High Brilliance Beamline ID02

1. Guidelines for sample preparation
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1. Guidelines for sample preparation

Liquid samples

The appropriate choice of sample cell is crucial for accurate SAXS measurements with fluid samples. To obtain the optimum scattered intensity, the sample thickness for many soft matter systems is in the range of 1.5 – 2.5 mm at 12.460 keV (standard energy at ID02 beamline). However, it may not be possible to use this optimum thickness in all cases because of multiple scattering at low angles and loss of angular resolution at wide angles.
The absorption and scattering by the empty sample cell should be as low as possible. This implies thin walls (10 µm) made of low density materials but at the same time providing sufficient uniformity and mechanical stability.

Samples with water-like viscosities

Commonly thin walled capillaries of glass, quartz or even polycarbonate are used as containers for samples with water-like viscosity. Accurate background subtraction is delicate because of the variations in scattering of the cell wall as well as the thickness of the sample at different positions. These uncertainties in background subtraction can be eliminated by using a flow-through cell (e.g., a quartz capillary of wall thickness 10 µm and diameter 2 mm). This set-up allows the background and the sample scattering to be measured at the same position.

Sample preparation:

- The samples can be provided in suitable vials with volumes of at least 0.05 ml – samples will be loaded to 2 mm quartz-capillaries during the experiment
  → measurements in *flow-through cell*
- Samples can also be prepared in pre-filled quartz-capillaries of 1.5 mm or 2 mm diameter (please take note of larger uncertainties in the background subtraction)
  → measurements in *capillary sample changer*
- For temperature studies below 5 °C and above 80 °C; samples have to be provided in quartz capillaries of 1 mm diameter
  → measurements in *Linkam heating stage*

In order to perform accurate background subtraction, you should provide the appropriate background samples as well (e.g. buffer solutions).

Paste-like samples

For viscous samples, cells with thin flat windows made of mica or Mylar or Kapton are preferred. Mica windows (thickness 10 – 15 µm) are usually found to be better for low angle SAXS but crystalline diffraction might be seen in the WAXS measurements.

Sample preparation:

- Appropriate Ni coated aluminium cells are available at the beamline and can be filled before the experiment.
  → measurement in *four position sample changer* or *industrial sample changer*
- Mica windows can be obtained from

  RICHARD JAHRE GMBH (http://www.jahregmbh.de)
  Mica windows / Glimmerscheiben (diameter: 16 µm): WN 1.1-R0x16-3 Ausf. 2 thickness 10 – 20 µm,Q.:41

  or can be purchased at the ESRF in packs of 100 windows (industrial users only)
- Sample cells can also be purchased at the ESRF for regular users

- An extra charge will be applied for mail-in industrial samples if the sample cells have to be filled at the ESRF

Powders

Powder samples should be prepared as thin and tightly packed columns.

Sample preparation:
- In quartz-capillaries of 1 mm diameter
  → measurements in *capillary sample changer*
- Sandwiched between mica or Kapton windows in the central hole of a washer (hole diameter of about 5 mm, thickness 1 mm)
  → adapted sample holder
- In the round sample cells mentioned above with thin mica or Kapton windows
  → measurement in *four position sample changer* or *industrial sample changer*

- Make sure that the cells/capillaries are homogeneously filled
- An extra charge will be applied for mail-in industrial samples if cells are not pre-filled

Solid samples

Solid samples should be provided in form of thin films.

Sample preparation:
- Thin films: thickness between several microns and 1 mm depending on scattering power of the sample
- Films should be prepared with a round or rectangular shape (between 3 mm and 15 mm diameter/edge length)

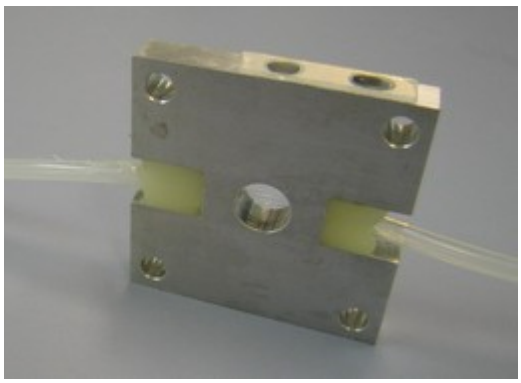
2. Sample environments

Standard sample environments

Flow-through cell

Two types of thin-walled capillaries are available: biocompatible polycarbonate or quartz-capillaries with a diameter of 2.0 mm. Using the flow-through cell allows reliable measurement of sample and solvent scattering of low scattering liquid samples (reliable background subtraction). A temperature range between 5 to 80 °C is accessible using a Peltier controlled stage.

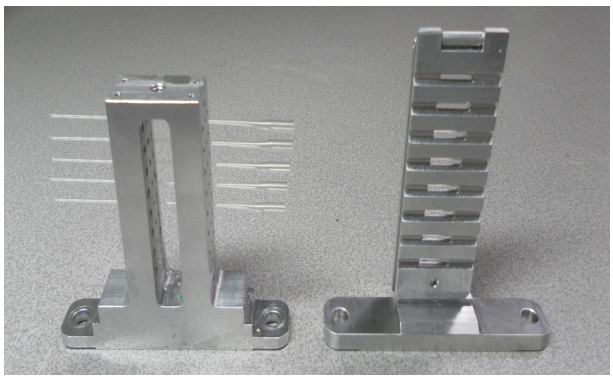
Only samples with viscosity up to a few Poise can be loaded.



Capillary sample changer

This set-up allows 8 sealed capillaries to be mounted of diameter ranging from 1 to 2 mm. The temperature can be varied from 5 to 80 °C using a Peltier controlled stage. Best adapted for measurements around 12.4 keV are quartz capillaries with a diameter of 1.5 or 2.0 mm.

Please make also sure to verify carefully the diameter of the capillaries before using the changer. This ensures faster loading of the samples and facilitates intensity calibration of the scattering curves.

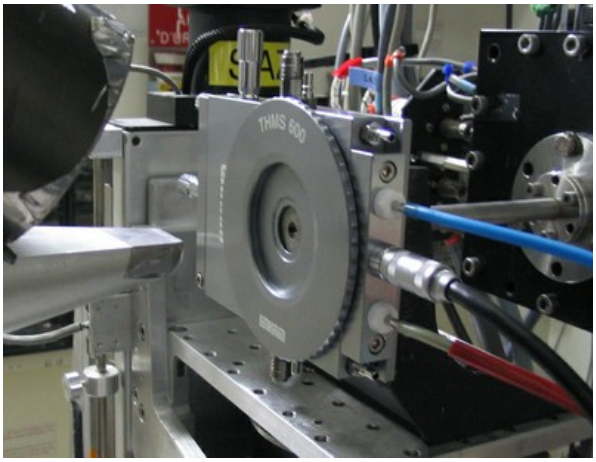


The set-up is less adapted for the study of low scattering liquid samples due the uncertainties in the background subtraction when using separate capillaries for sample and background (solvent, buffer, etc.).

Linkam heating stage

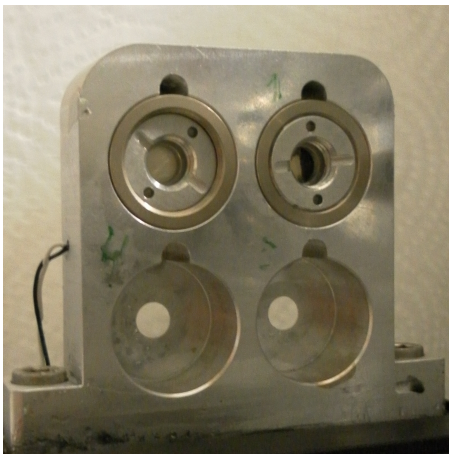
(THMS600/TMS94) This heating stage can perform fast temperature ramping and quenching (80 °C/min, nominal stability of 1 °C) primarily in crystallisation studies. This set-up can also be used to change the sample temperature over a wide temperature range (-100 to +600 °C).

An adaptor to mount solid samples (in the form of a circular disc - diameter 3 to 10 mm) and liquid samples (in sealed capillaries of diameter 1 mm) is available.



Four-position sample changer

The temperature (5 to 80 °C) is controlled by a Peltier element. The samples are contained in liquid tight aluminium cells with thin mica, Mylar or Kapton windows as described above. The sample thickness can be 0.5, 1.0 or 2.0 mm.



30-position sample changer

A more advanced version of the above set-up has explicitly been designed for high throughput industrial experiments. 30 sample cells can be placed at the same time.

The temperature is controlled by Peltier elements and can be varied from 10 to 120 °C in less than 20 minutes. The samples are contained in liquid tight aluminium cells with thin mica, Mylar or Kapton windows as described above. The sample thickness can be 0.5, 1.0 or 2.0 mm.

Further sample environments

not available for mail-in services – please contact us for more details

Stopped-flow rapid mixing device

The device is based on a commercial instrument (BioLogic, SFM-400) with four stepper motor driven syringes and three mixers. A home-made SAXS/ WAXS flow-through quartz capillary cell (diameter ~1.6 mm) is adapted to the mixing chamber. The flow can be stopped with millisecond accuracy using a solenoid driven stopper. The dead time of mixing is about 2 ms for flow rates exceeding 7 ml/sec.

A minimum sample volume of 2 ml per syringe is required (dead volume ~100 µl).

Stress controlled rheometer

The stress controlled rheometer is a commercial Thermo-Haake Mars II and the shaft drives an X-ray transparent couette cell. The concentric cylinders (inner diameter 20 mm, outer diameter 22 mm, and height 40 mm) are machined out of polycarbonate (lower background) or aluminium (for higher temperatures up to 170 °C and good organic solvents) with wall thickness around the beam position ~100 µm.

The minimum sample volume required is about 2.5 ml. For highly viscous samples, a plate-plate type shearing geometry is available.

Magnetic field

A magnetic field can be applied based on permanent magnets arranged periodically as in a synchrotron insertion device (undulator). The field can be varied from 0.1 mT to 1.5 T. The direction of the magnetic field can be either perpendicular or parallel to the beam direction.

An specific oven adapted for both capillaries (1 mm) and flat cells is also available to perform measurements in the range of temperature (25 to 300 °C).

3. Shipping samples to and from the ESRF

sending from/to ESRF

You have to use the ICAT Sample Tracking System.

ICAT (http://www.esrf.fr/ICAT) allows to create a parcel for a scheduled experiment session.

The sender has to be either on the A-form for the session (SMIS), or be the main proposer or one of the co-proposers on the proposal. In this case, he can add items like sample sheets or equipment to a shipment and **print a tracking code**, which needs to be **attached outside on the parcel**.

Updated informations on sample sheet and experiment session are automatically synchronized from SMIS to ICAT **after validation by the Safety group**.

Once the parcel has arrived at the ESRF Stores, the local contact will be notified via e-mail.

The ESRF label on the parcel should indicate the required storage condition such as room temperature, refrigerated, etc.

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returning from ESRF

If you want return shipment of your samples, then the ESRF return label should be furnished inside the parcel. Otherwise, samples will be safely disposed off.