Anton Paar

Measure
what is measurable
and make measurable
that which is not.

Galileo Galilei (1564-1642)

Reference Guide

HTK 1200N

High-Temperature Oven-Chamber with

CCU 1000 Control Unit

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Further information

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Reference Guide

HTK 1200N

High-Temperature Oven-Chamber with

CCU 1000 Control Unit

Contents

1 Safety Instructions

- Read the documentation before using HTK 1200N.
- Follow all hints and instructions contained in the documentation to ensure the correct use and safe functioning of HTK 1200N.
- The documentation is a part of the product. Keep this document for the complete working life of the product and make sure it is easily accessible to all people involved with the product. If you receive any additions or revisions to the documentation from Anton Paar GmbH, these must be treated as part of the documentation.

1.1 General safety instructions

Liability

- The documentation does not claim to address all safety issues associated with the use of the instrument and samples. It is your responsibility to establish health and safety practices and determine the applicability of regulatory limitations.
- Operate HTK 1200N with CCU 1000 Combined Control Unit from Anton Paar GmbH only.
- Anton Paar GmbH only warrants the proper functioning of HTK 1200N if no adjustments have been made to the mechanics, electronics, and firmware.
- Only use HTK 1200N for the purpose described in the documentation. Anton Paar GmbH is not liable for damages caused by incorrect use of HTK 1200N.

Installation and Use

- HTK 1200N is **not** an explosion-proof instrument and therefore must not be operated in areas with risk of explosion.
- HTK 1200N must not be used with hazardous (e.g. explosive or poisonous) gases.
- The installation procedure should only be carried out by authorized personnel who are familiar with the installation instructions.
- It is the responsibility of the customer to provide all the set-up (tubings, valves,..) necessary for proper work with HTK 1200N.
- Do not use any accessories or wearing parts other than those supplied or approved by Anton Paar GmbH.
- Make sure all operators are trained to use the instrument safely and correctly before starting any applicable operations.
- In case of damage or malfunction, do not continue operating HTK 1200N. Do not operate the instrument under conditions which could result in damage to goods and/or injuries and loss of life.
- Check HTK 1200N, especially the sample holder, for chemical resistance to the samples and cleaning agents.
- The results delivered by HTK 1200N not only depend on the correct function of the instrument, but also on various other factors. We therefore recommend to have the results checked (e.g. plausibility tested) by skilled personnel before consequential actions are taken based on the results.

Maintenance and Service

- Service and repair procedures may only be carried out by authorized personnel or by Anton Paar GmbH.

Disposal

- Concerning the disposal of HTK 1200N, observe the legal requirements in your country.

Returns

- For repairs send the cleaned HTK 1200N (instrument) to your Anton Paar representative. Only return the instrument together with the filled out RMA (Return Material Authorization) and the form "Safety Declaration for Instrument Repairs". Please download the Safety Declaration form from our website www.anton-paar.com.
- Do not return instruments which are contaminated by radioactive materials, infectious agents or other harmful substances that cause health hazards.

Precautions for highly inflammable samples and cleaning agents

- Observe and adhere to your national safety regulations for handling the measured samples

(e.g. use of safety goggles, gloves, respiratory protection etc.).

- Only store the minimum required amount of sample, cleaning agents and other inflammable materials near HTK 1200N.
- Do not spill sample/cleaning agents or leave their containers uncovered. Immediately remove spilled sample/cleaning agents.
- Make sure that the setup location is sufficiently ventilated. The environment of HTK 1200N must be kept free of inflammable gases and vapors.
- Supply a fire extinction unit.
- Ensure sufficient supervision of HTK 1200N during operation.

1.2 Radiation Safety

- The HTK 1200N High-Temperature Oven-Chamber represents an open system and is allowed to be operated only on diffractometers equipped with a radiation enclosure.
- Before starting the experiment, the user has to make sure that the radiation hazard equipment corresponds to the local requirements.

1.3 Conventions for safety messages

The following conventions for safety messages are used in this document:

DANGER

Description of risk.
Danger indicates a hazardous situation which, if not avoided, **will** result in death or serious injury.

WARNING

Description of risk.
Warning indicates a hazardous situation which, if not avoided, **could** result in death or serious injury.

CAUTION

Description of risk.
Caution indicates a hazardous situation which, if not avoided, could result in minor or moderate injury.

NOTICE

Description of risk.
Notice indicates a situation which, if not avoided, could result in damage to property.

CAUTION

Hot surface
This symbol calls attention to the fact that the respective **surface can get very hot**. Do not touch this surface without adequate protective measures.

CAUTION

High voltage
This symbol calls attention to the **risk of high voltage**. Do not proceed until the indicated conditions for averting this threat are fully understood and met.

CAUTION

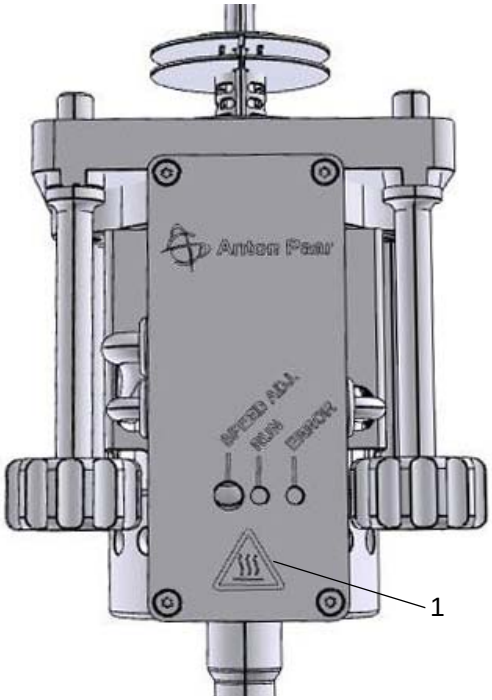
X-ray Radiation
This symbol calls attention to the **risk of radiation**. Do not proceed until the indicated conditions for averting this threat are fully understood and met.

Wear protective gloves
Wear protective gloves when handling the HTK 1200N.

Wear safety goggles
Wear safety goggles when handling the HTK 1200N.

TIP: *Tip gives extra information about the situation at hand.*

1.4 Safety Signs on the Instrument



1 CAUTION - Hot surface

Fig. 1: Position of warning signs on the sample holder flange

CAUTION

Hot surface
This symbol calls attention to the fact that the respective **surface can get very hot**. Do not touch this surface without adequate protective measures.

1 CAUTION

Fig. 2: Safety sign on Instrument

CAUTION

This symbol calls attention to the fact that the instrument must be connected to protective ground. Service has to be done by qualified personal only. Always disconnect power cord before serving.

Wear protective gloves
Wear protective gloves when handling the HTK 1200N.

Wear safety goggles
Wear safety goggles when handling the HTK 1200N.

NOTICE
Take care that the warning symbols remain clearly legible.

2 Overview

2.1 Description of the Instrument

The HTK 1200N High-Temperature Oven-Chamber is designed for non-ambient X-ray studies from room temperature to 1200 °C in different atmospheres.

The HTK 1200N High-Temperature Oven-Chamber can be easily adapted to most available goniometers.

Due to its environmental heating the temperature gradient in the sample is low and samples of several mm thickness can be investigated.

The possibility of sample spinning preserves good statistics of grain orientation distribution even if recrystallisation results in large-sized grains. This is necessary for obtaining reliable diffraction information and for subsequent data evaluation like Rietveld analysis.

The sample carriers can be easily exchanged and can accommodate various flat sample forms such as powders or bulk samples.
Thin films (on thick substrates) can also be studied with reliably measured surface temperature due to the environmental heating.
Sample carriers made of alumina provide high chemical resistance.

In addition to the sample holders for flat powder samples, a HTK 1200N Capillary Extension is available. With this extension, the HTK 1200N can be converted into a capillary heater up to 1000 °C.

To ensure reliable operation of the HTK 1200N High-Temperature Oven-Chamber, the instrument must be operated with:

- 135004 CCU 1000 Combined Control Unit
- 164877 Flow controller

TIP: *Please refer to Appendix Efor detailed information*

2.2 Design of the Instrument

The HTK 1200N is a heating stage for powder XRD with a temperature range from 25 to 1200 °C.

Basically, the HTK 1200N consists of:

1. Housing with safety switch box
2. Lid
3. Sample holder flange with sample holder (stationary or spinning)

TIP: *For detailed information on the materials used in the HTK 1200N, please refer to Appendix A.*

2.2.1 Housing with Safety Switch Box

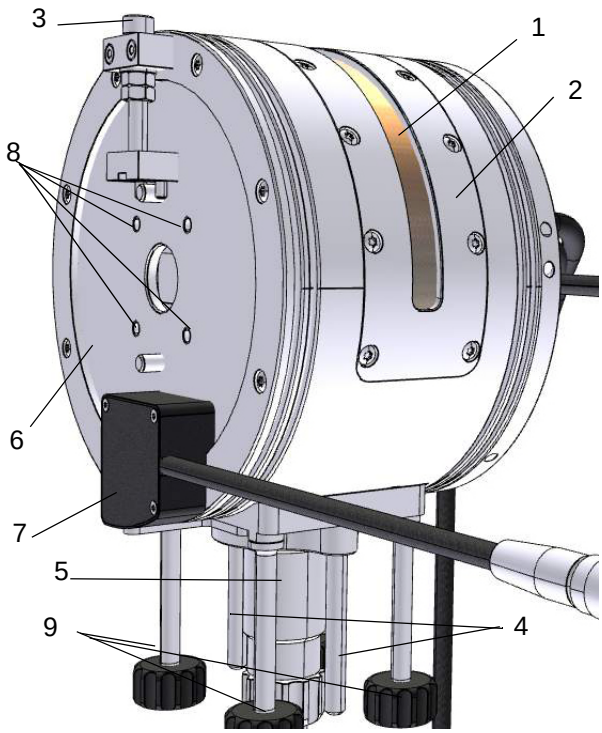


Fig. 3: Main components of the sample chamber

1 X-ray window
2 Window clamp
3 Height alignment screw
4 Sample holder guiding rods
5 Sample holder flange
6 Base plate
7 Safety switch box
8 Boreholes for adapter
9 Flange fixing screws

All components of the HTK 1200N housing are made of nickel-plated brass. Both the base plate and the housing are water-cooled.

The sample holder is inserted through the opening at the bottom. Two guiding rods provide for exact centering of the sample holder flange.

At the rear of the instrument is a small safety switch box that contains two safety switches:

- a bi-metallic thermo-switch that shuts off the

heater, if the housing temperature reaches 70 °C and

- a contact switch that detects whether the sample holder flange is mounted and which shuts off the heater, if it is not.

The adapter, which is used to mount the HTK 1200N on the goniometer is fixed to the housing's base plate. Vertical alignment of the HTK 1200N versus the goniometer axis is done by using the height alignment screw or the alignment stage.

Depending on the type of goniometer used, evacuation can be performed through the adapter or through the vacuum flange on the lid.

Along the radiation path (entrance and exit of X-rays), the housing is slotted. The window foil is pressed against an O-ring by means of two clamps and covers the window openings. The window foil is either an aluminium-coated Kapton foil of 125 µm thickness (for work up to 1000 °C) or a combination of a graphite foil and a non-coated Kapton foil of 125 µm thickness (for work above 1000 °C).

TIP: *The graphite foil and non-coated Kapton foil are mounted upon delivery.*

The oven consists of an electrical heater surrounding the sample holder and thermal insulation around the heater.

The electrical heater is formed in a special way to uniformly heat the sample, which is located in the center of the heater. The heating filament is made of Kanthal.

NOTICE
Risk of damage
At the surface of the Kanthal heater an oxide layer is formed which protects the wire.
After work in vacuum or inert gas, it is recommended to regularly regenerate this oxide layer by heating the HTK 1200N up to 1000 °C in air. This will extend the life time of the heater considerably.

The thermal insulation consists of foamed alumina and provides a very uniform temperature distribution inside the oven.

TIP: *The foamed alumina shell is very porous with a large internal surface. Even when operated with a vacuum pump, small amounts of adsorbed gas, in particular oxygen, are continuously released from the alumina. If the sample material is very sensitive to oxygen, this can lead to undesired sample oxidation at elevated temperature.*

2.2.2 Lid

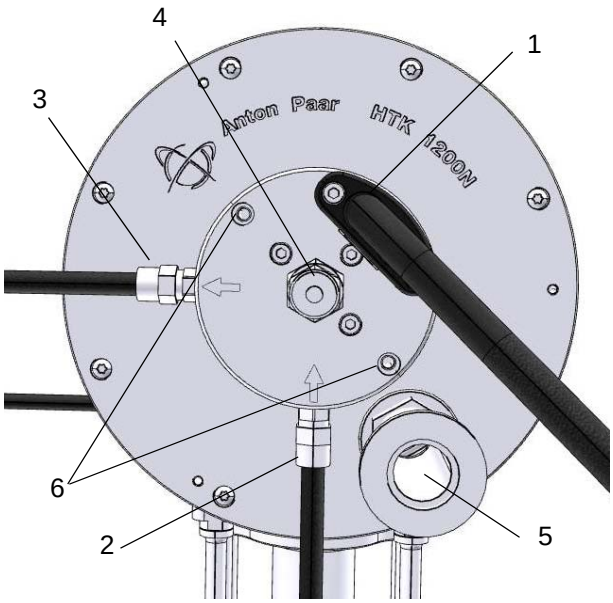


Fig. 4: Chamber Lid

1 Current cables for the electrical heater
2 Cooling water inlet
3 Cooling water outlet
4 Gas supply
5 Vacuum flange (DN16KF)
6 Tap holes for HTK 1200N Capillary Extension guiding rods (optional)

The lid is made of nickel-plated brass and is water-cooled. It is screwed to the housing and sealed with an O-ring.

The lid contains connectors for the water supply, the gas supply and the vacuum. The power supply for the electrical heater is provided by two cables which have to be connected to the CCU 1000.

The lid has two tap holes into which guiding rods are screwed when the HTK 1200N is used with the Capillary Extension (optional).

TIP: *For more information about the connectors, refer to chapter 4.*

For more information about the HTK 1200N Capillary Extension, refer to Appendix Eand to the HTK 1200N Capillary Extension Instruction Manual.

2.2.3 Sample Holder Flanges

Two different types of sample holder flanges are available for the HTK 1200N:

Stationary sample holder

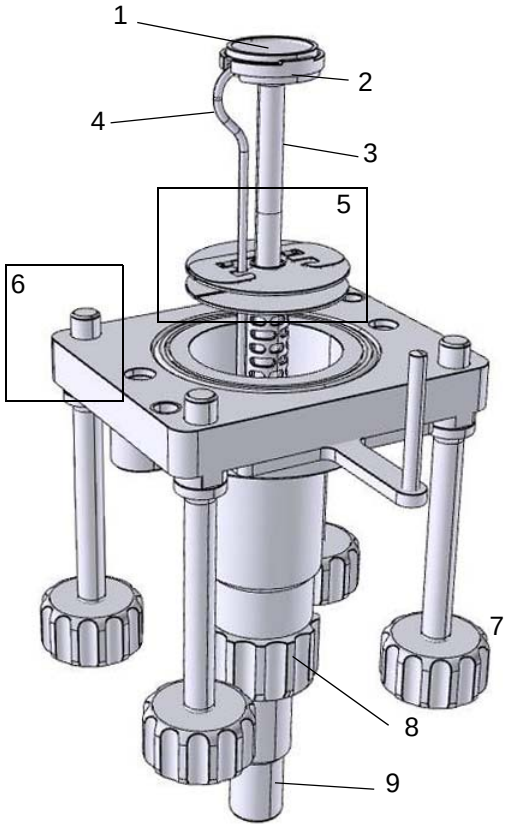


Fig. 5: Stationary sample holder

1 Sample carrier
2 Fixing ring for sample carrier
3 Sample holder body
4 Thermocouple
5 Temperature shielding with fixing clamp
6 Flange
7 Flange fixing screws (4 pcs)
8 Fixing nut
9 Cable connection between thermocouple and CCU 1000

Sample spinner

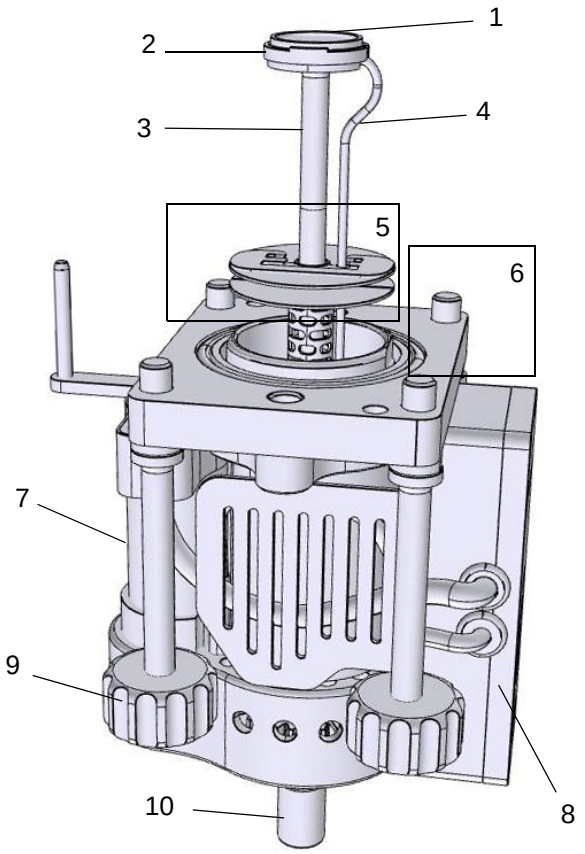


Fig. 6: Sample spinner

1 Sample carrier
2 Fixing ring for sample carrier
3 Sample holder body
4 Thermocouple
5 Temperature shielding with fixing clamp
6 Flange
7 Motor
8 Electronic motor control
9 Flange fixing screws (4 pcs)
10 Cable connection between thermocouple and CCU 1000

The sample holder assembly consists of the flange and the actual sample holder, which is fixed to the flange by a fixing nut.

TIP: *The sample spinner is actually not rotating but oscillating - still providing a good statistics of grain orientation in the sample.*

The sample holder comprises the sample carrier, a fixing ring and the sample holder body. All these parts are made of ceramics.

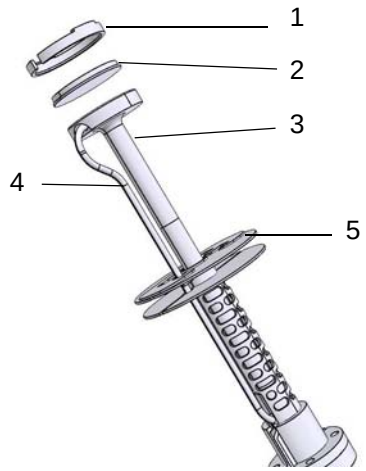


Fig. 7: Sample holder

1 Sample carrier
2 Fixing ring for sample carrier
3 Sample holder body
4 Thermocouple
5 Temperature shielding with fixing clamp

- The sample carrier is fixed to the top of the sample holder body by a fixing ring. Three different versions of the sample carrier with different depth are available: 0.8 mm deep, 0.4 mm deep and a flat one. Depending on the sample to be investigated sample carriers made of other materials are available on request.
- The fixing ring is flexible and is put over the edge of the sample carrier, allowing quick and easy exchange of the prepared sample.

NOTICE
Risk of damage
Make sure that the contact surfaces between sample carrier and top of the sample holder body are free of dust and sample residues to provide for a good thermal contact!

- The sample holder body contains a Pt10Rh-Pt (type S) thermocouple which is used for sample temperature measurement and control. It is located right underneath the sample position, thus providing good correspondence between the set temperature and the actual sample temperature.
  The thermocouple is connected to the connector box by the cable for sample holder flange (see also *chapter 4.4.2*).
  The temperature shielding closes the opening of the alumina insulation and provides a uniform temperature distribution.

TIP: *Please refer to chapter 7 for further information.*

The sample holder is inserted into the HTK 1200N chamber with two guide pins and fastened by four screws.

2.3 Connector Box

The connector box terminates the supply hose of the HTK 1200N, which contains the control wires for the temperature sensor, flange control and the sample spinner.

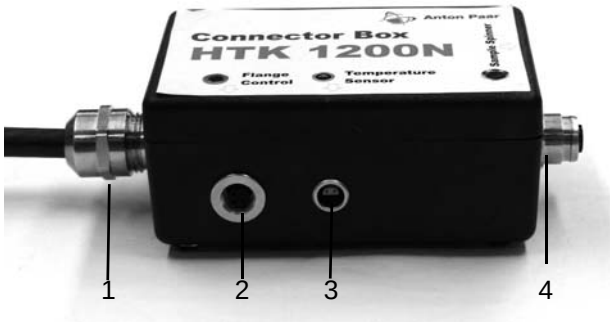


Fig. 8: Connector box

1 Connector cable to the CCU 1000
2 Connector for flange control
3 Connector for thermocouple
4 Connector for sample spinner

2.4 CCU 1000 Combined Control Unit

CCU 1000 is designed to control the sample holder temperature in HTK 1200N and to guarantee safe operation of the entire instrument. In order to reach and maintain the desired sample holder temperature, CCU 1000 controls the heater inside the sample chamber.

CCU 1000 can be operated manually by means of the push - buttons on the front panel, or it can be remote-controlled via a serial RS 232 interface.

2.4.1 CCU 1000 Front Panel

The front panel of CCU 1000 contains all control buttons, status LEDs and the display.

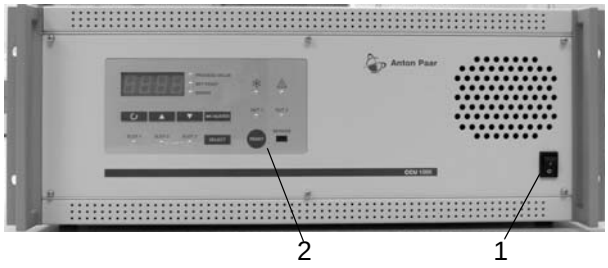


Fig. 9: CCU 1000 front panel with control elements

1 Mains switch
2 Display and keypad

All elements of the front panel are described in detail in chapter 6.2.

2.4.2 CCU 1000 Rear Panel

The rear panel of CCU 1000 contains all the connectors of the instrument.

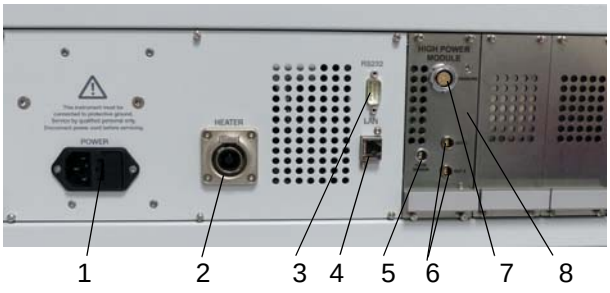


Fig. 10: CCU 1000 rear panel

1 Power connector
2 Connector Heater
3 RS 232 connector
4 LAN connector (currently not used)
5 Connector Flow Sensor
6 Connector OUT1/2 (optional connectors)
7 Connector Sensors/Heater
8 High power module for HTK 1200N

2.4.3 Overheat Protection

HTK 1200N has two devices to prevent overheating of the instrument. The **flow control unit** of HTK 1200N monitors the flow of cooling water through the housing of the sample chamber. It is a water flow dependent switch, which is connected to CCU 1000. If there is not enough water flow, CCU 1000 switches to Standby mode (heater off), the Error LED on the front panel starts to flash and shows an alarm message (E03).

In addition, a **thermoswitch** is mounted in the safety switch box for flange control (Fig. 3 (7)) to protect the device from being overheated in case of a malfunction. The thermoswitch interrupts the heating power circuit if the temperature of the chamber housing exceeds 70 °C. CCU 1000 automatically switches to Standby mode (heater off) and shows an alarm (E04).

CAUTION

Hot surface
When the cooling water supply to the chamber fails, the housing can have a **hot surface**. Carefully check the temperature of the HTK 1200N housing and make sure the housing is at room temperature before you touch it. Make sure that you have found and removed the cause of the error before you continue operation of HTK 1200N and CCU 1000.

TIP: *A list of all error messages can be found in chapter 10.1.*

3 Checking the Supplied Parts

HTK 1200N High-Temperature Oven-Chamber was tested and packed carefully before shipment. However, damage may occur during transport.

1. Keep the packaging material (box, foam piece, transport protection) for possible returns or questions from the transport and insurance company.
2. Check the delivery for completeness by comparing the supplied parts to those noted in the table below.
3. If a part is missing, contact the local representative of your diffractometer manufacturer or Anton Paar GmbH in Graz, Austria.
4. If a part is damaged, contact the transport company and either the local representative of your diffractometer manufacturer or Anton Paar GmbH in Graz, Austria.

Table 3-1: Supplied Parts

Pcs.	Item	Cat.No.
1	**HTK 1200N with stationary sample holder** including: Heating chamber Stationary sample holder Sample carrier, 0.8 mm deep (2x) Fixing ring for sample carrier Cable for sample holder flange	16407
or		
1	**HTK 1200N with sample spinner** including: Heating chamber Sample Spinner Sample carrier, 0.8 mm deep (2x) Fixing ring for sample carrier Cable for sample holder flange	16408
1 1 1 2 1 1 1	**CCU 1000 Combined Control Unit** Mains cables Heater cable Mains fuses RS232 cable to control PC USB cable Connector box	135004
1	Stand for sample holder flange	
1	Cooling water hoses (already mounted)	
1	Pressure hose and clamps (customer-specific part)	
1	Accessory box (content specified on label)	
1	Set of tools	
1 1 1	Instruction Manual Software manual Nambicon USB flash drive with Nambicon	

NOTICE

Risk of damage

Always use the original packing material when shipping the instrument or parts of it. Store the packing material carefully for later use!

TIP: *Check the supplied packages delivered with the HTK 1200N for completion by comparing the parts to those listed in the appropriate packing lists.*

4 Installation

CAUTION

X-ray radiation
The HTK 1200N High-Temperature Oven-Chamber represents an open system. It is only permitted to install the HTK 1200N on diffractometers equipped with a radiation enclosure.

NOTICE
Risk of damage
Do NOT touch the X-ray window during installation. When you touch the window, the graphite foil underneath the Kapton foil will tear.

4.1 Installation Requirements

TIP: *When HTK 1200N is ordered together with the required adapter, the adapter is mounted on the chamber on delivery.*

When HTK 1200N is ordered together with a motorized alignment stage, the adapter is mounted on the alignment stage.

Operation of HTK 1200N/CCU 1000 requires cooling water supply and electrical power. The corresponding requirements can be found below and in *Appendix A* and *Appendix B*.

4.1.1 Electrical Power and Computer Interface

CCU 1000:

- Mains voltage: AC 230 V ± 10 %
- Mains frequency: 50 or 60 Hz.
- Mains connector (1): IEC 60320 C13
- Mains fuses: 2 x T 8 A H 5 x 20 mm (ceramic tube)
- Remote control interface: RS 232 C
- RS 232 connector on CCU (2): D-Sub DE-9 (male)
- Cable type: null modem

4.1.2 Cooling Water Requirements

- flow rate: 0.7 to 1.5 l/min
- temperature: 15 to 25 °C
- pressure: max. 4 bar

4.2 Mounting the Adapter on HTK 1200N

An appropriate **adapter** is necessary to mount the HTK 1200N High-Temperature Oven-Chamber on a goniometer. For detailed ordering information, refer to *Appendix E*.

1 Alignment equipment
2 Allen screw (4 pcs)
3 Adapter (version depends on the goniometer used)
4 HTK 1200N base plate

To mount the adapter, proceed as follows:

1. Remove the alignment equipment (1) from the rear of the HTK 1200N base plate (4).
2. If required, clean the O-ring at the adapter flange and lightly coat the O-ring with vacuum grease.
3. Use the four Allen screws (2) to mount the adapter to the HTK 1200N base plate (4).

4. Fasten the lower part of the alignment equipment (1) to the adapter using the two screws.
5. Fasten the two upper parts of the alignment equipment (1) to the HTK 1200N base plate using the two screws in the front of the upper part. Make sure that the large screw on the top of the alignment equipment (1) is surrounded by the two halves of the upper part.

WARNING

Make sure that the system "chamber/alignment stage/goniometer" is properly earthed. Check with an ohmmeter if necessary: a low measuring value means sufficient earthing.

NOTICE
Risk of damage

- When using an adapter with alignment stage: Detailed mounting instructions are supplied with the alignment stage.
- A corresponding earthing connector is supplied with the alignment stage.

4.3 Mounting HTK 1200N on the Goniometer

The HTK 1200N High-Temperature Oven-Chamber (with adapter) is mounted on the goniometer instead of the standard (ambient) sample holder.

TIP: *Make sure that the X-ray beam is properly aligned before you remove the standard sample holder (see the Diffractometer Instruction Manual for detailed information). Good beam alignment is important, because HTK 1200N is aligned relative to the X-ray beam.*

Make sure that the chamber is mounted centrically on the goniometer.

Take care that the sample holder is as parallel as possible to the Θ-zero line of the goniometer.

Instructions for mounting the chamber are given in the instruction manual for your diffractometer

WARNING

Make sure that the HTK 1200N chamber and (if applicable) the alignment stage are properly connected to protective earthing. Refer to the instruction manual for your diffractometer for information about suitable terminals for protective earthing.

4.4 Installing CCU 1000

CCU 1000 Combined Control Unit is prepared for rack mounting in a 19" 4HE slot.

Mount CCU 1000 either in a suitable slot in the diffractometer or in a rack next to the diffractometer.

Make sure there is enough space (at least 10 cm) behind the CCU 1000 for all connectors and that the slot is sufficiently ventilated to avoid heat accumulation behind the CCU 1000.

CAUTION

Risk of damage
Always make sure that CCU 1000 is turned off before you connect or disconnect cables on the sample chamber or the CCU 1000.

The connector for temperature measurement, flange control and sample spinner are located in the connection box described in chapter 2.3.

The instrument is delivered together with the current cables for the HTK 1200N heater and the connection cable for the HTK 1200N temperature sensor.

Connect the electrical cable to the CCU 1000 rear panel as follows:

- Plug the fix mounted cable on the connector box to the connector on the rear panel of the CCU 1000 (see Fig. 10 (7)).
- Make sure the mains switch on CCU 1000 is OFF and connect the mains power cable to the POWER connector (see Fig. 10 (1)).

Connect the electrical cable to the sample chamber as described in the following chapters.

WARNING

Risk of injury
This instrument must be connected to protective ground.

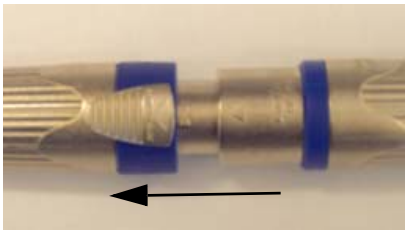
4.4.1 Power Supply for the Heater

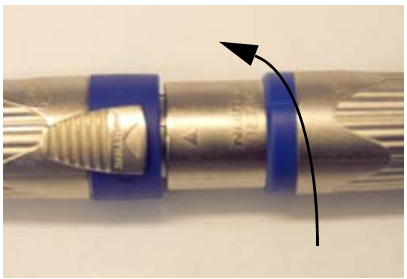
Power for the heater of the HTK 1200N is supplied with two high current cables. Plug the cable for the heater to the heater connector on the rear side of the CCU 1000 (Fig. 10 (2)). Connect these cables with the two high current cables at the lid of the HTK 1200N as described below:

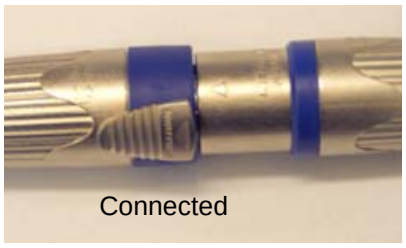
CAUTION

Risk of damage
Switch off the CCU 1000 and disconnect the cable for the mains supply, before disconnecting and connecting the high current cables and the thermocouple cable.

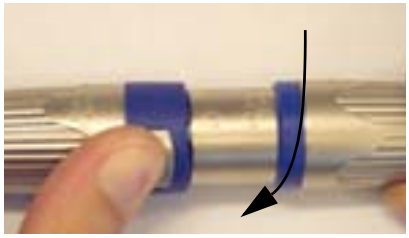
Connecting procedure:

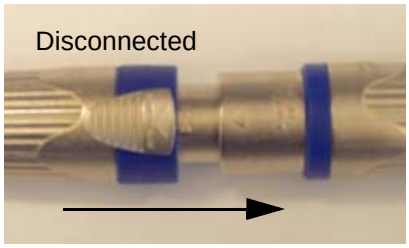






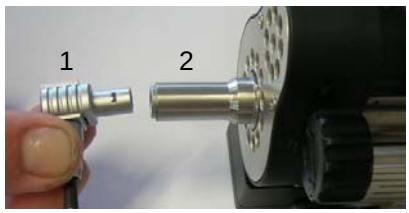
Disconnecting procedure:



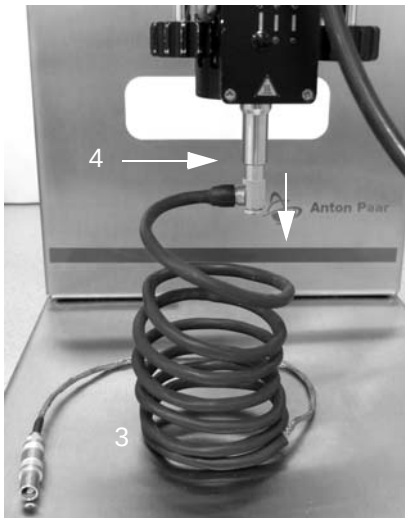


4.4.2 Thermocouple of Stationary Sample Holder and Sample Spinner

For correct installation please follow this instruction:



Stick the plug-in connection (1) of the cable for sample holder flange into the locknut (2) of the sample holder at the bottom of the sample holder flange. Then connect the other end of the cable to the connector "Temperature Sensor" on the connector box (Fig. 8 (3).



When operating the sample holder flange beware of the shape of the thermocouple cable.

Do NOT tear at the cable and prevent a complete straightening of the cable.

For safe operation with sample spinner preserve a minimum of four windings (3).

Before demounting the sample holder flange and changing the sample holder disconnect the cable from the sample holder at the bottom of the sample holder flange (4).

4.4.3 Connection of Sample Spinner Motor

The motor of the sample spinner has to be connected to the connector "Sample Spinner" on the connector box (Fig. 8 (4)).

4.4.4 Connection for Safety Circuit

At the rear of the HTK 1200N is a small safety switch box for flange control and overheating protection. Connect the extension cable from the safety switch box on the back of the HTK 1200N to the connector "Flange Control" on the connector box (*Fig. 8 (2)*).

4.5 Connecting CCU 1000 with a Computer

The RS 232 serial port at the rear panel of CCU 1000 allows you to connect the CCU 1000 to a computer for remote control (see Fig. 10 (3)).

A null modem RS 232 cable is required to connect the CCU 1000 to a PC.

PIN assignment:

Pin	
2	RX
3	TX
5	GND
Others	not connected

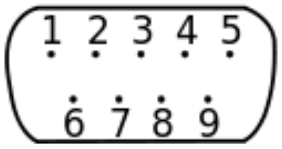


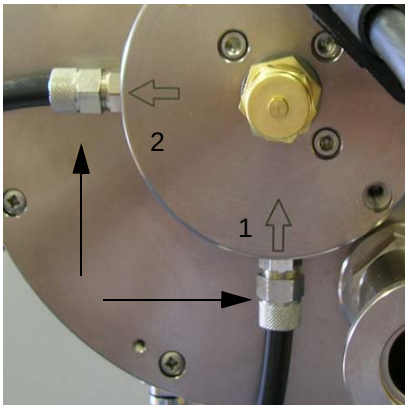
Fig. 11: RS 232 pins

NOTICE
According to EN60950 standards, the RS 232 serial interface represents a SELV circuit which must therefore only be connected to SELV circuits.

4.6 Installing the Cooling Water Circuit

The water flows through the HTK 1200N housing and the lid.

NOTICE
Take care that all hoses are secured on the nozzles against unintentional disconnection. Appropriate nuts are supplied with the instrument.



The HTK 1200N is supplied with mounted cooling water hoses with quick couplings. Water inlet (1) and oulet (2) are located on the HTK 1200N lid. Make sure that the water inlet is connected to nozzle 1 and the water outlet to nozzle 2!
Take care that the hoses connected to the nozzles are always protected against unintentional disconnection. Appropriate nuts (3) are supplied with the instrument.

The Water Flow Controller (part N° 164877) contains a pressure regulator and a flow control unit. The required flow rate is approx. 0.7 l/min at a pressure of 2 bar. The flow controller supplied by Anton Paar GmbH is set to this value. Refer to *Appendix B* for detailed information on the requirements for cooling water quality.

Connect the cable of the flow control unit to the WATER FLOW CONTROL connector on the rear panel of CCU 1000 (Fig. 10 (5)).

NOTICE
Risk of damage
Exceeding the maximum permissible pressure of 4 bar may result in bursting of the hoses.

NOTICE
Risk of damage
- In order to avoid damage, do not operate the HTK 1200N before the cooling water circuit is started.
- Check the proper functioning of the flow controller regularly!

4.7 Installing the Gas Supply

Depending on the specific measuring problem, the HTK 1200N can be operated under different gas atmospheres, preferably under inert gas.

NOTICE
Risk of Damage
Check the chemical resistance of the materials used in the HTK 1200N BEFORE you start an experiment with a gas other than specified in *Appendix A.*

The gas supply tubing is connected to the Swagelok fitting in the lid, see *chapter 2.2.2*.
The gas exhaust tubing can be connected to the adapter. The type of gas connector depends on the adapter type.

WARNING

Risk of injury
Do NOT use poisonous, explosive or aggressive gases.
Make sure that all fittings are tight and mounted properly, and an appropriate exhaust hose is connected to the gas outlet.

TIP: *Refer to Appendix F for further information.*

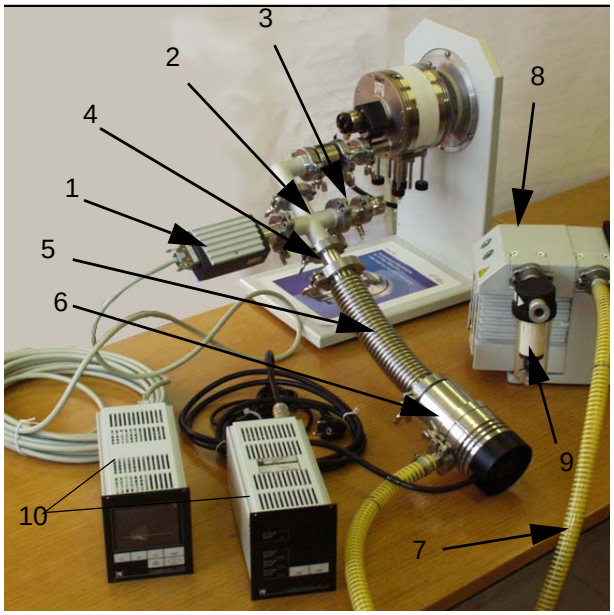
4.8 Installing the Vacuum Equipment

The HTK 1200N can be operated under a vacuum of approx. 10^{-4} mbar.

TIP: *Anton Paar GmbH offers a suitable vacuum equipment for the HTK 1200N. All required components for the installation are included in the delivery.*

Please refer to Appendix E.

Vacuum can be produced either through the adapter axis in the base plate or through the vacuum flange in the lid (see below).



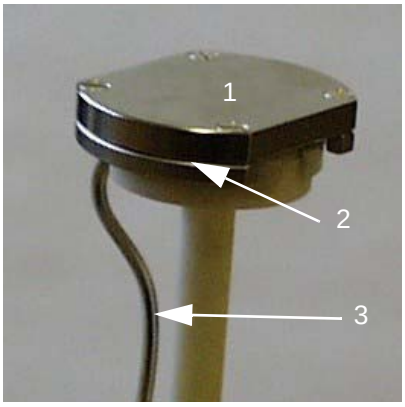
1 Combined vacuum gauge ITR 90
2 Cross piece DN25
3 Venting valve
4 Reducer DN25 / DN40
5 Flexible stainless-steel hose (0,25 m length)
6 Turbomolecular pump
7 Plastic hose (3 m length)
8 Rotary-slide vacuum pump
9 Oil-mist filter
10 Display

Fig. 12: Anton Paar GmbH high vacuum equipment

5 Putting HTK 1200N into Operation

5.1 Alignment of the Sample Holder

Proper alignment of the HTK 1200N High-Temperature Oven-Chamber is important to get good data quality. The surface of the sample holder must be parallel to the axis of the X-ray beam (Θ-zero line) and in the centre plane of the goniometer.



1 Alignment slit
2 Slit for X-ray beam
3 Thermocouple

Part of the HTK 1200N accessories is an alignment slit to enable precise alignment.

The alignment of the HTK 1200N consists of height (z-) alignment and rotation (ω–) alignment. The actual alignment procedure depends on the type of adapter (rigid or with motorized z-stage) and on the diffractometer type.

Read the related sections in the instruction manual for your diffractometer before you start the alignment.

NOTICE
Do not perform the **rotation (ω) alignment** yourself if you have a diffractometer with **alignment preserving sample stage fitting**. For such instruments the sample stage is usually pre-aligned.
Re-alignment should only be done by a service engineer from your diffractometer manufacturer.

Alignment is best done in two steps:

1. Coarse alignment with the upper edge of the empty sample holder.
2. Fine alignment with the slit for X-ray beam in the alignment slit

Make sure that the X-ray beam is properly aligned before you mount the HTK 1200N High-Temperature Oven-Chamber (read the *Diffractometer Instruction Manual* for detailed information). Good beam alignment is important, because HTK 1200N is aligned relative to the X-ray beam (θ-zero line).

5.2 Height adjustment with Rigid Adapter

1. Install the chamber into the already aligned goniometer (use the standard sample holder to align the goniometer)

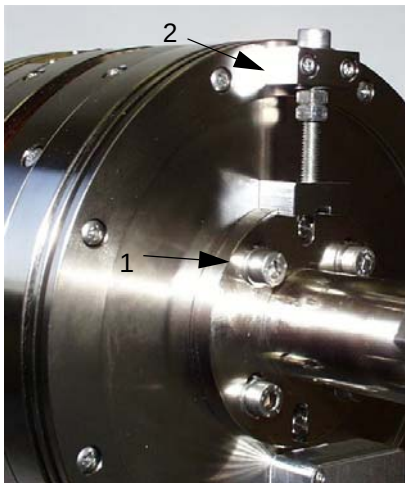
NOTICE
Risk of Damage
Perform the alignment of the HTK 1200N at room temperature and with embedded sample holder flange.
The **alignment slit melts**, if it is used at elevated temperatures!

2. Remove the sample holder flange and place the alignment slit onto the sample holder such that the 0.1mm slit is positioned in the beam path.
3. Insert the sample holder flange with mounted alignment slit in the chamber.

Align the height as follows:

The height (z-position) of the HTK 1200N relative to the X-ray beam can be adjusted with the alignment equipment mounted on the base-plate of HTK 1200N.

1. Slightly loosen the screws (1) which fix the adapter to the chamber base-plate so that the chamber can move up and down on the adapter.



2. Move the chamber up or down by means of the adjusting screw (2).
3. When you have found the desired position, tighten the fixing screws (1) again.

Adjusting screw:

- turn clockwise -> move chamber upwards
- turn anti-clockwise -> move chamber downwards

Alternative method

An alternative method of height adjustment is to let the X-ray beam pass above the alignment slit, and turn the HTK 1200N housing until the X-ray beam is achieving maximum intensity.

The aim of this method is to find the alignment slit more easily by lifting the sample holder (using the alignment equipment) until the intensity reaches a maximum again. Then, turn the HTK 1200N housing to exactly align the sample holder with the Θ-zero line.

Measure a standard sample to check for proper height alignment. Compare the measured values with known standard values.

NOTICE
Risk of Damage

- Remove the alignment slit before starting a measurement!
- Do not heat up the chamber when the alignment slit is still mounted! This will lead to destruction of the alignment slit and the sample carrier.

5.3 Height Adjustment with Motorized Alignment Stage

Motorized alignment stages are remote controlled. The height (z-position) of HTK 1200N can be adjusted from outside the diffractometer enclosure.

Benefits of a motorized alignment stage compared to manual alignment:

- The alignment stage makes the z-alignment procedure much easier: no more loosening of screws at the adapter, no inaccuracies due to tolerances at mechanical parts.
- The alignment stage allows re-alignment to be performed at any temperature and while the X-ray beam is on.
- The alignment stage offers an accuracy of < 5 µm.
- Digital display of the chamber position is possible.

TIP: *For information about the operation of the motorized alignment stage, read the Alignment Stage Instruction Manual.*

5.4 Adjustment of Chamber Rotation

The HTK 1200N chamber must be aligned on the goniometer so that the sample holder surface is parallel to the θ-zero line of the diffractometer (ω-alignment).

TIP: *The procedure for rotating HTK 1200N relative to the goniometer depends on the type of diffractometer. Refer to the Diffractometer Instruction Manual for details.*

5.5 Alignment procedure

NOTICE
Risk of Damage
The procedure described in this section requires that the detector is moved into the **primary X-ray beam**. Always **use** an **appropriate beam attenuator** and read the relevant sections in the *Diffractometer Instruction Manual* before you start the alignment.

If the chamber rotation is not aligned, carry out the procedure described below with the standard sample holder first (coarse alignment) and repeat it with the aligment slit.

If the chamber is rotated too much relative to the θ-zero line of the diffractometer, the X-ray beam will not pass through the alignment slit.

When the chamber is roughly aligned parallel to the θ-zero line, replace the standard sample holder with the alignment slit.

TIP: *Refer to chapter 7.1 for information about exchanging the standard sample holder and the alignment slit.*

Carry out the alignment as follows:

1. Put an appropriate beam attenuator in the primary beam path.
2. Generate a narrow X-ray beam (small beam height) and select a detector area which is larger than the beam.
3. Set the goniometer to θ = 0 ° / 2θ = 0 °.
4. Move the chamber to the θ-zero line as follows:

If you have mounted the standard sample holder:

- Lower the chamber with the alignment stage until no parts are in the X-ray beam path and determine the maximum beam intensity I_{max}.
- Lift the chamber until the X-ray beam is completely blocked and determine the background intensity I_{bg}.
- Move the chamber down until the beam intensity $I = ½ \bullet (I_{max} - I_{bg})$, which means that the upper edge of the alignment slit bisects the X-ray beam.

If you have mounted the alignment slit:

- Move the chamber up and down until the X-ray beam passes through the alignment slit with maximum intensity I_{max}.

5. Carry out an ω-scan and measure the beam intensity.
   Determine the ω-offset of the maximum of the intensity profile.

TIP: *For θ-θ diffractometers, rotate source + detector around the centre axis of the goniometer.*

For θ-2θ diffractometers, rotate the chamber around the centre axis of the goniometer.

When starting with the ω-alignment, do the ω-scan with ±3°. As the alignment gets better, reduce the scan range.

6. Depending on the ω-offset (Δω), rotate the chamber as described in the instruction manual for the diffractometer:
   ω-offset negative (–) -> rotate clockwise
   ω-offset positive (+) -> rotate anti-clockwise
7. Fix the chamber on the goniometer.
8. Set the goniometer back to θ = ω = 2θ = 0 ° and re-align the chamber height (z-position) as described in step 4.
9. Repeat steps 5 – 8 until the offset Δω is < 0.05 °.
10. Measure a reference sample to check the alignment. Compare the measured peak position values with known standard values.

NOTICE
Risk of Damage

- Remove the alignment slit before starting a measurement!
- Do not heat the chamber when the alignment slit is still mounted.

5.6 Checking the Instrument Condition

Before you start operation:

- Make sure that HTK 1200N is installed correctly and that no collisions between the sample chamber and components of the diffractometer can occur.
- Make sure that the cooling-water hoses connected to the inlet/outlet nozzles are protected against unintentional disconnection.
- Check whether all cables are correctly mounted before switching on the CCU 1000 Combined Control Unit.
- Check whether the foil on the X-ray windows of the sample chamber is undamaged and clean.
- Make sure a sample holder is mounted inside the sample chamber.

5.7 Turning on the Instrument

If possible, switch on CCU 1000 with the cooling water turned OFF in order to test the water flow monitor when starting operation.

Proceed as follows:

1. Press the mains switch on the front panel of CCU 1000.

The micro controller of CCU 1000 will initialize:

- All display elements and all LEDs on the front panel will light up for a few seconds.
- After a few seconds, the micro controller switches to the normal mode of operation. CCU 1000 is in the following status:
  - the temperature set point (SP) is set to the default value of 25 °C
  - the HEATER is off
  - the Error LED is flashing
  - the display shows E03 to indicate that there is no water flow

2. Turn on the cooling water supply.
   - The alarm message disappears and the flashing of the Error LED stops.
3. Press the HEATER button to start control of the sample holder temperature.

If the instrument condition is OK, the green HEATER LED lights up and all error messages disappear. The sample holder is now heated to 25 °C with the default heating rate SPR.

If the status of the system is not OK, an error code appears on the display, the controller turns off the heater and the Error LED is flashing.

6 Operating CCU 1000

This chapter describes the operation of the CCU 1000 Combined Control Unit. CCU 1000 can be operated manually or by remote control.

- Manual control is done with the keys on the front panel of CCU 1000. Manual operation is described in chapter 6.5.
- Remote control is done with a computer interfaced to CCU 1000 via the serial RS 232 interface and with suitable software. See chapter 6.6 for more information.

CCU 1000 can only store one target value for temperature and heating/cooling rate at a time. Programming temperature profiles into CCU 1000 is not possible on the controller itself. However, the delivered software for the controller allows to program a temperature profile (see the corresponding software manual that was delivered with the instrument).

6.1 Front Panel of the Instrument

The front panel of CCU 1000 contains the keypad, the status LEDs and the display which are described in the following section.

Fig. 13: Front panel of CCU 1000

6.2 Keypad

The keypad of CCU 1000 has 6 keys to select parameters and enter values.

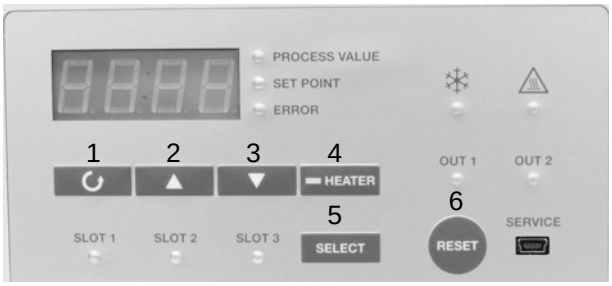


Fig. 14: Keypad of CCU 1000

No	<Key>	Function
1	SCROLL	Switches to next parameter.
2	UP	Increases the value of the currently displayed parameter.
3	DOWN	Decreases the value of the currently displayed parameter.
4	HEATER	Switches the heater on and off.
5	SELECT	Short: selects a parameter.
6	RESET	Resets an error.

6.2.1 Status LEDs

There are three types of status LEDs. The first type of LEDs is located on the right side of the display and gives additional information on the value that is shown on the display.

Fig. 15: LEDs for value of CCU 1000

LED on	Display shows
Process value	actual temperature
Set Point	the actual set point
Error	an occurring error

The next type of LEDs on the right side of the front panel gives information about the status of the instrument.

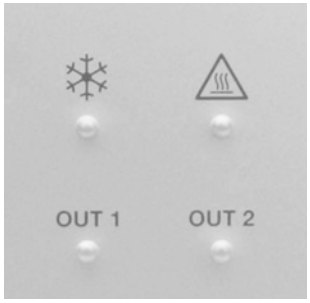


Fig. 16: LEDs for status of CCU 1000

LED	Condition
COOLING	cooling active
HEATING	heater active
OUT1	additional connector OUT1 on rear panel of CCU 1000 is active
OUT2	additional connector OUT2 on rear panel of CCU 1000 is active

The HEATING LED has a special function besides giving information about active/inactive heating process. If this LED is blinking, the instrument is in the status STANDBY. This status is active if the sample holder flange of the instrument is not mounted. After mounting the sample holder, the LED stops blinking and the instrument is ready to use.

The last type of LEDs indicates the actual active slot of CCU 1000. At delivery only slot 1 is used.

Fig. 17: LEDs for active slots of CCU 1000

6.3 Display

The display consists of four 7-segments elements that show depending on the LEDs on the right side of the display (see chapter 6.2.1) different information. In the normal operation mode (process value LED is green), the display shows the actual temperature of the sample holder of HTK 1200N.

By pressing the SCROLL button, the set point LED is green, which means that the target set point temperature can now be changed by using the UP or DOWN button.

By pressing the SCROLL button twice, the Error LED is red. The display in this case contains information about the number of errors that are currently active and also the corresponding error code. This is shown in the following way:

XExy

X…number of actual active errors

E…indicates an error

xy…corresponding error code

By pressing the UP/DOWN button it is possible to switch between all active error codes.

The complete list of all possible errors is shown in chapter 10.1.

6.3.1 Navigation Diagram of CCU 1000

The complete navigation diagram is shown below:

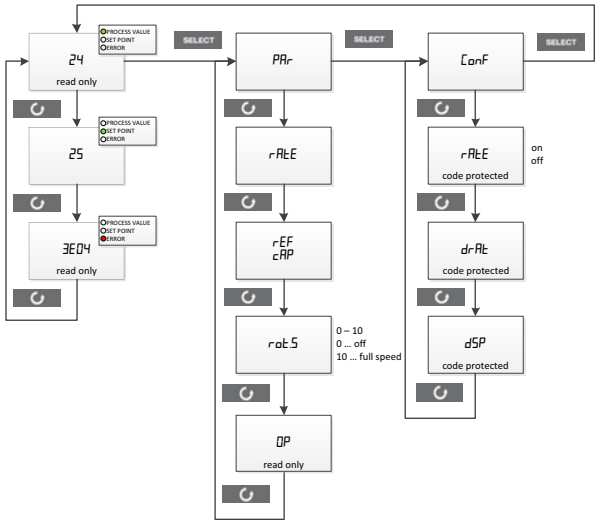


Fig. 18: Navigation diagram

The navigation diagram of CCU 1000 contains three different branches. The first, which contains the process value, the set point and the error messages is already described in the previous chapter.

By pressing the SELECT button it is possible to switch between the branches of the navigation diagram. Pressing the button once, gives access to the parameter page.

Parameter Page

The following parameters can be found in the parameter page:

Parameter	Description	Note
rAte	Heating/Cooling Rate value [°C/min]	Default value: 20 °C/min
rEF cAP	Configuration for reflection or for use with capillary extension. Limits the operating temperature depending on the used equipment.	Reflection: max. 1200 °C Capillary Extension: max. 1000 °C
rot.S	Sample holder rotation activation; values between 0 (no rotation) and 10 (full rotation speed) can be adjusted	Only for measurements with the capillary sample holder of HTK 1200N
OP	shows the actual heating power of the system in %.	

Changing of the values within the parameter page is possible by using the UP or DOWN buttons.

Config Parameters

Pressing the SELECT button twice gives access to the config mode of the instrument. Parameters that can be changed in the config mode are described in the following table.

Parameter	Description	Note
rAte: On Off	Allows to deactivate the heating rate. This results in max. heating power all the time.	This parameter is password protected.
drAt	Allows changing of the default heating/cooling rate (20 °C/min) from 1 - 150 °C	This parameter is password protected.
dSP	Allows changing of the default set point (25 °C) from 25 - 50 °C.	This parameter is password protected.

6.4 Turning the Heater On and Off

Pressing the heater button turns on the heating power supply to the heater inside the sample chamber. The red and the green HEATER LED lights up and the sample holder is heated to the temperature set-point SP with the default heating rate. Changing of the heating rate is described in the previous chapter.

If the heater button is pushed again, heating is stopped. Temperature set-point SP and ramp rate SPR are reset to the default values. The green HEATER LED is switched off.

WARNING

Hot surface.
There can be dangerously hot surfaces inside the sample chamber even if the heater has been switched off. Always check on the display of CCU 1000 that the sample holder temperature is below 50 °C before you open the sample chamber and touch any parts inside.

TIP: *After pressing the heater button it takes a short time until a reaction of the CCU 1000 is visible.*

6.5 Manual Control of CCU 1000

Manual operation of HTK 1200N is done with the keys on the front panel of CCU 1000. Proceed as follows to heat or cool the sample to the desired temperature:

1. Make sure that the heater is switched on.
2. Set the desired heating/cooling rate on the parameter page of CCU 1000. (See chapter 6.3.1.)
3. Push the scroll button one time (SP LED green) and set the desired target temperature by using the UP/DOWN buttons.
4. Wait until the displayed sample holder temperature has reached the target value.

TIP: *If you have chosen a large heating/cooling rate, wait 1 minute before you start the X-ray scan to make sure the sample surface has reached a constant temperature.*

6.6 Remote Control of CCU 1000

Usually CCU 1000 is integrated in the software that controls the X-ray diffractometer. For more information about controlling HTK 1200N, read the instruction manual of the diffractometer control software.

Alternatively it is also possible to remote control the instrument with the delivered Nambicon software (Non-AMBIent-CONtrol) by Anton Paar GmbH. For further information see the corresponding manual of the Nambicon software.

The NAMBICON software is included in the delivery of the instrument. Please follow the instructions in the manual of the software for corresponding installation and use.

7 Handling the Sample Holder and Sample

Two different types of sample holder flanges are available for the HTK 1200N: the **stationary sample holder** (Fig. 19) and the **sample spinner** (Fig. 20).

The pedestal and the cup of the sample holder are made of alumina providing high corrosion resistance.

Fig. 19: Stationary sample holder

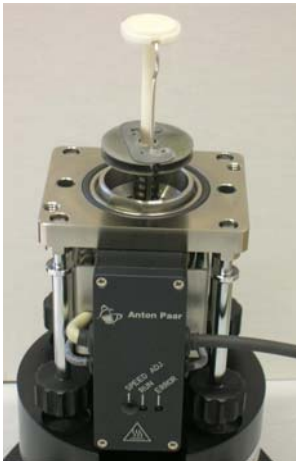


Fig. 20: Sample spinner

7.1 Mounting/Demounting the Sample Holder Flange

The HTK 1200N is delivered with the sample holder mounted.

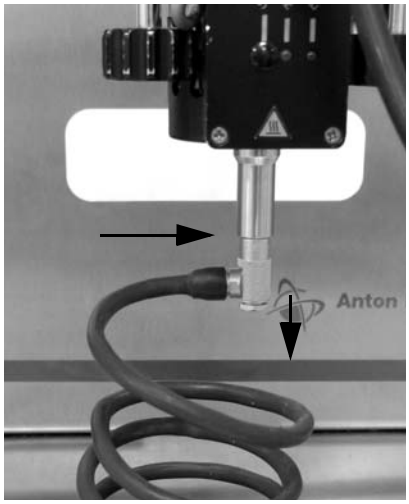
The procedure of mounting the sample holder is the same for both sample holder flanges.

To replace the sample holder, proceed as follows:

CAUTION

Hot surface
Make sure that the heater is off and the sample holder is at room temperature before touching it. We recommend using a suitable meter for checking the temperature of the sample holder surface.

1. Disconnect the cable from the sample holder at the bottom of the sample holder flange. Prepare the supplied stand for the sample holder flange.



2. Hold the sample holder flange with one hand and unscrew the four fixing screws with the other hand. Remove the sample holder flange carefully from the chamber and put it on the stand.

3. You can now mount or demount the sample carrier and the fixing ring or the alignment slit.
   Refer also to *chapter 7.2*.
   To replace the sample holder from the sample holder flange please see *chapter 7.1.1*.
4. Insert the sample holder flange again into the chamber and fix the four screws.
   Be careful not to harm the guiding rods.

Before mounting the sample holder to the chamber again, some general requirements have to be fulfilled.

- Carefully clean the guiding rods and surfaces of both the flange and the chamber.
- Lightly coat the built-in O-ring with vacuum grease!

5. Connect the cable to the sample holder on the sample holder flange.

NOTICE
Risk of damage
Before operating the **rotating** sample holder beware of the shape of the cable for sample holder flange. For safe operation do NOT tear at the cable and preserve a minimum of four windings.

7.1.1 Exchanging the sample holder body

To replace the sample holder body from the sample holder flange first demount the sample holder flange from the chamber (refer to chapter 7.1) and then follow the instruction below:

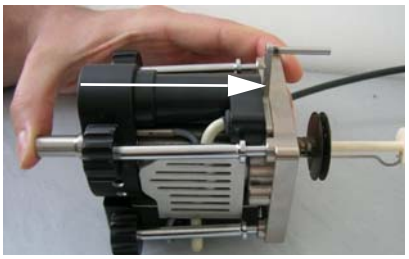
1. Unscrew the two screws at the bottom of the sample holder flange and demount the panel.

2. Loosen the locknut at the bottom of the sample holder flange with 4-5 rotations.

3. Remove the whole sample holder unit from the flange as it is described step by step in the instruction below.

NOTICE
Risk of damage
- Be careful not to harm the thermocouple!
- Do not apply lateral force to the sample holder!
- Always store the sample holders in the accessory box included in the delivery.

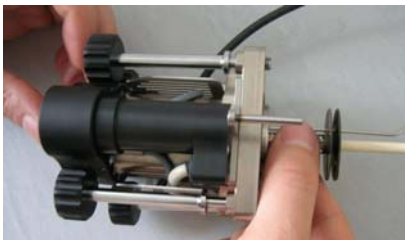
   a. Press the loosened locknut against the sample holder flange.



b. Then completely unscrew the locknut by holding the sample holder at the temperature shieldings.

c. Remove the locknut.

d. Carefully push against the thermocouple plug to slide the whole sample holder out of the sample holder flange.

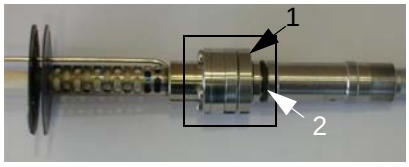


4. Carefully mount the new sample holder into the flange.
5. Fix the locknut at the bottom of the sample holder flange.
6. Mount the panel and fix the two screws at the bottom of the sample holder flange.
7. Insert the sample holder flange into the chamber as described in *chapter 7.1*.

7.1.2 Matching Sample Holder Height to Sample Thickness

The sample holder can be manually aligned by ± 2.5 mm with the alignment equipment of the HTK 1200N or automatically by ± 3.5 mm using the motor-driven alignment stage.

For samples thicker than 2.5 mm (3.5 mm), the sample holder height can be varied by demounting the spacer ring (1). The 4 mm spacer ring is included in the delivery and is already mounted.



CAUTION

Hot surface
Make sure that the sample holder is at room temperature before touching it. We recommend using a suitable meter for checking the temperature of the sample holder surface.

TIP: *Before removing the spacer ring make sure that the HTK 1200N is aligned properly! If not, re-align by means of the alignment slit (refer to chapter 5 for further information).*

To remove the spacer ring (1), demount the sample holder first (see *chapter 7.1* and *chapter 7.1.1*). Then remove the O-ring (2) and afterwards the spacer ring. Mount the O-ring and then the sample holder again.

When mounting the spacer ring again, demount the O-ring first and make sure that the centering pins of the spacer ring rest in the corresponding grooves. Then mount the O-ring again and then the sample holder.

7.2 Applying a Sample

CAUTION

Hot surface
Make sure that the sample holder is at room temperature before touching it. We recommend using a suitable meter for checking the temperature of the sample holder surface.

The sample holder of the HTK 1200N consists of the sample holder body, the sample carrier and a fixing ring.

For faster work, the sample is prepared on the sample carrier and the sample carriers are simply exchanged.

Proceed, as follows:

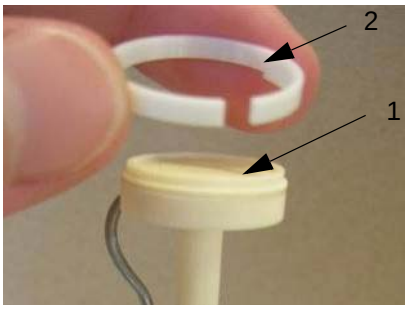
1. Disconnect the cable for sample holder flange from the sample holder.
2. Hold the sample holder flange and unscrew the four fixing screws. Remove the sample holder flange from the chamber and put it on the supplied stand.

NOTICE
Risk of damage
When taking the sample holder flange out of the chamber, immediately put it on the supplied stand to avoid any damage.

3. Apply a sample to an empty sample carrier, as described in *chapter 7.2.1* and *chapter 7.2.2*.
4. Place the sample carrier (1) with the sample on top of the sample holder body.

TIP: *Make sure that the contact surfaces between sample carrier and top of the sample holder body are free of dust and sample residues to provide for a good thermal contact!*

5. Put the fixing ring (2) around the edge of the sample carrier and carefully pull it downwards to fix the sample carrier.



6. Insert the sample holder flange into the chamber

NOTICE
Risk of damage
Before operating the **rotating** sample holder beware of the shape of the thermocouple cable. For safe operation do NOT tear at the cable and preserve a minimum of four windings.

NOTICE
Risk of damage
The fixing ring is very fragile.
Please don‘t press or extend the fixing ring.

7.2.1 Powder Samples

NOTICE
Risk of damage
Make sure that the materials of the HTK 1200N are chemically resistant against the sample and reaction products! Please refer to *Appendix A*.

To apply the sample, use a spatula to press the powder into the groove of the sample carrier and subsequently smooth the powder's surface by means of a glass platelet.

TIP: *Make sure that the contact surfaces between sample carrier and top of the sample holder body are free of dust and sample residues to provide for a good thermal contact!*

7.2.2 Solid Samples

NOTICE
Risk of damage
Make sure that the materials of the HTK 1200N are chemically resistant against the sample and reaction products! Please refer to *Appendix A*.

Simply place solid samples into the groove of the sample carrier.

The alignment equipment respectively the alignment stage allows you to compensate for the difference in height between sample and sample holder groove.

TIP: *Make sure that the contact surfaces between sample carrier and top of the sample holder body are free of dust and sample residues to provide for a good thermal contact!*

7.3 Temperature Validation

The two most important aspects for non-ambient XRD are a highly uniform temperature in the sample and a reliable temperature measurement. Both conditions are fulfilled in the HTK 1200N:

- The **design of the oven** provides a high temperature uniformity in the interior and consequently guarantees a homogeneous temperature distribution in the sample.
- The sample temperature is measured and controlled with a Pt10Rh-Pt (type S) **thermocouple**. Its tip, which is the most temperature-sensitive part, is located **right underneath the sample position**. This set-up provides a very good correspondence between the measured temperature and the actual sample temperature.

TIP: *A possible temperature deviation in the HTK 1200N is not only influenced by its design, but also by the thickness, thermal conductivity, emissivity of the sample and the thermal contact of the sample to the sample holder.*

To account for temperature deviations in heating attachments for non-ambient X-ray diffraction, a temperature calibration should be performed.

Temperature calibration can be done by observing **known phase transitions:** either melting points or more preferably solid-solid phase transitions.
For some information on this issue refer to e.g. "Industrial Applications of X-ray diffraction", edited by F.H. Chung and D.K. Smith, Marcel Dekker Inc., 2000, pages 891-901.

Alternatively the **thermal expansion of known materials** e.g. oxides such as MgO or alumina can be used to calibrate the temperature.
The thermal expansion data of different materials are collected in "Thermophysical Properties of High Temperature Solid Materials", Y.S.Touloukian et. al, Macmillan, New York, 1977.

TIP: *Make sure that the heating of the sample is performed slowly to prevent that the sample is detached from the sample carrier and to guarantee temperature equilibration of the sample.*

Please note that there are currently no standard reference materials available for the calibration of non-ambient XRD attachments.

8 Operation

The HTK 1200N allows for measurements under various atmospheric conditions (air, vacuum, inert gas) in the temperature range from room temperature to 1200 °C.

8.1 Before Turning on Power

NOTICE
Risk of damage
Make sure that the HTK 1200N is installed according to the instructions in *chapter 4*.

Take care that the cooling water hoses connected to the inlet/outlet nozzles are protected against unintentional disconnection.

Refer to *Appendix B*- for detailed information on the requirements for cooling water quality.

Check the flow controller of the CCU 1000 periodically for proper functioning.

Only turn on the mains supply of the CCU 1000 Combined Control Unit, when the instrument is fully installed.

Always operate the HTK 1200N with the cooling water circuit on. This is absolutely necessary to avoid effects of the working temperature on the goniometer.

Do not exceed the maximum permissible pressure of 4 bar to prevent bursting of the hoses.

Make sure that the alignment slit is demounted from the sample holder before operating the chamber.

8.2 Operation under Air Atmosphere

8.2.1 Operation at Room Temperature

Operation is performed in the same way as with the standard sample holder. No special precautions have to be taken.

8.2.2 Operation as Heating Chamber

Operation is performed in the same way as with the standard sample holder.

The CCU 1000 Combined Control Unit by Anton Paar GmbH is available for the HTK 1200N. For detailed ordering information, see *Appendix E*. The temperature control unit is used to control the sample temperature by means of the chamber's heating system as well as the thermocouple.

Keep the following sequence, when starting the operation of the HTK 1200N at high temperatures:

1. Mount the sample and insert the sample holder flange into the chamber, as described in *chapter 7.1* and *chapter 7.2*.

CAUTION

Hot surface
During operation and even after turning off the instrument, temperatures at the sample holder can reach up to 1200 °C. Make sure that the sample holder is at room temperature before handling it.

NOTICE
Risk of damage
Check the chemical resistance of the materials in the HTK 1200N BEFORE the experiment.

2. Switch on the cooling water circuit.
3. Switch on the temperature controller and set the required sample temperature.

NOTICE
Risk of damage
Make sure that the HTK 1200N is fully installed, refer to *chapter 4*, before switching on the CCU 1000 Combined Control Unit.

Always operate the HTK 1200N with the cooling water circuit on!

Refer to *Appendix B* for detailed information on the requirements for cooling water quality.

Take care that the cooling water hoses are protected against unintentional disconnection!

Do not exceed a maximum cooling water pressure of 4 bar!

Check the flow controller of the CCU 1000 periodically for proper functioning.

8.3 Operation under Vacuum

Dependent on your actual measuring problem, it may be necessary to evacuate the chamber. A vacuum of approx. 10^{-4} mbar can be reached in the HTK 1200N with a turbo-molecular pump.

Anton Paar GmbH offers a vacuum equipment as an option for the HTK 1200N. For further information refer to *Appendix E*.

The evacuation of the chamber is performed through the vacuum flange in the lid or through the adapter axis.

Keep the following sequence, when starting the operation of the HTK 1200N under vacuum:

1. Mount the sample and insert the sample holder flange into the chamber, as described in *chapter 7.1* and *chapter 7.2.*

CAUTION

Hot surface
During operation and even after turning off the instrument, temperatures at the sample holder can reach up to 1200 °C. Make sure that the sample holder is at room temperature before handling it.

NOTICE
Risk of damage
Check the chemical resistance of the materials in the HTK 1200N BEFORE the experiment.

2. Apply vacuum.
3. Switch on the cooling water circuit.

NOTICE
Risk of damage
Always operate the HTK 1200N with the cooling water circuit on!

Take care that the cooling water hoses are protected against unintentional disconnection!

Do not exceed a maximum cooling water pressure of 4 bar!

Check the flow controller of the CCU 1000 periodically for proper functioning.

4. Switch on the temperature controller and set the required sample temperature.

NOTICE
Risk of damage
Make sure that the HTK 1200N is fully installed, refer to *chapter 4*, before switching on the CCU 1000 Combined Control Unit.

Make sure that venting of the chamber is carried out slowly to prevent that the sample is detached from the sample holder.

Venting of the chamber should be carried out shortly before turning off the vacuum pump to prevent oil from being sucked back into the vacuum hose and even into the chamber.

Take care that all gas connectors are sealed by temporary covers.

At the surface of the Kanthal heater an oxide layer is formed which protects the wire.
After work in vacuum or inert gas, it is recommended to regularly regenerate this oxide layer by simply heating the HTK 1200N up to 1000 °C in air. This will extend the life time of the heater considerably

8.4 Operation using Various Gases (except air)

The HTK 1200N is made of chemically very resistant materials and can be also operated using various gases (preferably inert gas). Refer to *chapter 8.2.2* for general comments on the operation and to *chapter 4.6* for further information on the gas supply.

NOTICE
Risk of damage
Check and replace the gas hoses regularly!

Before applying aggressive gases, make sure that the materials in the HTK 1200N are resistant to these gases! Refer to *Appendix A*

It is the responsibility of the customer to provide all the set-up (tubings, valves,..) necessary for proper work with the HTK 1200N. This implies that the local safety regulations especially for work at elevated temperatures need to be fulfilled.

Make sure that all fittings are tight and mounted properly and an appropriate exhaust hose is connected to the safety valve's outlet.

Do NOT work with poisonous, aggressive or explosive gases.

At the surface of the Kanthal heater an oxide layer is formed which protects the wire.
After work in vacuum or inert gas, it is recommended to regularly regenerate this oxide layer by simply heating the HTK 1200N up to 1000 °C in air. This will extend the life time of the heater considerably.

9 Putting HTK 1200N out of Operation

When you stop using HTK 1200N for a short period without removing it from the diffractometer, proceed as follows:

1. Cool down the sample holder to room temperature.
2. Turn off the heater with the HEATER button.
3. Remove the sample and put the sample holder back into the sample chamber.
4. Turn off the cooling water.
5. Turn off CCU 1000 completely.

In order to remove HTK 1200N from the diffractometer continue as follows:

1. Completely turn off CCU 1000 (mains switch).
2. Disconnect all electrical cables from the sample chamber.
3. Disconnect the couplings of the cooling water hoses.
4. Dismount the sample chamber from the goniometer.
5. Store the sample chamber in a safe place, preferably on a stage mount offered by Anton Paar GmbH.

10 Troubleshooting

10.1 Error Messages

Alarm Message	Alarm Name	Description
E01	Sensor Break	The electronic circuit of the temperature sensor is interrupted.
E02	Loop Break	The electronic circuit of the heater is interrupted.
E03	No Water Flow	There is no or too little water flow.
E04	Housing Overtemperature	The protective thermoswitch in the sample stage housing is activated.
E05	Air Cooling Failure	Air cooling flow is off or insufficient.*
E06	Sample Holder not mounted	The contact switch does not detect the sample holder.
E07	Chamber not closed	The contact switch does not detect the lid of the instrument.*
E08	Current cable disconnected	The contact switch does not detect the current cable.*
E09	CCU 1000 Air Cooling Fail	The protective thermoswitch inside CCU 1000 is activated.*
E10	Other Messages	Internal errors of CCU 1000 --> contact Anton Paar GmbH.
E11	Sensor Spread Failure	Activated in case of too big temperature difference between the sensors (sample holder and heater).*
E12	Flow-Control Extension missing	Activated in case of missing flow controller.*
E98	No / Unknown Instrument present	Activated in case that there is no connection between chamber and CCU.
E99	No / Unknown module present	Activated in case that there is no power module present.

* not applicable for HTK 1200N (valid for other non ambient attachments)

10.2 General Messages on Display

Message	Description
8888	Normal during booting. If the message doesn´t disappear after booting, the power module doesn´t boot properly. Check the error code.
9999	This value is shown in case of sensor break. Check the error code.

10.3 Error Analysis

1. CCU 1000 does not start after turning it on.

cause	action
mains cable not properly connected	Make sure the CCU 1000 is properly connected to mains.
wrong **supply voltage**	Required voltage AC 230 V ± 10 %.
mains fuse blown	Replace mains fuses (see chapter 11.4).

cause	action
internal thermoswitch switched off (CCU 1000 was overheated)	reset the thermoswitch --> contact Anton Paar GmbH.

2. Sensor Break (E01)

cause	action
temperature sensor cable not properly connected	Check temperature sensor cable and connectors.
Thermocouple damaged	Visual check of Thermocouple for damage.
plug of Thermocouple cable	Connect the plug of Thermocouple cable on the connector box and the sample holder.
HTK 1200N or CCU 1000 malfunction	⇒ Contact Anton Paar GmbH.

3. Loop Break (E02)

cause	action
heater fuse blown	• Check the heater resistance. --> contact Anton Paar GmbH. • Replace heater fuse --> contact Anton Paar GmbH.
heater defective	⇒ Contact Anton Paar GmbH.
heater cable not properly connected	check heater cable and connectors

4. No water flow (E03)

cause	action
water hoses disconnected	Connect the water hoses on HTK 1200N to the cooling water circuit.
no **cooling water**	Turn on the cooling water.
flow controller disconnected	Connect the water flow controller to the CCU 1000.
insufficient **cooling water supply**	Make sure the water pressure is between 2 – 4 bar and the flow rate is 0.7 – 1.5 l/min.
flow controller does not switch	⇒ Contact Anton Paar GmbH
cooling ducts inside **HTK 1200N** housing choked	Check the cooling water quality (algae?). Disconnect the water hoses, try to blow out choking material with compressed-air and rinse with clean water.

5. Housing Temperature too high (E04)

cause	action
no cooling of housing	• Check if HTK 1200N water hoses are connected to the cooling water circuit • Check if cooling water is turned on.
insufficient cooling of housing	• Check if the cooling water flow before HTK 1200N meets the requirements. • Check if the cooling water flow after HTK 1200N meets the requirements. • If the flow resistance of the HTK 1200N chamber is too high, contact Anton Paar GmbH.
cable to thermoswitch defective	⇒ Contact Anton Paar GmbH.

6. Communication errors with the control software

cause	action
bad **RS 232 connection**	check RS 232 cable (null modem required) and connectors
wrong settings in **diffractometer software**	check settings for *COM port*, *baud rate*, *string format* and *device address*
conflicting settings in CCU 1000 and diffractometer software	⇒ contact your diffractometer manufacturer or Anton Paar GmbH

7. No / Unknown Instrument present (E98)

cause	action
Cables between chamber and CCU not properly connected	Check connections between chamber and CCU.

8. No / Unknown module present (E99)

cause	action
High-Power module not installed correctly	Make sure the High-Power module seats correctly (no gap on the side or inserted crookedly). If not, contact your diffractometer manufacturer or Anton Paar GmbH.
High-Power module not installed	Check if the High-Power module is installed. If not, contact your diffractometer manufacturer or Anton Paar GmbH.

10.4 Technical Support

If you need technical support, please contact the local service organization of your diffractometer manufacturer or Anton Paar GmbH in Graz, Austria.

Contact details of Anton Paar GmbH:

Anton Paar GmbH
Anton-Paar-Strasse 20
A-8054 Graz
AUSTRIA / Europe

Tel: +43 316 257-0
Fax: +43 316 257-257

E-mail: info@anton-paar.com
Web: www.anton-paar.com

11 Maintenance

11.1 Routine Maintenance

The HTK 1200N represents a mechanical precision instrument and thus has to be handled with particular care. The chamber is virtually maintenance-free. Always keep the chamber with the sample holder flange installed.

To guarantee trouble-free and reliable operation, please follow the hints below:

- Remove the protective cap of the vacuum feedthrough only shortly before fitting the appropriate connector.
- Periodically remove residual sample material and clean the chamber.
- Periodically check cooling water circuit.
- Periodically check the thermocouple for proper functioning. If required, validate the temperature readout with an appropriate standard sample.
- Make sure that the O-ring of the sample holder flange is always slightly coated with vacuum grease.
  Periodically clean the O-Ring, the groove and the sealing surface with alcohol.

NOTICE
Risk of damage.
After long-term operation, we recommend cleaning both the O-ring and the groove using a lint-free paper towel wetted with a suitable solvent, e.g. alcohol. After this, slightly cover the O-ring with vacuum grease (contained in the accessory box).

11.2 Check the Functioning of the Flow Controller

- Check the proper functioning of the flow controller once a week as described in chapter 5.7.

NOTICE
Risk of damage.
Do not operate the HTK 1200N chamber if the water flow controller does not work properly.

11.3 Exchanging of Parts

11.3.1 Switching off the Instrument

1. Switch off the CCU 1000 by using the power switch on the front side of the instrument.
2. Unplug all cables from the rear side of the instrument.

Perform the recommended maintenance work regularly to ensure the smooth long-term operation of the instrument.

11.3.2 Replacing the Window Foil

The window in the housing is covered by either of the following foils:

- When operating the HTK 1200N at temperatures **up to 1000 °C** an aluminium-coated Kapton foil of 125 µm thickness is used to cover the housing window. This foil guarantees minimum absorption.
- For temperatures **above 1000 °C** a combination of a graphite foil and a non-coated Kapton foil of 125 µm thickness is used.

NOTICE
Risk of damage
Above 1000 °C it is crucial to mount the combination of a graphite foil and a non-coated Kapton foil! The graphite foil protects the Kapton foil from any damage due to high temperature!

To seal the window, the foil is pressed towards an O-ring using two clamps.

The combination of graphite foil and non-coated Kapton foil is mounted upon delivery.

NOTICE
Risk of damage
Check the window foil regularly and exchange it, if it is leaky or foggy!

To mount or exchange the window foil, proceed as follows:

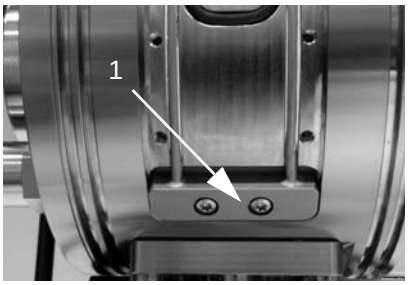
1. Loosen the screws of the two clamps with the screw driver contained in the accessory box.

2. Remove the clamps and the foil.

3. If required, clean the area of window, O-ring and O-ring groove. Slightly coat the O-ring using vacuum grease.
4. *Aluminium-coated Kapton foil:*
   Put the new foil centrically over the window opening. The aluminium-coated side has to be inside.

Combination of graphite foil and non-coated Kapton foil:
Place the graphite foil within the O-ring first and then put the non-coated Kapton foil centrically over the graphite foil.

5. Mount the two clamps again.
6. Fasten the screws of the clamps, beginning from the side where the cooling tube (1) is fastened.



7. Only a minimum slit should remain between the clamps.

11.3.3 Replacing the Thermocouple

If the thermocouple is defective, demount the sample holder as described in chapter 7.1.

It is of crucial importance for reliable temperature measurement that the thermocouple is thoroughly embedded in the sample holder. For this reason the thermocouple cannot be exchanged on its own, but only together with the sample holder.

Contact your local Anton Paar GmbH representative for exchange.

11.3.4 Replacing the O-rings

Generally, commercially available O-rings made of Viton are used.

1. Remove the O-ring from the O-ring groove.

NOTICE
Rsik of damage

- Do not use pointed tools.
- Do not damage the sealing surface or the edges of the groove.

2. Clean the groove and the sealing surface with a suitable solvent, e.g. alcohol.
3. Coat the new O-ring with a thin film of vacuum grease (contained in the accessory box).
4. Insert the O-ring into the O-ring groove.

11.4 Checking and Replacing Main Fuses

DANGER

High voltage
Make sure that BEFORE performing any service/maintenance work of the CCU 1000, the instrument is disconnected from the mains supply.
Only use the specified fuse types

Service and/or maintenance procedures which involve checking and replacing fuses may only be performed by authorized service personnel.

Two mains fuses are located in the fuse box (1) on the backside of the CCU 1000.

Exchange the mains fuse as follows:

1. Switch off CCU 1000 and disconnect all cables from the rear side.
2. Use a small screw driver to push out the fuse holder on both sides.
3. Check the fuse or exchange the defective fuse. (The two mains fuses are made of ceramic. Spec.: 2 x T 8 A H)

NOTICE
Risk of damage.
Only use the specified fuse types to avoid damage.

4. Put the fuse box back into the cavity of the power supply.

Appendix A: Technical Data

Temperature range

Operating temperature:	Room temperature to 1200 °C Helium: Room temperature to 1000 °C

Atmospheres

Atmospheres applicable:	Vacuum (10^{-4} mbar), air, inert gas
Max. operating pressure:	1 bar above atmospheric pressure

Temperature measurement & control

Control unit:	CCU 1000
Typical heat-up time - RT to 1200°C	In air: 15 min.
Temperature sensor:	Pt10Rh-Pt thermocouple (type S)
Temperature accuracy[1)]:	≤ ±2 °C
Temperature stability[1)]:	≤ ±0.5 °C
Heating method:	Oven heater

[1)]Sample holder temperature; values depending on operating conditions

Sample spinner

Voltage supply:	DC 6.5 V and 12 V
Power consumption:	max. 2 Watt

X-ray window:

Angle of incidence:	0 - 164 ° 2Θ
Window opening:	10 mm / 200°
Foil material:	Aluminium-coated Kapton (up to 1000 °C) or Combination of graphite and Kapton foil (above 1000 °C)

Chamber:

Diameter:	145 mm
Height adjustment alignment equipment (manual) alignment stage (automatic)	 ± 2.5 mm ± 3.5 mm
Housing:	Nickel-plated brass
Weight:	6.5 kg

Sample holder:

Sample holder:	Alumina (other materials on request)
Sample diameter:	max. 16 mm
Sample thickness	max. 5 mm

Materials:

Insulation:	Foamed alumina
Heater:	Kanthal APM (22 % Cr, 5.8 % Al, Fe)

CCU 1000 Dimensions:

Width x Depth x Height:	450 x 410 x 180 mm
Weight:	approx. 26 kg

CCU 1000 Electrical Data (For use with HTK 1200N):

Voltage (mains):	AC 230 V ± 10 %
Frequency (mains):	50 ... 60 Hz
Power consumption:	max. 1671 VA
Mains fuses:	2 x T 8 A H 5 x 20 mm (ceramic tube)
Overvoltage category:	II according to EN 61010-1
Voltage output:	AC 45 V
Current output:	max. AC 35 A
Thyristor fuse:	Semiconductor protection fuse FF 12.5 A 10 x 38 mm

Cooling water

Flow rate:	0.7 to 1.5 l/min
Temperature:	15 to 25 °C
Pressure:	Max. 4 bar

Ambient conditions

Ambient temperature:	15 - 35 °C
Ambient humidity:	80 % max., not condensing
Maximum operating altitude:	3000 m above sea level
Pollution degree	2 according to EN 61010-1

The instrument is designed for INDOOR USE ONLY!

The HTK 1200N with CCU 1000 Combined Control Unit is CE-compliant according to:

- **Electromagnetic compatibility (2014/30/EU, OJ L 96/79 of 29.3.2014)**

Applied standards:

EN 61326-1:2013 Electrical equipment for measurement, control and laboratory use - EMC requirements - Part 1: General requirements

The product is classified as a class B equipment and is not intended for the use in industrial area.

- **Low Voltage Directive (2014/35/EU, OJ L 96/357 of 29.3.2014):**

Applied standards:

EN 61010-1:2010 Safety requirements for electrical equipment for measurement, control and laboratory use - Part 1: General requirements

IEC 61010-2-010: 2014 Safety requirements for electrical equipment for measurement, control and laboratory use - Part 2: Particular requirements for laboratory equipment for the heating of materials

Appendix B: Cooling water

The cooling water is required to avoid heat transfer from the chamber to the goniometer.

TIP: *For more information on the installation of the water supply, refer to chapter 4.6*

The cooling water hoses can be connected directly to the mains water supply provided that the water quality requirements are fulfilled (see table B.1) and an appropriate filter is used.

If the cooling water does not fulfill the quality requirements in table B.1, a closed cooling unit must be used. This unit must contain water of the required quality.

However, in order to prevent the formation of deposits and corrosion in the unit, we recommend the following water quality instead of tap water:

- Use distilled water.
- Add 1,68 grams (20 mmol) of $NaHCO_3$ per liter of distilled water.
- Check the pH value; it must be between 8.0 and 8.3.
- If the pH value is too low, add Na_2CO_3 to the mixture until the pH is in the above mentioned range.
- If the pH value is too high, add $NaHCO_3$ to the mixture until the pH is in the above mentioned range.

NOTICE
Risk of damage
Do not use plain de-ionized or bidistilled water!

Table 11-1: Requirements on cooling water quality

Cooling water	Condition	Remarks
Water quality	Tap water	If suitable.
Saturation index (see below)	< 1	
Ryznar index (see below)	5.5 to 7.5	

Table 11-1: Requirements on cooling water quality

Cooling water	Condition	Remarks
Dissolved organic material	use of $KMnO_4$ < 20 mg/l	
Chloride	< 250 mg/l	
Flow rate	0.7 to 1.5 l/ min	
Temperature	15 to 25 °C	
Pressure	4 bar max.	
Water filter (supplied with the Anton Paar flow controller)	16 µm filtering	Part of the pressure reducer.

Saturation index: SI = pH - pH's

Ryznar Index: RI = 2 pH's - pH

pH: Is obtained and corrected by the addition of chemicals as stated before.

pH's: Is calculated according to

$$pH's = (9,3 + A + B) - (C + D)$$

with the following values:

A = Conversion value of total dissolved material in milligrams per liter (mg/l)

B = Conversion value of the temperature of the cooling water in degrees Celsius (°C)

C = Conversion value of the calcium hardness (NOT the total hardness), in German degrees (°D) (1 °D = 10 mg CaO per liter)

D = Conversion value of the bicarbonate content in chemical equivalent per liter (1 chemical equivalent/liter = 61 mg/l for bicarbonate)

The conversion values A, B, C and D can be determined from table B.2.

Table 1: Conversion values for the calculation of Saturation index and Ryznar index

Total dissolved material		**Temperature**		**Ca-hardness* Ca++**		**Bi-carbonate content HCO_3**	
mg/l	A	Deg. C	B	Deg. D	C	chem.	D eq/l
50-300	0.1	10	2.3	0.6	0.6	0.2	1.0
400-1000	0.2	15	2.2	1.4	1.0	0.5	1.4
		24	2.0	2.2	1.2	1.3	1.8
		35	1.8	3.5	1.4	2.0	2.0
		47	1.6	5.5	1.6	3.2	2.2
		60	1.4	7.0	1.7	4.1	2.3
		76	1.2	9.0	1.8	5.2	2.4
				11.5	1.9	6.4	2.5
				14.5	2.0	7.9	2.6
				18.0	2.1		
				22.2	2.2		
				1 D = 10 mg/l CaO		1 chem = 61 mg/l	

** This means Calcium hardness and not total hardness*

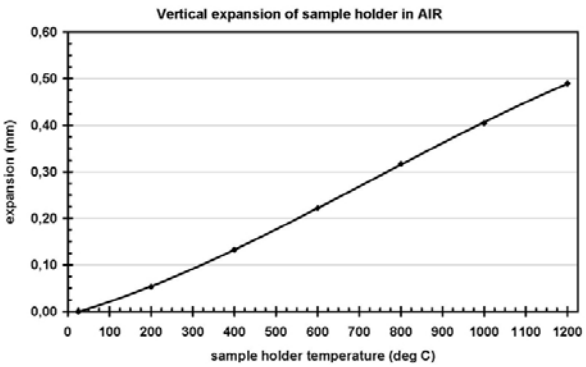
Appendix C: Thermal Expansion of the Sample Holder

Due to the thermal expansion of the used materials the height of the sample holder expands with increasing temperature which makes regular realignments during high- temperature XRD investigations necessary.

The tables and diagrams below show measurements of vertical expansion of the rigid sample holder in vacuum and air. The indicated values are comparable for the rigid sample holder and for the sample spinner and are supposed to give a rough idea about the magnitude of vertical expansion.

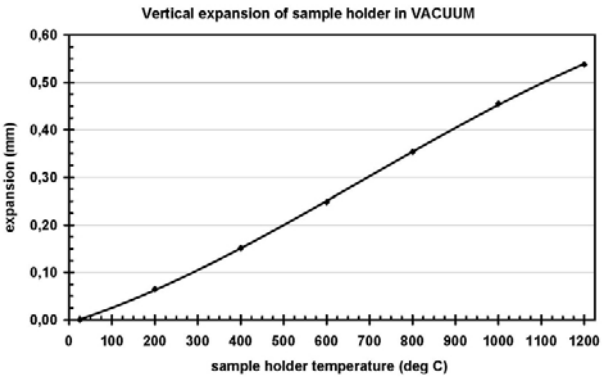
Vertical sample holder expansion **in air**:

Temperature [C°]	Vertical expansion [mm]
25	0,00
100	0,02
200	0,06
300	0,09
400	0,13
500	0,18
600	0,22
700	0,27
800	0,32
900	0,36
1000	0,41
1100	0,45
1200	0,49



Vertical sample holder expansion **in vacuum**:

Temperature [C°]	Vertical expansion [mm]
25	0,00
100	0,02
200	0,06
300	0,11
400	0,15
500	0,20
600	0,25
700	0,30
800	0,35
900	0,40
1000	0,45
1100	0,50
1200	0,54



Appendix D: Warranty

The warranty regulations for the HTK 1200N High-Temperature Oven-Chamber are in accordance with "General Terms of Delivery" of the Austrian Electrical and Electronic Industry.

Anton Paar GmbH gives no warranty for:

- ceramic parts.

Appendix E: Accessories

Temperature Controller	
135004	CCU 1000 Combined Control Unit
164877	Flow Control Unit HTK 1200N
Option	
27406	HTK 1200N CAPILLARY EXTENSION
Accessories and Spare Parts	
58963	HIGH VACUUM EQUIPMENT
176757	HIGH VACUUM EQUIPMENT (100V)
15913	HTK 1200N SAMPLE CARRIER 0.8MM DEEP
16411	HTK 1200N SAMPLE CARRIER 0.4MM DEEP
16412	HTK 1200N SAMPLE CARRIER FLAT
15914	FIXING RING FOR SAMPLE CARRIER HTK 1200N
75850	SET OF WINDOW FOILS (AL-COATED KAPTON)
2600	SET OF GRAPHITE FOILS
2601	SET OF KAPTON FOILS
75851	SET OF O-RINGS
6650	SPARE PARTS PACKAGE
182226	CABLE FOR SAMPLE HOLDER FLANGE

Appendix F: Swagelok tube fittings

Appendix F.1: Installation instructions

Swagelok tube fittings come to you completely assembled, finger-tight and are ready for immediate use. Disassembly before use is unnecessary and can result in dirt or foreign material getting into fitting and causing leaks.

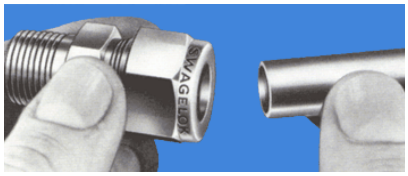
NOTICE

Risk of damage

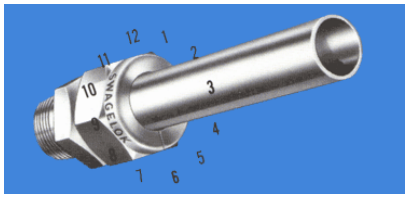
In case of unwanted disassembling, assemble the fitting as shown in the following picture!.

Swagelok tube fittings are installed in three easy steps

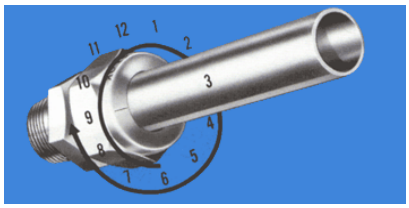
1. Simply insert the tubing into the Swagelok tube fitting. Make sure that the tubing rests firmly on the shoulder of the fitting and that the nut is finger-tight.



2. Before tightening the Swagelok nut, scribe the nut at the 6 o'clock position.



3. Hold the fitting body steady with a backup wrench and tighten the nut 1 1/4 turns. Watch the scribe mark, make one complete revolution and continue to the 9 o'clock position. By scribing the nut at the 6 o'clock position as it appears to you, there will be no doubt as to the starting position. When the nut is tightened 1 1/4 turns to the 9 o'clock position, you can easily see that the fitting has been properly tightened. Use of the gap inspection gage (1 1/4 turns from finger-tight) ensures sufficient pull-up.



NOTICE

Risk of damage

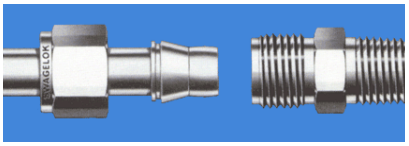
For 1/16, 1/8, 3/16 inch, 2, 3, and 4 mm size tube fittings proceed as described above, but use a 3/4 turn instead of the 1 1/4 turn.

Appendix F.2: Retightening instructions

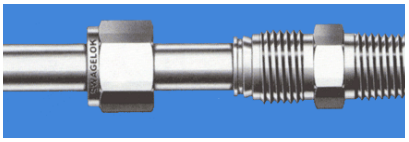
Connections can be disconnected and retightened many times.

The same reliable leak-proof seal can be obtained every time the connection is remade.

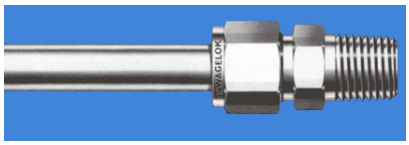
1. Fitting shown in the disconnected position.



2. Insert tubing with pre-swaged ferrules into the fitting body until the front ferrule sits.



3. Tighten nut by hand. Rotate nut to the original position with a wrench. An increase in resistance will be encountered at the original position. Then tighten slightly with the wrench. Smaller tube sizes will take less tightening to reach the original position, while larger tube sizes will require more tightening. The wall thickness will also have an effect on tightening



Appendix G: Declaration of Conformity

EU Declaration of Conformity

(original)

Anton Paar

The Manufacturer **Anton Paar GmbH**, Anton-Paar-Str. 20, A-8054 Graz, Austria – Europe hereby declares that the product listed below

Product designation:	**HTK 1200N OVEN-CHAMBER WITH RIGID SAMPLE HOLDER**
	HTK 1200N OVEN-CHAMBER WITH SAMPLE SPINNER
	CCU 1000 COMBINED CONTROL UNIT
Model:	**HTK 1200N, CCU 1000**
Material number:	16407, 16408, 135004

is in conformity with the relevant European Union harmonisation legislation. This declaration of conformity is issued under the sole responsibility of the manufacturer.

Electromagnetic Compatibility (2014/30/EU, OJ L 96/79 of 29.3.2014)

Applied standards:

- EN 61326-1:2013 — Electrical equipment for measurement, control and laboratory use - EMC requirements - Part 1: General requirements

The product is classified as a class B equipment and is not intended for the use in industrial area.

Low Voltage Directive (2014/35/EU, OJ L 96/357 of 29.3.2014)

Applied standards:

- EN 61010-1:2010 — Safety requirements for electrical equipment for measurement, control and laboratory use - Part 1: General requirements
- EN 61010-2-010:2014 — Safety requirements for electrical equipment for measurement, control and laboratory use - Part 2-010: Particular requirements for laboratory equipment for the heating of Materials

RoHS Directive (2011/65/EU, OJ L 174/88 of 1.7.2011)

Place and date of issue: Graz, 2018-02-28

ppa P. Kettisch

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Head of Material Characterization
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