NETZSCH

Proven Excellence.

Operating Manual

DSC 214

Polyma® / Nevio

35689 / Version 1.4 / November 2020

DOCUMENTATION

206030005 80032061 2020-12-03

EC declaration of conformity

according to the EU Machinery Directive 2006/42/EC, Annex II 1. A

NETZSCH

Manufacturer

NETZSCH-Gerätebau GmbH

Wittelsbacher Str. 42

DE - 95100 Selb

Person established in the Community authorised to compile the relevant technical documentation

NETZSCH-Gerätebau GmbH

Wittelsbacher Str. 42

DE - 95100 Selb

Description and identification of the machinery

Product / Article	Differential Scanning Calorimeter
Serial number	ab SN.DSC21400A-0010-L
Project number	PRJ-2015-01-22-0002
Commercial name	DSC214 Polyma / DSC214 Nevio
Model	DSC21400A00.000-00
Function	Differential Scanning Calorimetry (DSC) determines transition temperatures and changes of enthalpy in solids and liquids during controlled temperature changes.

It is expressly declared that the machinery fulfils all relevant provisions of the following EU Directives or Regulations:

2006/42/EC	Directive 2006/42/EC of the European Parliament and of the Council of 17 May 2006 on machinery, and amending Directive 95/16/EC (recast) (1) Published in L 157/24 of 09.06.2006
2014/30/EU	Directive 2014/30/EU of the European Parliament and of the Council of 26 February 2014 on the harmonisation of the laws of the Member States relating to electromagnetic compatibility (recast) Published in L 96/79 of 29.03.2014

Reference to the harmonised standards used, as referred to in Article 7 (2):

EN ISO 12100:2010-11	Safety of machinery - General principles for design - Risk assessment and risk reduction (ISO 12100:2010)

Selb, 05.12.2018

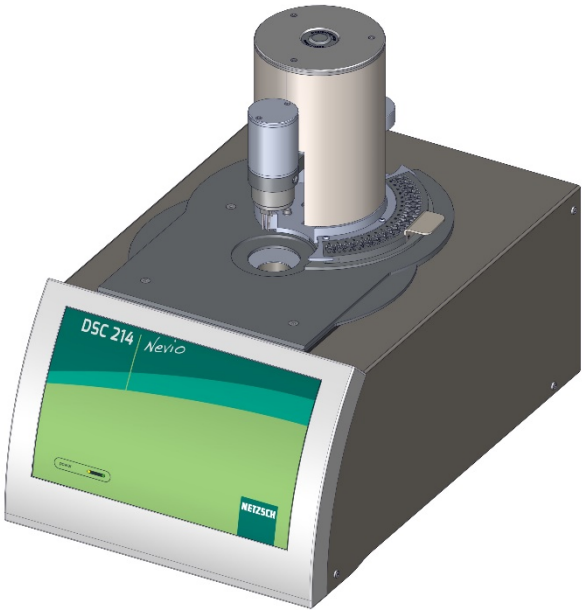
Place, Date

Signature
Dr. Thomas Denner
managing director

Dear Customer,

congratulations to the purchase of your NETZSCH Instrument.
Thank you for the trust you have placed in us, you have made the right decision.

This manual includes additional information regarding the *Nevio* instruments, specifically dedicated for applications in the fields of pharmacy, cosmetics and food.
Supplementary notes describe further information and deviating operating procedures to standard instruments.



CONTENTS

DSC 214

NETZSCH

Chapter I – General Information & Safety Instruction

206030005 80032061 2020-12-03

Information

Prior to installation and operation of your measuring system or individual components, intently read this generally admitted chapter. The content of this chapter may exceed the information required for your instrument.
Used notes, information and symbols in the following chapters vary corresponding to your instrument configuration.

Duplication

Any duplication or transmission of this document, both electronic and mechanical, requires a written prior permission of NETZSCH-Gerätebau GmbH.

Technical Data

All technical data, instrument features and other information described in these operating instructions are compiled with great diligence, elaborated to the best of our knowledge and corresponding to the instruments' technical standards at the time of printing. We reserve the right of technical modifications.

Proposals

We welcome any comments, suggestions or new ideas concerning the instrument, its operating instructions as well as software or service:

NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42
D - 95100 SELB
Phone: 09287/881- 0
Fax: 09287/881- 144
E-Mail: at@netzsch.com
Internet: http://www.netzsch-thermal-analysis.com

Service and Maintenance

Service and maintenance have to be executed by NETZSCH Customer Service personnel.

It is our pleasure to offer service contracts for our customers.

In case there is no existing service contract for your device, we invite you to send us back the form enclosed with the appliance documentation.

Safety Instructions

State-of-the-Art	Your instrument has been produced with state-of-the-art technology and is safe to operate.
Authorized Operation	Any operation of the instrument other than as authorized requires consultation with NETZSCH. Any use exceeding the (expanded) authorized operation is considered unauthorized. The manufacturer will not be liable for any damage resulting from such use.
Manufacturer's Requirements	Authorized operation of the instrument includes compliance with manufacturer's requirements regarding installation, commissioning, operation and maintenance.
Qualified Personnel	Installation, use and maintenance of the instrument must be carried out by authorized personnel only. These personnel must be introduced to possible origin of danger.
Responsibility	Responsibility for commissioning, operation and maintenance must be clearly defined and compliance has to be ensured. The resulting responsibilities can be clearly resolved under the aspect of safety.
Unauthorized Access	The operator has to make sure that only trained personnel work on the instrument.
Improper operations	Any type of operation which interferes with the safety of the user and the operability of the instrument should be avoided.
Unauthorized Changes to the System	Unauthorized modifications and changes which affect the safety of the instrument are not permitted.
Maintenance Obligation	The operator of the system is committed to provide immaculate operating conditions at every time.
Proper Set-up of the Work Stations	The operator has to ensure clear arrangement and cleanliness of work stations at the instrument by means of appropriate instruction and inspections.

Shut-down	For all maintenance work, the instrument must be switched off and unplugged.
Electrical Energy Danger	The unit may only be opened by qualified personnel. Moreover it is absolutely essential to previously turn it off and disconnect it from power supply! Any work on electrical supply, electrical lines or electrical components has to be carried out by qualified personnel only (electrical professionals).
Removal of Protective Devices	Removing protective devices of the Instrument is solely permitted when switched off and unplugged. Furthermore, protective devices are imperative to be replaced before restarting the instrument.
Checks following Maintenance or Repair Work	After maintenance or repair work, a check is necessary to ensure that all protective devices are in place and operate properly. Only then should the instrument be restarted.
Industry-specific Accident Prevention Regulations	The operator must observe the relevant regulations and protective measures when handling required gases. In any case, the industry-specific and local accident prevention regulations are also valid for the instrument.
Disposal of Production Materials	Production materials are to be disposed of according to local regulations.
Preventive Maintenance or Repair	Products sent in for preventive maintenance or repair should, to the extent possible, be free of harmful substances (e.g. radioactive, toxic, caustic or microbiological materials). Otherwise, the type of contamination must be declared. Products not explicitly declared to be "free of harmful substances" will be decontaminated at the expense of the sender. Permission of NETZSCH-Gerätebau GmbH is required for repairs.
Operating Instruction	Using the Operation Manual, the operator should prepare an operating instruction which specifies the actions and tasks required for safe operation. The operating instruction should be placed in a suitable location at the work place and has to be performed by the employees.

Lighting at the Workplace

The lighting level on the working environment should be determined in such a way that safe work and an identification of risks at an early stage are possible at all times. The minimum provided lighting level amounts 300 lx in the laboratory and 500 lx on the working environment.

Gases

When handling with gases, take into account the relevant regulations and protective measures. Consider possible reactions with materials.

Warning signs should be posted in appropriate places and be in accordance with national and regional regulations.

Sealing material

The Chemical resistance of used sealing material is hardly to be obligatory estimated due to the multitude of inserted purge -and reaction gases, concentrations, temperatures and contaminations.

Please make sure they meet the requirements, since we cannot assume any guarantee.

Safety Symbols

Below described symbols could be used to simplify orientation in this manual.

 **NOTE:**	**NOTE:** This sets particularly important information apart from the rest of the text.
 **ATTENTION!**	**ATTENTION!** These instructions must be followed exactly to avoid injury to the user and damage to the instrument.
	This symbol refers to more detailed information which can be found elsewhere, e.g. in the Software Manual.
	The tools listed next to this symbol are required for the installation or modification of your instrument.
	**DANGER!** Hot surface! Danger of burn!
	**DANGER!** Cold surface! Danger of frostbite!
	**DANGER!** Danger of hand injury!
	**DANGER!** Danger of laser radiation!
	**DANGER!** Danger of optical radiation!
	**DANGER!** Danger of electric shock!
	**DANGER!** Toxic hazard!
	Refer to instruction manual / booklet
	Wear safety footwear
	Wear protective gloves
	Opaque eye protection must be worn

	Wear face shield
	Wear a mask
	**CAUTION!** The instrument may only be opened when it is turned off and disconnected from the power supply!

Information for the usage of Purge Gases

Fundamentally, using filler or purge gas requires consideration of the following safety-related information to avoid environmental hazards or damage to the instrument:

To reach operating temperatures of more than 1000°C it is inevitably necessary to use ceramic components in the reaction zone (sample / furnace room). These components are exposed to continuous changes in temperature. This might trigger crack formation and further lead to leakages, especially in the hot area. The mentioned defects are hardly to be calculated in advance due to special properties of ceramic components. As a result they can't be avoided through preventive maintenance (replacement).

Dry, inert gases are recommended for purging. Prior to the introduction of gas, a leakage test should be carried out whilst pre-evacuating the system in order to assure the purity of the sample atmosphere. We also recommend to pass the expelled purge gas into a suitable exhaust hood. Depending on the measuring conditions (sample material, atmosphere, temperature range), derivative products can also be formed due to thermal reactions when using inert gases. These products (e.g. HCN, CO, SO_2, dioxin), even in small amounts, are very detrimental to health and must not be allowed to pass into the work room.

The user must decide in advance whether toxic gases might be released during a measurement. Should that be the case, safety precautions are absolutely essential!

Humid gas mixtures may only be used if condensations inside furnace and measuring system can be avoided. If a humid gas mixture is cooling down, the water begins to condensate at a certain temperature. This temperature is called the dew point. The temperature must not fall below this limit. NETZSCH offers special furnace types.

Should the measurement necessitate the usage of special gases in the sample chamber, a safety check of the hazard potential of the gas or gas mixture is imperative. Therefore, the following aspects have to be taken into consideration:

- Do explosive gas mixtures or explosive compounds evolve when the sample has contact with oxygen (air)?
- Can be ensured that no toxic compounds will evolve from the gases or gas mixtures when using the chosen application temperatures? In this connection, possible reactions between the purge gas and the reaction gas should also be investigated.
- Is it exclusionary that the gases might corrode neither the leads nor the seals used in either the system or the surrounding equipment (valves, manometer or flow meter)? Otherwise leakages are to be expected.
- What side effects do the gases or gas mixtures have on the system's accessories? These effects might not result in leakages but could considerably increase wear and tear and further end up in a total failure of the system.

The following standards need to be observed and followed unconditionally:

ATTENTION!

Explosive gas mixtures may not on any term be utilized!

Gases or gas mixtures escaping from the measuring part unconditionally need to be deduced through am extractor hood.

The measuring part has to be purged sufficiently with inert gas after measurement.

Since restrictions for some components of the devices are valid in an oxygen free environment, we direly recommend consultation with the manufacturer. (E.g. certain heating elements cannot be operated up to their specified maximum temperature in inert environment since they are in need of oxide-formation for preserving the protective layer).

Information concerning the possibility of using various gases can be found in appropriate technical literature or can be requested from the manufacturer or retailer of gases.

Subsequent we offer a selection of purge gases including their limiting properties.

The list makes no claim to be complete.

Helium (He)	– chemically inert (inert gas) – no technical safety limitations
Argon (Ar)	– chemically inert (inert gas) – asphyxiating – no technical safety limitations – argon should not be used for low temperature investigations
Nitrogen N_2	– largely inert – asphyxiating – no technical safety limitations, but sample reactions are possible in the high temperature range
Air	– oxidizing – no technical safety limitations, but sample reactions are possible; application is possible above room temperature (maximum application temperature is determined by furnace material, sample carrier type, crucible material) – the oxygen could liquefy if used in the low temperature range (LN2)
Oxygen (O_2)	– Increases flammability (must not come into contact with fats or oils) – no technical safety limitations, but hefty reactions are possible with the sample; application is possible above room temperature (maximum application temperature is determined by furnace material, sample carrier type, crucible material) – liquefaction could occur in the low temperature range (LN2)

Carbon dioxide (CO_2)	– non-toxic – german occupational exposure limit value: 5000 ml/m³ (ppm) – non-flammable – application is possible above room temperature
Hydrogen (H_2)	– flammable – the instrument is not destined and suitable for usage in hydrogen atmosphere; exposable mixtures might be forming inside the system during the respective experiments
Ammonia (NH_3)	– toxic – german occupational exposure limit value: 20 ml/m³ (ppm) – flammable – danger of explosion when getting in contact with oxygen. – for safety reasons it is forbidden to use an instrument in ammonia atmosphere, that has been installed in the normal way (corrosion of seals is possible)
Carbon monoxide (CO)	– toxic – german occupational exposure limit value: 30 ml/m³ (ppm) – flammable – corrosive – for safety reasons it is forbidden to use an instrument that has been installed in the normal way CO - atmosphere
Hydrogen sulphide (H_2S)	– toxic – german occupational exposure limit value: 5 ml/m³ (ppm) – flammable, corrosive – for safety reasons it is forbidden to use an instrument that has been installed in the normal way H2S- atmosphere

Other reducing gases or gas mixtures

ATTENTION!

The instrument is unsuitable for usage in atmosphere of gas mixtures or reducing gases; performing such experiments might provoke explosive mixtures inside the system

Chlorine (Cl_2)	– very toxic – german occupational exposure limit value: 0,5 ml/m³ (ppm) – non-flammable, corrosive, caustic – for safety reasons it is forbidden to use an instrument that has been installed in the normal way in Cl2 - atmosphere
Hydrogen chloride (HCl)	– toxic – german occupational exposure limit value: 2 ml/m³ (ppm) – non-flammable, corrosive, caustic – for safety reasons it is forbidden to use an instrument that has been installed in the normal way in HCl- atmosphere
Sulphur dioxide (SO_2)	– toxic – german occupational exposure limit value: 1 ml/m³ (ppm) – non-flammable, corrosive – for safety reasons it is forbidden to use an instrument that has been installed in the normal way in SO2 - atmosphere
Fluorine (F_2)	– very toxic – german occupational exposure limit value: 1 ml/m³ (ppm) – encourages burning, corrosive, caustic – for safety reasons it is forbidden to use an instrument that has been installed in the normal way in F_2 - atmosphere
Hydrogen fluoride (HF)	– very toxic – german occupational exposure limit value: 1 ml/m³ (ppm) – very caustic, corrosive – for safety reasons it is forbidden to use an instrument that has been installed in the normal way in HF- atmosphere
Gaseous hydrocarbon	– can form explosive gas mixtures when getting in touch with air; for safety reasons it is forbidden to use an instrument that has been installed in the normal way in such atmosphere

The AGW rates indicated are based on TRGS 900, and published in the GESTIS-database! (*http://gestis.itrust.de*)
State: August 2015

Relevant national regulations are to be strictly adhered to!

Safety Regulations for Handling Nitrogen/ Liquid Nitrogen LN2

Characteristics

- Colorless, inert, harmful and incombustible liquid. Due to its odorlessness there´s a lack of noticeable signs for its suffocating impact.
- Leaking liquid is cryogenic (**-196°C**) and vaporizes rapidly.
- Liquid leads to frostbites and causes severe skin burn and eye damage.
- Gas is lighter than air.

Safety Instructions

- Handling a higher quantity of Nitrogen requires the availability of a self-contained breathing apparatus in or in front of the workspace.
- Fix an information sign!
- Treat containers, equipment and vessels carefully. Before refilling, make sure they are well dried off. Eliminate the risk of falling over. Do not operate vessels abruptly or jerkily. Perform leak tests at appropriate intervals.
- Icing of containers or equipment is to be removed by using warm air or hot water only. Do not apply direct flame or incandescent materials for defrosting.
- For transportation do solely employ low pressure containers isolated by glass wool or slag. For small quantities use metal-cased Dewar vessels!
- Filling or decanting, make sure to prevent spillage. Only use well dried containers!
- Pay attention to solely fill containers up to the upper limit (5 cm below the rim) to avoid excessive pressure.
- Discharge overfilled containers.
- In case of nutrient nitrogen ensure fresh air supply. Possibly provide for suction and if necessary use a heavy breathing apparatus.
- When expanding, there can occur rapid formation of cold fog that might spread widely.
- If leakage of a high quantity of Liquid Nitrogen occurs in a closed room (e.g. as a result of pipe break or vessel breach) leave the area immediately, provide sufficient fresh air supply and take immediate steps to eliminate the leak. Possibly make use of a self-contained breathing apparatus since there might be a lack of essential oxygen.
- Keep in mind that eating, drinking or storing aliments is prohibited in the working area.
- Protective clothing, safety glasses and possibly even protective screens and safety gloves need to be worn!
- Entering a container or tank that is suspected to contain Nitrogen urgently requires the employment of a self-contained

	breathing apparatus! – Breathing apparatuses containing filter inserts are, in any case, inadmissible. – Rotational Instruction of employees and inscription in the receipt book are necessary. Mind the employment ban according to regional regulations.
Information concerning case of fire or damage	– Neither gaseous nor Liquid Nitrogen are flammable and do not by themselves constitute a fire or explosion risk. – Firefighting claims to be coordinated in consideration of the vicinity. – Form a great security zone being located on the leeward side. – Apply self-contained breathing apparatus.
Information concerning health protection	– Notwithstanding the fact that Nitrogen is physiologically ineffective, an accumulation of about more than 81% impedes vital respiration, eventually causing suffocation. – Nitrogen doesn't announce itself by any irritant or warning effect. – Liquid Nitrogen causes frost damages. These are skin damages similar to burn injuries.
First aid	– Inform doctor or emergency hospital as quickly as possible. – Body party affected by the cold liquid need to be defrosted by water. Remove clothing carefully. – Do not rub affected skin parts. Cover them with sterile dressing. – Immediately provide fresh air in case of unconsciousness. If necessary perform artificial respiration. Keep airways free. – In case of unconsciousness place patient stably in side position, also for transportation.
Additional Information	– Mind safety regulations according to regional standards.

Never touch objects, which have been in contact with Liquid Nitrogen with your bare hands.
Always wear special safety glasses and gloves when handling Liquid Nitrogen.

Please pay attention to all information for handling LN2 described in the operating manuals.

Safety Regulations for LN2 Dewar Vessels

The dewar vessel complies with the current valid Directive for Pressure Equipment and meets requirements up to a pressure of 1.5 bar.

- The Dewar vessel is designed for use with Liquid Nitrogen only.
- For pressurization of the Dewar vessel use gaseous nitrogen exclusively (GN)!
- Not at any terms should the Dewar vessel be opened until the pressure has completely dissipated! Monitor the pressure reduction.
- The Dewar vessel has to be kept vertical always.
- Escaping Liquid Nitrogen might cause serious injuries!
- A closed room storing Liquid Nitrogen vessels constantly contains evaporated nitrogen.1 l Liquid Nitrogen releases about 700l nitrogen gas. Notwithstanding the fact that Nitrogen behaves inertly and has no toxic impact, it displaces the atmospheric oxygen. Should the rate of atmospheric oxygen drop below the minimum of 19%, danger of physical harm occurs.
- All rooms and areas housing vessels containing Liquid Nitrogen should be well ventilated at all times and equipped with at least one oxygen gauge. All personnel should be informed of the risks associated with the use of nitrogen.
- When removing the supply system from the Dewar vessel it can be extremely cold. **Danger of frostbite!** In any case wait until the supply system has heated up to room temperature.
- In order to avoid damages to the Dewar head / withdrawal head it is forbidden to use hot air instruments for deicing the components!
- Should it be impossible to let the cooling device completely dry down outside the storage vessel, a non-negligibly amount of water will accumulate inside. It is important to remove the water regularly by completely emptying the storage vessel, removing the ice/ water and letting the vessel dry down entirely.
- Only in-house transport is allowed when vessel is upright, unpressurized.
- Never seal the Dewar vessel hermetically. Use only the stopper provided, the Dewar head or withdrawal head.
- The Dewar vessel requires being transported emptily, in its original packaging and in compliance with the current national and international regulations. Never stack vessels on top of each other.

The Dewar vessel containing Dewar head / withdrawal head is characterized by several precautions to prevent possible dangers handling with LN2.

- Withdrawal head with integrated safety equipment:
  Possible excess pressure is degrading through a safety valve (opening pressure: 1.5 bar).

- LN2-cooler CC 200 F3, control head operating by means of compressed gas (N2) endowed with software driven magnetic valve:
  Possible excess pressure is degrading through a safety valve (opening pressure: 0.5 bar).

- Dewar head CC300 head with heating device enabling controllable generation of pressure (working pressure) in the storage vessel:
  Possible excess pressure is degrading through a safety valve (opening pressure: 0.8 bar).
  An additional safety valve (opening pressure 1.2 bar) allowing to degrade possible excessive pressure in the storage vessel, caused by a defect in isolation or heating.

ATTENTION!

Should there be any suspicion that the integrity of the equipment has been compromised (for example as a result of damage sustained during transit or during use), it needs to be withdrawn from service. Make sure that the withdrawn equipment cannot be accidentally used by others. The defective equipment should be handed over to authorized technicians for inspection.

Handling of Fused Silica Parts and Al_2O_3-Parts

Fused Silica

Handling

Fused silica parts may only be touched by cotton or powder free latex gloves. Alternatively a fuzz free tissue can also be used. Otherwise body perspiration or dirt particles could bake into the material permanently when touching fused silica parts with the hands. Moreover the properties of the material might change.

Cleaning

In case of soiled fused silica parts these parts have to be cleaned instantly or latest before heating up. For cleaning purposes we recommend the use pure alcohol (e.g. ethanol).

Al_2O_3

Handling

Alumina parts also may only be touched by cotton or powder free latex gloves. Alternatively a fuzz free tissue can be used. Touching these parts directly with the skin can cause that perspiration or pollutants stick to the surface and will be visible there permanently.

Cleaning

If nevertheless pollutants are on the alumina they have to be cleaned before any further processing. The cleaning can be done with pure alcohol, e. g. ethanol or acetone, ethyl alcohol, petroleum ether. If necessary heat it up to 1600°C in air atmosphere and make an immediate controlled cooling down to at least < 1000°C.

NOTE:

For further cleaning purposes contact qualified NETZSCH service staff!

Please take care to all safety regulations and laboratory instructions for the handling of chemicals!

Before using any cleaning or decontamination methods except those specified by the manufacturer, these methods should be checked in coordination with the manufacturer to avoid any damage of equipment!

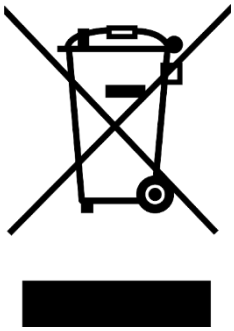
DSC 214

NETZSCH

Chapter II - Installation

206030005 80032061 2020-12-03

Waste disposal

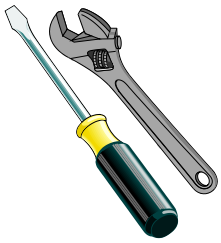


In the European Economic Area (EEA) the disposal of waste equipment is regulated in the "Directive of the European Parliament and of the Council on Waste Electrical and Electronic Equipment (WEEE)". The current official journal on this matter is available on the European Parliament's homepage.

The symbol for the separate collection of electrical and electronic equipment is a crossed-out trash can.

Disposal with household waste (unsorted waste) or similar collections of municipal waste is not permitted! Contact an authorized waste disposal contractor in your country.

Installation



In most cases, your measuring system will be set up and put into commission by one of our customer service engineers.

If you would like to set up your instrument yourself, please read through the following sections carefully.

On request, the computerized system can addionally be qualified (IQ = installation qualification, OQ = operational qualification) by the service engineer. Please contact your responsible sales or service representative in such case.

Packaging

- If possible, keep the original packaging in which your instrument was delivered. Should repairs become necessary or should you wish to add additional equipment to your instrumentation, you can reuse the packaging to ensure damage-free return transport. Our environment will thank you as well.
- After unpacking, please check all delivered components for possible transport damage, using the supplied delivery note as a checklist for the individual items.
- Should an item be missing, please contact us immediately.

NOTE!

Important notes for sending goods:

⇨ NETZSCH cannot accept any liability whatsoever for damage caused by improper packaging. To avoid damage during transport, always ship components in their original packaging and suitable secondary packaging!

⇨ If the original packaging or the secondary packaging is damaged or no longer available, packages can be purchased from NETZSCH to ensure a safe transport of your goods.

Placement Requirements

When considering the best placement for your instrument, select:

- Rooms with low traffic and little vibration
- Rooms that are as dust-free as possible
- Rooms with the most constant temperature conditions possible (room temperature)
- Sturdy lab tables, concrete bases or stable wall brackets. (When using weighting tables with air or rubber damping, please ensure that the damping behavior is appropriate for the instrument.)

Avoid:

- Direct sunlight to the instrument
- Drafts from windows, doors and air conditioning
- Placement in rooms with cast plaster or wooden flooring
- Installation in the direct vicinity of transfer stations or systems with strong magnetic stay fields
- Setting up thermoanalytical instruments near doors or walls bordering a hallway or an elevator

Refer to the separate booklet “Installation Notes” for installation plans.

Connecting the System Components

Requirements
The detailed requirements for installation are given in the enclosed installation schematic.

Connecting the Computer System

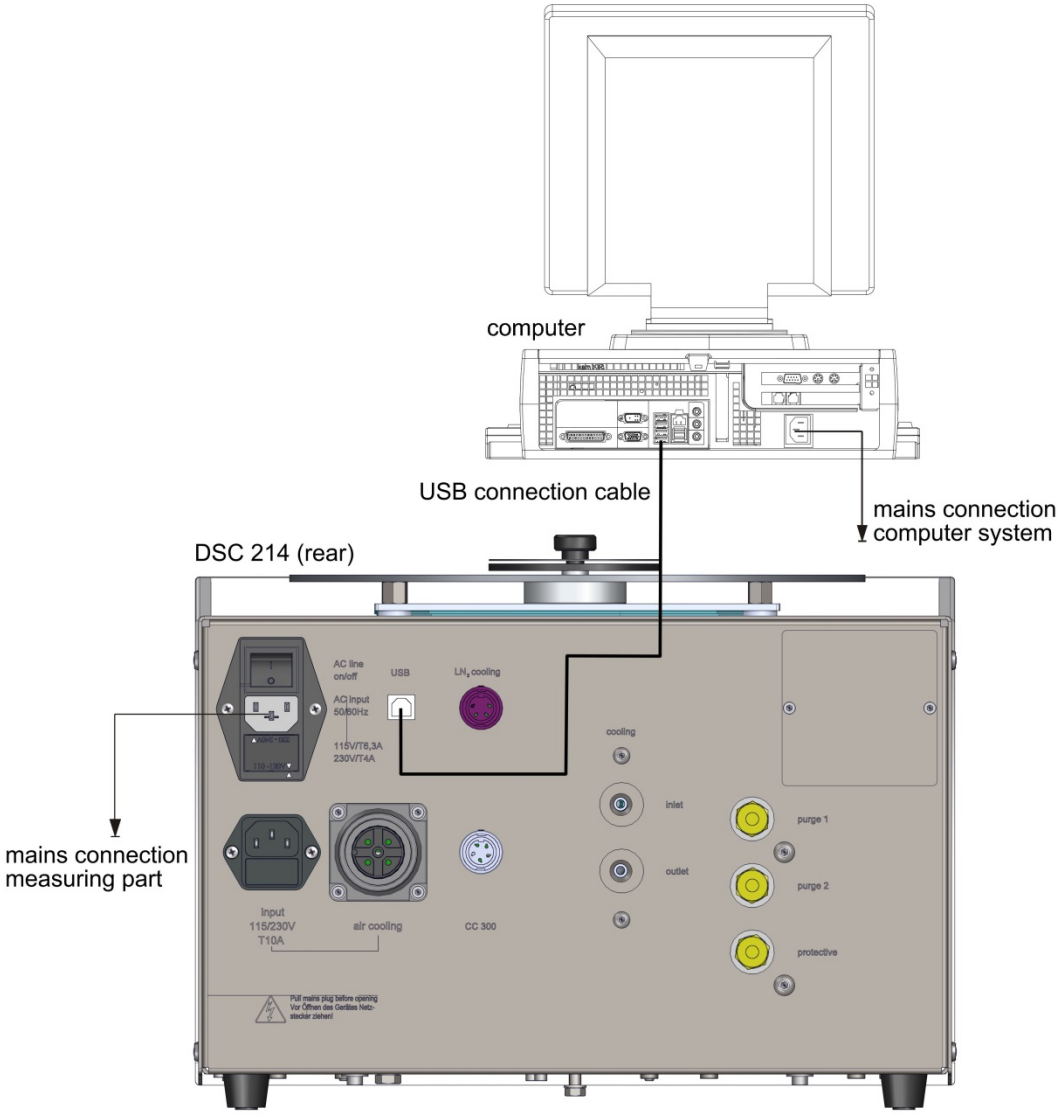


Figure 1: connecting computer system

For connecting the computer system, see also the technical documentation of the computer manufacturer.

Your DSC 214 instrument communicates with the PC using the USB interface of the PC. This requires the installation of suitable drivers delivered together with the Proteus Software.
Before initially connecting the DSC 214 the Proteus Software must be installed first.

For details of installation, please read chapter 3 and chapter 4.2 in the document "NETZSCH *Proteus* Software".

- ⇨ Do not use USB hubs (connect your DSC 214 directly to the PC).
- ⇨ Use the original USB cable delivered from NETZSCH.
- ⇨ If you accidentally change the USB interface on the PC the Plug&Play procedure will start again to install the drivers for this interface. Please note that this procedure has to be done for any "not yet" connected USB interface.

Note for *Nevio* instruments:

Core element of the *Nevio* instrument series is *Proteus Protect*, a supplement to the 8th generation of the *Proteus* software meeting the requirements of 21 CFR Part 11.
Proteus Protect offers:

- ⇨ Access control (password management)
- ⇨ User management (assignment of permissions, etc.)
- ⇨ Audit Trail (documentation of all relevant actions performed)
- ⇨ Inactivity observer (automatic log-out of the user elapsing a defined time interval of inactivity)
- ⇨ Electronic signatures

The separate **Philosophy** document provides some background information.

Prior to the installation of *Proteus Protect*, some pre-arrangements have to be made at the customer site which are summarized in a separate document entitled **Installation Requirements**.
Please don´t forget to send the completed questionnaire (which can be found on the two last pages of the document) to the responsible service organization before installation and commissioning of the instrument will be scheduled.
Thank you very much for this.

Please find further instructions for the software adaptation in the separate **Users Guide**.

Connecting the Linear Small Compressor

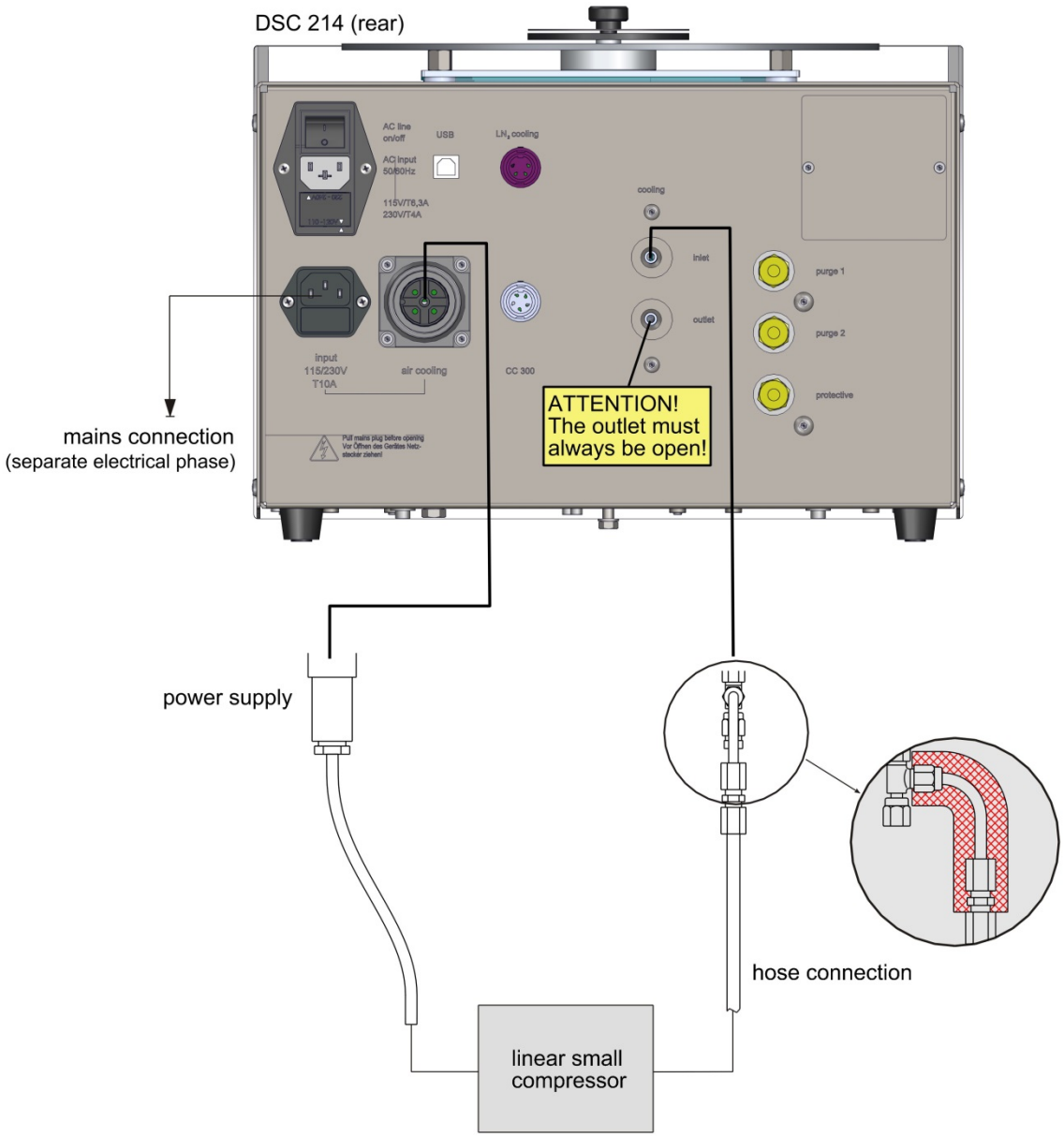


Figure 2: connecting the linear small compressor

Connecting the On-Off Valve Kit for Compressed Air Cooling

input pressure max. 2 bar absolute (1 bar overpressure)

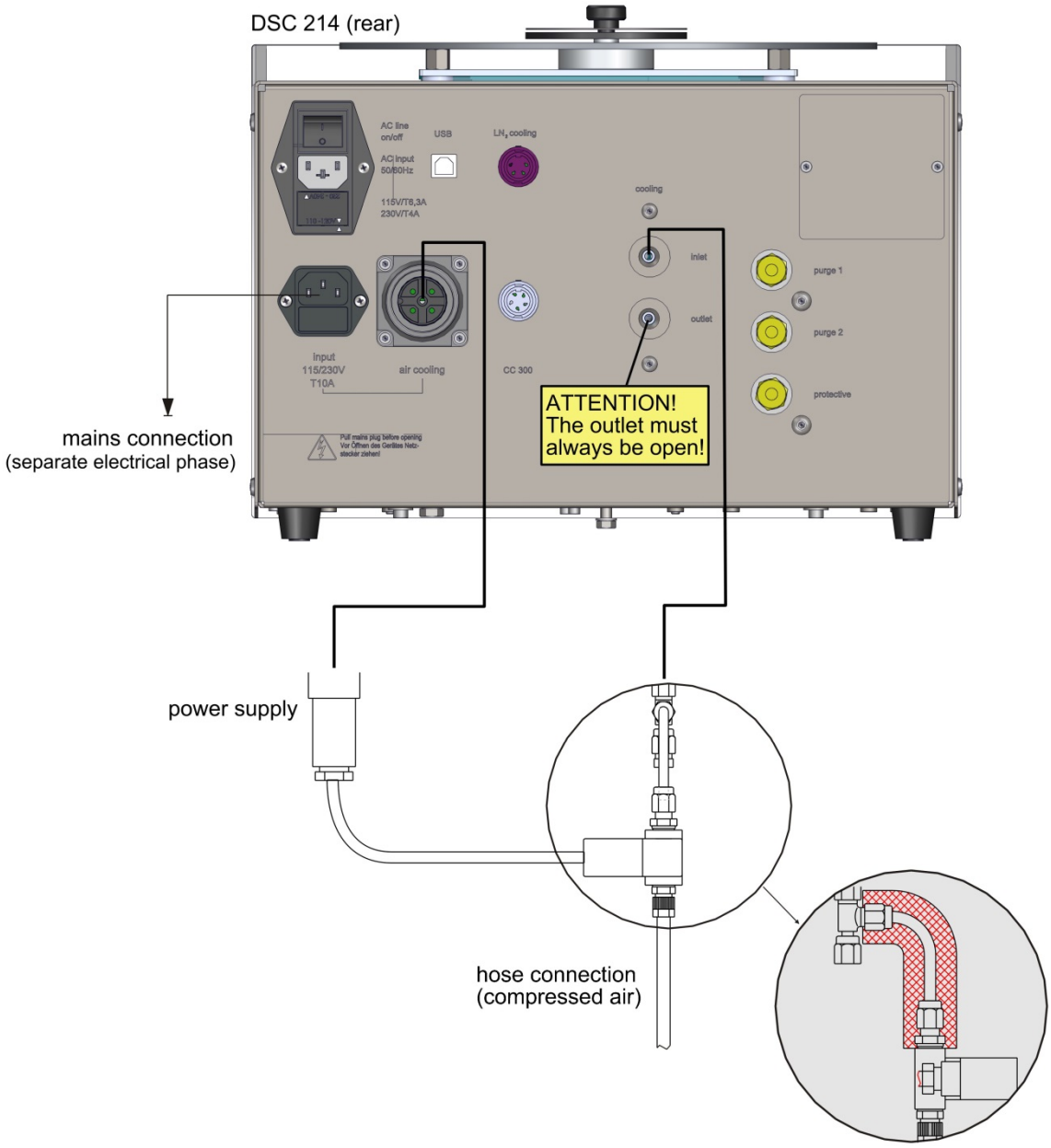


Figure 3: connecting the on-off valve kit for compressed air cooling

Connecting the Cooling device for Pressurized Air

For minimum temperature of 0°C or below (dependent on air pressure). Includes cold air generator and software controlled magnetic valve.

Requirements at customers site:
Pressurized air, throughput approx. 200 l/min, min. pressure 6 bar, max. pressure 10 bar, oil-free, dry (pressure dew point <5 °C), filtered (25 µm or better)

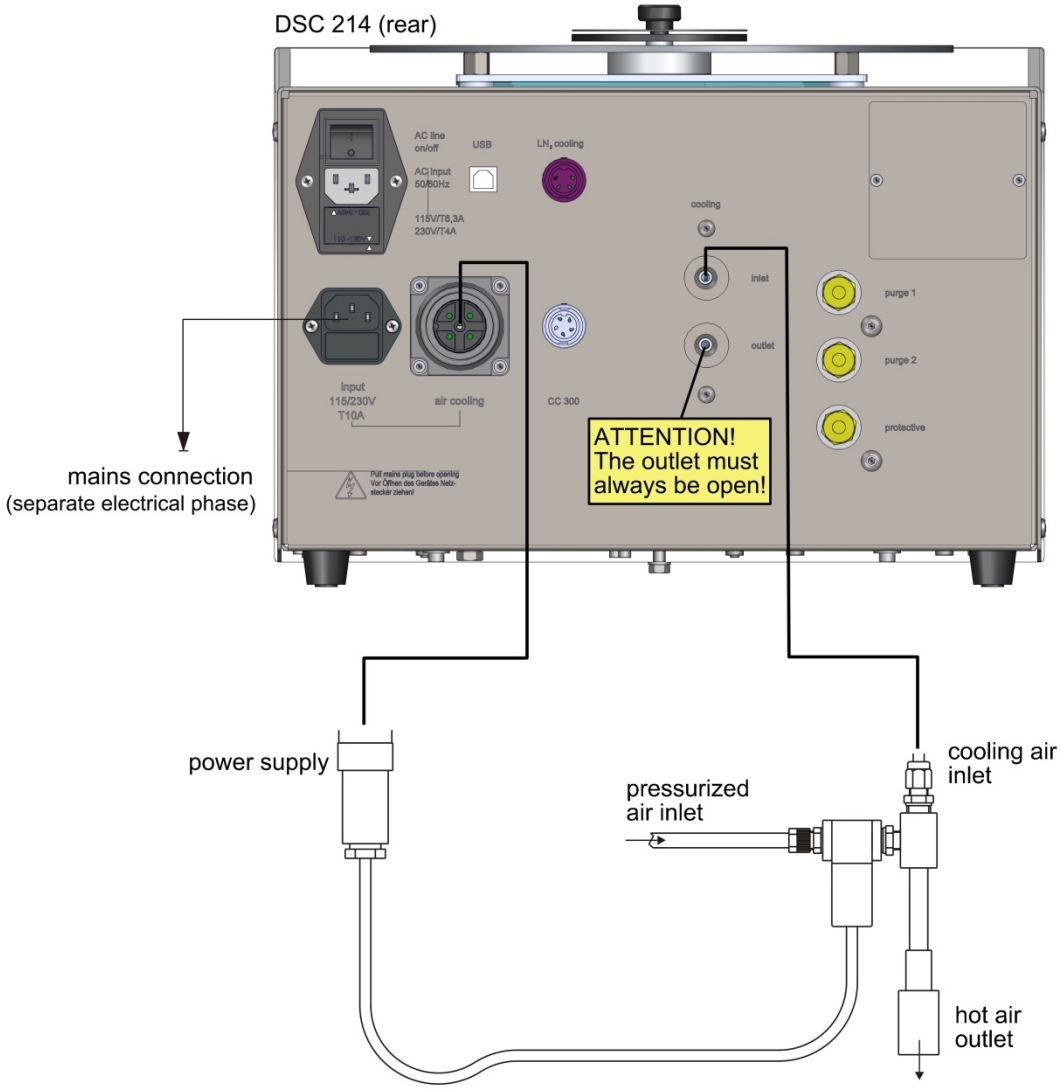


Figure 4: connecting the cooling device for pressurized air

Connecting the Intracooler 40

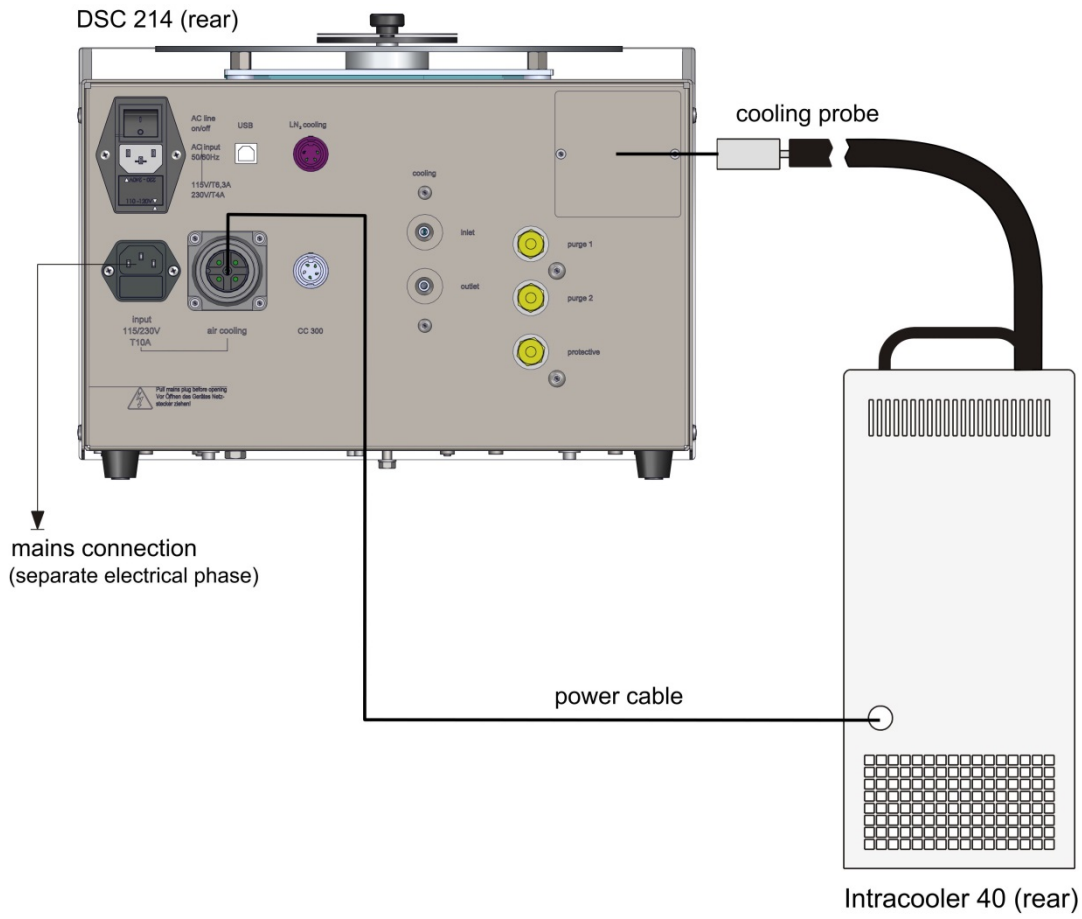


Figure 5: connecting the Intracooler 40

See separate manual Intracooler 40!

The installation of the Intracooler 40 should only be carried out by NETZSCH service staff!

Connecting the Intracooler 70 (230 V / 50-60 Hz)

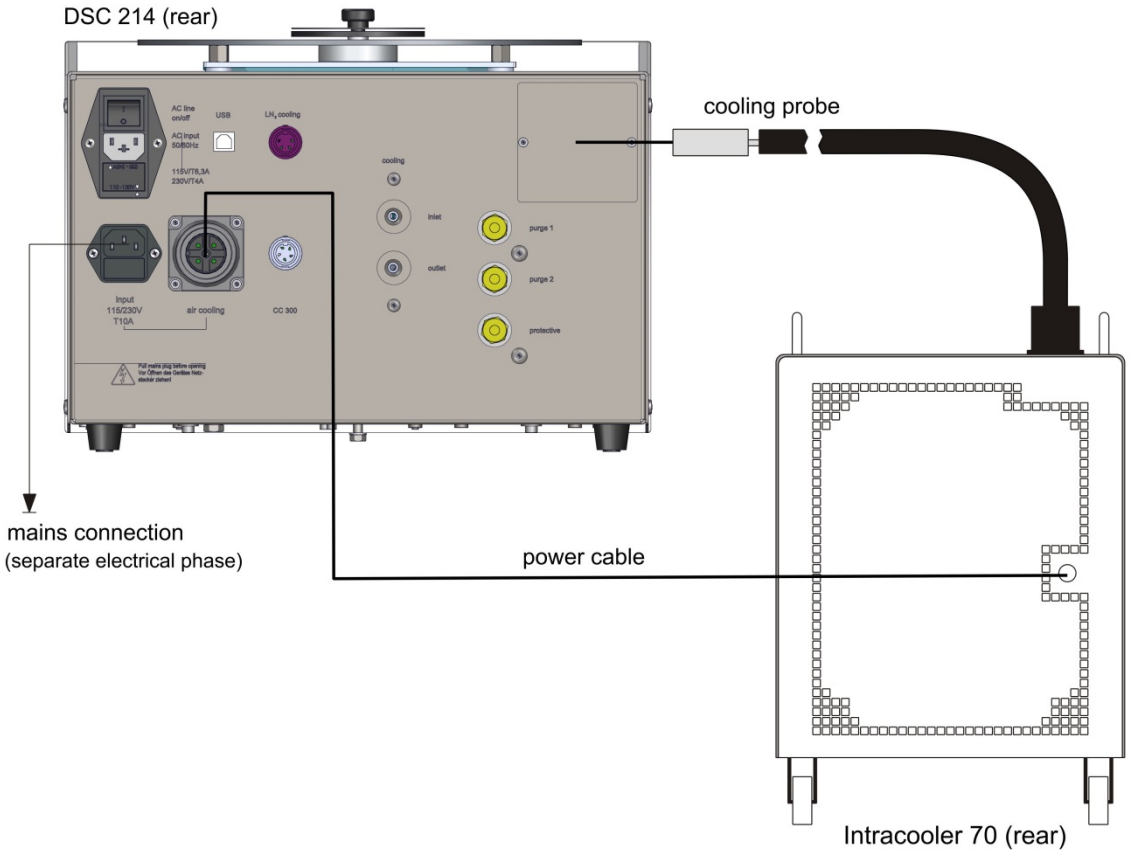
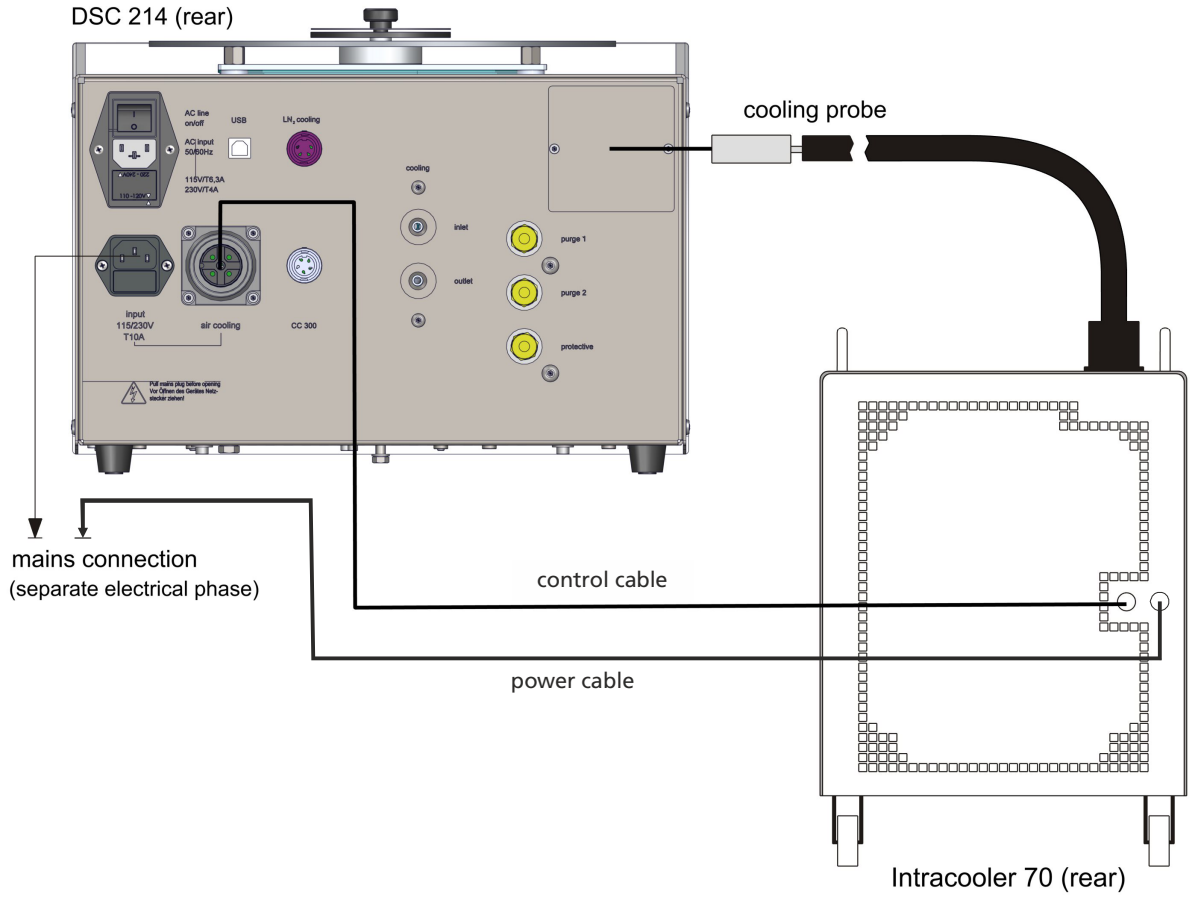


Figure 6: connecting the Intracooler 70 (230 V / 50-60 Hz)

See separate manual Intracooler 70!

The installation of the Intracooler 70 should only be carried out by NETZSCH service staff!

Connecting the Intracooler 70 (115 V / 60 Hz)



The mains connection of the IC 70 (115 V / 60 Hz) must be the same as the additional AC input (5)!

Figure 7: connecting the Intracooler 70 (115 V / 60 Hz)

See separate manual Intracooler 70!

The installation of the Intracooler 70 should only be carried out by NETZSCH service staff!

Connecting the LN2 Cooler CC 200 F3

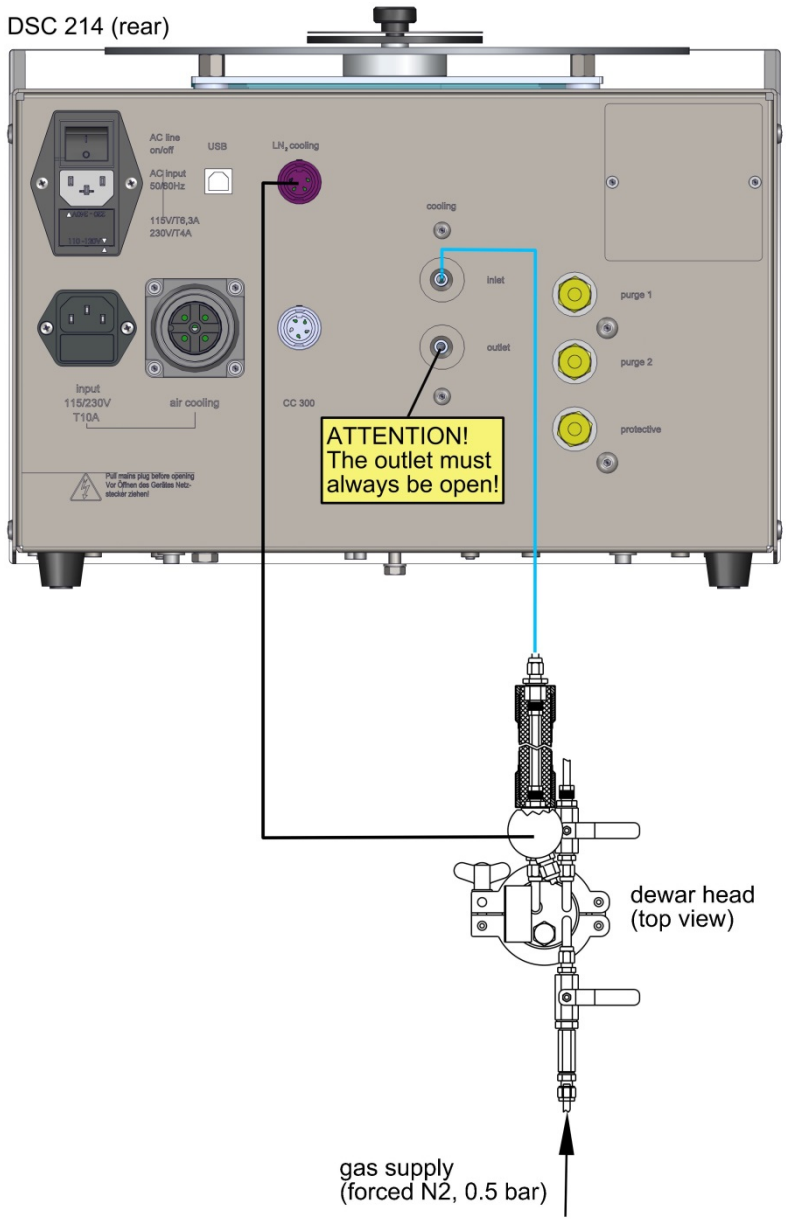


Figure 8: connecting the LN2 cooler CC 200 *F3* with magnetic valve

Please pay attention to all safety regulations for handling liquid nitrogen (see chapter I)!

Connecting the LN2 Cooler CC 300

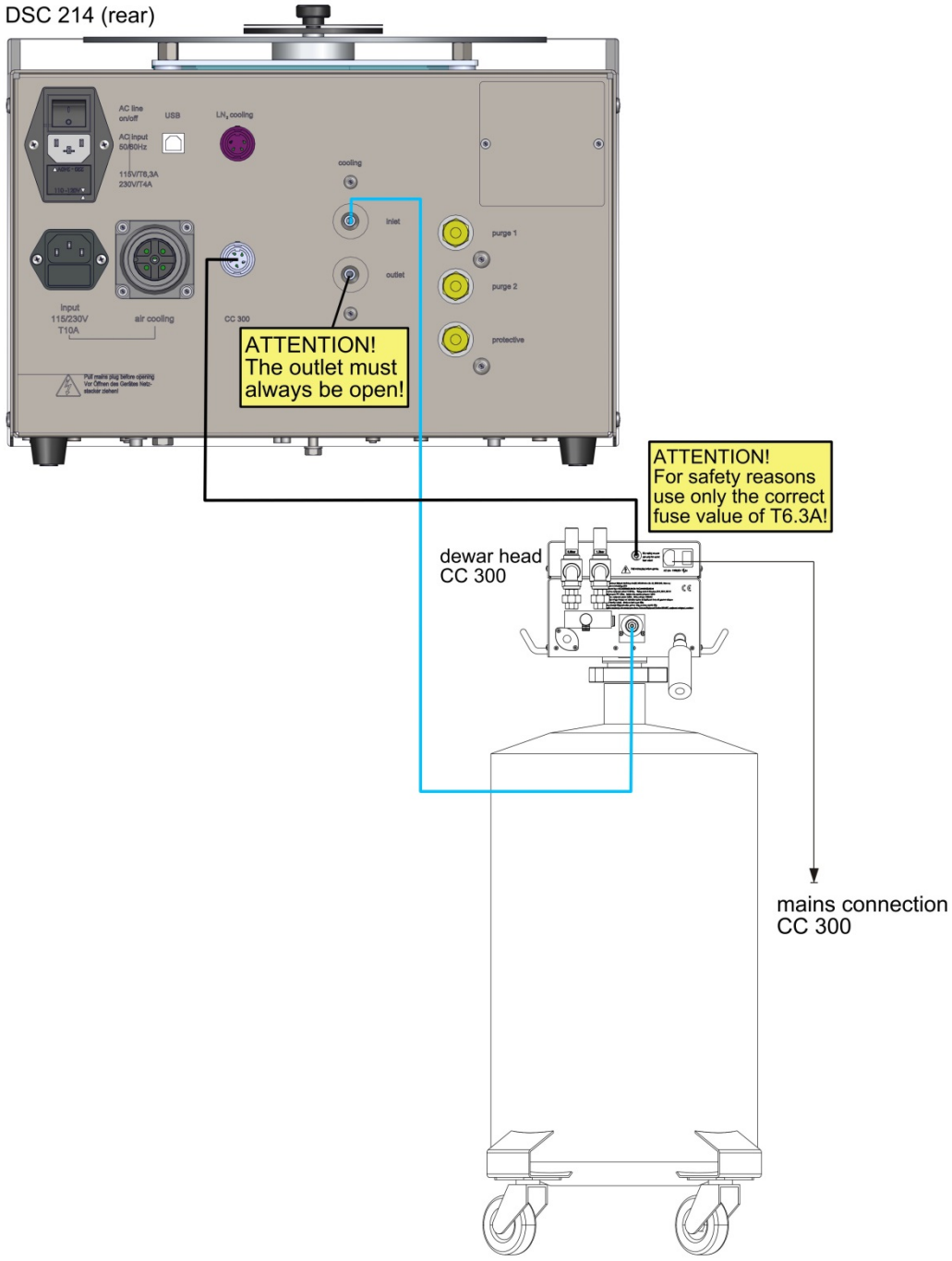


Figure 9: connecting the LN2 cooler CC 300

See separate manual CC 300!

Connecting the Gas Supply

Depending on the equipment of the DSC 214 instrument, the gas supply can be connected to the following components:

- sintering inserts
- gas flow control device (6.240.20-40.0.00)
- mass flow controllers (MFCs)

Sintering Inserts

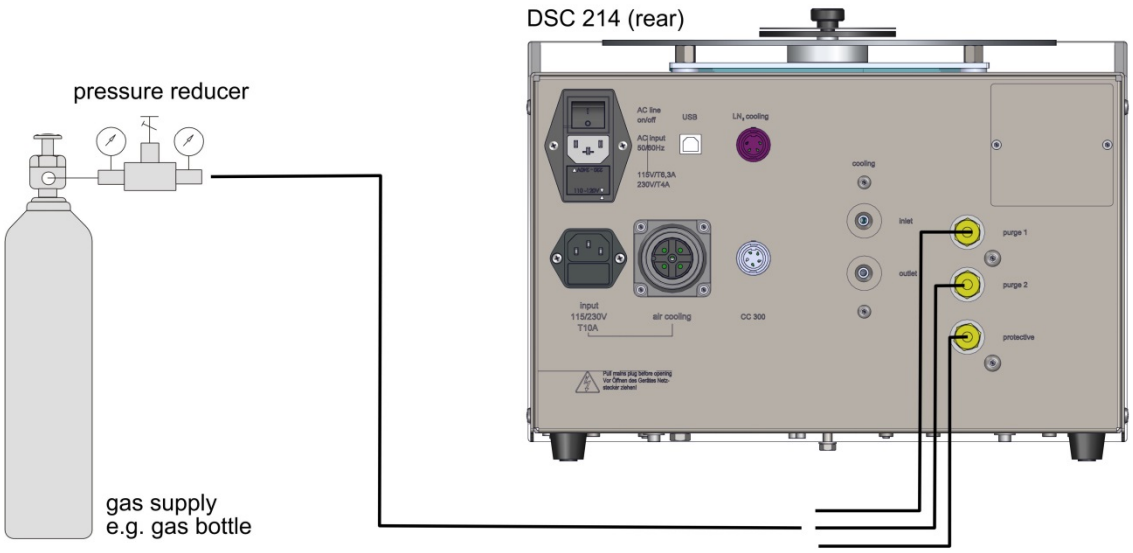


Figure 10: connecting the gas supply (sintering inserts)

- Connect the gas hoses as shown in Figure 10.

Adjusting the gas flow: see chapter IV

See also recommendations for the use of reactive purge gases (chapter I)!

Connecting the Gas Flow Control Device

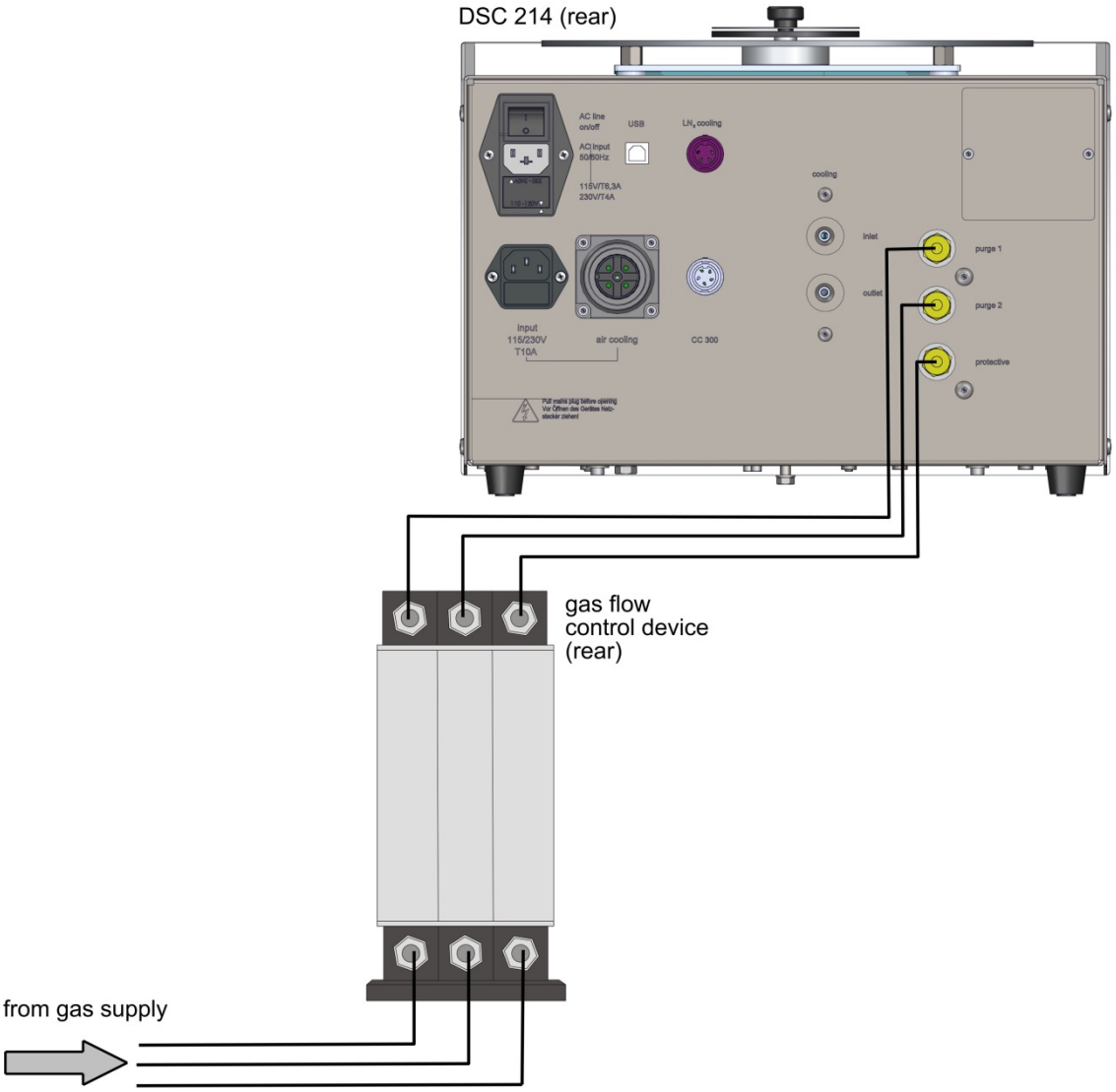


Figure 11: connecting the gas flow control device (6.240.20-40.0.00)

- Connect the gas hoses as shown in Figure 11.

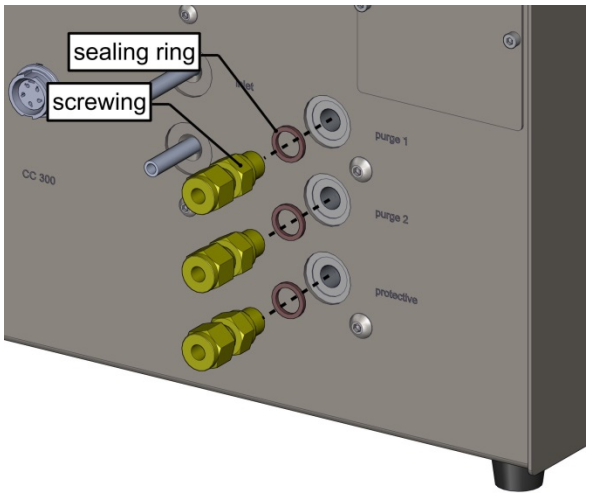
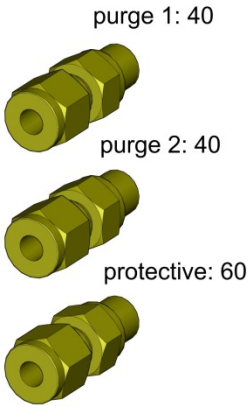
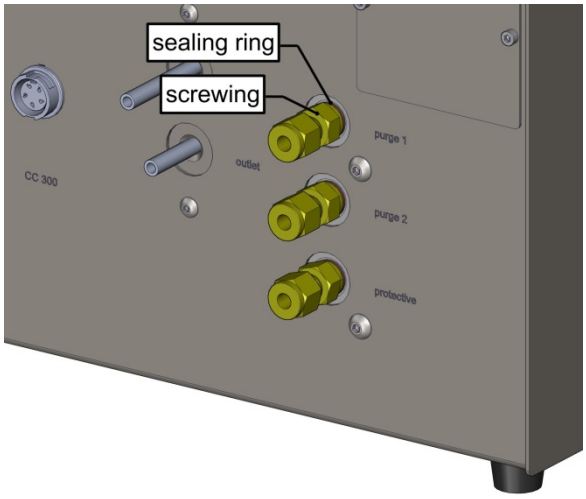
Max. input pressure 0.5 bar!

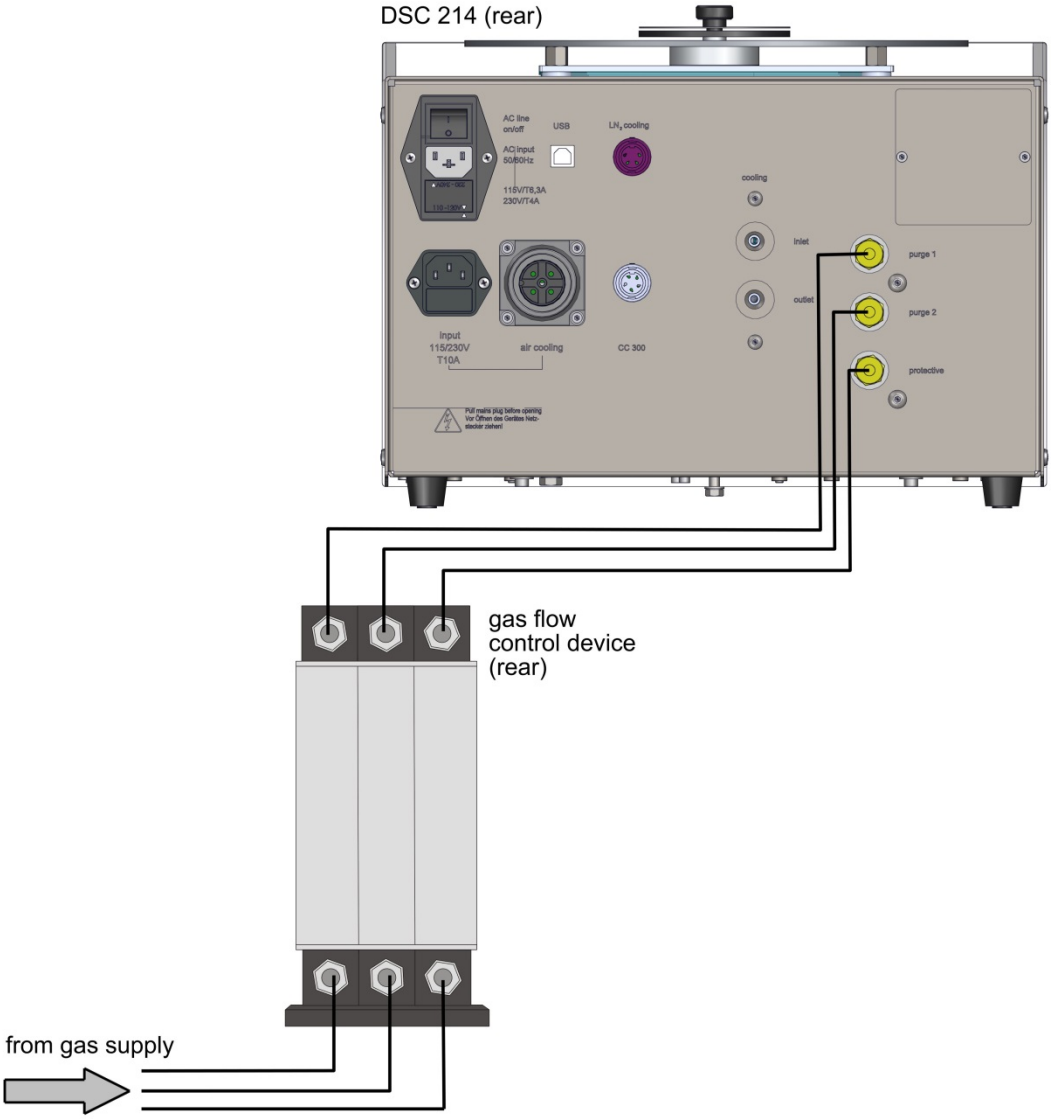
Adjusting the gas flow: see chapter IV!

See also recommendations for the use of reactive purge gases (chapter IV)!

If the gas flow control device is added a later date, check that the sintering inserts are removed.

Removing the Sintering Inserts

	• Remove the screwing (3x) and sealing ring (3x).
	• The screwings are marked: **40** (40 ml/min = gas flow adjusted by sintering insert) **60** (60 ml/min = gas flow adjusted by sintering insert) If you reinstall the nuts: Use for "purge 1" and "purge 2" the nuts marked with 40. Use for "protective" the nut marked with 60. Do not forget the seal.
	• Install in the screwing (3x) without sintering inserts. Do not forget the seals (3x).



- Connect the gas flow control device as shown in the figure above.

Max. input pressure 0.5 bar (only for gas flow control device).

See also recommendations for the use of reactive purge gases!

Connecting the Mass Flow Controllers (MFCs)

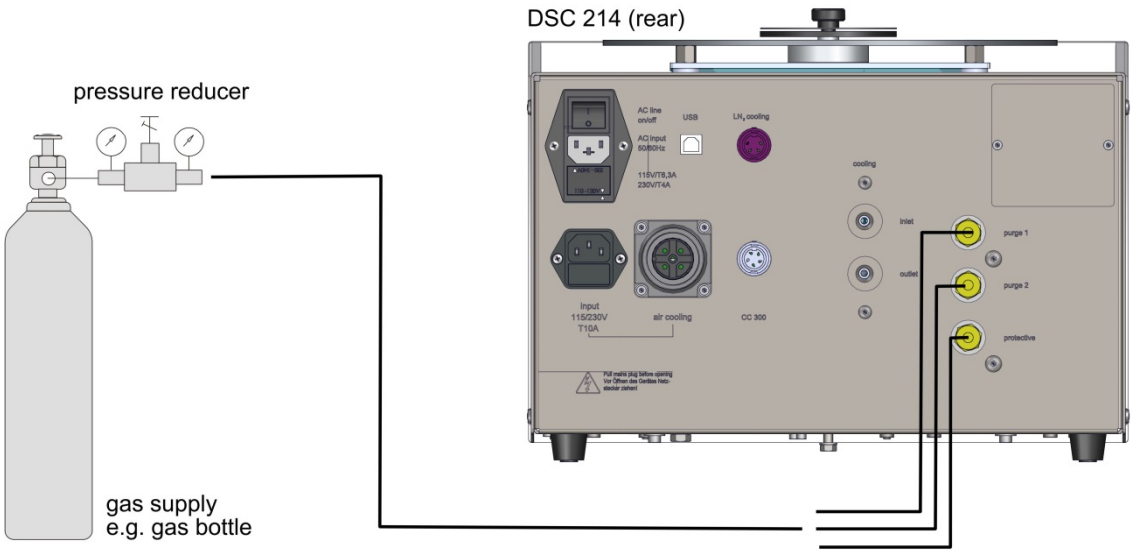


Figure 12: connecting the gas supply (MFCs)

The gas supply unit (e.g. gas bottle) should be equipped with a gas pressure reducer.
The recommended input pressure at the gas inlet of the instrument is 0.5 bar overpressure.

Max. input pressure 1.0 bar overpressure!

A higher input pressure than 1.0 bar overpressure might damage the MFC. Therefore, the input pressure should be adjusted at an appropriate gas pressure reducer of the gas supply unit before the gas hoses are connected.

The gas hoses should be disconnected when the maximum pressure might be exceeded, for example after an exchange of the gas bottle and reinstallation of the gas pressure reducer.

The mass flow controllers are not designed for the use of corrosive, flammable or reducing gases!
See also all recommendations for the use of reactive purge gases (chapter I)!

After setting the gas pressure reducer to the recommended value of 0.5 bar overpressure connect the gas hoses for purge 1, purge 2, and protective at the rear of the instrument.
The gas flow for all gases can be adjusted by means of MFCs via the software.

206030005 80032061 2020-12-03

DSC 214

NETZSCH

Chapter III – Specific Information of the Instrument

206030005 80032061 2020-12-03

Operating Principle

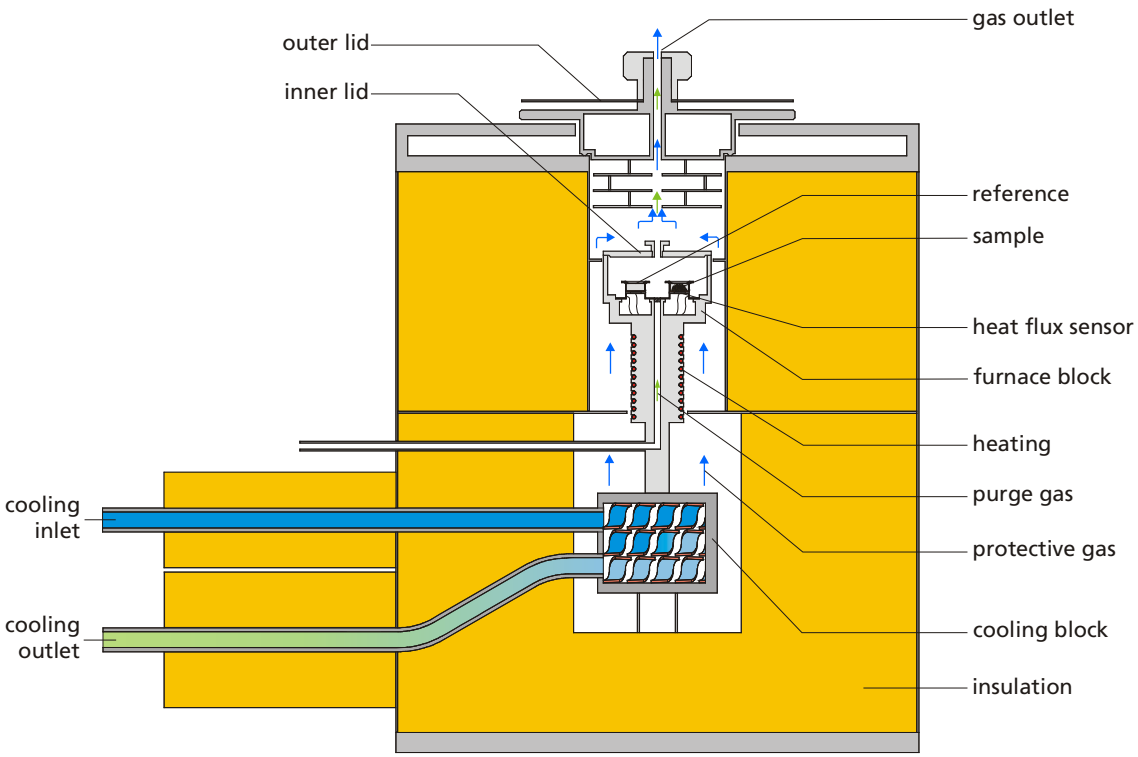


Figure 13: DSC 214 cross-section of the measuring cell

Differential Scanning Calorimetry (DSC)

Technique in which the difference in heat flow to a sample and to a reference is monitored as a function of temperature or time, while the sample is subjected to a controlled temperature program. (DIN 51007, ASTM E 474).

Furnace	• block • miniature jacketed heater • furnace temperature is measured by a thermocouple which is integrated into the furnace wall
Sample chamber	• closed with two additional lids • the measuring cell is sealed with the instrument lid
Cooling	• nitrogen cooling (liquid) • air cooling (compressor), compressed air (electrovalve)
Sensor	• DSC sensor system type E with high sensitivity
Purge gas	• two separate purge gas inlets (sample chamber), connected via a tee piece • protective gas (furnace jacket)

Front Panel of the Measuring Unit

Figure 14: front panels with control LED

LEDs	
green LED on:	instrument is switched on
orange LED on:	measurement is running

Rear of the Measuring Unit

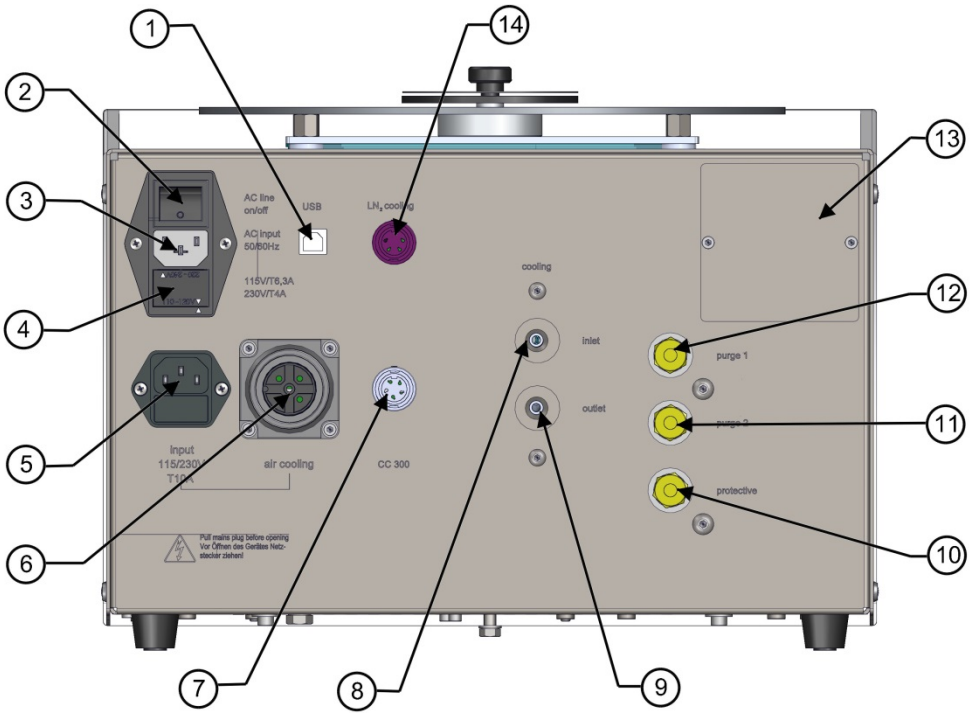


Figure 15: rear of the measuring unit

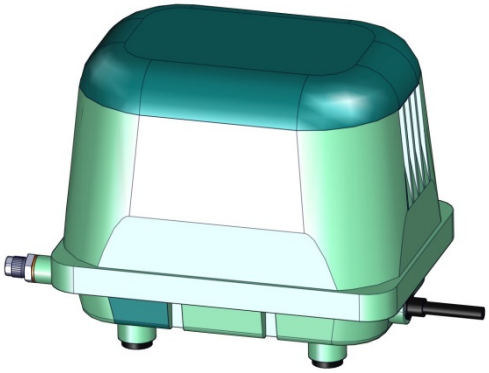
No.	label	function
1	USB	computer interface connection (USB)
2	AC line on/off	power switch: instrument "on/off"
3	AC input 50/60 Hz	power connection 230 (115) V
4	115V/T4A; 230V/T2A	mains fuses
5	AC input 50/60 Hz	power connection 230 (115) V for cooling equipment
6	Air cooling	plug connection: air cooling (compressor or electrovalve)
7	CC 300	plug connection: liquid nitrogen cooling CC 300 (0CC30060B10.000-00 or 15.000-00)
8	cooling in	inlet for air and nitrogen cooling
9	cooling out	outlet for air and nitrogen cooling
10	protective	screw connection: protective gas (jacket)
11	purge 2	screw connection: purge gas 2 (sample chamber)
12	purge 1	screw connection: purge gas 1 (sample chamber)
13	cover plate	connection for Intracooler
14	LN2 cooling	plug connection: liquid nitrogen cooling (6.351.35-00.0.00)

Cooling Devices

Linear small compressor

Linear small compressor for cooling purposes

- Temperature range: 700°C to 40°C
- Quickest possible cooling time from 600°C to 40°C: 20 min
- Linear cooling at 20K/min to 160°C and at 10K/min to 100°C



Technical Specifications:

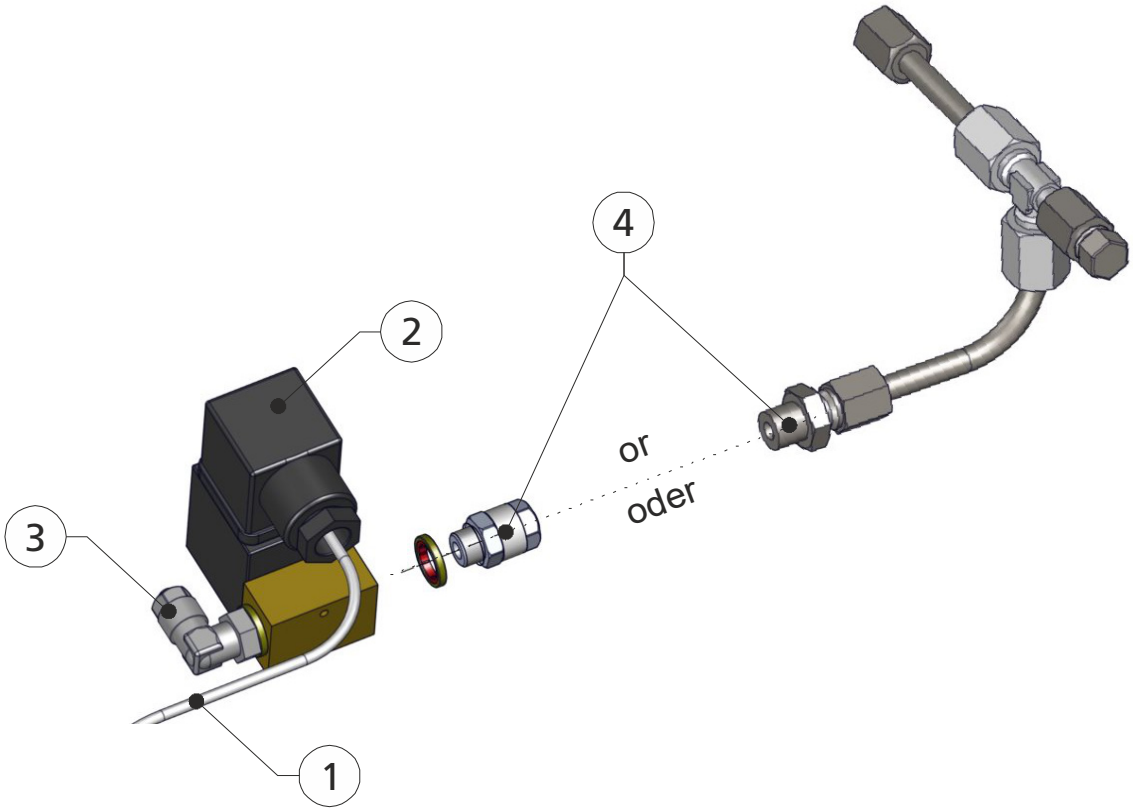
Voltage, alternating current:	230 V, 50/60 Hz or 115 V, 60 Hz
Type of motor protection:	IP 20
Pressure, suction side:	1 bar abs.
Pressure, pressure side:	1 bar abs.
Delivery efficiency at atm. pressure (measured in air at 20°C):	approx. 39 l/min
Allowable temperature of the medium:	+5°C to 40°C
Allowable temperature of the environment:	+5°C to 40°C
Gas connection:	hose, inside diameter 6 mm outside diameter 8 mm
Flow medium:	air
Weight:	6.6 kg

See seperate operating manual!
The installation is described in Chapter II !

Cooling Device for Compressed Air Cooling

Requirements on-site at customer's location for compressed air:

- pressure: max. 2 bar absolute (1 bar overpressure)
- oil-free
- pure
- filtered



No.	Description
1	control cable
2	magnetic valve
3	compressed air connection (inlet or outlet, depending on device type)
4	compressed air connection (inlet or outlet, depending on device type)

The installation is described in Chapter II

Cooling Device for Pressurized Air (Vortex)

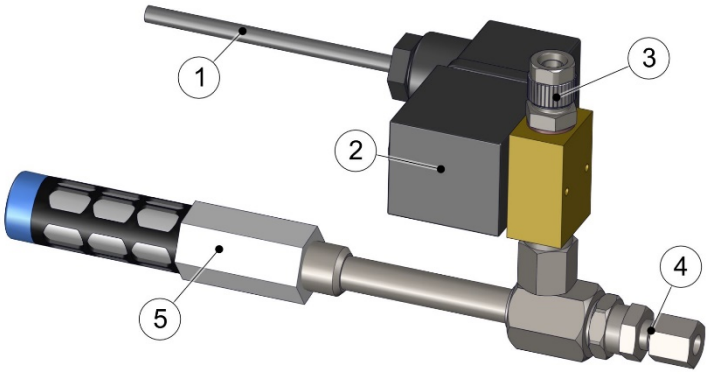
The cooling device for pressurized air (Vortex) includes a cold air generator and a software-controlled magnetic valve. Temperatures of ≤ 0°C can be reached (depending on the air pressure).

Requirements on-site at the customer's location for compressed air:

- flow rate of approx. 200 l/min
- min. pressure: 6 bar
- max. pressure: 10 bar
- oil-free
- dry (pressure dew point of < 5°C)
- filtered (25 µm or better)

NOTE:

- Level of noise: up to 70 dB (A)
- Tube between cooling inlet (instrument) and cold air outlet (Vortex tube) should be insulated.

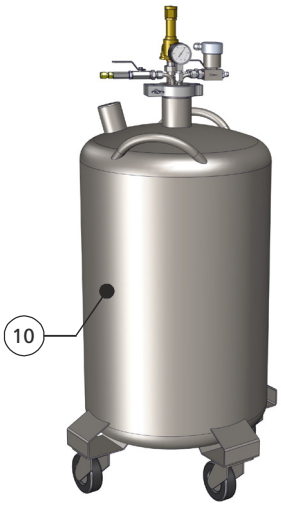


No.	Description
1	control cable
2	magnetic valve
3	gas connection (compressed air inlet)
4	gas connection (cold air outlet)
5	outlet (hot air)

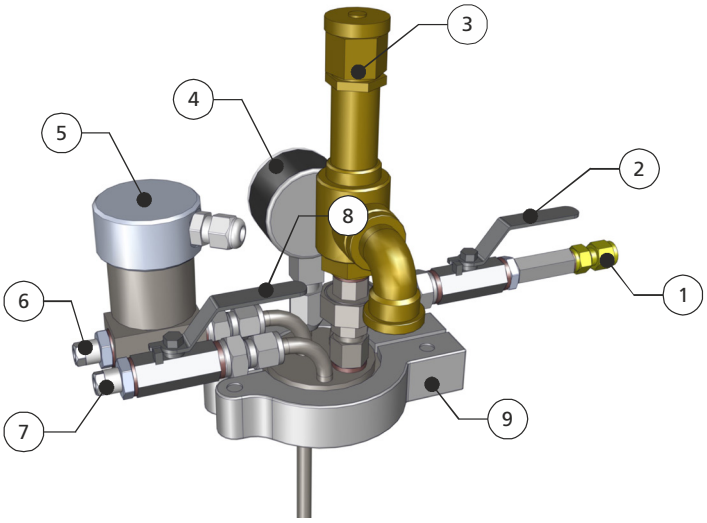
The installation is described in Chapter II

LN2 Cooling System CC 200 F3

LN2 cooling system, operated with compressed gas (N2), software-controlled magnetic valve for switching the LN2 supply on and off, with LN2 storage vessel.



Storage vessel (CS 60 S)



Dewar head (CC 200 *F3*)

Height:	950 mm
∅ Storage vessel:	450 mm
Width:	555 mm
Volume:	60 l
Weight empty / full:	46 / 98 kg

Width / Depth:	~ 300 mm / ~ 120 mm
Height (outside vessel):	210 mm
Weight of Dewar head:	4 kg

No.	Description
1	gas inlet (gas supply, forced N2 , 0.5 bar)
2	shut-off valve (from gas supply)
3	pressure relief valve (open at overpressure 0.5 bar)
4	manometer
5	magnetic valve
6	gas outlet (to instrument)
7	gas outlet (pressure relief)
8	shut-off valve (pressure relief)
9	clamping ring and centering ring
10	vessel 60 l

Please carefully heed all safety regulations for handling liquid nitrogen (LN2) or gaseous nitrogen (GN2)!

The installlation is described at Chapter II .

LN2 Cooling System CC 300

The LN2 cooling system CC300 for liquid and low-temperature gaseous nitrogen is used as an additional cooling supplement for NETZSCH thermoanalytical instruments.

Dewar head with supply system (without refilling device)

Dewar head with supply system and refilling device (optional)

Storage vessel

Dewar head	
Height (outside vessel):	250 mm
Weight of Dewar head:	14 kg
Weight of refilling device:	2 kg
Voltage:	115/230V / 50/60Hz
Power consumption (max):	550 VA

CS 60 S	
Height:	950 mm
∅ Storage vessel:	450 mm
Width:	555 mm
Volume:	60 l
Weight empty / full:	46 / 98 kg

See seperate operating manual!
The installation is described in Chapter II .

Before using liquid nitrogen (LN2) or nitrogen (N2) it is of crucial importance to familiarize oneself with the corresponding safety instructions. Those can be read through in Chapter I !

Intracooler 40

Cooling system supplement (compressor cooling) for installation into DSC 214 / DSC 200 *F3* / DSC 3500

Temperature range for above mentioned instrument: -40 ... +600°C

Intracooler IC40

Height: 430 mm (w/o hose)
Width: 200 mm
Depth: 360 mm
Weight 24 kg

See seperate operating manual!
The installation bis described in Chapter II .

Intracooler 70

Cooling system supplement (compressor cooling) for installation into DSC 214 / DSC 200 *F3* / DSC 3500

Temperature range for above mentioned instrument: -70 ... +600°C

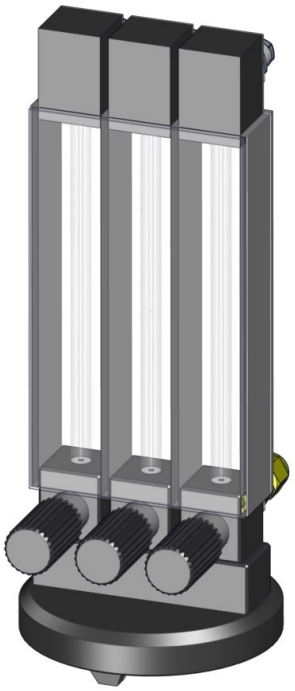
Intracooler IC70

Height: 600 mm (w/o hose)
Width: 380 mm
Depth: 550 mm
Weight 50 kg

See seperate operating manual!
The installation is described in Chapter II .

Gas Flow Control Device

Gas flow control for 3 gas pipes with 3 regulating valves and 3 flow meters.



Gas flow control for 3 gas pipes

Free-standing, including connectors with frits instead of the device related standard frits, connecting and hose material.

measuring range for purge gases: 2x 6 ... 70 ml/min

measuring range for protective gas: 1x 5 ... 190 ml/min

Maximum input pressure 0.5 bar!

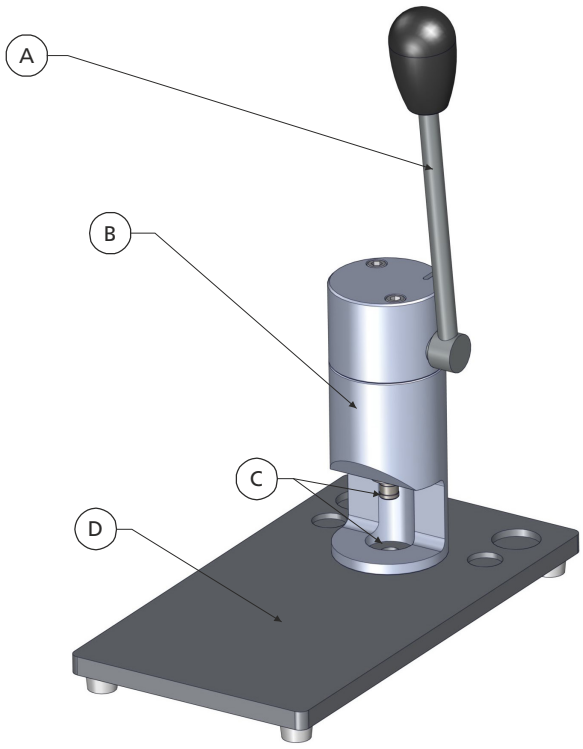
The installation is described in Chapter II .

See also recommendations for the use of reactive purge gases in Chapter I.

Sealing Press

Sealing press for cold welding or pressure-tight sealing of crucibles. The toolkits must be selected according to the desired sample container.

Unit



A Lever

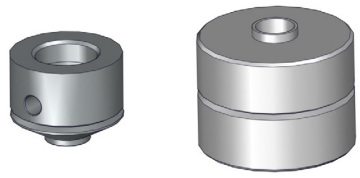
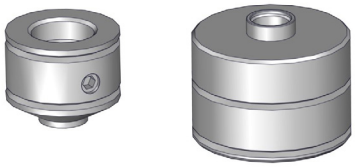
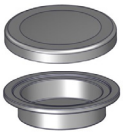
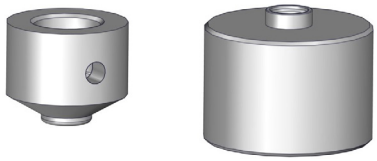
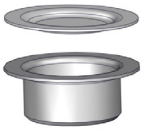
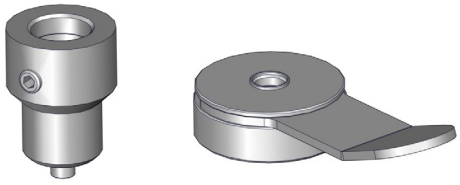
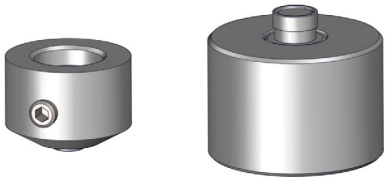
B Press housing

C Toolkit fastener

D Base plate

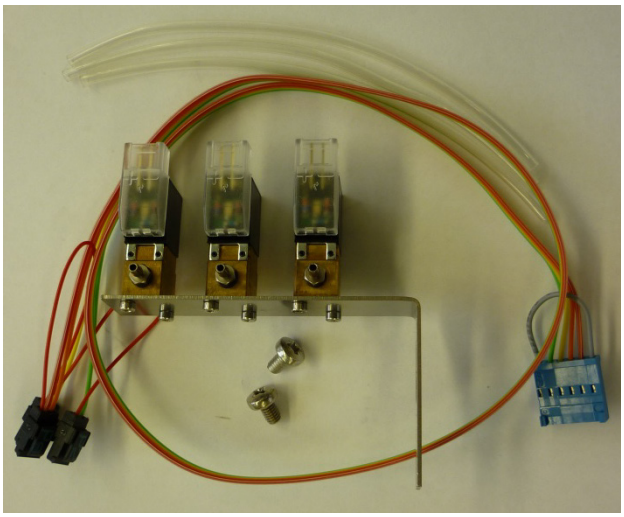
Sealing press no. 6.240.10-80.0.00 is used for sealing different types of crucibles. Each type of crucible requires a different toolkit (see available toolkits). To seal a crucible, position it on the lower part of the toolkit fastener along with its corresponding lid. Then lower the lever with a single continuous motion to close or cold weld the crucible. The necessary force is defined by a spring in the press housing.

Available Toolkits:

6.240.10-81.0.00 	Type of crucible: 	Toolkit for pressure-tight cold welding of Al crucibles, Ø 6 mm: Series 6.239.2-64.5xx
6.240.10-82.0.00 	Type of crucible: 	Toolkit for pressure-tight cold welding of Al crucibles, Ø 6 mm: Series 6.240.10-65.1xx
6.240.10-83.0.00 	Type of crucible: 	Toolkit for pressure-tight sealing of the medium-pressure crucibles: 6.240.1-68.1.00
6.240.10-85.0.00 	Type of crucible: 	Toolkit for pressure-tight cold welding of Concavus pans: Series DSC21400A66.xxx NGB814672
DSC21400A80.030-00 	Type of crucible: 	Toolkit for inserting the slide-in lids (NGB815051) into the Concavus pan (NGB814672). Especially for polymer foil samples.
6.240.10-84.0.00 	Type of crucible: 	Stamping tool kit for aluminum crucibles for SFI-measurements (Solid Fat Index): NGB810405 (SFI-measurements)

Purge Gas Switching (O.I.T Extension)

3 magnetic valves (two for purge gas inlet to the sample and one for protective gas for the measuring cell) for programmed ON/OFF of the gas flow.



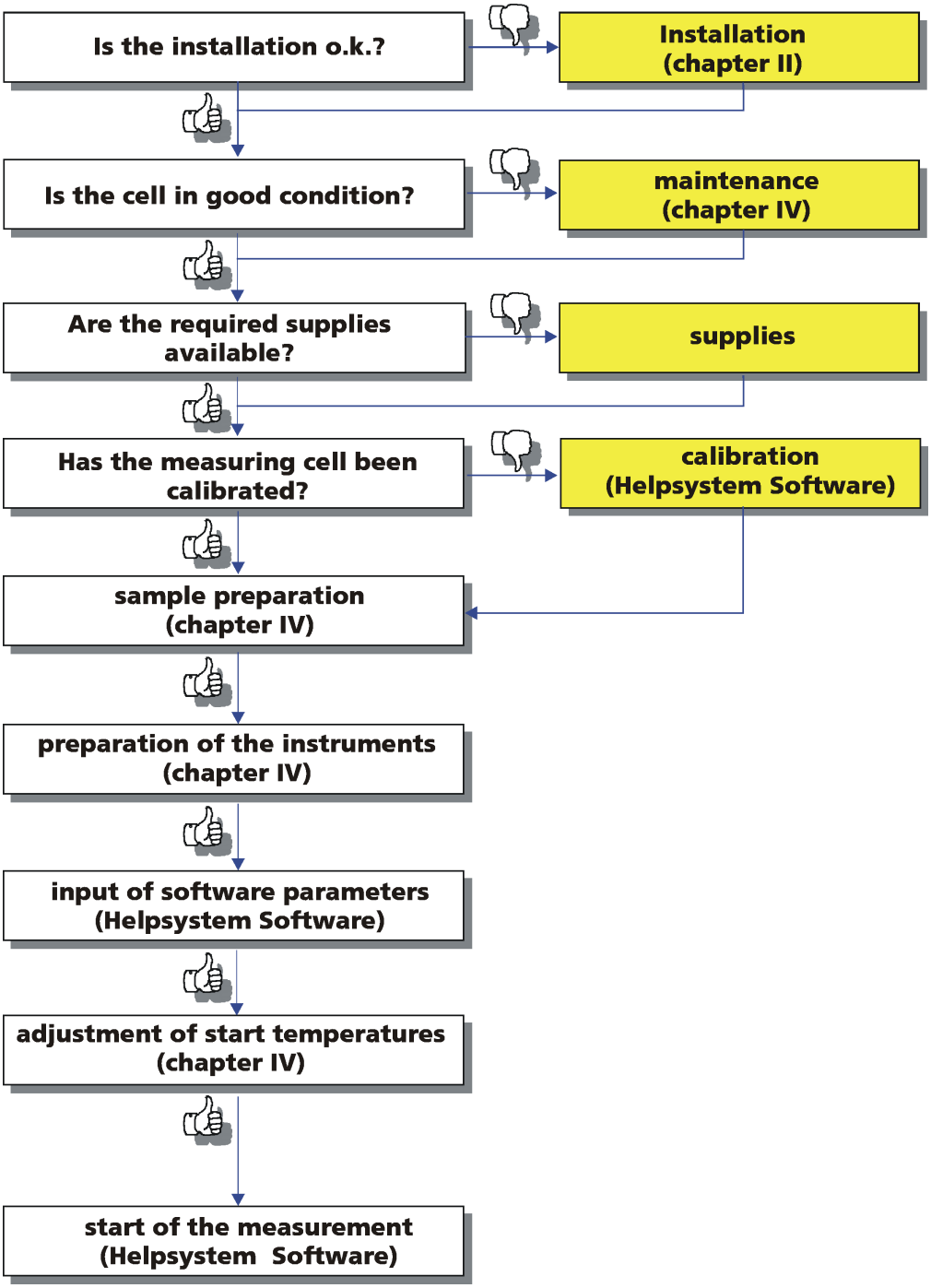
DSC 214

NETZSCH

Chapter IV – Operate the Instrument

206030005 80032061 2020-12-03

Operating the Instrument



206030005 80032061 2020-12-03

Preparing the Measurement

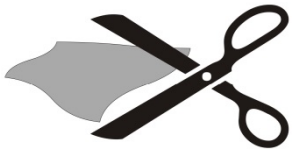
In thermoanalytical investigations, the test parameters have a significant influence on the results.

Information regarding the specific effects of the individual test parameters on the results of DSC measurements can be found in the technical literature.

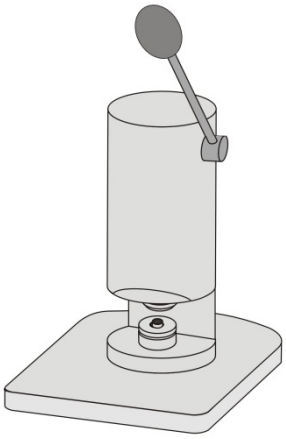
The following parameters should be considered when preparing the measurement:

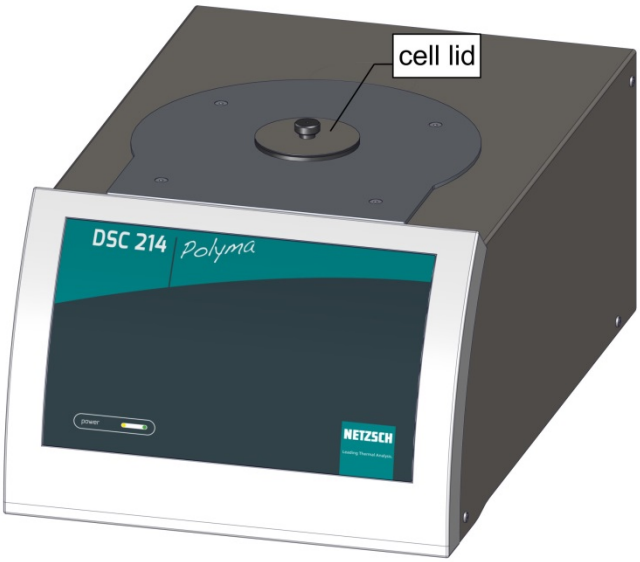
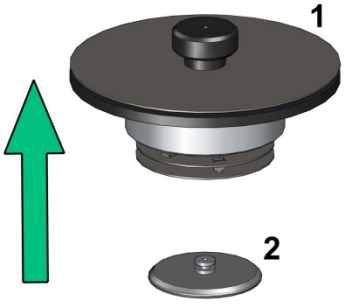
- calibration
- sample preparation
- sample weight
- reference material
- sample chamber temperature
- temperature program
- atmosphere

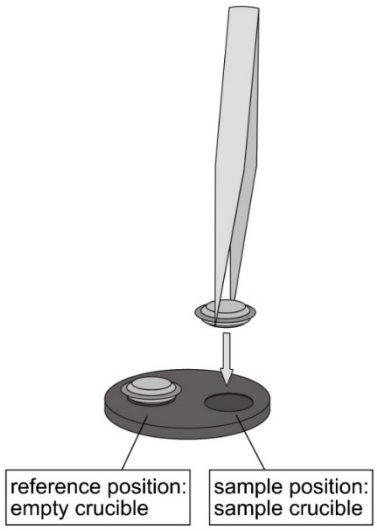
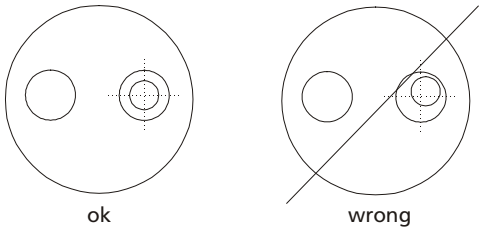
What do you need for the measurement?	Assemble the required materials before starting the measurement: • purge gases (sample chamber and furnace jacket) liquid nitrogen (cooling), forced air (cooling) • sealing press for cold sealing of Al crucibles • sealing tool for pressure-tight crucibles • tweezers (pointed for crucibles, offset for lids)
Calibration	Observe below listed **important notes**: ⇨ Calibrate the instrument at least once per year. ⇨ Measurements within limit ranges causes shorter calibration cycles. ⇨ It is Operator´s responsibility to define different calibration cycles/ verification cycles according to valid industry specific standards. ⇨ Calibration is required if other equipment (e.g. LN_2 cooling device) has been added or removed from the instrument. ⇨ Calibration is required after maintenance or repair (for example exchange of sensor, thermocouples or electronic components). ⇨ The calibration must be verified (using Indium) at least once per month.  Please refer to separate Document “Temperature- and Sensitivity Calibration for DSC214/ DSC204F1/ DSC3500” for further information regarding calibration of DSC Instruments.
Calibration Kit	A calibration kit is supplied with the DSC 214. NETZSCH calibrate your instrument prior to the delivery.

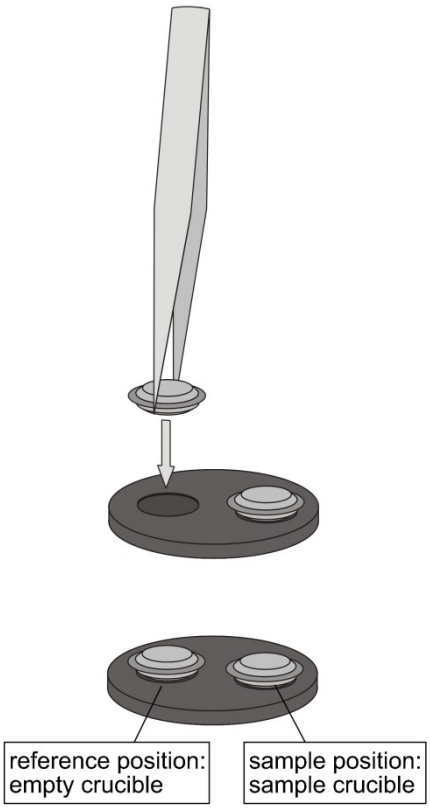
Preparing the Sample	
	When preparing the sample, the effects to be interpreted and the consistency of the sample must be considered. Good thermal contact between the sample and heat-flux sensor is an indispensable requirement for optimum results. The methods most frequently used for preparation of solid and liquid samples are described below.
Powdered solids	The sample is evenly distributed in the bottom of the sample crucible.
Compact solids	Compact solids, e.g. rubber or thermoplastic, are cut into thin slices with a knife, scalpel or razor blade. For the analysis, a hollow drill is used to punch out a sample disc of a suitable size from this larger disc (taken from the center of the whole sample). If it is not possible to punch out a suitably sized disc, the bottom of the crucible is covered with thin slices of the material. ⇨ A sample crucible must always be used. Direct application of the sample material increases the danger of contaminating the heat-flux sensor! ⇨ **PVC** PVC samples should only be heated in inert gas atmospheres (N_2) to a maximum of 220°C. Otherwise, HCl can evolve and cause serious damage to the sensor.
Films	Discs are punched from films with a hollow drill or punch pliers. The discs should completely cover the bottom of the crucible. In order to improve the contact between the sample and crucible bottom, the lid should be placed on the crucible, with the convex side down, and sealed.
Fibers	The fiber can be cut into small pieces, which are then spread parallel on the bottom of the crucible. The fiber is wound around a small rod. The coiled fiber is then removed from the rod and placed in the crucible. A bundle of fibers is wrapped with aluminum foil and cut at both ends. (The weight of the sample can be increased with voluminous fiber materials.) The fiber material with the foil wrapping is then placed in the crucible. The significance of the experimental results can be increased in all cases by adding a drop of silicone oil (improves the heat transfer).

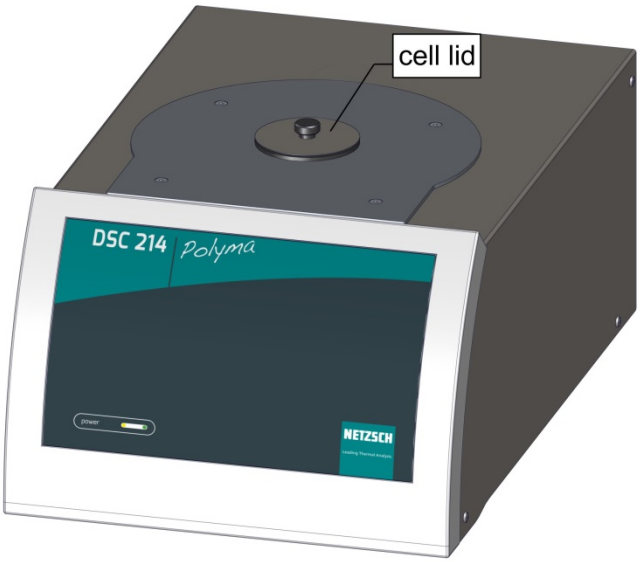
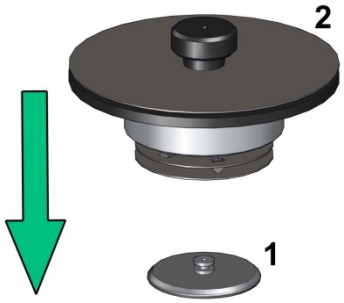
Liquids	Depending on the viscosity, liquid samples can be dropped into the crucible with a thin glass rod, a micro-pipette or a syringe.
Unstable samples	Unstable samples are tested in special pressure-tight crucibles (optional). The measuring cell must be recalibrated when pressure-tight crucibles are used.
Evaporation reactions	When investigating evaporation reactions, e.g. evaporation of water of crystallization, a closed crucible with a small hole in the center of the lid should be used. The hole should be punched prior to sealing to avoid deformation of the crucible later.
Measurements in defined atmospheres	For measurements in defined atmospheres, a needle should be used to pierce one to five holes in the lid of the crucible. The holes should be made prior to sealing to avoid deformation of the crucible later. It is also an option to run the measurement without a lid on the crucible.
Select the sample crucible	 See chapter V: crucibles
Weigh the sample	**Weigh** the sample with an analytical balance. Accuracy: ± 0.01 mg  ⇨ **Clean** the crucible and lid with acetone or alcohol prior to use. ⇨ When filling the crucible, no sample material may remain on the edge of the crucible. Otherwise, when cold-sealed, the bond between the crucible and lid may not be tight. ⇨ After crimping, the bottom of the crucible must be plane and free of scratches. If the bottom of the crucible is uneven, the heat transfer between the heat-flux sensor and the sample is impaired!  ⇨ For application temperatures above 350°C, the crucible lids should be pierced.

Seal the crucible 	Certain types of crucibles (see chapter V: crucibles) can be cold-sealed with a special sealing press.  **The operation of the sealing press is described in a separate manual!**

Insert the crucibles	*The lids could still be hot. Danger of burns!* *If possible, lay the lid on the metal plate of the measuring cell.*  • Remove the cell lid and the interior lid.  *Use the tweezers for lid for removing the separate cell lids.*

Sample crucible	• **Pick up** the sample crucible from the top with the tweezers and place it exactly in the center of the right-hand support of the heat-flux sensor.  

Reference crucible	 The heat-flux resistance is primarily determined by the crucible and not by the relatively small sample weight. In general, an empty crucible is used as the reference. If a strong baseline drift occurs with samples with a high specific heat, this asymmetry can be compensated by using an inert reference material. The properties of the reference material (thermal conductivity, specific heat, etc.) should be similar to those of the sample. • **Pick up** the reference crucible from the top with the tweezers and place in the center of the **left-hand** support of the heat-flux sensor. 

Place the cell lids	 • Replace the interior lid and the cell lid.  *Prior to inserting the lid wait until the furnace has cooled down to at least 100°C. If the lids are installed at higher temperatures and eventually canted, a kind of welding between the surfaces (furnace and cover) can occur, caused by the fast warming of the silver cover. Then the only way is to loosen the lid violently, that can involve a damage of the furnace and cover.* *Use the tweezers for lid for inserting the separate cell lids.*

Generate Purge Gas Atmosphere

Measurements can be run in static or dynamic atmospheres.

Conducting measurements in a protective gas atmosphere has a positive influence on the service life of the measuring cell.

Recommended:

Use a dry, inert purge gas in the sample chamber above 500°C.

Absolutely necessary:

For operation of a LN_2 cooling device or of an Intracooler, the use of a dry, inert protective gas for the furnace chamber (gas inlet "protective") is absolutely necessary.

The purge gas inlets purge 1 and purge 2 are connected to one another with a tee inside the measuring cell. This allows a gas change during the measurement.
If possible, carry out the gas change within a short, isothermal program step. You will thus achieve a clean separation of the atmospheres.

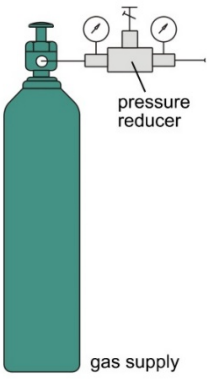
Recommended purge gas rates:

purge 1 approx. 40 ml/min
purge 2 approx. 40 ml/min
protective approx. 60 ml/min

If one of the purge gas inlets is not used it has to be closed with a blind plug.

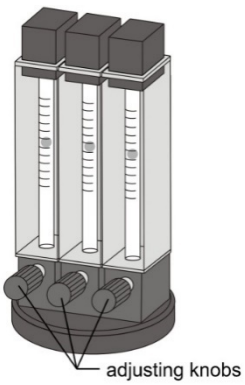
Adjusting the gas flow

Option 1:
(sintering inserts)



If no gas flow device is available, the actual gas flow value is determined by the overpressure which

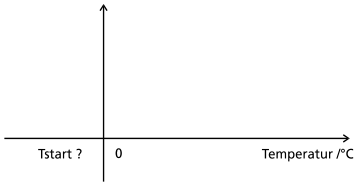
Option 2:
(gas flow control device)



If your DSC 214 is equipped with a gas flow control device the gas flow can be adjusted via the adjusting knobs on the front.

	is set at the gas pressure reducer at the gas bottle. For each gas flow channel, i.e. purge 1, purge 2 and protective a calibration table is given for each instrument where it is shown which nitrogen overpressure has to be set in order to achieve a desired nitrogen gas flow.	**Option 3:** (MFCs) The gas flow for purge 1, purge 2 and protective is controlled by the means of MFCs and can be adjusted via the software (see Help System Software).
Hints for special gases by using sintering inserts	For special gas types the overpressure to be set can be calculated according the formula: overpressure to be set for special gas flow = overpressure to be set for nitrogen* calibration factor where the calibration factor in good approximation is given by: $calibrationfactor = geometryfactor \cdot \sqrt{\rho_{special\ gas} / \rho_{nitrogen}}$ Calibration factors for special gases: Oxygen 1.01 Argon 1.23 Helium 0.97 The accuracy of the gas flow setting by adjusting the overpressure at the gas pressure reducer is strongly dependant on the quality of the gas pressure reducer. Approximately ± 5 ml/min can be achieved.	
Purge gas switching (O.I.T. extension)	 If your instrument is equipped with internal magnetic valves (6.240.20-04.0.00), the gas flow can be programmed switched on/off via the software (two for purge gas inlet to the sample and one for protective gas for the measuring cell).	

Set the start temperature for the measurement

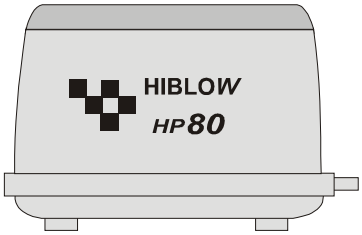


The start temperature for the measurement is entered via the software.

It is frequently the case that the temperature in the sample chamber is higher than the start temperature for the measurement.

In order to reach the required start temperature, there are several cooling options available.

Cooling with the Linear Small Compressor



The measuring cell can be cooled to above room temperature through the introduction of air (linear small compressor).

Use air-cooling to effectively cool the cell to approx. 100°C.

Air-cooling can be switched on via the software.

Handling

Be sure that the linear small compressor is connected to the measuring unit (see: chapter II – Installation)

The air cooling is switched on and off depending on the software presettings.

The compressed air leaves the outlet of the cooling channel with high speed. Depending on the furnace temperature it might be hot. Please ensure that no damage can be caused for these reasons.

Cooling with Liquid Nitrogen

General information

Cooling with liquid nitrogen is required, if:

- cooling processes are being investigated, i.e. if the start temperature is higher than the end temperature.
- measurements that are to be started below 0°C are normally carried out at a heating rate ≥ 5 K/min.

To prevent icing of the measuring cell:

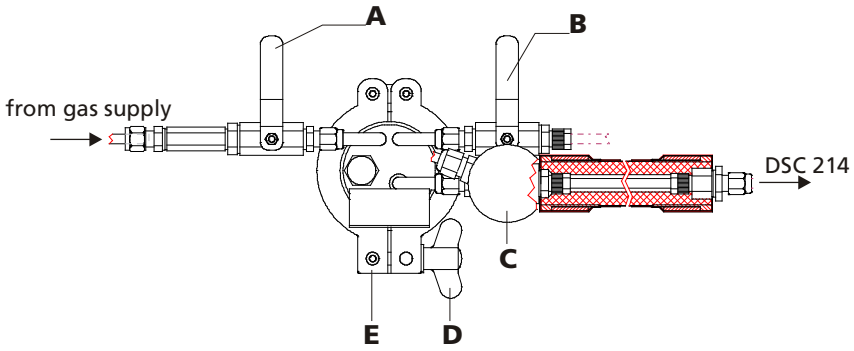
- **Feed** a dry protective gas, e.g. N_2 (60…100 ml/min) into the furnace jacket (use gas inlet "protective").
- **Purge** the sample chamber (N_2, 40…50 ml/min, use gas inlet "purge 1" or "purge 2").
- Due to the good heat transfer, helium **can** also **be used** as a purge gas at very low temperatures (reduces the sensitivity of the sensor).
- **Do not change** the gas flow rate (protective, purge) during the measurement. This will eliminate effects on the sensitivity of the measuring cell.

ATTENTION!

Please pay attention to all safety regulations for handling liquid nitrogen (see chapter I).

How to operate the CC300 cooling system (0CC30060B10.000-00 or 15.000-00)?
See separate operating manual CC 300!

Cooling with the LN2 Cooler CC 200 F3 (magnetic valve controlled)



Handling	Be sure that the LN2 cooler is connected to the measuring unit (see: chapter II – Installation) It is possible to use a dry, inert gas for pressure generation. In that case open your gas supply. Open the shut-off valve (A). Wait until the pressure in the dewar vessel reaches 0.3 bar. Pressure generation is enhanced when the dewar is not completely filled. It is not necessary to keep shut-off valve (A) open when the content of the dewar is less than 50%. The magnetic valve opens and closes the nitrogen cooling depending on the software presettings.  Be sure that shut-off valve (A) is closed when dewar is empty.
Removing the LN2 cooler from the dewar vessel	Close your gas supply. Close the shut-off valve (A). Open the shut-off valve (B) to reduce pressure in the dewar vessel. Watch the manometer. If normal pressure has been reached: Loosen the butterfly nut (D). Remove the clamping ring (E). Draw out the LN_2 cooler.

On-Off Valve Kit for Compressed Air Cooling

Handling	Be sure that the on-off valve kit for compressed air cooling is connected to the measuring unit (see: chapter II – Installation). The air cooling is switched on and off depending on the software presettings.
⚠	The compressed air leaves the outlet of the cooling channel with high speed. Depending on the furnace temperature it might be hot. Please ensure that no damage can be caused for these reasons.
⚠	If liquid nitrogen cooling (6.351.23/24/25/35) is installed the highest allowed air pressure is 1 bar overpressure. If liquid nitrogen cooling (6.351.23/24/25/35) is **not** installed the highest allowed air pressure is 2 bar overpressure.

Cooling Device for Pressurized Air

Handling	Be sure that cooling device for pressurized air is connected to the measuring unit (see: chapter II – Installation). The air cooling is switched on and off depending on the software presettings.
⚠	The compressed air leaves the outlet of the cooling channel with high speed. Depending on the furnace temperature it might be hot. Please ensure that no damage can be caused for these reasons. **It is not possible to use the cooling device for pressurized air with simultaneous connected liquid nitrogen cooling!**

Intracooler 40

Temperature range: -40°C up to 600°C

See separate manual **Intracooler 40**!

The use of a dry, inert protective gas for the furnace chamber (gas inlet “protective”) is absolutely necessary.

Intracooler 70

Temperature range: -70°C up to 600°C

See separate manual **Intracooler 70**!

The use of a dry, inert protective gas for the furnace chamber (gas inlet “protective”) is absolutely necessary.

Maintenance

- **Make** absolutely sure before every measurement that the measuring cell is clean.
- **Clean** the measuring cell with an organic solvent (acetone, alcohol), using a lint-free cloth or paper towel.
- Carefully **remove** heavy soil from the surface with a glass fiber brush or a fine diamond sponge.

  Extreme caution should be exercised when using a diamond spo remove soil.

  Do not apply too much force!
- After **cleaning** remove all particles (vacuuming).
- Afterwards, the cell must be cleaned with acetone and baked out.
- If not absolutely required to achieve the goal of the investigation, the measurement should not be run to the maximum temperature of the cell.
- Do not operate the instrument at the maximum temperature for measurements of longer duration.
  To achieve an optimal lifetime of the measuring cell it is recommended to prevent isothermal segments above 500°C and heating rates < 10 K/min (exception: baking out the measuring cell for cleaning purposes, see below).
- **Begin** cooling just above the sample reaction (e.g. 20 - 30 K above the melting point).
- By setting a reasonable temperature program, you **minimize** the danger of contaminating the cell.
- Whenever possible, **use** a protective gas in the sample chamber.

- **Bake out** the sample chamber if it is necessary due to contaminations.

  Procedure:
- Heat the cell to 600°C (operation with Intracooler: 570°C) and hold the temperature constant for maximum 30 minutes.
- If possible, **avoid** measurements with samples that create heavy soot or soil.
- It is imperative that the sensor surfaces remain scratch-free.
- Do not introduce LN_2 into the system when temperatures are above 450°C.

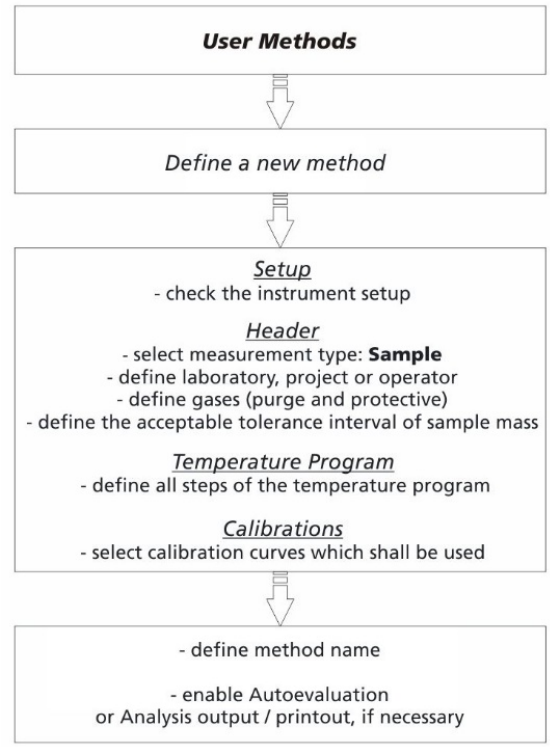
Short instructions – How to Start a Measurement

Explained below is how to:

- **set up** a method**,**
- and **measure** a sample

Detailed information on the individual steps can be found in the hardware and software manuals.

Set up a Method

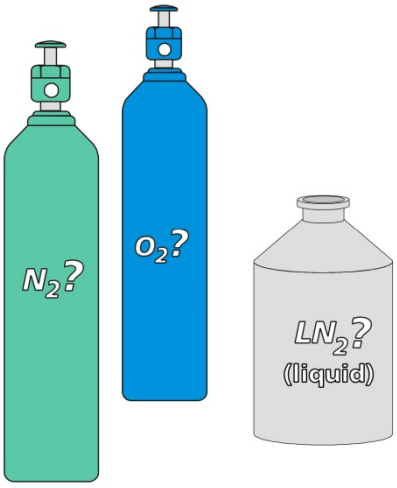
Gehen Sie wie folgt **vor**, um eine Methode zu erstellen:	

Execute a Measurement

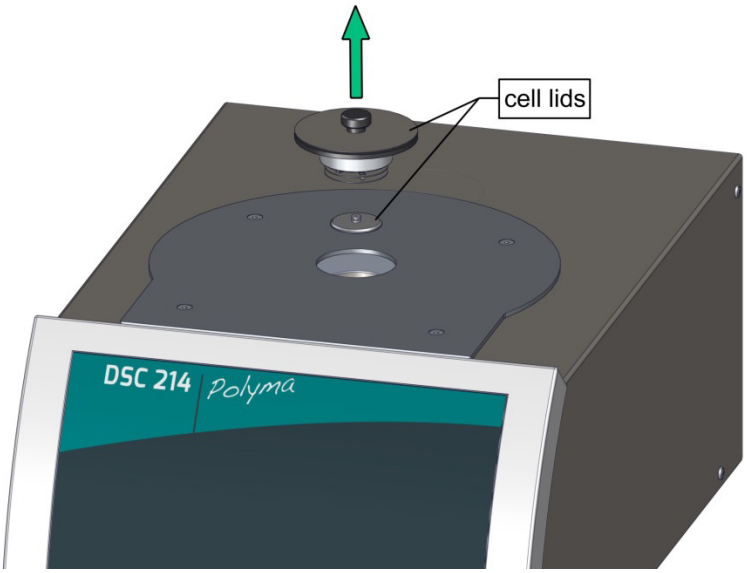
1. **Make sure** that all system components are correctly connected and that the cell is clean.

2. **Switch on** the computer system and measuring unit approx. 60 min. before starting the measurement.

3. Depending on your measurement conditions, have available the required supplies:
liquid nitrogen,
purge gases, etc.



4. **Open** the measuring cell by removing the cell lids.



5. **Set** the required amount of purge gas.

Option 1:
Sintering inserts

Option 2:
Gas flow control device

Option 3:
MFCs

HARDWARE

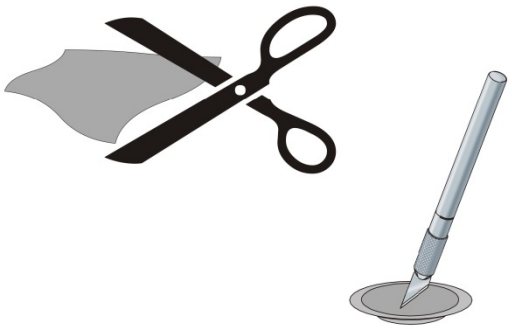
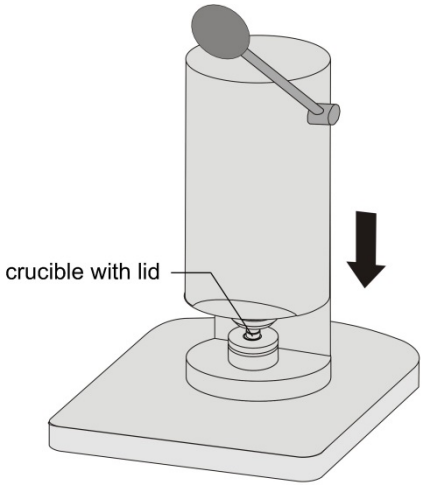
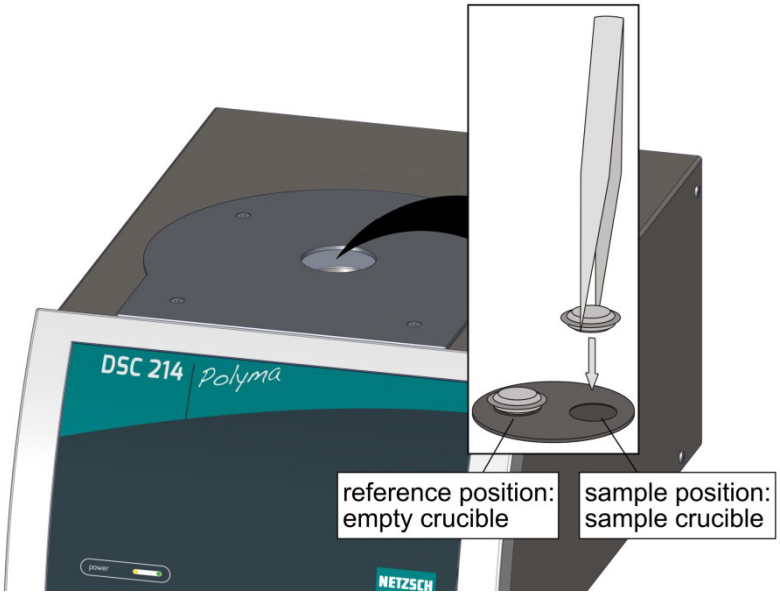
Chapter IV
page 62

6. **Start** the DSC 214 measurement program.

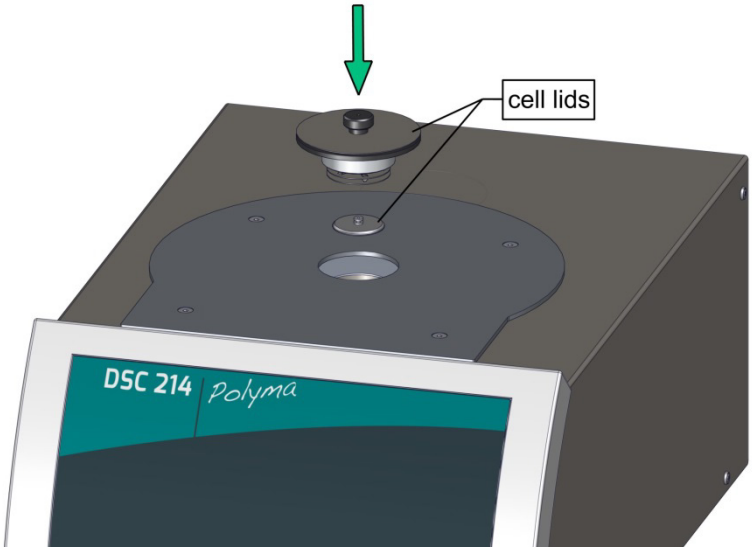
Note for *Nevio* instruments:
While working with *Proteus Protect* you must have the necessary permissions to start the measurement part of the software. This will be checked during the authorization process when you type in your ID and password.

Your possibilities in software handling depend on your permissions as well. Therefore it could be that some of the following steps cannot be realized in the described way.

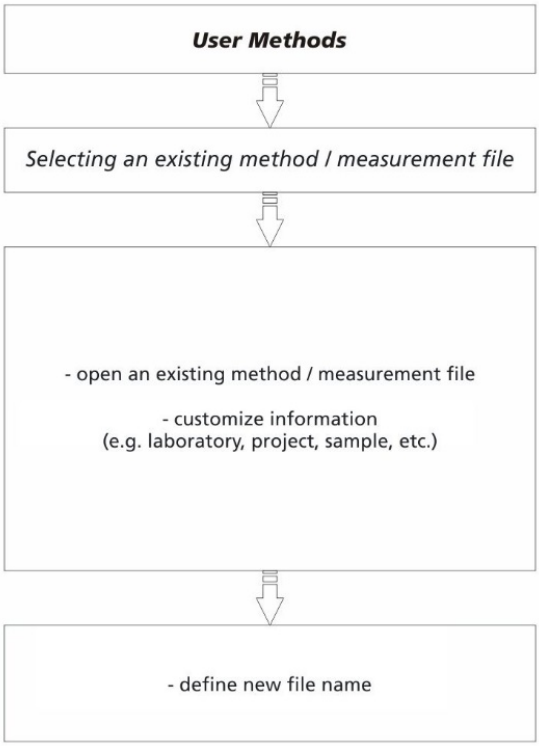
Please ask the *Proteus Protect* software administrator of your lab for further information.

7. **Prepare** the sample and, if necessary, **pierce** the crucible lid.		***HARDWARE*** Chapter IV page 55
8. **Weigh** the sample.		***HARDWARE*** Chapter IV page 56
9. **Seal** the crucible with the lid.		***HARDWARE*** separate manual
10. **Insert** the crucibles.		

11. Replace the cell lids.



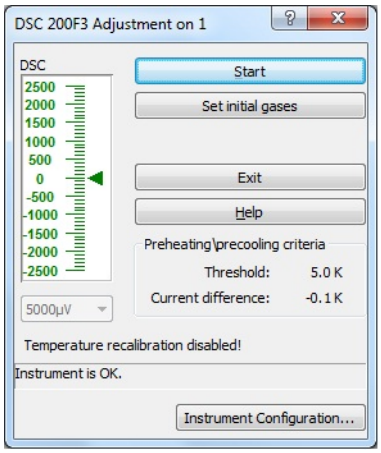
12.Proceed as shown to start a method:



For DSC measurements a baseline run is only necessary in special cases, for example, if the expected thermal effects are very small or for cp (=specific heat capacity) determination. In all other cases it is sufficient to perform just sample runs. A baseline run is a measurement using two empty crucibles.

13. **Switch on** the initial conditions if necessary.

Start the measurement.



14. **Open** the NETZSCH Proteus Analysis program.

After the measurement is finished, you can **begin** with the evaluation.

If the used method includes already evaluation procedures, the analysis software will execute these procedures automatically.

Note for *Nevio* instruments:

While working with *Proteus Protect* you must have the necessary permissions to start *Proteus* Analysis. Therefore it can happen that the evaluation of measurement curves is not possible.

Please ask the *Proteus Protect* software administrator of your lab for further information.

Using *Proteus Protect*:

- When the evaluation is finished the resulting state can be signed by persons who have the permission to do that.
- Evaluation procedures, which are included in the used method, will be executed independent of user permissions for opening the analysis software.

206030005 80032061 2020-12-03

Hints for Using Purge Gases

In dynamic mode the sample chamber is purged with gas during measurement.

Inert gases, air and some reaction gases (non toxic, non flammable) can be used.

In order to avoid environmental hazards or damage to the instrument, the following instructions must be followed when using purge gas.

Dry, inert gases are recommended for purging the sample chamber. We also recommend that the expelled purge gas be passed into a suitable exhaust hood. Depending on the measuring conditions (sample material, atmosphere, temperature range), derivative products can also be formed due to thermal reactions when using inert gases. These products (e.g. HCN, CO, SO_2, dioxine), even in small amounts, are very detrimental to health and must not be allowed to pass into the work room.

The user must decide in advance whether toxic gases might be released during a measurement. Should this be so, then safety precautions are absolutely essential.

When the measurement requires special reaction gases to be used in the sample chamber, then a safety check of the potentially hazardous gas or gas mixture is imperative. In this case the following aspects have to be taken into consideration:

Do explosive gas mixtures or explosive compounds evolve when the sample has contact with oxygen (air)?

Is it certain that no toxic compounds can evolve from the gases or gas mixtures when using the chosen application temperatures? In this connection, possible reactions between the purge gas in the weighing chamber and the reaction gas in the sample chamber should also be investigated.

Can it be ruled out that the gases neither corrode the leads nor the seals which are used in either the system or the surrounding equipment (valves, manometer or flow meter)? If not, then leakages are to be expected.

What side effects do the gases or gas mixtures have on the system's accessories? These effects do not result in leakages but would considerably increase wear and tear or could lead to a total failure of the system.

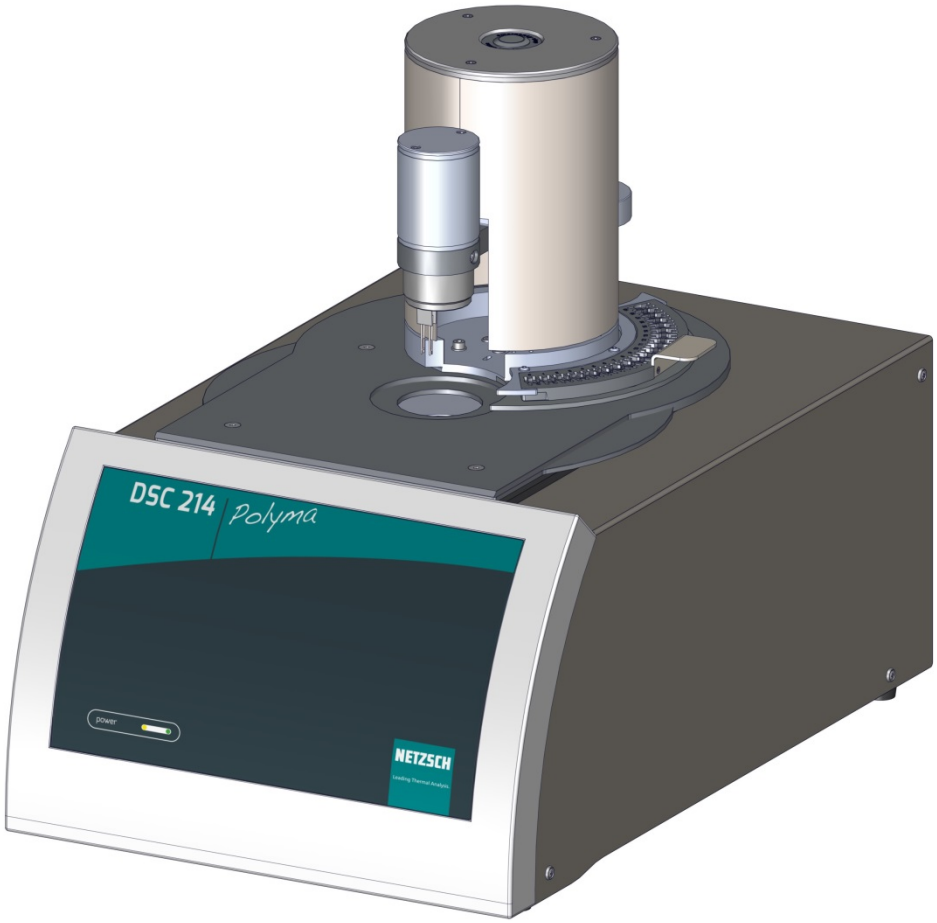
Under no circumstances may explosive gas mixtures be used. The mass flow controllers are not designed for the use of corrosive, flammable or reducing gases!

Information concerning the possibilities of using various gases can be found in the appropriate technical literature or can be requested from the manufacturer or retailer of gases.

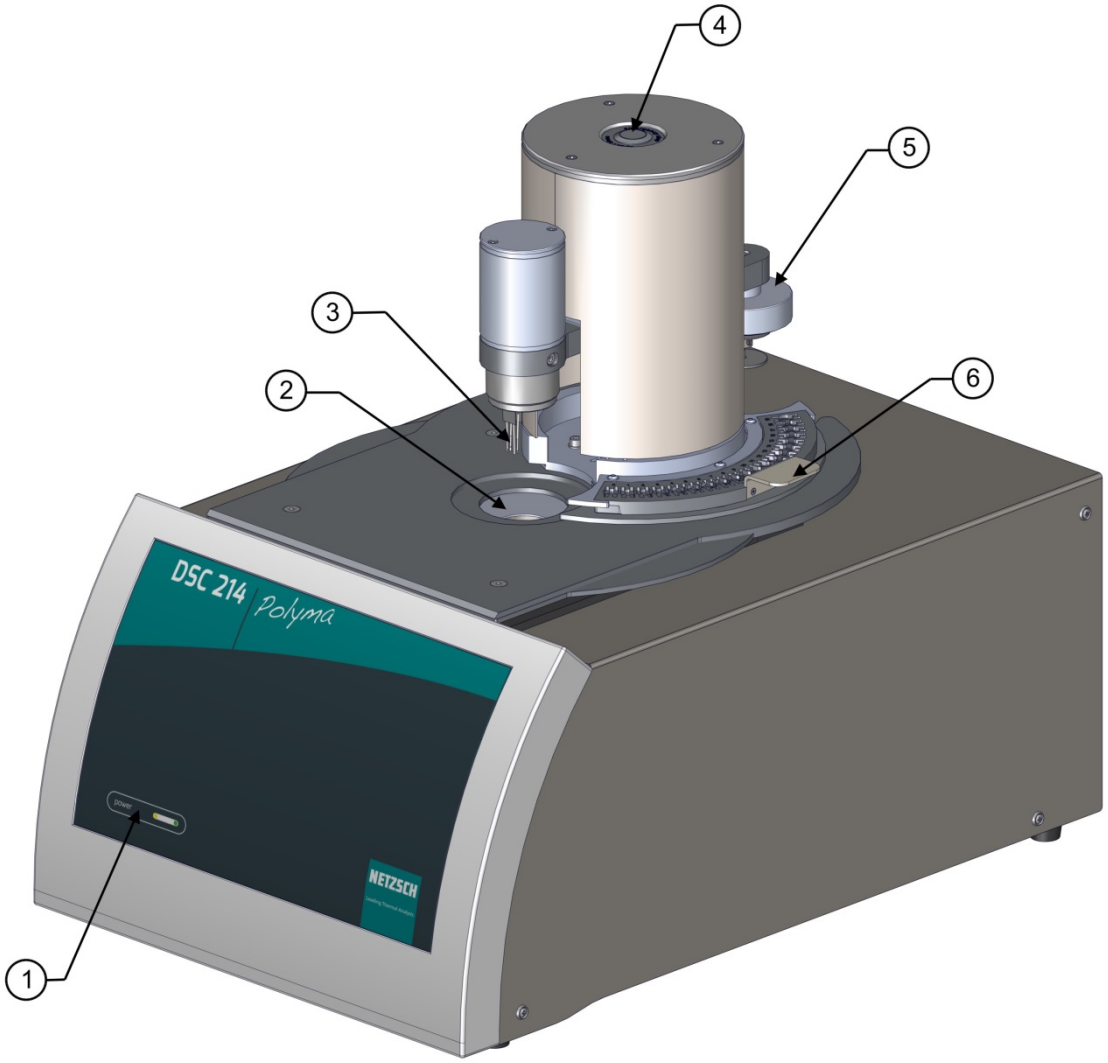
For more information refer to the list of selected purge gases, see section “**Fehler! Verweisquelle konnte nicht gefunden werden.**”, page **Fehler! Textmarke nicht definiert.** and following pages.

DSC 214 with Automatic Sample Changer (ASC)

The ASC sample changer is an extension of the DSC 214 *Polyma* and DSC214 *Nevio*.
When turning on the instrument the ASC is automatically switched on. The instrument executes a reference run automatically at the first motion request. As soon as the gripper has reached its rest position the ASC is ready for operation.

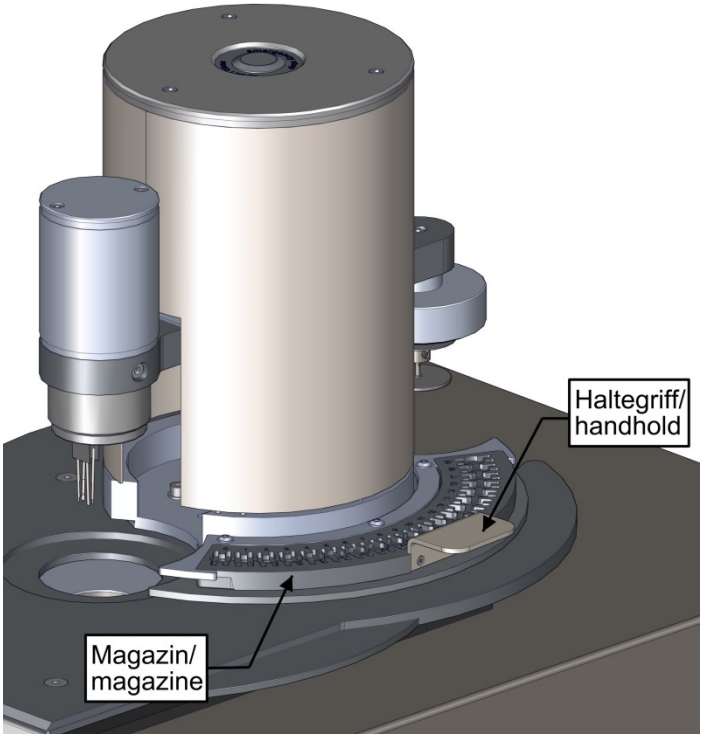


Measuring Part

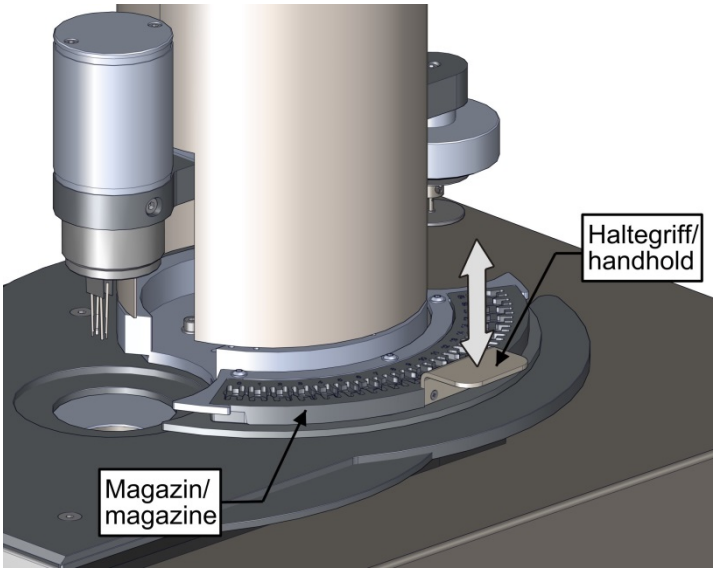


1	*control display (on/off)*
2	*measuring cell*
3	*gripper*
4	*function key (open/close, emergency stop)*
5	*cell cover*
6	*crucible magazine*

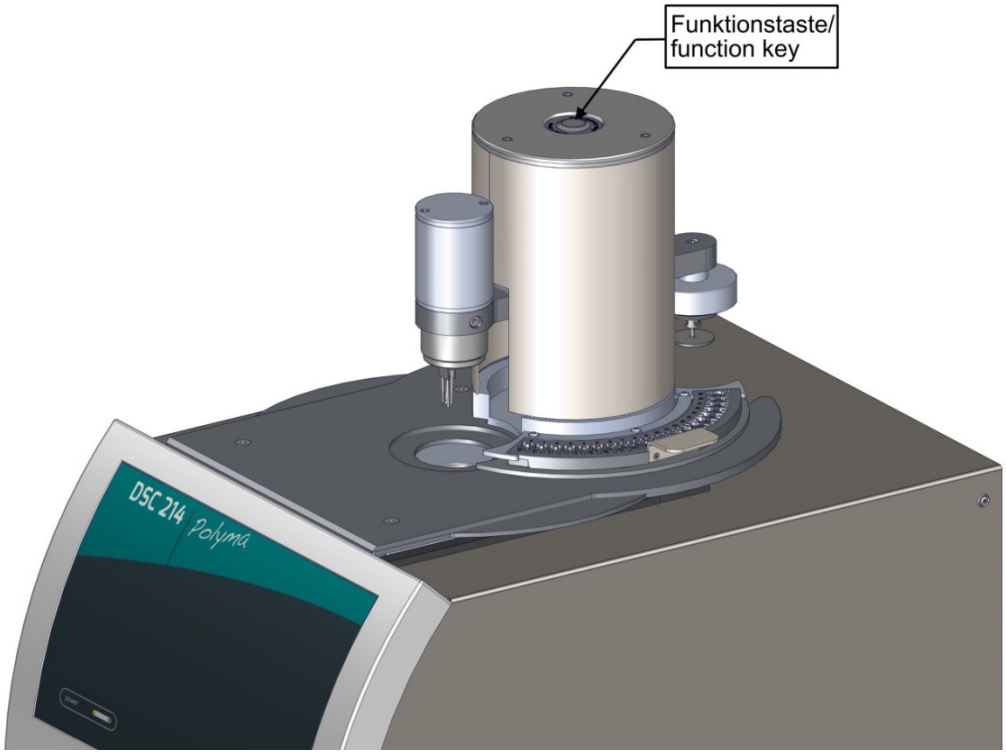
Magazine



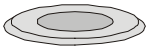
The magazine can be loaded with up to 20 crucibles. It can be removed from the instrument by means of the handhold.



Control Panel ASC



Function key

	***Triple assignment of the function key:*** • ***Manual mode (ASC manger off)*** *open and close the measuring cell and emergency reset during movement of the ASC* • ***ASC mode*** *emergency reset during movement of the ASC*

Prior to restarting:

- *check crucibles and their correct positioning*

ATTENTION!

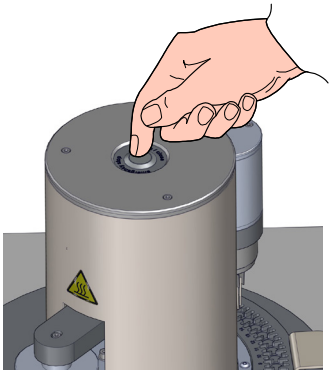
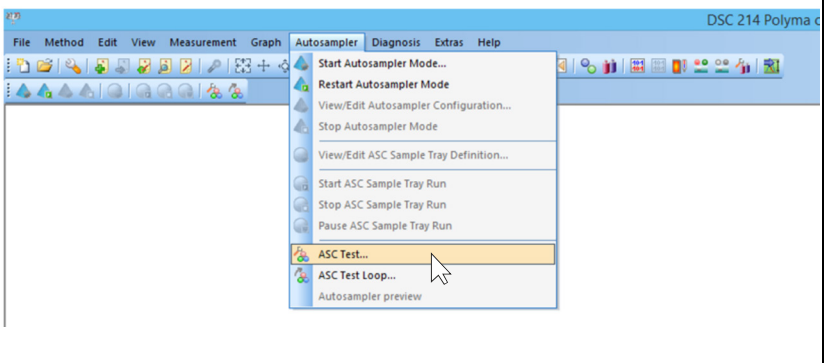
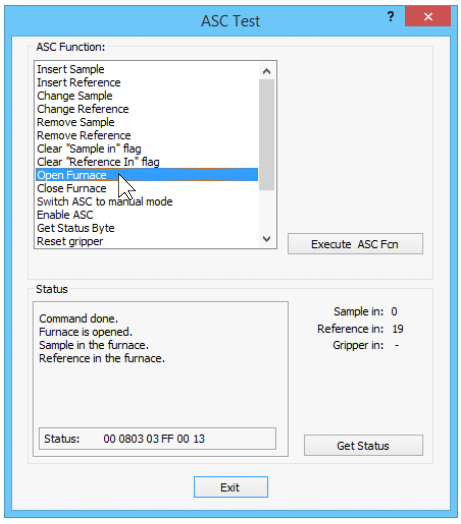
If the sample still remains in the gripper after an error (e.g. sample sticks on the gripper) or after switching on the measuring part, the gripper is moved to the rear position. To open the gripper, push the function key and remove the sample. After the sample was removed, the measuring cell is completely opened and operation can be continued.

Crucible Waste Receptacle

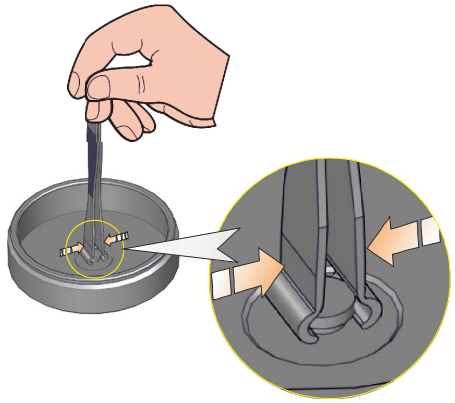
For disposing of used sample crucibles.

NOTE

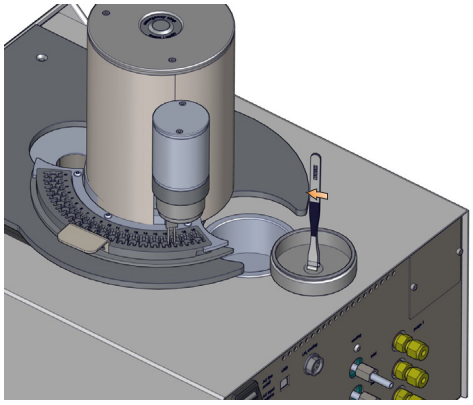
Only available for DSC 214 *Polyma* with ASC.

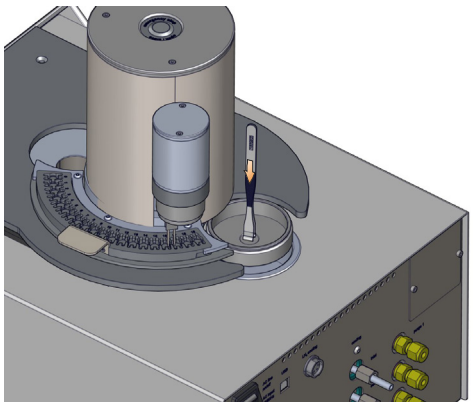
ATTENTION!	**There are two different ways to move the ASC into the initial state:**
Manual Mode: Open the measurement cell by means of the "*open/close*" function key of the ASC.	
ASC Mode: Open the measurement cell by means of the "*Open Furnace*" command in the "*ASC Test*" section of the Proteus software.	 

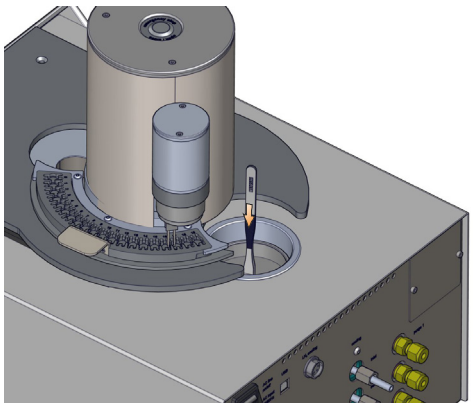
Operation: Insert Crucible Waste Receptacle



- Grab the crucible waste receptacle at the knob in the center, by means of a pair of tweezers.

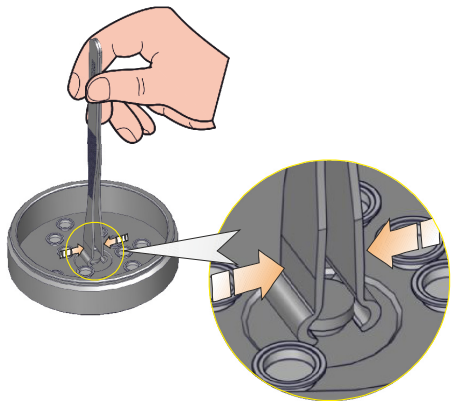




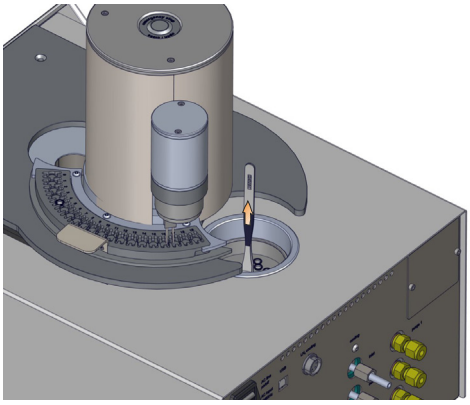


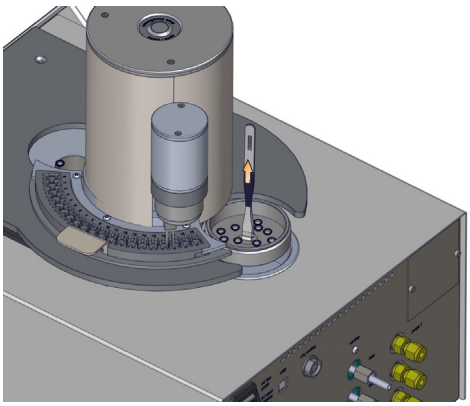
- To insert, move the crucible waste receptacle first sideways and then downwards, as shown in the figure below.

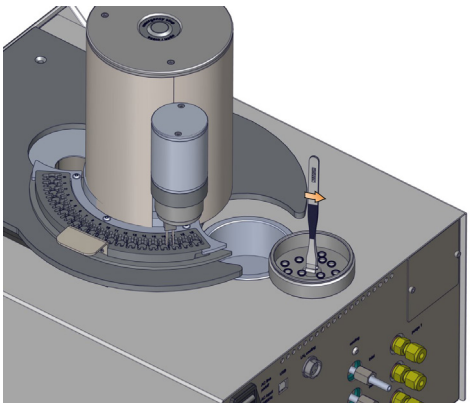
Operation: Remove Crucible Waste Receptacle



- Grab the crucible waste receptacle at the knob in the center, by means of a pair of tweezers.







- To remove, move the crucible waste receptacle first upwards and then sideways, as shown in the figure below.

Transport

ATTENTION!

In order to lifting up and transport the measuring part two people are required. Touch the measuring part on the bottom plate as shown in the figure to lift it upwards.


Cleaning

ATTENTION!

For cleaning purposes contact qualified NETZSCH service staff!

ATTENTION!

Please take care to all safety regulations and laboratory instructions for the handling of chemicals!
Before using any cleaning or decontamination methods except those specified by the manufacturer, responsible bodies should check with the manufacturer that the proposed method will not damage the equipment!

DSC 214

NETZSCH

Chapter V - Appendix

206030005 80032061 2020-12-03

Technical Specifications

Heating System	
Temperature range:	-170°C … 600°C
Heating rates:	0.001 … 500 K/min (dependent on final temperature)
Cooling rates:	0.001 … 500 K/min (dependent on final temperature)
Cooling System (cooling time, free cooling, 600°C to RT)	
liquid nitrogen:	approx. 3 min
linear small compressor:	approx. 45 min (0.2 bar excess pressure, RT=40°C)
Intracooler 40:	approx. 4 min
Intracooler 70:	approx. 4 min
Measuring System	
DSC measuring range:	+/- 750 mW
Technical resolution:	0.1 µW
RMS noise:	1 µW
Peak-to-Peak noise:	5 µW
Enthalpy precision: Enthalpy accuracy:	0.05% (for indium) < 1% for metals, < 2% for most materials
Atmosphere	
Gas atmosphere	inert, oxidizing, static and dynamic operation

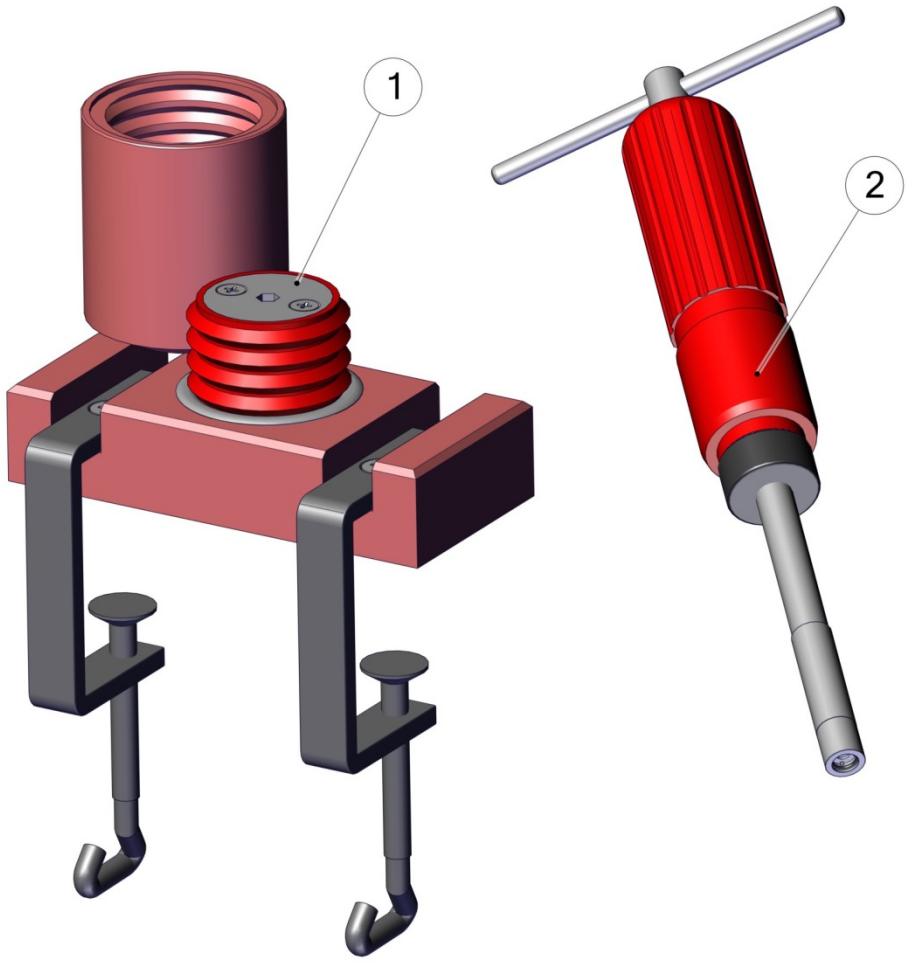
Dimensions in mm (w x d x h) / Weight (net) in Kg	
Measuring unit:	350 x 560 x 280 / 23
Power Supply	
	230 V / 115 V, 50 Hz / 60 Hz, < 600 W (without cooling accessory)
Operational Conditions	
	Indoor use (laboratory) Ambient temperature 20°C +/- 5°C Relative air humidity 60% +/- 20% Atmospheric pressure 1013 hPa +/- 30 hPa Wall distance min. 30 cm

Technical data subject to change

Sealing Tool

General Information

Sealing press for high-pressure sample crucibles 6.239.2-92.3.00, -92.31.00, -92.6.00, -92.8.00, -92.9.00, -93.3.00, -93.31.00, -93.4.00, and -93.41.00 consisting of base plate, two-part press and adjustable torque wrench, with operating instructions.



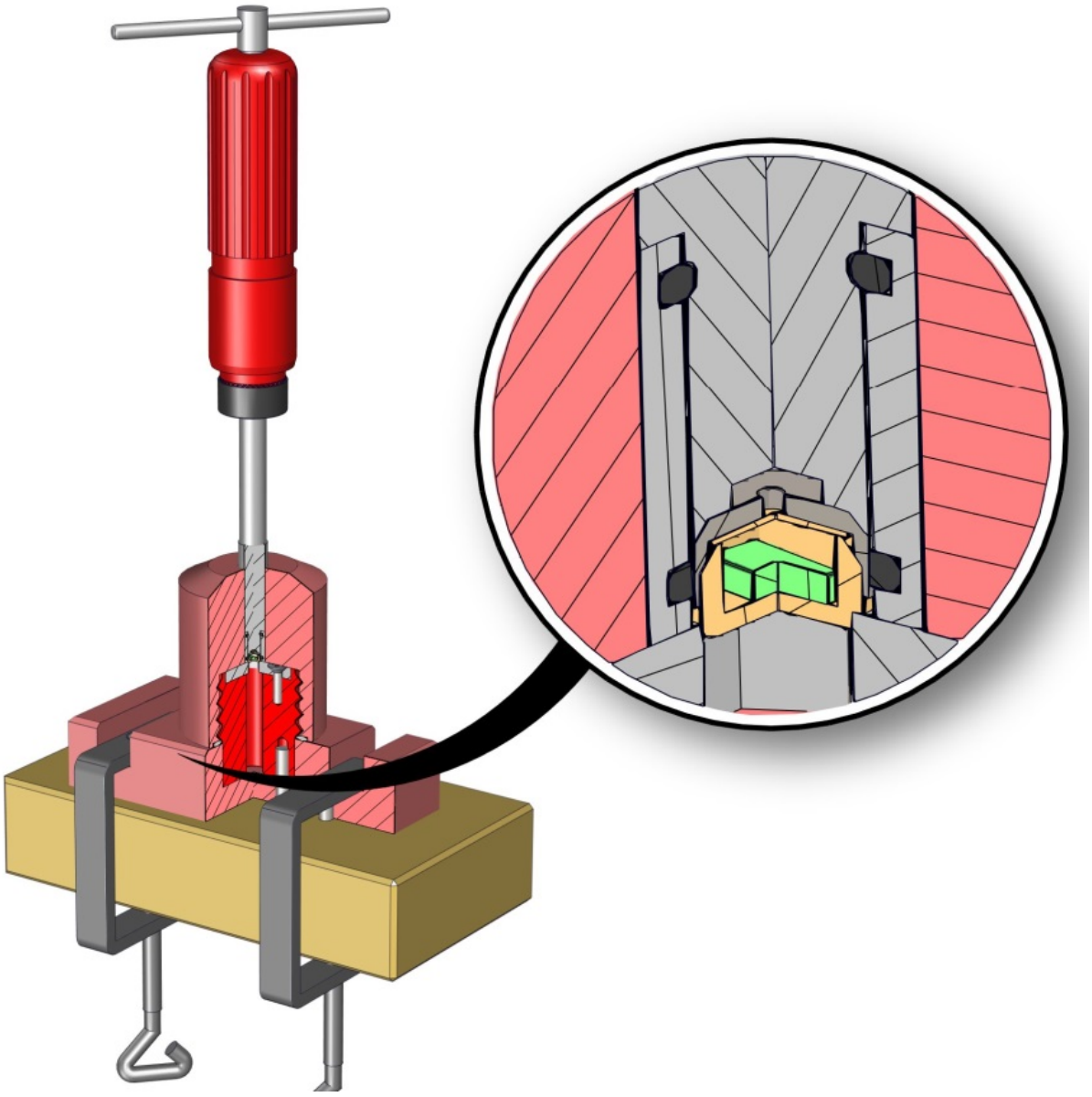
Complete sub-assembly
(6.239.2-92.4.00)

Sealing press for high-pressure sample crucibles

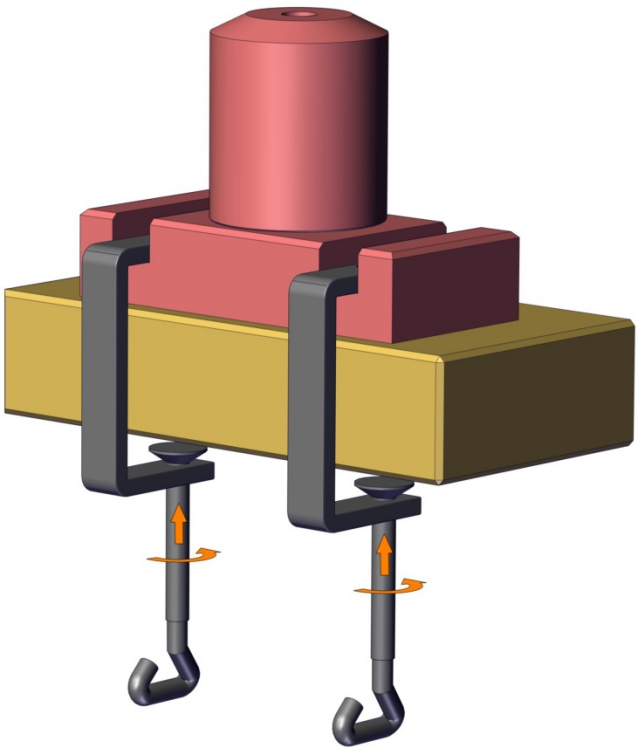
No.	Description
1	support fixture for high-pressure crucibles (GB396775)
2	dynamometric key for high-pressure crucibles (GB396776)

Intended Use

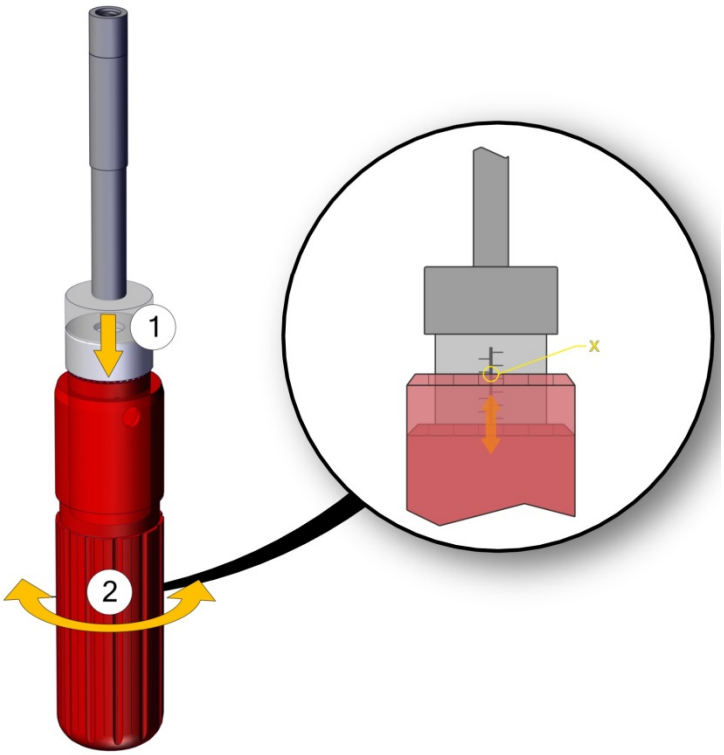
The support fixture of the sealing press set is used to correctly position the high-pressure crucible with sample, which is then closed firmly and tightly with a defined moment of force by means of a torque wrench.



Mounting the Support Fixture for High-Pressure Crucibles

	• Mount the support fixture to your table's work plate using the butterfly screws.

Adjusting the Torque Wrench



x = area for reading the value for the desired moment of force

- By pulling the locking sleeve ① downward, you will be able to adjust the desired moment of force with the handle ②.

NOTE!

Please also refer to the manufacturer's operating manual, which is included in the scope of delivery!

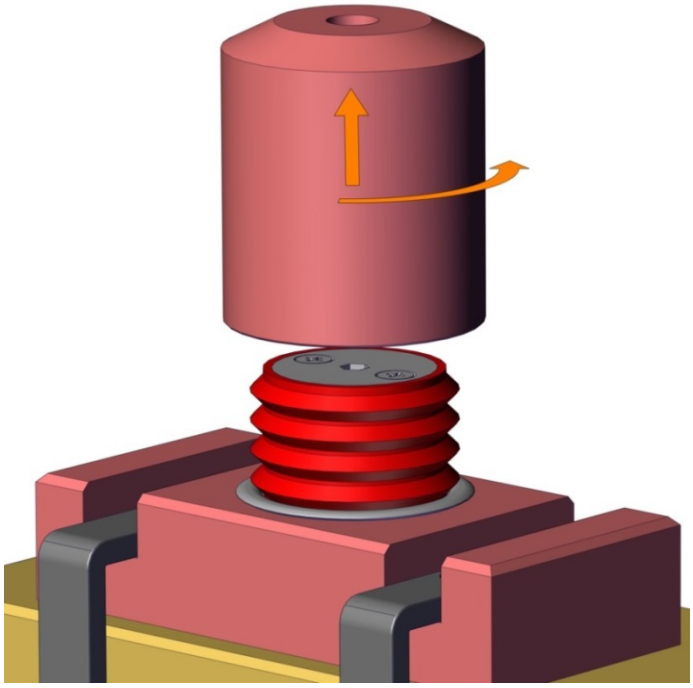
NOTE!

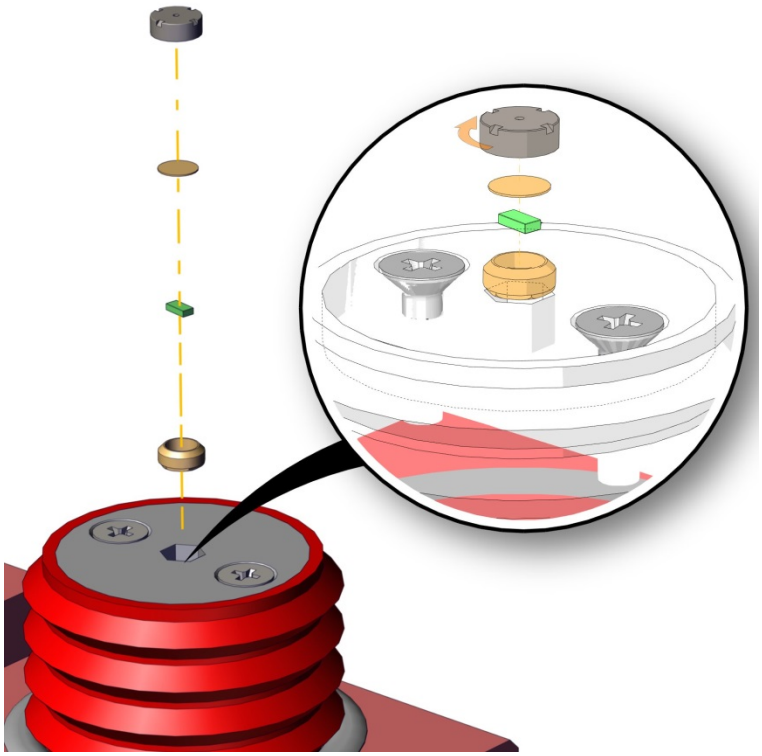
We recommend a moment of force of **3.7 Nm** for correct sealing of the crucible!

- Make sure that the locking sleeve is returned to its upward position after adjusting the moment of force. If necessary, slide it into this position manually.

Using the Sealing Press Set with a High-Pressure Crucible

	NOTE! The Au sealing discs which are included in the high-pressure crucible set serve as bursting discs (a safety device). Before using, please note the following information: • The Au sealing discs are intended for single usage only. • Care must be taken that the protective function of the Au sealing disc as a bursting disc is not affected by contamination from the sample itself or by out-gassing of any substance from the sample. It is the operator's responsibility to ensure this.

	• Unscrew the guide.

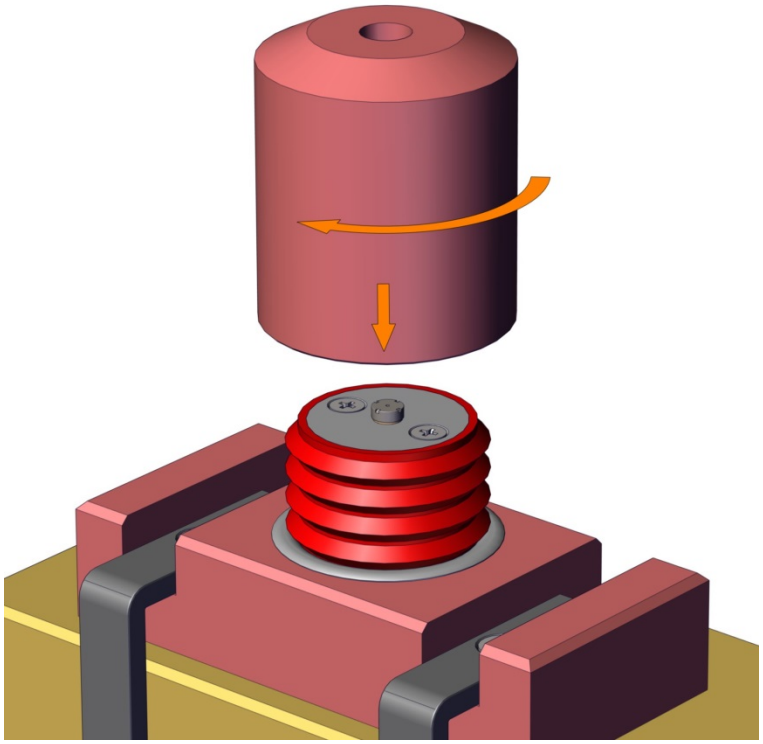


- Insert the high-pressure crucible into the hexagon support.
- Next, insert the sample.
- Then insert the gold sealing disc centered on top of the high-pressure crucible.
- Finally, screw on the high-pressure lid securely by hand.

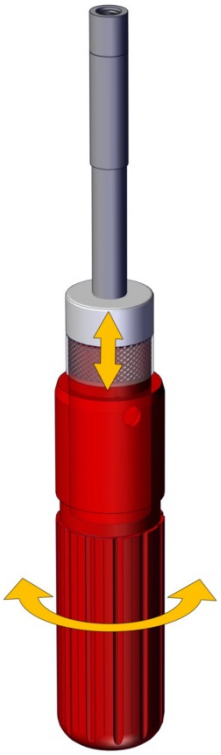
NOTE!

For inserting the sample and positioning the gold sealing disc, the use of a pair of tweezers is recommended!

For clean handling, the use of appropriate gloves is recommended.



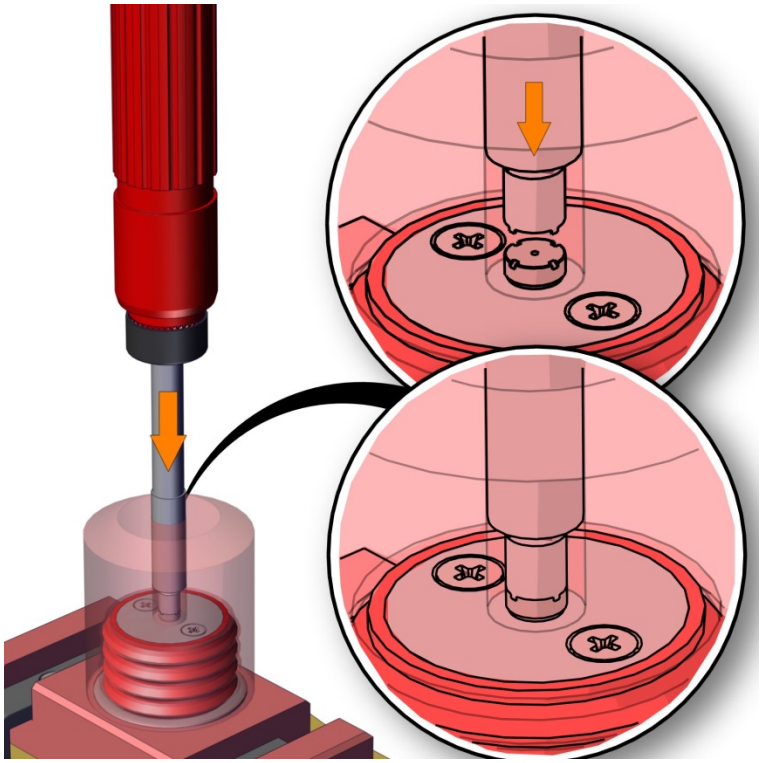
- Screw the guide securely back onto the foundation by hand.



- Adjust the torque wrench. See the “Adjusting the Torque Wrench” section.

ATTENTION!

Please be sure to apply the prescribed moment of force here!



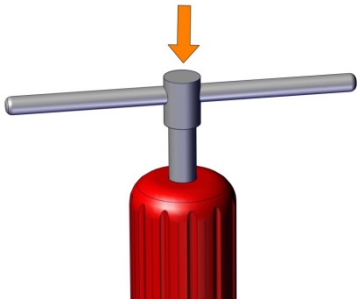
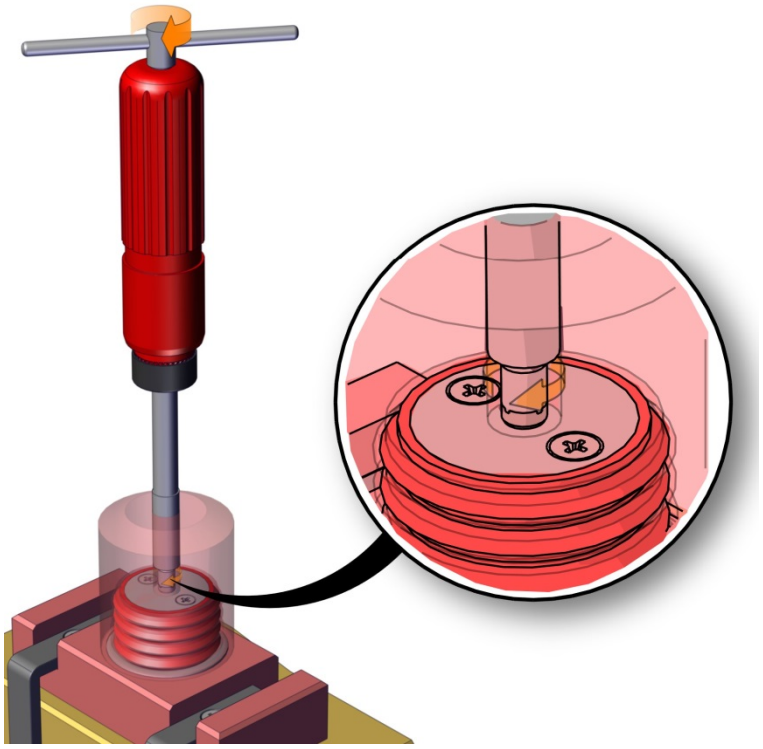
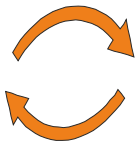
- Now insert the torque wrench into the drill hole of the guide and move it downward carefully.
- While carefully rotating the torque wrench, seek out the locking notches on the high-pressure lid.

ATTENTION!

Use extreme caution so that you do not damage the grooves on the lid or the notches on the key!

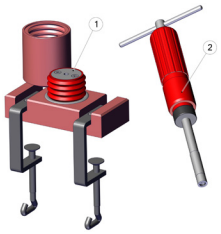
NOTE!

For this step, use the torque wrench provided!

	• Next, insert the square key into the torque wrench.
	• Hold the handle with one hand and rotate the square key at the top with the other until the torque wrench clicks and the desired moment of force has been reached.
	• Afterward, remove the torque wrench. • Unscrew the guide. • Remove the tightly closed high-pressure crucible containing your sample. You can now use it for your measurement. **NOTE!** Heed the maximum allowable pressure and temperature for each crucible!

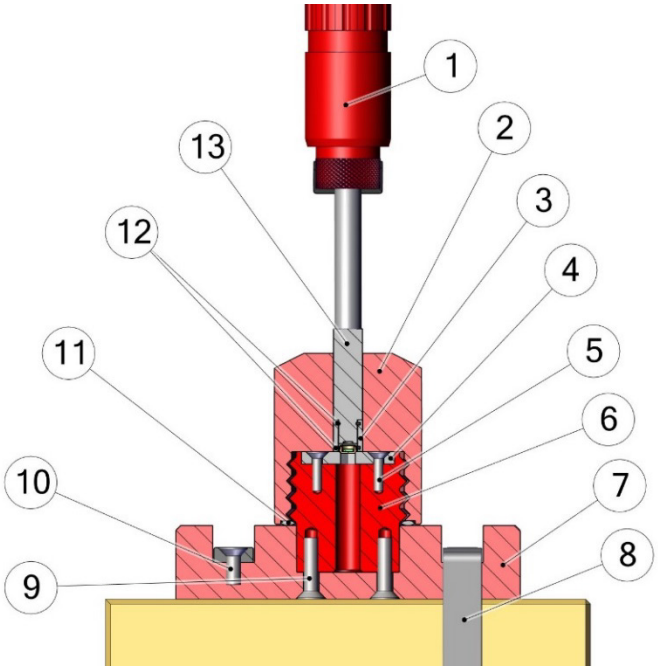
Spare Parts List

Sealing press for high-pressure sample crucibles 6.239.2-92.3.00, -92.31.00, -92.6.00, -92.8.00, -92.9.00, -93.3.00, -93.31.00, -93.4.00 and -93.41.00 consisting of base plate, two-part press and adjustable torque wrench, with operating instructions



6.239.2-92.4.00

Component Parts of 6.239.2-92.4.00:



No.	Description	Quantity	Order No.:
1	Torque wrench for sealing tool 6.239.2-92.4.00	1	GB396776
2	Guide	1	400 01 02 *
3	Lid support for sealing tool 6.239.2-92.4	1	NGB802623
4	Plate with hexagon insertion for sealing tool 6.239.2-92.4	1	NGB803373
5	Countersunk screw M4x16	2	400 01 05 *
6	Foundation	1	400 01 06 *
7	Base	1	400 01 08 *
8	Setscrew No.6	2	400 01 11 *
9	Countersunk screw M6x16	2	400 01 10 *
10	Countersunk screw M4x10	2	400 01 09 *
11	O-ring Ø 47x4	1	400 01 12 *
12	O-ring seal made of Viton (FKM), Ø 8 x 1.5 mm	2	NGB800590
13	Bit	1	NGB802622

*= only on request

Literature

Kaisersberger E.; Möhler H.
NETZSCH Annual for Science and Industry, Volume 1
DSC on Polymeric Materials
Würzburg, Selb 1991

Kaisersberger E.; Knappe S.; Möhler H.
NETZSCH Annual for Science and Industry, Volume 2
TA for Polymers and Additives DSC-TG-DMA
Selb, Würzburg 1993

Kaisersberger E.; Knappe S.; Möhler H.; Rahner S.
NETZSCH Annual for Science and Industry, Volume 3
TA for Polymers and Additives DSC-TG-DMA-TMA
Selb, Würzburg 1994

Brown
Introduction to Thermal Analysis
Chapman and Hall, London, New York, 1988

DIN 51005
Thermische Analyse [TA] Begriffe
Beuth-Verlag GmbH, Berlin, 1983

Hill [ICTA]
For Better Thermal Analysis and Calorimetry
ICTA, 1991

Mackenzie
Differential Thermal Analysis
[Vol. 1, Fundamental Aspects]
Academic Press, 1970

Mackenzie
Differential Thermal Analysis
[Vol. 2, Applications]
Academic Press, 1972

Hemminger, H.K. Cammenga
Methoden der Thermischen Analyse
Springer-Verlag, Berlin/Heidelberg/New York/
London/Paris/Tokyo, 1989

Turi
Thermal Analysis in Polymer Characterization
Heyden & Son, Philadelphia, London, Rheine, 1996

Wendlandt
Thermal Analysis
Vol.19, 3rd edition
John Wiley & Sons, New York/London/Sydney/Toronto, 1986

Scientific journals

Thermochimia Acta
Elsevier Science Publishers B.V.

Journal of Thermal Analysis
John Wiley & Sons, Chichester
Academiai Kiado, Budapest

Standards for Thermal Analysis

DIN 51 005	Thermal Analysis (TA) terms
DIN 51 006	Thermal Analysis (TA); Thermogravimetry (TG) principles
DIN 13346	Temperature, temperature difference, basic concept and units
DIN 43760	Basic values for measuring resistors
ASTM D3418	Transition temperature of polymers by Thermal Analysis
ASTM E473	Standard definitions of terms relating to Thermal Analysis
ASTM E793	Heats of fusion and crystallization by DSC
ASTM E472	Standard practice for reporting Thermoanalytical Data

ASTM = **A**merican **S**ociety for **T**esting and **M**aterials

DIN = **D**eutsches **I**nstitut für **N**ormung e.V.

NETZSCH-Gerätebau GmbH

Wittelsbacherstraße 42
95100 Selb · Germany
Tel.: +49 9287 881-0
Fax: +49 9287 881 505
at@netzsch.com

Customer Service
Tel.: +49 9287 881-555
ngb-service@netzsch.com

www.netzsch.com

206030005 80032061 2020-12-03