# XAS chamber: sample loading & unloading procedure

The instructions on how to place the sample in the chamber are outlined in here.

Every time the Load-lock chamber is vented, 3 sample holders can be loaded on what is called the “ladder”. Only one sample holder can be introduced from the ladder to the main chamber when the load-lock chamber reaches the required vacuum.

## Controlled vent of the Load-lock chamber

From the PGM CONTROL PANEL check the following requirements:

- the negative high voltage (-1450 V) on the fluorescence (FL) detector is OFF (ramped down and off);
- the End-Station gate valve (connecting to the upstream of the beamline) is **CLOSED**.

From the Endstation check that:

- The MANUAL gate valve between the main chamber and the load-lock chamber is **TIGHTLY CLOSED**.

After all three requirements are verified, you can start venting the load-lock chamber:

i. Close the manual “speedy” valve to the scroll pump.
ii. Switch off the turbo pump of the load-lock chamber (pushing the start/stop button)
iii. Slowly bring the load-lock up-to-air (~700 Torr) using the Nitrogen gas-line. Check the pressure in the main valve; it will raise however it should never go higher then $2x10^{-6}$ Torr (i.e. $5x10^{-6}$ Torr).
iv. Open the viewport door in the glove box and raise the ladder.

## Sample loading procedure

With the load-lock section up-to-air, load the samples on the ladder. Lower the ladder, close and finger-tighten the viewport door.

Pump down the load-lock chamber to the required vacuum:

i. SLOWLY open the manual “speedy” valve to the scroll pump monitoring at the same time the pressure in the load-lock section.
ii. Start the Turbo pump (pushing the start/stop button).
iii. Wait few minutes until the Turbo pump reaches NORMAL and the pressure in the load-lock is better than 6.7 mTorr (i.e. 6.5 mTorr).

Open the manual gate valve between the load-lock and the main chamber; the pressure in the main chamber should stay better than $2x10^{-6}$ Torr (i.e. $1.8x10^{-6}$ Torr).

Using the transfer arm, grab the sample. Push the transfer arm into the main chamber and gently slide the sample into the holder.

Fully retract the transfer arm back into the load-lock and close the manual gate valve.

Align the sample with the help of the camera cross.

If the pressure in the main chamber is reasonable (better than $5x10^{-7}$ Torr; i.e. $4.8x10^{-7}$ Torr) from the PGM CONTROL PANEL switch **OPEN** the End-Station gate valve between the main chamber and the upstream of the beamline and turn ON the FL negative high voltage (-1450 V).

## Sample un-loading procedure

From the PGM CONTROL PANEL check the following requirements:

- the negative high voltage (-1450 V) on the fluorescence (FL) detector is OFF (ramped down and off);
- the End-Station gate valve (connecting to the upstream of the beamline) is **CLOSED**.

Open the manual gate valve connecting the load-lock to the main chamber.

Push the transfer arm into the main chamber. Lock onto your sample.

Smoothly fully retract the transfer arm back into the load-lock, CAREFUL not to open the jaws. Place the sample onto the ladder.

Close the manual gate valve.

Do you have more samples on the ladder that need to be analyzed? Proceed with loading the next sample in the main chamber.

Have you analyzed all the samples on the ladder? Proceed with the controlled venting of the load-lock to remove or replace the samples.