Anton Paar

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

Galileo Galilei (1564-1642)

Reference Guide

DHS 1100

Domed Hot Stage with

CCU 100 Control Unit

Disclaimer

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Reference Guide

DHS 1100

Domed Hot Stage with

CCU 100 Control Unit

Contents

1 Safety Instructions

- Read the documentation before using DHS 1100.
- Follow all information and instructions contained in the documentation to ensure the correct use and safe functioning of DHS 1100.
- 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.
- Anton Paar GmbH only warrants the proper functioning of DHS 1100 if no adjustments have been made to the mechanics, electronics, and firmware.
- Only use DHS 1100 for the purpose described in the documentation. Anton Paar GmbH is not liable for damages caused by incorrect use of DHS 1100.

Installation and use

- DHS 1100 is **not** an explosion-proof instrument and therefore must not be operated in areas with risk of explosion.
- The installation procedure should only be carried out by authorized personnel who are familiar with the installation instructions.
- Do not use any accessories or spare 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 DHS 1100. Do not operate the instrument under conditions which could result in damage to goods and/or injuries and loss of life.
- Check DHS 1100 for chemical resistance to the samples and cleaning agents.

Maintenance and service

- The results delivered by DHS 1100 not only depend on the correct functioning of the instrument, but also on various other factors. We therefore recommend you have the results checked (e.g. plausibility tested) by skilled personnel before consequential actions are taken based on the results.
- Service and repair procedures may only be carried out by authorized personnel or by Anton Paar GmbH.

Disposal

- Concerning the disposal of DHS 1100, observe the legal requirements in your country.

Returns

- For repairs send the cleaned DHS 1100 (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 the DHS 1100.
- 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 DHS 1100 must be kept free of inflammable gases and vapors.
- Supply a fire extinguisher.
- Ensure the sufficient supervision of DHS 1100 during operation.

Radiation Safety

- The DHS 1100 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 protection equipment corresponds to the local requirements.
- Do not operate DHS 1100 with explosive or poisonous gases. Do not measure samples that evaporate explosive or poisonous gases.

1.2 Conventions for safety messages

The following conventions for safety messages are used in this instruction manual:

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

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.

WARNING

Electrostatic sensitive device
Warning indicates a situation which, if not avoided, could result in damage to property.

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

1.3 Safety Signs on the Instrument

1 CAUTION - Hot Surface

Fig. 1: Position of warning signs on the instrument

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.

NOTICE
Take care that the warning symbols remain clearly legible.

2 Overview

2.1 Description of the Instrument

The DHS 1100 Domed Hot Stage is a heating attachment for four-circle X-ray diffractometers. It is designed for X-ray diffraction experiments in reflection geometry with sample temperatures from 25 °C to 1100 °C.

The unique design of the instrument provides:

- Sample temperatures from 25 °C to 1100 °C
- High temperature uniformity across the sample
- Easy sample mounting
- Compact design, low weight and high flexibility
- Versatile applications

The special, dome-shaped X-ray window of the DHS 1100 sample stage combines two important properties: gas-tight enclosure of the sample and X-ray transparency over the complete half space above the sample. Because of the gas tightness, samples can be investigated in vacuum or inert gas to avoid oxidation or other chemical reactions of the sample at high temperatures.

Due to the design of dome, heater and sample fixing, very low angles of incidence can be reached and the following types of X-ray measurements can be carried out:

- pole figures
- grazing incidence diffraction
- grazing incidence in-plane diffraction

Cooling of the dome and the housing of the DHS 1100 attachment is achieved by using compressed-air. The cooling air supply is controlled with the CCU 100 Combined Control Unit. The CCU 100 monitors if sufficient cooling air is available and automatically turns on the cooling air if the heater temperature exceeds 200 °C.

Below 200 °C heater temperature, the instrument can be run without dome and without cooling air.

The main applications for the DHS 1100 are to investigate the temperature dependence of

- crystalline textures
- crystal stress and strain
- temperature-induced phase transitions
- structural properties of thin films

To ensure reliable operation of the DHS 1100, the instrument must be operated with:

- 135000 CCU 100 Combined Control Unit

In order to avoid contamination of the DHS 1100 by the cooling air, Anton Paar GmbH provides an **air service unit** with pressure regulator and air filters as an accessory:

- 6931 AIR SERVICE UNIT

If vacuum operation is required, Anton Paar GmbH offers:

- 58974 Standard Vacuum Equipment

If a PEEK dome is required instead of the standard graphite dome, an additional cooling nozzle is necessary as the PEEK dome has to be cooled also on the top side of the dome. Two cooling nozzles are optionally available as an accessory:

- 135038 DOME COOLING NOZZLE DHS 1100 (600 °C)
- 159567 HT DOME COOLING NOZZLE DHS 1100 (900°C)

TIP: *Refer to appendix E for detailed information.*

2.2 Design of the Instrument

The DHS 1100 consists of:

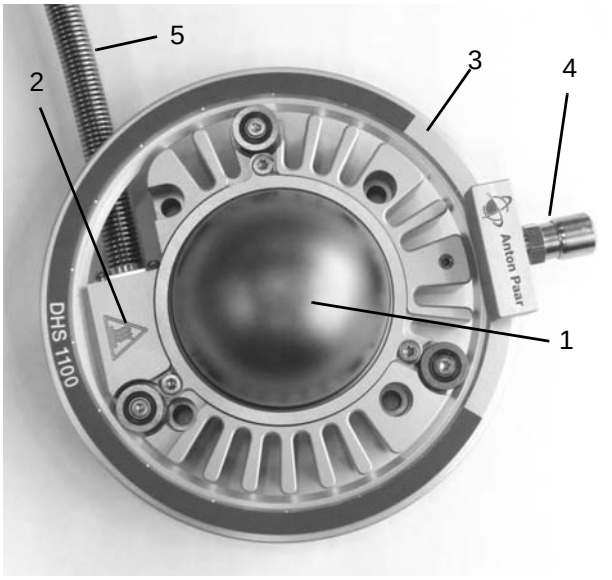


Fig. 2-1: DHS 1100

1 Dome
2 Housing
3 Turnable cooling ring with outlets for cooling air
4 Quick coupling connection for cooling air supply
5 Supply hose for gas/vacuum and temperature control

NOTICE
Risk of damage
Do not disconnect the supply hose from the DHS 1100 housing, because the connecting wires from the thermocouple and the heater can be damaged.

The DHS 1100 has an air cooling system, which has the advantages that only one hose is needed for the cooling agent and that no liquid can leak inside the cabinet in case of a defect.
The cooling gas, preferably clean compressed-air, is distributed around the chamber inside the turnable cooling ring (3). Small outlets in the ring direct the gas onto the housing (2) and the dome (1). The cooling ring is turnable so that the connector for the cooling air hose (4) can be placed in a position where it does not interfere with the incident and diffracted X-ray beams.
The cooling air supply is controlled with the CCU 100 Combined Control Unit, which automatically turns on the cooling air when the heater temperature reaches 200 °C.

In order to achieve the maximum movability of the DHS 1100 on the goniometer, the number of hoses to the instrument is kept to a minimum. Besides the hose for the cooling gas there is only one additional hose (5) which contains the electrical wires for thermocouple and heater and which is used to evacuate the dome or to fill it with gas.

2.2.1 Dome

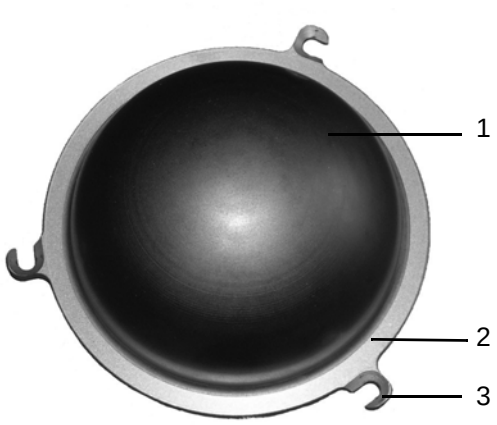


Fig. 2: Graphite Dome

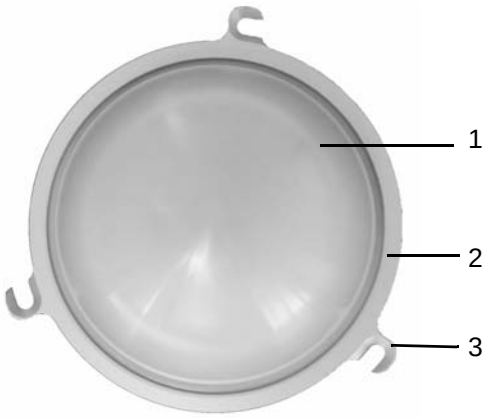


Fig. 3: PEEK Dome

1 Dome
2 Fixing ring
3 Clasps

The graphite dome is made of graphite, which exhibits an excellent combination of good mechanical properties, high temperature resistance and excellent X-ray transmission.

The PEEK dome is made of PEEK, a synthetic material based on polyetheretherketone resin. This advanced plastic exhibits a unique combination of good mechanical properties, high temperature resistance and excellent chemical resistance. The glass transition temperature of PEEK is in the range of 140 °C.

The dome is fixed to the housing of the DHS 1100 by a fixing ring made of aluminium with three clasps.

Vacuum tightness is guaranteed by an O-ring in the housing.

CAUTION

- Always lift the dome by the fixing ring. Do not touch the graphite dome.
- Make sure that the air cooling is on to guarantee sufficient cooling of the DHS 1100 housing and the dome during operation at temperatures higher than 200 °C.
- Never touch the dome during operation, especially while it is evacuated or at elevated temperatures!
- If the inner surface of the dome has become coated with evaporated sample components, clean it with a dry cloth.
- Always use a dome cooling nozzle for operation with PEEK dome.

TIP: *For a diffractogram of graphite and PEEK, please refer to Appendix C: Diffraction Patterns.*

The dome is also available as spare part, refer to chapter Appendix E: Parts List.

Anton Paar GmbH gives no warranty for the dome if it is not handled according to our instructions.

2.2.2 Dome Cooling Nozzle for PEEK Dome

2.2.2.1 Dome Cooling Nozzle for T_{max} 600 °C

The Dome Cooling Nozzle is for operating the DHS 1100 with PEEK dome up to a temperature limit of max. 600 °C.

In order to use the cooling nozzle (1), a special cooling-air connector (2) is required. Since the cooling-air connector and the cooling-air ring are one inseparable unit in the standard configuration, the complete cooling-air ring must be exchanged. The ring with connector is part of the accessory.

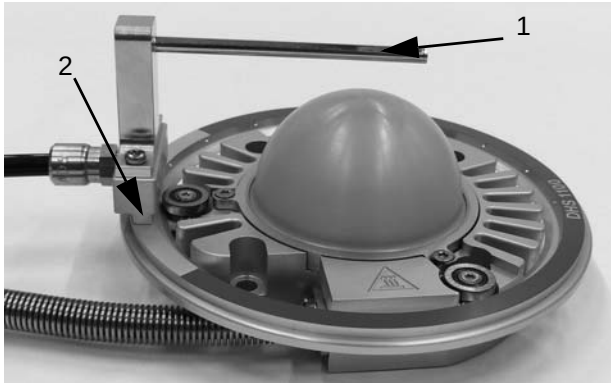
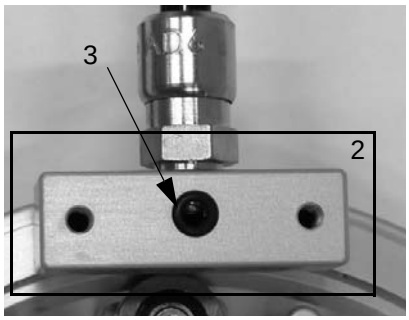


Fig. 4: DHS 1100 with cooling nozzle



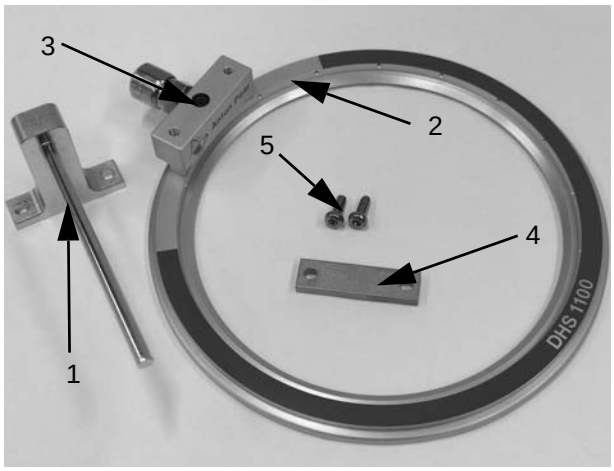


Fig. 5: Main parts included in DHS 1100 accessory Cat.No. 135038

1 Cooling nozzle
2 Cooling-air ring with special connector block with gas outlet
3 O-ring 3x1 mm
4 Blind cover
5 Screws M3x8 mm

For operation with a graphite dome and without cooling nozzle, a blind cover must be mounted on the connector (2) instead of the cooling nozzle (see Fig. 6).

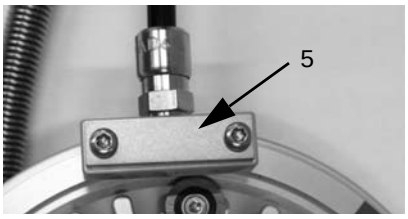


Fig. 6: DHS 1100 with blind cover

2.2.2.2 HT Dome Cooling Nozzle for T_{max} 900 °C

The HT Dome Cooling Nozzle is for operating the DHS 1100 with PEEK dome up to a temperature limit of max. 900 °C.

The nozzle of the HT Dome Cooling Nozzle is designed larger in size and is equipped with additional boreholes for the effusing cooling air.

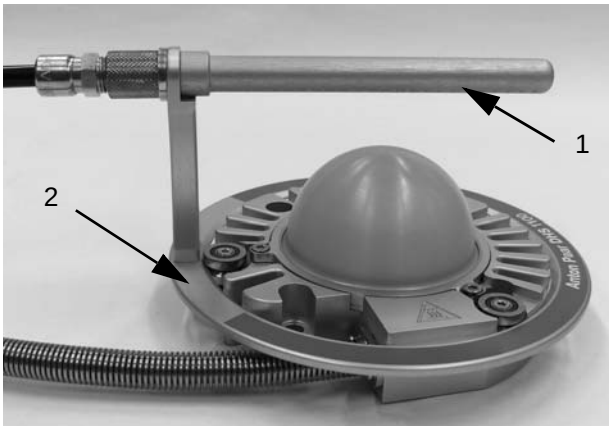


Fig. 7: Main parts included in DHS 1100 accessory Cat.No. 159567

1 Cooling nozzle
2 Cooling-air ring with special connector block with gas outlet

These parts cannot be separated.

2.2.3 Housing and Internal Parts

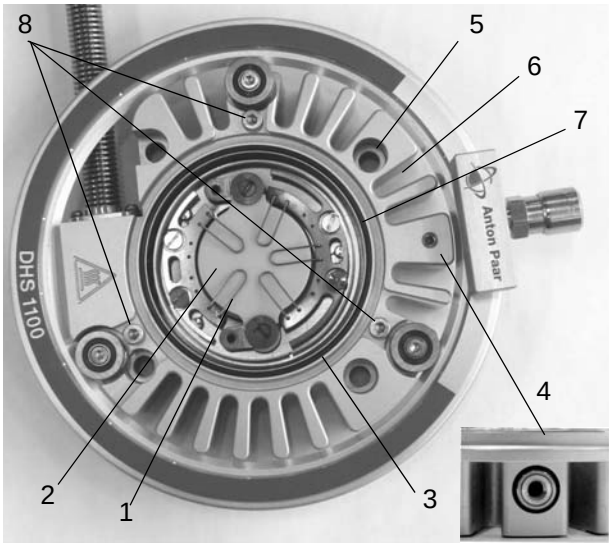


Fig. 8: Main parts of DHS 1100

1 Springs for fixing the sample
2 Sample plate
3 Temperature shieldings
4 Pressure relief valve
5 Bore holes for mounting the DHS 1100
6 Cooling fins
7 O-ring for the dome
8 Fixing screws for the dome

The housing of the DHS 1100 is made of anodized aluminium and is therefore very light. It contains cooling fins which provide sufficient heat transport from the heating plate of the DHS 1100 to the surrounding atmosphere.

The heating plate of the DHS 1100 is made of aluminium nitride (AlN), a ceramics with excellent temperature conductivity and good chemical resistance. In case of damage, the sample plate can be easily replaced by a new one.

The springs for fixing the sample are made of Inconel and can be loosened very easily by using tweezers.

TIP: *For a diffractogram of aluminium nitride and Inconel, please refer to Appendix C: Diffraction Patterns*

The heater is located just underneath the AlN heating plate. The design and the excellent temperature conductivity of the AlN plate guarantee high temperature uniformity across the heating plate. The thermocouple is located below the middle of the heating plate.

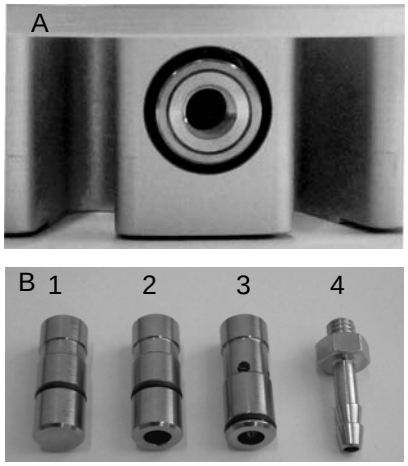
The connecting wires from the thermocouple and the heating filament run within the black hose (supply hose) to the connection device described in *chapter 2.2.4 Connection Device*.

NOTICE

Risk of damage

Do not disconnect the supply hose from the DHS 1100 housing because the connecting wires from the thermocouple and the heating filament can be damaged.

The housing of the DHS 1100 contains a bore hole (A) for various connectors (B). The following connectors are supplied with DHS 1100:



1 Blind plug
2 Feed-through adapter
3 Pressure relief valve
4 Hose connector

A blind plug (B.1) is provided for measurements under vacuum.

The feed-through adapter (B.2) can be used together with the hose connector (B.4) for additional gas discharge.

The pressure relief valve (B.3) is a safety device that opens if an overpressure of 0.35± 0.05 bar relative pressure is generated in the dome.

TIP: *Additional information for replacing the connectors can be found in chapter 7.5.2.3: Operation under Vacuum.*

2.2.4 Connection Device

The connection device terminates the supply hose of the DHS 1100, which contains the electrical wires of the instrument and which is used to evacuate the instrument or to fill it with gas. It is a vacuum-tight interface box with connectors for the temperature sensor and the heater inside the DHS 1100 and a flange to connect a vacuum pump or the gas supply.

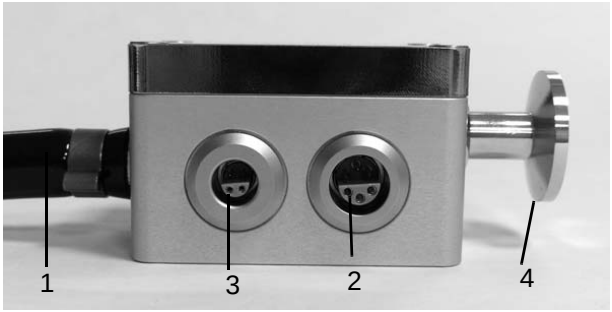


Fig. 9: Connection device

1 Supply hose with wires to thermocouple and heater
2 Connector for thermocouple (6 poles)
3 Connector for heating (4 poles)
4 Flange for gas supply / vacuum pump (DN16KF)

CAUTION

Place the connection device for gas/vacuum and temperature control in a way that the hose is not caught by any parts of the goniometer.

2.3 CCU 100 Combined Control Unit

CCU 100 is designed to control the sample plate temperature in DHS 1100 and to guarantee safe operation of the entire instrument. In order to reach and maintain the desired sample temperature, CCU 100 controls the resistance heater inside the sample chamber and the compressed-air cooling.

CCU 100 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.

The main parts of CCU 100 are the main board, the micro controller for the temperature control and the power module. The micro controller controls the sample plate temperature based on the measured temperature and the user defined target values. The power module provides variable electrical power for the heater inside DHS 1100, depending on the control signal from the micro controller.

2.3.1 CCU 100 Front Panel

The front panel of CCU 100 contains all control buttons, status LEDs and the display.

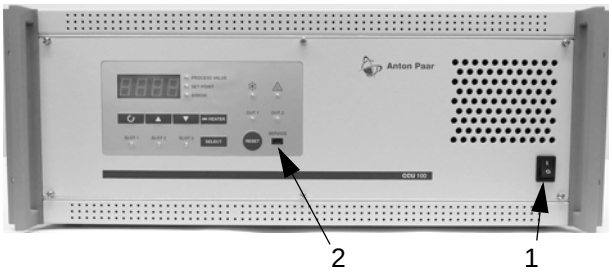


Fig. 10: CCU 100 front panel with control elements

1 Mains switch
2 Display and keypad

All elements of the front panel are described in detail in *Chapter 6.2 Keypad*

2.3.2 CCU 100 Rear Panel

The rear panel of CCU 100 contains all the connectors of the instrument.

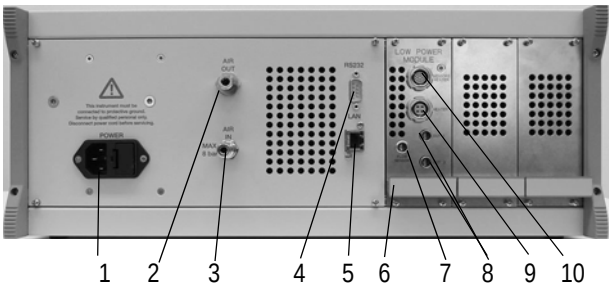


Fig. 11: CCU 100 rear panel

1 Power connector
2 Connector air out
3 Connector air in
4 RS 232 connector
5 LAN connector (currently not used)
6 Low power module for DHS 1100
7 Connector Flow Sensor (not used for DHS 1100)
8 Connector OUT1/2 (optional connectors)
9 Connector Heater
10 Connector Sensors/Heater

2.3.3 Control of the Compressed-Air Cooling

The CCU 100 controls the compressed-air cooling equipment.

In order to be able to cool the dome and housing, the compressed-air source has to be connected to the connector AIR IN on the rear panel of CCU 100. If cooling is required (depending on the configuration this could be dSP + 1 °C or < 200 °C, see Chapter 6), a valve inside CCU 100 connects the AIR IN to the AIR OUT connector, which is connected to the quick coupling connector for cooling air supply at the DHS 1100 (Fig. 2-1,4).

In case that no cooling is required, the valve between AIR IN and AIR OUT closes.

2.3.4 Overheat Protection

DHS 1100 has a protective thermoswitch, which is mounted inside the housing, to prevent overheating of the instrument in case the air cooling system fails. The thermoswitch interrupts the heating power circuit if the temperature of the chamber housing exceeds 115 °C.

CCU 100 automatically switches to Standby mode (heater off) and shows an alarm (E04).

CAUTION

Hot surface
When the compressed-air supply to the device fails, the housing can have a **hot surface**.
Carefully check the temperature of the DHS 1100 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 DHS 1100 and CCU 100.

TIP: *A list of all error messages can be found in Chapter 9.1 Error Messages.*

2.4 Compressed-Air Cooling Equipment

In order to meet the requirements for the compressed air regarding pressure and purity, Anton Paar GmbH recommends to use the following pressure regulator with filters:

- Air Service Unit (cat.no 6931)

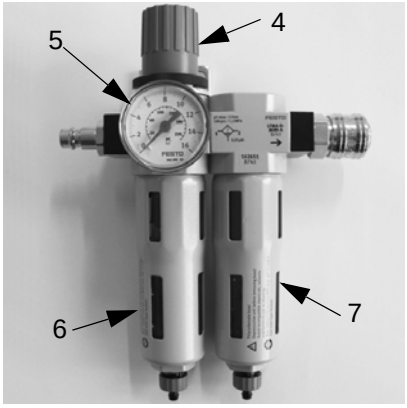


Fig. 12: Air service unit

1 air pressure regulator
2 air pressure gauge
3 coarse filter cartridge
4 fine filter cartridge

For further details see *Chapter 6.5 Controlling the Compressed-Air Cooling*

3 Checking the Supplied Parts

The DHS 1100 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 and further questions from the transport and insurance company.
2. Check the delivery for completion by comparing the supplied parts to those listed in Table .
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 1: Supplied Parts

Pcs.	Article Description	
1	DHS 1100 Domed Hot Stage	25012
1	CCU 100 Combined Control Unit*	135000
and/or 1	Low-Power Module CCU	176732
1	Accessory box (content specified on label)	---
1	Instruction Manual	---
1	Software Manual Nambicon	---
1	USB flash drive with Nambicon	---

** The CCU 100 is able to control more than one chamber. In case of ordering the chamber and controller, the CCU 100 together with the preinstalled Low-Power Module is included; in case of adding a second chamber to an existing CCU 100, only the Low-Power Module is included.*

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!

4 Installation

4.1 Installation Requirements

4.1.1 X-Ray Diffractometer Requirements

CAUTION

Radiation
The DHS 1100 represents an open system and therefore is only allowed to be operated on goniometers equipped with a radiation enclosure

4.1.2 Mounting Adapter

On certain goniometer supports an adapter is necessary to mount DHS 1100 to the goniometer.

4.1.3 Electrical Requirements CCU 100

- Mains voltage: AC 100 - 240 V
- Mains frequency: 50 or 60 Hz.
- mains connector (1): IEC 60320 C13
- mains fuses: 2 x T 6.3 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

4.1.4 Compressed-Air Cooling

For operation of DHS 1100 with compressed-air cooling, the main requirements for the compressed-air supply are:

- Cooling air pressure: min. 2 bar rel. / max. 4 bar rel. (rel. = relative to atmospheric pressure).
- Air flow rate at 2 bar: 0.12 m³/min
- The air must be free of oil and dust

NOTICE
We recommend to use the Air Service Unit by Anton Paar GmbH (refer to *Appendix E: Parts List*).

4.1.5 Vacuum Equipment Requirements (Optional)

Vacuum equipment requirements:

- Pumping speed acc. DIN 28400: ≥ 4.2 m^3/h
- Ultimate partial pressure acc. DIN 28400: ≤ 1E-4 mbar
- Ultimate total pressure acc. DIN 28400: ≤ 2E-3 mbar
- Vacuum connector on sample chamber: DN16 ISO-KF

4.2 Unpacking the Instrument

NOTICE
Risk of damage

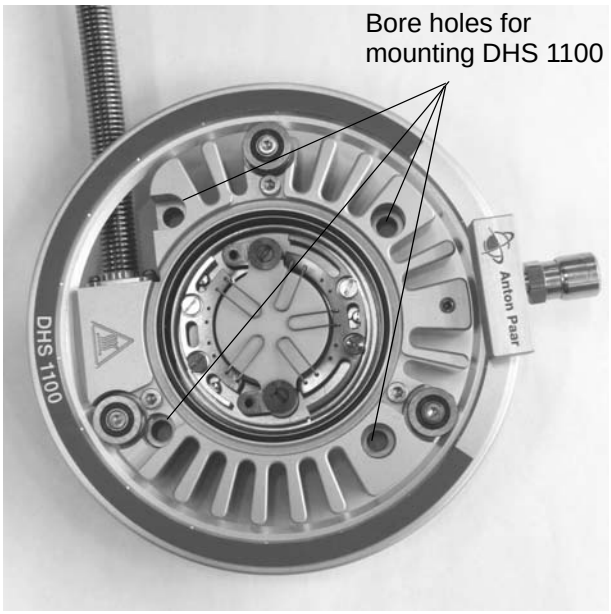
- Unpack the system with care.
- Do not drop the instrument.
- The graphite dome is a fragile item, handle it with particular care.
- Always lift the dome by the fixing ring, do not touch the graphite dome.

TIP: *Anton Paar GmbH gives no warranty for the dome if it is not handled according to our instructions.*

If the dome is already broken when unpacking DHS 1100, please take pictures that show explicit the broken dome with the original packing material. Otherwise no warranty can be accepted by Anton Paar.

4.3 Mounting the DHS 1100 on the Goniometer

The DHS 1100 is mounted to the goniometer via the four bore holes, as shown below.



For detailed information on the installation refer to the *Instruction Manual* of your *Diffractometer*.

4.4 Installing the Compressed Air Cooling

Cooling of the dome and the housing of the DHS 1100 heating attachment is achieved by using clean compressed-air. If no compressed-air supply is available in the laboratory, a small compressor can be used to generate the cooling air.

NOTICE
A special Air Service Unit can be used to clean the cooling gas from dust and oil (see also *Appendix E: Parts List*).

CAUTION

Do not connect the DHS 1100 directly to the cooling air supply.
The cooling air must be supplied through the CCU 100 Combined Control Unit.

A black hose for the cooling air is supplied with the DHS 1100. Install this supply hose as follows:

1. Connect the end of the hose inside the cabinet to the appropriate quick coupling connector (1) on the turnable ring of the DHS 1100.

2. Feed the air hose through the diffractometer enclosure and connect it to the AIR OUTLET connector on the rear panel of CCU 100.

Install the compressed-air cooling device on the sample chamber as follows:

NOTICE
When you dismount the compressed-air cooling unit, push it upwards towards the DHS 1100 before you turn it to avoid damage to the locking slit.

CAUTION

Risk of damage
Make sure that the compressed-air cooling unit is securely locked in the connector before you start to use the instrument. Gently try to pull it downwards. It must be fixed on the connector.

Connect the compressed-air supply to CCU 100:

1. Connect the female connector of the pneumatic hose supplied with CCU 100 to the AIR INLET connector on the rear panel of CCU 100.

2. Connect the *Air Service Unit* to the compressed-air supply of the laboratory.
3. Connect the male connector of the pneumatic hose to the outlet of the *air service unit*.

CAUTION

Do not apply more than 4 bar rel. pressure to the compressed-air connector of CCU 100. Exceeding the pressure limit may cause hoses to burst and can damage the instrument.
If necessary, use an appropriate pressure regulator between the compressed-air source and CCU 100.

4. Set the pressure on the *Air Service Unit* to 2 bar.

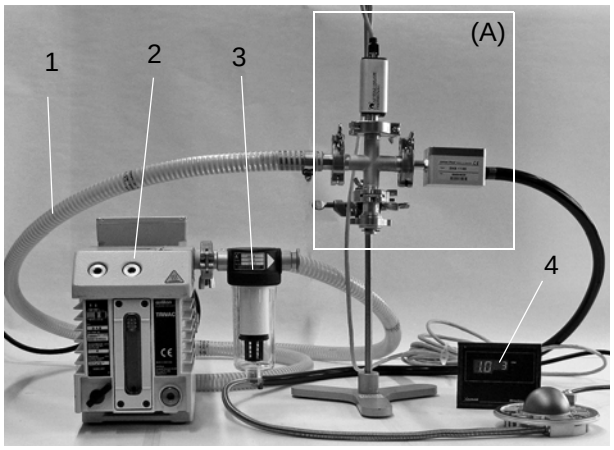
TIP: *2 bar is the standard value. The pressure can be varied between 2 to 4 bar. Higher pressure provides more cooling power, lower pressure less air consumption.*

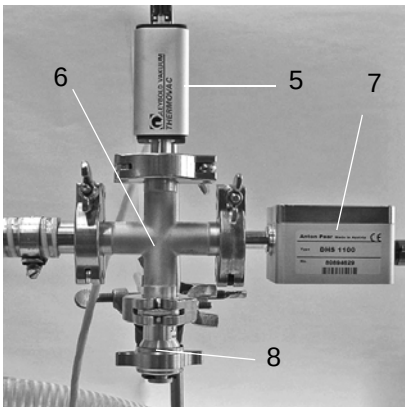
5. After you have put CCU 100 in operation, change the CCU 100 configuration to *Cooling Type* "cOn" or "cOFF" as described in *Chapter 6.3.1 Navigation Diagram of CCU 100*.

4.5 Connecting the Gas Supply/Vacuum Equipment

The graphite dome allows the investigation not only in air, but also under vacuum or in inert gas to avoid oxidation or other chemical reactions of the sample at high temperatures.

Feed an appropriate gas/vacuum hose through the cabinet of the goniometer and connect it to the flange (DN16KF) on the connection device of the DHS 1100.





1 Vacuum hose
2 Rorary vane pump (RVP)
3 Oil mist filter
4 Display for vacuum gauge
5 Vacuum gauge
6 4-way-cross DN16KF
7 Connection device DN16KF
8 Vent valve DN10KF

The connection device for the gas supply/vacuum equipment is described in *chapter 2.2.4 Connection Device*.

TIP: *For further information about operation of the DHS 1100 under vacuum or various gases, please refer to chapter 7.5.2 Creating a Sample Environment.*

4.6 Installing the CCU 100 Combined Control Unit

CCU 100 Combined Control Unit is prepared for rack mounting in a 19" 4HE slot.

Mount CCU 100 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 100 for all connectors and that the slot is sufficiently ventilated to avoid heat accumulation behind the CCU 100.

CAUTION

Risk of damage
Always make sure that CCU 100 is turned off before you connect or disconnect cables on the connection device or the CCU 100.

The connector for temperature measurement and the connector for heating are located in the connection device for gas/vacuum and temperature control, described in chapter 2.2.4 Connection Device.

The instrument is delivered together with two cables for the connection of DHS 1100 to CCU 100. At one end of the cables a cable fixation (1) is mounted. This side is connected to CCU 100.

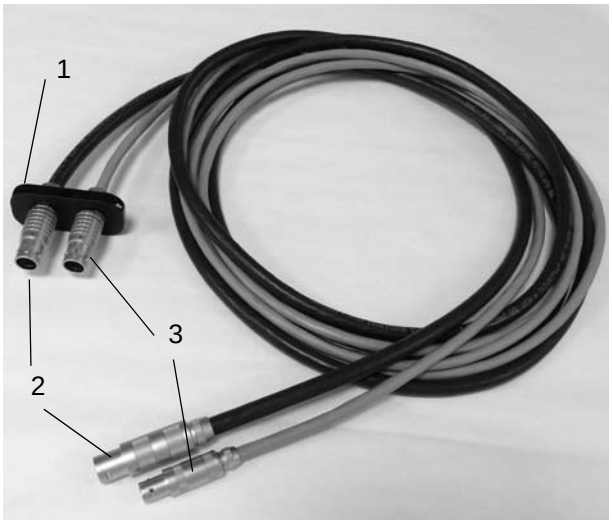


Fig. 13: Connection cables

1 Cable fixation
2 Temperature sensor cable
3 Heater cable

Connect the electrical cables to the connection device as follows:

1. Feed the cables through the appropriate port of the diffractometer cabinet as described in the diffractometer instruction manual.
2. Connect the heater cable to the connection device of DHS 1100.
3. Connect the temperature sensor cable to the connection device of DHS 1100. The temperature sensor cable contains the wires for the type S thermocouple, measuring the sample temperature inside the DHS 1100, and the wires for the thermoswitch which protects the DHS 1100 housing from being overheated.

Connect the electrical cables to the CCU 100 rear panel as follows:

1. Connect the side with the cable holder to the connector sensors/heater on the rear panel of CCU 100 (see Fig. 11).
2. Make sure the mains switch on CCU 100 is OFF and connect the mains power cable to the POWER connector.

WARNING

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

4.7 Connecting CCU 100 with a Computer

The RS 232 serial port at the rear panel of CCU 100 allows you to connect the CCU 100 to a computer for remote control (see Fig. 11(4)).

A null modem RS 232 cable is required to connect the CCU 100 to a PC.

PIN assignment:

Pin	
2	RX
3	TX
5	GND
Others	not connected

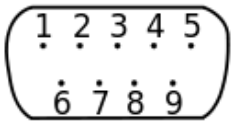


Fig. 14: RS 232 pins

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

4.8 Installing the Cooling Nozzle

4.8.1 Specification for Operation with PEEK Dome

The following specification applies for operation of DHS 1100 with a PEEK dome:

	Dome Cooling Nozzle	HT Dome Cooing Nozzle
Permitted atmospheres:	air, nitrogen, inert gases, vacuum	air, nitrogen, inert gases, vacuum
Temperature range:	**25 to 600 °C** with air, nitrogen, vacuum **25 to 400 °C** with helium	**25 to 900 °C** with air, nitrogen, vacuum **25 to 750 °C** with helium
Heating rate:	max. 300 °C/min	max. 300 °C/min
Cooling air pressure:	**min. 2.5** / max. 4 bar rel.	**min. 2** / max. 4 bar rel.
All other specifications are the same as for DHS 1100 with graphite dome.		

CAUTION

- For operation of DHS 1100 with PEEK dome, the cooling nozzle must be mounted properly.
- If possible, limit the range for the temperature set-point to the specified values in the software that controls CCU 100.
- The range of the set-point SP in CCU 100 cannot be changed (Graphite Dome 25 - 1100 °C; PEEK dome 25 - 900 °C).

NOTICE

Anton Paar GmbH accepts no warranty claims for PEEK domes which have been overheated on a DHS 1100.

4.8.2 Installing of the Dome Cooling Nozzle for T_{max} 600 °C

Proceed as follows to **mount** the cooling nozzle:

1. Turn off the heater on CCU 100 and wait until the temperature of the sample plate is close to 25 °C.
2. Turn off the cooling air supply.
3. Disconnect the cooling air hose from the quick coupling connector on the turnable cooling ring of the DHS 1100

4. Remove the dome from DHS 1100.

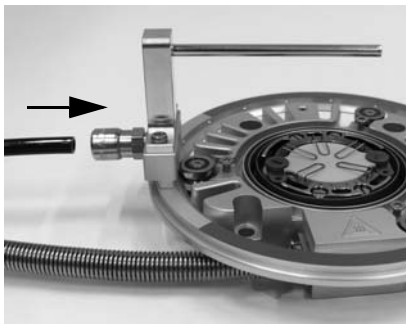
5. Dismount the bearing next to the connection of the supply hose shown with a Torx T7.
   To facilitate pulling out the bearing, gently tilt the ring and/or the bearing.

6. Lift the cooling ring and slightly shift it to be able to pull out the ring from the other two bearings.

7. Thread the ring of the Dome Cooling Nozzle into these two bearings and gently place the cooling ring in its position.

8. Remount the bearing.

9. Connect the cooling air hose to the quick coupling connector on the turnable cooling ring of the DHS 1100.

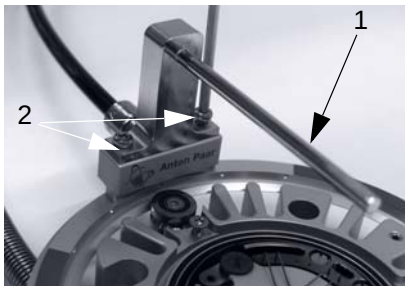


10. Mount the PEEK dome.

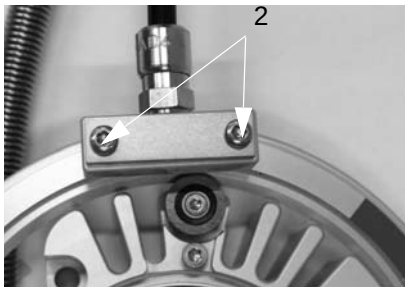
11. Turn on the air-cooling and make sure there is no leak between the cooling nozzle and the cooling-air connector.
12. If the nozzle is mounted tightly, resume operation.

Proceed as follows to **dismount** the cooling nozzle and mount the blind cover:

1. Turn off the heater on CCU 100 and wait until the temperature of the sample plate is close to 25 °C.
2. Turn off the cooling air supply.
3. Remove the dome from DHS 1100.
4. Loosen the two scews (2) and remove the cooling nozzle (1) from the cooling-air connector.



5. Make sure that the O-ring is clean and lying properly in the O-ring groove.
6. Mount the blind cover with the two screws M3 x 8 (2) supplied with the blind cover.



7. Turn on the air-cooling and make sure there is no leak between the blind cover and the cooling-air connector.
8. If the cover is mounted tightly, resume operation.

4.8.3 Installing of the HT Dome Cooling Nozzle for T_{max} 900 °C

1. Turn off the heater of the CCU 100 and wait until the temperature of the heating plate is nearly 25 °C
2. Turn off the cooling air supply
3. Disconnect the cooling air hose from the quick coupling connector on the turnable cooling ring of the DHS 1100.

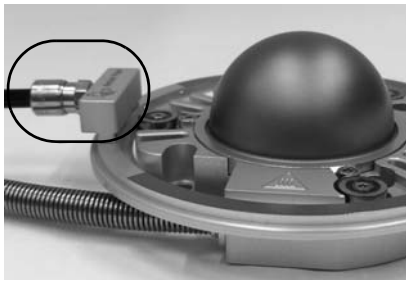
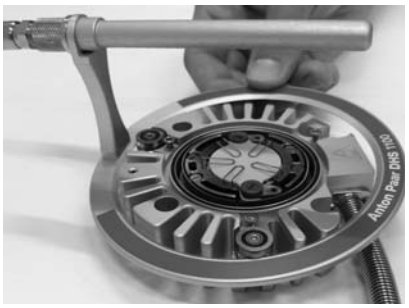


Fig. 15: Disconnection of the cooling air hose

4. Dismount the dome
5. Dismount the bearing next to the connection of the supply hose shown with a Torx T7.
   To facilitate pulling out the bearing, gently tilt the ring and/or the bearing.

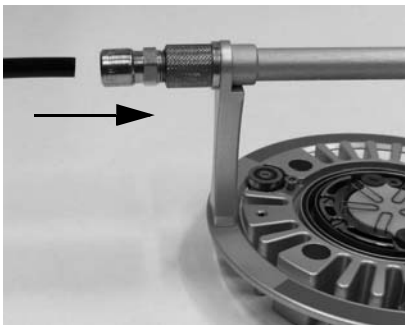
6. Lift the cooling ring and slightly shift it to be able to pull out the ring from the other two bearings.

7. Thread the ring of the HT Dome Cooling Nozzle into these two bearings and gently place the cooling ring in its position.

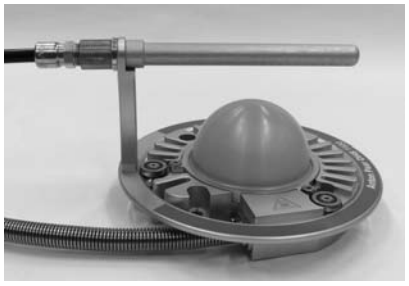


8. Remount the bearing.

9. Connect the cooling air hose to the quick coupling connector on the turnable HT Dome Cooling Nozzle ring.



10. Mount the PEEK dome.



5 Putting DHS 1100 into Operation

5.1 Alignment

The standard alignment of the DHS 1100 is carried out at room temperature.

CAUTION

Radiation
The DHS 1100 represents an open system and therefore is only allowed to be operated on goniometers equipped with a radiation enclosure.

For very accurate measurements, e.g. $\theta/2\theta$ scans at 2θ angles < 60°, it may be necessary to repeat the alignment at the temperature of the measurement to compensate the thermal height expansion of sample stage and sample. Data for the thermal expansion of the sample stage are shown in *Appendix A: Technical Data*.

To align the sample height, proceed as follows:

1. Mount the sample as described in *chapter 7.2 Mounting the Sample*.
2. Perform the standard alignment of the sample, referring to the *Diffractometer Instruction Manual*.

5.2 Starting CCU 100

Proceed as follows:

1. Press the mains switch on the front panel of the CCU 100.

The micro controller of the CCU 100 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 100 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

2. Check whether the correct Cooling Type parameter is set, as described in *Chapter 6.3* :
   - cON ... turns the compressed-air on/off at a temperature of dSP + 1 °C.
   - cOFF ... turns the compressed-air on at a temperature of > 200 °C and off at a temperature < 195 °C.
3. Press the HEATER button to start control of the sample plate temperature.

If the instrument condition is OK, the green HEATER LED lights up and all error messages disappear. The sample plate 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 remains in Standby mode (flashing HEATER LED) and the Error LED is flashing.

6 Operating CCU 100

This chapter describes the operation of the CCU 100 Combined Control Unit. CCU 100 can be operated manually or by remote control.

Manual control is done with the keys on the front panel of CCU 100. Manual operation is described in *Chapter 6.6 Manual Control of CCU 100*.

Remote control is done with a computer interfaced to CCU 100 via the serial RS 232 interface and with suitable software. See *Chapter 6.7* for more information.

CCU 100 can only store one target value for temperature and heating/cooling rate at a time. Programming temperature profiles into CCU 100 is not possible on the controller itself. However, the delivered control software NAMBICON 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 100 contains the keypad, the status LEDs and the display which are described in the following section.

Fig. 6-1: Front panel of CCU 100

6.2 Keypad

The keypad of CCU 100 has 6 keys to select parameters and enter values.

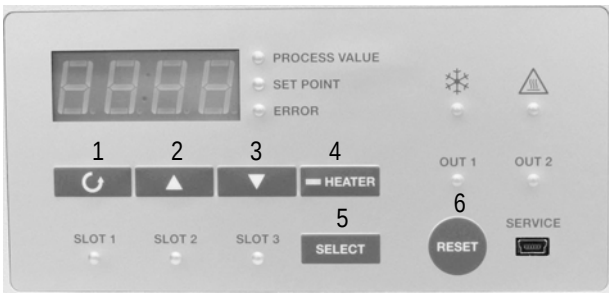


Fig. 16: Keypad of CCU 100

No	<Key>	Function
1	SCROLL	Switches to next parameter
2	UP	Increase the value of the current 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. Long: selects a slot.
6	RESET	Resets an error.

6.2.1 Satus 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.

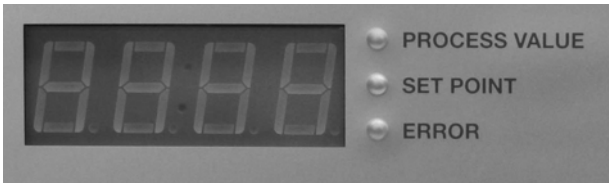


Fig. 17: LEDs for value of CCU 100

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. 18: LEDs for status of CCU 100

LED	Condition
COOLING	cooling active*
HEATING	heater active
OUT1	additional connector OUT1 on rear panel of CCU 100 is active
OUT2	additional connector OUT2 on rear panel of CCU 100 is active

** Only for chambers with active SAMPLE cooling. Not valid for DHS 1100.*

The last type of LEDs indicates the actual active slot of CCU 100.

Fig. 19: LEDs for active slots of CCU 100

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 plate of DHS 1100.

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 9.1* .

6.3.1 Navigation Diagram of CCU 100

The complete navigation diagram is shown below:

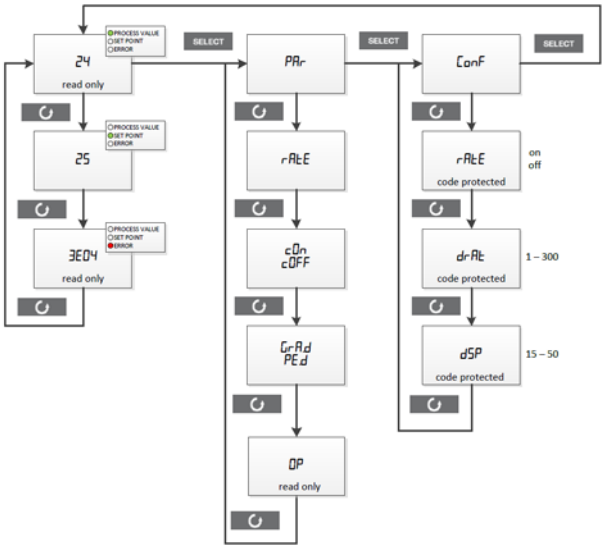


Fig. 20: Navigation diagram

The navigation diagram of CCU 100 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
cOn cOFF	Allows to switch the cooling of the instrument on and off.	cON: Cooling of the housing is switched on above dSP + 1 °C. cOFF: Cooling of the housing is switched on at temperatures above 200 °C.
GrA.d PE.d	Configuration for graphite dome and PEEK dome. Limits the operating temperature depending on the used dome.	Graphite dome: max. 1100 °C PEEK dome: max. 900 °C
OP	shows the actual heating power of the system in %.	

NOTICE

Risk of damage

- The max. operating temperature for graphite and PEEK depends on the used atmosphere and dome.
- The max. operating temperature for the PEEK dome depends on the used cooling nozzle.

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 - 300 °C	This parameter is password protected.
dSP	Allows changing of the default set point (25 °C) from 15 - 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 LEDs light up and the sample plate 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.

CAUTION

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 100 that the sample plate temperature is below 50 °C before you open the sample chamber and touch any parts inside.

6.5 Controlling the Compressed-Air Cooling

The cooling device is automatically turned on and off by CCU 100 whenever it is needed.

There are two modes available:

1. cOFF: An operation of the DHS 1100 without a dome is possible below 200 °C requiring that the compressed-air cooling is switched off. Exceeding 200 °C the CCU 100 automatically switches on the compressed-air cooling.
2. cON: Compressed-air cooling is continuously switched on above dSP + 1 °C. This mode allows faster cooling between 200 °C and room temperature compared to cooling mode cOFF.

6.6 Manual Control of CCU 100

Manual operation of DHS 1100 is done with the keys on the front panel of CCU 100. 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 100. (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 plate 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.*

6.7 Remote Control of CCU 100

Usually CCU 100 is integrated in the software that controls the X-ray diffractometer. For more information about controlling DHS 1100, 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 Operation

Measurements with the DHS 1100 are performed in the same way as when using the standard sample stage for the diffractometer.

7.1 Preparing the Instrument

Before starting to work with the DHS 1100 make sure that the following safety requirements are fulfilled:

CAUTION

Radiation
The DHS 1100 represents an open system and therefore is only allowed to be operated on goniometers equipped with a radiation enclosure

CAUTION

Hot surface
During operation and even after turning off the heating, the sample stage and the dome can be hot. Make sure that all parts of the DHS 1100 are below 50°C before touching the instrument.

WARNING

Make sure that no inflammable material is lying below or near the DHS 1100 during operation.

In the temperature range between room temperature and 200 °C it is possible to work without the cooling gas and without the dome. At temperatures above 200 °C the dome has to be mounted and the air cooling must be on to guarantee sufficient heat removal from the dome and the housing of the DHS 1100.

For operation with a PEEK dome at elevated temperatures a modification of the cooling system with a cooling nozzle for DHS 1100 is necessary.

CAUTION

Never touch the dome during operation especially when it is evacuated or at elevated temperatures, because this may easily damage the dome.

For information about the temperatures of the dome, the housing and the goniometer after operation at 1100 °C for 1 hour, refer to *Appendix A: Technical Data*.

7.2 Mounting the Sample

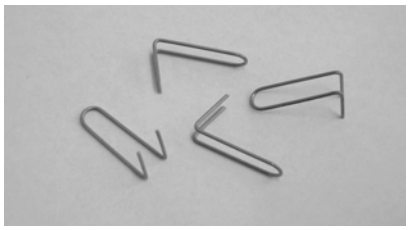
7.2.1 Flat Samples

The DHS 1100 heating attachment is preferably used for flat and/or flake-shaped samples with a maximum diameter of 25 mm and a maximum thickness of 2 mm. The recommended sample diameter is 15 – 20 mm and the recommended sample thickness is ≤ 1 mm. This ensures good clamping of the sample to the heating plate and maximum temperature homogeneity across the sample.

CAUTION

- Make sure that the sample components do not react with the sample stage at elevated temperatures.
- Check the melting point of the sample **before** the experiment and choose the temperature of the investigation correspondingly.

The sample is fixed on the sample plate with springs. The springs are supplied in the accessory box and can be easily exchanged by pulling them out of the small bore holes. Depending on the sample size, use either the large or the small springs for fixation.



CAUTION

Hot surface
During operation and even after turning off the heating, the sample stage and the dome can be hot. Make sure that all parts of the DHS 1100 are below 50 °C before touching the instrument.

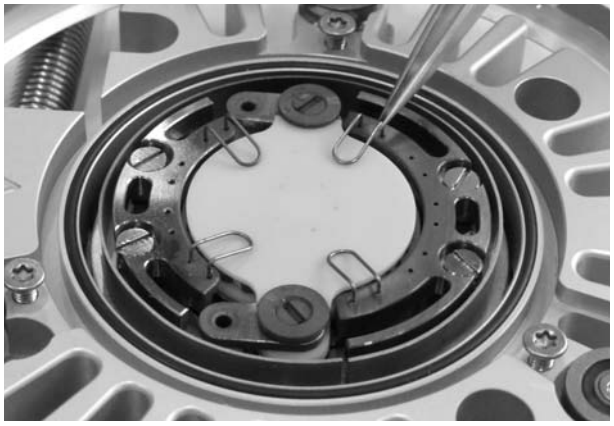
CAUTION

Use a pair of tweezers for fixing the sample on the sample stage.

- Carefully mount the sample with the fixing springs and do **not** bend or deform the springs.
- Used fixing springs can be bend and used for further measurements or replaced (refer to *Appendix E: Parts List*).

To mount the sample, proceed as follows:

1. Loosen the fixing springs by using tweezers.



2. Place the sample in the middle of the heating plate to make sure that the X-ray beam hits the sample. If the X-ray beam hits the heating plate or the springs, diffraction and fluorescence from the heating plate or the springs may be detected. In this case mention notice *Appendix C: Diffraction Patterns*).
3. Insert and push down the springs to fix the sample. Make sure that the sample has a very tight contact to the heating plate to guarantee optimum heat transfer. Fix the sample tightly so that it does not move during the measurement (upon movement of the goniometer).

7.2.2 Powder Samples

Optionally, a powder sample carrier (PSC) and corresponding anti-scatter slits (ASS) are available for the DHS 1100 (see Appendix E: parts list).

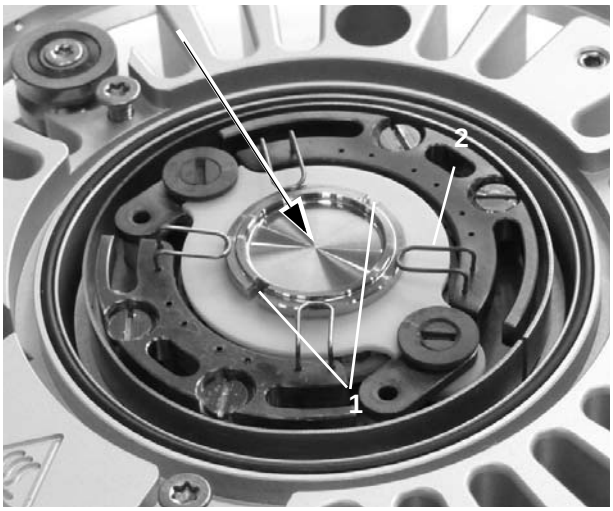


Fig. 7-1: Powder Sample Carrier (PSC)

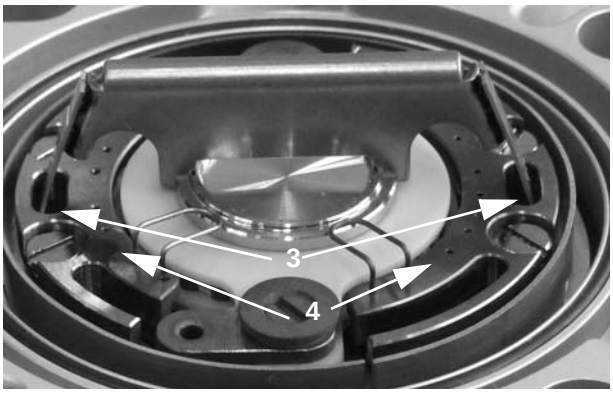


Fig. 21: PSC with Anti-Scatter slit (ASS)

Both, the powder sample carrier (cup depth: 1mm; cup diameter: 15 mm) and the anti-scatter slit are made of Inconel. Two different heights of the anti-scatter slit are available: 0.5 mm and 1 mm.

Mounting without Anti-Scatter Slit

CAUTION

Make sure that the temperature of the heating plate of DHS 1100 is < 50 °C before you mount/ dismount the PSC and ASS.

1. Put the PSC in the middle of the heating plate.
2. Fix the PSC with the short (5 mm) fixing springs (Fig. 7-1: 2) delivered with DHS 1100.

Mounting with Anti-Scatter Slit

The Anti-Scatter slit has springs on either side (Fig. 21: 3) with which it is fixed in the segments (4) around the heater as shown in Fig. 21: . Grooves in the PSC (Fig. 7-1: 1) position the ASS on the PSC.

Fig. 22: Mounting of the ASS

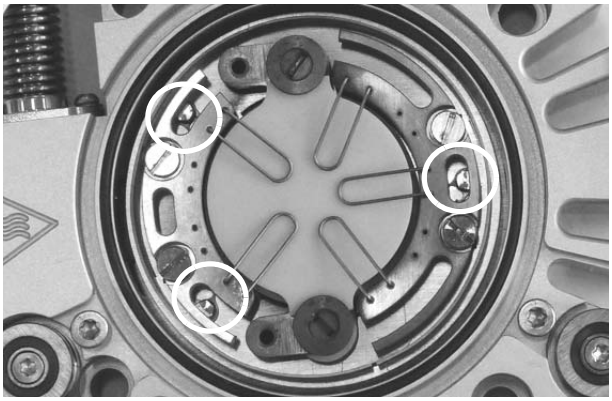


Fig. 23: Screws underneath the segment

1. Put the PSC in the middle of the sample cup.
2. Insert one side of the ASS into the opening in one of the segments and connect the PSC and the ASS as shown in s. Be careful not to scratch the heating plate with the ASS.

NOTICE
Risk of damage
We recommend to use the two segments shown in Fig. 7-3. In case you cannot push down the ASS completely due to the screws, the other segments are also possible however make sure, that the spring does not touch the heater (1) as a short circuit may occur otherwise.

3. Press the spring on the other side inwards and insert it into the ring. Slightly move the PSC so that the ASS rests in both grooves and fully push down the ASS onto the PSC.
   When you mount the ASS as shown in Fig. 22: , mind the screw underneath the segment (marked in Fig. 23:) and position the ASS beside the screw. Otherwise you cannot push it down completely.
4. Secure the PSC with min. two springs.

The PSC increases the temperature deviation between the sample surface (probed with the X-ray beam) and the value displayed on the CCU 100. We recommend to carry out a temperature validation measurement to determine this deviation before starting with the actual experiments.

NOTICE
Risk of damage
The springs of the ASS loose their elastic force at temperatures above ~ 750 °C. We recommend to use the ASS only at temperatures below this value.

7.2.3 Sample Temperature

Be aware that, depending on the sample thickness and the thermal conductivity of the sample, the temperature of the heating plate will be different from the temperature of the sample surface.

The temperature gradients across the sample are much smaller in inert gas atmosphere than in vacuum.

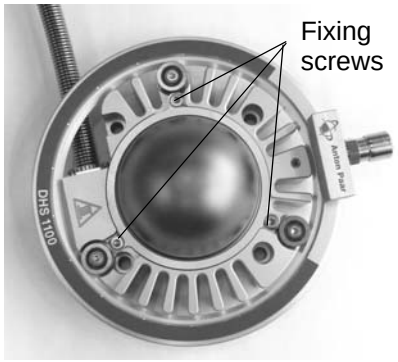
7.3 Mounting and Removing the Dome

CAUTION

- Always check the dome before you mount it. In case of a defect do not mount it.
- The graphite dome is a fragile item, handle it with particular care.
- Always lift the dome by the fixing ring, do not touch the graphite dome.
- Always make sure that all parts of the instrument are cooler than 50 °C before you start to remove the dome.

To **mount the dome**, carry out the following steps:

1. Make sure the O-ring and the contact surface of the dome are free from dust.



2. Place the dome on the O-ring.
3. Turn the dome until the three clasps rest underneath the fixing screws.
4. Fix the 3 screws using the screw driver from the accessory box.
5. Check whether the dome is mounted firmly and cannot fall off the instrument due to the movement on the goniometer.

To **remove the dome**, carry out the following steps:

1. Turn off the heater on the CCU 100 temperature control unit (refer to the *CCU 100 Instruction Manual*).
2. Make sure all parts of the DHS 1100 are cooler than 50 °C.
3. Loosen the 3 screws using the screw driver from the accessory box.
4. Take the dome by the fixing ring and turn it until the three clasps are free.
5. Lift the dome off the housing.

7.4 Operation of DHS 1100 with PEEK Dome

- For operation with a PEEK dome, the
  - dome cooling nozzle or
  - the HT dome cooling nozzle must be mounted.

NOTICE
Risk of damage
Operation without cooling nozzle and blind cover will cause insufficient cooling and overheating of the dome.

- When you operate DHS 1100 with a graphite dome:
  - remove the dome cooling nozzle and mount the blind cover or
  - remove the HT dome cooling nozzle and replace it by the standard cooling ring delivered with the DHS 1100.
- Only evacuate and vent DHS 1100 when the sample plate temperature is < 50 °C. Otherwise the dome can implode.

CAUTION

Hot surface
Do not touch the PEEK dome while heating the sample plate.

7.5 Performing a Measurement

After having mounted and aligned the sample you are now ready to start with your measurements (for sample mounting and alignment refer to *chapter 7.2 Mounting the Sample* and *chapter 5 Putting DHS 1100 into Operation*).

If the sample stage temperature does not exceed 200 °C during the entire measurement, the measurement can be carried out without dome and cooling air. Above 200 °C, dome and cooling air are necessary. Use the configuration cOFF on the CCU 100.

Use cOn on the CCU 100, if it is required to start cooling the sample stage at a starting temperature of dSP + 1 °C or to cool down to dSP + 1 °C continuously.

7.5.1 Before Starting

Before you start the measurement:

- Check the dome. In case of any defect do **not** use it.
- Make sure that the dome is mounted correctly and the three fixing screws provide tight connection between dome and the DHS 1100 housing.
- Make sure that the pressure relief valve is not blocked or closed with a blind plug.
- Make sure that the temperature of your experiment is below the melting point of the sample and that no sample components react with the sample stage.
- Make sure that the goniometer is operated in a way so that it **always** moves back to its starting position. This is important to avoid that the hoses wind up.
  If changes in the diffractometer software are necessary, contact the diffractometer manufacturer.

NOTICE
We recommend to move the goniometer with the DHS 1100 on it over the complete range of angles intended for the measurement to ensure that the hoses are not caught by any parts of the diffractometer.

- Make sure that cooling air is supplied to the CCU 100 temperature controller and that the cooling air is connected to the DHS 1100 (refer to *chapter 4.4 Installing the Compressed Air Cooling*).
- If the PEEK dome is required, make sure that the dome cooling nozzle is installed (see *chapter 4.8: Installing the Cooling Nozzle*).

7.5.2 Creating a Sample Environment

With DHS 1100 samples can be investigated in vacuum or different gas atmospheres.

CAUTION

- The temperature control of the DHS 1100 is optimized for **stationary atmospheres** around the sample. Do not measure with a gas flow onto the sample or through the dome.
- The atmosphere inside the dome has an influence on the maximum achievable sample heating/cooling rates and on the temperature offset between sample stage and probed sample surface.
- When the DHS 1100 is filled with **helium**, the **maximum operating temperature** is **limited** (see technical specifications appendix A) due to the large heat transfer from the heating plate to the dome.

7.5.2.1 Operation with Air

No special measures must be taken if the sample is measured in air. The sample stage can be heated up to the maximum temperature of 1100 °C, depending on the used dome.

7.5.2.2 Operation with Non-Reactive Gases

In order to prevent oxidation of samples, the DHS 1100 can be filled with non-reactive gases like helium or nitrogen. We recommend not to use argon because of its high X-ray absorption.

CAUTION

All pressure values in this section are relative to atmospheric pressure!

The **pressure relief valve** in the DHS 1100 housing is set to 0.35 ± 0.05 bar. This is the maximum pressure which can be achieved inside the dome.

CAUTION

- Use a suitable pressure reducer/regulator with a range from 0 to ≤ 5 bar and an accuracy of ± 0.1 bar to control the gas pressure supplied to the DHS 1100.
- Make sure the pressure relief valve is not blocked or closed with a blind plug.
- Avoid pressure bursts, because they can destroy the dome.
- For flushing the instrument with gas, never apply more than 1 bar pressure. This can destroy the dome.

To **fill the sample stage with gas**, proceed as follows:

1. Feed a suitable gas hose through the cabinet of the diffractometer and connect it to the flange (DN16KF) on the connection device of the DHS 1100 (refer to *chapter 4.5 Connecting the Gas Supply/Vacuum Equipment*).
2. Connect the other end of the gas hose to the pressure reducer/regulator of your gas supply.
3. In order to remove the air from the sample stage, apply a pressure of approx. 0.5 bar. This opens the pressure relief valve and the instrument is flushed with the non-reactive gas.
4. After a few minutes, depending on the permitted amount of residual air, reduce the pressure to 0.2 - 0.3 bar to close the pressure relief valve and create a stationary atmosphere.
5. To remove the gas from the DHS 1100, close the gas supply, slowly loosen the fixing screws of the dome to allow the gas to escape and take off the dome.

The feed-through adapter can be used together with the hose connector (for hoses with an inner diameter of 4 mm) for additional gas discharge after removing the pressure relief valve or the blind plug.

7.5.2.3 Operation under Vacuum

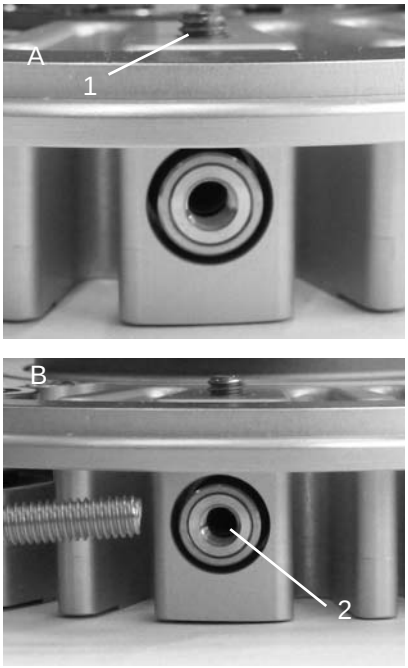
Inside the dome of the DHS 1100 a vacuum of < $6x10^{-1}$ mbar can be achieved with a standard vacuum pump with 4.2 m^3/h capacity.

When applying vacuum to the instrument, the pressure relief valve is automatically closed. For vacuum measurements we recommend to mount the blind plug, which is supplied in the DHS 1100 accessory box (see also *chapter 2.2.3 Housing and Internal Parts*).

To **replace the pressure relief valve**, proceed as follows:

The pressure relief valve can be replaced with the blind plug by loosening the Allen screw on top of DHS 1100 with a 2 mm Allen key (A). Insert a screw (M4, not supplied by Anton Paar) into the embedded valve to pull it out of the DHS 1100 (B).

Then insert the blind plug and tighten the Allen screw.



1 Allen screw
2 Pressure relief valve

To **evacuate the sample stage**, proceed as follows:

1. Feed an appropriate vacuum hose through the cabinet of the diffractometer and connect it to the flange (DN16KF) on the connection device of the DHS 1100 (refer to *chapter 4.5 Connecting the Gas Supply/Vacuum Equipment*).
2. Connect the vacuum hose to a suitable vacuum pump, e.g. a rotary pump.
   Between vacuum pump and DHS 1100 connec-

tion device you should have

- a valve to close the vacuum pump
- a valve to vent the DHS 1100 sample stage
- a pressure gauge.

3. To apply vacuum, close the venting valve and open the valve to the vacuum pump.

CAUTION

Be aware that it takes some time until the vacuum is good enough for the measurement (approx. 15 min for $6x10^{-1}$ mbar with a pump with 4.2 m^3/h capacity).

NOTICE

Anton Paar GmbH offers a suitable vacuum equipment for the DHS 1100. All required components for the installation are included in the delivery. Please refer to *Appendix E: Parts List*.

7.5.2.4 Reactive, Explosive, and Poisonous Gases

WARNING

Do **not** operate the DHS 1100 with reactive, explosive or poisonous gases or with gases which form explosive mixtures with air.
In particular, do **not** operate DHS 1100 with hydrogen.

7.5.3 Setting the Sample Temperature and Heating Rate

Sample temperature and heating/cooling rate are controlled by the CCU 100 Combined Control Unit.

There are two ways of defining the desired sample temperature and heating/cooling rate:

1. Sample temperature and heating/cooling rate can be set manually by using the keypad on the front panel of the CCU 100. Refer to the *CCU 100 Instruction Manual* for detailed information.
2. Sample temperature and heating/cooling rate can be defined in the diffractometer control software. Refer to the *Diffractometer Instruction Manual* for detailed information.

The **maximum heating and cooling rate** which can be set in the CCU 100 (parameter SPR) is 300 °C/min. Be aware that this is not the maximum heating or cooling rate for the sample plate which you can actually achieve with the instrument. The achievable heating and cooling rates for the sample plate depend on the operating conditions (gas type, temperature range). Information about the physical range of heating and cooling rates is given in *Appendix B: Temperature Control Data*.

Temperature control of CCU 100 has been optimized in such a way that it can be used for all specified sample environments without any adjustments to the internal control parameters. Due to the different thermal properties of the specified gases, a temperature overshoot of a few °C may occur under certain operating conditions. The temperature overshoot can be reduced by reducing the heating rate.

7.5.4 Recording the X-ray Scan

Before you start the scan make sure that the connector on the turnable cooling ring does not block the incident or diffracted beam.

Refer to the *Diffractometer Instruction Manual* for information about recording an X-ray scan.

8 Putting DHS 1100 out of Operation

When you stop using DHS 1100 for a short period without removing it from the diffractometer, proceed as follows:

1. Cool down the sample plate to room temperature
2. Turn off the heater with the HEATER button.
3. Remove the sample, clean the sample plate and put it back into the sample chamber.
4. Turn off the compressed-air supply.
5. Turn off CCU 100 completely (optional).

In order to remove DHS 1100 from the diffractometer continue as follows:

1. Completely turn off CCU 100 (mains switch).
2. Disconnect all electrical cables from the connection device.
3. Disconnect the compressed-air hose from the quick coupling connection for cooling air supply.
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.

9 Troubleshooting

9.1 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 plate not mounted	The contact switch does not detect the sample plate.*
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 100 Air Cooling Fail	The protective thermoswitch inside CCU 100 is activated.
E10	Other Messages	Internal errors of CCU 100 ⇒ 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 DHS 1100 (valid for other non ambient attachments)*

9.2 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.

9.3 Error Analysis

1. CCU 100 does not start after turning on.

cause	action
mains cable not properly connected	Make sure the CCU 100 is properly connected to mains.
wrong **supply voltage**	Required voltage AC 100 - 240
mains fuse blown	Replace mains fuses (see Chapter 10.4.2).

2. Sensor Break (E01)

cause	action
temperature sensor cable not properly connected	Check temperature sensor cable and connectors.
temperature sensor cable broken	Check continuity on the connection box.
DHS 1100 or **CCU 100** 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.
set temperature cannot be reached in the predefined time interval	Check the heater resistance. –––> Contact Anton Paar GmbH.
heater defective	⇒ Contact Anton Paar GmbH
heater cable not properly connected	Check heater cable and connectors.

4. Housing Temperature too high (E04)

cause	action
no cooling of housing	• Check if DHS 1100 is connected to the compressed-air. • Check if compressed-air is turned on.
insufficient cooling of housing	• Increase the compressed-air to max. 4 bar • Make sure all air hose connections on CCU 100 and DHS 1100 are tight.
cable to thermoswitch defective	⇒ Contact Anton Paar GmbH.

5. Insufficient air cooling (E05)

cause	action
no **compressed-air supply** to CCU 100	• Make sure compressed air is turned on. • Make sure all air hose connections on the supply unit and CCU 100 are tight.
air supply **pressure** too low	• Increase compressed-air pressure to max. 4 bar. • Make sure compressed-air is turned on • Make sure all air hose connections on CCU 100 and DHS 1100 are tight.

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 100 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
Low-Power module not installed correctly	Make sure the Low-Power module seats correctly (no gap on the side or inserted crookedly). If not, contact your diffractometer manufacturer or Anton Paar GmbH.
Low-Power module not installed	Check if the Low-Power module is installed. If not, contact your diffractometer manufacturer or Anton Paar GmbH.

9.4 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

When you seek technical assistance, please provide:

- The serial number of the instrument (component).
- A problem description containing the following information:
  - Is the instrument new or used?
  - Did the problem occur when starting operation or during operation?
  - What were the operating conditions when the problem occurred? (atmosphere, temperature range, ramp rate, type of samples, ...)
  - Which diffractometer is used?

9.5 Returning Items for Repair

An RMA number is needed to return an item for repair. Please contact the local service organization of your diffractometer manufacturer or Anton Paar GmbH in Graz, Austria before you send the item (see *Chapter 9.4 Technical Support* for contact details).

Clean the sample chamber before you send it back. Polluted instruments will not be repaired. If it is not possible to clean the sample chamber from all residues, safety data sheets for all possible contaminations must be sent together with the instrument.

10 Maintenance

10.1 Checking the Dome

The dome should be regularly checked, depending on the operating hours, the usual operating temperatures and the applied atmospheres.

CAUTION

- Always lift the dome by the fixing ring.
- Never touch the dome during operation, especially when it is evacuated or at elevated temperatures.
- Check the dome for cracks and coatings on the inner surface.
- If the inner surface of the dome has become coated with evaporated sample components, clean it with a dry cloth.

If cracks are observed, replace the dome (refer to *Appendix E: Parts List*).

10.2 Replacing the O-rings

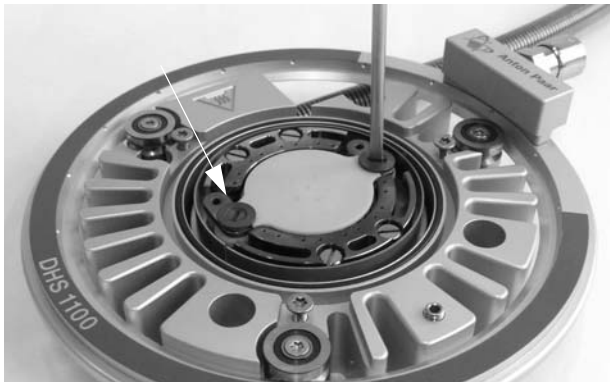
In general, commercially available O-rings made of Viton are used.

While detaching an O-ring, make sure that the sealing surfaces remain undamaged.

Before fitting a new O-ring, clean the sealing surfaces with a suitable solvent.

10.3 Changing the Hot Plate

1. Open the sample plate fixing screws.



2. Remove the old hot plate.

NOTICE
The cavities between the heating wires are filled with a ceramic paste. This paste provides a good thermal contact between the heater and the hot plate. Moreover, it acts as electrical insulation between the heating wires. Depending on the previous thermal treatment different cases can occur.

- The surface of the ceramic is rough.

Please use an abrasive paper and gently smoothen the surface to provide a good thermal contact to the hot plate.

- Parts of the ceramic powder are brittle and fall off the heater. The resulting cavities would give poor thermal accuracy.

Please send the DHS 1100 to Anton Paar to repair the heater.

3. Mount the new hot plate and remount the screws.

Tip: *Before you remount the screw, lubricate the screw with the high temperature lubricant, Molykote grease from Dow Corning, which you can find in your spare parts package.*

NOTICE
It is recommended to validate the temperature of the new hot plate by either measuring the transition points of known standards or by using the thermal expansion method of known substances.

10.4 Exchanging Electronic Components

10.4.1 Switching off the instrument

1. Switch off the CCU 100 by using the power switch on the front side of the instrument.
2. Unplug all cables from the rear side of the instrument.

CAUTION

High voltage
CCU 100 contains electrical components which may carry dangerous voltage. Always switch off the CCU 100 and disconnect it from mains before you open the housing.
Do not operate the CCU 100 when the housing is open.

10.4.2 Checking and Replacing Main Fuses

CAUTION

High voltage
Make sure that BEFORE performing any service/maintenance work of the CCU 100, the instrument is disconnected from the mains supply.
Only use the specified fuse types

NOTICE
Risk of damage.
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 100.

Exchange the mains fuse as follows:

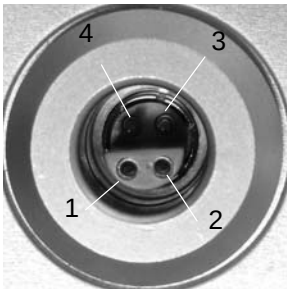
1. Switch off CCU 100 and disconnect all cables from the rear side.
2. Use a small screw driver to push out the fuse holder on both sides.
3. Press the plastic bracket together and take the fuses out.
4. Check the fuse or exchange the defective fuse. (The two mains fuses are made of ceramic. Spec.: 2 x T 6.3 A)

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

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

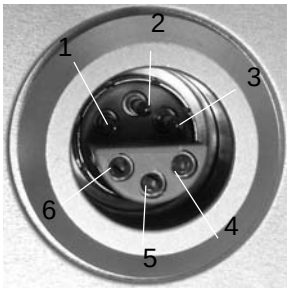
10.5 Connector Pin Assignment

Pin assignment of the heater/sensor connector on the connection device (3 in Fig. 9):



Pin	
1,2	Heater +
3,4	Heater -

Pin assignment of the thermocouple connector on the connection device (2 in Fig. 9):



Pin	
1	Thermocouple (+)
2	I-Button
3	Thermocouple (-)
4	DHS 1100 housing thermoswitch
5	I-Button
6	DHS 1100 housing thermoswitch

Appendix A: Technical Data

Temperature range

Graphite Dome:	
- Air, N_2, vacuum:	25 to 1100 °C
- Helium:	25 to 1000 °C
PEEK Dome (with Dome Cooling Nozzle):	
- Air, N_2, vacuum:	25 to 600 °C
- Helium:	25 to 400 °C
PEEK Dome (with HT Dome Cooling Nozzle):	
- Air, N_2, vacuum:	25 to 900 °C
- Helium:	25 to 750 °C
Heating rate:	max. 300 °C/min

Atmospheres

- Gases:	Air, N_2, inert gas
- Overpressure inside dome:	max. 0.35 ± 0.05 bar rel.
- Vacuum:	< 6 x 10^{-1} mbar

Temperature measurement & control

Temperature Control Unit:	CCU 100
Temperature sensor:	thermocouple Pt–Pt10Rh (type S)
Heater type:	Resistance heater

Dimensions/Weight

Diameter:	128 mm
Weight (without adapter):	430 g

Sample plate

Sample plate diameter:	29 mm
Sample diameter:	max. 25 mm
Sample thickness:	max. 2 mm
Height above housing base plate:	22 mm
Parallelism to housing base plate:	≤ 0.05 mm
Thermal height expansion:	see graph below
Sample fixing:	Inconel springs

Dome

Diameter:	63 mm (66 mm incl. cooling ring)
Wall thickness:	0.25 mm
X-ray transmission: (primary + diffracted beam)	CrKα 40 % CuKα 65 % MoKα 95 %
Angular range: - 2 Theta (2Θ) - Omega (ω) - Psi (ψ) - Phi (φ)	 0 to 166° 0 to 83° 0 to 85° 0 to 360°

Cooling requirements

Cooling air pressure: - with Graphite Dome - with PEEK Dome (Dome Cooling Nozzle) - with PEEK Dome (HT Dome Cooling Nozzle)	 min. 2 bar rel. / max. 4 bar rel. min. 2.5 bar rel. / max. 4 bar rel min. 2 bar rel. / max. 4 bar rel
Air flow rate at 2 bar:	0.12 m³/min
Gas hose - material: - inner x outer diameter:	 polyamide 4 x 6 mm

List of materials

Sample plate:	aluminium nitride
Sample fixing springs:	Inconel
Thermocouple:	Pt–Pt10Rh (type S)
Heater:	Kanthal
Dome:	PEEK, graphite
Housing:	aluminium, Inconel
Supply hose:	stainless steel + PVC
Insulation of electrical wires:	PTFE
Connection device box:	aluminium, Cr-plated brass

CCU 100 Dimensions

Width x Depth x Height:	450 x 410 x 180 mm
Weight:	approx. 13 kg

CCU 100 Electrical Data (for use with DHS 1100)

Voltage (mains):	AC 100 - 240 V
Frequency (mains):	50 ... 60 Hz
Power consumption:	max. 594 VA
Mains fuses:	2 x T 6.3 A H 5 x 20 mm (ceramic tube)
Overvoltage category:	II according to EN 61010-1
Voltage output:	max. DC 48 V
Current output:	max. DC 11.5 A
Heater fuse:	15 A (Blade Fuse)

Ambient conditions

Ambient temperature:	5 °C to 35 °C
Atmospheric humidity:	max. 80 % relative, not condensing
Pollution degree:	2 according to EN 61010
Maximum operating altitude:	3000 m above sea level

The DHS 1100 and the CCU 100 have been designed for INDOOR USE ONLY!
Protect the instruments from moisture!

Disposal

The instrument should be disposed of in accordance with the local regulations.

The DHS 1100 with CCU 100 Combined Control Unit 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: Temperature Control Data

The following measurements were done with a 1 mm thick AIN sample plate.

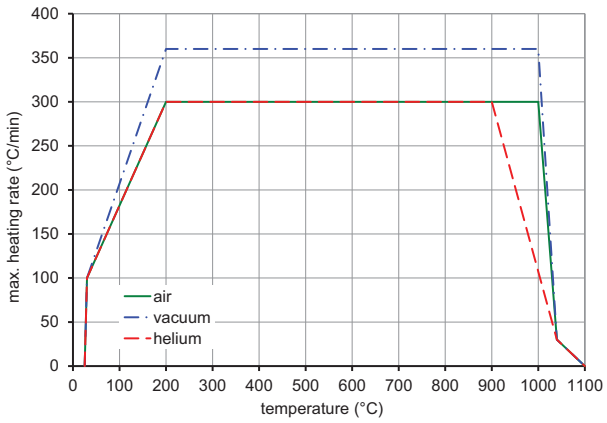


Fig. B-1: Maximum heating rate for different atmospheres

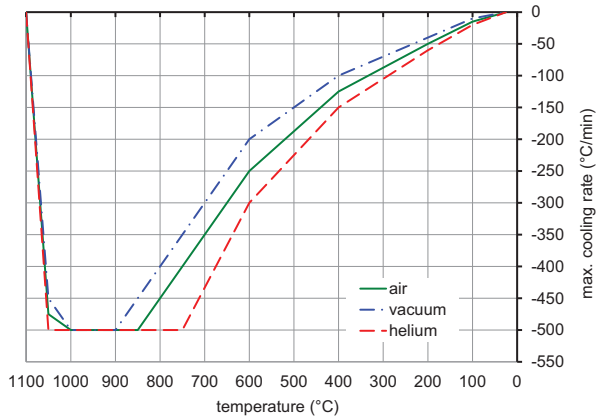


Fig. B-2: Maximum cooling rate for different atmospheres

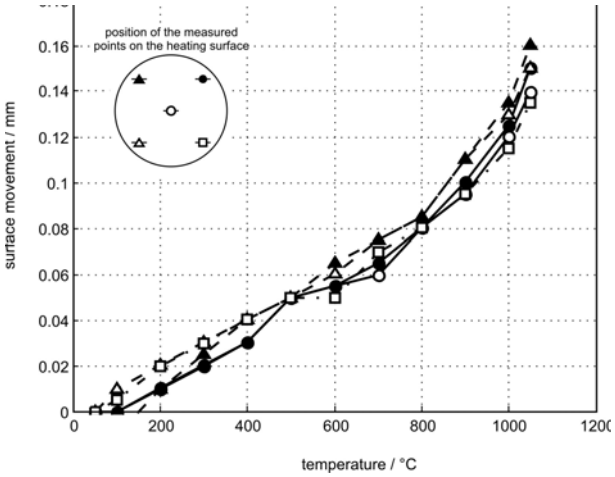


Fig. B-3: Thermal height expansion at five different sites

on the heating plate

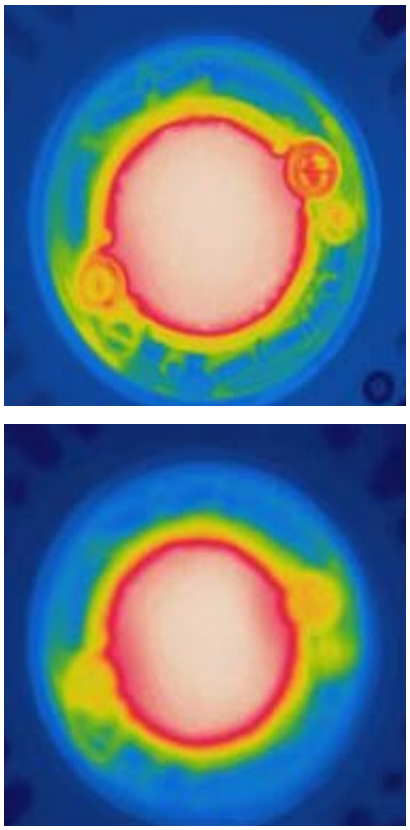


Fig. B-4: Temperature distribution across the heating plate

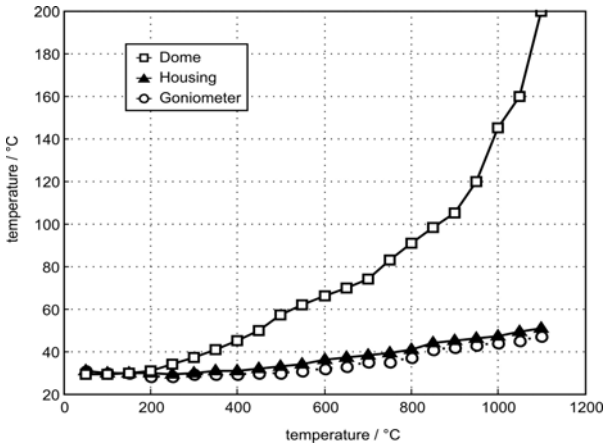


Fig. B-5: Temperature at dome, housing and goniometer after operation for 1 hour depending on the set temperature

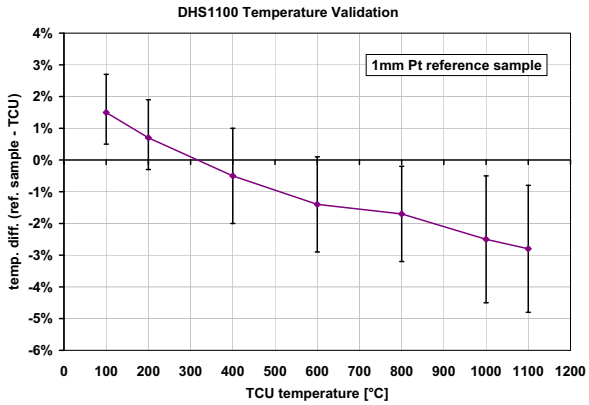


Fig. B-6: Sample surface temperature

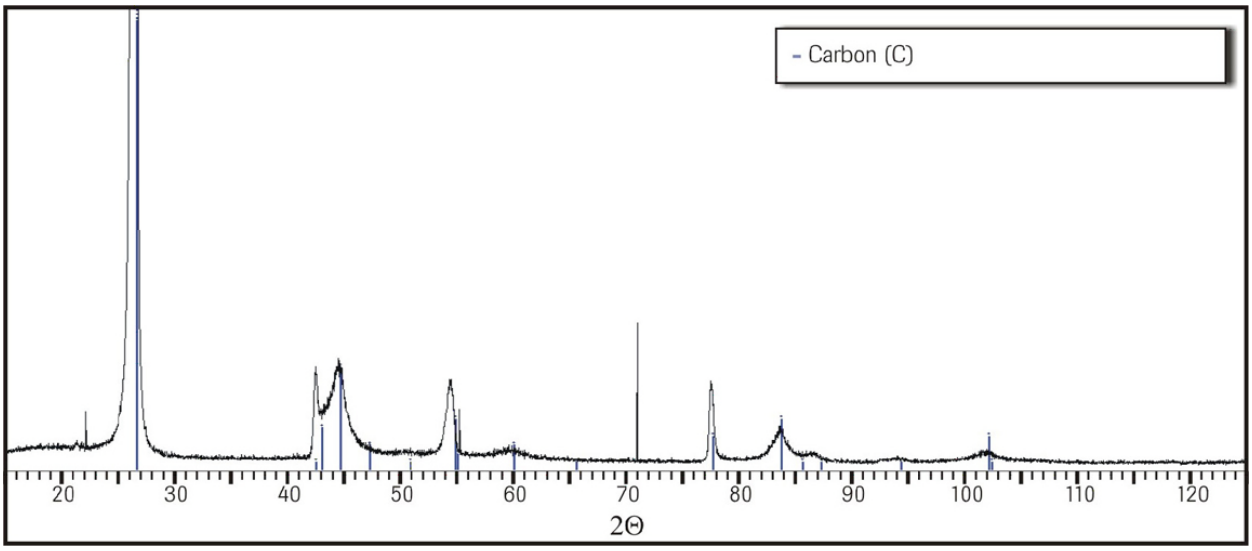
A 1 mm thick 10 x 10 mm platinum plate with a type S thermocouple spot- welded to the surface was used as a reference sample to give an indication of the temperature offset between the stage temperature displayed on the CCU 100 Temperature Control Unit and the surface of a sample scanned with the X-ray beam. The measurements were done in air.

The temperature deviation for your actual sample will depend on the sample thickness, the thermal contact between sample stage and sample, the thermal properties of the sample and the sample environment (gas/vacuum).

Appendix C: Diffraction Patterns

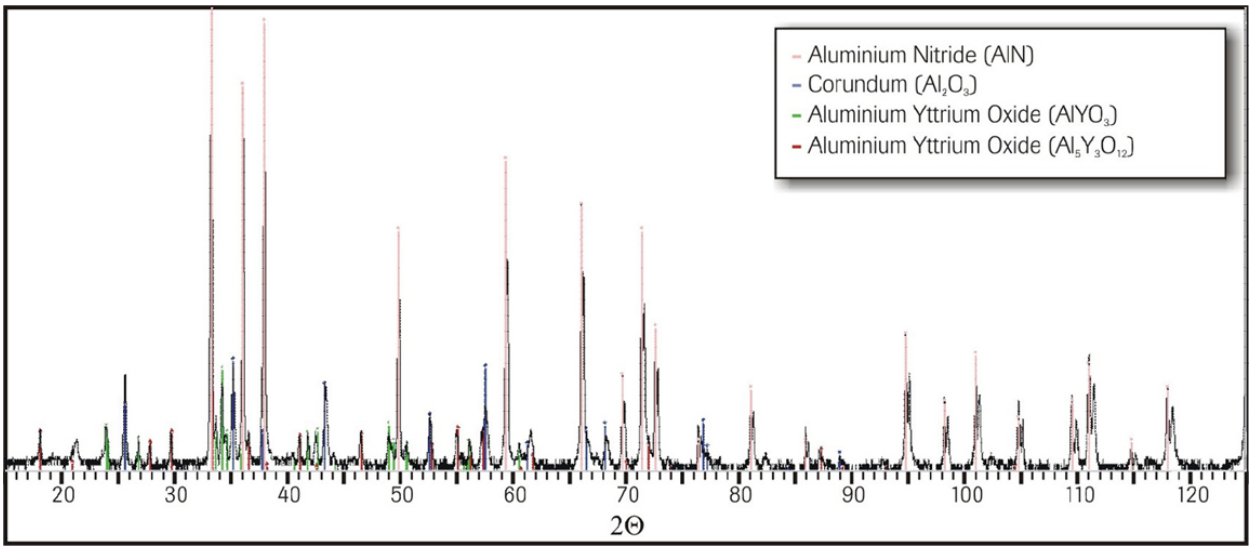
All diffraction patterns were measured with CuKα radiation in Bragg-Brentano geometry.

- Graphite



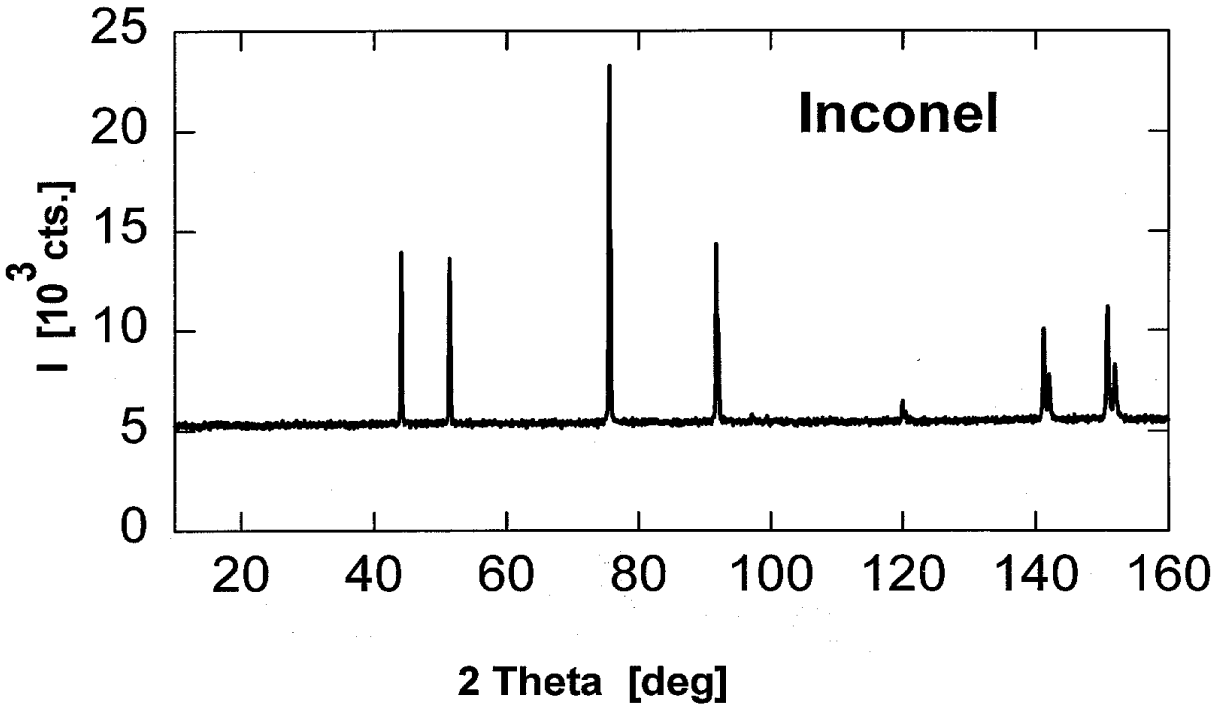
Set temperature 900 °C

- Aluminium nitride



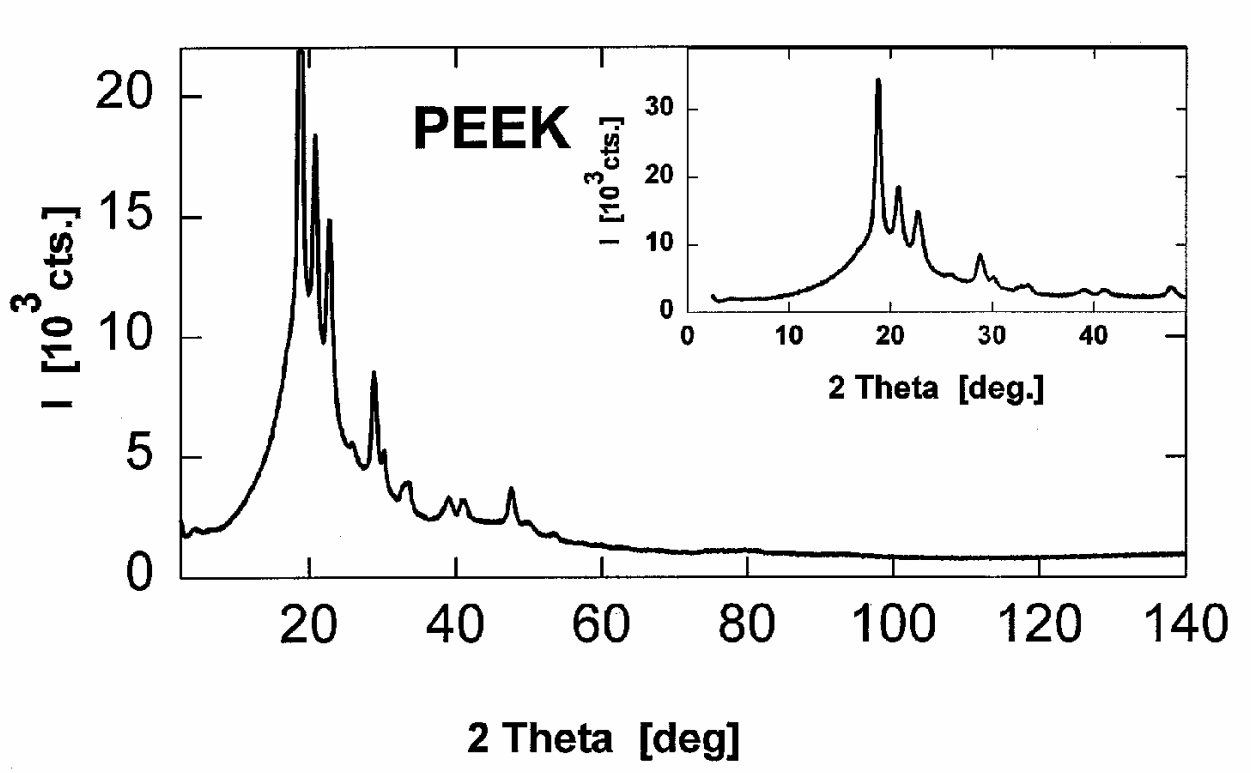
Set temperature 500 °C

- Inconel



Set temperature 25 °C

- PEEK



Appendix D: Warranty

The warranty regulations for the DHS 1100 are in accordance with the "General Terms of Delivery" of the Austrian Electrical and Electronic Industry.

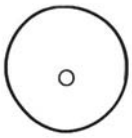
Anton Paar GmbH gives no warranty for the dome if it is not handled according to our instructions (see also *chapter 2.2.1 Dome* and *chapter 4.2 Unpacking the Instrument*).

Appendix E: Parts List

	Item	Cat. no.
	DHS 1100 DOMED HOT STAGE FOR FOUR-CIRCLE GONIOMETERS	25012
	CCU 100 COMBINED CONTROL UNIT	135000
	LOW-POWER MODULE FOR DHS 1100	176732

Adapter		
	Item	**Cat. no.**
	ADAPTER FOR BRUKER CRADLE AND GADDS XYZ STAGE	8201

Accessories		
	Item	**Cat. no.**
	AIR SERVICE UNIT	6931
	DOME COOLING NOZZLE DHS 1100 (600 °C)	135038
	DOME COOLING NOZZLE DHS 1100 (900 °C)	159567

	PEEK DOME DHS	7580
	ANTI-SCATTER SLIT SET DHS 1100 includes - 1mm SLIT (1 pc) - 0.5mm SLIT (1 pc)	143952
	SET OF POWDER SAMPLE CARRIERS DHS 1100 (3#)	143953
Spare Parts		
	Item	**Cat. no.**
	GRAPHITE DOME DHS	24998
	DHS 1100 HOT PLATE	22010
	FILTER CARTRIDGE (0.01 µm PORE SIZE)	8415
	O-RING SET DHS	7626
	SET OF CLAMPS FOR LARGE SAMPLES	7624
	SET OF CLAMPS FOR SMALL SAMPLES	7625

	SPARE PARTS PACKAGE DHS 1100 Consits of: 1x 24998 Dome 3x 22010 DHS 1100 hot plate 1x 7624 Set of clamps for large samples 1x 7625 Set of clamps for small samples 1x 7626 Set of O-Rings 1x 8415 Filter cartridge (0.01 µ pore size)	92920

Appendix F: 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:	**Domed Hot Stage with Combined Control Unit**
Model:	**DHS 1100 + CCU 100**
Material number:	**25011, 25012, 96450, 104030, + 135000**

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

Place and date of issue: Graz, 2016-12-06

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