

Ultimate experimental flexibility for SAXS/WAXS applications

# The right solution for every sample – SAXS samples stages from Anton Paar

Anton Paar offers a wide range of off-the-shelf high-quality and high-precision sample stages and holders for its SAXSpoint 5.0 and SAXSpace.

Whatever your application needs are, these sample stages and holders give you the utmost flexibility to characterize your nanostructured material under ambient and non-ambient conditions.

The sample stages precisely control temperature, atmosphere, and pressure. They position the sample accurately, which is of special importance for sample mapping and GISAXS studies. All sample stages are fully integrated in the software and hardware of SAXSpoint 5.0 and SAXSpace and provide:

- Automatic recognition and configuration of any sample stage with Stagemaster
- Self-alignment of the sample stage together with all components with the X-ray beam with TrueFocus
- Automatic g-range optimization of the sample stage within the measuring chamber with TrueSWAXS

# Choose from the following classes of sample stages and holders according to your application needs:

# Temperature-controlled sample stages and holders

For single samples

# Specific sample stages

GISAXS Stage Rotation Module Shear Cell SAXS

# Method combinations with SAXS and customized solutions

UV/Vis-SAXS Cell FTIR-SAXS Cell

# **High-throughput solutions**

Autosampler



For multiple samples

Tensile Stage RheoSAXS Module Humidity Stage

SEC-SAXS Customized solutions

# Temperature-controlled stages and holders for single samples

# Analyze your samples in a temperature range from -150 °C to +600 °C with the highest temperature accuracy.

The temperature-controlled TCStages are the sample stages of choice for sequential single SAXS and WAXS measurements. The TCStages are easily installed in the SAXSpoint 5.0 and SAXSpace instruments. They ensure accurate temperature control during heating and cooling within the temperature range specified. The inside temperature measurement during operation is performed with a Pt 100 sensor according to DIN 43760.

Using the TCStage you benefit from a broad variety of sample holders to measure high- and lowvolume liquid samples, solids, and paste samples. Special sample holders to increase sample throughput, to investigate samples under defined pressure, or to prevent sedimentation are also available. To keep flexibility high you can use disposable capillaries filled with sample.

## **Benefit from:**

- Accurate temperature control
- Fast cooling/heating rates
- Maximum thermal contact between the heating/ cooling block and the sample holder
- Counter-cooling of the TCStages by coolingwater circuit of SAXSpoint 5.0 and SAXSpace
- Versatile sample holders

TCStage 150 1 Temperature range: -30 °C to +150 °C

TCStage 350 1 Temperature range: ambient to +350 °C

TCStage 600 (2) Temperature range: ambient to +600 °C

# TCStage 350-c 🔞

Temperature range: -150 °C to +350 °C Heating: with a highly precise resistance heater Cooling: with liquid nitrogen



# Sample holders for liquids

## Quartz capillary

- +5 °C to +90 °C

# μCell

- +5 °C to +90 °C

## FlowCell

- +5 °C to +90 °C

## **TubeCell**

- Polymer flow-through cell
- non-removable samples
- -30 °C to +150 °C

# Sample holders for solids and viscous samples

# TCS sample holder for solids

- -150 °C to +600 °C

# PasteCell N

- -150 °C to +300 °C

# **Special sample holders**

# RotorCell

- during measurement
- -30 °C to +120 °C

# **High-pressure Cell**

- non-explosive gases

# **TCS Capillary Holder**

- disposable capillary

Δ

- Standard capillary for liquid samples

- Low-volume capillary for precious liquid samples - Minimum sample volume: 8 µL

- High-throughput measurements - Low-noise 1 mm quartz capillary - Automation with an autosampler unit

- High chemical and thermal stability - For highly contaminating, non-cleanable, or - Automation with an autosampler unit

- For pastes, gels, and powders - Sample is placed between exchangeable windows (Kapton or other materials)

- For sample spinning to prevent sedimentation - Disposable polymer capillaries

- For studies under pressures up to 100 bar - Usable gases: air, inert gas, CO<sub>2</sub>, methane, other - 0 °C to +90 °C (100 bar up to 60 °C)

- Holder for disposable capillaries - Temperature range depends on the used

# Temperature-controlled stages and holders for multiple samples

# Multiple samples in the spotlight of X-rays from -150 °C to +350 °C

Temperature-dependent investigations of different types of samples belong to the standard SAXS/ WAXS measurements to characterize materials' properties. With Anton Paar's modern sample stages, Heated/Cooled Sampler and Heated Sampler, it is possible to do a fully automatic scan of up to 20 different samples in one go. The sample holder is easily mounted to the sample stage.

SAXSpoint 5.0 recognizes the Heated/Cooled Sampler and Heated Sampler automatically when plugged in the measuring chamber. Benefit from an easy setup of your experimental details by predefining in SAXSpoint 5.0's control software in which order, at which temperature, and at which sample-to-detector distance (SDD) your samples are to be measured.

Standard sample holders for multiple measurements of solids, foils, films, powders, gels, pastes, and liquids are available. To extend your experimental flexibility Heated Sampler can be equipped with a Cryo Module to extend the covered temperature range for cryogenic temperatures down to -150 °C.

# Benefit from:

- A wide temperature range of -150 °C to +350 °C
- Diverse sample holders to analyze multiple solids, gels, pastes, and liquids
- High degree of temperature accuracy
- Quick heating/cooling rates
- Maximum heating homogeneity at all sample positions
- Automated multiple sample measurement of up to 20 samples
- Plug and play feature for simple installation



Heated Sampler 1 Temperature range: ambient to +350 °C

Heated/Cooled Sampler 2 Temperature range: -10 °C to 120 °C

Cryo Module for Heated Sampler 😣 Temperature range: -150 °C to 350 °C

# Sample holder for multiple samples - solids

Multiple solid sample holder - Loading of up to 20 samples

# Special sample holders

SiN Cell

In case special customized multiple sample holders are required, e.g. with a different number of sample positions, Anton Paar supports you in designing and manufacturing one to suit your requirements.

## Sample holders for multiple samples – liquids

## Multi-cuvette holder

- Loading of up to 5 single sample holders - Optional use of quartz capillary, µCell, PasteCell N, FlowCell, TubeCell, and sample holder for solids - Utmost flexibility to insert different single sample holders in the same multi-cuvette holder

## Holder for disposable capillaries

- Loading of up to 5 disposable capillaries - Separate holders for horizontal or vertical positioning of disposable capillaries

## Sample holder for multiple samples - pastes/gels

PasteCell HS - Loading of up to 4 samples

- Unique low-parasitic flow-through cell for
  - measurement of liquids
- Temperature range: -10 °C to 120 °C
- Ensures an extremely low scattering background,
  - which is beneficial for weakly scattering samples

# Specific sample stages

Get the best out of your sample with dedicated sample stages for special investigations.







# Grazing-incidence small-angle X-ray scattering (GISAXS)

Get insights into the surface nanostructure of thin-film samples GISAXS applications include the characterization of mesoporous thin films, surface-deposited nanoparticles, metal deposits on oxide surfaces, and - more recently - soft matter systems such as polymer/block copolymer thin films and biological materials which are attached to surfaces.

## **GISAXS Stage**

Anton Paar's GISAXS Stage is a high-resolution and high-precision motorized sample stage for performing GISAXS/GIWAXS/GIXD (grazing-incidence SAXS/WAXS/diffraction) studies of thin-film samples. It is equipped with a  $\Phi$ -rotation module allowing sample rotation from 0° to 345°. The samples can be tilted in an angular range of  $-4^{\circ}$  to  $+5.6^{\circ}$  to vary the penetration depth of the incident beam. Anton Paar's GISAXS Stage can be equipped with the GISAXS Heating Module to perform temperature-dependent studies up to 500 °C or with the Heated/Cryo Module for covering a temperature range from -150 °C to +350 °C.

# Tensile studies combined with SAXS/WAXS investigations

Investigate stress/strain under precisely defined mechanical load Typical samples for tensile measurements are fibers, foils, and thin films for use in special tissues, clothes, cover or composite materials.

### **Tensile Stage SAXS**

Anton Paar's Tensile Stage SAXS is the only commercial sample stage specifically designed for in-situ SWAXS investigations. The Tensile Stage SAXS is used in transmission geometry and allows you to put samples under strain with a force of up to 600 N. You have the choice between two load cells depending on whether you need a high-resolution (0.01 N to 5 N) or a full-range (1 N to 600 N) load cell for your studies. In the Tensile Stage's software you are free to program complex force-controlled straining including cyclic strain and creep experiments. To cover the full possible temperature range for stress/strain studies Tensile Stage SAXS can be equipped with a Heated/Cryo Module for temperature-dependent measurements from -150 °C to 350 °C.

# Isotropic and anisotropic fiber and film studies

Fibers and films often require special measuring equipment so that a full picture of the sample from all angles can be obtained.

### **Rotation Module SAXS**

The Rotation Module SAXS enables measurement during rotation of the sample perpendicular to the X-ray beam at ambient temperatures. If the sample is anisotropic you can use the Rotation Module SAXS to align the sample in order to receive the

full scattering pattern.

# Specific sample stages

Combine complementary techniques in one setup: full rheological characterization or simple shear experiments during SAXS investigations as well as humidity studies to optimize your material for its intended use



# RheoSAXS – a unique combination of rheology and SAXS in one lab-scale instrument

RheoSAXS provides the unique chance to directly correlate structural changes recorded on the nanometer scale with macroscopic properties determined in rheological investigations. This method combination is of particular interest for people investigating modern smart materials exhibiting uncommon properties. Typical application fields are cosmetics, colloidal dispersions, polymers, nanomaterials, and nanomicromaterials.

# **RheoSAXS Module**

The RheoSAXS Module comprises a dedicated cylindrical Rheo-SAXS measuring cell for axial and tangential measurement mounted into SAXSpoint's 5.0 measuring chamber and the DSR 502 dynamic shear rheometer measuring head.

The RheoSAXS measuring cell is temperature-controlled in a range from -10 °C up to 90 °C. The fully integrated DSR 502 measuring head allows all standard measuring capabilities e.g. rotational and oscillatory measurement modes.

You further benefit from accurate measurements of even low-viscosity dilute suspensions with a minimum torque of 10 nNm (in rotation) or 2 nNm (in oscillation). For in-depth analysis of rheological results with the RheoSAXS Module use the RheoCompass software.



# Shear rate and nanostructure investigations

For quick and easy shear measurements Anton Paar offers its new Shear Cell SAXS for liquids such as emulsions, dispersions, or suspensions. It is used to study the change in the nanostructure of complex fluids under defined shear rates and temperature. Typical applications are polymers, liquid crystals, and other binary liquids.

# Shear Cell SAXS

Anton Paar's Shear Cell is a special sample holder mounted to the Heated/Cooled Sampler or Heated Sampler. You benefit from quick and simple shear-rate-dependent investigations, which can be performed in the range of 0.001/s to 15000/s with a varied frequency from 0 Hz to 20 Hz and a temperature range from ambient to +350 °C.

Choose defined heating rates from 0.01 °C/min up to 60 °C/min depending on your application needs. The control and the programming of the shear experiments are fully integrated in the SAXSpoint 5.0 software. You get all the structural information at a defined shear at once.



# Humidity and nanostructure investigations

Investigations into structural changes under the influence of humidity are important for determining e.g. a sample's stability during storage and transport or under the conditions for its intended use and over its life cycle. Degeneration of materials and the exchange of the affected materials are often costly, so thorough investigations under controlled conditions are important.

# Humidity Stage

To perform temperature- and humidity-dependent studies you need Anton Paar's dedicated Humidity Stage. Designed for powders and solids (films, fibers) it enables measurements under humid conditions at 5 % to 95 % relative humidity (RH) at temperatures ranging from +10 °C to +60 °C and under vacuum, air, or inert gas in a temperature range from +10 °C to +110 °C.

The Humidity Stage is operated with the Modular Humidity Generator which exactly controls the relative humidity. Highly precise temperature and humidity sensors close to the sample guarantee uniform and well-defined humidity conditions and maximum thermal homogeneity. You benefit from a sophisticated design which provides maximum interaction of sample and humid atmosphere.

# Method combinations with SAXS and customized solutions

Combine SAXS with other methods to obtain data from both measurements in one go.



# **UV/Vis-SAXS** combination

UV/Vis spectroscopy is a common method to detect the molecule type and impurities. Combined with SAXS all the structural information such as size and molecular weight is received in parallel from the same sample.

This is of advantage during e.g. protein screening as it saves time and increases throughput by measuring both UV/Vis and SAXS in one go. Typical applications for the UV/Vis-SAXS combination can be found in biosciences and the pharmaceutical field.

The Anton Paar equipment for inline spectroscopy enabling **simultaneous UV/Vis and SAXS measurements** consists of a high-performance UV/Vis spectrophotometer and a special combined inline SAXS and UV/Vis measuring stage. The UV/Vis-SAXS stage is designed so that the fiber-optic microprobe is inserted into the inline UV/Vis-SAXS stage and is completely immersed in the sample. In that way accurate results are ensured. Measurements can be performed in a temperature range of -10 °C up to +120 °C.



# **SEC-SAXS** combination

Size exclusion chromatography (SEC) is used for fast purification of e.g. proteins, peptides, and nucleic acids and is a typical step in each lab working with proteins before starting any further analysis. In parallel to determining proteins' structure it is important to know which fraction contains the desired sample.

Anton Paar's setup for online SEC-SAXS uses Anton Paar's standard FlowCell which is connected to a high-performance chromatography system. The UV signal from the chromatography system triggers the start of the SAXS measurement once a protein is detected. Thus the time stamps of both measurements are perfectly synchronized.

Benefit from the flexibility to choose whether the sample should be kept and collected in an optional fraction collector for further analysis or discarded after the SAXS measurement. You can also run the SEC system in offline mode, which gives you the freedom to collect the fractions of interest without blocking your SAXS system. This can be used for additional independent SAXS measurements during purification cycles on the chromatography system.



# **SAXS-FTIR Cell**

FTIR absorption spectra deliver a molecular "fingerprint" of the sample. When FTIR is combined with SAXS you benefit from the samples' nanostructure information in parallel after just one measurement on the sample.

Anton Paar's unique combination of SAXS and FTIR ensures **simultaneous measurement of FTIR and SAXS**. This is realized by a specially designed SAXS-FTIR cell which is mounted on the Heated/Cooled Sampler of Anton Paar's SAXSpoint 5.0 system offering a temperature range from -10 °C to +120 °C and maximum temperature homogeneity.

The FTIR probe is encapsulated and thus made vacuum-tight and located as close as possible to the sample surface, guaranteeing excellent measuring results. While the FTIR cell is also vacuum-tight, SAXS-FTIR measurements are performed under atmosphere. Anton Paar's SAXS-FTIR cell is designed for solid samples, foils, and powders and contains three different special sample holders.



# **Customized solutions**

Thanks to the longstanding experience in design and production of sample environments (stages and holders) for SAXS and XRD systems Anton Paar can provide expert support for the implementation of customized sample holders/ chambers as well as for the integration and combination with other instruments.

We also offer the design and implementation of customized environments according to customer needs.

# High-throughput autosampler solution



# **ASX autosampler for liquids**

Measure up to 192 liquid samples in one run with the ASX autosampler at ambient conditions. With the ASX c version you can measure down to 4 °C. This is especially important for temperature-sensitive samples in order to protect them from denaturation and evaporation, ensuring consistent and reliable results.

The autosampler is fully integrated into the SAXS systems' hardware and software providing high userfriendliness. Different washing cycles can be programmed to avoid any contamination of samples during measurement. The autosampler is designed for minimum sample volumes down to 10  $\mu$ L.

The ASX autosampler is used in combination with Anton Paar's flow-through cells such as the FlowCell, TubeCell or SiN Cell.

# Overview table

| Type of stage  | Stage                       | Sample holders   |   | SAXSpace     | SAXSpoint 5.0 |
|--|-----------------------------|--|---|--------------|---------------|
| Temperature-controlled<br>stages for single<br>samples   | TCS 150                     | Quartz capillary<br>FlowCell<br>PasteCell N<br>RotorCell<br>TCS capillary holder | µCell<br>TubeCell<br>TCS sample holder for solids<br>High-pressure cell | ~            | ~             |
|  | TCS 350                     | Quartz capillary<br>FlowCell<br>PasteCell N<br>RotorCell<br>TCS capillary holder | µCell<br>TubeCell<br>TCS sample holder for solids<br>High-pressure cell | ~            | ×             |
|  | TCS 350-c                   | Quartz capillary<br>FlowCell<br>PasteCell N<br>RotorCell<br>TCS capillary holder | µCell<br>TubeCell<br>TCS sample holder for solids<br>High-pressure cell | ~            | ×             |
|  | TCS 600                     | Quartz capillary<br>FlowCell<br>PasteCell N<br>RotorCell<br>TCS capillary holder | µCell<br>TubeCell<br>TCS sample holder for solids<br>High-pressure cell | $\checkmark$ | ~             |
| Temperature-controlled<br>stages for multiple<br>samples | Heated Sampler 2.0          | Multi-cuvette holder<br>PasteCell HS<br>SiN Cell                                 | Holder for disposable capillaries<br>Multiple solid sample holder       | ×            | $\checkmark$  |
|  | Heated/Cooled Sampler       | Multi-cuvette holder<br>PasteCell HS<br>SiN Cell                                 | Holder for disposable capillaries<br>Multiple solid sample holder       | ×            | $\checkmark$  |
| Specific sample stages                                   | GISAXS stage                |  |   | $\checkmark$ | $\checkmark$  |
|  | Tensile Stage SAXS          |  |   | $\checkmark$ | $\checkmark$  |
|  | Rotation Module SAXS        |  |   | $\checkmark$ | $\checkmark$  |
|  | RheoSAXS Module             |  |   | ×            | $\checkmark$  |
|  | Shear Cell SAXS             |  |   | ×            | $\checkmark$  |
|  | Humidity Stage              |  |   | $\checkmark$ | $\checkmark$  |
| Method combinations with SAXS                            | UV/Vis-SAXS                 |  |   | ×            | $\checkmark$  |
|  | SEC-SAXS                    |  |   | $\checkmark$ | $\checkmark$  |
|  | SAXS-FTIR                   |  |   | ×            | $\checkmark$  |
| High-throughput<br>autosampler solution                  | ASX autosampler for liquids |  |   | $\checkmark$ | $\checkmark$  |

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