Anton Paar

Non-Ambient
X-Ray Diffraction

Sample Stages Overview

Setting the Pace in **Non-Ambient XRD**

Non-ambient X-ray diffraction (XRD) has become an indispensable technique to understand the influence of temperature, atmosphere, or pressure on materials of any kind. Besides its relevance for conducting research, this knowledge is essential for optimizing technical processes and performing quality control in industry. Temperature is the most important parameter in non-ambient XRD. Depending on the sample type, the temperature, and the atmosphere, two different methods of heating are applied.

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ANTON PAAR'S
X-RAY ANALYTICAL SOLUTIONS

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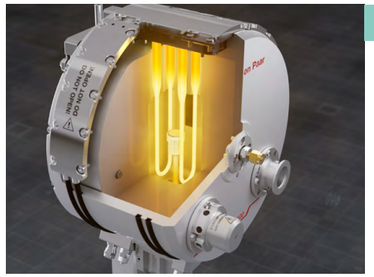


RADIATION HEATING – HTK 1500 | HTK 1200N | XRK 900

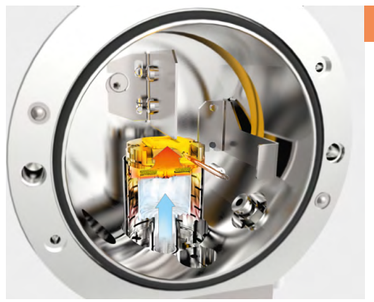
Homogeneous sample temperature up to 1,500 °C

- → Inert material sample holders don't react with sample
- → Sample rotation for better statistics
- → Operation in oxidizing or reducing atmospheres and vacuums
- → Applicable for any sample type

DIRECT HEATING – HTK 16N | HTK 2000N | DHS 1100

Highest sample temperatures of up to 2,300 °C

- → Fast temperature changes
- → Sample holders made of inert material available
- → Operation in oxidizing or reducing atmospheres, and vacuum
- → Fine powders, thin films

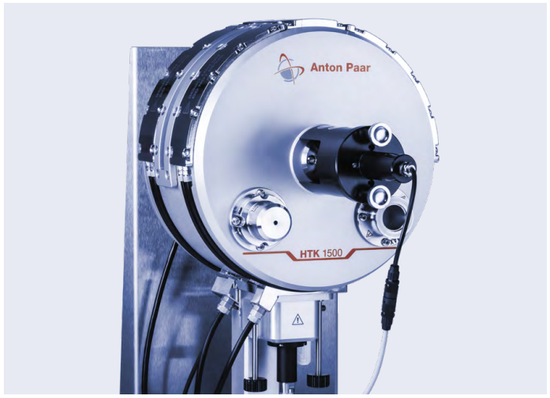


HEATING AND COOLING – TTK 600 | CHC PLUS+ | DCS 500 | BTS 150/500

Low and medium sample temperatures from -190 °C to +600 °C

- → Operates in air, inert gas, and vacuum
- → Lowest temperatures in vacuum
- → Applicable for any sample type
- → Multipurpose instruments

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

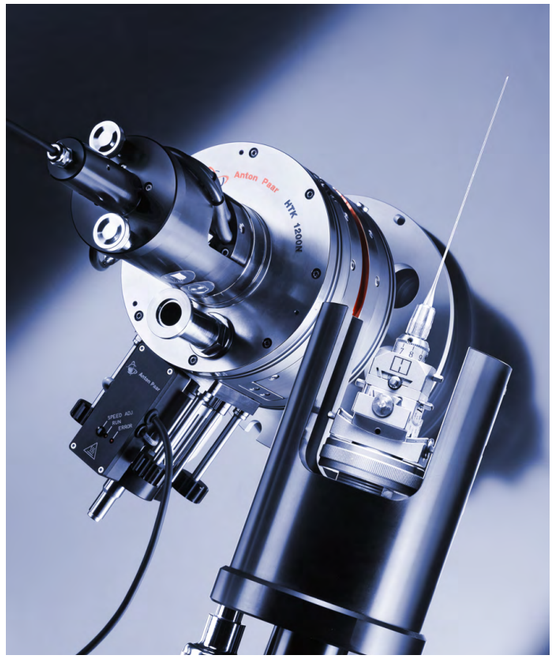
Atmospheres: air, vacuum (10^{-1} mbar), inert gases

X-ray geometry: reflection and transmission

Sample spinning option

Optional capillary extension

Environmental Heating for Homogeneous Sample Temperature



HTK 1200N | HTK 1200N Capillary
The 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 novel 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 of up to 5 mm thickness.

The sample spinning option provides highly random grain orientation, which is 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 repeatable temperature measurement.

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 a particular sample.

The HTK 1200N is the first “two-in-one” attachment for combined reflection and transmission high-temperature XRD.

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

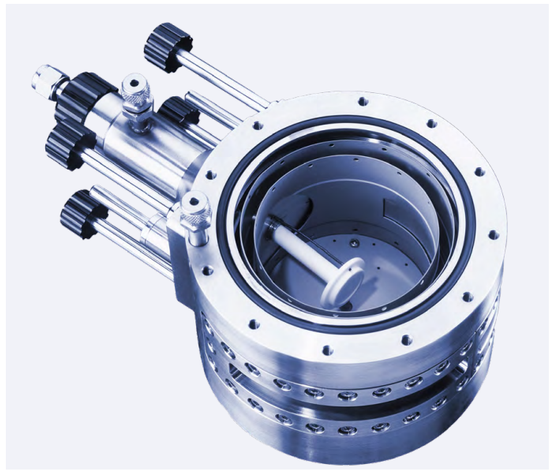
Technical data
Temperature range: 25 °C to 1,200 °C

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

X-ray geometry: reflection and transmission

Optional capillary extension

In Situ XRD Investigations of Solid State Reactions



XRK 900
The 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.

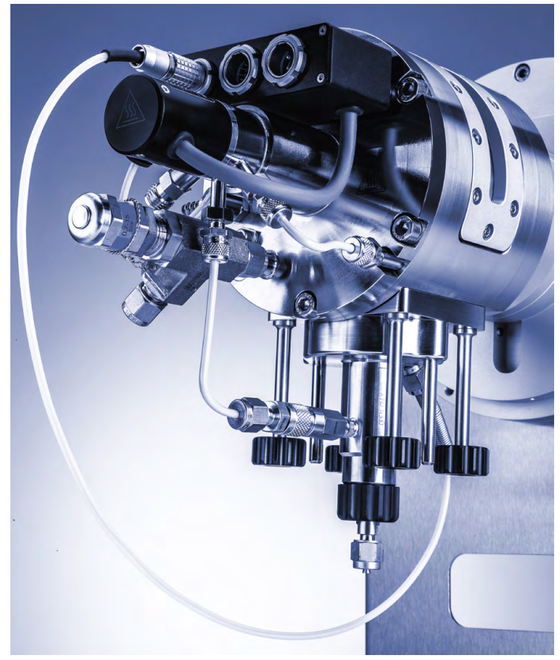
The special arrangement of the electrical heater inside the furnace eliminates temperature gradients in the sample. Two thermocouples reliably measure and control the sample temperature.

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.

The XRK 900 is a unique tool for in-situ XRD investigations of solid state reactions — unmatched in robustness and performance.



Typical applications
- → Dynamic structure changes
- → Studies of solid state reactions
- → Simultaneous investigation of structural and catalytic parameters of catalysts
- → Analysis of materials which are unstable under ambient conditions
- → Kinetic investigations of solid state reaction processes

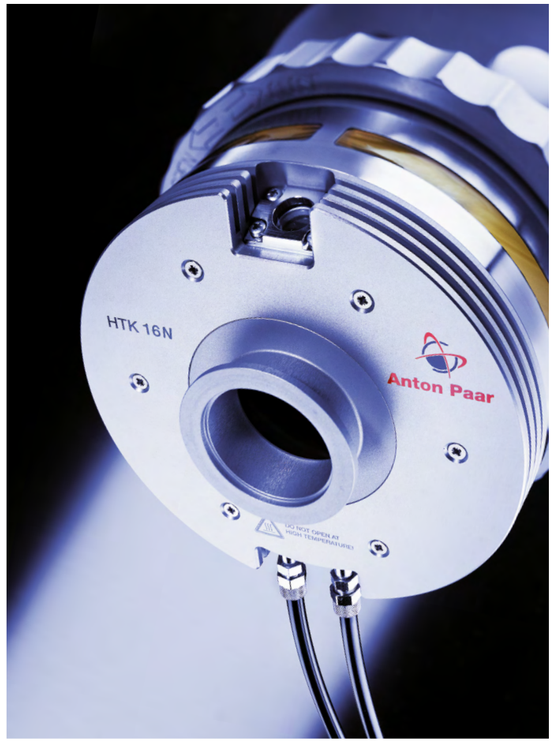
Technical data
Temperature range: 25 °C to 900 °C

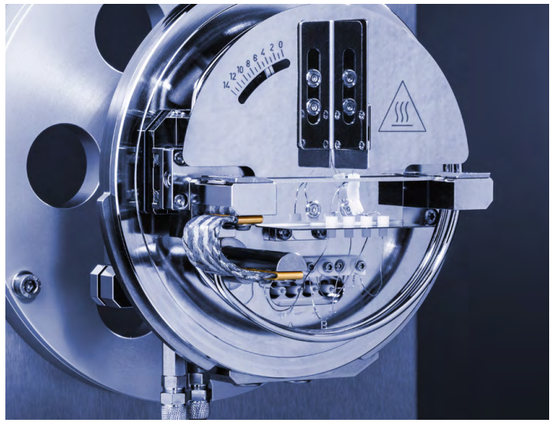
Pressure range: 1 mbar to 10 bar

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

X-ray geometry: reflection

Filament 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 used heating filament (Pt, Ta, W, C, or others on request).

The design of the chambers is optimized for minimum temperature gradients in the sample. A thermocouple, spotwelded 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.

HTK 16N and HTK 2000N are well-proven instruments for many of our customers' applications, providing high-grade materials, superior design, and simple handling.

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; up to 1,600 °C
Vacuum (10^{-4} mbar); up to 2,300 °C

X-ray geometry: reflection

A Unique Heating Attachment
for Four-Circle Goniometers

DHS 1100
The DHS 1100 is an advanced heating attachment for in-situ diffraction studies on four-circle goniometers up to 1,100 °C. It fits all common four-circle goniometers, replacing the standard sample holder. The instrument is extremely compact and lightweight. Due to the compact design with a minimum of supply hoses, the DHS 1100 does not restrict the movements of the goniometer.

The design of the heating plate guarantees a high temperature uniformity and good position stability at elevated temperatures.

The unique, dome-shaped X-ray window, made of graphite, allows the analysis of samples under vacuum and under inert gas conditions to avoid oxidation or other chemical reactions of the sample at high temperatures.

The replaceable sample holder plate of the DHS 1100 is made of aluminum nitride (AlN), a material with outstanding thermal conductivity and high chemical resistance.

Extensive cooling of the dome and the DHS 1100 housing is achieved by using compressed air.

The unique design of the DHS 1100 is registered and provides all the features you need: compactness, safety, and high performance.

Typical applications
- Temperature-induced phase transition investigations
- Texture measurements
- Stress analysis
- Profile analysis
- Grazing incidence investigations
- High-resolution studies
- Investigation of layered structures

Technical data
Temperature range: 25 °C to 1,100 °C

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

Diameter / height / weight: 128 mm / 51 mm / 450 g

X-ray geometry: reflection

Low-Temperature XRD Studies
between -190 °C and +600 °C

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 for thermal lattice expansion
- → Investigation of polymer materials

Technical data

Temperature range: -190 °C to +600 °C (liquid-nitrogen cooling), -10 °C to +600 °C (compressed-air cooling)

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

X-ray geometry: reflection and transmission

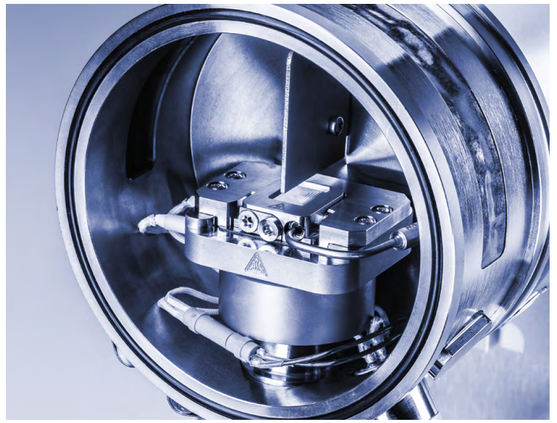
Optional capillary extension

TTK 600
The 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 measuring 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 CCU Combined Control Unit guarantees maximum temperature stability and economical use of liquid nitrogen or compressed air, respectively. The different sample holders can be easily exchanged without Z-alignment. Air-sensitive samples can be safely transferred into TTK 600 by using an antechamber.

TTK 600 is a unique all-rounder for XRD studies in the low temperature range.

Studies under Controlled **Temperature and Relative Humidity Conditions**



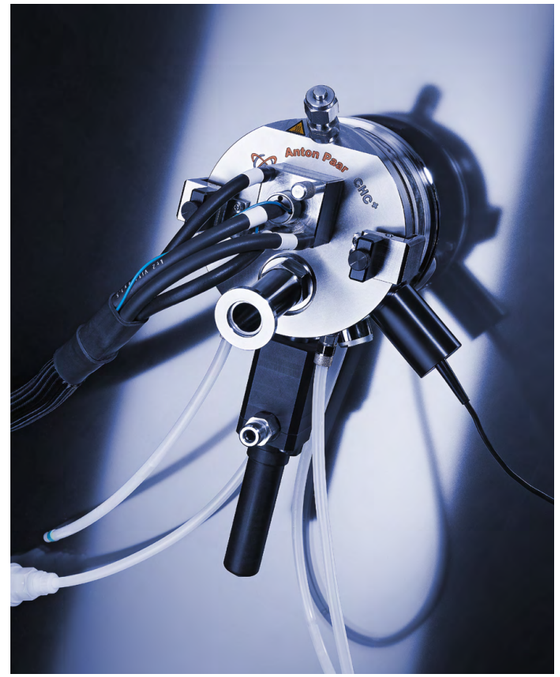
CHC plus⁺
CHC plus⁺ is a unique combination of the multi-purpose CHC Cryo & Humidity Chamber and an advanced relative humidity (RH) generator for in-situ X-ray diffraction studies at low and high temperatures from -180 °C to +400 °C, as well as under controlled humidity conditions.

The gas humidifier is mounted directly on the CHC plus⁺ 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.

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

The large temperature range, combined with the possibility to control the humidity around the sample, make 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 pharmaceutical substances and food ingredients
- → Polymorphism in APIs
- → Hydration/dehydration of zeolites and clay minerals
- → 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)

X-ray geometry: reflection

Low-Temperature Attachment for Four-Circle Goniometers and XYZ Stages

DCS 500
The DCS 500 is a novel attachment for in-situ X-ray diffraction studies between -180 °C and +500 °C on four-circle goniometers and XYZ stages. The clever design of the instrument provides high temperature uniformity and good position stability of the sample over the whole temperature range.

The X-ray transparent dome, made of graphite, allows samples to be investigated in controlled atmospheres. Vacuum or inert gas prevent chemical reactions of the sample at high temperatures or condensation at low temperatures. Cooling or heating of the dome isn't necessary.

The DCS 500 housing is temperature-controlled with water to avoid condensation at low temperatures. The layout of all supply hoses provides the best possible flexibility.

The combination of a liquid nitrogen flow, induced by the underpressure of a venturi nozzle, and a temperature control unit guarantees short cooling and heating cycles. High-precision temperature measurement is performed in the sample holder.

With the DCS 500 Domed Cooling Stage, Anton Paar offers another valuable analytical tool for materials scientists.

Typical applications
- → Temperature-induced phase transition investigations
- → Texture measurements
- → Profile analysis
- → Stress analysis
- → Investigation of layered structures

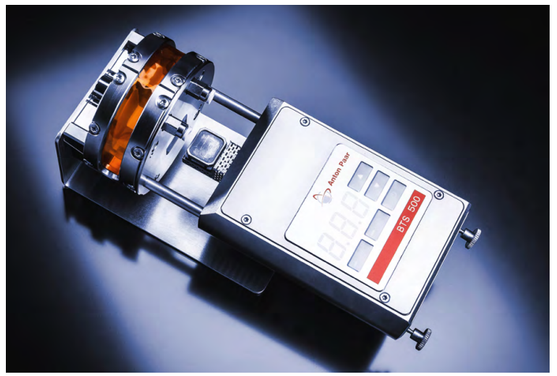
Technical data
Temperature range: -180 °C to +500 °C

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

Diameter / height / weight: 115 mm / 65 mm / 850 g

X-ray geometry: reflection

In Situ XRD Studies on Benchtop Diffractometers



BTS 150/500
The BTS 150/500 Benchtop Heating Stages are the first commercial non-ambient stages for benchtop diffractometers and extend their applicability to in-situ XRD studies. Measurements can be performed between -10 °C and +150 °C with BTS 150 and from ambient to 500 °C with BTS 500.

Both instruments are extremely compact in design to fit into the restricted space of typical benchtop diffractometers. The control electronics are integrated in the heating stages without the need of an external control unit.

Samples are heated by a Peltier (BTS 150) or a resistance heater (BTS 500). Excellent insulation and air cooling avoid heat transfer to any components of the benchtop diffractometer. The Pt100 temperature sensor is located close to the sample and guarantees reliable and repeatable temperature measurements.

The BTS 150/500 sample holders are easily accessible. Studies may be carried out either under vacuum, air, or inert gas conditions. The instrumental design guarantees minimum thermal expansion of the sample holder and therefore a correct geometrical position of the sample in the X-ray beam throughout the experiment.

The unique design of BTS 150/500 is patented and provides all the features required by benchtop diffraction applications: compactness, reliability, and ease of use.

Typical applications
- → In-situ phase characterization
- → Structure determination
- → Phase properties (cell parameters, crystallite size, lattice strain)
- → Dynamic structure changes
- → Rietveld analysis

Technical data
Temperature range: -10 °C to +150 °C (BTS 150)
Ambient to 500 °C (BTS 500)

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

X-ray geometry: reflection

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