Anton Paar

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

Galileo Galilei (1564-1642)

Reference Guide

TTK 600

Low Temperature Chamber with

CCU 100 Control Unit

Further information

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Reference Guide

TTK 600

Low Temperature Chamber with

CCU 100 Control Unit

Contents

1 Safety Instructions

- Read the documentation before using TTK 600.
- Follow all hints and instructions contained in the documentation to ensure the correct use and safe functioning of TTK 600.
- 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

- This 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 TTK 600 if no adjustments have been made to the mechanics, electronics, and firmware.
- Only use TTK 600 for the purpose described in the documentation. Anton Paar GmbH is not liable for damages caused by incorrect use of TTK 600.

Installation and use

- TTK 600 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 (see chapter 3).
- 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 TTK 600. Do not operate the instrument under conditions which could result in damage to goods and/or injuries and loss of life.
- Check TTK 600 for chemical resistance to the samples and cleaning agents.

Maintenance and service

- The results delivered by TTK 600 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 TTK 600, observe the legal requirements in your country.

Returns

- For repairs send the cleaned TTK 600 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 TTK 600.
- 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 TTK 600 must be

kept free of inflammable gases and vapors.

- Connect TTK 600 to the mains via a safety switch located at a safe distance from the instrument. In an emergency, turn off the power using this switch instead of the power switch on TTK 600.
- Supply a fire extinguisher.
- Ensure the sufficient supervision of TTK 600 during operation.

Work with liquid nitrogen

- For detailed safety information on working with liquid nitrogen, please refer to appendix E.

1.2 Conventions for safety messages

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

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

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

WARNING

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

Wear protective gloves
Wear protective gloves when handling the TTK 600.

Wear safety goggles
Wear safety goggles when handling the TTK 600.

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

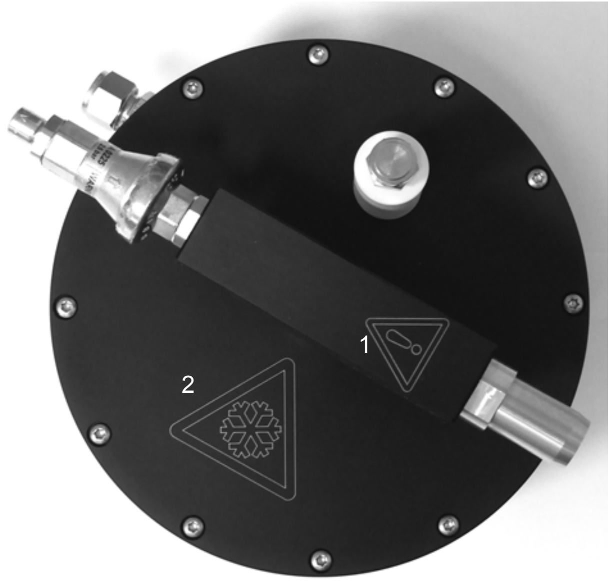
1.3 Safety Signs on the Instrument

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.



1 *CAUTION*
2 *CAUTION - Cold surface*

Fig. 2: Position of warning signs on the safety box

CAUTION

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

Wear protective gloves
Wear protective gloves when handling the TTK 600.

Wear safety goggles
Wear safety goggles when handling the TTK 600.

NOTICE
Take care that the warning symbols remain clearly legible.

2 TTK 600 - An Overview

The TTK 600 Low-Temperature Chamber is an attachment for 2-circle X-ray diffractometers with vertical goniometer to cool and heat samples for in-situ X-ray diffraction.

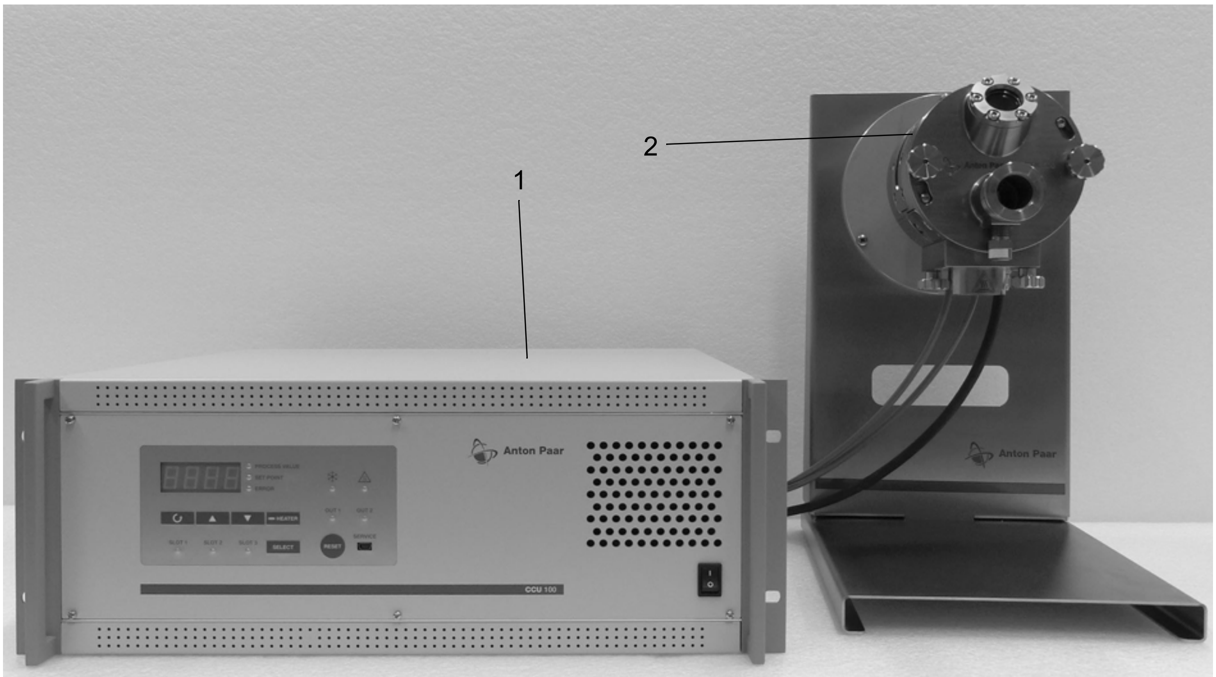


Fig. 3: Main Components of the instrument

1 *Combined Control Unit CCU 100 to control the sample temperature*
2 *Sample chamber (mounted on the stage mount)*

2.1 Intended Use of the Instrument

The TTK 600 Low-Temperature Chamber has been designed for use on powder X-ray diffractometers with a vertical goniometer. The sample chamber is mounted on the goniometer instead of the standard (ambient) sample holder in order to heat or cool the sample to a defined temperature while scanning the sample with X-rays.

Different types of sample holders are available that allow to perform X-ray scans either in reflection geometry, transmission geometry using capillaries or transmission geometry for foils, pastes and also powders. Two cooling options are available for the instrument. With the liquid nitrogen cooling equipment, the temperature range is -190 °C to 600 °C. With the compressed-air cooling equipment the temperature range is -20 °C to 600 °C. The sample can be cooled/heated in vacuum or various gas atmospheres.

The sample temperature is controlled by a control unit (CCU 100). This control unit is installed in special compartments in the diffractometer or in an external rack beside the diffractometer.

TTK 600 should be used for powder samples (all three types of sample holders) or foils and pastes (transmission sample holder). The following sample materials must not be investigated with TTK 600:

- radioactive samples
- samples that are self-igniting in air or when exposed to humid gas.

CAUTION

Risk of injury
Observe that in case of an unlikely malfunction the highest possible temperature in the instrument can be up to 900 °C. Do not use samples that are self-igniting or form poisonous gases at that temperature.

If TTK 600 is operated under dynamic gas conditions, gas flows through the chamber and sample material can be released to the environment. This must be taken into account when measuring samples that present a health hazard.

TTK 600 can be operated under vacuum or standard gases like nitrogen or air. Certain other gases can be used as well, but the following types of gases are not permitted:

- poisonous gases
- explosive gases
- corrosive gases
- gases which can create explosive gas mixtures with air.

TTK 600 must not be pressurized to more than 2 bar rel. under all operating conditions.

2.2 Operating Principle

TTK 600 uses so called direct heating/cooling to control the temperature of the samples. Heating is done by a resistive heater underneath the sample holder. Cooling is provided by either compressed air or liquid nitrogen which cools down a cryostat which is located directly under the heater of the instrument. Measurement of the temperature is done by a thermocouple inside the heater and depending on the actual setup a second temperature is measured with a Pt100 which is directly integrated in the sample holder to ensure accurate temperature measurement.

The following table gives an overview of the accessible temperature range in dependence of the used atmosphere and the used type of sample holder.

Standard Sample Holder			
Cooling Equipment	Ambient Air	Dry Air/ N2/He	Vacuum
Liquid Nitrogen Cooling	+25 °C[1] +450 °C	-150 °C +450 °C	-190 °C +600 °C
Compressed Air Cooling	+25 °C[1] +450 °C	-10 °C[2] +450 °C	-20 °C[2] +600 °C

Transmission Sample Holder			
Cooling Equipment	Ambient Air	N2	Vacuum
Liquid Nitrogen Cooling	+25 °C[1] +450 °C	-175 °C +600 °C	-190 °C +600 °C
Compressed Air Cooling	+25 °C[1] +450 °C	-10 °C[2] +600 °C	-20 °C[2] +600 °C

Capillary Sample Holder		
Cooling Equipment	Ambient Air	N2
Liquid Nitrogen Cooling	+25 °C[1] +450 °C	-175 °C +600 °C
Compressed Air Cooling	+25 °C[1] +450 °C	-10 °C[2] +600 °C

Battery Sample Holder			
Cooling Equipment	Ambient Air	N2	Vacuum
Liquid Nitrogen Cooling[3]	+25 °C[1] +130 °C	-150 °C +130 °C	-180 °C +130 °C
Compressed Air Cooling	+25 °C[1] +130 °C	-10 °C[2] +130 °C	-15 °C[2] +130 °C

1 *This is a nominal limit. The true limit is the dew point temperature of the (ambient) air.*

2 *Observe the pressure dew point requirements for the compressed-air given in appendix A in order to avoid icing of the cryostat.*

3 *Recommended **heating rate/slope** for the **battery sample holder transmission**: < 20 °C for the temperature range < CL (cooling limit; refer to chapter 6.3.1).*

The standard sample holder of the instrument is used in reflection geometry, whereas the transmission sample holder and the capillary sample holder are made for transmission geometry.

2.3 TTK 600 Sample Chamber

The following chapters describe the chamber and its parts in detail.

2.3.1 Chamber Main Parts

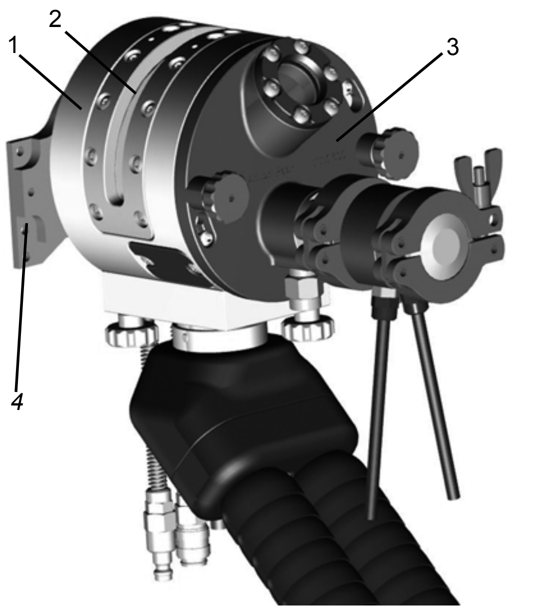


Fig. 4: Main components of the sample chamber

1 Chamber housing
2 X-ray beam window
3 Lid
4 Adapter for z-alignment stage (mechanical interface)

A suitable mechanical interface is necessary to mount TTK 600 on the goniometer. Two types of interfaces are available:

- Rigid adapters with a simple z-alignment device which requires manual alignment of the sample stage.
- Motorized z-alignment stages for PC-controlled alignment of the sample stage.

If a motorized z-alignment stage is used, the alignment stage is mounted on the base-plate of TTK 600 and the interface to the goniometer is mounted on the z-alignment stage.

The sample position can change due to thermal movement of the sample holder or sample shrinking/expansion. If correction of the sample surface displacement during a series of X-ray scans is required, we recommend to use a motorized z-alignment stage. For TTK 600 the sample holder displacement during heating and cooling is approx. ±0.1 mm.

2.3.2 Chamber Housing with Connectors

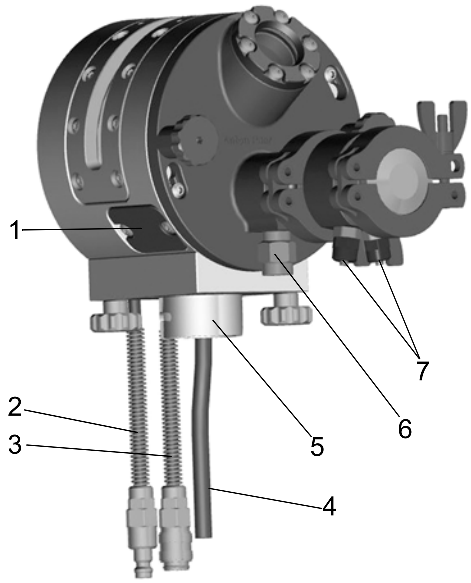


Fig. 5: Chamber housing with connectors

1 Blind plug (for optional additional connections)
2 Cooling water hose inlet
3 Cooling water hose outlet
4 Connection cable to CCU 100
5 Connector for sample cooling (liquid nitrogen or compressed air)
6 Overpressure relieve valve (3 bar)
7 Gas connectors

The TTK 600 chamber housing is made of aluminum and is water-cooled. The housing is slotted along the radiation path (entrance and exit of X-rays). The X-ray window is covered with a Kapton foil, which is pressed onto an O-ring by two clamping pieces.

There is one cable to connect TTK 600 to CCU 100. This cable is responsible for providing the heating current to the heater and for transferring the temperature read-out and the signals from the sensors within the instrument.

At the bottom of the housing is the connector for the sample cooling device, either the liquid nitrogen cooling equipment or the compressed-air cooling unit.

2.3.3 Chamber Lid

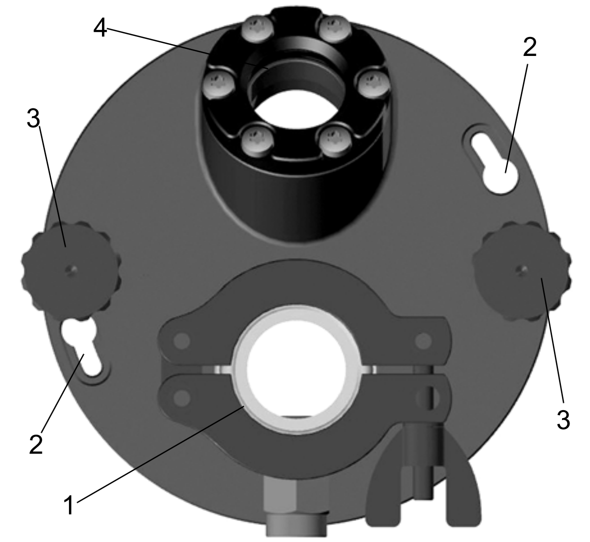


Fig. 6: Chamber lid

1 Vacuum flange
2 Bayonet fitting
3 Fixing screws
4 Viewing window

The lid of the TTK 600 is fixed to the housing by two bayonet fittings as well as two fixing screws and is sealed with an O-ring.

TIP: *There is a special lid for the use of the Capillary Sample Holder. For further information refer to chapter 2.3.5.3 and the Instruction manual for the Capillary Sample Holder.*

2.3.4 Internal Parts

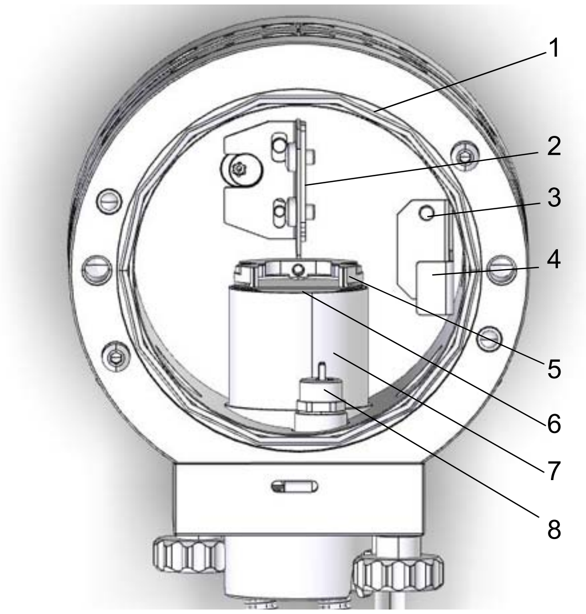


Fig. 7: Main parts inside the sample chamber

1 O-ring seal
2 Knife edge
3 Guiding rods for beam stop
4 Beam Stop (only for transmission configuration)
5 Sample holder fixing ring
6 Graphite foil
7 Cryostat
8 Pt100 plug connector

For detailed information on the materials used in the TTK 600, please refer to appendix A.

The temperature of the sample holder is controlled with a combination of resistance heater and cryostat. The cryostat can either be cooled with liquid nitrogen or compressed air (refer to chapter 2.4).

Temperature measurement is performed with a Pt100 temperature sensor which is embedded in the sample holder. For sample exchange the sensor can be disconnected with a plug.

2.3.5 Sample Holders

There are four sample holders for the TTK 600. Detailed information is contained in the chapters below.

2.3.5.1 Standard Sample Holder

Fig. 8: Standard Sample Holder

1 *Sample holder*
2 *Fixing screw for sample holder*
3 *Sheath resistance Pt100*
4 *Pt100 connector*

The standard sample holder of TTK 600 is optimized for measurements in reflection geometry. The sample holder is made of nickel. To provide good heat transfer from the heater to the sample holder, a graphite foil is placed in between. The sample holder is fixed onto the heating/cooling block with a screw (see Fig. 8-2).

2.3.5.2 Standard Sample Holder Heating Environment

Optionally, to minimize the deviation between the real sample surface temperature and the displayed temperature a standard sample holder environment can be used which inhibits the loss of heat to the surrounding atmosphere inside the TTK 600. The sample holder environment can be equipped with a graphite, nickel or Kapton foil (others on request). To mount the sample holder environment see chapter 8.4.2 Mounting the Standard Sample Holder Heating Environment.

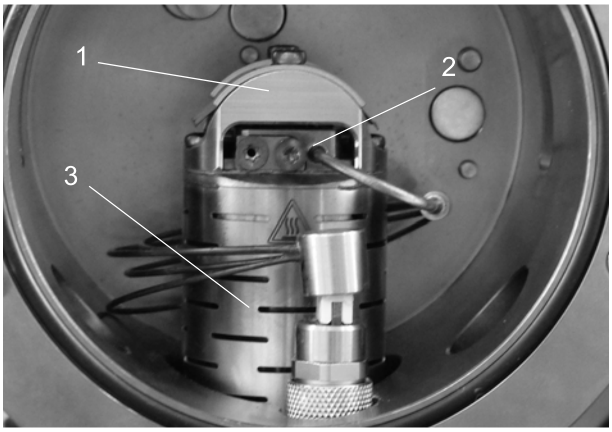


Fig. 9: Standard sample holder heating environment

1 *Standard sample holder heating environment*
2 *Sample holder*
3 *Heater insulation*

2.3.5.3 Capillary Sample Holder

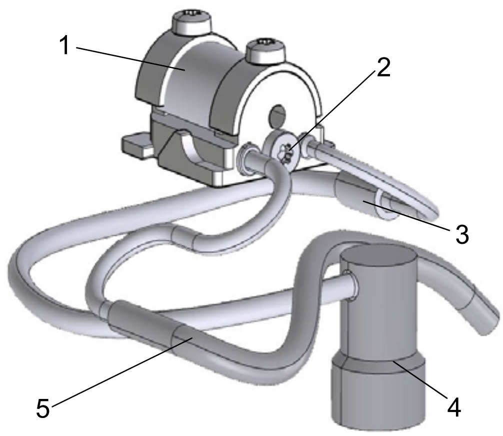


Fig. 10: Capillary Sample Holder

1 *Window foil of the capillary sample holder*
2 *Fixing screw for sample holder*
3 *Sheath resistance Pt100*
4 *Pt100 connector*
5 *Hose for convection heating of the capillary*

The Capillary Sample Holder is used for transparent samples in transmission geometry. The temperature distribution around the capillary is improved by the use of an additional convection heater. For further information on the Capillary Sample Holder, refer to the instruction manual
Capillary Sample Holder for TTK 600.

2.3.5.4 Transmission Sample Holder

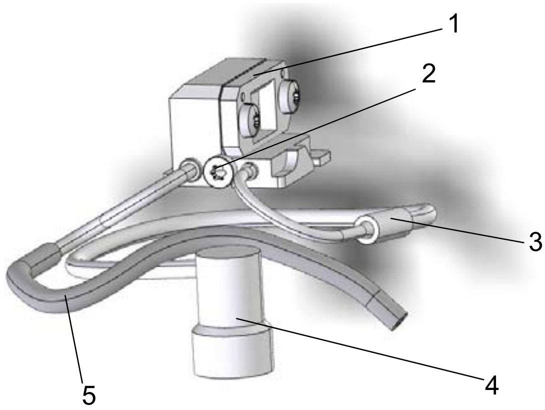


Fig. 11: Transmission Sample Holder

1. *Transmission sample holder heating environment*
2. *Fixing screw for sample holder*
3. *Sheath resistance Pt100*
4. *Pt100 connector*
5. *Hose for convection heating of the sample*

The Transmission Sample Holder is used for powders, foils and pastes in transmission geometry. The temperature distribution around the sample is improved by the use of an additional convection heater (necessary for Kapton foil only).

2.3.5.5 Antechamber for TTK 600

The Antechamber for TTK 600 is used for the transfer of air-sensitive samples to TTK 600.

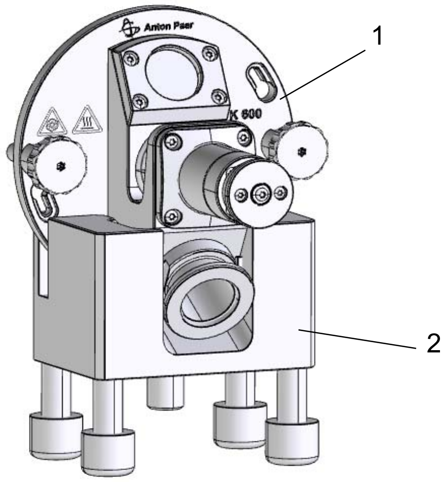


Fig. 12: Antechamber for TTK 600

1. *Antechamber TTK 600*
2. *Mount of Antechamber*

This can be done by loading the sample to the antechamber within a glove box. Detailed information about the necessary steps can be found in chapter 8.4.5.

The sample holder for the antechamber is different compared to the standard sample holder. The most important difference is that this type of sample holder has no integrated Pt100 temperature sensor. If the antechamber for TTK 600 is used, the temperature of the thermocouple inside the heater is used to measure the temperature.

For the recognition of this type of sample holder, the Pt100 blind plug, which is delivered together with the antechamber has to be connected to the Pt100 plug connector.

2.3.5.6 Battery Sample Holder

The battery sample holder is used to measure coin cell battery samples in either reflection or transmission geometry. An anode and a cathode are located on either side of the sample so that the behavior of the coin cell can be monitored in-situ and in-operando.

Reflection:

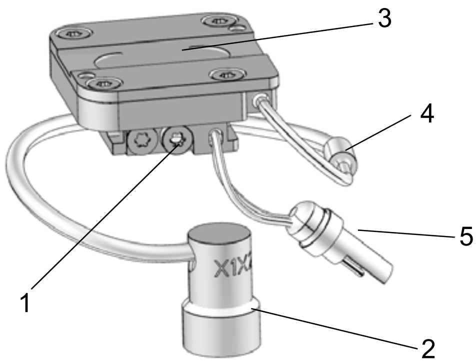


Fig. 13: Battery Sample Holder (Reflection)

1. *Fixing screw for sample holder*
2. *Pt100 connector*
3. *Lid of battery sample holder*
4. *Sheath resistance Pt100*
5. *Electrode cable*

Transmission:

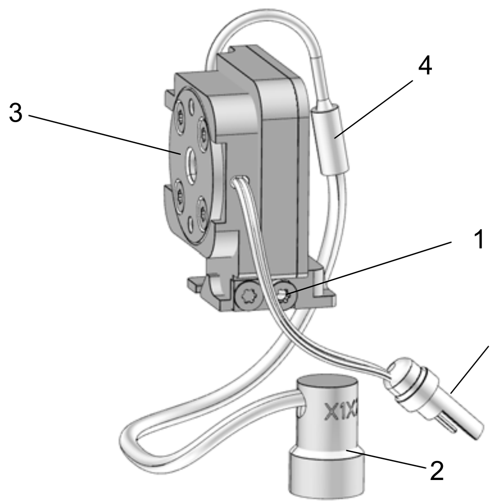


Fig. 14: Battery Sample Holder (Transmission)

1 Fixing screw for sample holder
2 Pt100 connector
3 Lid of battery sample holder
4 Sheath resistance Pt100
5 Electrode cable

2.4 Sample Cooling

TTK 600 Low-Temperature Chamber can be operated with two types of sample cooling equipment, depending on the desired temperature range:

- liquid nitrogen cooling
  temperature range -190 °C to +600 °C
- compressed-air cooling
  temperature range -20 °C to +600 °C

Both types of cooling equipment are connected to TTK 600 via the connector for sample cooling (see chapter 2.3.2) and controlled by CCU 100.
CCU 100 automatically turns off the cooling when it is not needed to save liquid nitrogen/compressed-air.

2.4.1 Liquid Nitrogen Cooling Equipment

The LN2 cooling equipment consists of the following components:

- Low Temperature Equipment for Venturi Nozzle (cat. no. 164578)
- Liquid Nitrogen Container, Dewar 60 L (cat.no.166462)

or

- Liquid Nitrogen Container, Dewar 100 L (cat.no.166463)

Other dewar vessels can be used, provided they have the same specification and opening dimension as the ones offered by Anton Paar GmbH (see chapter 4.2.4 for further information).

Fig. 15: Low Temperature Equipment mounted on TTK 600

1 LN2 connector
2 LN2 supply hose
3 Exhaust hose for evaporated nitrogen

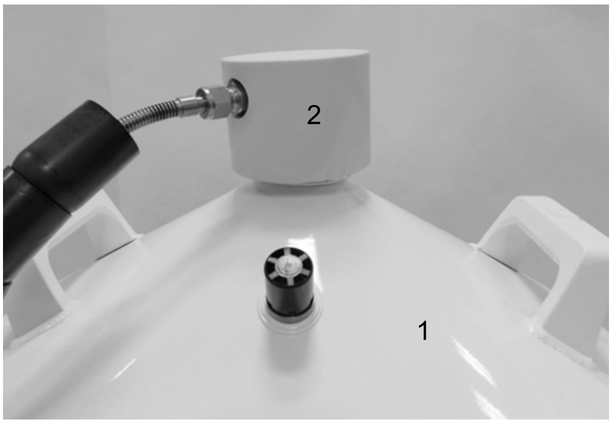


Fig. 16: LN2 Dewar

1 LN2 Dewar vessel
2 LN2 inlet connection

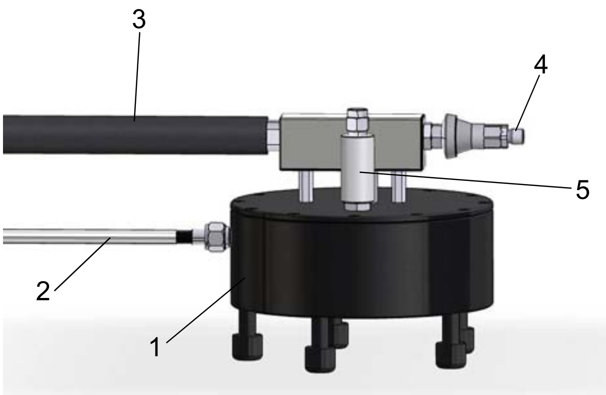


Fig. 17: "Venturi-Nozzle - Safety Box"

1 *"Venturi-Nozzle - Safety Box"*
2 *N2 inlet hose*
3 *N2/Air exhaust hose*
4 *Compressed-air connector*
5 *Overpressure-relieve valve*

On top of the safety box is a Venturi nozzle which creates a vacuum if the compressed air is switched on. This vacuum ensures that liquid nitrogen is sucked from the Dewar vessel through TTK 600 and provides in this regard the cooling power that is needed for low-temperature experiments. The evaporated nitrogen is removed through the exhaust hose of the Low-Temperature Equipment and directed into the Venturi-Nozzle - Safety Box. This box is made as additional safety device for the cooling system.

Liquid nitrogen that may escape from the gas exhaust hose of the Low-Temperature Equipment would accumulate in the Venturi-Nozzle - Safety Box until a certain filling level is reached and a floater inside the box closes the gas outlet of the box to a minimum level. Due to that the flow through the system is decreased which results in a warming of the whole cooling system. For the error case that the outlet hose of the box is blocked, an additional overpressure relief valve that opens at a pressure of 3 bar is integrated in the lid of the box. In this way a safe use of liquid nitrogen is ensured that prevents liquid nitrogen to escape from the system in case of an error or mishandling.

The compressed air that is needed for the activation of the Venturi nozzle is provided by CCU 100. Active cooling is only needed to cool the sample holder to temperatures around room temperature and below.The so-called Cooling Limit (CL) defines the threshold temperature for active cooling. The value can be changed between 20 and 55 °C (see chapter 6.3.1 for further information). If the temperature set point is <CL, CCU 100 opens the compressed-air connection to the Venturi nozzle and enables in this way the cooling of the system. If the sample is heated above the threshold temperature, CCU 100 closes the compressed-air connection and thus stops the supply of LN2 to the cryostat of TTK 600.

DANGER

Risk of injury.
Never open the lid of the Venturi-Nozzle - Safety Box during operation.

For further details see chapter 6.5.1 Controlling the Liquid Nitrogen Cooling.

2.4.2 Compressed-Air Cooling Equipment

The compressed-air cooling equipment consists of the following components:

- Air-Cooling Set TTK 600 (cat.no.165420)

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)

For frequent or long-time cooling of the sample holder to < 10 °C Anton Paar GmbH recommends to use the following component in order to prevent problems with condensed water or ice inside the cooling unit:

- Compressed-air Dryer (cat.no. 81393)

When compressed-air is supplied to the cooling unit, it generates a flow of cold gas which is directed into the cryostat inside the sample chamber and cools the sample holder. The supply of compressed-air to the cooling unit is controlled by CCU 100.

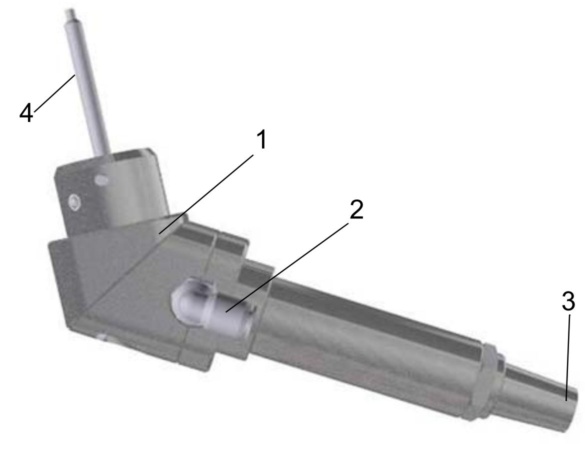


Fig. 18: Compressed-air cooling

1 *Air cooling unit*
2 *Compressed air connector*
3 *Air exhaust*
4 *Cooling nozzle*

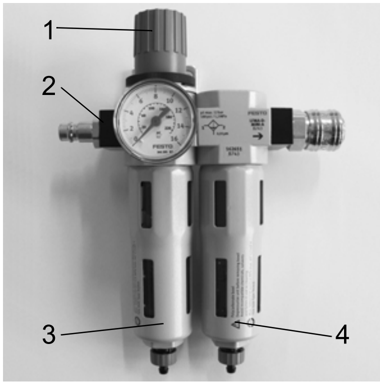


Fig. 19: Air service unit

1 *Air pressure regulator*
2 *Air pressure gauge*
3 *Coarse filter cartridge*
4 *Fine filter cartridge*

For further details see chapter 6.5.2 Controlling the Compressed-Air Cooling.

2.5 CCU 100 Combined Control Unit

CCU 100 is designed to control the sample holder temperature in TTK 600 and to guarantee safe operation of the entire instrument. In order to reach and maintain the desired sample holder temperature, CCU 100 controls the resistance heater inside the sample chamber and the cooling devices (liquid nitrogen cooling equipment or compressed-air cooling unit.)

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 holder temperature based on the measured temperature and the user defined target values. The power module provides variable electrical power for the heater inside TTK 600, depending on the control signal from the micro controller.

2.5.1 CCU 100 Front Panel

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

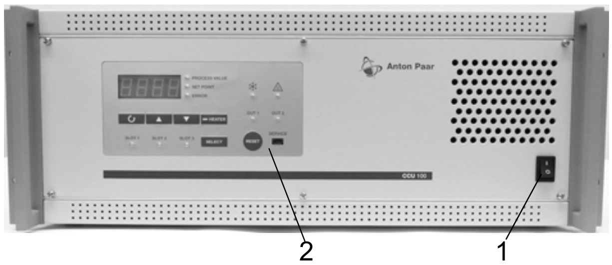


Fig. 20: CCU 100 front panel with control elements

1 *Mains switch*
2 *Display and keypad*

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

2.5.2 CCU 100 Rear Panel

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

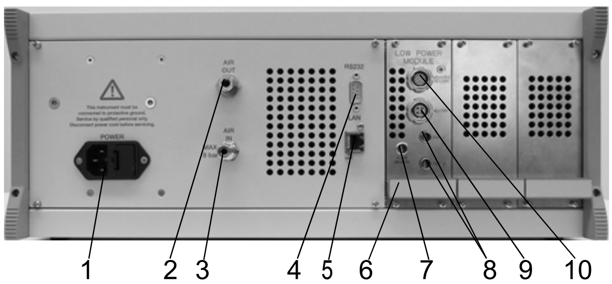


Fig. 21: CCU 100 rear panel

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 TTK 600*
7 *Connector Flow Sensor*
8 *Connector OUT1/2 (optional connectors)*
9 *Connector Heater (not used for TTK 600)*
10 *Connector Sensors/Heater*

2.5.3 Control of the Sample Cooling

Depending on the instrument configuration, CCU 100 controls either the liquid nitrogen cooling (LN2) or the compressed-air cooling equipment.

In order to be able to cool the sample, the compressed-air source has to be connected to the connector AIR IN on the rear panel of CCU 100. If the controlling of the LN2 cooling equipment is active, CCU 100 switches a valve inside which is connected to the AIR OUT connector. By switching this valve the compressed air is directed to the Venturi nozzle on the safety box or not. The Venturi nozzle is creating a vacuum in the hose to the Dewar vessel if the compressed air is on. Due to that liquid nitrogen is transported from the Dewar vessel to the cryostat of TTK 600. If no cooling is required, the compressed air is switched off.

Cooling automatically starts if the set point temperature is below the Cooling Limit (CL) of the instrument. This value can be changed between 20 and 50 °C. For further details on how to change this parameter see chapter 6.3.1.

If the compressed air cooling equipment is used, the compressed air supply is also connected to the AIR IN connector on CCU 100. If cooling is required, a valve inside CCU 100 connects the AIR IN to the AIR OUT connector, which is connected to the air cooling unit on the sample chamber.

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

2.5.4 Overheat Protection

TTK 600 has two devices to prevent overheating of the instrument. The water flow controller (cat.no. 164877) 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 100. If there is not enough water flow, CCU 100 switches to Standby (heater off) mode, the Error LED on the front panel starts to flash and shows an alarm message (E03).

In addition, a thermoswitch is mounted on the sample chamber 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 100 automatically switches to Standby mode (heater off) and shows an alarm (E04).

WARNING

Risk of injury.
When the cooling water supply to the chamber fails, the housing can have a hot surface. In case of an E03 error, turn off the heater by pressing the HEATER button and do not touch TTK 600. Carefully check the temperature of the TTK 600 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 TTK 600 and CCU 100.

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

2.6 Water Flow Controller

The Water Flow Controller (cat.no. 164877) consists of two components to control and monitor the cooling water flow through the TTK 600 sample chamber housing. The pressure regulator is used to reduce the pressure from the cooling water supply and set it to the required value of 2 bar. The water flow monitor is a flow dependent switch which detects if the water flow is below the required minimum value. The flow monitor is connected to CCU 100 and switches off the heating power in case of insufficient water flow.

Fig. 22: Pressure regulator

Fig. 23: Water flow monitor

3 Checking the Supplied Parts

TTK 600 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 noted in Table 3-1.
3. If a part is missing, contact your Anton Paar representative.
4. If a part is damaged, contact the transport company and your Anton Paar representative.

Table 3-1: Supplied Parts

Pcs.	Article Description	Mat. No.
1	**TTK 600 Low Temperature Chamber**	159900
1	Adapter or z-alignment stage	*
8 m	Cooling water hoses with quick coupling connectors (already mounted)	
1	Standard Sample Holder (depth 0.8mm)	159930
1	Gas/vacuum connector	
1	Accessory box (content specified on label)	
1	**CCU 100 Combined Control Unit****	135000
1	Mains cables	
1	Connection cable to TTK 600 sample chamber	
2	Mains fuses	
1	RS232 cable to control PC	
and/or		
1	**Low-Power Module CCU**	176730
1	**Flow Controller CCU**	164877
1	Pressure regulator	
1	Water flow monitor	
	with Liquid-Nitrogen Cooling Equipment	
1	Low-temperature equipment for Venturi nozzle	164578
1	Liquid nitrogen container, Dewar 60L (optional)	166462
1	Liquid nitrogen container, Dewar 100L (optional)	166463
	with Compressed-air Cooling Equipment	
1	Air cooling set TTK 600	165420
1	Air service unit (optional)	6931
1	Compressed-air dryer (optional)	81393
1	**Instruction manual English**	
1	**Software manual Nambicon**	
1	**USB flash drive with Nambicon**	

** The catalog number of the adapter/alignment stage depends on the type of diffractometer.*

*** 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.*

4 Installation

This chapter describes the installation of TTK 600 on the X-ray diffractometer.

4.1 Installation Overview

The following schematic diagrams show how to set up TTK 600 with liquid nitrogen cooling equipment and with compressed-air cooling equipment. The connectors referenced with numbers are described in the installation requirements following the schematic diagrams.

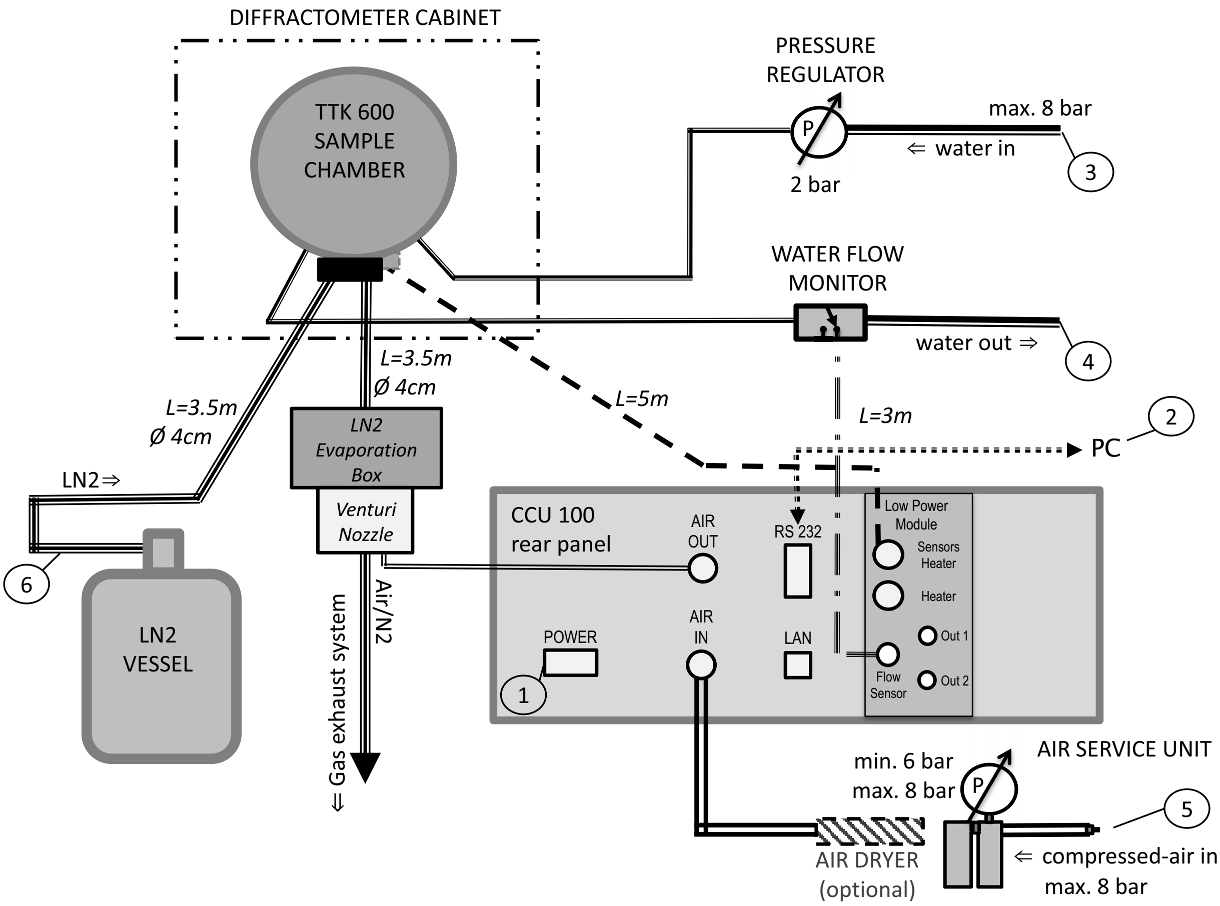


Fig. 24: Installation diagram for TTK 600 with liquid nitrogen cooling equipment.

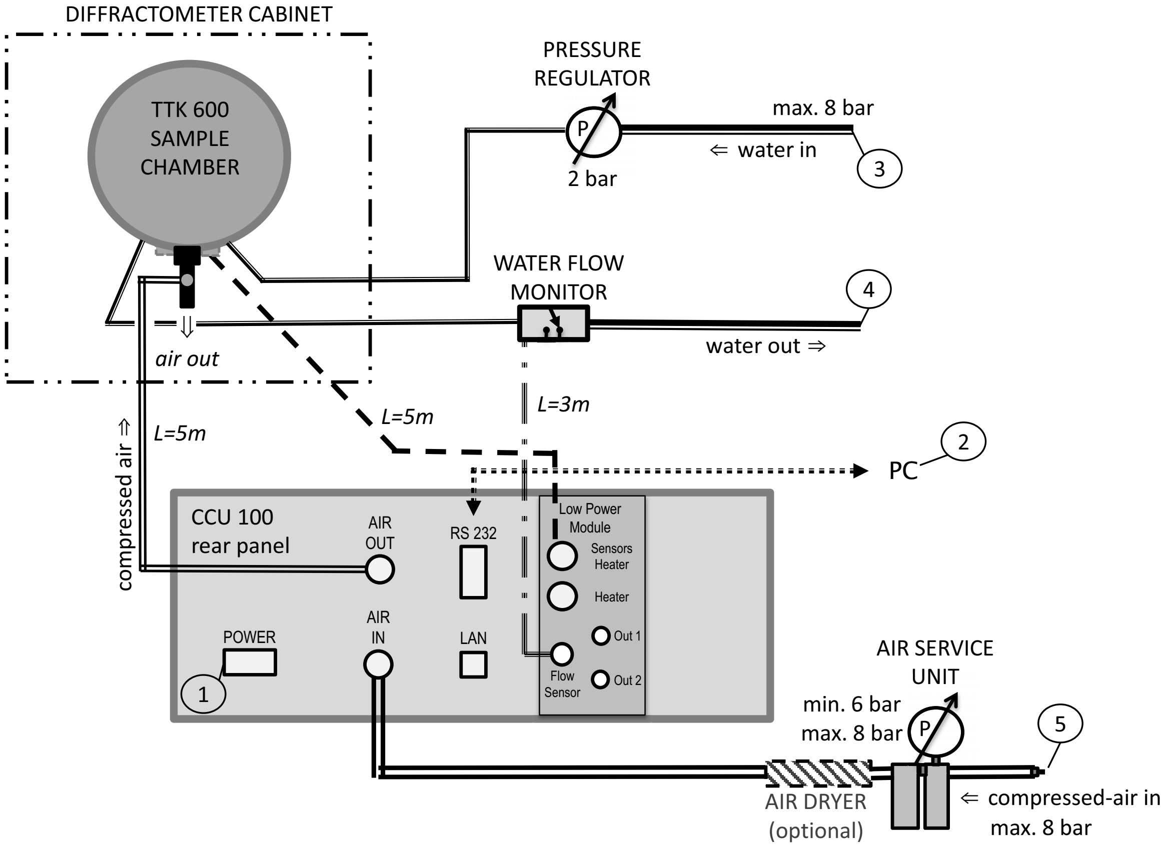


Fig. 25: *Installation diagram for TTK 600 with compressed-air cooling equipment.*

4.2 Installation Requirements

The setup location and surroundings should meet the requirements of a typical laboratory.

Allow the equipment to reach ambient temperature before installation. This is very important if the equipment has been stored or transported at lower temperatures.

To ensure temperature stability and trouble-free measurement never locate your instrument:

- next to a heating facility
- near an air conditioning, ventilation system or an open window
- in direct sunlight

Keep the instrument away from magnetic fields.

Read the Safety Instructions in chapter 1.

Find all Technical Data in appendix A.

4.2.1 X-Ray Diffractometer Requirements

- Radiation safety enclosure

WARNING

Risk of injury.
Operation of TTK 600 Low-Temperature Chamber on an X-ray diffractometer without radiation safety enclosure can cause exposure to ionizing radiation.
Exposure to ionizing radiation above natural dose can lead to skin and eye damage and can have carcinogenic and mutagenic effects in your body.

- Vertical θ-θ goniometer
  Operation on θ-2θ goniometer is possible, but during low temperature operation the LN2 hoses get stiff and rotatability of the sample chamber may be limited.
- Clearance around goniometer axis:
  at least ∅ 125 mm
  => see also appendix A
- Feedthroughs for two LN2 hoses with ∅ 40 mm each, two water hoses with 4x6 mm, two electrical cables with ∅ 5 mm and a vacuum hose with

∅ 25 mm.

- A suitable mechanical adapter between the sample chamber and the goniometer interface is required.

4.2.2 Electrical Requirements

Mains voltage: AC 100-240 V
Mains frequency: 50 ... 60 Hz.

CCU 100 mains connector to (1): IEC 60320 C13

CCU 100 mains fuses: 2 x T 6,3 A H 5x20 mm (ceramic tube)

Remote control interface: RS 232
RS 232 connector on CCU 100 (2): D-Sub DE-9 (male)
Cable type: null modem

4.2.3 Cooling Water Requirements

Temperature: 15 to 25 °C
and above the dew point of the ambient air

Supply pressure: 2 to 8 bar

Water drain: pressureless

Water flow: 0.7 to 1.5 l/min

Water quality: specific conductivity:
10 to 250 µS/cm
hardness: 0.2 to 0.7 mMol/l (1 to 4 °dH)
acidity: 7.5 to 9.5 pH
filtered/filter size: max. 50 µm
=> no de-ionized or plain distilled water
=> no anti-freeze additives

Connectors (3,4): suitable for water hose ID/OD 8/14 mm

4.2.4 Requirements for Liquid Nitrogen Cooling

- Liquid nitrogen container (Dewar) with
  - a self-evaporation rate of at least 0.2 L/d.
  - compliance to the local safety regulations
- Required opening dimension of the Dewar: DN50KF (6)
- compressed air requirements for the liquid nitrogen cooling equipment:
  > 70 L/min @ > 2.5 bar rel.

TIP: *Anton Paar GmbH offers suitable Dewars with 60 L and 100 L volume (see appendix B).*

WARNING

Risk of injury.
Accumulation of nitrogen gas in the room can lead to **asphyxiation** due to lack of oxygen.
The laboratory must have sufficient ventilation or a gas exhaust system to dissipate 4 m³/h nitrogen gas.

4.2.5 Requirements for Compressed-air Cooling

Pressure: 6 to 8 bar rel.

Flow rate: 120 l/min at 7 bar

pressure dew point (@ 7 bar):
for operating temperature ≥ +5 °C: < +25 °C
for operating temperature < +5 °C: < +10 °C

Purity (acc. DIN 8573-1): solid particle class 3 or better oil class 2 or better

Connector of air-supply (5): pneumatic coupling DN 7.2 (Rectus™ series 26 KA)

TIP: *Anton Paar GmbH recommends to use the Air Service Unit to meet the requirements (see appendix B).*

TIP: *Anton Paar GmbH offers a suitable compressed-air dryer for reducing the dew point of the compressed air (see appendix B).*

4.2.6 Vacuum Equipment Requirements (optional)

Vacuum inside the TTK 600 sample chamber is required for cooling the sample holder below -150 °C or to heat it above +450 °C.

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: DN25 ISO-KF

Vacuum capable tubing with minimum ID 16 mm.

TIP: *Anton Paar GmbH offers a suitable Vacuum Equipment (see appendix B).*

4.3 Mounting the Adapter on the Sample Chamber

> **NOTICE**
> If TTK 600 has been ordered together with the rigid adapter or motorized z-alignment stage, the adapter/z-alignment stage is mounted on delivery.

If it is necessary to mount a motorized z-alignment stage, see the mounting instructions delivered with the alignment stage. The adapter to the chamber is mounted on the z-alignment stage on delivery.

In order to mount a rigid adapter, proceed as follows:

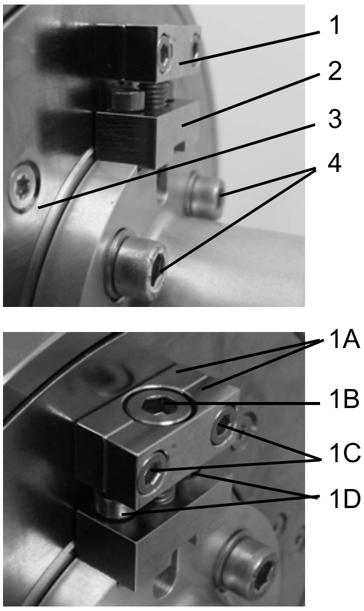


Fig. 26: Mounting the rigid adapter stage on the sample chamber

1. Loosen the two Allen screws (1C) and remove the alignment equipment (1) from the base-plate (3) of TTK 600.
2. If required, clean the O-ring on the adapter flange and lightly coat the O-ring with vacuum grease.
3. Use the four Allen screws (4) to mount the adapter on the TTK 600 base-plate (3).
4. Fasten the lower part of the alignment equipment (2) with the two screws (1D).
5. Fasten the two upper parts (1A) of the alignment equipment to the TTK 600 base-plate using the two screws (1C) in the front of the upper part. Make sure that the alignment screw (1B) is surrounded by the two parts (1A).

> **NOTICE**
> The rigid height alignment tool for the rigid adapter is optional (mat.no. 166148) and only needed for diffractometers without a z-stage.

Adapter for Battery Sample Holders:

When changing from standard sample holders to the battery sample holders, ensure that the guiding pins of the TTK 600 are aligned in the correct position on the adapter. There are different positions for standard sample holders, the reflection battery sample holder, and the transmission battery sample holder.

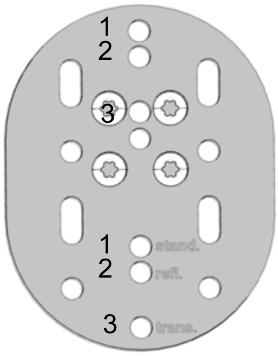


Fig. 27: Adapter for TTK 600

1 Mounting position for standard sample holders
2 Mounting position for battery sample holder reflection
3 Mounting position for battery sample holder transmission

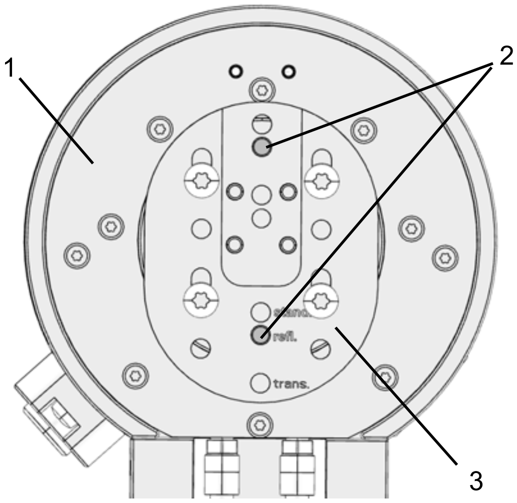


Fig. 28: Mounting the adapter stage on the chamber for use with different sample holders

1 TTK 600 back side
2 Guiding pins
3 Adapter for TTK 600

4.4 Mounting the Chamber on the Goniometer

The TTK 600 (with adapter) is mounted on the goniometer instead of the standard (ambient) sample holder. Instructions for mounting the chamber are given in the instruction manual for your diffractometer.

NOTICE

- Before you take off the standard (ambient) sample holder make sure that the X-ray beam is properly aligned (see the diffractometer instruction manual for detailed information). Good beam alignment is important, because TTK 600 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 inside TTK 600 is as parallel as possible to the Θ-zero line of the goniometer.

4.5 Installing CCU 100

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 sample chamber or the CCU 100.

Connect the electrical cable to the sample chamber as follows:

1. Feed the cable through the appropriate port of the diffractometer cabinet as described in the diffractometer instruction manual.
2. Connect the cable for the heater/temperature sensor to the connection cable of TTK 600 (see Fig. 5, 4) on the sample chamber.

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

- Plug the cable for the heater/temperature sensor to the connector Sensors/Heater (see Fig. 21(10)).
- Make sure the mains switch on CCU 100 is OFF and connect the mains power cable to the POWER connector (see appendix 21 (1)).

WARNING

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

4.6 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. 21(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

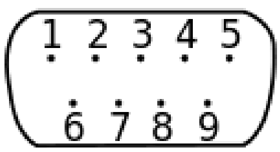


Fig. 29: RS 232 pins

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

4.7 Installing the Cooling Water Circuit

Prepare the following items:

- the two additional hoses (ID/OD 4x6 mm) with quick couplings supplied with the sample chamber
- the pressure regulator and the water flow monitor from the Water Flow Controller set (see Fig. 22 and Fig. 23).
- sufficient suitable water hose ID/OD 8x14 mm to connect the pressure regulator and the flow monitor to the water source and drain

NOTICE

- Secure all hose connections against unintentional disconnection with a suitable nut or clamp.
- Connect the water circuit to a pressureless water drain, otherwise the water flow monitor will not work properly.

The flow direction of the water through the TTK 600 chamber housing does not matter.

Install the cooling water circuit as follows:

1. Feed the two thin hoses with quick couplings through a port in the diffractometer cabinet so that the couplings are inside the cabinet.
2. Connect one of the hoses to the corresponding connector on the water pressure regulator and secure it with the nut.
3. Connect the other hose to the corresponding connector on the water flow monitor and secure it with the nut.
4. Connect the outlet of the water flow monitor to the water drain with the 8x14 mm hose.
   Secure the hose on both sides with a suitable clamp.
5. Connect the inlet of the pressure regulator to a suitable water tap or water chiller with the 8x14 mm hose.
   Secure the hose on both sides with a suitable clamp.
   Observe the cooling water requirements given in chapter 4.2.3.
6. Connect the quick couplings inside the diffractometer cabinet to the corresponding couplings on the sample chamber.
7. Connect the cable of the water flow monitor to the WATER FLOW CONTROL connector on the rear panel of CCU 100.

TIP: *The couplings tightly seal the hoses. Nevertheless, we recommend to turn off the water supply if you don't use TTK 600 for a longer period of time.*

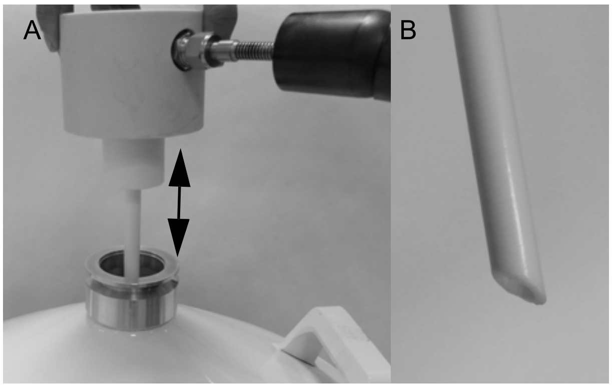
4.8 Installing the Liquid Nitrogen Equipment

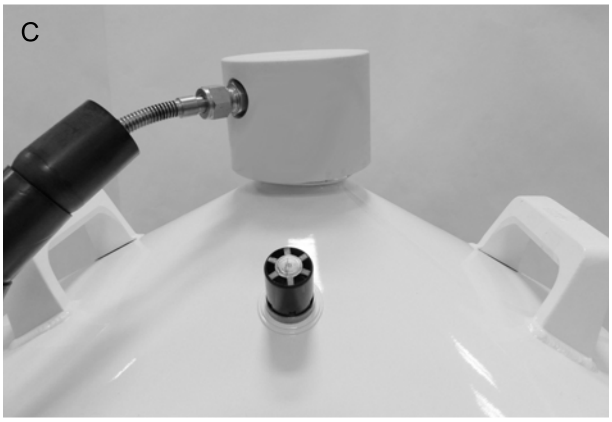
DANGER

Risk of injury.
Liquid nitrogen is extremely cold and can cause freezing of tissue and cryogenic burns, similar to frostbite, to eyes or skin upon contact.
Wear protective clothing (eye and face protection, thermally insulating gloves) when (dis)connecting the Low Temperature Equipment to (from) the liquid nitrogen container.
Read and observe the safety instructions given in appendix E and in the instruction manual of the liquid nitrogen container.

Install the liquid nitrogen (LN2) cooling equipment as follows:

1. Feed the LN2 supply hoses through a suitable port in the diffractometer cabinet so that the LN2 connector is inside the cabinet (see Fig. 15).
2. Place the LN2 inlet connection in the Dewar (C in the following figure). If the system is not ordered with the Dewars provided by Anton Paar GmbH, the Teflon tube of the inlet connection which is placed in the Dewar has to be cut to the appropriate length. To cut the length of the tube proceed as follows:
   - place the LN2 inlet connection in the Dewar (A)
   - measure the distance from the top of the Dewar to the bottom of the LN2 inlet connection (indicated by the arrow in A of the following figure); this is the length that has to be shortened
   - cut the tube with the delivered saw. Please make sure to make the cut in an angle of approximately 45° (see B in the following figure).





3. Make sure that there are no electronic devices or components underneath the parts of the LN2 exhaust hose that are not insulated. Ice can form on these parts and water can drop down. If necessary, add insulation material.
4. Connect the exhaust hose for evaporated nitrogen to the Venturi-Nozzle - Safety Box at the inlet (2) in Fig. 17. Connect the N2/Air exhaust hose of the box (3) in Fig. 17 with the exhaust gas hose and direct it to a suitable gas exhaust system. Connect the compressed air connector on the box (4) in Fig. 17 to the connector AIR OUT on the rear side of CCU 100 (see Fig. 21).
5. Connect the compressed air supply hose to the AIR IN connector on the rear side of CCU 100 (see Fig. 21).

> **WARNING**
>
> The liquid nitrogen cooling equipment of TTK 600 can release up to 5 m³ nitrogen gas per hour, which can lead to reduced oxygen concentration in the room. Make sure that the room is sufficiently ventilated or that the gas is removed through the exhaust gas hose to a gas exhaust system.

6. To connect the Low Temperature Equipment to the TTK 600 sample chamber proceed as follows:

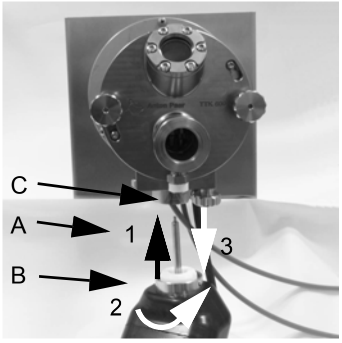


Fig. 30: Low Temperature Equipment

1. Insert the nozzle (A) of the LN2 connector (B) into the cooling device connector (C) on the sample chamber.
2. Rotate the LN2 connector (B) with a gentle push upwards until it fully slides into the cooling device connector (C).
3. Turn the LN2 connector (B) counter clockwise so that it is parallel to the goniometer to lock it on the cooling device connector (C).

After you have put CCU 100 in operation, check if the CCU 100 configuration is set to *Cooling Type "LN2"* as described in section 6.3.1.

4.9 Installing the Air-Cooling Equipment

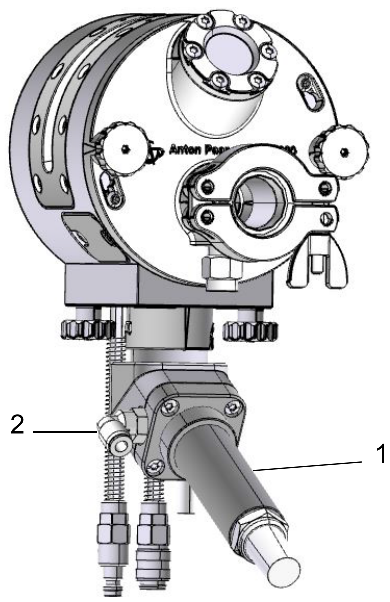


Fig. 31: Air-cooling equipment

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

1. Take the air-cooling unit (1) so that the hose coupling (2) is on the left side.
2. Turn the cooling unit clockwise to 8 o'clock and insert it from the bottom into the cooling device connector (see Fig. 3 - 3).
3. Take care that the bolt inside the cooling unit slides into the slit in the connector.
4. Turn the cooling unit counter-clockwise so that the hose coupling is again on the left side to lock it on the connector.

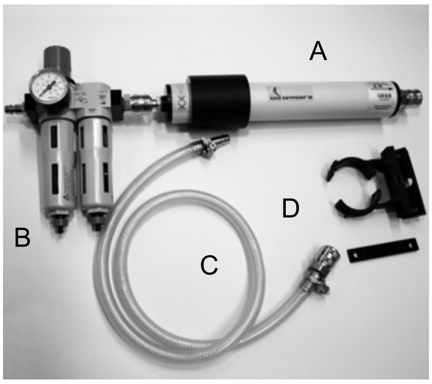
In order to connect the compressed-air hose (3) supplied with the instrument, proceed as follows:

5. Insert and push the compressed-air hose as far as possible into the fitting and check the tightness by applying compressed air.
6. Feed the air hose through the diffractometer enclosure and connect it to the AIR OUT connector on the rear panel of CCU 100.

> **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. If a compressed-air dryer (A) is used, combine the air dryer and the air service unit (B). This can be done either directly or with the short hose (C) supplied with the instrument.

> **NOTICE**
> Do not mount the compressed-air dryer directly on the air service unit without support or wall mounting. Mounting lugs (D) are supplied with the compressed-air dryer.

3. Connect the Air Service Unit to the compressed-air supply of the laboratory.
4. Connect the male connector of the pneumatic hose to the outlet of the air service unit or the compressed-air dryer, respectively.

> **CAUTION**
>
> ***Risk of damage.***
> Do not apply more than 8 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.

5. Set the pressure on the Air-Service Unit to 7 bar.

TIP: *7 bar is the standard value. The pressure can be varied between 6 to 8 bar. Higher pressure provides more cooling power, lower pressure less air consumption.*

After you have put CCU 100 in operation, change the CCU 100 configuration to Cooling Type "CAir" as described in section 6.3.1.

4.10 Cooling Mode Switching Valve (optional)

The cooling mode switching valve is optionally available and allows to easily switch between compressed-air cooling and liquid nitrogen cooling without the need of connecting or disconnecting any hoses at the backside of CCU 100.

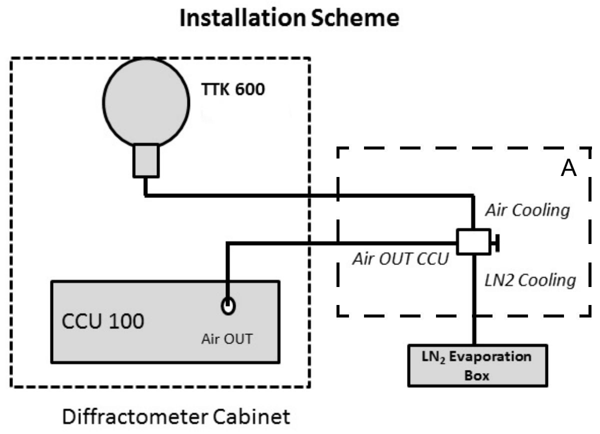


Fig. 32: Installation diagram

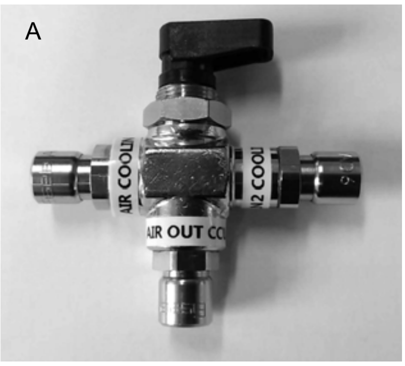


Fig. 33: Cooling mode switching valve

The valve is delivered with 4 m additional hose.

Install the cooling mode switching valve as follows:

1. Connect the AIR OUT of the cooling mode switching valve to the connector AIR OUT on the rear side of CCU 100.
2. Install the Liquid Nitrogen Equipment as described in chapter 4.8. In step 4 connect the compressed air connector on the box (4) in Fig. 17 to the LN2 COOLING connector of the cooling mode switching valve instead of connection to the CCU 100.
3. Install the Air Cooling equipment as described in chapter 4.9. In step 6 connect the compressed-air hose to the AIR COOLING connector of the cooling mode switching valve instead of connection to the CCU 100.
4. In order to use Air Cooling or Liquid Nitrogen switch the cooling mode switching valve to the corresponding position and install the air-cooling equipment (Fig. 31) or the Low Temperature Equipment (Fig. 30) to the chamber (5 on Fig. 5).

4.11 Mounting the Gas/Vacuum Connector

The gas/vacuum connector allows to supply and extract gas from the TTK 600 and to apply vacuum to the chamber. The gas/vacuum connector is mounted to the lid of the TTK 600.

4.11.1 Installing the Gas Supply (optional)

Depending on the measuring problem, the TTK 600 can be operated with different gas atmospheres, e.g. air, inert gas or nitrogen.

> **DANGER**
>
> ***Risk of damage.***
> Poisonous, explosive or corrosive gases must not be used.

Apart from the explicitly specified gases, other non-hazardous gases can be used, provided the gas itself and possible reaction products with the sample do not damage the instrument. A list of chamber materials is given in appendix A.

Gases can be supplied to and extracted from the chamber by using the gas/vacuum connector and a blind cover delivered with the chamber (see Fig. 34). The gas/vacuum connector is equipped with two hoses for gas inlet and outlet. The directions of in/outlet are marked on the gas/vacuum connector. Each hose contains a quick coupling connector with a valve to open/close the gas inlet/outlet.

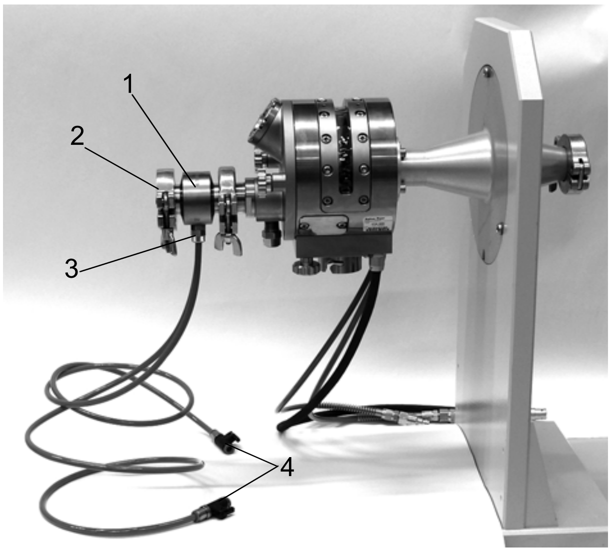


Fig. 34: Gas supply

1 *Gas/vacuum connector*
2 *Blind cover*
3 *In/out gas hoses*
4 *Quick coupling connectors with valves*

The TTK 600 sample chamber can only hold an overpressure of approx. 2 bar. At a pressure of 3 bar the overpressure relieve valve on the lid of the instrument opens. Use a suitable pressure reducer/ regulator with the pressure range of 0-2 bar on the gas supply to TTK 600.

NOTICE
Anton Paar GmbH does not provide components for gas supply systems.

CCU 100 Combined Control Unit has been configured for operation with stationary gas filling. Gas flow through the chamber will have negative effects on temperature control and the accuracy of sample temperature measurement.

4.11.2 Installing the Vacuum Equipment (optional)

The vacuum equipment can be mounted directly to the flange (DN25 KF) on the lid of the TTK 600 in case no gas supply is required (see Fig. 35).

Detailed information for the installation of the vacuum equipment are supplied in the manual of the vacuum equipment.

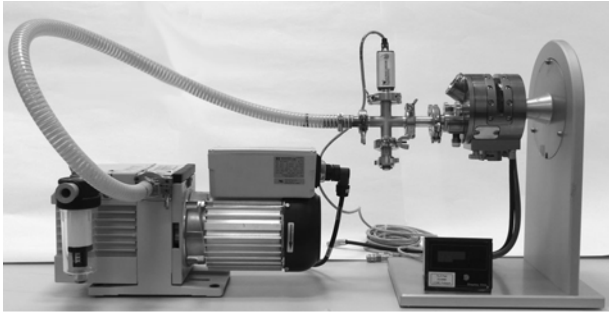


Fig. 35: Anton Paar GmbH standard vacuum equipment

TIP: *Make sure that both the housing's sealing surface and the O-ring of the lid are clean and slightly coated with vacuum grease.*

If oxygen-free sample environment is needed, connect the vacuum system to the gas/vacuum connector instead of the blind plug. Between the gas/ vacuum connector and the cross piece a valve MUST be used, to protect the vacuum gauge and the pump from pressure (see Fig. 36).

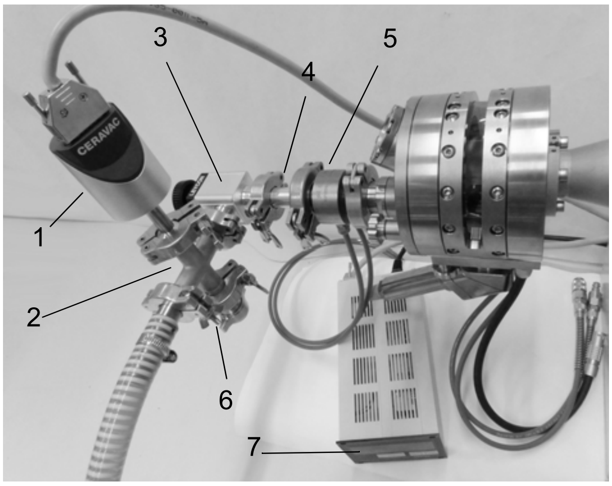


Fig. 36: Gas/vacuum supply

1 *Pirani tube*
2 *Cross piece DN16*
3 *Corner valve*
4 *Reducer DN25/DN16*
5 *Gas/vacuum connector*
6 *Venting valve*
7 *Display for Pirani tube*

NOTICE
The corner valve is not included in the vacuum equipment but can be ordered as an accessory (refer to appendix D).

To protect the vacuum equipment from pressure a valve MUST be installed between gas/vacuum connector and the vacuum equipment. BEFORE opening the gas inlet and applying pressure the valve has to be closed as damage of the vacuum parts will occur.

Anton Paar GmbH does not provide components for gas supply systems.

4.11.3 Installing the Gas/Vacuum system for using the Antechamber

As the antechamber is used for air-sensitive samples in most cases a combination of gas purge and evacuation of the chamber will be required. The vacuum equipment is mounted to the antechamber via the vacuum flange of the antechamber. A ball valve between chamber and vacuum parts is necessary to protect the vacuum system from overpressure.

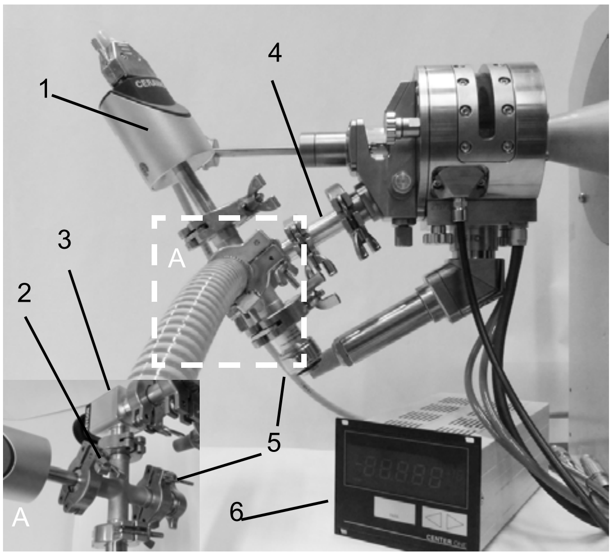


Fig. 37: Gas/vacuum supply for Antechamber

1 Pirani tube
2 Cross piece DN16
3 Corner valve
4 Reducer DN25/DN16
5 Venting valve
6 Display for Pirani tube

NOTICE
The corner valve is not included in the vacuum equipment but can be ordered as an accessory (refer to appendix D).

To protect the vacuum equipment from pressure a valve MUST be installed between gas/vacuum connector and the vacuum equipment. BEFORE opening the gas inlet and applying pressure the valve has to be closed as damage of the vacuum parts will occur.

Anton Paar GmbH does not provide components for gas supply systems.

Gas is supplied and extracted via two gas connectors on both sides of the chamber. The gas connectors are mounted instead of the blind covers (refer to chapter 4.12 how to exchange the blind cover with the gas connector). Add a suitable pressure regulator (from 0 - 2 bar) in the gas line to avoid pressures inside the chamber above the limit of 2 bar rel. Additionally valves have to be installed on the supply and exhaust lines to close the hoses during evacuation.

NOTICE
Anton Paar GmbH does not provide components for gas supply systems.
CCU 100 Combined Control Unit has been configured for operation with stationary gas filling. Gas flow through the chamber will have negative effects on temperature control and the accuracy of sample temperature measurement.

4.12 Convection Heater

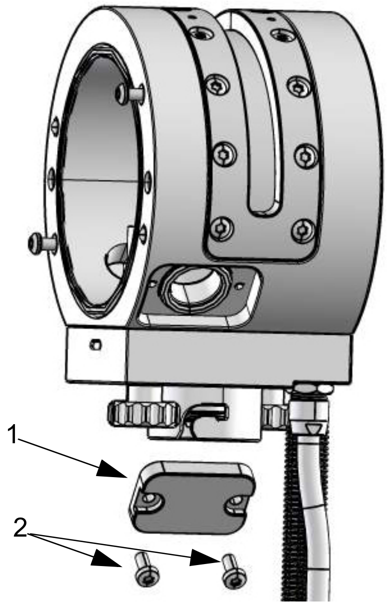
When using the transmission sample holder or the capillary sample holder an additional convection heater is required for minimizing temperature deviations between heater and sample when using a Kapton foil.

NOTICE
Risk of damage.
Use of the Kapton foil for the capillary heating environment limits the max. temperature to 230 °C

For installation of the convection heater proceed as follows:

1. Dismount the blind cover on the right side of the housing (unscrew the two screws)

2. Replace the blind cover with the gas connector. Do not forget to place the O-rings.



1 Blind cover
2 Screws

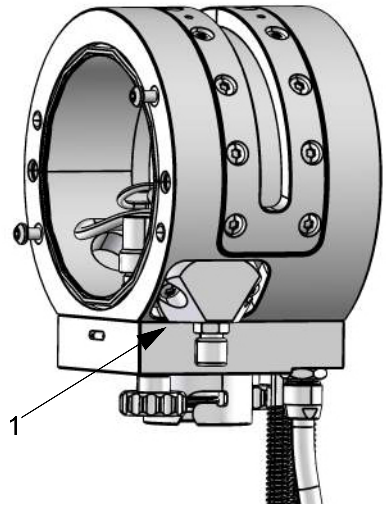


Fig. 38: TTK 600 with Gas Connector

1 Gas Connector

3. Connect the gas hose leading from the sample holder to the gas connector (OD 4 mm; black; shorten the hose to an appropriate lengths; 5 in Fig. 57)
4. Connect the gas hose from the gas line to the gas connector (OD 6 mm; black)

NOTICE
A separate flow controller has to be mounted in this gas line in order to be able to adjust the gas flow accordingly. This item is not delivered by Anton Paar GmbH. It is recommended to use a flow controller that allows to adjust the flow between 0.5 and 3 L/min.

Both hoses are delivered with the transmission or capillary sample holder.

For uninstalling the connection heater repeat the above mentioned points in reverse order.

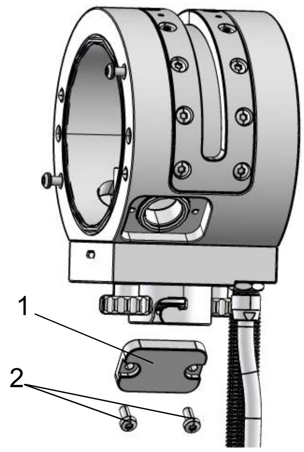
Do not forget to place the O-rings.

4.13 Mounting the Electrode Connector and the Connector Box

For the use of the battery sample holder, it is necessary to mount the electrode connector and the connector box to TTK 600 in order to be able to put an electrical load on the battery materials under investigation.

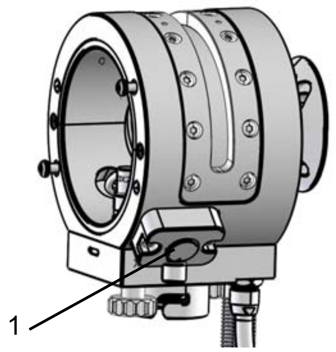
For the installation of these parts, proceed as follows:

1. Dismount the blind cover on the right side of the housing (unscrew the two screws).



1 Blind cover
2 Screws

2. Replace the blind cover with the electrode connector. Do not forget to place the O-rings.



1 Electrode connector

3. Connect the plug of the connector box to the electrode connector on TTK 600.

Fig. 39: Connector box for battery sample holder

The connector box has two banana-type connectors for the connection of an external electrical measuring device.

5 Putting TTK 600 into Operation

5.1 Alignment of the Standard Sample Holder

Proper alignment of the TTK 600 Low-Temperature 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 center plane of the goniometer.

An alignment block with a small slit for accurate chamber alignment is available as an option (mat. no. 162029)

The alignment of the TTK 600 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), on the diffractometer type and on the used sample holder.

Fig. 5-1: Alignment sample holder

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

It is important that the X-ray beam height and the detector are properly aligned before you start to align TTK 600, because the sample chamber is aligned relative to the X-ray beam (Θ-zero line).

Perform the alignment of TTK 600 at room temperature.

Alignment is best done in two steps:

1. Coarse alignment with the upper edge of the empty sample holder.
2. Fine alignment with the alignment slit in the alignment block.

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.

5.1.1 Height Alignment with Rigid Adapter

The height (z-position) of the sample chamber relative to the X-ray beam can be adjusted with the alignment equipment mounted on the base-plate of TTK 600.

The alignment sample holder is available as an option (mat. no. 162029).

Fig. 40: Rigid Adapter

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 to move the chamber upwards.

Turn anti-clockwise to move the chamber downwards.

5.1.2 Height Alignment with Motorized Alignment Stage

Motorized alignment stages are remote controlled. The height (z-position) of TTK 600 can be adjusted from outside the diffractometer enclosure.

- The sample height can be aligned while the X-ray beam is on.
- The sample height can be re-aligned during the experiment at any temperature.
- The alignment stage offers an accuracy of < 5 µm.
- Digital display of the chamber position is possible.

For information about the operation of the motorized alignment stage, read the instruction manual of the alignment stage.

5.1.3 Alignment of the Chamber Rotation

The TTK 600 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).

The procedure for rotating TTK 600 relative to the goniometer depends on the type of diffractometer. Refer to the instruction manual for the diffractometer for details.

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 instruction manual for the diffractometer before you start the alignment.

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. Therefore start the alignment of the chamber rotation with the standard sample holder mounted in the chamber (coarse alignment).

When the chamber is roughly aligned parallel to the Θ-zero line, replace the standard sample holder with the alignment block and repeat the procedure.

5.2 Alignment of the Standard Sample Holder

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 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 $I = ½ \cdot (I_{max} - I_{bg})$, which means that the upper edge of the alignment block bisects the X-ray beam.

If you have mounted the alignment block:

- 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 center axis of the goniometer.*
For Θ-2Θ diffractometers, rotate the chamber around the center 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 = 2*Θ* = 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 a tabulated value.

> **NOTICE**
> Remove the alignment slit before starting a measurement. Heating the alignment slit will destroy it.

5.3 Preparing the Cooling Equipment

5.3.1 Preparing the Liquid-Nitrogen Cooling

> **WARNING**
>
> ***Cold Surface***
> Liquefied nitrogen can cause freezing of tissue, or cryogenic burns, similar to frostbite to eyes or skin upon contact. Always wear protective clothing (eye and face protection, thermally insulating gloves) when handling the Low Temperature Equipment or the liquid nitrogen container.
> Follow all instructions given in appendix E and in the instruction manual for the liquid nitrogen container.

Fig. 41 shows a picture of the Liquid Nitrogen Equipment (LTE) mounted on the liquid nitrogen container (Dewar).

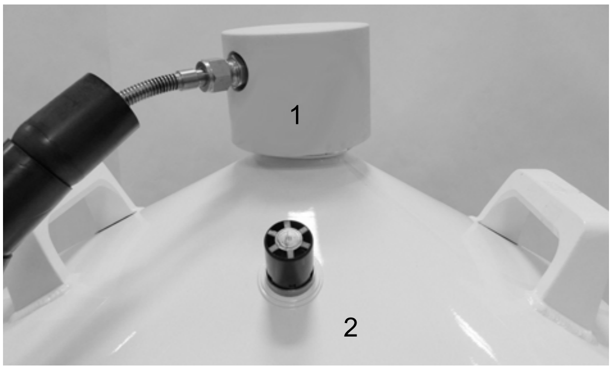


Fig. 41: LTE on Dewar

1 Low-Temperature Equipment (LTE)
2 Liquid Nitrogen Container (Dewar)

Read the instruction manual for the Dewar before you carry out the following steps. Proceed as follows to put the liquid nitrogen equipment in operation:

1. Remove the LTE (or the plug) from the Dewar.
2. Fill the Dewar as described in the instruction manual for the Dewar.
3. Place the LTE in the Dewar.
4. Open the compressed-air source.
5. Set the pressure regulator to a pressure of > 2.5 bar.

> **NOTICE**
> Always adjust the length of the cooling period to the filling level of the Dewar. Do not operate the cooling equipment with an empty Dewar. This results in condensation of water inside the Dewar.

5.3.2 Preparing the Compressed-Air Cooling

Prepare the compressed-air cooling as follows:

1. Open the compressed-air source.
2. Set the pressure regulator to 6 bar.

TIP: *The cooling power and the air consumption increase with increasing pressure. Adjust the air pressure within the specified limits depending on your experimental needs.*

5.4 Checking the Instrument Condition

Before you start operation:

- Make sure that TTK 600 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 100 Temperature 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.

When you are using the liquid nitrogen cooling equipment:

WARNING

Cold surface
Liquefied nitrogen can cause freezing of tissue, or cryogenic burns, similar to frostbite to eyes or skin upon contact. Always wear protective clothing (eye and face protection, thermally insulating gloves) when handling the Low Temperature Equipment or the liquid nitrogen container.
Follow all instructions given in appendix E and in the instruction manual for the liquid nitrogen container.

- Make sure the exhaust hose of the Safety Box is connected to an exhaust gas system or ends in a sufficiently ventilated area.

WARNING

Risk of injury.
Release of large amounts of nitrogen can lower the oxygen concentration in the room and lead to asphyxiation.

- Check the filling level of the Dewar.

If you are using the compressed-air cooling equipment:

- Make sure that the air cooling unit is fixed on the TTK 600 cooling device connector and that the compressed-air hoses are attached firmly to the air cooling unit and CCU 100.

5.5 Turning on the Instrument

If possible, switch on CCU 100 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 100.

The micro controller of 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
  - 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. Check whether the correct Cooling Type parameter is set, as described in chapter 6.3:
   LN2…for liquid nitrogen cooling
   CAir… for compressed-air cooling
4. 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 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 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 100 contains the keypad, the status LEDs and the display which are described in the following section.

Fig. 42: Front panel of CCU 100

6.2 Keypad

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

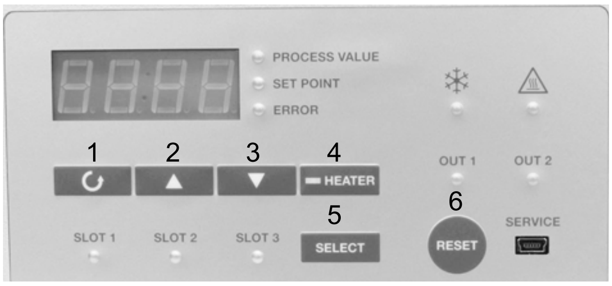


Fig. 43: Keypad of CCU 100

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. Long: selects a slot.
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. 44: 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.

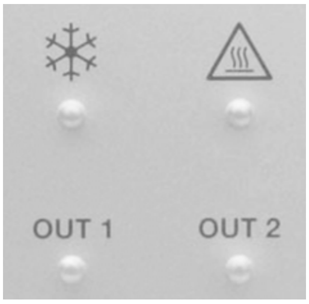


Fig. 45: LEDs for status of CCU 100

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

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 lid of the instrument is not mounted. After mounting the lid, the LED stops blinking and the instrument is ready to use.

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

Fig. 46: 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 holder of TTK 600.

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 100

The complete navigation diagram is shown below:

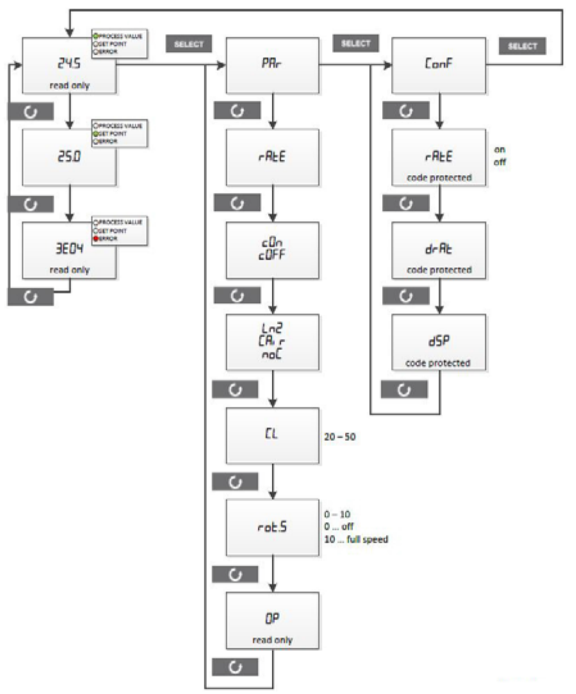


Fig. 47: Navigation diagram

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	

Parameter	Description	Note
Ln2 CAir noC	Toggle between the CCU 100 configurations for LN2 cooling, compressed-air cooling and no cooling	
CL	Temperature below which cooling is permanently activated. Values can be set between 20 °C and 50 °C.	Default value: 24 °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 TTK 600
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 - 300 °C	This parameter is password protected.

Parameter	Description	Note
dSP	Allows changing of the default set point (25 °C) for all three types of cooling modes 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 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 or cold surface.
There can be dangerously hot or cold surfaces inside the sample chamber even if the heater has been switched off. Always check on the display of CCU 100 that the sample holder temperature is below 50 °C before you open the sample chamber and touch any parts inside.

6.5 Controlling the Cooling Device

The temperature of the sample holder is controlled with a combination of cooling it with the cooling device and (counter) heating with the electrical heater. The cooling device is automatically turned on and off by CCU 100 whenever it is needed. At the same time CCU 100 controls the power of the heater to keep the sample holder on the defined temperature. Active cooling from high temperatures (> 300 °C) is not activated until the sample temperature reaches 300 °C. Going below this limit the CCU 100 automatically switches on the used cooling equipment.

6.5.1 Controlling the Liquid Nitrogen Cooling

LN2 cooling is turned ON if the temperature set point is below the Cooling Limit (CL). The COOLING LED (see chapter 6.2.1) indicates that CCU 100 supplies liquid nitrogen to the sample chamber and that the LN2 cooling is active.

The LN2 cooling is automatically switched OFF if CCU 100 changes to Standby mode because of an error and if the instrument is switched off completely.

TIP: *If you want to switch off the LN2 cooling at the end of a measurement (target temperature = 25 °C), you can set the Cooling Limit on CCU 100 to a value below 25 °C).*

6.5.2 Controlling the Compressed-Air Cooling

CCU 100 turns ON the compressed-air cooling under the following conditions:

- Air cooling is turned on, if the temperature setpoint (SP) is 5 °C lower than the actual temperature of the sample holder.
- Air cooling is permanently turned on if the temperature set point is below a threshold value called Cooling Limit (CL). CL can be set between 20 °C and 50 °C on the Parameter Page of CCU 100.

The cooling is automatically switched OFF if CCU 100 changes to Standby mode because of an error and if CCU 100 is switched off completely.

TIP: *Always set the air cooling limit (CL) higher than the temperature of the cooling water which runs through the chamber.*

TIP: *If you want to switch off the air cooling at the end of a measurement (target temperature = 25 °C), you can set the Cooling Limit on CCU 100 to a value below 25 °C).*

6.6 Manual Control of CCU 100

Manual operation of TTK 600 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 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.*

6.7 Remote Control of CCU 100

Usually CCU 100 is integrated in the software that controls the X-ray diffractometer. For more information about controlling TTK 600, 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 Operating the LN2 Cooling Equipment

This chapter gives specific information for the operation of the liquid nitrogen cooling equipment of TTK 600.

7.1 LN2 Consumption and Operating Time

The typical consumption of LN2 does not exceed 4 L/h. This corresponds to a measuring time of max. 25 hours in case that a Dewar vessel with a volume of 100 L is used.

7.2 Typical Cooling Curves

For typical sample holder cooling curves with maximum cooling rate for the liquid nitrogen cooling equipment and the compressed-air cooling equipment refer to chapter 8.8.2.1.

8 Performing a Measurement

This chapter describes all activities related to using TTK 600 for an in-situ X-ray diffraction measurement.

8.1 Adjusting the Knife Edge

TTK 600 is equipped with a knife edge, which can be used to reduce the X-ray background signal for small 2θ angles. The minimum height H of the knife edge above the sample surface must be adjusted to the required maximum 2θ angle and the beam height (divergence slit):

$$H[mm] \geq \frac{R \times \sin\delta}{\cos(\omega_m - \delta)}$$

where R= radius of the goniometer in [mm]
δ= $^1/_2$ x beam divergence angle in [deg]
ω_m= $^1/_2$ x maximum 2θ angle in [deg]

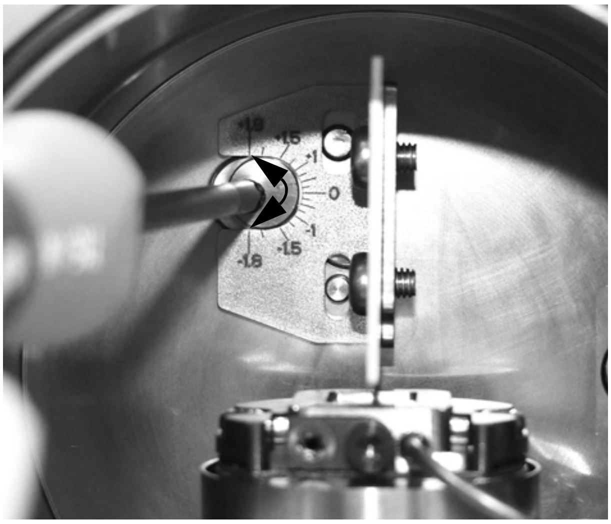


Fig. 48: Adjusting the knife edge

The knife edge is easily attached and detached as it is magnetic. To put it in, just hold it close to the adapter at the back of the chamber and it will attach in the correct position automatically.To take the knife edge out, extract it from its position.

NOTICE
- Adjust the height of the knife edge before you prepare the sample.
- If you don’t measure at small 2θ angles, remove the knife edge from TTK 600 to avoid accidental blocking of the beam and to make sample holder handling more convenient.

The height of the knife edge can be adjusted as follows:

Use the delivered Torx screw driver to adjust the height of the knife edge. Clockwise rotation increases the height, whereas counter-clockwise rotation decreases the height.

NOTICE
There are separate Knife Edges for all three types of sample holders available.

8.2 Preparing the Sample

8.2.1 Additional Information for the Standard Sample Holder

The sample holder (1) of the TTK 600 is made of nickel, providing a good thermal conductivity and mechanical properties as well as high corrosion resistance.

Three sample holders with different depths of the sample cavity (0,8 mm, 0,2 mm or flat) are available.

In addition a zero background holder with a specially cut silicon insert can be used to avoid X-ray background signals from the sample holder. This is particularly useful for organic samples with high X-ray transmission.

A Pt100 sheath resistance temperature sensor (2) is encapsulated in the sample holder (1), providing exact temperature measurement. A connector for the Pt100 (3) permits fast and easy exchange of the sample holder.

Before the instrument is used for the first time, the insulation of the heater has to be installed. This can be done in the following way:

1. Remove the sample holder (refer to chapter 8.2.4) and the beam knife (refer to

chapter 8.1).

2. Take out the heater insulation from the accessory box delivered with the TTK 600 and place it over the heater. See Fig. 9for further details.
3. Mount the heating block by reversing the actions from point 1.

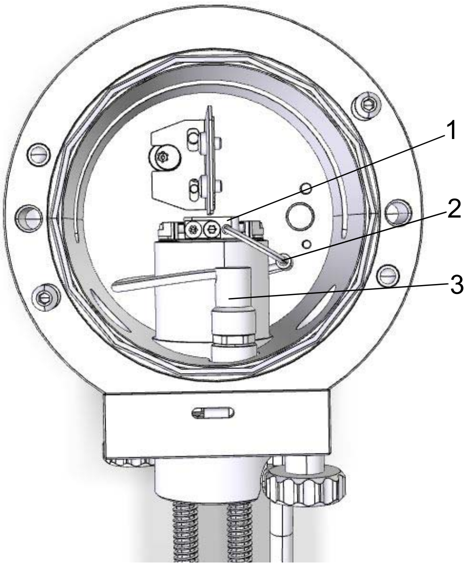


Fig. 49: Sample on standard sample holder

NOTICE
Risk of damage.
Take care that the graphite foil that provides improved thermal contact between sample holder and heating/cooling block is in a good condition (no visible scratches). Otherwise it is strongly recommended to replace the graphite foil with a new one in order to minimize the temperature deviation between displayed and real sample temperature.

8.2.2 Additional Information for the Transmission Sample Holder

The transmission sample holder (1) of TTK 600 is made of nickel providing a good thermal conductivity and mechanical properties as well as high corrosion resistance.

A Pt100 sheath resistance temperature sensor (2) is encapsulated in the sample holder (1), providing exact temperature measurement. A connector for the Pt100 (3) permits fast and easy exchange of the sample holder. An additional convection heating is provided by the connection of the gas hose (4) to the gas connector on the right side of the instrument (5). This additional heating mechanism ensure a minimization of the temperature deviation between displayed and real sample temperature. To protect the detector, use the beamstop (6) at low 2Θ angles.

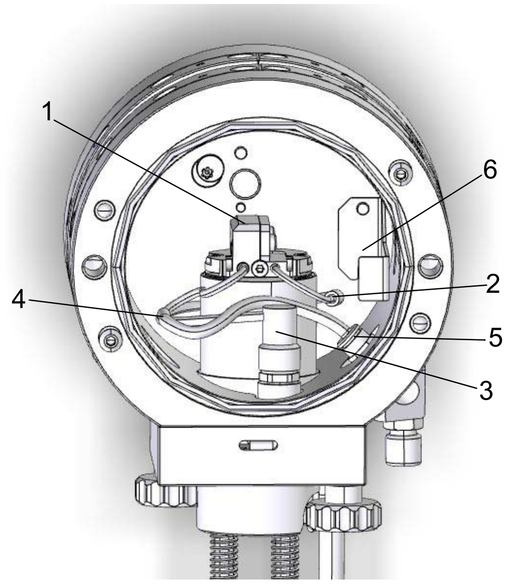


Fig. 50: Sample on transmission sample holder

NOTICE
Risk of damage.
Take care that the graphite foil that provides improved thermal contact between sample holder and heating/cooling block is in a good condition (no visible scratches). Otherwise it is strongly recommended to replace the graphite foil with a new one in order to minimize the temperature deviation between displayed and real sample temperature.
Detailed information on the replacement of the graphite foil are given in the service manual of TTK 600.

8.2.3 Samples for the Capillary Sample Holder

For information on preparing samples for the capillary sample holder, refer to the instruction manual Capillary Sample Holder for TTK 600.

8.2.4 Taking out the Sample Holder

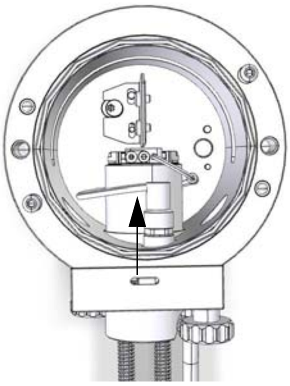
8.2.4.1 Standard Sample Holder

CAUTION

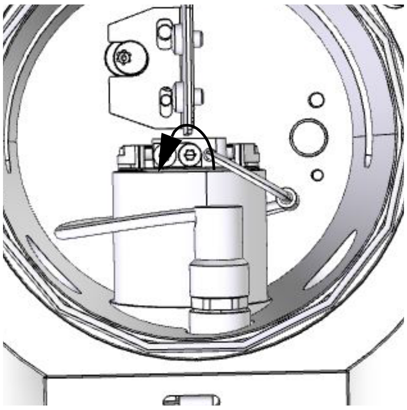
Hot surface
During operation and even after switching off the heater there can be dangerously hot or cold surfaces inside the sample chamber. Always check on the display of CCU 100 that the sample holder temperature is below 50 °C before you open the sample chamber and touch any parts inside.
If CCU 100 shows an error message, use a suitable temperature sensor to measure the actual temperature of the parts inside the sample chamber. Always switch off the heater before you take out the sample holder.

In order to take the sample out of TTK 600, proceed as follows:

1. Swich off the heater of CCU 100
2. Open the lid.
3. Disconnect the Pt100 connector.



4. Unscrew the fixing screw of the sample holder with the supplied Torx screw driver.



5. Take the sample holder carefully out of the fixing block by gentle pulling on the sheat resistance temperature sensor (right side) while lifting the cable of the temperature sensor over the heating block (left side)
6. Place the sample holder on the corresponding mount as shown in the following picture.

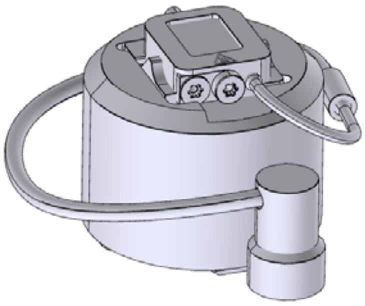


Fig. 51: Sample holder on mount

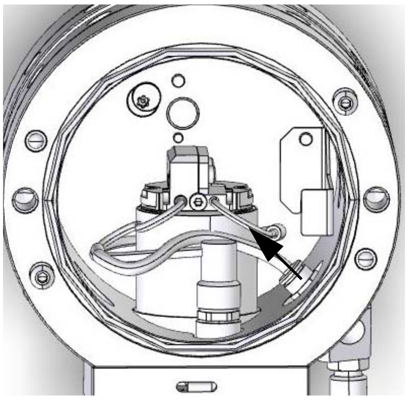
8.2.4.2 Transmission Sample Holder

CAUTION

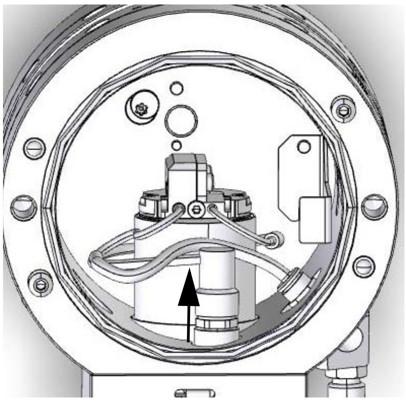
Hot surface
During operation and even after switching off the heater there can be dangerously hot or cold surfaces inside the sample chamber. Always check on the display of CCU 100 that the sample holder temperature is below 50 °C before you open the sample chamber and touch any parts inside.
If CCU 100 shows an error message, use a suitable temperature sensor to measure the actual temperature of the parts inside the sample chamber. Always switch off the heater before you take out the sample holder.

In order to take the sample out of TTK 600, proceed as follows:

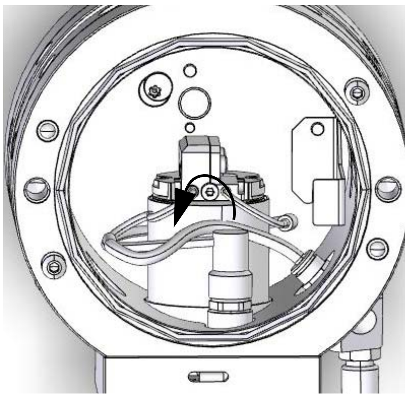
1. Switch off the heater of CCU 100.
2. Open the lid.
3. Disconnect the Pt100 connector.



4. Disconnect the Gas Connector.



5. Unscrew the fixing screw of the sample holder with supplied Torx screw driver.

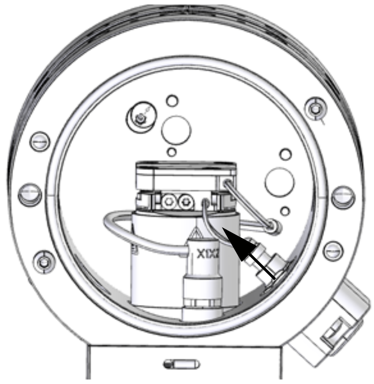


6. Take the sample holder carefully out of the fixing block by gentle pulling on the sheat resistance temperature sensor (right side) while lifting the cable of the temperature sensor over the heating block (left side).

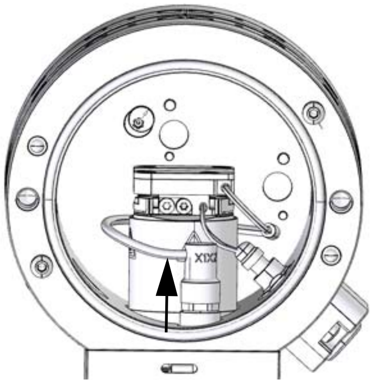
8.2.4.3 Battery Sample Holder

In order to remove the sample from the TTK 600, proceed as follows:

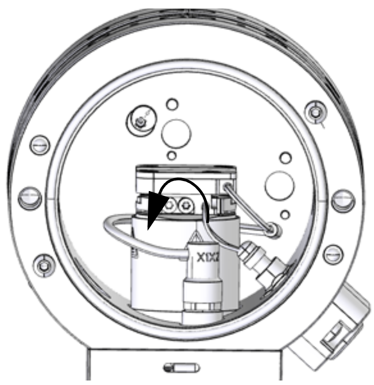
1. Switch off the heater of the CCU 100.
2. Open the lid.
3. Disconnect the electrode connector from the TTK 600 housing.



4. Disconnect the Pt100 connector.



5. Unscrew the fixing screw of the sample holder with the supplied Torx screwdriver.



6. Carefully take the sample holder out of the fixing block by gently pulling on the sheath resistance temperature sensor (right side) while lifting the cables over the heating block (right side).

8.2.5 Applying the Sample

In general the most suitable way of sample preparation strongly depends on the sample, temperature range and goal of the experiment. Therefore, regard the following instructions as suggestions only.

NOTICE
Risk of damage.
Sample components might react with the sample holder and destroy it.
- Check the chemical resistance of the sample holder for the respective sample in the applied atmosphere and at the desired temperature BEFORE the experiment.
- Refer to appendix A for technical data of the TTK 600 Low -Temperature Chamber.

8.2.5.1 Powder Samples for the Standard Sample Holder

Prepare the sample as follows:

Use a spatula to press the powder into the groove of the sample holder and then smooth the powder's surface with a glass platelet.

If required, mix the powder with a small quantity of diluted Zapon lacquer solution.

1. Again, use the spatula to uniformly apply this "slurry" into the groove of the sample holder.
2. Allow sufficient time for the sample to dry and become an adhesive and solid substance.

NOTICE
Risk of damage.
Do not heat fast, because rapidly evaporating liquid creates holes in the sample.
- Make sure that the sample completely covers the sample holder. Holes in the sample can cause large background signal from the sample holder.
- For organic samples with high X-ray transmission, use the zero back- ground holder.

8.2.5.2 Bulk Samples for the Standard Sample Holder

Bulk samples are usually difficult to measure because of irreproducible deviations between the temperature displayed on the CCU 100 and the temperature of the sample surface scanned with the X-ray beam.

Main properties which determine the temperature gradient are:

- thermal conductivity and thickness of the sample
- flatness and smoothness of the sample surface.

NOTICE
Make sure that the sample is thermally contacted to the sample holder to achieve a uniform temperature distribution within the sample.

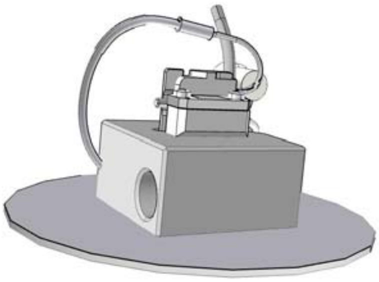
Place solid samples into the groove of the sample holder. The depth of the groove in the sample holder is 0.8 mm (a sample holder with a depth of 0.2 mm and a flat sample holder are also available). The alignment stage allows you to compensate for the difference in height between the sample holder and sample holder groove.

NOTICE
If possible, do not measure bulk samples in vacuum. Heat transfer in vacuum is usually very bad. Use inert gas (He) instead of vacuum.
Bulk samples often bend upon heating, which changes the thermal contact and the temperature gradients in an undefined way.

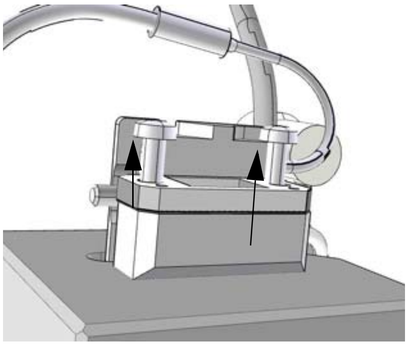
8.2.5.3 Powders and Pastes for the Transmission Sample Holder

The procedure for preparing powder and paste samples for the transmission sample holder is described below:

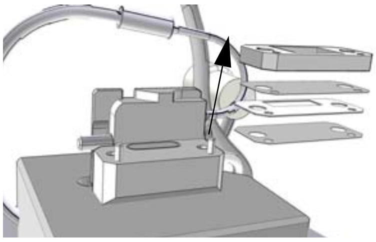
1. Place the delivered mounting tool on a flat surface and place the transmission sample holder



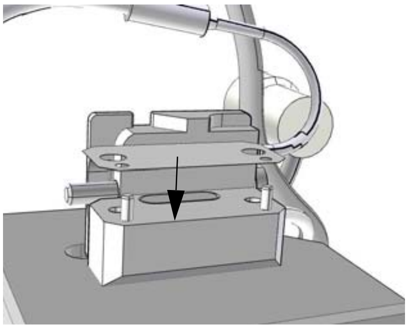
2. Remove the two screws to open the sample holder.



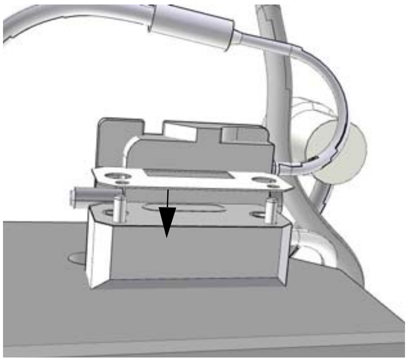
3. Remove all the pieces and foils from the sample holder.



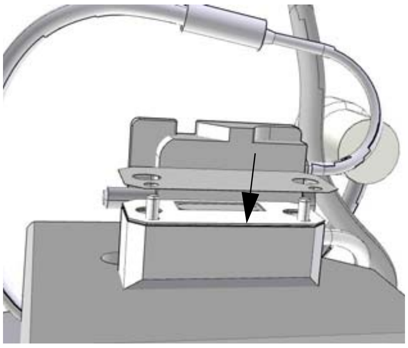
4. Place the first foil over the first clamping piece.



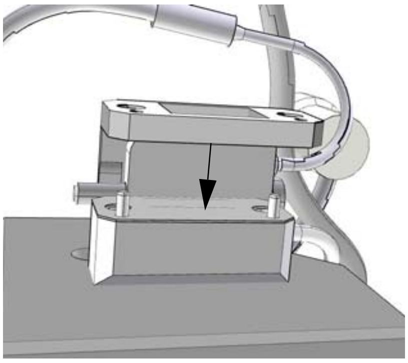
5. Add the distance piece to the stack. Different distance pieces are available: 1 mm, 0.5 mm and 0.25 mm.



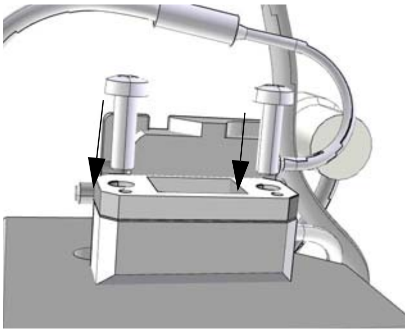
6. Fill the central room in the distance piece with your sample. Powders and pastes can be used. Use a spatula to compress the sample within the compartment.
7. Add the second foil to the stack.



8. Add the clamping piece.



9. Fix the stack with the two screws.



8.2.5.4 Foils for the Transmission Sample Holder

Foils can be mounted in a similar way as described in the previous chapter. In this case only the two clamping pieces together with the foil have to be used.

In order to be able to mount the foil correctly between the two clamping pieces, ensure that the foil has the following dimensions: 12 x 16 mm.

8.2.5.5 Coin Cells for the Battery Sample Holder

Battery Sample Holder (Reflection):

The procedure for preparing the coin cells for the battery sample holder (reflection) is described below:

1. Place the battery sample holder together with all necessary components for the assembly of the battery in a glove box.
2. Remove the four screws on the lid (1) of the battery sample holder.
3. Remove the foils (2+3) and cathode (4) of the battery sample holder.
4. Place the assembled coin cell (5) on the anode (6).
5. Place the cathode on the coin cell.
6. Place first the Kapton foil (3) on the cathode, followed by the graphite foil (2). (The use of the graphite foil is optional. See also chapter 8.3.4).
7. Mount the lid (1) of the battery sample holder again by the use of the 4 Torx screws.

An exploded-view drawing showing all the components (including the assembled coin cell) is shown below.

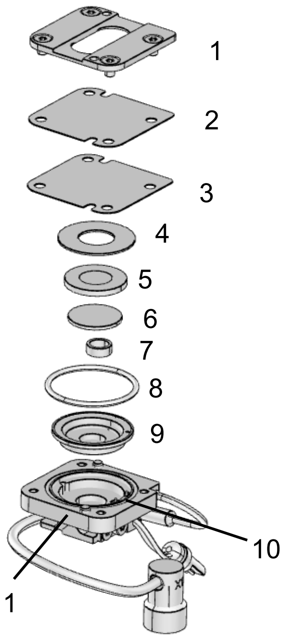


Fig. 52: Exploded-view drawing of the battery sample holder (reflection)

1 Lid of holder
2 Foils: kapton + graphite
3 Cathode
4 Coin cell
5 Anode
6 Isolation inlet
7 O-ring
8 Basis
9 Electrode contacts

After the complete assembly of the battery sample holder, the sample holder can be extracted from the glove box and mounted on the heater of TTK 600 (see chapter 8.4.6 for further details).

NOTICE

Risk of damage.

When closing the battery sample holders ensure that the cathode is correctly aligned in the slot in the sample holder base. Rotate and visually inspect the sample holder after closing to ensure there are no gaps between the sample holder base and the lid. Not closing the sample holder correctly can lead to problems aligning the sample.

Battery Sample Holder (Transmission):

The procedure for preparing the coin cells for the battery sample holder (transmission) is described below:

1. Place the battery sample holder together with all necessary components for the assembly of the battery in a glove box.
2. Remove the four screws on the lid (1) of the battery sample holder.
3. Remove the foils (2+3) and cathode (4) of the battery sample holder.
4. Place the assembled coin cell (5) on the anode (6).
5. Place the cathode on the coin cell.
6. Place first the Kapton foil (3) on the cathode, followed by the graphite foil (2). (The use of the graphite foil is optional. See also chapter 8.3.4).
7. Mount the lid (1) of the battery sample holder again by the use of the 4 Torx screws.

An exploded-view drawing showing all the components (including the assembled coin cell) is shown below.

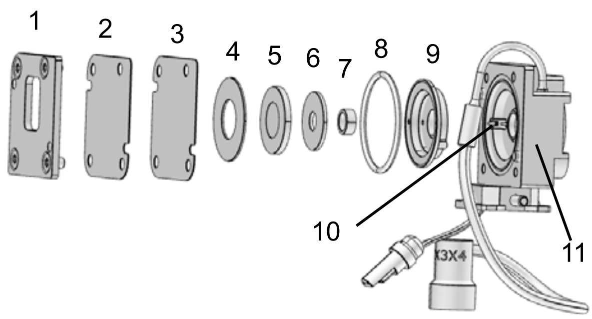


Fig. 53: Exploded-view drawing of the battery sample holder (Transmission)

1 Lid of holder
2 Graphite foil
3 Kapton foil
4 Cathode (aluminum)
5 Coin cell
6 Anode (copper)
7 Spring
8 O-ring
9 Isolation inlet
10 Electrode contacts
11 Basis of sample holder

After the complete assembly of the battery sample holder, the sample holder can be extracted from the glove box and mounted on the heater of TTK 600 (see also chapter 8.4.6 for further details).

8.3 Guideline for the correct choice of conditions and foil materials

The following guideline can be used for the optimal choice of the foil material for the transmission sample holder.

8.3.1 Temperature Range

Whenever the Kapton foil is used, the max. temperature of the experiment must not exceed 230 °C. Higher temperatures would result in a destruction of the foil material.

NOTICE
Risk of damage.
Use of the Kapton foil limits the max. temperature to 230 °C

Both, nickel and graphite, do not limit the temperature range of the experiment.

For operation in air the max. temperature is limited to 450 °C.

8.3.2 Temperature accuracy and influence of convection heater

The thermal conductivity of the offered foils is as follows:

Kapton ~ 0.2 W/mK
Nickel ~ 80 W/mK
Graphite ~ 140 W/mK

The better the thermal conductivity of the foil, the lower is the temperature difference between displayed temperature on CCU 100 and real temperature on the surface of the sample. As a consequence of this, for nickel and graphite, the additional convection heater is obsolete. Due to the high thermal conductivity, these foils transport the heat very efficiently and do not need the additional heat transfer mechanism of convection.

Kapton has a quite poor thermal conductivity. In this case the additional convection heating improves the temperature accuracy of the instrument. The optimal gas flow for the Kapton foil is 0.5 L/min.

Therefore the temperature accuracy and the conditions used for the different foils can be summarized as follows:

Graphite (0 L/min) > Nickel (0 L/min) > Kapton (0.5 L/min)

8.3.3 Background of the foil materials

Another factor that has to be kept in mind for the right choice of the foil material is the background of the foils. The foil should be chosen in a way that the ROI (region of interest) does not overlap with signal of the foil.

The background of the different foils is summarized below:

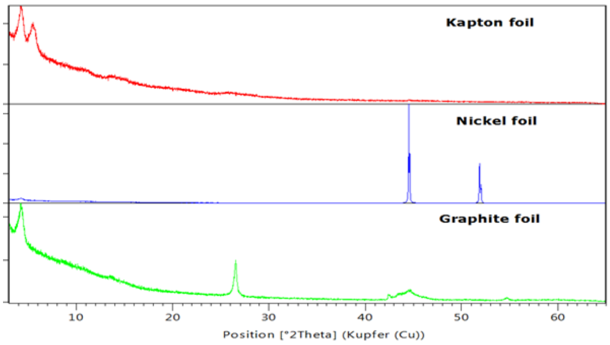


Fig. 54: Transmission measurements of the used foil materials in TTK 600

The Kapton foil shows two peaks in the low 2θ-region at ~ 4.4 and 5.7°. The first one can be attributed to the Kapton foil on the housing of TTK 600 (It is also visible for all other measured foils).

The Nickel foil shows strong reflections at 44.5 and 51.9° 2θ whereas graphite shows a medium intense reflection at 26.6° and an amorphous reflection from 42.3 to 47.4° 2θ.

The thicknesses of the used foil materials are as follows:

Foil material	Thickness/ µm	Transmission for Cu-radiation
Kapton	25	96
Nickel	10	43
Graphite	80	86

From the comparable low values of transmission for Nickel it can be clearly seen that Nickel should only be used as foil material if the background of graphite does interfere significantly with the ROI from the investigated substance.

8.3.4 Foil Materials for the Battery Sample Holders

The battery sample holders (both reflection and transmission) contain Kapton foils to ensure the gas tightness of the sample holder. As the thermal conductivity of Kapton is relatively poor, additional graphite foils can be provided to ensure the smallest possible difference between the temperature on the CCU 100 and the actual sample temperature. The graphite foils must be used in combination with the Kapton foils to ensure the gas tightness of the sample holder.

The absolute temperature differences for the different foil combinations on the reflection and transmission holders in a nitrogen atmosphere are shown below:

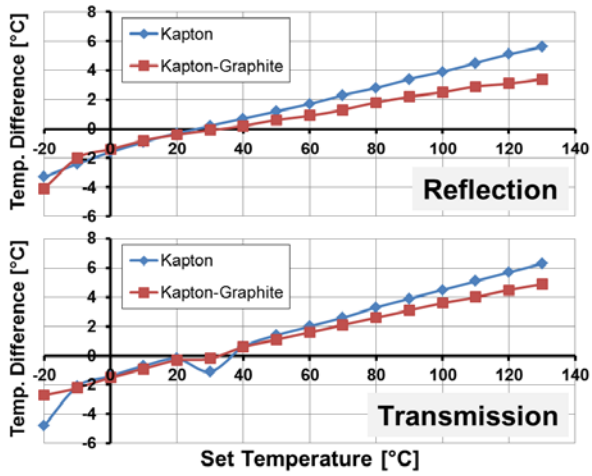


Fig. 55: Difference between the set temperature of the CCU 100 and the actual sample temperature for different foil combinations of the battery sample holders under N_2 atmosphere.

The temperature differences are fairly small across the entire measured range (typically < 5 %) when only a Kapton foil is used and smaller when the graphite foil is also used. However, especially in the case of the reflection battery sample holder, use of the graphite foil leads to strong additional diffraction peaks so this must also be considered.

In addition, the use of the graphite foil leads to a reduction in the diffracted intensity. For both the reflection and transmission battery sample holders, the diffracted intensity is ~ 68 % of that obtained when only Kapton foils are used.

Due to the influence that the graphite foils have on the diffraction pattern (see chapter 8.3.3) and the reduced X-ray transmission, it is recommended in most cases to use only a Kapton foil; graphite foils are optionally available when the smallest possible temperature deviations from the displayed temperature of the CCU are needed.

8.4 Mounting the Sample Holder

NOTICE
Risk of damage
Make sure the graphite foil for heat conduction is not scratched. Deep scratches will cause insufficient heat transfer which will eventually lead to severe damage of the heating system.
For changing the graphite foil refer to the service manual of TTK 600.

8.4.1 Mounting the Standard Sample Holder

In order to mount the standard sample holder proceed as follows:

1. Insert the sample holder in the groove of the sample holder fixing ring (1) while guiding the cable of the Pt100 (2) behind the heating/cooling block.
2. Use the Torx screw driver that was delivered with the instrument to fix the sample holder to the fixation ring by tightening the screw (3) until the first click of the Torx screw driver occurs. (corresponds to a force of 1.1 Nm)
3. Connect the Pt100 to the Pt100 plug connector (4).
4. Switch on the heater of CCU 100.

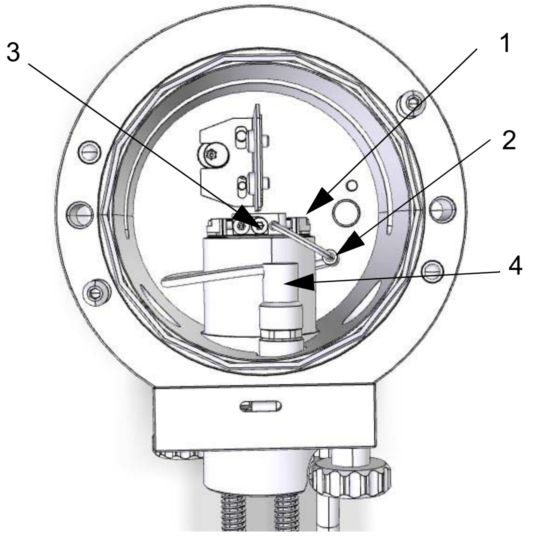


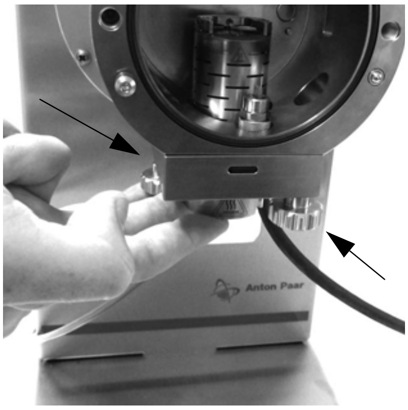
Fig. 56: Standard Sample Holder

1 Sample holder fixing ring
2 Pt100
3 Fixing screw
4 Pt100 plug connector

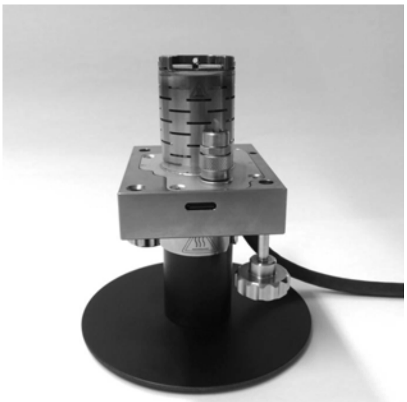
8.4.2 Mounting the Standard Sample Holder Heating Environment

To mount the sample holder environment proceed as follows:

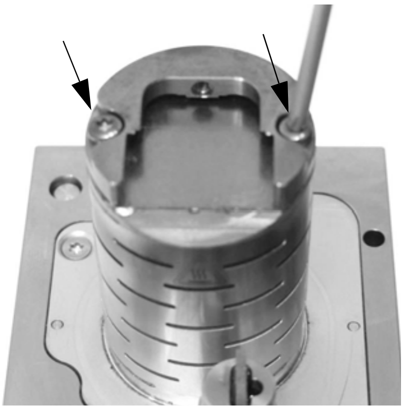
1. Remove the sample holder.
2. Remove the cooling equipment from the instrument.
3. Demount the heating/cooling block of the instrument from the housing by unscrewing the knurled screws as shown in the following picture. Take care to hold the heating block while unscrewing the last screw. (Should the knurled screws at the bottom be stuck, use the supplied Torx 10 to loosen them.).



4. Place the heating/cooling block on the delivered stand as shown in the picture.



5. Remove the sample holder fixing ring by unscrewing the two screws with the supplied Torx driver.



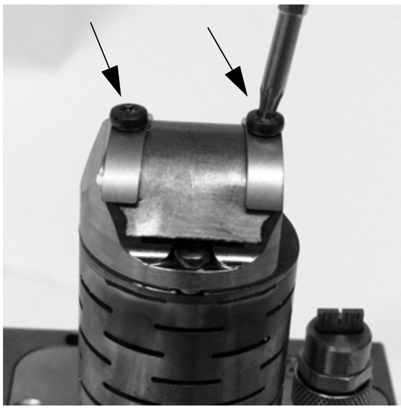
6. Take of the sample holder fixation ring and replace it with the sample holder heating environment.

7. Mount the sample holder heating environment with the two screws.
8. Mount the sample holder as described in chapter 8.4.1 Mounting the Standard Sample Holder.

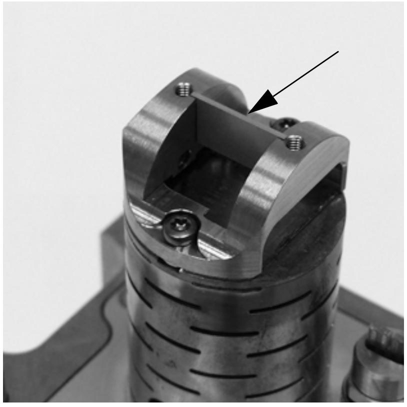
Nickel and graphite are available as foil materials for the standard sample holder heating environment. The choice depends mainly on the ROI (region of interest) of the diffractogram. Background measurements for Nickel and Graphite can be found in chapter 8.3.

To replace the foil of the sample holder heating environment and to demount/mount the antiscatter shield for the sample holder heating environment proceed as follows:

1. Unscrew the two screws of the foil clamps and take of the clamps and the foil



2. Mount/demount the antiscatter shield.



3. Replace the foil and ensure that the notches of the foil are placed over the holes for the screws.

4. Mount the foil clamps with the two screws.

8.4.3 Mounting the Capillary Sample Holder

For information on mounting the capillary sample holder, refer to the instruction manual Capillary Sample Holder for TTK 600.

8.4.4 Mounting the Transmission Sample Holder

In order to mount the transmission sample holder proceed as follows:

1. Dismount the blind cover on the right side of the housing and replace it with the gas connector. Install the gas line for the connection heater (see chapter 4.12).
2. Insert the sample holder in the groove of the sample holder fixing ring (1) while guiding the cable of the Pt100 (2) behind the heating/cooling block.
3. Use the Torx screw driver that was delivered with the instrument to fix the sample holder to the fixation ring by tightening the screw (3) until the first click of the Torx screw driver occurs. (corresponds to a force of 1.1 Nm)
4. Connect the Pt100 to the Pt100 plug connector (4).
5. Connect the tube for the convection heating to the corresponding convection heater connector (5).
6. Switch on the heater of CCU 100.
7. Connect the outside connector of the gas connector to the delivered gas hose of the instrument.
8. Connect the other end of the gas hose to the gas reservoir.
9. The optimal flow that is needed depends on the choice of the foils. Further information can be found in chapter 8.3 Guideline for the correct choice of conditions and foil materials.

NOTICE
A separate flow controller has to be mounted in this gas line in order to be able to adjust the gas flow accordingly. This item is not delivered by Anton Paar GmbH. It is recommended to use a flow controller that allows to adjust the flow between 0.5 and 3 L/min.

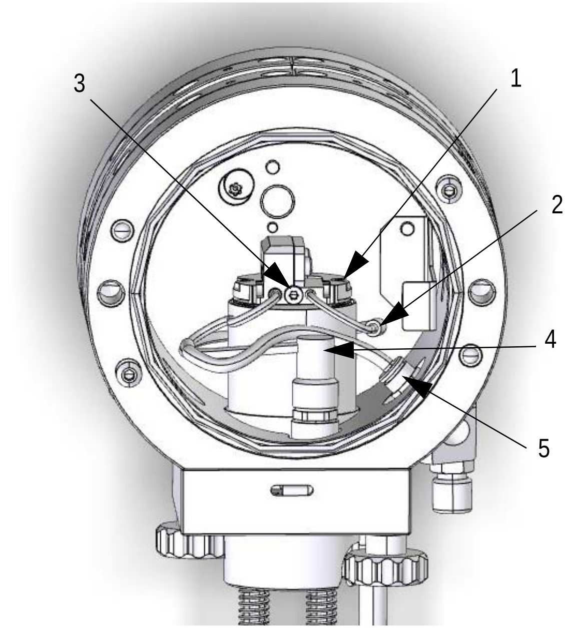


Fig. 57: Transmission Sample Holder

1 Sample holder fixing ring
2 Pt100
3 Fixing screw
4 Pt100 plug connector
5 heater connector

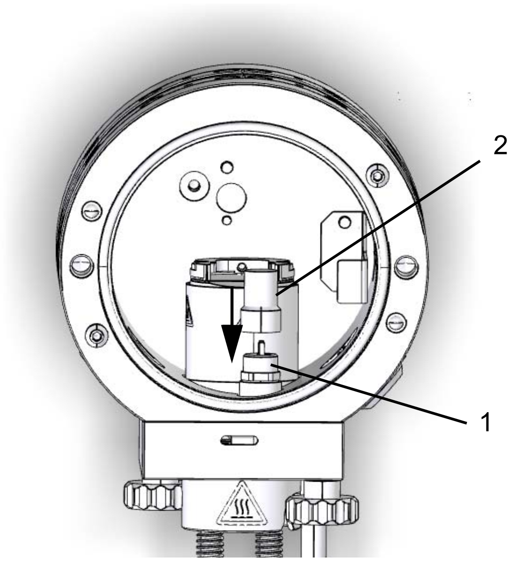
In order to prevent the detector from being damaged by the primary beam at low 2Θ angles, a beam stop is delivered with the transmission sample holder.

The beam stop can be mounted/demounted easily as it is magnetically coupled to the housing of the instrument. It is simply mounted by placing it over the corresponding guiding rods (3 in Fig. 7) on the right backside of the instrument.

8.4.5 Mounting and use of the Antechamber for TTK 600

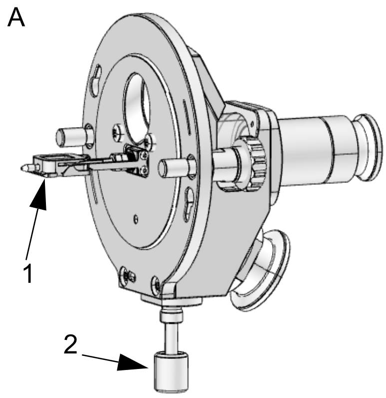
In order to use the antechamber for TTK 600, the normal lid of the instrument has to be removed. In order to transfer a sample without interaction with air from a glove box to TTK 600, the following steps are necessary:

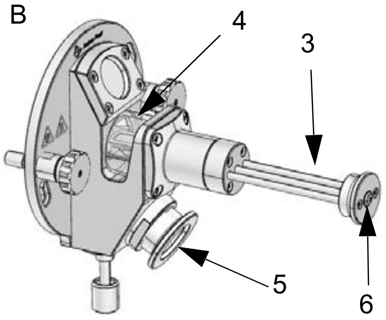
Before the sample is loaded to the antechamber, connect the Pt100 blind plug (2) that was delivered together with the antechamber to the Pt100 plug connector (1).



1 Pt100 plug connector
2 Pt100 blind plug

This is necessary to change the control behavior of CCU 100 from the Pt100 to the thermocouple inside the heater.





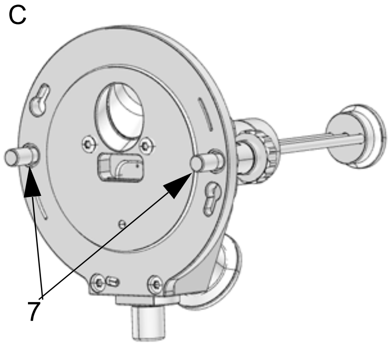


Fig. 58: Antechamber TTK 600

1 Sample holder for antechamber
2 Closing mechanism
3 Sample holder loader
4 Gas tight compartment
5 Vacuum flange
6 Torx screw for sample holder fixation
7 Mounting screws

1. Place the antechamber as depicted in Fig. 58 (A) in the glove box. In order to do so a separate mount is contained in the delivery of the antechamber (see appendix 12).
2. Fill the sample into the sample holder (1).
3. Pull back the sample loader (3).
4. Close the gas tight compartment (4) by pushing in the closing mechanism (2) like shown in Fig. 58 (B).
5. Extract the antechamber from the glove box and mount it on TTK 600. The mounting is done in exactly the same way as for the standard lid of the instrument by using the two mounting screws (7).
6. Connect the gas vacuum connector to the vacuum flange (5) of the antechamber.
7. Remove residual air from TTK 600 by purging gas and/or evacuating.
8. In the final step of air removal bring TTK 600 back to ambient pressure by purging with inert gas.

9. Open the gas tight compartment (4) by pulling down the closing mechanism (2).
10. Insert the sample holder (1) into the fixing ring of TTK 600 by pushing the sample holder loader (3) inwards.
11. Fix the sample holder (1) to the fixing ring by the use of the Torx screw (6) on the sample holder loader. To do so, use the constant force Torx screw driver that was delivered with the instrument. Screw in till the first click occurs.
12. Pull back the sample holder loader (3).
13. Close the gas tight compartment by pushing in the closing mechanism.
14. Sample is now ready for the experiment.

In order to remove the sample from TTK 600, proceed as follows:

1. Open the gas tight compartment (4) by pulling down the closing mechanism.

NOTICE
If vacuum has been applied during the experiments before, always bring the instrument back to ambient pressure before you try to open the gas tight compartment.

2. Push the sample holder loader (3) inwards.
3. Use the torx screw (6) on the sample holder loader to unscrew the sample holder from the fixing ring of the instrument.
4. Insert the sample to the gas tight compartment by pulling back the sample holder loader(3).
5. Close the gas tight compartment by pushing in the closing mechanism.
6. Remove the antechamber from TTK 600 by unscrewing the two mounting screws.
7. Bring the sample back to the glove box if necessary.

8.4.6 Mounting and Use of the Battery Sample Holders

Before mounting the battery sample holder, ensure that the chamber is mounted on the adapter in the correct position for the sample holder which will be used. Details for the mounting of the adapter can be found in chapter 4.3.

Battery Sample Holder (Reflection):

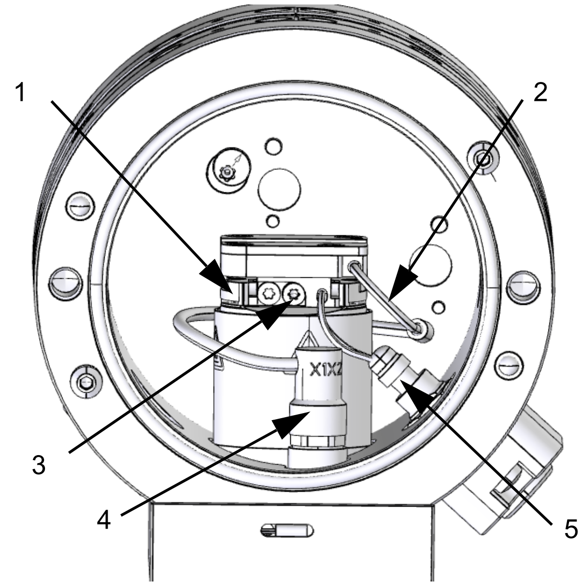


Fig. 59: Reflection sample holder

To mount the battery sample holder (reflection), proceed as follows:

1. Insert the sample holder in the groove of the sample holder fixing ring (1) while guiding the cable of the Pt100 (2) behind the heating/cooling block.
2. Use the Torx screw driver that was delivered with the instrument to fix the sample holder to the fixing ring by tightening the screw (3) until the first click of the Torx screw driver occurs (corresponds to a force of 1.1 Nm).
3. Connect the Pt100 to the Pt100 plug connector (4).
4. Connect the electrode cable to the electrode connector in the housing of TTK 600 (5).
5. Close the lid of the TTK 600.
6. Switch on the heater of CCU 100.

To align the sample in the beam, the standard I1/2 method can first be used (see chapter 6.2). If the X-ray tube and detector are correctly aligned, the surface of the sample holder should first block the X-ray beam during the height scan, before a small peak in intensity appears representing the gap between the top part of the sample holder and the Kapton foil. The sample holder height should be aligned to the position of the reflection battery sample holder lid. So that the sample is correctly aligned in the X-ray beam and peak shifts due to height errors are avoided, the height should be increased by the following amounts depending on the foils being used:

- only Kapton foil: +2.28 mm
- Kapton and graphite foils: +2.32 mm

NOTICE
Risk of damage
Take care that no setpoint > 130 °C is applied to the instrument as this can cause destruction of the components of the battery sample holder.

Battery Sample Holder (Transmission):

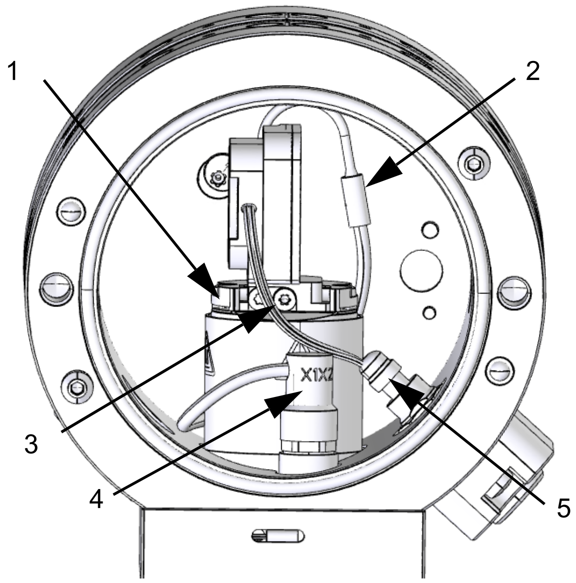


Fig. 60: Transmission sample holder

To mount the battery sample holder (transmission), proceed exactly as described for the battery sample holder (reflection).

To align the sample in the beam, a height scan should be performed with ? = 0° and 2? = 0°. This will show intensity where the window opens on the back side of the sample holder. The sample height should be aligned to be in the middle of this intensity peak.

8.5 Creating a Gas/Vacuum Environment

An overview of the possible in-situ XRD experiments that are possible with the TTK 600 Low-Temperature Chamber is given in chapter 2.2.

CAUTION

Risk of injury.
The gas inside the chamber expands when heated. Above 3 bar rel. the gas will be released through the overpressure relieve valve on the lid of the instrument.
This overpressure relieve valve MUST NOT be used for the control of the pressure inside the instrument. Instead a separate adjustable overpressure relieve valve has to be installed in the gas line.

8.5.1 Operation with Air

CAUTION

Risk of injury.
Be aware that experiments in air can be performed at temperatures between room temperature and 450 °C for the standard sample holder.

There are no special requirements for the operation with air inside the chamber.

Leave the vacuum flange open to allow release of gas during heating in order to avoid overpressure inside the chamber.

8.5.2 Operation under Vacuum

TTK 600 can be evacuated to approx.$1x10^{-2}$ mbar with a rotary pump (4.2 m^3/h).

In case that high vacuum is needed, TTK 600 can also be evacuated to approx. $1x10^{-4}$ mbar using a turbomolecular pump.

Experiments in vacuum can be performed in the full temperature range of the TTK 600 Low-Temperature Chamber from -190 °C to 600 °C.

TIP: *The thermal conductivity of powder material is much lower than that of the corresponding bulk material. As a consequence, significant temperature deviation between the sample holder and the*

sample surface can occur.
Ways to avoid this problem:
Measure in inert gas (e.g. He) instead of vacuum.
Make a slurry of the sample powder with a suitable solvent, e.g. diluted Zapon lacquer.

Anton Paar GmbH offers a standard vacuum equipment and a high vacuum equipment as an accessory for the TTK 600. Refer to appendix D for further information.

TTK 600 can be evacuated through the vacuum flange or in combination with the gas/vacuum connector and a ball valve depending on the experimental requirements.

The ball valve (corner valve) is not included in the vacuum equipment but can be ordered as an accessory (refer to appendix D).

NOTICE
Risk of damage.
To protect the vacuum equipment from pressure a ball valve MUST be installed between gas/vacuum connector and the vacuum equipment. BEFORE opening the gas inlet and applying pressure the ball valve has to be closed as damage of the vacuum parts will occur.

Vent the chamber slowly to avoid the sample being detached from the sample holder.

Detailed information about the set-up for operation with vacuum are given in the instruction manual for the vacuum equipment.

8.5.3 Operation with Gases Other than Air

WARNING

Risk of injury.
Do not use TTK 600 with hazardous (e.g. explosive or poisonous) gases. Use of these gases can lead to an exposure to hazardous gases.

Standard gases (beside air) for measurements with TTK 600 are inert gases and nitrogen. Other non-hazardous gases can be used, provided the gas itself and possible reaction products of the gas with the sample material do not damage the sample holder and the chamber in the complete temperature range of interest.

NOTICE
Risk of damage.
Check the chemical resistance of the sample holder and the materials inside the chamber against your sample and the applied atmosphere up to the required maximum temperature BEFORE the experiment.

Never operate the TTK 600 at temperatures higher than 450 °C, when working under oxidizing atmosphere to avoid damage to the interior of the chamber.

The gas inside the chamber expands when heated. Above 3 bar rel. the gas will be released through the overpressure relieve valve on the lid of the instrument.

Use a suitable pressure regulator for the pressure range 0 - 2 bar rel.

Temperature control of TTK 600 is optimized for stationary gas filling of the chamber. A gas flow through the chamber during heating/cooling will deteriorate temperature control and cause a temperature offset between sample holder temperature (displayed) and sample surface.

Gas can be supplied and extracted from the TTK 600 through the gas/vacuum connector which can be connected to the lid of the instrument.

If you need oxygen-free sample environment, combine the gas supply system with a vacuum equipment.

NOTICE
Risk of damage.
To protect the vacuum equipment from pressure a ball valve MUST be installed between gas/vacuum connector and the vacuum equipment. BEFORE opening the gas inlet and applying pressure the ball valve has to be closed as damage of the vacuum parts will occur.

A typical gas supply system consists of:

- gas supply with pressure reducer and pressure regulator
- gas hoses of appropriate materials
- vacuum equipment (if applicable)

TIP: *Anton Paar GmbH does not provide components for gas supply systems.*

8.6 Heating and Cooling the Sample

WARNING

Risk of cryogenic burns.
Never disconnect the cooling device or any other components of the sample cooling circuit during operation. The liquid nitrogen cooling equipment contains liquefied nitrogen. Liquefied nitrogen can cause freezing of tissue, or cryogenic burns, similar to frostbite to eyes or skin upon contact. The compressed-air cooling components are under pressure. Disconnected components present a mechanical hazard.

Sample heating/cooling can be done in two ways:

- You can enter the desired heating/cooling rate and the target temperature manually into CCU100 (see chapter chapter 6.4).
- You can use a computer with the software delivered by Anton Paar GmbH to control TTK 600.

TIP: *Usually, TTK 600 is integrated in the software that controls the X-ray diffractometer. Read the instruction manual of your diffractometer control software.*

In order to heat the sample with manual control, proceed as follows:

1. Turn on the heater with the HEATER button.
2. Use the <scroll> button to select the set point (SP). After this step the LED switches from Process value to Set Point. Use the <Up> or <Down> keys to select a set point temperature between -190 °C and 600 °C.
3. The TTK 600 starts to heat/cool according to the settings. The set-point temperature will be held until the set-point is changed or the heater button is pushed (switching off the heater).
4. Wait until the set-point temperature has been reached. Depending on your experimental conditions, it may take some time until the sample temperature is stable due to the required heat transfer between sample holder and sample and the heat conduction through the sample.

8.7 Carrying out the Measurement

To perform the XRD measurement, proceed as follows:

1. Check whether CCU 100 displays a temperature close to room temperature.
2. Turn off the heater and remove the sample holder.
3. Prepare the sample and insert the sample holder with the sample.
4. Close the lid.
5. Turn on the heater.
6. If necessary, align the sample height at 25 °C before starting an experiment. This can be done by measuring the peak position of a known phase in the sample and re-adjusting the sample (chamber) height until the peak is at the correct 2θ-angle.

TIP: *If you measure powder samples and prepare the sample as described in chapter 8.2.5, it is not necessary to align the sample height.*

7. Create the desired experimental conditions.
8. Heat or cool your sample to the desired temperature.
9. Start the X-ray scan or batch program.

TIP : *Detailed information about diffractometer setup and X-ray scans are given in the instruction manual of your diffractometer.*

After you have finished your experiment, allow the instrument to reach 25 °C before you open the chamber and remove the sample.

CAUTION

Risk of injury.
During operation and even after turning off the instrument, parts inside the sample chamber can have extremely hot or cold surfaces. Make sure that the sample holder is at room temperature before handling it.

8.8 Temperature Control Information

This chapter provides general information related to heating and cooling of the samples.

8.8.1 Sample Temperature Accuracy

All specified temperature values in this instruction manual refer to the temperature of the sample holder and not the sample.

Depending on the operating conditions, the temperature of the sample surface probed with the X-ray beam can deviate significantly from the temperature of the sample holder displayed on CCU 100.

Especially for measurements with vacuum inside the TTK 600 sample chamber, the deviation can reach several 10 °C.

The deviation between the temperature of the sample surface and the displayed temperature mainly depends on the following properties:

- temperature range
- atmosphere inside TTK 600 (gas type, vacuum)
- sample (thermal properties, surface)
- condition of graphite foil

Anton Paar GmbH only ensures the accurate measurement and correct display of the sample holder temperature. All parts are checked before delivery.

In order to avoid experimental errors caused by the inevitable difference between sample holder temperature and sample surface temperature, Anton Paar GmbH strongly recommends that you do temperature validation measurements for your particular experimental conditions. Such validation measurements are done with reference materials with accurately known thermal properties, using either a crystalline phase transformation or thermal lattice expansion. Lists of reference materials with thermal data and instructions for doing validation measurements can be found in the XRD literature.

TIP: *Try to avoid measurements in vacuum, because temperature deviation in vacuum is much larger than if the chamber is filled with gas. Whenever possible, use dry nitrogen or helium gas instead of vacuum.*

8.8.2 Effective Heating and Cooling Rate

Independent of the maximum set-point rate (SPR) value which you can define, the maximum heating/cooling rate which you can actually achieve depends on your experimental conditions.

Due to the response time of the system, the CCU 100 needs some time to reach the pre-set heating/cooling rate (SPR). In addition, the heating/cooling rate must be reduced by the CCU 100 when the temperature approaches the temperature set-point to avoid temperature overshoot.

As a consequence:

- the total effective heating/cooling rate for the complete temperature step is a little smaller than SPR, depending on SPR and the amount of temperature change.
- it does not make sense to define small temperature changes with very large heating rates, because the effective heating rate is limited.

SPR should be chosen in such a way that the theoretical time for the temperature change is ≥ 1 min.

8.8.2.1 Standard Sample Holder

Exemplary maximum heating and cooling rates for the standard sample holder can be found in the graphs below.

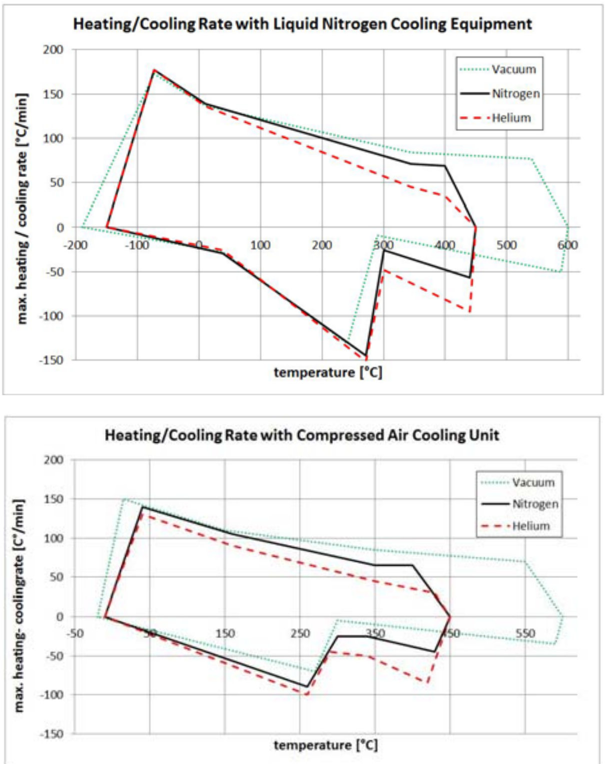


Fig. 61: Exemplary maximum heating and cooling rates for the configuration with liquid nitrogen cooling equipment (upper graph) and compressed-air cooling unit (lower graph) as a function of the temperature and the used atmosphere.

9 Putting TTK 600 out of Operation

When you stop using TTK 600 for a short period without removing it from the diffractometer, proceed as follows:

1. Cool down/heat up 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. When using the liquid nitrogen cooling equipment:

I. Turn off the low temperature equipment.

II. Leave the LN2 inlet connection in the Dewar until the system has reached room temperature again. In case that the liquid nitrogen of the Dewar has to be refilled, proceed as follows:

1. Take out the inlet connection of the Dewar.
2. Close the tube by using the delivered tube plug (1).
3. Refill the Dewar as described in the instruction manual of the Dewar.
4. Remove the plug of the inlet connection.

5. Place the inlet connection in the Dewar again.
6. Start the next experiment.

TIP: *The above mentioned procedures prevent condensation of moisture inside the liquid nitrogen cooling equipment.*

> **NOTICE**
> ***Risk of damage.***
> Some parts of the Low Temperature Equipment are covered with ice. Make sure that no water drops on electronic or otherwise sensitive parts and regularly wipe while it is warming up.

7. When using the compressed-air cooling equipment:

III. Turn off the compressed-air supply.

1. Turn off the cooling water.
2. Turn off CCU 100 completely.

In order to remove TTK 600 from the diffractometer continue as follows:

1. Completely turn off CCU 100 (mains switch).
2. Disconnect all electrical cables from the sample chamber.
3. Disconnect the cooling device.
4. Disconnect the couplings of the cooling water hoses.
5. Dismount the sample chamber from the goniometer.
6. Store the sample chamber in a safe place, preferably on a stage mount offered by Anton Paar GmbH.
7. When using the liquid nitrogen cooling equipment, follow the instruction for storing the liquid nitrogen container given in the container's instruction manual.

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 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.*
E13	Power Stage Fault	Contact Anton Paar GmbH.
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 TTK 600 (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 100 does not start after turning it 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 11.4)

2. Sensor Break (E01)

cause	action
temperature sensor cable not properly connected	Check temperature sensor cable and connectors.
Pt100 damaged	Visual check of Pt100 for damage.
plug of Pt100 cable inside the chamber disconnected	Connect the plug of Pt100 cable.
TTK 600 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. • Check whether the cooling equipment is switched off correctly above the threshold temperature.
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 TTK 600 to the cooling water circuit.
no **cooling water**	Turn on the cooling water.
flow controller disconnected	Connect the water flow controller to the CCU 100.
insufficient **cooling water supply**	Make sure the water pressure is between 2 – 3 bar and the flow rate is 0.7 – 1.5 l/min.
flow controller does not switch	⇒ Contact Anton Paar GmbH
cooling ducts inside **TTK 600** 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 TTK 600 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 <u>before TTK 600</u> meets the requirements. • Check if the cooling water flow <u>after TTK 600</u> meets the requirements. • If the flow resistance of the TTK 600 chamber is too high, contact Anton Paar GmbH.
cable to thermoswitch defective	⇒ Contact Anton Paar GmbH.

6. Insufficient Sample Cooling (air cooling and liquid nitrogen cooling)

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 ≥ 6 bar.
insufficient **compressed-air supply** to TTK 600	• Make sure compressed-air is turned on • Make sure all air hose connections on CCU 100 and TTK 600 are tight.
cryostat clogged with ice	Remove the compressed-air cooling unit, heat the sample holder inside TTK 600 to 200 °C for one hour and blow dry air or nitrogen through the cryostat. In case of the LN2 cooling equipment, remove the LN2 inlet connection from the Dewar. Close it by the use of the delivered plug and wait until all parts reached room temperature. After that heat the sample holder of TTK 600 to 200 °C for one hour and blow dry air or nitrogen through the cryostat by removing the plug and inserting a suitable gas hose in the inlet connection.

NOTICE

Risk of damage.

The pressure of the drying gas (dry air or nitrogen) **must not exceed 0.4 bar** during the drying of the cryostat.
Higher pressures could damage the heater of the instrument.

7. 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

8. Sensor Spread Failure (E11)

cause	action
Graphite foil between sample holder and heating/cooling block damaged	Change the graphite foil as described in chapter 11.3.2
Pt100 of the sample holder defect	a. Check this case by performing a measurement with another sample holder (if possible) b. ⇒ contact your diffractometer manufacturer or Anton Paar GmbH

9. No / Unknown instrument present (E98)

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

10.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.

10.4 Technical Support

If you need technical support, please contact the local representative of your diffractometer manufacturer or Anton Paar GmbH.

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

Always keep the chamber closed, otherwise the vacuum sealing in the housing, the heating/cooling block or the Pt100 thermo sensor can be damaged.

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

- Remove the protective cap of the vacuum feed through only shortly before fitting the appropriate connector.
- Periodically clean the chamber to remove residual sample material.
- Periodically check the cooling-water circuit.
- Periodically check the Pt100 temperature sensor for proper functioning. If required, validate the temperature with an appropriate standard.
- Periodically check the floater of the Liquid Nitrogen Evaporation Box for mobility by gently shaking the box and listening for the noise of the floater.
- Make sure that the O-ring of the housing (the seal between the housing and the lid) is always slightly coated with vacuum grease.
  Periodically clean the O-Rings, 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 5.5.

NOTICE
Risk of damage.
Do not operate the TTK 600 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 100 by using the power switch on the front side of the instrument.
2. Unplug all cables from the rear side of the instrument.

WARNING

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.

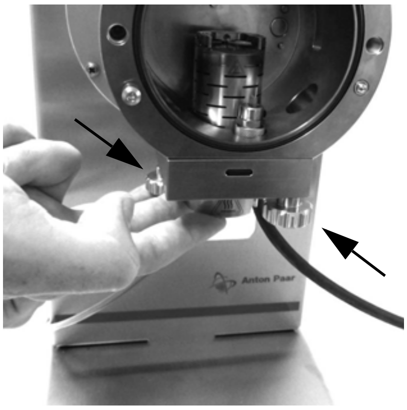
Perform the recommended maintenance work regularly to ensure the smooth long-term operation of the instrument.

11.3.2 Exchanging the Graphite Foil

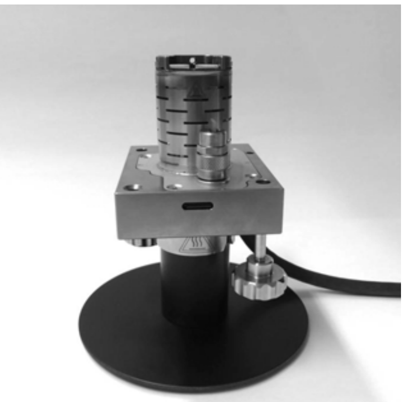
The graphite foil between the sample holder and the heating/cooling block ensures good heat transfer from the heater to the sample holder. Therefore it is essential to regularly check the condition of the graphite foil. It is recommended to exchange the graphite foil after several exchanges of the sample holder or if visible scratches can be seen on the foil.

In order to exchange the graphite foil proceed as follows:

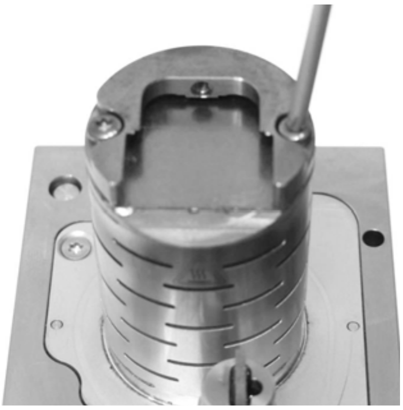
1. Remove the sample holder.
2. Remove connected cooling equipment from the instrument.
3. Demount the heating/cooling block of the instrument from the housing by unscrewing the knurled screws as shown in the following picture. Take care to hold the heating block while unscrewing the last screw. (Should the knurled screws at the bottom be stuck, use the supplied Torx 10 to loosen them.)



4. Place the heating/cooling block on the delivered mount as shown in the next picture



5. Remove the sample holder fixing ring by unscrewing the two screws with the supplied Torx driver.



6. Remove the sample holder fixation ring and the old graphite foil.
7. Replace the old graphite foil with a new one by placing it centrically over the heater.

Mount the instrument by reversing the above mentioned steps.

11.3.3 Replacing the Window Foil

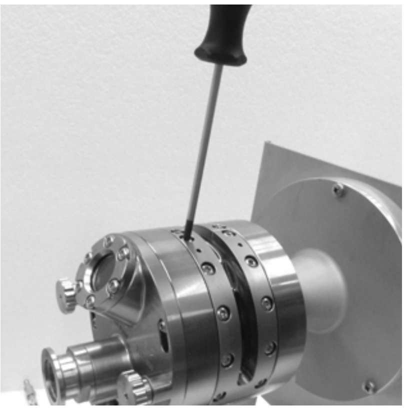
A Kapton foil with 50 µm thickness is used to cover the X-ray window in the housing. To seal this window the Kapton foil is pressed onto an O-ring with two clamping pieces.

CAUTION

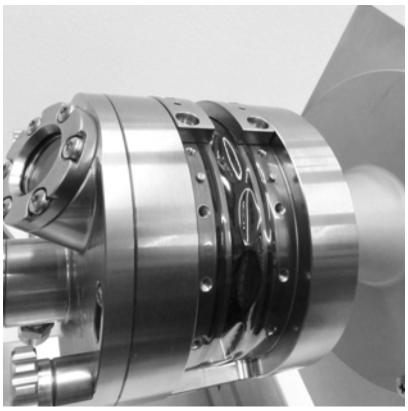
- Check and replace the window foil regularly.
- Once the foil of the radiation window is leaky or foggy, it has to be replaced. A set of foils is contained in the accessory box.

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

1. Unscrew the 20 screws that fix the clamping pieces to the housing of the instrument.



2. Remove the clamping pieces from the instrument.



3. Remove the used foil.

4. Remove the O-ring and use a suitable solvent, e.g. alcohol to clean both the O-ring and the groove.

5. Slightly coat the O-ring with vacuum grease and mount it again in the groove.
6. Place the window foil over the nipples that hold the foil in place.
   Make sure the holes for the screws in the foil are in the correct places over the holes for the screws of the chamber.
7. Place the clamping pieces back on the chamber.
8. Tighten the 20 screws beginning with the uppermost four.

11.3.4 Replacing the O-rings

Commercially available O-rings made of Viton are used.

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

Before fitting the new O-rings, clean the sealing surfaces with an appropriate solvent (e.g. alcohol) and slightly coat them using vacuum grease.

11.4 Checking and Replacing Mains Fuses

WARNING

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.

Appendix A: Technical Data

Temperature range

Upper limit:	
- Nitrogen, Air, inert gas:	+450 °C
- Vacuum (10^{-2}mbar):	+600 °C
Low limit with compressed-air cooling:	
- Nitrogen, helium, dry air:	-10 °C
- Vacuum (10^{-2} mbar):	-20 °C
Low limit with liquid nitrogen cooling:	
- Nitrogen, helium, dry air:	-150 °C
- Vacuum:	-190 °C

Atmospheres

Atmospheres:	air, inert gas, N_2 vacuum (10^{-4} mbar)
Pressure:	max. 2 bar relative

Temperature measurement & control

Control unit:	CCU 100
Temperature sensor1:	TC Type K
Temperature sensor2:	Pt100
Temperature accuracy[1)]:	≤ ±2 °C
Heating method:	Resistance heater
Cooling methods:	compressed-air cooling or liquid nitrogen cooling
Compressed-air consumption:	@ 6 bar 120 l/min
Liquid-nitrogen consumption:	max. 4 l/h

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

X-ray window

Scan range:	0 - 164° 2θ
Width / Length:	12 mm / 195°
Foil material:	Kapton (50 µm)

Sample Chamber Dimension and Materials

Diameter:	125 mm (122 mm depth)
Weight:	approx. 3 kg
Housing surface material:	nickel plated aluminum
Heating/cooling unit surface material	Nickel

Sample Holder

Sample holder surface:	Nickel
Sample cavity area (L x W):	14 x 10 mm
Sample cavity depth:	0,8 mm, 0,2 mm or flat (other depths on request)
Zero background holder:	Si(510), thickness 0.5 mm

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 TTK 600):

Voltage (mains):	AC 100-240 V
Frequency (mains):	50 ... 60 Hz
Power consumption:	max. 250 VA
Mains fuses:	2 x T 6,3A H 5x20 mm (ceramic tube)
Overvoltage category:	II according to EN 61010-1
Voltage output:	DC 48 V
Current output:	max. DC 4 A
Heater fuse:	2 x T4 A (SMD)

Cooling water requirements:

Flow rate:	0.7 to 1.5 l/min
Temperature:	15 to 25 °C
Pressure:	2 to 8 bar

Compressed-air requirements of air cooling:

Pressure:	6 - 8 bar rel.
Flow rate:	≥ 120 l/min at 6 bar
pressure dew point (@ 7 bar): - for operating temperature ≥ +5 °C - for operating temperature < +5 °C	at 7 bar < +25 °C < +10 °C
Purity (acc. DIN 8573-1):	solid particle class 3 or better oil class 2 or better

Compressed-air requirements for LN2 cooling:

Pressure:	> 2.5 bar rel.
Flow rate:	≥ 70 l/min at 2.5 bar

Dewar requirements:

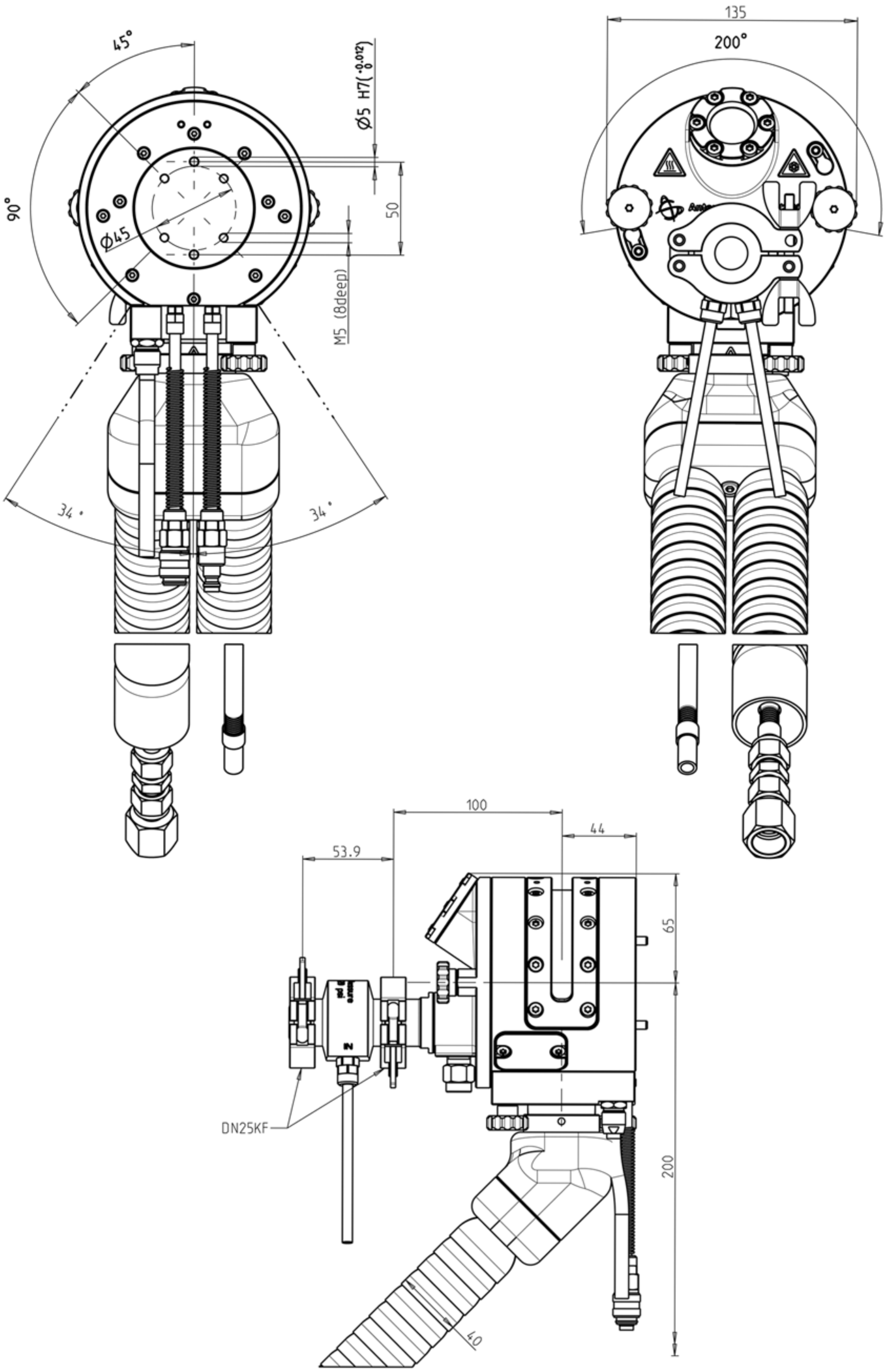
Self-evaporation rate:	> 0.2 L/d
Opening dimension:	DN 50 KF

Ambient conditions

Ambient temperature:	5 - 35 °C
Ambient humidity:	80 % max. not condensing
Maximum operating altitude:	3000 m above sea level
Pollution degree:	2 according to EN 61010-1

TTK 600 is for INDOOR USE ONLY!
Protect the instruments from moisture!

Appendix B: Sample Chamber Dimensions



Appendix C: 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:	**TTK 600 LOW TEMPERATURE CHAMBER + CCU 100 COMBINED CONTROL UNIT**
Model:	**TTK 600 + CCU 100**
Material number:	**159900 + 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, 2016-05-17

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Last modification 2016-05-17
Created by Bernhard Pammer

D52 TTK 600 + D54 CCU 100_CE Declaration B.docx
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Appendix D: Spare Parts and Accessories

Liquid nitrogen cooling equipment with accessories	
164578	LOW TEMPERATURE EQUIPMENT FOR VENTURI NOZZLE
166462	LIQUID NITROGEN CONTAINER, DEWAR 60L
166463	LIQUID NITROGEN CONTAINER, DEWAR 100L
Compressed-air cooling equipment with accessories	
165420	AIR-COOLING SET TTK 600
6931	AIR SERVICE UNIT
81393	COMPRESSED-AIR DRYER
Vacuum equipment	
58974	STANDARD VACUUM EQUIPMENT
58963	HIGH VACUUM EQUIPMENT
Accessories and spare parts	
159930	STANDARD SAMPLE HOLDER WITH Pt100 (0,8mm DEPTH)
162262	STANDARD SAMPLE HOLDER TTK 600 WITH Pt100 (0,2mm DEPTH)
162263	STANDARD SAMPLE HOLDER TTK 600 WITH Pt100 (FLAT)
163290	CAPILLARY SAMPLE HOLDER TTK 600
164863	TRANSMISSION SAMPLE HOLDER TTK 600
25420	ZERO BACKGROUND INSERT TTK
164864	SET OF GRAPHITE FOILS TTK 600 (10 PCS)
70175	VACUUM GREASE 20g
164865	SET OF KAPTON FOILS (5 pcs)
164866	SET OF O-RINGS
8415	FILTER CARTRIDGE (0.01 µm PORE SIZE)
165748	SPARE PARTS PACKAGE TTK 600
166554	CORNER VALVE TTK 600
168401	STANDARD SAMPLE HOLDER HEATING ENVIRONMENT
166940	ANTECHAMBER TTK 600
171089	SET OF FOILS FOR HEATING ENVIRONMENT
171085	SET OF FOILS FOR TRANSMISSION SAMPLE HOLDER
162029	ALIGNMENT SAMPLE HOLDER
166148	RIGID HEIGHT ALIGNMENT TOOL

176383	COOLING MODE SWITCHING VALVE

Appendix E: Safety Data Sheet for Liquid Nitrogen

MATERIAL SAFETY DATA SHEET

Prepared to U.S. OSHA, CMA, ANSI and Canadian WHMIS Standards

1. PRODUCT IDENTIFICATION

CHEMICAL NAME; CLASS: LIQUID NITROGEN

SYNONYMS: Nitrogen NF; LIN; Cryogenic Liquid Nitrogen; Nitrogen, Refrigerated Liquid
CHEMICAL FAMILY NAME: Inert Gas
FORMULA: N_2

2. COMPOSITION and INFORMATION ON INGREDIENTS

CHEMICAL NAME	CAS #	mole %	EXPOSURE LIMITS IN AIR					
			ACGIH-TLV		OSHA-PEL		NIOSH	OTHER
			TWA ppm	STEL ppm	TWA ppm	STEL ppm	IDLH ppm	ppm
Nitrogen	7727-37-9	99.99 %	There are no specific exposure limits for Nitrogen. Nitrogen is a simple asphyxiant (SA). Oxygen levels should be maintained above 19.5%.					
Maximum Impurities		<0.01%	None of the trace impurities in Liquid Nitrogen contribute significantly to the hazards associated with the product. All hazard information pertinent to Liquid Nitrogen has been provided in this Material Safety Data Sheet, per the requirements of the OSHA Hazard Communication Standard (29 CFR 1910.1200) and State equivalents standards.					

NE = Not Established. See Section 16 for Definitions of Terms Used.
NOTE: ALL WHMIS required information is included in appropriate sections based on the ANSI Z400.1-1998 format. This product has been classified in accordance with the hazard criteria of the CPR and the MSDS contains all the information required by the CPR.

3. HAZARD IDENTIFICATION

EMERGENCY OVERVIEW: Liquid Nitrogen is a colorless, odorless, cryogenic liquid. The main health hazard associated with releases of this gas is asphyxiation, by displacement of oxygen. The cryogenic liquid will rapidly boil to the gas at standard temperatures and pressures. The liquefied gas can cause freezing of tissue, or cryogenic burns , similar to frostbite to eyes or skin upon contact.

HAZARDOUS MATERIAL IDENTIFICATION SYSTEM

HEALTH HAZARD	(BLUE)	3
FLAMMABILITY HAZARD	(RED)	0
PHYSICAL HAZARD	(YELLOW)	0

PROTECTIVE EQUIPMENT

EYES	RESPIRATORY	HANDS	BODY
	SEE SECTION 8		SEE SECTION 8

For Routine Industrial Use and Handling Applications

SYMPTOMS OF OVER-EXPOSURE BY ROUTE OF EXPOSURE: The most significant routes of over-exposure for this gas are by inhalation, and contact with the cryogenic liquid.

INHALATION: High concentrations of this gas can cause an oxygen-deficient environment. Individuals breathing such an atmosphere may experience symptoms which include headaches, ringing in ears, dizziness, drowsiness, unconsciousness, nausea, vomiting, and depression of all the senses. Under some circumstances of over-exposure, death may occur. The following effects associated with various levels of oxygen are as follows:

<u>**CONCENTRATION**</u>	<u>**SYMPTOM OF EXPOSURE**</u>
12-16% Oxygen:	Breathing and pulse rate increased, muscular coordination slightly disturbed.
10-14% Oxygen:	Emotional upset, abnormal fatigue, disturbed respiration.
6-10% Oxygen:	Nausea and vomiting, collapse or loss of consciousness.
Below 6%:	Convulsive movements, possible respiratory collapse, and death.

CONTACT WITH SKIN or EYES: Contact of the liquid with the skin can lead to severe cryogenic burns or dermatitis (red, cracked, irritated skin), depending upon concentration and duration of exposure. Contact of the liquid with the eyes can cause pain, redness, severe cryogenic burns, and prolonged exposure could cause blindness. Contact with the undiluted liquid will cause frostbite, ulceration of the skin (which may be delayed in appearance for several hours), blistering, and pain. Contact with rapidly expanding gas poses a frostbite hazard.

OTHER POTENTIAL HEALTH EFFECTS: Contact with cryogenic liquid or rapidly expanding gases (which are released under high pressure) may cause frostbite. Symptoms of frostbite include change in skin color to white or grayish-yellow. The pain after contact with liquid can quickly subside.

HEALTH EFFECTS OR RISKS FROM EXPOSURE: An Explanation in Lay Terms. Over-exposure to Nitrogen may cause the following health effects:

ACUTE: The most significant hazard associated with this gas is inhalation of oxygen-deficient atmospheres. Symptoms of oxygen deficiency include respiratory difficulty, ringing in ears, headaches, shortness of breath, wheezing, headache, dizziness, indigestion, nausea, and, at high concentrations, unconsciousness or death may occur. The skin of a victim of over-exposure may have a blue color. Contact with cryogenic liquid or rapidly expanding gases (which are released under high pressure) may cause frostbite. Symptoms of frostbite include change in skin color to white or grayish-yellow. The pain after contact with liquid can quickly subside.

CHRONIC: There are currently no known adverse health effects associated with chronic exposure to this gas.

TARGET ORGANS: ACUTE: Respiratory system. CHRONIC: None known.

4. FIRST-AID MEASURES

RESCUERS SHOULD NOT ATTEMPT TO RETRIEVE VICTIMS OF EXPOSURE TO LIQUID NITROGEN WITHOUT ADEQUATE PERSONAL PROTECTIVE EQUIPMENT. At a minimum, Self-Contained Breathing Apparatus should be worn.

Remove victim(s) to fresh air, as quickly as possible. Trained personnel should administer supplemental oxygen and/or cardio-pulmonary resuscitation, if necessary. Only trained personnel should administer supplemental oxygen. Victim(s) must be taken for medical attention. Rescuers should be taken for medical attention, if necessary. Take copy of label and MSDS to physician or other health professional with victim(s).

4. FIRST-AID MEASURES (Continued)

EYE EXPOSURE: If liquid is splashed into eyes, or if irritation of the eye develops after exposure to liquid or gas, open victim's eyes while under gentle running water. Use sufficient force to open eyelids. Have victim "roll" eyes. Minimum flushing is for 15 minutes. Seek medical assistance immediately, preferably an ophthalmologist.

SKIN EXPOSURE: In case of frostbite remove any clothing that may restrict circulation to any frozen area. Do not rub frozen parts as tissue damage may occur. As soon as practicable, place any affected area in warm water bath which has a temperature that does not exceed 105°F (40°C). NEVER USE HOT WATER. NEVER USE DRY HEAT. If area of frostbite is extensive, and if possible, remove clothing while showering with warm water. If warm water is not available, or is impractical to use, wrap the affected parts gently in blankets. Alternatively, if the fingers or hands are frostbitten, place the affected area of the body in the armpit. Encourage victim to gently exercise the affected part while being warmed. Seek immediate medical attention.

Frozen tissue is painless and appears waxy, with a possible yellow color. Frozen tissue will become swollen, painful and prone to infection when thawed. If the frozen part of the body has been thawed by the time medical attention has been obtained, cover the area with a dry sterile dressing and a large bulky protective covering.

MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE: Pre-existing respiratory conditions may be aggravated by over-exposure to Liquid Nitrogen.

RECOMMENDATIONS TO PHYSICIANS: Treat symptoms and reduce over-exposure.

5. FIRE-FIGHTING MEASURES

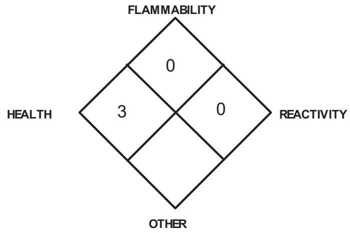
FLASH POINT: Not applicable.

AUTOIGNITION TEMPERATURE: Not applicable.

FLAMMABLE LIMITS (in air by volume, %):

Lower (LEL): Not applicable.
Upper (UEL): Not applicable.

NFPA RATING



FIRE EXTINGUISHING MATERIALS: Non-flammable, inert cryogenic liquid. Use extinguishing media appropriate for surrounding fire.

RESPONSE TO FIRE INVOLVING CRYOGEN: Cryogenic liquids can be particularly dangerous during fires because of their potential to rapidly freeze water. Careless use of water may cause heavy icing. Furthermore, the relatively warm water greatly increases the evaporation rate of Nitrogen. If large concentrations of Nitrogen gas are present, the water vapor in the surrounding air will condense, creating a dense fog that may make it difficult to find fire exits or equipment. Liquid Nitrogen, when exposed to the atmosphere, will produce a cloud of ice/fog in the air upon its release.

UNUSUAL FIRE AND EXPLOSION HAZARDS: Nitrogen does not burn; however, containers, when involved in fire, may rupture or burst in the heat of the fire. Liquid Nitrogen when accidentally released will vaporize rapidly, forming an oxygen deficient vapor cloud. Evacuate this vapor cloud area. Visibility may be obscured in its vapor cloud. Pressure in a container can build-up due to heat and it may rupture if pressure relief devices should fail to function. Contact with cold liquid or gaseous Nitrogen may cause frostbite.

Explosion Sensitivity to Mechanical Impact: Not Sensitive.
Explosion Sensitivity to Static Discharge: Not Sensitive.

SPECIAL FIRE-FIGHTING PROCEDURES: Structural fire-fighters must wear Self-Contained Breathing Apparatus and full protective equipment. If possible, remove Nitrogen cryogenic containers from fire area or cool with water. Do not direct water spray at the container vent. Evacuate area. Other information for pre-planning can be found in the North American Emergency Response Guidebook.

6. ACCIDENTAL RELEASE MEASURES

RESPONSE TO CRYOGENIC RELEASE: Clear the affected area and allow the liquid to evaporate and the gas to dissipate. After the gas is formed, follow the instructions provided below. Alternatively, to increase the rate of vaporization, spray large amounts of water on to the leak from an upwind position. If the area must be entered by emergency personnel, SCBA, leather or insulated gloves, and safety shoes must be worn. Personnel responding to a release must avoid all contact with the liquid.

Minimum Personal Protective Equipment should be **Level B: leather or thermally insulated gloves and Self-Contained Breathing Apparatus.** Locate and seal the source of the leaking gas. Allow the gas, which is lighter than air to dissipate. Monitor the surrounding area for oxygen level. The atmosphere must have at least 19.5 percent oxygen before personnel can be allowed in the area without Self-Contained Breathing Apparatus.

If leaking incidentally from the container or valve, contact your suppler.

7. HANDLING and USE

WORK PRACTICES AND HYGIENE PRACTICES: Be aware of any signs of dizziness or fatigue; exposures to fatal concentrations of Liquid Nitrogen could occur without any significant warning symptoms, due to oxygen deficiency.

STORAGE AND HANDLING PRACTICES: Cryogenic containers should be stored in dry, well-ventilated areas away from sources of heat, ignition and direct sunlight. Store containers away from heavily trafficked areas and emergency exits. Store away from process and production areas, away from elevators, building and room exits or main aisles leading to exits. Protect containers against physical damage.

Containers should be stored upright and be firmly secured to prevent falling or being knocked-over. Containers can be stored in the open, but in such cases, should be protected against extremes of weather and from the dampness of the ground to prevent rusting. Cryogenic containers are equipped with pressure relief devices to control internal pressure. Under normal conditions, these containers will periodically vent small amounts of product. Some metals such as carbon steel may become brittle at low temperatures and will easily fracture. Prevent entrapment of liquid in closed systems or piping without pressure relief devices. Use a check valve or other protective device in the discharge line to prevent hazardous backflow. Never tamper with pressure relief valves and containers.

Keep the smallest amount on-site as necessary. Full and empty containers should be segregated. Use a first-in, first-out inventory system to prevent full containers from being stored for long periods of time.

SPECIAL PRECAUTIONS FOR HANDLING CRYOGENIC CONTAINERS: Cryogenic liquids can present significant safety hazards. Never allow any unprotected part of the body to touch uninsulated pipes or vessels which contain cryogenic fluids. The extremely cold metal of the container will cause the flesh to stick fast and tear when one attempts to withdraw from it. The following rules are applicable to work situations in which cryogenic containers are being used.

Before Use: Move containers a suitable hand-truck. Do not drag, slide or roll containers. Do not drop containers or permit them to strike each other. Secure containers firmly.

During Use: Use designated CGA fittings and other support equipment. Do not use adapters. Do not use oils or grease on valve fittings or equipment. Leak-check system with leak detection solution. Immediately contact the supplier if there are any difficulties associated with operating container valve.

After Use: Close main container valve. Mark empty container “EMPTY”.

NOTE: Use only DOT or ASME code containers designed for cryogenic gas storage. Close valve after each use and when empty. Containers must not be recharged except by or with the consent of owner. For additional information, refer to Section 16, Other Information, for additional available literature.

OTHER SPECIAL PRECAUTIONS: Use piping and equipment adequately designed to withstand pressures and temperatures to be encountered. Use a check valve or other protective apparatus in any line or piping from the container to prevent reverse flow. To prevent cryogenic liquids or cold gas from being trapped in piping between valves, the piping shall be equipped with pressure relief devices. Only transfer lines designed for cryogenic liquids shall be used. It is recommended that all vents be piped to the exterior of the building.

STANDARD VALVE CONNECTIONS FOR U.S. AND CANADA: Use the proper CGA connections, DO NOT USE ADAPTERS:

THREADED:	CGA 295
PIN-INDEXED YOKE:	Not applicable.
ULTRA HIGH INTEGRITY:	Not applicable.

PROTECTIVE PRACTICES DURING MAINTENANCE OF CONTAMINATED EQUIPMENT: Follow practices indicated in Section 6 (Accidental Release Measures). Make certain application equipment is locked and tagged-out safely. Always use product in areas where adequate ventilation is provided.

8. EXPOSURE CONTROLS - PERSONAL PROTECTION

VENTILATION AND ENGINEERING CONTROLS: Use with adequate ventilation. Local exhaust ventilation is preferred, because it prevents gas dispersion into the work place by eliminating it at its source. If appropriate, install automatic monitoring equipment to detect the level of oxygen.

RESPIRATORY PROTECTION: Maintain oxygen levels above 19.5% in the workplace. Use supplied air respiratory protection if oxygen levels are below 19.5% or during emergency response to a release of Liquid Nitrogen. If respiratory protection is required, follow the requirements of the Federal OSHA Respiratory Protection Standard (29 CFR 1910.134), or equivalent State standards.

HAND PROTECTION: Wear loose-fitting, thermally insulated or leather gloves. Otherwise, wear glove protection appropriate to the specific operation for which Liquid Nitrogen is used. If necessary, refer to U.S. OSHA 29 CFR 1910.138 and appropriate Standards of Canada.

8. EXPOSURE CONTROLS - PERSONAL PROTECTION (Continued)

BODY PROTECTION: Use body protection appropriate for task. Safety shoes are recommended when handling containers, as well as long sleeve shirts and trousers. Safety shoes are recommended when handling cylinders. If a hazard of injury to the feet exists due to falling objects, rolling objects, where objects may pierce the soles of the feet or where employee's feet may be exposed to electrical hazards, use foot protection, as described in U.S. OSHA 29 CFR 1910.136.

9. PHYSICAL and CHEMICAL PROPERTIES

GAS DENSITY @ 0°C (32°F) and 1 atm: .072 lbs/cu ft (1.153 kg/m^3)
BOILING POINT: -195.8°C (-320.4°F)
FREEZING/MELTING POINT @ 10 psig: -210°C (-345.8°F)
SPECIFIC GRAVITY (air = 1) @ 21.1°C (70°F): 0.906
SOLUBILITY IN WATER vol/vol @ 0°C (32°F) and 1 atm: 0.023
EVAPORATION RATE (nBuAc = 1): Not applicable.
ODOR THRESHOLD: Not applicable. Odorless.
VAPOR PRESSURE @ (21.1°C) 70°F psig: Not applicable.
COEFFICIENT WATER/OIL DISTRIBUTION: Not applicable.
pH: Not applicable.
MOLECULAR WEIGHT: 28.01
EXPANSION RATIO: Not applicable.
SPECIFIC VOLUME (ft^3/lb): 13.8
APPEARANCE, ODOR AND COLOR: Liquid Nitrogen is a colorless, odorless cryogenic liquid.
HOW TO DETECT THIS SUBSTANCE (warning properties): There are no unusual warning properties associated with a release of Liquid Nitrogen, except the extreme cold, which may form a vapor cloud.

10. STABILITY and REACTIVITY

STABILITY: Normally stable in gaseous state. With cryogenic liquid, when exposed to air, oxygen in the air may condense into the Liquid Nitrogen. Liquid Nitrogen contaminated with oxygen may present the same hazards as Liquid Oxygen and could react violently with organic materials, such as oil and grease.
DECOMPOSITION PRODUCTS: None.
MATERIALS WITH WHICH SUBSTANCE IS INCOMPATIBLE: Titanium is the only element that will burn in Nitrogen. Lithium reacts slowly with Nitrogen at ambient temperatures. Also, use of Liquid Nitrogen in cryogenic grinding of fatty materials can lead to an explosion. A mixture of magnesium powder and Liquid Nitrogen reacts very violently when lit with a fuse, forming magnesium nitride. Liquid Nitrogen is not corrosive to metals, but the extreme cold can make some metals brittle.
HAZARDOUS POLYMERIZATION: Will not occur.
CONDITIONS TO AVOID: Contact with incompatible materials. Cryogenic containers exposed to high temperatures or direct flame can rupture or burst.

11. TOXICOLOGICAL INFORMATION

TOXICITY DATA: The following toxicology data for pure Nitrogen are listed below.

Eye Irritation (rabbit): Liquid Nitrogen poured into the eye for one or two seconds with the lids held apart, produced no discernible injury. When the exposure was extended to five seconds, slight lesions of the cornea were observed. By the next day, all eyes were entirely normal.

SUSPECTED CANCER AGENT: Nitrogen is not found on the following lists: FEDERAL OSHA Z LIST, NTP, CAL/OSHA, IARC; therefore it is not considered to be, nor suspected to be a cancer-causing agent by these agencies.
IRRITANCY OF PRODUCT: Contact with the cryogenic liquid or rapidly expanding gases can cause frostbite and damage to exposed skin and eyes.
SENSITIZATION OF PRODUCT: Nitrogen is not a sensitizer.
Listed below is information concerning the effects of Liquid Nitrogen on the human reproductive system.
Mutagenicity: Nitrogen is not reported to cause mutagenic effects in humans.
Embryotoxicity: Nitrogen is not reported to cause embryotoxic effects in humans.
Teratogenicity: Nitrogen is not reported to cause teratogenic effects in humans.
Reproductive Toxicity: Nitrogen is not reported to cause adverse reproductive effects in humans.

A mutagen is a chemical which causes permanent changes to genetic material (DNA) such that the changes will propagate through generation lines. An embryotoxin is a chemical which causes damage to a developing embryo (i.e. within the first eight weeks of pregnancy in humans), but the damage does not propagate across generational lines. A teratogen is a chemical which causes damage to a developing fetus, but the damage does not propagate across generational lines. A reproductive toxin is any substance which interferes in any way with the reproductive process.

BIOLOGICAL EXPOSURE INDICES (BEIs): Currently, Biological Exposure Indices (BEIs) have not been determined for Nitrogen.

12. ECOLOGICAL INFORMATION

ENVIRONMENTAL STABILITY: Nitrogen occurs naturally in the atmosphere. The gas will be dissipated rapidly in well-ventilated areas.
EFFECT OF MATERIAL ON PLANTS or ANIMALS: Any adverse effect on animals would be related to oxygen deficient environments, or the extreme cold of the cryogenic gas.. No adverse effect is anticipated to occur to plant-life, except for frost produced in the presence of rapidly expanding gases, or freezing from direct exposure to the cryogenic liquid.
EFFECT OF CHEMICAL ON AQUATIC LIFE: As an inert gas, this product would have no effect on aquatic life.

13. DISPOSAL CONSIDERATIONS

PREPARING WASTES FOR DISPOSAL: Waste disposal must be in accordance with appropriate Federal, State, and local regulations. Return cryogenic containers with any residual product to Air Liquide. Do not dispose of locally.
For emergency disposal, discharge slowly to the atmosphere in a well-ventilated area or outdoors.

14. TRANSPORTATION INFORMATION

THIS GAS IS HAZARDOUS AS DEFINED BY 49 CFR 172.101 BY THE U.S. DEPARTMENT OF TRANSPORTATION.

PROPER SHIPPING NAME:	Nitrogen, refrigerated liquid
HAZARD CLASS NUMBER and DESCRIPTION:	2.2 (Non-Flammable Gas)
UN IDENTIFICATION NUMBER:	UN 1977
PACKING GROUP:	Not Applicable
DOT LABEL(S) REQUIRED:	2.2 (Non-Flammable Gas)

NORTH AMERICAN EMERGENCY RESPONSE GUIDEBOOK NUMBER (2000): 120
MARINE POLLUTANT: Nitrogen is not classified by the DOT as a Marine Pollutant (as defined by 49 CFR 172.101, Appendix B).
SPECIAL SHIPPING INFORMATION: Cryogenic containers should be transported in a secure position, in a well-ventilated vehicle. The transportation of compressed gas cryogenic containers in automobiles or in closed-body vehicles present serious safety hazards and should be discouraged.
NOTE: Shipment of compressed gas cryogenic containers which have not been filled with the owners consent is a violation of Federal law (49 CFR, Part 173.301 (b).

TRANSPORT CANADA TRANSPORTATION OF DANGEROUS GOODS REGULATIONS: This gas is considered as Dangerous Goods, per regulations of Transport Canada. The use of the above U.S. DOT information from the U.S. 49 CFR regulations is allowed for shipments that originate in the U.S. For shipments via ground vehicle or rail that originate in Canada, the following information is applicable.

PROPER SHIPPING NAME:	Nitrogen, refrigerated liquid
HAZARD CLASS NUMBER and DESCRIPTION:	2.2 (Non-Flammable Gas)
UN IDENTIFICATION NUMBER:	UN 1977
PACKING GROUP:	Not Applicable
HAZARD LABEL(S) REQUIRED:	2.2 (Non-Flammable Gas)
SPECIAL PROVISIONS:	None
EXPLOSIVE LIMIT & LIMITED QUANTITY INDEX:	0.12
ERAP INDEX:	None
PASSENGER CARRYING SHIP INDEX:	None

PASSENGER CARRYING ROAD OR RAIL VEHICLE INDEX: 75
MARINE POLLUTANT: Nitrogen is not a Marine Pollutant.

15. REGULATORY INFORMATION

ADDITIONAL U.S. REGULATIONS:
U.S. SARA REPORTING REQUIREMENTS: Liquid Nitrogen is not subject to the reporting requirements of Sections 302, 304 and 313 of Title III of the Superfund Amendments and Reauthorization Act.
U.S. SARA THRESHOLD PLANNING QUANTITY: There are no specific Threshold Planning Quantities for this gas. The default Federal MSDS submission and inventory requirement filing threshold of 10,000 lb (4,540 kg) may apply, per 40 CFR 370.20.
U.S. TSCA INVENTORY STATUS: Nitrogen is listed on the TSCA Inventory.
U.S. CERCLA REPORTABLE QUANTITIES (RQ): Not applicable.

15. REGULATORY INFORMATION (Continued)

ADDITIONAL U.S. REGULATIONS (continued):
OTHER U.S. FEDERAL REGULATIONS:

- Generally recognized as safe (GRAS), as a direct human food ingredient when used as a propellant, aerating agent and gas, per 21, CFR, 184.1540. Nitrogen NF is regulated by the FDA as a prescription drug.
- Nitrogen does not contain any Class I or Class II ozone depleting chemicals (40 CFR part 82).
- Depending on specific operations involving the use of Liquid Nitrogen, the regulations of the Process Safety Management of Highly Hazardous Chemicals may be applicable (29 CFR 1910.119). Under this regulation Nitrogen is not listed in Appendix A.
- Nitrogen is not listed as a Regulated Substance, per 40 CFR, Part 68, of the Risk Management for Chemical Releases.

U.S. STATE REGULATORY INFORMATION: Nitrogen is covered under the following specific State regulations:

Alaska - Designated Toxic and Hazardous Substances: No.
California - Permissible Exposure Limits for Chemical Contaminants: Nitrogen.
Florida - Substance List: No.
Illinois - Toxic Substance List: No.
Kansas - Section 302/313 List: No.
Massachusetts - Substance List: No.
Minnesota - List of Hazardous Substances: No.
Missouri - Employer Information/Toxic Substance List: No.
New Jersey - Right to Know Hazardous Substance List: Nitrogen.
North Dakota - List of Hazardous Chemicals, Reportable Quantities: No.
Pennsylvania - Hazardous Substance List: Nitrogen.
Rhode Island - Hazardous Substance List: Nitrogen.
Texas - Hazardous Substance List: No.
West Virginia - Hazardous Substance List: No.
Wisconsin - Toxic and Hazardous Substances: No.

CALIFORNIA SAFE DRINKING WATER AND TOXIC ENFORCEMENT ACT (PROPOSITION 65: Liquid Nitrogen is not on the California Proposition 65 lists.
ADDITIONAL CANADIAN REGULATIONS:
CANADIAN DSL/NDSL INVENTORY STATUS: Liquid Nitrogen is included in the DSL Inventory.
CANADIAN ENVIRONMENTAL PROTECTION ACT (CEPA) PRIORITY SUBSTANCES LISTS: Liquid Nitrogen is not on the CEPA Priorities Substances Lists.
WHMIS CLASSIFICATION: Liquid Nitrogen is categorized as a Controlled Product, Hazard Class A, as per the Controlled Product Regulations.
OTHER CANADIAN REGULATIONS: Not applicable.

16. OTHER INFORMATION

MIXTURES: When two or more gases or liquefied gases are mixed, their hazardous properties may combine to create additional, unexpected hazards. Obtain and evaluate the safety information for each component before you produce the mixture. Consult an Industrial Hygienist or other trained person when you make your safety evaluation of the end product. Remember, gases and liquids have properties which can cause serious injury or death.
Further information about Nitrogen can be found in the following pamphlets published by: Compressed Gas Association Inc. (CGA), 4221 Walney Road 5th floor, Chantilly, VA 20151-2923. Telephone: (703) 788-2700.

G-10.1	*"Commodity Specification for Nitrogen"*
P-1	*"Safe Handling of Compressed Gases in Containers"*
P-9	*"Inert Gases, Argon, Nitrogen and Helium"*
P-12	*"Safe Handling of Cryogenic Liquids"*
P-14	*"Accident Prevention in Oxygen-Rich, Oxygen-Deficient Atmospheres"*
SB-2	*"Oxygen Deficient Atmospheres "*
AV-1	*"Safe Handling and Storage of Compressed Gases"*
AV-5	*"Safe Handling of Liquefied Nitrogen and Argon"*
	"Handbook of Compressed Gases"

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AIR LIQUIDE

Appendix F: Warranty

The warranty regulations for TTK 600 are in accordance with the „General Terms of Delivery“ of the Austrian Electrical and Electronic Industry.