XSD Beamline 8ID-E, 8ID-I

Standard Operating Procedure for Linkam Thermal Stage

Updated 3/7/2019

0. Work Planning

Use of the Linkam requires authorization of an ESAF properly completed with these items:

a. The Description tab should state "We will use the beamline's Linkam thermal stage to control the sample temperature over the range XXX to YYYY deg. C. The stage requires Liquid Nitrogen for cooling. We will follow the beamline's SOP for using the stage and handling Liquid Nitrogen."
b. On the Description tab, the current version of this SOP should be attached.
c. The Materials tab will include Liquid Nitrogen (CAS # 7727-37-9), quantity 2L, with the Other box checked and the comment inserted below "Liquid Nitrogen is a cryogen."
d. On the Equipment tab, check the Cryogenics box and the Heater box.

1. Installation

Only qualified beamline staff will install the Linkam stage. The platform for installation is the GIXS (Nealey) chamber base, with the sample selection stage (vphic) and the wedge removed. The Linkam stage is mounted onto the vsamx/vsamy stage via the Linkam stage adapter. The components and top chamber to be installed depend on the q-range and the temperature range of the experiment. Beamline staff will discuss experimental plans with the users prior to installation and install the appropriate components. Staff will check that the GIXS chamber is equipped, as usual, with a pressure relief valve (Accu Glass, PRV ON A ISO NW40 KF FLANGE, PRESSURE RELIEF VALVE Part Number: 113160, rated for 0.5 psi)

Temperature range	Components needed
< room temperature	Liquid nitrogen Dewar, T96 Liquid nitrogen pump. Vacuum operation only.
Room temperature-250 °C	Liquid nitrogen Dewar, T96 liquid nitrogen pump. Operation under vacuum or inert atmosphere only.

Staff will inspect the stage and especially the tubing for any defects at each installation.

If liquid nitrogen is needed, beamline staff or qualified users will fill the 2L Dewar approximately 2/3 full and replace the lid with the siphon attached, but will not fasten the catches until liquid nitrogen stops bubbling. Note that when removing the Dewar lid, it is important to place it with the black capillary tube pointing upwards to avoid damaging or creasing the tubing! Staff will wear appropriate PPE, including gloves and faceshields, when handling liquid nitrogen. Staff should also wear long sleeves, cuffless trousers and shoes with non-absorbent (leather) uppers.

Staff will make sure that the pedestal (X95 post) for the Dewar is secured in place. Staff will position the Dewar onto the pedestal and then connect the Dewar to the chamber via the vacuum feedthrough connection. Staff will also secure a second X-95 post near the Dewar pedestal to hold the Dewar lid while the Dewar is removed for filling. The Dewar must be covered with a foam lid while being moved.

Staff will disable the rotation stage within the GIXS chamber (vphif) or limit its rotational range to < 5 degrees when liquid nitrogen is being used. Tubing will not support rotation by 90 degrees.

2. Operation

Users and staff will wear safety glasses when operating the stage with the liquid nitrogen pump.

Users will return the sample stage to room temperature before venting the vacuum chamber and changing samples. For the GIWAXS top chamber, users will wear hearing protection and keep the Pilatus detector covered when venting/pumping the chamber. Users will handle samples with forceps.

Users will only operate the stage within the temperature range compatible with the installed components, consistent with discussion prior to experiments.

Before pumping vacuum on the sample chamber, users should place the lid on the chamber, but they should not clamp the lid tightly. This will provide a backup pressure relief mechanism.

3. Removal

Only qualified beamline staff will remove the Linkam stage and components from the GIXS instrument. Staff will be sure to handle components carefully and stow them properly to avoid damage.