, and Rough Pump. The 'Binary Sensors' section shows 'Bake Intlk: Bake Fault', 'Probe Status: Retracted', 'V1 closed status: closed', 'V1 open status: not open', and 'V4 Gun Turbo Iso: V4 Closed'. The 'Sensors' section shows 'Intro Chamber Conv.: 0.000E+000', 'Main Chamber Ion Gauge: 1.900E-007', and 'Intro Chamber Cold Cathode: 2.450E-004'.



The screenshot shows the Watcher software interface after the task change. The 'Task Control' dropdown menu is now set to 'Task: Transfer Sample'. The 'Task Status' field now displays 'Transfer Sample - Task Complete'. The 'Control' panel schematic is identical to the first screenshot. The 'Binary Sensors' section is identical. The 'Sensors' section shows 'Intro Chamber Conv.: 0.000E+000', 'Main Chamber Ion Gauge: 2.800E-007', and 'Intro Chamber Cold Cathode: 2.360E-004', with the latter value highlighted by a red box.

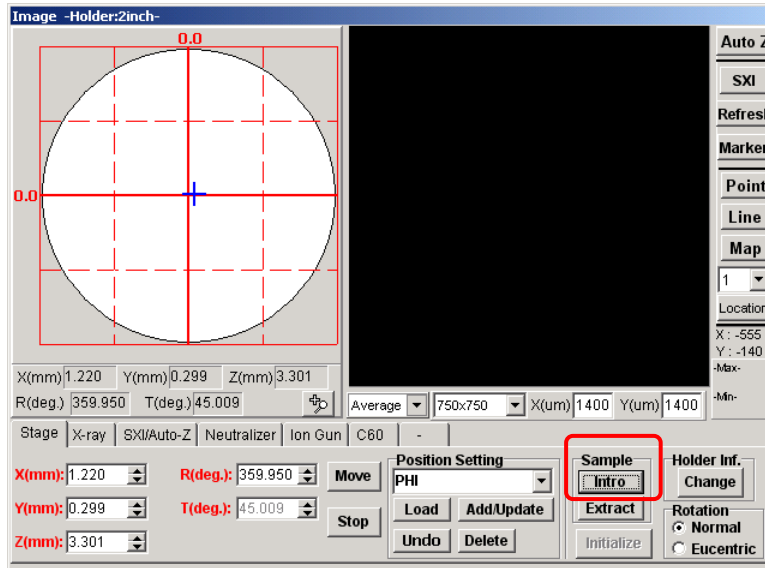
- Click **Pump Intro** in the **Vacuum Watcher** window to initiate the pump down sequence. The routine takes ca 6 min
- The sample can be left in the **Intro chamber** for **Cold Cathode Reaches** $1 \text{ e-}4$!!!
- Depending on the sample and the processing condition this time can vary min 20-120 min and up to hours and in some cases over night

Take enough time for sample out gassing

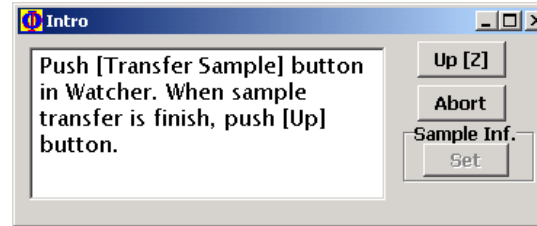
Possible consequences

- 1. System will shut down immediately and the vacuum need to be recovered
(few days typically if there is no need for backing out the chamber)**
- 2. It will increase the frequency of the need for bake out**
- 3. It might contaminate the electron gun and as a result there will be no X-ray. So the system will be marked as down till the service engineer come to the site**

Sample Introduction

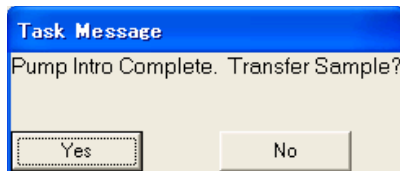


- Sample holder (platen) introduction in main chamber: Click the **Intro** button in the **Summitt – Image - Stage** window, the stage moves to a pre-calibrated home position. This dialog box appears:

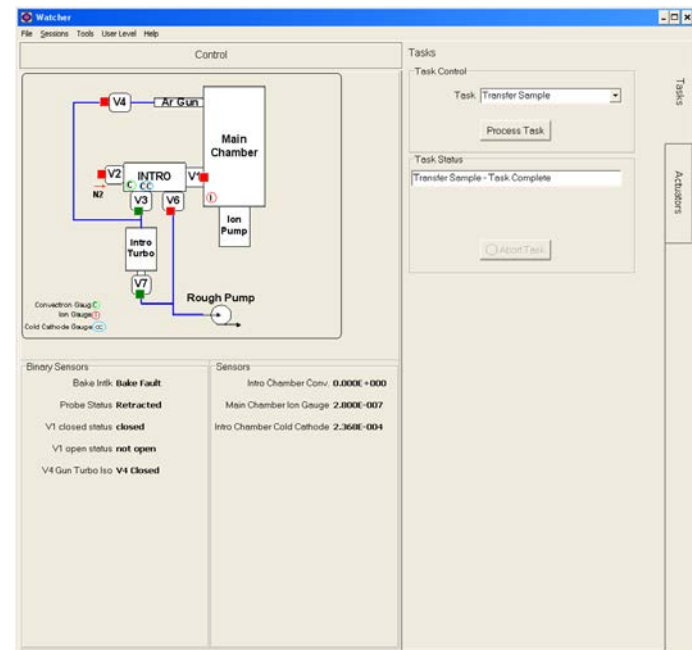
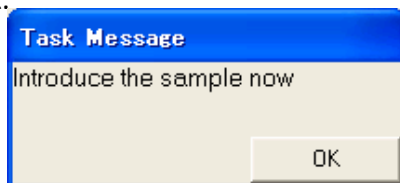


- Do not click Up[Z] !!

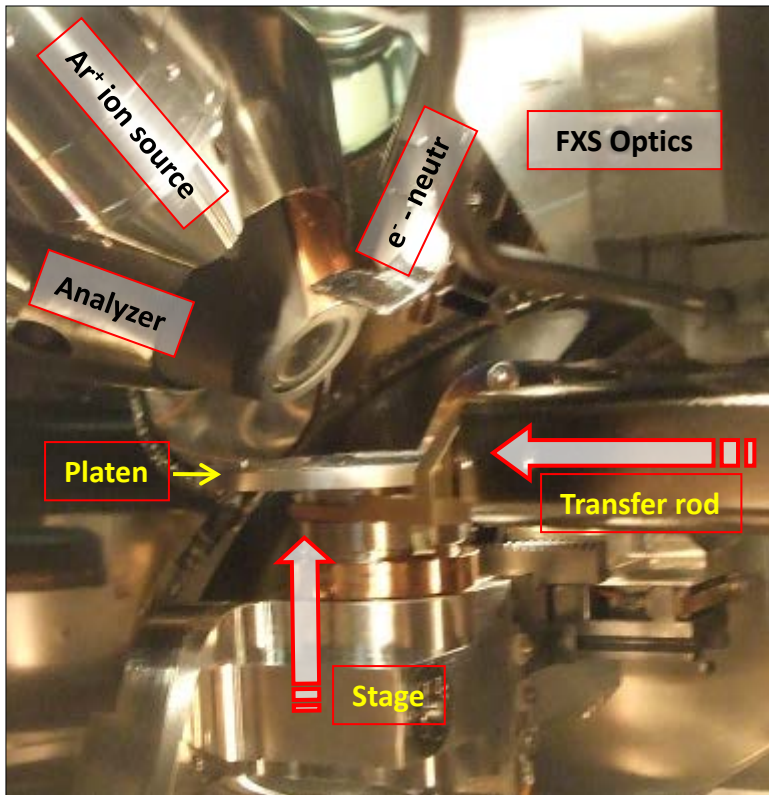
- Click **Transfer Sample** button in **Vacuum Watcher** window to initiate the sequence for opening the main chamber valve V1. This dialog box appears, **Click Yes**



- When V1 opens another **Watcher** Task message appears, **click OK**:

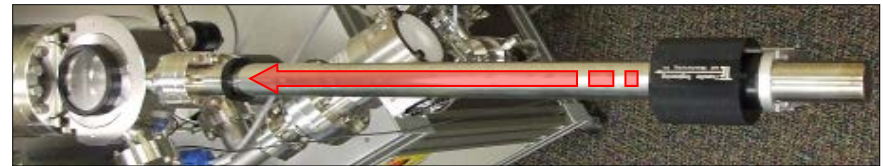


Sample Introduction

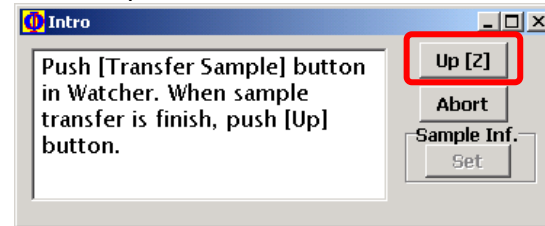


View of sample position in main chamber

- Insert fully the sample holder (platen) into the main chamber above the stage using the transfer rod



- In the **Intro** dialog box click **Up [Z]** to raise the stage to pick-up the sample holder



The stage must fully engage the sample holder; Verify through the viewport that the sample holder is secure on the stage

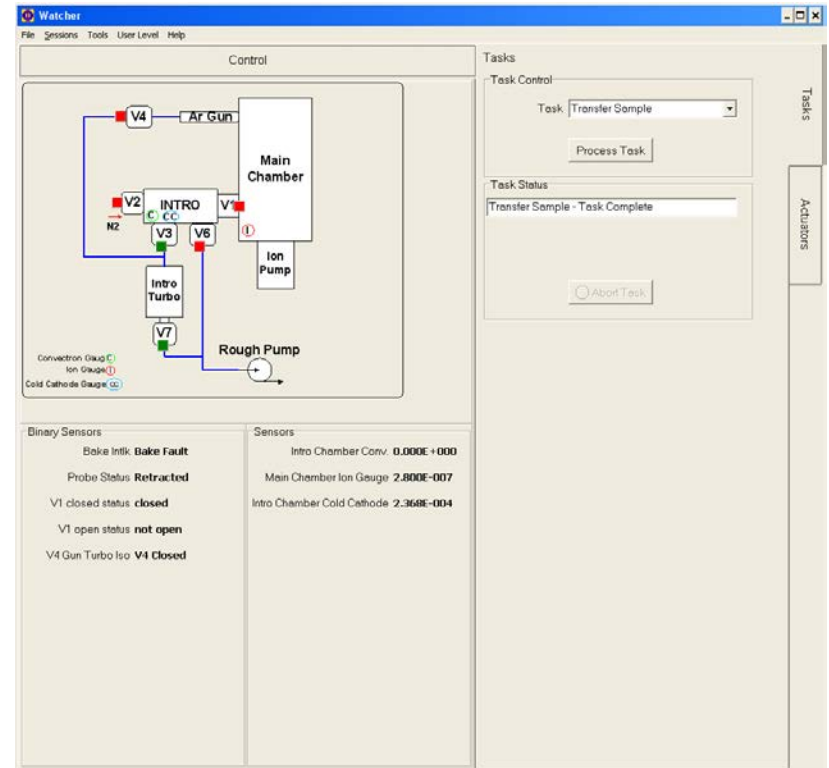
- Retract Transfer Rod completely out of the analysis chamber. Valve V1 in **Vacuum Watcher** will automatically close once the transfer rod is fully retracted (see next slide)



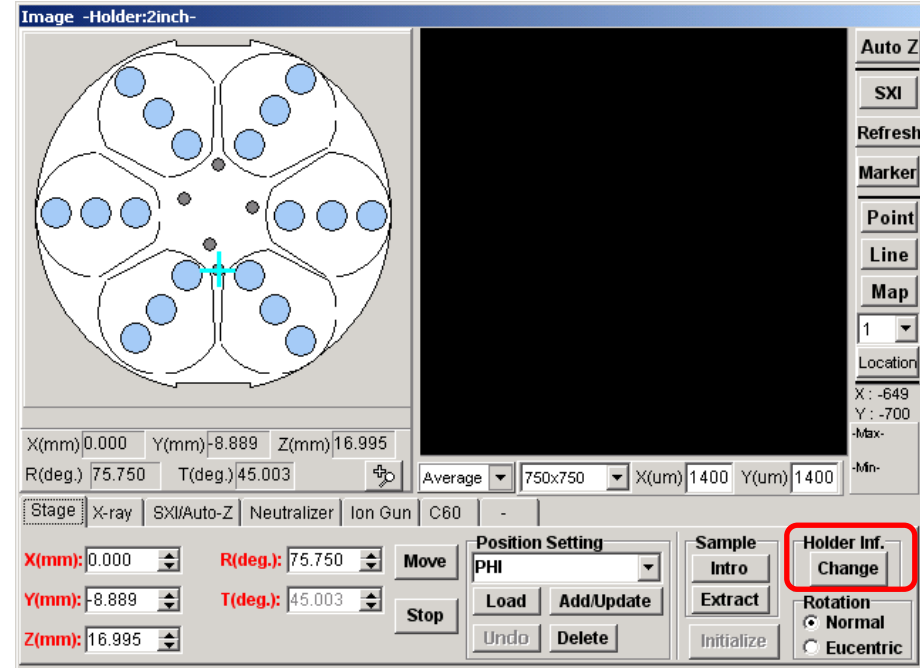
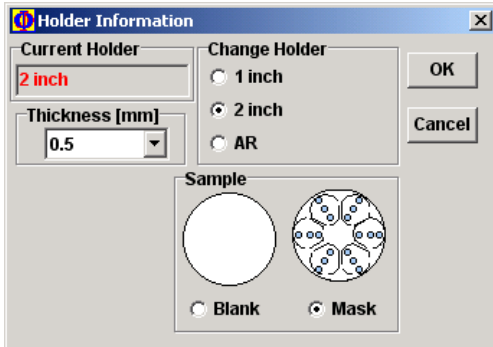
Sample Introduction

After **Transfer sample** is completed the following dialog box appears:

Click **OK** if you want to keep the existing sample platen info
Click **Set** to choose sample height and select platen size



Platen Type/Height Selection



- The platen type may be also specified by clicking **Holder Inf./Change** on the **Stage** menu in **Image** window