

# Sample Stages

## Overview

XRDynamic 500

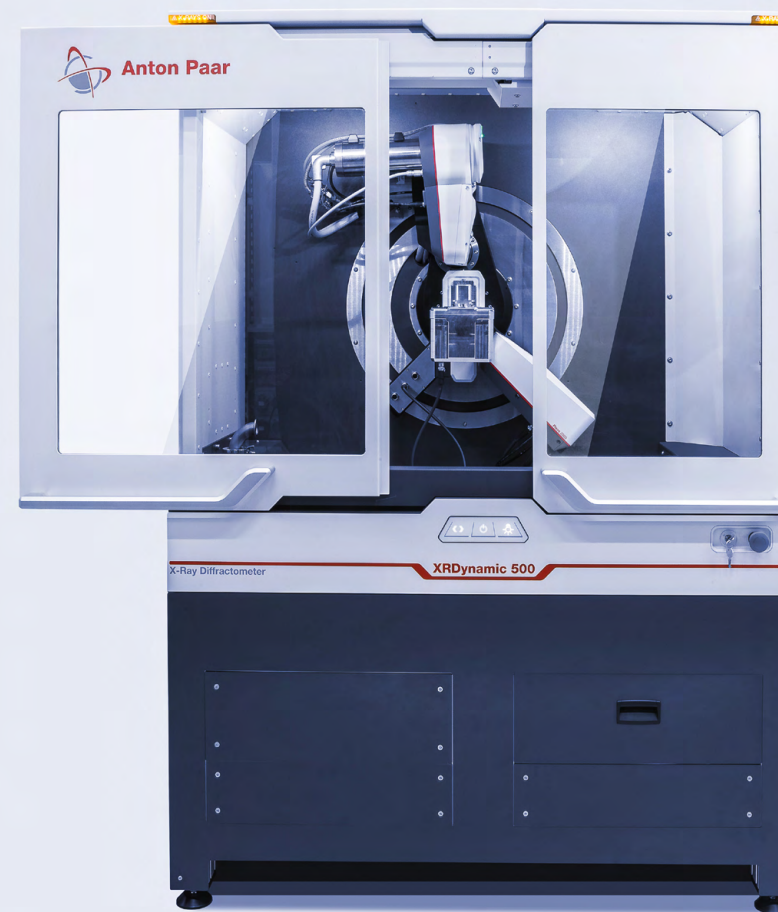


# A Solution for Any Application

XRDynamic 500, with its wide range of sample stages and components, is your future-proof diffractometer for any kind of XRD application. Whether you perform measurements in reflection, transmission, with capillaries, or under non-ambient conditions, XRDynamic 500 offers sample stages for every eventuality.

Almost all stages are mounted on a motorized Z (height alignment) stage, which is included with XRDynamic 500 as standard. Convenient component recognition and highly precise, automatic alignment routines for all ambient and non-ambient stages allow fast exchange between different configurations, ensuring that you can always measure with the optimized configuration for your current sample.

**Whatever the sample and application, look no further than XRDynamic 500.**



**XRDynamic 500**

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X-RAY ANALYTICAL  
SOLUTIONS



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**FIXED STAGE** (Reflection, Transmission)  
**SAMPLE SPINNER** (Reflection, Transmission)  
**CAPILLARY SPINNER** (Transmission)  
**XY STAGE (INCL. AUTOSAMPLER)** (Reflection, Transmission)  
**EVAC MODULE (FOR XRD AND SAXS)** (Reflection, Transmission)  
**HTK 1500** (Reflection, Transmission, High Temperature, Low Temperature)  
**HTK 1200N** (Reflection, Transmission, High Temperature, Low Temperature)  
**HTK 16N/2000N** (Reflection, High Temperature)  
**XRK 900 (INCL. HIGH PRESSURE)** (Reflection, High Temperature)  
**TTK 600** (Reflection, Transmission, High Temperature, Low Temperature)  
**CHC plus+ (INCL. HUMIDITY)** (Reflection, High Temperature, Low Temperature)

Reflection    Transmission    High Temperature    Low Temperature



# Flexible Sample Stages

## for All Samples



### Fixed stage | Sample spinner (reflection/transmission)

The fixed sample stage is delivered with XRDynamic 500. During measurement, the sample is kept in a fixed position, which makes XRD measurement of all sample types straightforward.

With the sample spinner, measure all sample types and spin the sample during measurement for improved measurement statistics. To study samples with different orientations, you can also conduct measurements at defined  $\phi$  positions.

Both are standard sample stages for powder XRD measurements using a wide variety of sample holders with standardized dimensions. Both let you measure in reflection and transmission geometry, depending on the sample holder, which gives you maximum measuring flexibility. From standard frontloading powder holders and gas-tight holders to transmission holders, the stages are compatible with the full range of XRDynamic 500 sample holders.

Conveniently, both the fixed sample stage and sample spinner are mounted on the Z (height alignment) stage of XRDynamic 500 so you can automatically align samples of differing dimensions.

### Typical applications

- XRD of all types of sample materials with a wide range of sample holders
- Grazing incidence XRD (GIXRD) of solids and thin layers
- Residual stress analysis of solids and complete work pieces

### Technical data

Sample types: powders, solids, thin films, foils, fibers  
Measurement geometry: reflection and transmission  
Sample spinning option

# Capillary Measurements

## Made Easy



### Capillary spinner stage

With the capillary spinner stage, measure samples in both open-ended funnel capillaries and sealed capillaries in transmission geometry. The capillary is spun during measurement to ensure excellent measurement statistics, and it's pre-aligned to guarantee a constant sample position in the X-ray beam.

The alignment microscope for the capillary spinner stage is delivered with the stage and makes pre-alignment of the capillary easy and stress-free.

When used in tandem with focusing beam optics, capillary measurements on XRDynamic 500 provide an unbeatable mix of both excellent intensity and outstanding resolution. This is accomplished by maximizing the X-ray intensity on the sample while focusing X-rays to a small spot on the detector. Since you can use all detectors for XRDynamic 500 near or in the direct X-ray beam without risk of damage, there's no need for beam stops that could restrict the maximum  $2\theta$  range.



### Typical applications

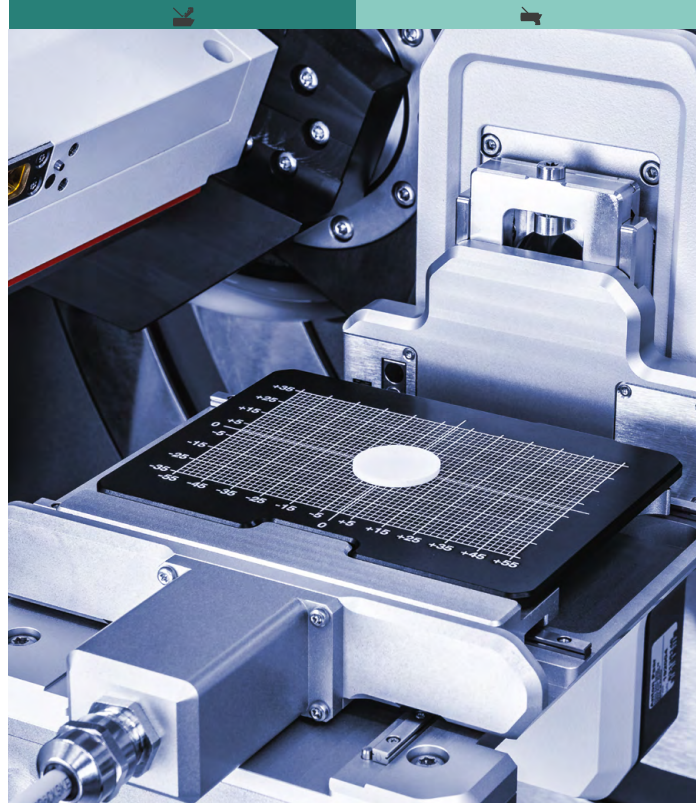
- XRD of organic or sensitive materials
- Measurement of small sample quantities
- Crystal structure solution of new materials
- PDF analysis of amorphous or semicrystalline materials

### Technical data

Sample types: powders, fibers, slurries, liquids  
Measurement geometry: transmission  
Sample spinning option



# Solutions for Solid and Thin Film Samples



## XY stage

The XY stage for XRDynamic 500 is a versatile sample stage that can be configured with various adapter plates to utilize the stage for either bulk, solid/thin film samples, or as an automatic sample changer.

When configured with the adapter plate for solid samples, the XY stage is suitable for measuring samples with dimensions of up to 110 mm x 75 mm, thicknesses of up to 28.5 mm, and weights of up to 3 kg. Samples can range from bulk solid materials and complete workpieces to thin films deposited on various substrates.

The XY stage lets you perform scans at different positions on the sample surface for phase mapping measurements in the X, Y, or XY directions.

The XY stage is mounted on the Z (height alignment) stage of XRDynamic 500, which facilitates automatic alignment of both the sample height and surface tilt. This ensures precise sample positioning no matter the sample size.

## Typical applications

- XRD of solid and bulk samples
- Grazing incidence XRD (GIXRD) of solids and thin layers
- Residual stress analysis of solids and complete workpieces
- Phase mapping across a sample surface

## Technical data

Sample types: powders, solids, thin films  
Measurement geometry: reflection

# Maximum Efficiency Sample Changers



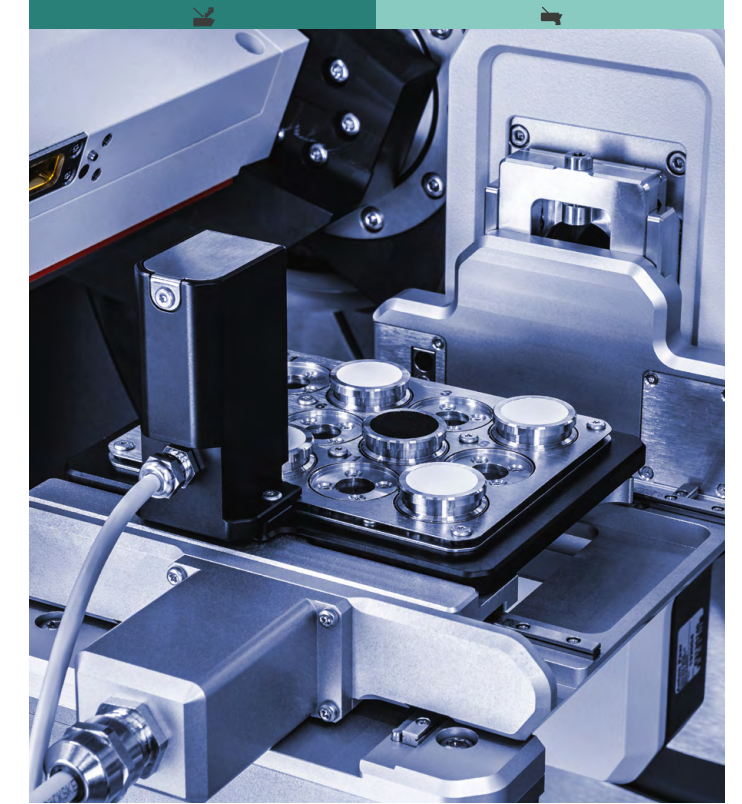
## Autosampler option for XY stage

Reconfigure the XY stage with several different adapter plates to transform it into a sample changer for up to 12 samples. For all sample changer adapter plates, perform measurements in both reflection and transmission geometry. You can even spin samples during measurement for excellent measurement statistics.

As the XY stage is mounted on the Z (height alignment) stage, each sample can be aligned before measurement to ensure precise peak positions even with samples of varying dimensions.

The autosampler options for XRDynamic 500 maximize the potential of the automated TruBeam™ concept, letting you measure different sample types with different instrument configurations in one run – no operator intervention needed.

With TruBeam™, measure one sample with a standard powder XRD configuration and the next one with a grazing incidence XRD setup. Or switch from reflection to transmission geometry from one sample to the next, maximizing measurement efficiency. Our components recognition technology reduces the chance of operator error for all TruBeam™ optics.



## Typical applications

- High-throughput XRD of all types of sample materials
- Automated batch measurement of different sample types using different instrument configurations

## Technical data

Sample types: powders, solids, thin films, foils, fibers  
Measurement geometry: reflection and transmission  
Sample spinning option



# Unique Evacuated Chamber for XRD and SAXS



## EVAC module

Our patented EVAC module lets you measure XRD and SAXS on a diffractometer like never before. With over 95 % of the beam path under vacuum, the EVAC module delivers the best-possible signal-to-noise ratio by almost completely eliminating parasitic air scattering. Since there's a significantly reduced absorption of X-rays in vacuum, you can measure faster, while still enjoying the full usable angular range of XRDynamic 500.

The outer housing tilts with the detector while the sample remains horizontal so there's no risk of the sample moving. Suitable for measurements in both reflection and transmission geometries, you can configure the EVAC module for both XRD and SAXS measurements via specialized sample holder mounts to offer maximum flexibility in how you measure.

The dedicated SAXS optics incorporated in the SWAXS configuration of the EVAC module let you reach a resolution of  $q_{\min} = 0.05 \text{ nm}^{-1}$ . With a data quality close to that of a stand-alone line-collimation SAXS system, XRDynamic 500, combined with the EVAC module, is a true 2-in-1 system for both XRD and SAXS.

## Typical applications

- Ultra-low background measurements of all types of sample materials
- Identification and quantification of minor phase fractions
- SWAXS measurements for analysis of nanomaterials
- Determination of particle size, shape, and internal structure
- PDF analysis of amorphous and semicrystalline materials

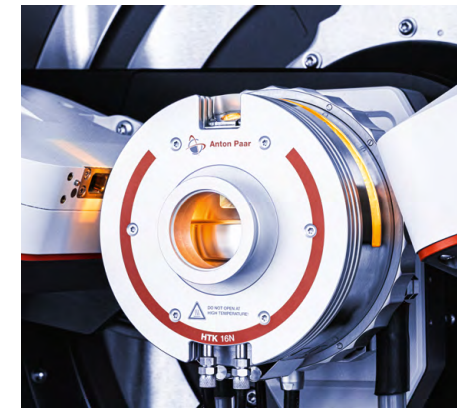
## Technical data

Samples types: powders, solids, thin films, foils, fibers, liquids, dispersions, pastes

Measurement geometry: reflection and transmission

# An Instrument Designed for Non-Ambient XRD

With over half a century of experience in non-ambient XRD, we understand what's needed for a successful non-ambient XRD experiment. Our philosophy is to make non-ambient XRD measurements as straightforward as any standard ambient XRD measurement. The non-ambient XRD concept of XRDynamic 500 makes the most of our experience and expertise in this specialized field to deliver the best non-ambient XRD solution possible.



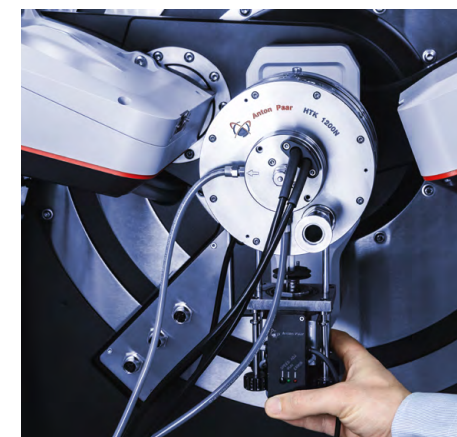
## The largest range of non-ambient XRD attachments on the market

With the broadest portfolio of non-ambient stages on the market, our non-ambient XRD attachments offer the widest specifications range. Conduct XRD measurements over a temperature from  $-190 \text{ }^\circ\text{C}$  to  $+2,300 \text{ }^\circ\text{C}$ . Do this in vacuum or under different gas atmospheres, under reactive gas conditions, and with controlled humid atmospheres.



## Built-in non-ambient control unit to simplify experimental setup

XRDynamic 500 features a built-in CCU (combined control unit), compatible with all available non-ambient XRD attachments, to simplify experiment setup. The CCU is fully integrated into XRDynamic 500's XRDdrive instrument control software so you can seamlessly set up non-ambient measurement plans with various temperature set points and heating rates. All non-ambient XRD attachments are automatically detected via component recognition.



## Convenient non-ambient connections in the instrument housing

XRDynamic 500 also features convenient non-ambient supply connections built into the instrument housing to further simplify your non-ambient experiment setup. Connections for cooling water, compressed air, and gas/vacuum supply means that all of the most commonly required connectors are already present in the instrument and just need to be plugged in. For more specialized connectors, cables/hoses can be easily fed into the wide opening on the side of the instrument to make experiment setup as easy as possible, no matter the conditions.



# Environmental Heating for Homogeneous Sample Temperature



## HTK 1500

HTK 1500 High-Temperature Chamber is an environmental heater for high-temperature X-ray diffraction (XRD) that allows you to heat all types of sample materials - including crystalline powders and bulk samples - homogeneously up to 1,500 °C. The position of the heating rods, together with a highly accurate temperature measurement by the use of a thermosensor inside the ceramic sample holder, makes HTK 1500 the only 'true' environmental heater for XRD up to 1,500 °C.

Two versions of HTK 1500 are available: a static version and a version comprising a sample spinning option. Sample spinning guarantees the highest-quality XRD data as preferred orientation and graininess effects due to improper sample preparation can be overcome.

With the possibility to change between different measurement geometries – reflection geometry and transmission geometry by use of a capillary extension – a wide variety of sample types can be investigated. The innovative slider for the sample holder allows for quick and reproducible mounting and dismounting of the sample holder, ensuring easy handling and sample exchange. The added benefit of chemically inert sample carrier materials means that any reactions with the sample are minimized.

## Typical applications

- Define lattice parameters and structure analysis
- Identification of thermal expansion coefficients
- Measure temperature-induced phase changes
- Studies of chemical reactions and processes
- Observe annealing, sintering, calcination processes, and more

## Technical data

Temperature range: 25 °C to 1,500 °C

Atmospheres: air, inert gas, vacuum ( $10^{-1}$  mbar)

Measurement geometry: reflection and transmission

Sample spinning option

Optional capillary extension

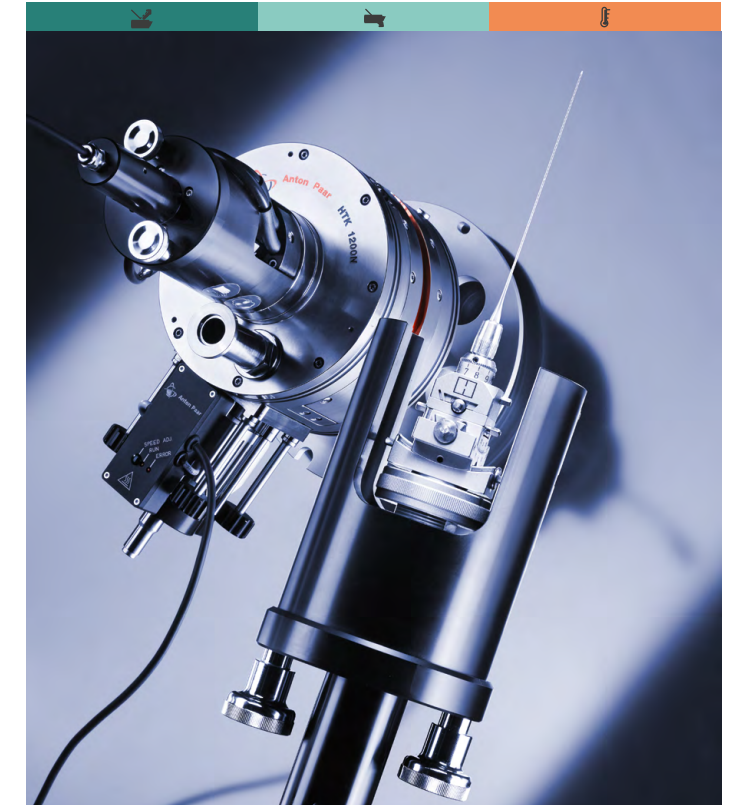


## HTK 1200N

HTK 1200N has been the attachment of choice for in situ XRD studies on flat samples up to 1,200 °C for many years. The smart capillary extension turns this well-known oven chamber into a powerful capillary heater. Due to its environmental heater, there's virtually no temperature gradient in the sample, even in samples that are up to 5 mm thick.

The sample spinning option provides highly random grain orientations, which are necessary for good diffraction data quality and subsequent profile fitting routines. The temperature sensor is located right underneath the sample in a protective ceramic sample holder, guaranteeing reliable and reproducible temperature measurements.

The alumina sample carriers can be easily exchanged and can accommodate various sample forms like powder samples, bulk samples, and thin layers. With the capillary extension, a variety of capillary holders and capillaries can be used, depending on the specific properties of particular samples.



## Typical applications

- Structure determination
- Coefficients of thermal expansion
- Investigation of phase diagrams
- Studies of chemical reactions
- Dynamic structure changes
- Lattice parameter measurements

## Technical data

Temperature range: 25 °C to 1,200 °C

Atmospheres: air, inert gas, vacuum ( $10^{-4}$  mbar)

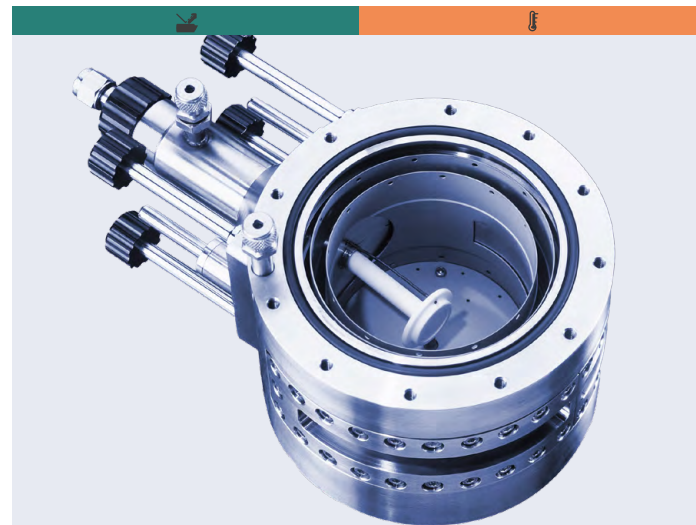
Measurement geometry: reflection and transmission

Sample spinning option

Optional capillary extension



# In Situ XRD Investigations of Solid-State Reactions



## XRK 900

XRK 900 is a well-proven reactor chamber for X-ray diffraction experiments up to 900 °C and 10 bar. Its robust and sophisticated design lets you perform studies of solid-state and solid-state gas reactions at high temperatures and pressures.

The special arrangement of the electrical heater inside the furnace guarantees the absence of temperature gradients in the sample. Two thermocouples reliably measure and control the sample temperature.

For solid-state gas reactions, defined atmospheric conditions are an important precondition. The design permits homogeneous flushing with reaction gas, as well as gas flow through the sample.

The housing can be heated up to 150 °C to prevent condensation of reaction products.

The sample spinning option provides highly random grain orientation, necessary for good diffraction data quality and subsequent profile fitting routines. Different sample holders made of stainless steel or ceramics are available.

### Typical applications

- Dynamic structure changes
- Studies of solid-state reactions
- Simultaneous investigation of structural and catalytic parameters of catalysts
- Analysis of materials that are unstable under ambient conditions
- Kinetic investigations of solid-state reactions

### Technical data

Temperature range: 25 °C to 900 °C

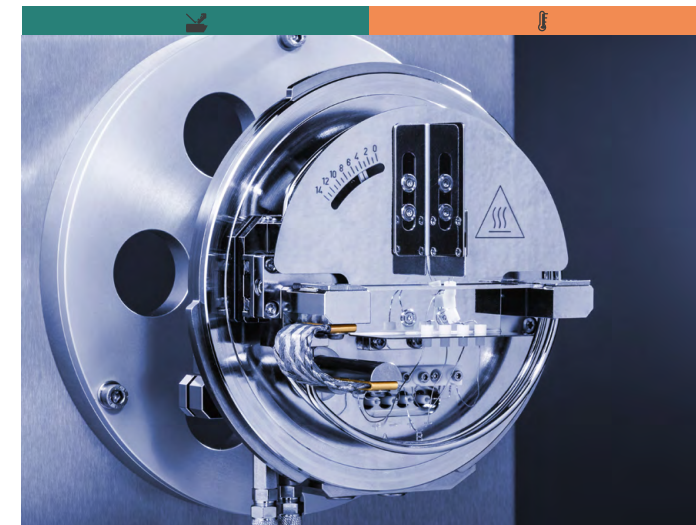
Pressure range: 1 mbar to 10 bar

Atmospheres: air, inert gas, certain reactive gases, vacuum (1 mbar)

Measurement geometry: reflection

Sample spinning option

# Strip Heating Up to 2,300 °C



## HTK 16N | HTK 2000N

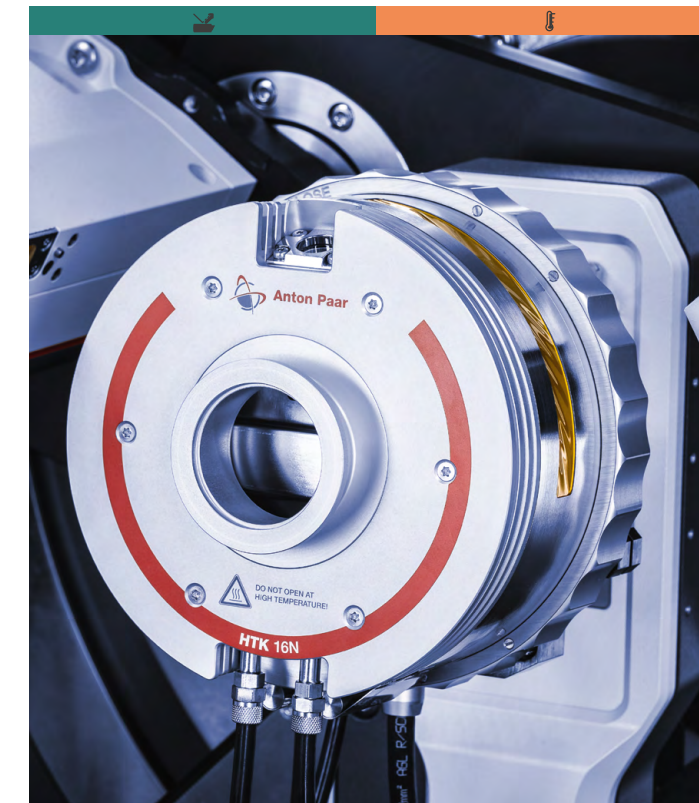
HTK 16N and HTK 2000N are strip-heater type chambers for X-ray studies with direct sample heating at very high temperatures up to 2,300 °C.

Investigations can be carried out under vacuum or in various gases depending on the experiment and the heating strip used (Pt, Ta, W, C, or others on request).

The design of the chambers is optimized for minimum temperature gradients in the sample. A thermocouple, spot welded onto the heating strip, provides reliable, accurate temperature measurement and control under all operating conditions.

Prestressing of the heating strip with a sophisticated linear stage guarantees high stability of the sample position over the complete temperature range. Integrated alignment slits allow exact height alignment of the strip at all temperatures.

The graphite heating filament with inert sample support plate offers the advantages of better temperature homogeneity in the sample and a higher chemical resistance. The front cover of the chamber has a bayonet catch for easy sample exchange.



### Typical applications

- Structure analysis
- Mineralogical studies
- Investigations of chemical reactions
- Aging processes
- Annealing
- Crystallographic characterization

### Technical data

Temperature range: 25 °C to 1,600 °C (HTK 16N)  
25 °C to 2,300 °C (HTK 2000N)

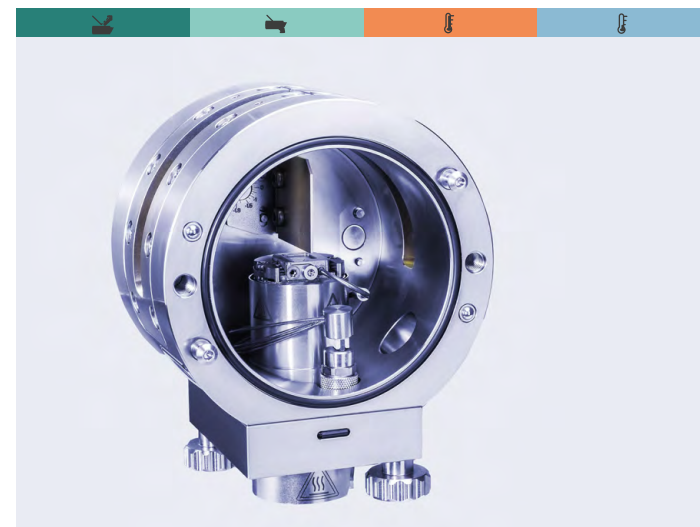
Atmospheres: air, inert gas; both up to 1,600 °C  
Vacuum (10<sup>-4</sup> mbar); up to 2,300 °C

Measurement geometry: reflection



# Low-Temperature XRD Studies

between -190 °C and +600 °C



## TTK 600

TTK 600 Low-Temperature Chamber is a versatile sample stage for X-ray diffraction studies in the temperature range from -190 °C to +600 °C.

Different types of sample holders ensure maximum flexibility concerning sample types and measurement geometries. The standard sample holder enables studies in reflection geometry. The optional capillary and transmission sample holders are used to investigate powders, foils, and paste samples in transmission geometry. Special sample holders for the in-operando investigation of coin cells (in reflection or transmission geometry) are also available.

The sample temperature is measured with an accurate Pt100 sensor right underneath the sample. The heat transfer between heater and all sample holders is optimized by design. The capillary and the transmission sample holders both work with additional convection heaters to enable fast temperature changes and improved temperature distribution. The temperature control algorithms guarantee maximum temperature stability and economical use of liquid nitrogen or compressed air.

## Typical applications

- In situ characterization of the crystal structure of pharmaceutical substances and food ingredients
- Changes in chemical composition during solid-solid and solid-gas reactions
- Accurate determination of coefficients of thermal expansion
- Investigation of polymer materials

## Technical data

Temperature range:

- 190 °C to +600 °C (liquid nitrogen cooling)
- 20 °C to +600 °C (compressed air cooling)

Atmospheres: air, inert gas, vacuum ( $10^{-4}$  mbar)

Measurement geometry: reflection and transmission

Optional capillary extension

# Studies under Controlled Temperature and Relative Humidity Conditions



## CHC plus+

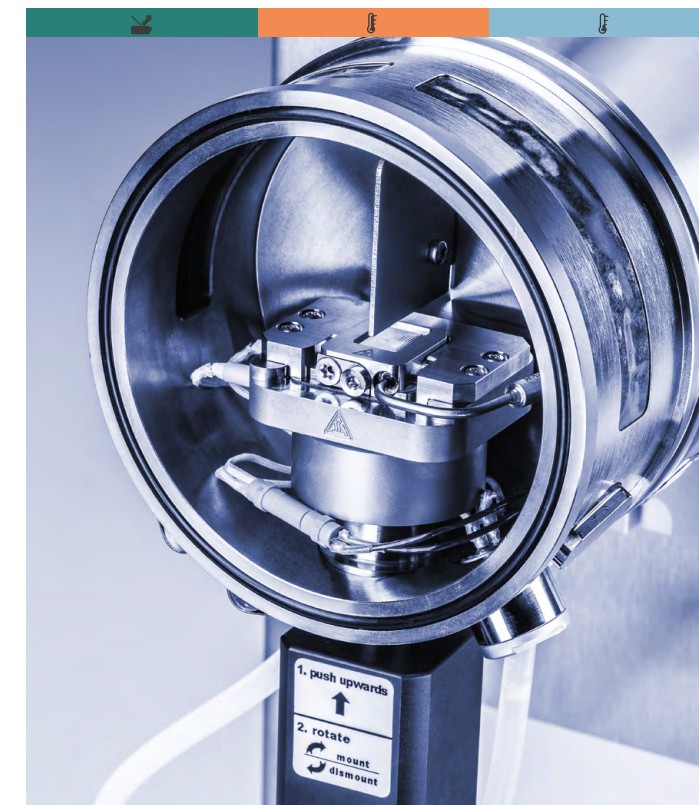
CHC plus+ is a unique combination of multipurpose CHC Cryo & Humidity Chamber and an advanced relative humidity (RH) generator for in situ X-ray diffraction studies at low and high temperatures from -180 °C to +400 °C as well as controlled humidity conditions.

The gas humidifier is mounted directly on the chamber, and the humidity is controlled with a calibrated RH sensor located inside CHC plus+ close to the sample.

The chamber housing is temperature-controlled with a water bath. This setup, together with the excellent control performance of the RH generator, provides uniform and well-defined humidity conditions around the sample without the risk of condensation.

All types of experiments can be done in one go without removing the sample. Sample conditioning without the need for realignment after sample exchange speeds up measurement preparations.

The large temperature range, combined with the possibility to control the humidity around the sample, makes CHC plus+ the ideal tool for XRD studies of temperature- and humidity-induced changes of crystal structures.



## Typical applications

- Temperature- and humidity-induced changes in pharmaceuticals and food ingredients
- Polymorphism in APIs
- Hydration/dehydration of zeolites and clays
- Hardening processes in building materials

## Technical data

RH range: 5 % RH to 95 % RH from +10 °C to +60 °C  
5 % RH to 70 % RH at +80 °C

Temperature range: -180 °C to +400 °C (vacuum)  
-120 °C to +300 °C (dry air)

Atmospheres: air (humid), inert gas, nitrogen, vacuum ( $10^{-2}$  mbar)

Measurement geometry: reflection



