Operating Instructions

Thermo Microbalance

TG 209 *F1* Libra®

CONFORMITY DECLARATION

according to ISO/IEC Guide 22 and EN 45014

NETZSCH

Manufacturer's name:	**NETZSCH-Gerätebau GmbH**
Manufacturer's address:	**Wittelsbacherstr. 42** **D-95100 Selb**

declares that the product:

product name:	**Thermo-Microbalance Apparatus**
model number:	**TG 209 *F1* Libra®**

conforms to the following product specifications:

EN 61326:1997+A1:1998+A2:2001
EN 61000-3-2:2000
EN 61000-3-3:1995+Corrigendum 1997+A1:2001
EN 61326:1997 table 3
EN 61000-3-2:2000
EN 61326:1997 table A.1
EN 61000-4-2:1995+A1:1998+A2 :2001
EN 61000-4-3:2002+A1:2002
EN 61000-4-4:1995+A1:2001+A2:2001
EN 61000-4-5:1995+A1:2001
EN 61000-4-6:1996+A1:2001
EN 61000-4-8:1993+A1:2001
EN 61000-4-11:1994+A1:2001
EN 61010-1:2002+Corrigendum 1:2002+2:2004

We hereby certify that the product is in accordance with the EU Electromagnetic Compatibility Directive 2004/108/EC and the EU Low Voltage Directive 2006/95/EC.

Selb, January 2013

N E T Z S C H
Gerätebau GmbH

(Dr. T. Denner)
Managing Director

Operating Instructions TG 209 *F1* Libra®

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Chapter I

General Informationen

Information

In the design of your instrument, we endeavor to take individual solutions into account and to include these in the documentation.

However, in order to keep the scope of the technical documentation at a reasonable level, we must limit the description to a standard model.

We ask for your understanding, if additional information particular to your instrument is not included within the scope of the standard instructions.
This additional information can always be found on the corresponding information sheets.

All technical data, instrument features and other information described in these operating instructions are presented to the best of our knowledge and in accordance with the technical standards of the instrument at the time of printing.

We welcome any comments, suggestions or new ideas concerning the instrument and these operating instructions. Please address them to:

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Telefax: 09287/881- 144
E-Mail: at@netzsch.com
Internet: http://www.netzsch-thermal-analysis.com

Maintenance and service must be carried out by NETZSCH Customer Service personnel.

A service contract is available for our customers.

This instruction manual is provided to give the customer information on proper operation of the instrument. NETZSCH-Gerätebau GmbH will accept no liability for damage resulting from improper use.

Notes on the Use of this Manual

In this manual, the symbols described below are used to simplify orientation.

NOTE

This sets particularly important information apart from the rest of the text.

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 after this symbol are required for the installation or modification of your instrument.

Basic Safety Information

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.
Training of Personnel	Your instrument may only be operated and maintained by authorized, trained and individually instructed personnel who have been informed of possible sources of danger.
Responsibility	Responsibility for commissioning, operation and maintenance must be clearly defined and compliance must be ensured. The resulting responsibilities can be clearly resolved under the aspect of safety.
Unauthorized Access	The operator must make sure that only trained personnel work on the instrument.
Improper Operation	Any type of operation which reduces the safety of the user and the operability of the instrument should be avoided.
Unauthorized Changes to the System	Unauthorized modifications and changes which effect the safety of the instrument are not permitted.
Obligation to Report Changes to the System	The user is obligated to immediately report to the operator in charge any changes to the instrument which effect the safety of the system.

Maintenance Obligation	The operator of the system must ensure that the instrument is operated in perfect condition at all times.
Proper Set-up of the Work Stations	The operator must guarantee the openness and cleanliness of the work stations at the instrument through 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, and only when it is turned off and disconnected from the power supply! Any work on the electrical supply, electrical lines and electrical components may only be carried out by qualified personnel (electrical professionals).
Removal of Protective Devices	Protective devices may only be removed when the instrument is switched off and unplugged. It is imperative that the protective devices be replaced before starting the instrument.
Checks following Maintenance or Repair Work	After maintenance or repair work, a check should be made to ensure that all protective devices are in place and operate properly. Only then should the instrument be started.
Industry-specific Accident Prevention Regulations	The operator must observe the relevant regulations and protective measures when handling the required gases. In addition, possible reactions with the materials used must be considered. At operating temperatures above 55°C, protective gloves must be worn. 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.
Maintenance or Repair	Products sent in for 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.

Lighting at Work	The lighting level on the working environment should be determined in such a way that a safe work and an identification of risks in time are possible at all times. The minimum provided lighting level in the laboratory is 300 lx and on the working environment 500 lx.
Operating Instructions	Using the Operation Manual, the operator should prepare operating instructions which specify the actions and tasks required for safe operation. The operating instructions should be placed in a suitable location in the work place and complied with by the employees.
Gases	Observe the relevant regulations and protective measures when handling the required gases. Consider possible reactions with the materials. Warning signs should be hung in the appropriate places in accordance with national and regional regulations.

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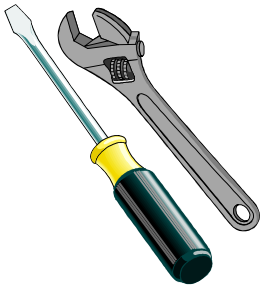
Chapter II

Installation

Contents

Installation



In most cases, your TG 209 *F1* is set up and commissioned by one of our customer service engineers.

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

Packing and Delivery

We deliver all components in separate cartons. The measuring unit, electronics, computer system etc. are packed in form-molded foam to protect against damage during transport.

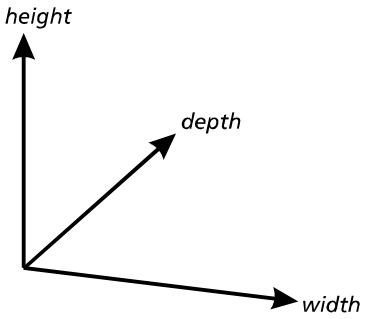
- ✓ We recommend keeping the original cartons. Should repairs or an extension of the system become necessary, the cartons can be reused, thus ensuring a relatively safe return to the manufacturer.
- ✓ Prior to shipping, we carefully test all components of the system for mechanical and electrical operability.
- ✓ After unpacking, please check all delivered components for possible transport damage and compare the individual items against the delivery note supplied.
- ✓ Should an item be missing, please contact us immediately.

Location Requirements

✓ **Select** the optimal component arrangement for the space you have available.

The location of your instrument must meet the following requirements:

✓ constant temperature conditions (room temperature) to the extent possible
✓ no direct sunlight on the instruments
✓ dust-free environment to the extent possible



measuring system A stable, shock-absorbent table with a working surface of approx. 2.5 m x 1 m is required for the measuring device (measuring cell, computer, printer).

thermostat The thermostat cooling system requires 0.5 m x 0.5 m space. The cooling system should be placed beside the table.

An installation schematic of the measuring system follows on the next page.

Installation schematic

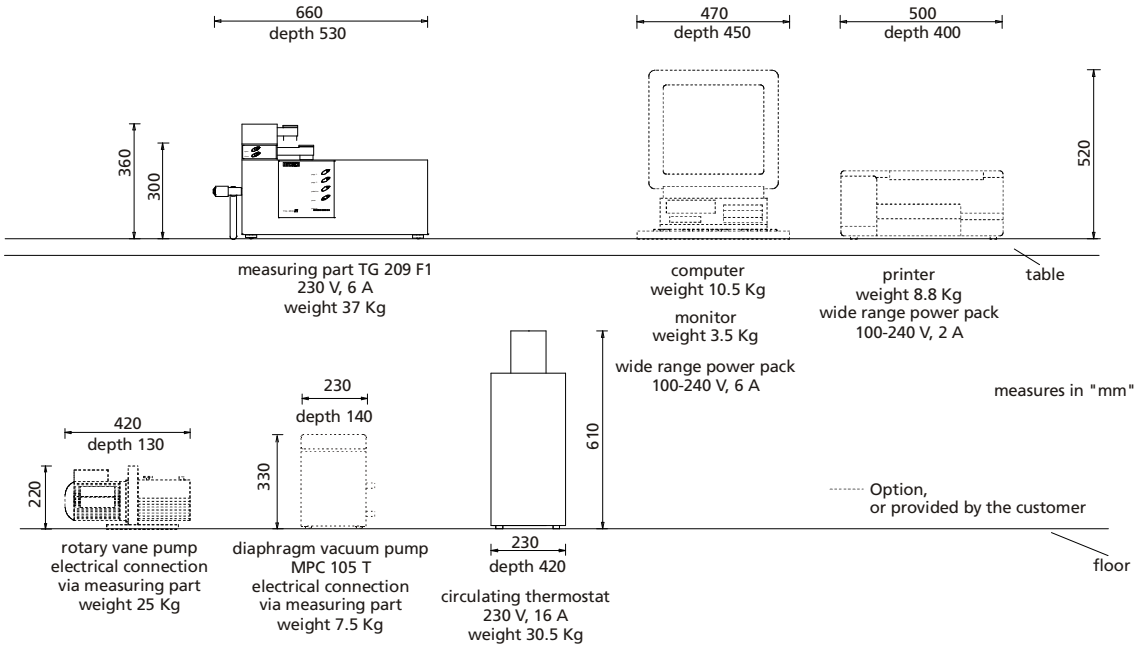


Figure 1: Installation schematic TG 209 *F1*

Alignment of the measuring unit

- Place the measuring unit on a table.
- Align the balance by turning the knurled nuts (A) on the front and rear of the adjustable feet up or down.
- Use the spirit level (B) to align the measuring unit.

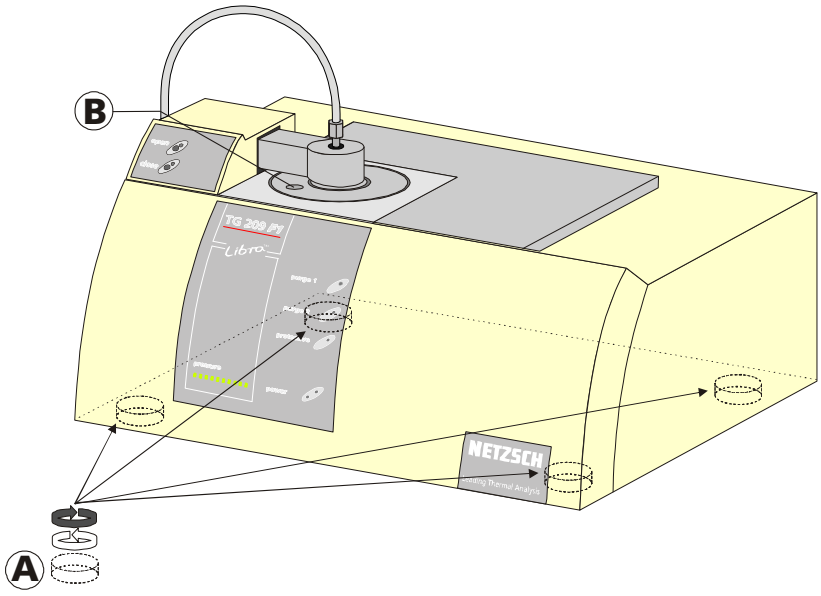


Figure 2: Alignment measuring unit

Connections between the instruments

Electrical connections

- Follow the figure below to connect measuring part and computer system.
- Connect the measuring part and the computer system via USB cable.
- Connect the measuring part, computer system and thermostat to the power supply.

Connect computer system, printer and measuring part to one phase! The thermostat must be connected to a separate electrical phase! The vacuum pump is connected via the measuring part!

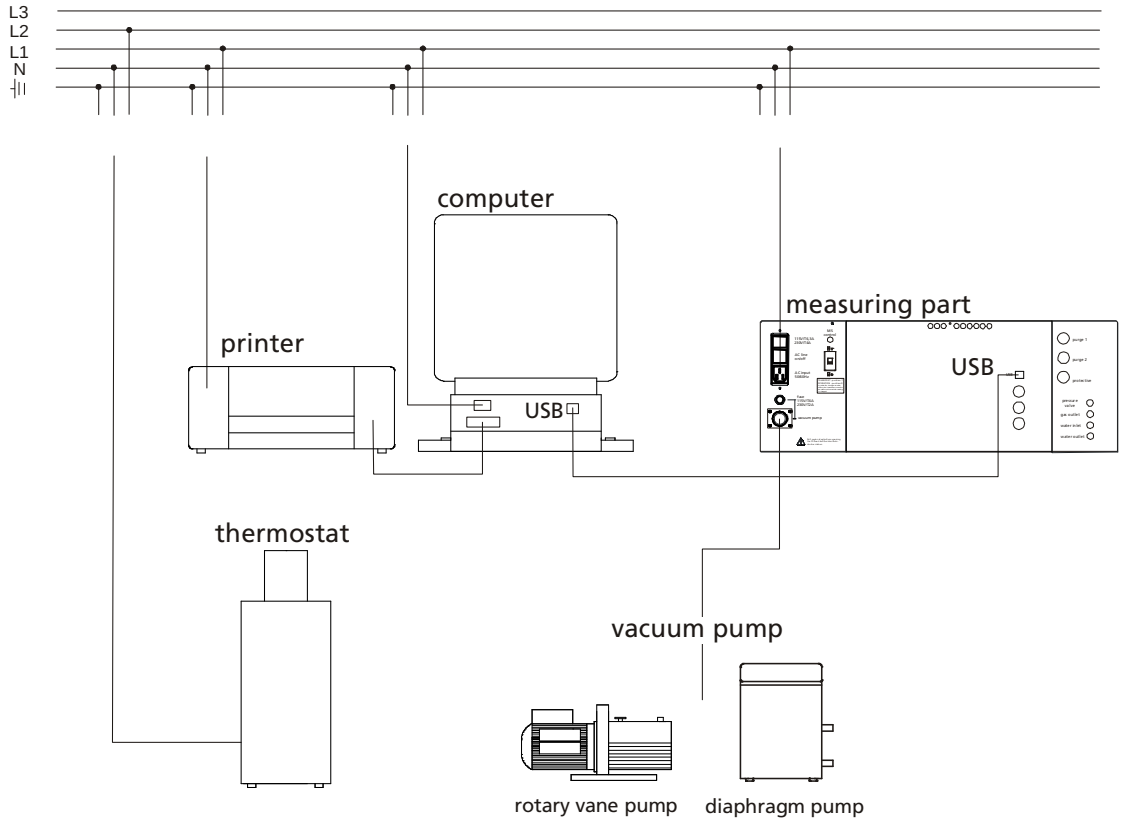


Figure 3: Electrical connections

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

Important notes for Software installation

Your TG 209 *F1* 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 TG 209 *F1* 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".

Notes:

- Do not use USB hubs (connect your TG 209 *F1* 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.

Connecting the thermostat

The measurement part must be thermostatically controlled to guarantee a constant temperature.

Attach the cooling water hoses from the thermostat to the appropriate connections on the back of the measuring unit.

ATTENTION!
Carefully read the manual of the thermostat and pay attention to all described safety regulations!

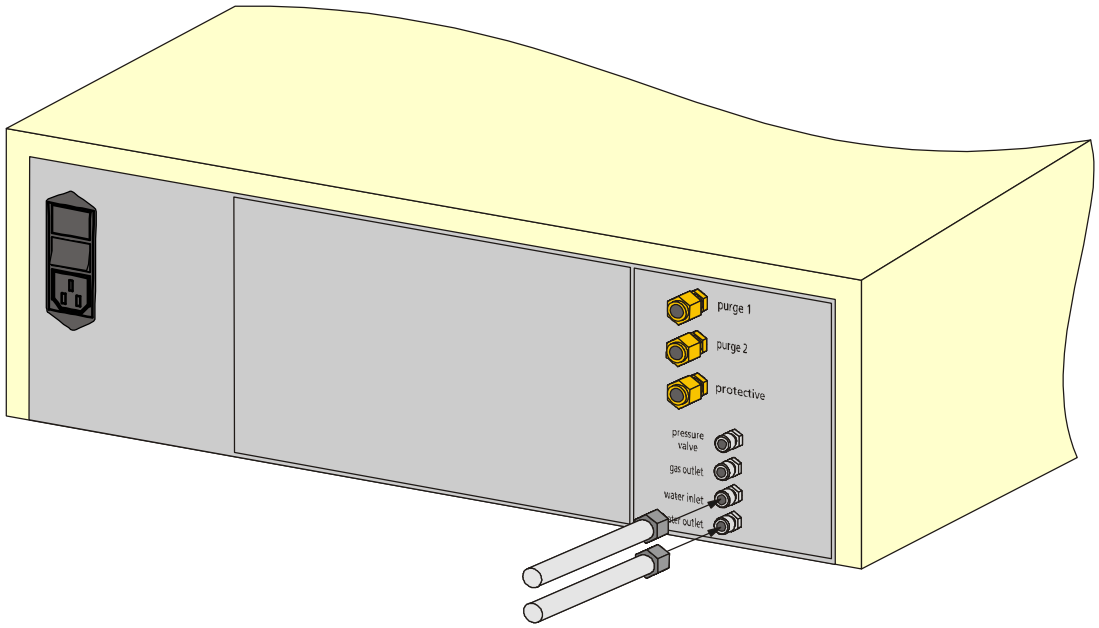


Figure 4: Connecting thermostat

Adjust the thermostat temperature 2-3°C higher than room temperature. See manual thermostat.

Max. input pressure 5 bar!

Connecting the evacuating system

An evacuating system can be connected to the measuring unit.

- Connect the magnetic valve on the measuring unit.
- Connect the vacuum pump to the magnetic valve.

The vacuum pump is switched on/off via the software (For it the main switch of the vacuum pump must be activated). The magnetic valve opened automatically to prevent a too fast evacuation of the sample chamber.

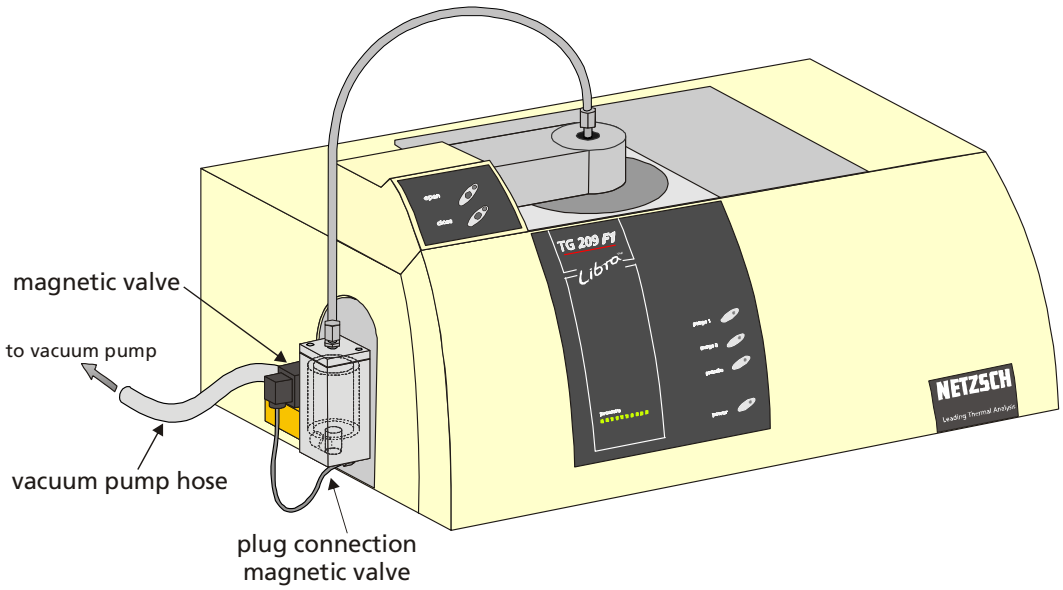


Figure 5: Connecting the evacuating system

Electrical connection

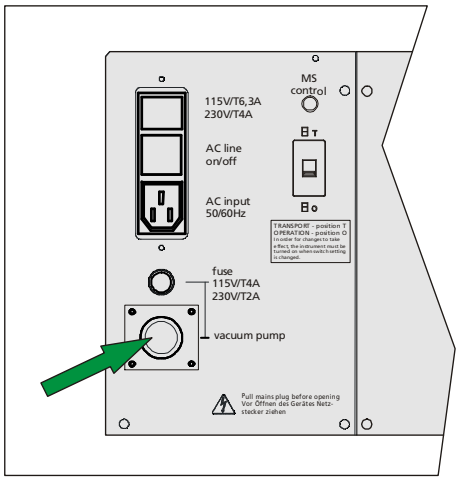


Figure 6: Electrical connection of the evacuating system

Connect the power supply of the evacuating system to the connection **vacuum pump** at the rear of the measuring unit.

Connecting the gas supply

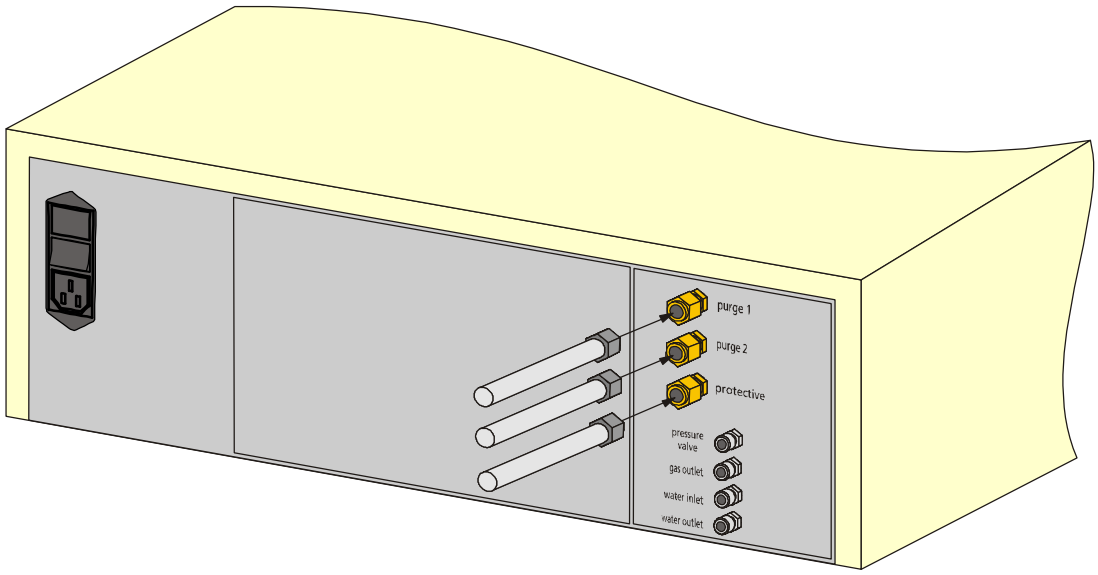


Figure 7: Connecting gas supply

Recommended purge gas rates:

purge1, 2	approx. 20 ml/min
protective	approx. 5-10 ml/min

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 IV)!

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.

Connecting the gas outlet tube

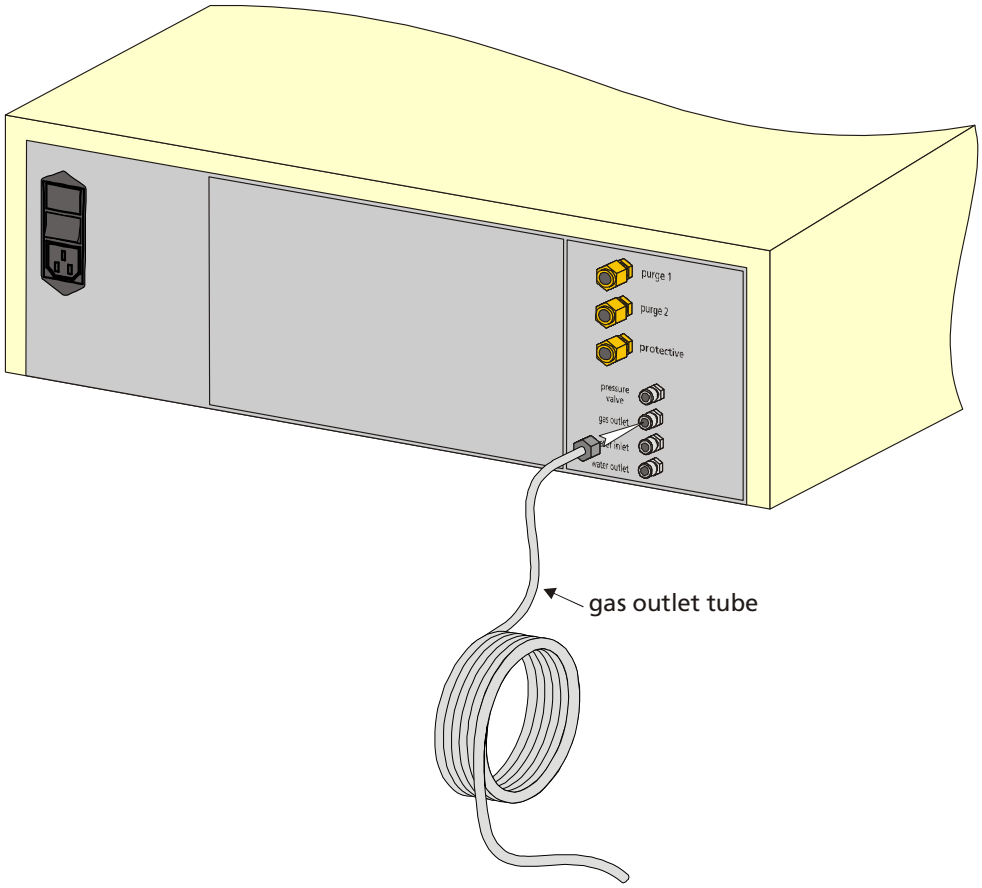


Figure 8: Connecting gas outlet tube

Function

The TG 209 *F1* can be equipped with a separate gas outlet tube (∅ 3 mm, length about 5 m; included in delivery) on the gas outlet of the instrument. The gas outlet tube is used to minimize fluctuations in external room pressure which can affect the measurement.

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Chapter III

System Components

Contents

Measuring unit

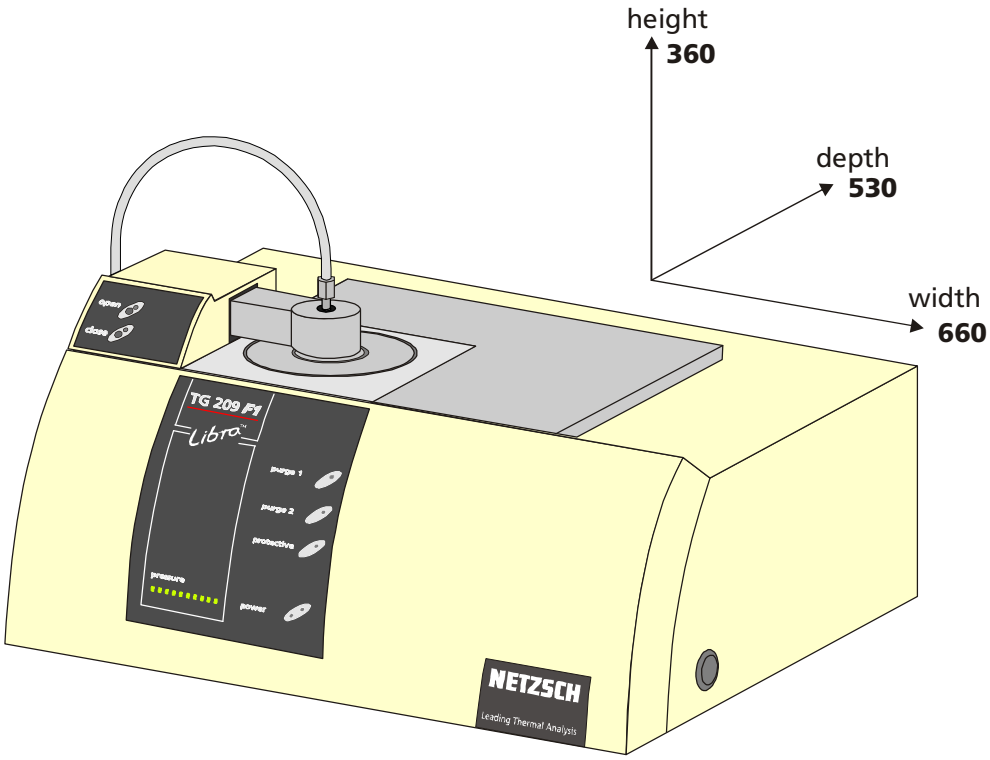


Figure 1: TG 209 *F1* measuring unit

Principle of operation

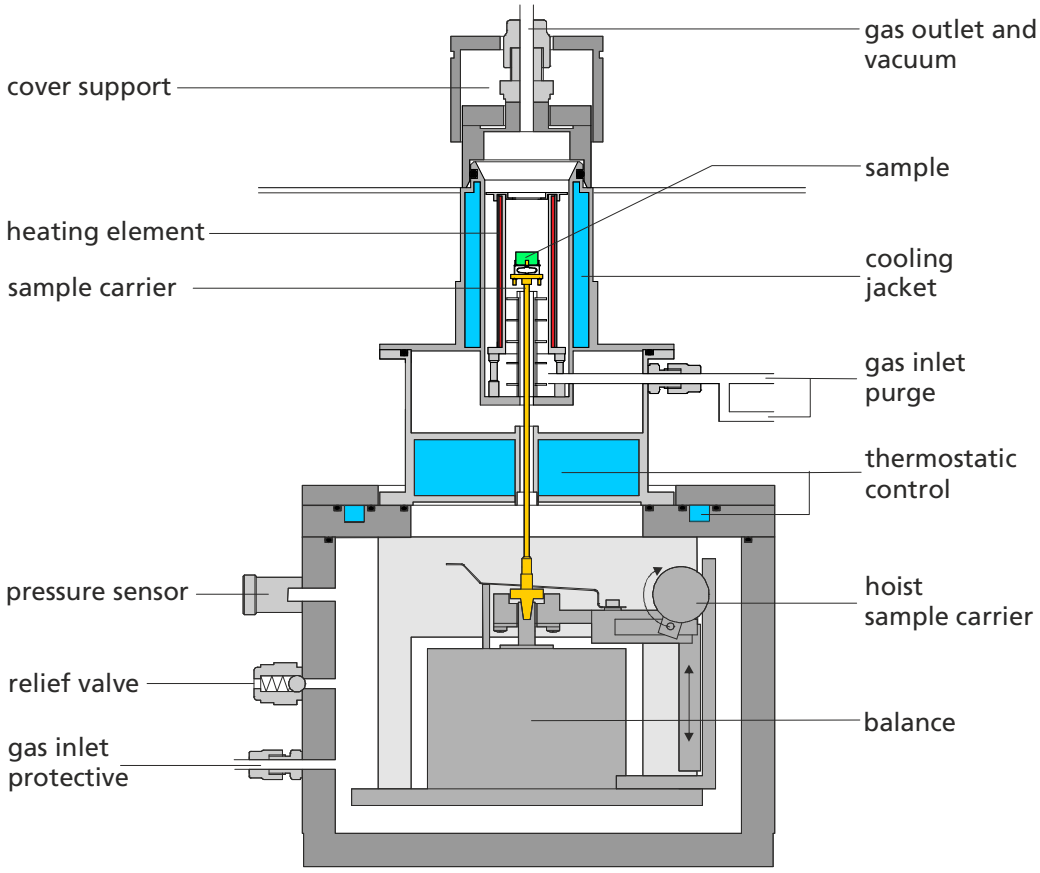


Figure 2: TG 209 *F1* measuring cell (schematic)

Microbalance System

The **microbalance** works according to the principle of electromagnetic power compensation and is enclosed by a **vacuum-tight housing**. The balance housing is thermostatically controlled to avoid temperature influences. The **sample holder** is located above the microbalance and is connected to the balance by a **sample holder support**.

Furnace

The sample is **heated** by a **microfurnace** which is enclosed by a **cooling jacket**. Water is used as the cooling agent. Thus, cooling times of approx. 15 minutes are possible for uncontrolled cooling from 1100°C to room temperature. The heating rate can be varied between 0.001 K/min and 200 K/min. The **control thermocouple** is exchangeable integrated in the ceramic furnace.

The **radiation shield** and **thermostatic control** prevent the influence of heat radiated from the furnace on the balance system, which could lead to incorrect data.

The thermostatic control and furnace cooling are combined in a common cooling cycle. The cooling agent (water) flows first through the thermostatic control and then to the cooling jacket to cool the furnace.

Front panel of the measuring unit

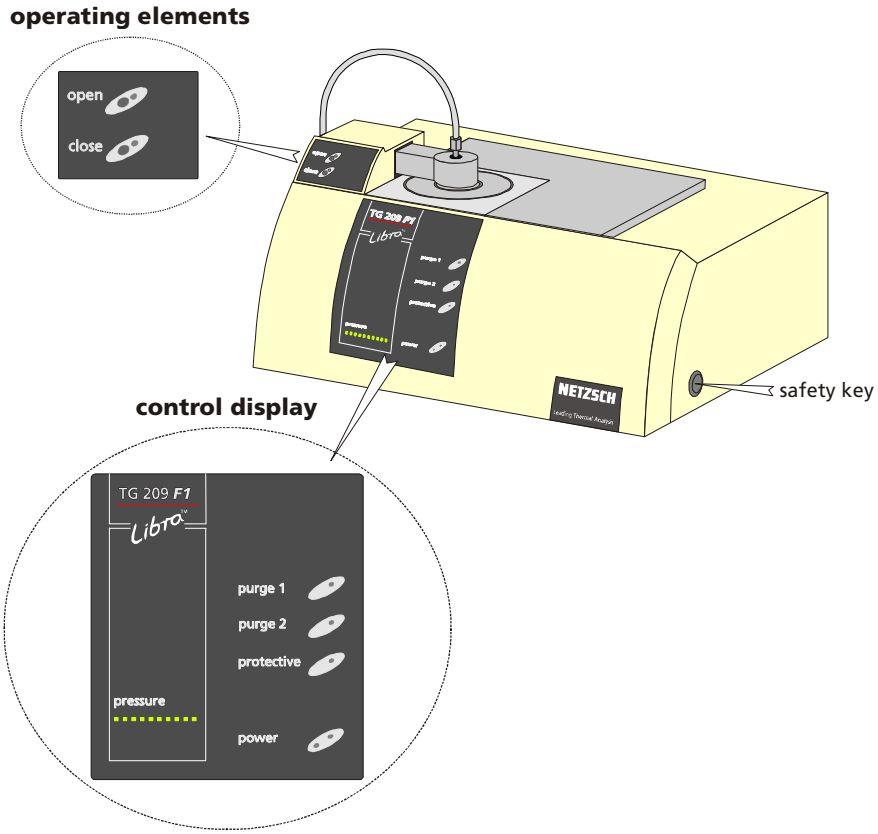


Figure 3: front panel

operating elements

label	function
"open"+"safety key"	opens the cover of the measuring unit (green LED on: upper end position reached)
"close"+"safety key"	closes the cover of the measuring unit (green LED on: lower end position reached)

control display

label	function
purge 1	control LED: purge gas 1 (sample chamber) "on/off"
purge 2	control LED: purge gas 2 (sample chamber) "on/off"
protective	control LED: protective gas "on/off"
power	green LED on: instrument is switched on orange LED on: measurement is running
pressure	normal pressure: only the right green LED is shining decreasing pressure: orange lights from right to the left go out
	The gases "protective", "purge 1" and "purge 2" can be activated via the software (see Help system Software).

Rear of the measuring unit

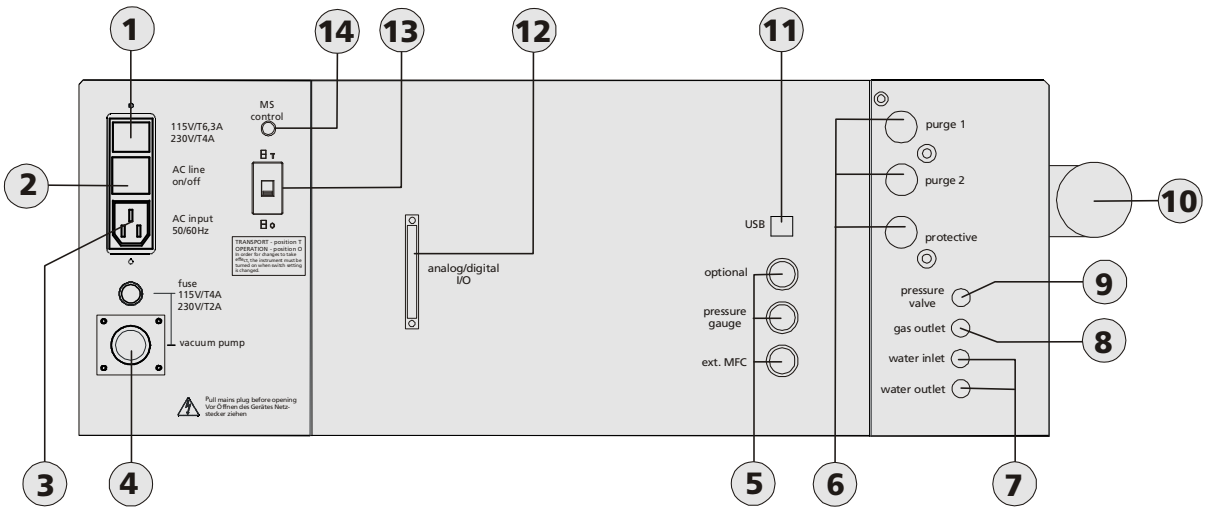


Figure 4: rear of the measuring unit

No.	label	function
1	115V/T4A; 230V/T2A	mains fuses
2	AC line on/off	power switch instrument "on/off"
3	AC input 50/60Hz	power connection 230 (115) V
4	vacuum pump	plug connection for vacuum pump
5	optional pressure gauge ext. MFC	optional connection for peripheral equipment optional connection for pressure gauge optional connection for external NETZSCH MFCs
6	purge 1 purge 2 protective	plug-in connection purge gas 1 plug-in connection purge gas 2 plug-in connection protective gas
7	thermostat	connection thermostat inlet/outlet
8	gas outlet	gas outlet sample chamber
9	pressure valve	outlet pressure relief valve sample chamber
10	vacuum flange	hose connection vacuum pump
11	USB	connection computer system
12	analog digital I/O	analog digital input/output
13	transportation lock	O = Operation T = Transport
14	MS control	trigger signal for QMS-Aëolos (only for QMS 403 C)

Gas outlet

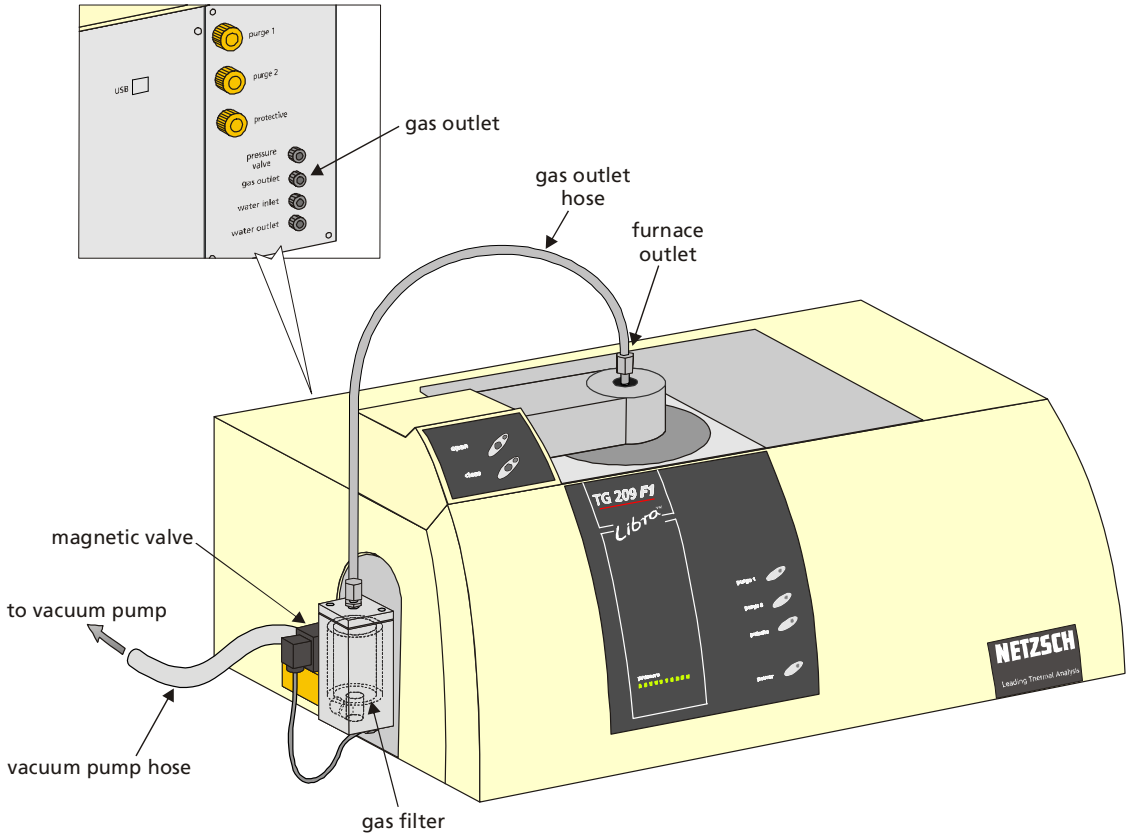


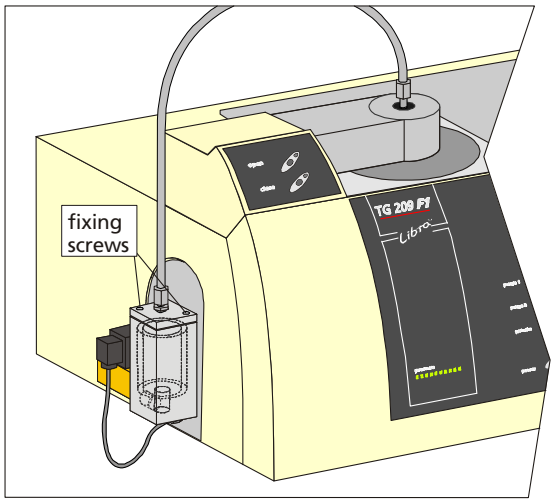
Figure 5: gas outlet

Through a hose connected to the gas filter and magnetic valve the gas is released from the furnace outlet to the gas outlet on the left. The magnetic valve can be switched on/off via the software (see HELP-system in the software). The hose at the gas outlet serves to reduce the cross-section at the gas outlet. External pressure fluctuations which affect the TG signal are thus isolated from the balance chamber to a great extent.

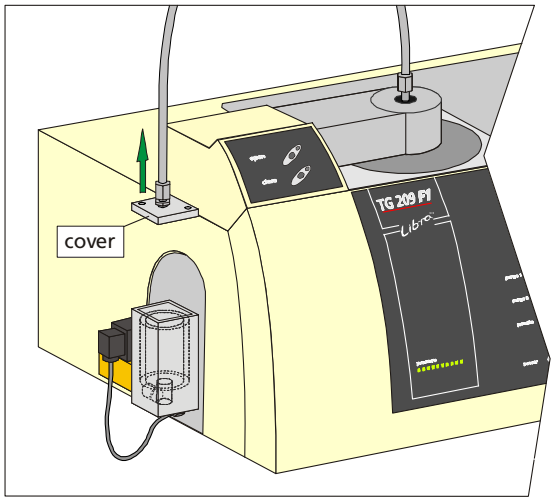
Attention!

The gas filter and the hose should be changed periodically as required.

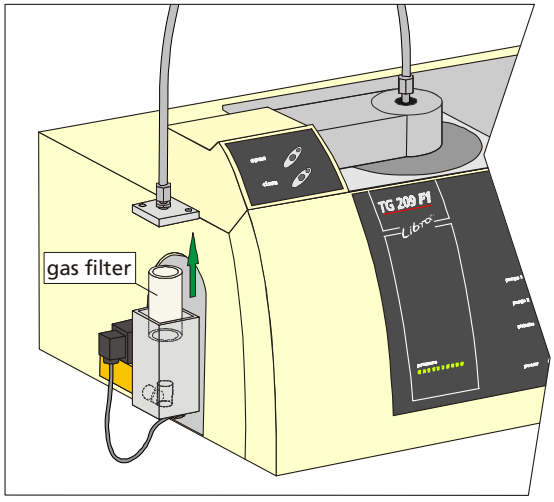
Changing the gas filter



- Unscrew the fixing screws in the cover of the filter block.

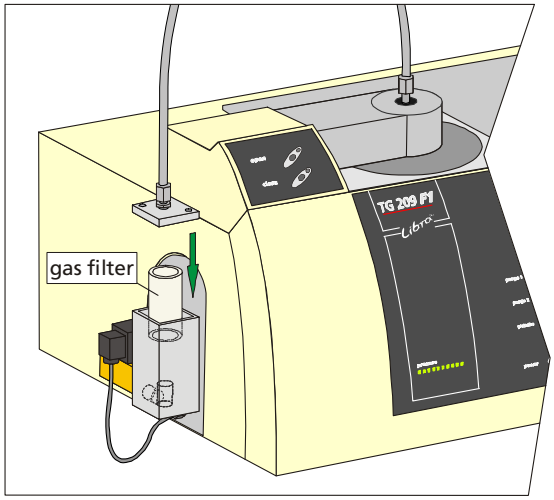


- Lift off the cover from the filter block.

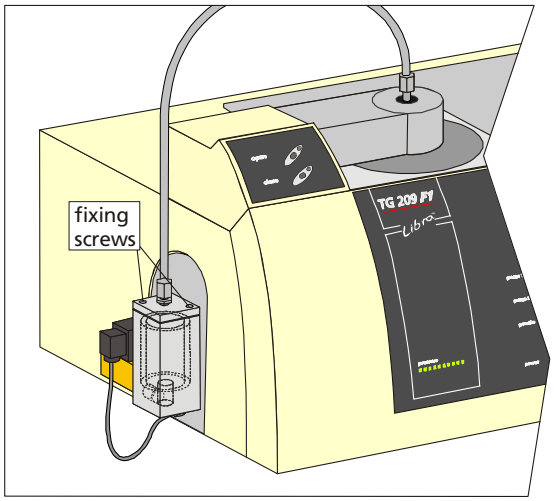


- Remove the gas filter.

New gas filter



- Insert the new gas filter.



- Affix the cover with fixing screws.

Evacuation system

The balance and sample chamber can be evacuated up to a vacuum of approx. 10^{-2} mbar via the software.

Rapid ventilation of the measuring unit is possible via the gas inlet connection plug "Protective" on the rear of the measuring unit.

The balance chamber is also provided with a safety valve (pressure relief valve), which opens at an excess pressure of 0.3 bar.

Pressure gauge (front of the measuring unit)

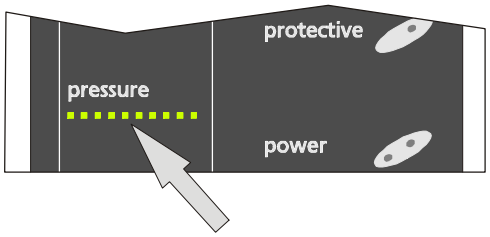
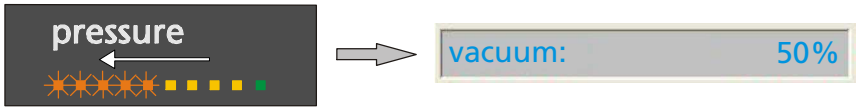
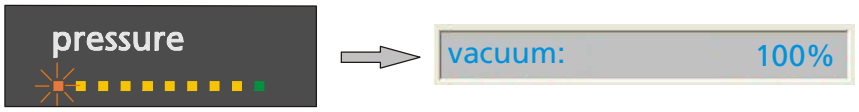
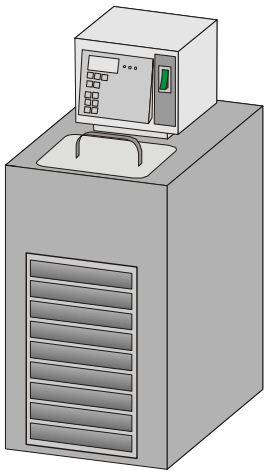


Figure 6: pressure gauge

normal pressure:	 only the right green LED is on → 0% vacuum
decreasing pressure:	 orange LEDs from right to the left go out → % value rises
minimum pressure:	 only the left orange LED is on → 100% vacuum

Thermostat



A thermostat is required to operate the instrument. This handles thermostatic control of instrument components relevant to the measurement and cooling of the furnace system. The thermostat is connected to the **thermostat (inlet-outlet)** connections on the back panel of the measuring unit.

Technical data for the thermostat can be found in the accompanying operating instructions.

ATTENTION!

Carefully read the manual of the thermostat and pay attention to all described safety regulations!

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Chapter IV

Operating the Instrument

Contents

Operation of the instrument

Measurement preparation

The test parameters should be carefully selected prior to the measurement according to the nature of the sample material and the expected effects.
Specific effects that the individual test parameters have on the TG curves are discussed in detail in the literature [1, 2].
Special attention should be given the following parameters during preparation for a measurement:

- sample preparation
- sample weight
- temperature program
- purge gas flow rate
- crucible material

Calibration

When calibrating the TG 209 *F1*, a distinction must be made between the temperature calibration and calibration of the balance.

Temperature calibration

Measurement of the sample temperature occurs outside the sample, resulting in systematic deviations from the true sample temperature. The deviations between the measured and "true" sample temperature can be determined with the aid of calibration materials. Chemical reactions are less suited for calibration, since the reaction temperatures are also dependent on atmosphere, pressure and heating rate.

Several materials (e.g. Ni, Fe) undergo reproducible changes in the magnetic properties at certain temperatures and can thus be used for temperature calibration. It must be pointed out here that the transition temperatures of ICTA and NBS materials are experimental mean values. Therefore, even a temperature calibration of a thermobalance, carried out using magnetic reference materials, can be subject to a certain degree of uncertainty.

Temperature calibration with calibration materials

you require:

- TG 209 *F1* with c-DTA® equipment
- c-DTA® Software
- Temperature Calibration Set 6.221.5-91.1 (includes 6 substances for calibrating)

procedure: See HELP-System in the software!

Calibration of the balance

A complete recalibration of the balance should be carried out at least once per year and additionally after transport, repairs and considerable changes in the test parameters (e.g. changing the sample holder). The weighing system has an inbuilt calibration weight for automatic calibration.

Proceed as follows:

- Remove the sample crucible. The sample holder remains in the instrument (from instrument No. 220 10 074).
- Open the NETZSCH Measurement Software.
- Select the menu item **Diagnosis**.
- Select **Balance Calibration**.
- Start Calibration.
- Wait until the calibration procedure has been finished.

See HELP-System in the software!

Sample preparation

The sample preparation is dependent on the consistency of the sample. Temperature variations occurring within the sample during the measurement can be minimized with appropriate sample preparation. The transmission of heat in larger samples can also be influenced by the sample preparation, e.g. reduction of the sample size.

Powder samples

The samples are evenly spread over the bottom of the sample crucible. If crucibles of various diameters are available, radial temperature gradients can be ruled out with the selection of a crucible diameter that is not too large.

Solid samples

Solid samples can be weighed into the crucible in one piece or they can be broken up. The sample should be broken up (increased surface area) if oxidation reactions are to be investigated.
During the investigation of oxidation reactions (change from neutral to oxidizing atmosphere), the atmosphere should only be changed in the sample chamber because its lower volume makes it possible to accomplish this quickly.

To the extent possible, the investigation of samples which form a great deal of carbon black should be avoided. If this is not possible, the tests should be run in an oxidizing atmosphere (synthetic air or oxygen) in order to prevent the formation of carbon black

During the testing of certain samples, intermediate or final products can condense on the colder parts of the furnace. Therefore, we recommend that the furnace be heated up at regular intervals and, when possible, that the measurements always be carried out under dynamic conditions (gas purge).

Fiber samples

Fibers can be cut into small pieces or placed in the crucible as fiber bundles.

Liquid samples

Depending on the viscosity, liquids are dripped into the sample crucible with a thin glass rod, a micropipette or microsyringe.

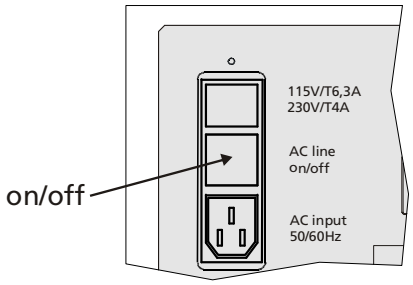
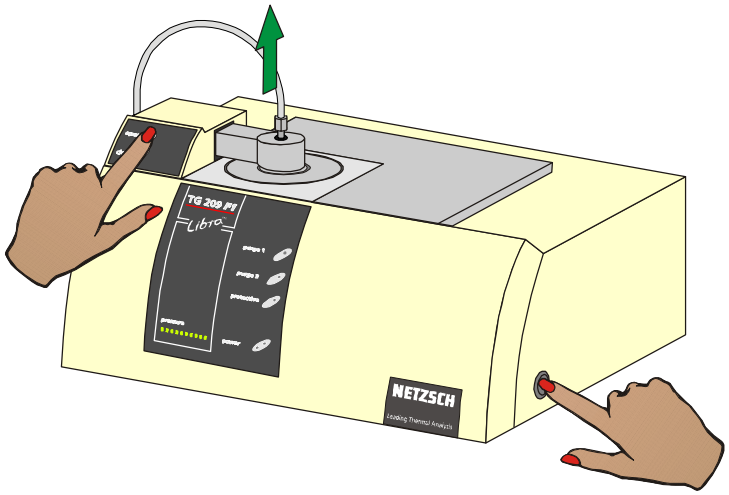
Instrument preparation

Weighing the sample

The sample is weighed with an analytical balance in an appropriate sample crucible (see table below).

Material	T_{max}/°C	Ø in mm	Capacity	Remark	Order No.
Al_2O_3	1000	6.8	85 µl	pan (lid: 399.973)	399.972
Quartz glass	1000	6.7	85 µl	pan (lid: 399.975)	399.974
Pt/Rh	1000	6.8	85 µl	pan (lid: 399.860)	399.205
Al (99.99)	550	6.7	85 µl	pan (lid: 399.971) / minimum purchase quantity: 100 pieces	399.970
ZrO_2	1000	6.7	85 µl	pan (lid: 397.052)	397.053
Graphite	1000	6.7	85 µl	pan (lid: 399.957)	399.956
Au	900	6.7	85 µl	pan (lid: 398.191)	398.190
Ag	750	6.7	85 µl	pan (lid: 398.193)	398.192

Insertion and adjustment of the sample holder

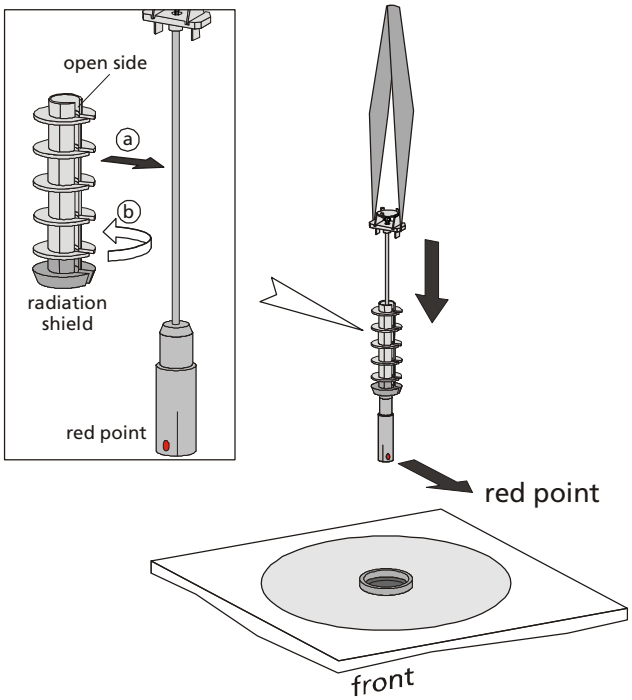
• **Switch on** the measuring unit.	
• Press **"open"+"safety key"** at the same time and move the cover into the upper position (green LED on the **"open key"** is shining). The sample carrier goes up automatically.	

Mounting of radiation shield:

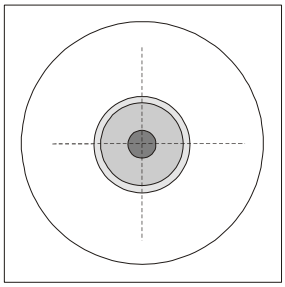
a) Attach the open side of the radiation shield to the sample holder.

b) The radiation shield can be prevented from falling off by twisting the radiation shield components.

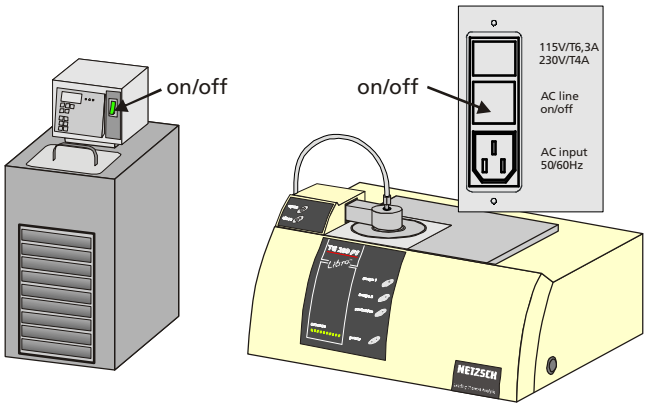
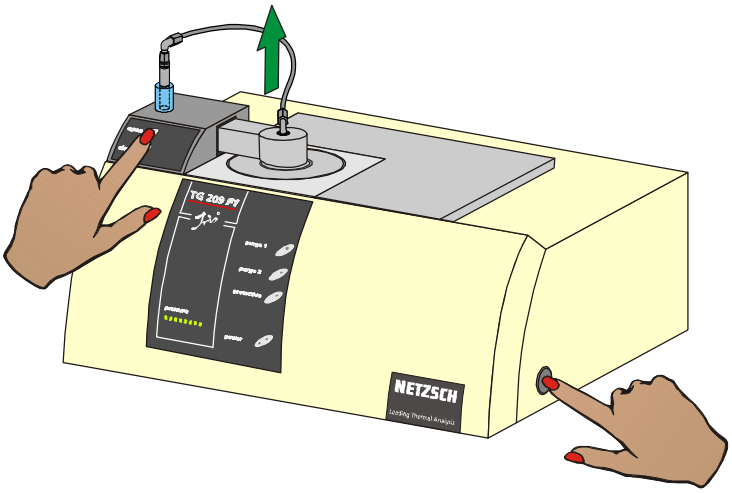
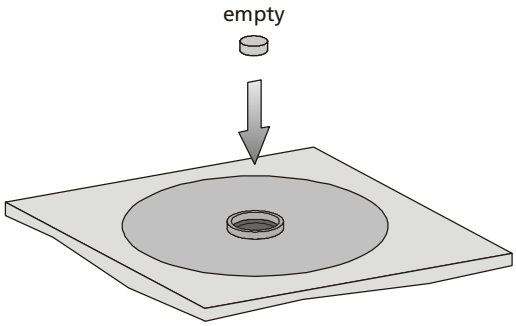
- Insert the **sample holder** from the top.
- The **red point** on the plug of the sample holder must face the front of the instrument (as shown in the figure).

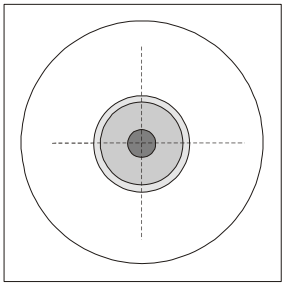
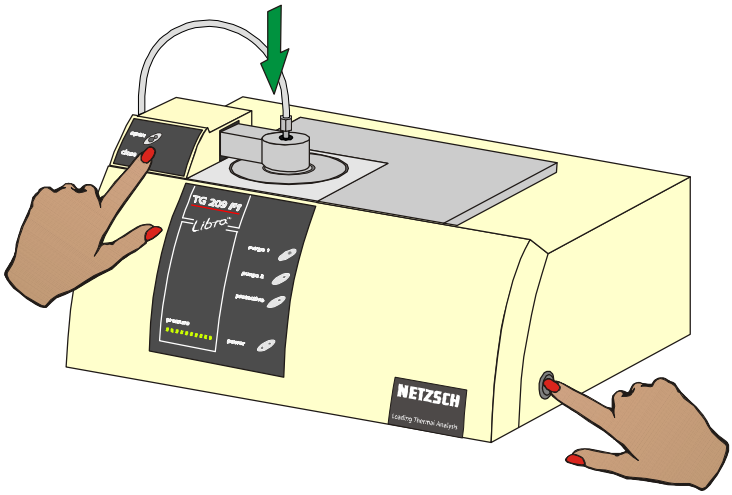
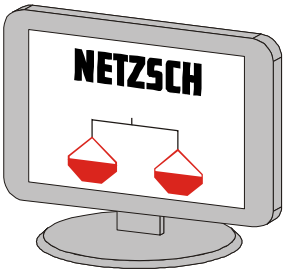
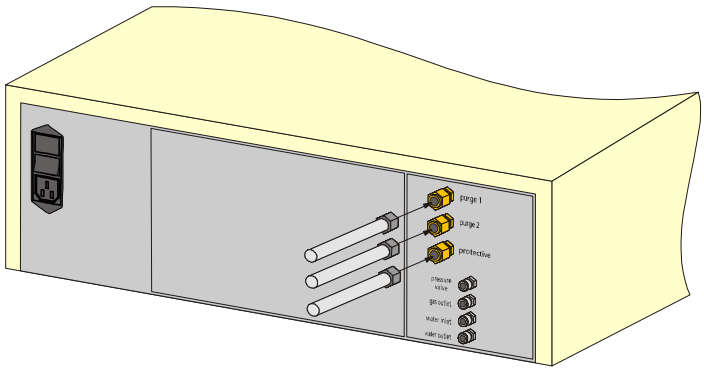


- **Check** the position of the sample holder from above.
- The sample holder should be centered.



Starting a measurement

1. **Switch on** the **thermostat** and the **TG 209 *F1*** measuring unit at least 60 min before starting the measurement. Allow sufficient time between switching on the thermostat and starting the measurement. Switch on the measuring part, computer, printer approximately 30 min before starting a measurement.	
2. Open the measuring unit. Press **"open"+"safety key"** at the same time and move the cover into the upper position (green LED on the **"open key"** is shining). The sample carrier goes up automatically.	
3. Place an empty **crucible***. *for baseline (correction measurement)	

4. Check that the **crucible** has been **correctly centered** by looking at it from above.	
5. Close the measuring unit. Press **"close"+"safety key"** at the same time and move the cover into the lower position (green LED on the **"close key"** is shining). The sample carrier goes down automatically.	
6. **Start** the TG 209 *F1* **measurement program**.	
7. **Set** the required amount of gases in the software.	

8. **Proceed** as shown **to create** a baseline: ➔

File Menu

⇩

New

⇩

Measurement Definition

- *Setup*
  - check the instrument setup

⇩

- *Header*
  - select measurement type: **Correction**
  - select temperature calibration file
  - define all sample settings (e.g. Identity, Name)
  - define gases (purge and protective)

⇩

- *Temperature Program*
  - define all steps of the temperature program

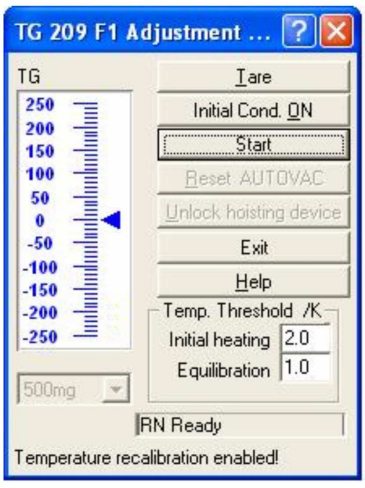
⇩

- *Last Items*
  - define Filename

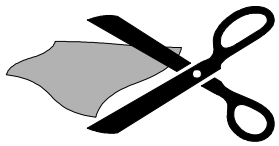
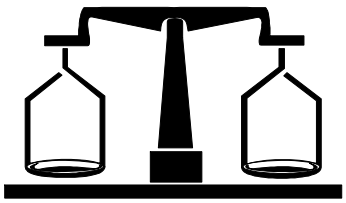
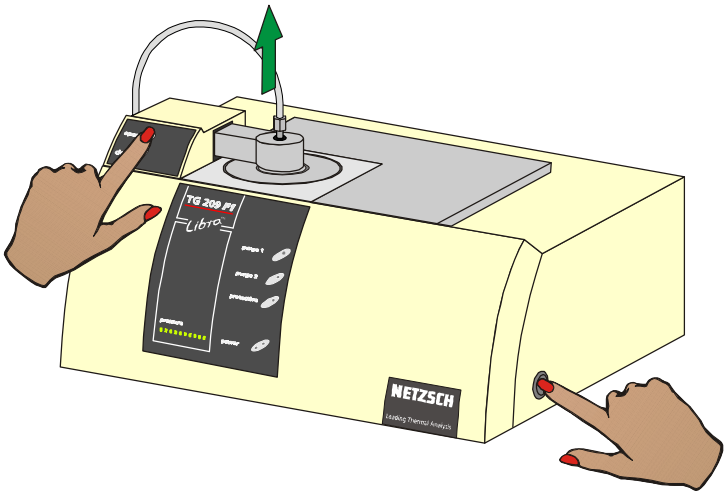
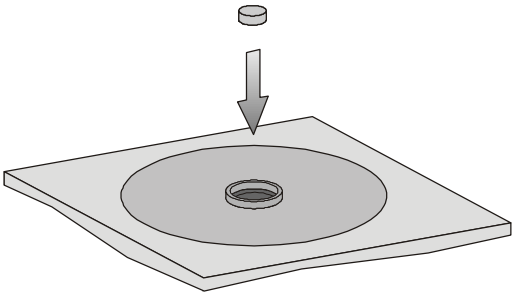
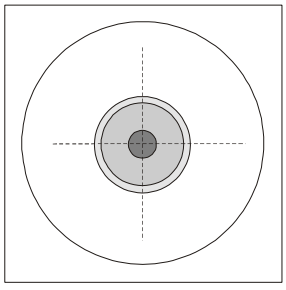
⇩

*click **Measure** -*
the program switches to the adjustment window

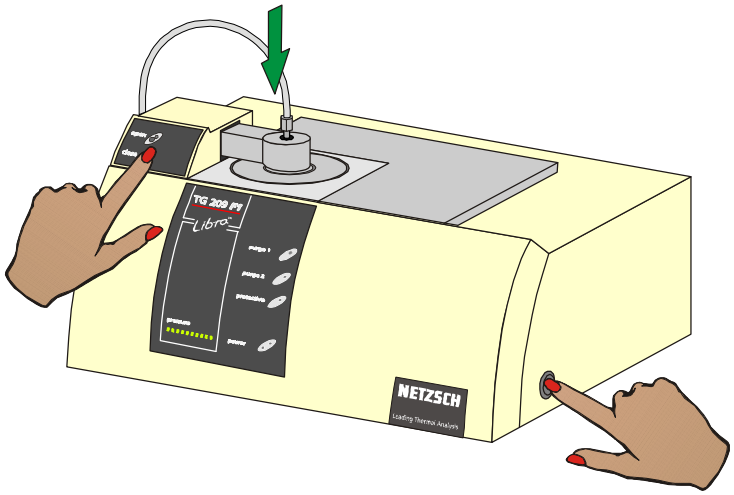
9. **Set** the **Initial Cond. ON** and wait until the TG signal is stable.



10. Then **start** the measurement.

11. **Prepare** the sample.	
12. **Weigh** the sample with an analytical balance or in the TG 209 *F1* instrument.	
13. Open the measuring unit. Press **"open"+"safety key"** at the same time and move the cover into the upper position (green LED on the **"open key"** is shining). The sample carrier goes up automatically.	
14. **Place** the **sample crucible**.	
15. Check that the **crucible** has been **correctly centered** by looking at it from above.	

16. Close the measuring unit. Press **"close"+"safety key"** at the same time and move the cover into the lower position (green LED on the **"close key"** is shining). The sample carrier goes down automatically.



17. **Proceed** as shown to combine the sample measurement with the correction measurement: ➔

File Menu

⇩

Open
use file from correction measurement!
sample mass = x mg!

⇩

Measurement Definition

• *Fast Definition*
- select measurement type: **Correction+Sample**
- enter the sample mass in mg!

⇩

- define sample settings
e.g. to accept the settings from the correction measurement (e.g. Identity, Name) set the checkmark for
Get these properties from just opened measurement

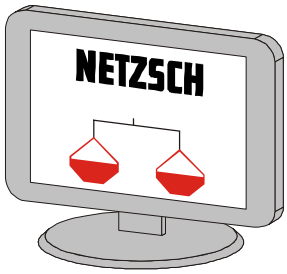
⇩

- define Filename

⇩

*click **Measure** -*
the program switches to the adjustment window

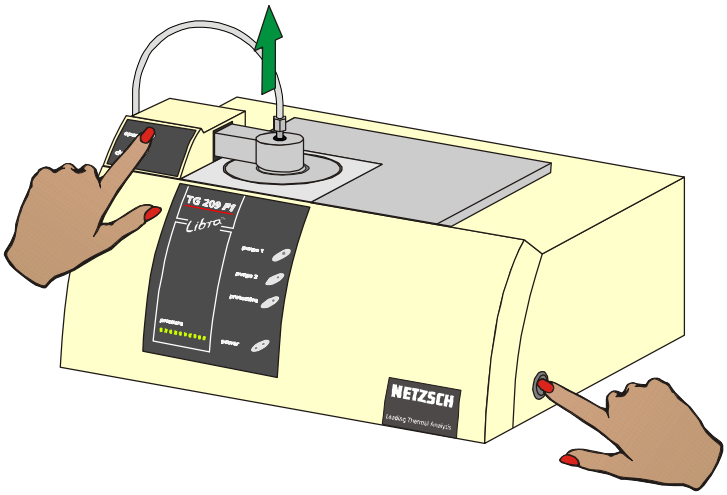
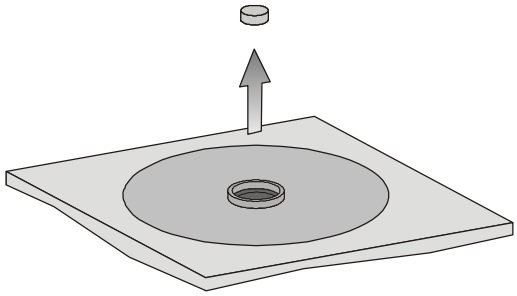
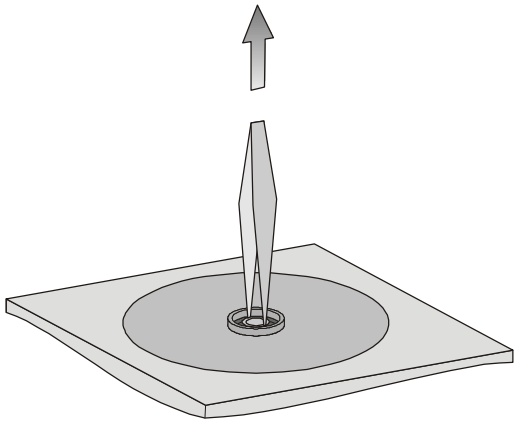
Start

18. **Open** the NETZSCH Proteus TA Analysis program. After the measurement is finished, you can begin with the evaluation.	

Changing the sample holder

The following procedure for changing the sample holder is valid only for the so-called "normal case", i.e. if your sample holder is badly worn or has been slightly damaged.

Should unexpected damage occur to the sample holder during a measurement and it cannot be changed according to the following procedure, please call our Customer Service Department immediately.

1. Press **"open"+"safety key"** at the same time and move the cover into the upper position (green LED on the **"open key"** is shining). The sample carrier goes up automatically.	
2. Remove the crucible.	
3. Take hold of the **sample holder** on the ceramic part with the tweezers and carefully lift it out.	

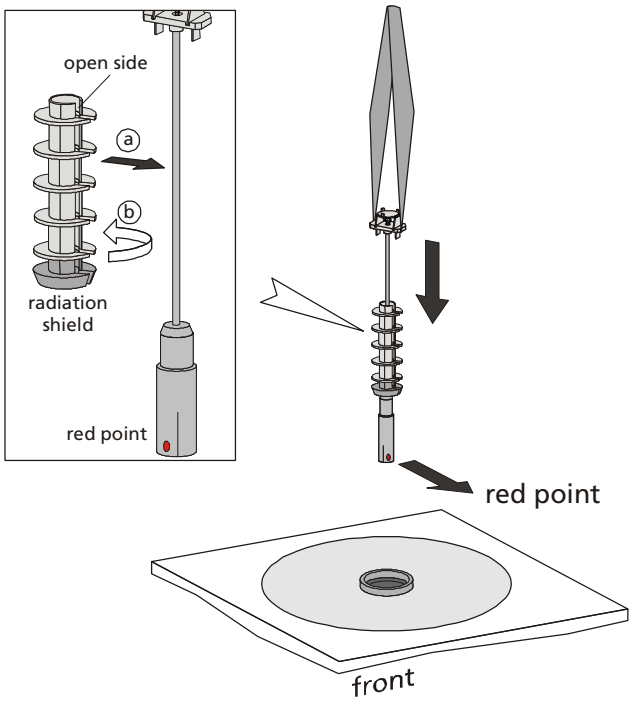
New sample holder

4. **Mounting of radiation shield:**

a) Attach the open side of the radiation shield to the sample holder.

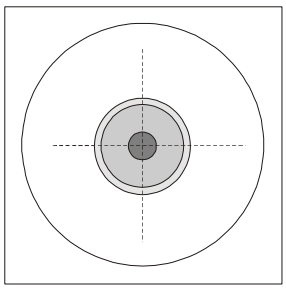
b) The radiation shield can be prevented from falling off by twisting the radiation shield components.

- Insert the **sample holder** from the top.
- The **red point** on the plug of the sample holder must face the front of the instrument (as shown in the figure).

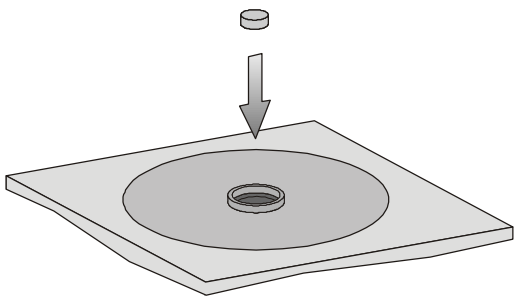


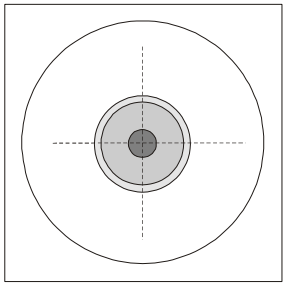
5. **Check** the position of the sample holder from above.

   The **sample holder** must be centered.



6. **Insert** the **crucible**.



7. With the sample holder raised, check that the **crucible** has been **correctly centered** by looking at it from above.	

Safety specifications for the use purge gases in the sample chamber

The balance chamber must always be purged with protective gas via the separate gas inlet (**protective)**.

For purging the sample chamber, certain reactive gases (non-toxic, non-flammable) can also be introduced via the inlets **purge 1** and **purge 2**.

Attention!

To avoid danger to the user and damage to the instrument, it is imperative that purge gases be used only in accordance with the following instructions:

The TG 209 *F1* Libra® has a vacuum-tight, gastight design. Thus, it is possible to conduct measurements in controlled sample atmospheres.

Dry, inert gases are recommended for purging the balance and sample chamber of the TG 209 *F1* Libra®. Prior to admission of the gas, a leakage test should be performed during the required pre-evacuation of the system. This serves to ensure the purity of the sample atmosphere. It is also recommended that the purge gas expelled be drawn into a suitable exhaust hood. Depending on the measurement conditions (sample material, atmosphere, temperature range), thermal reactions can give rise to derivative products even when inert gases are used. The smallest amounts of these derivative products (e.g. HCN, CO, SO_2, dioxins) are extremely hazardous to health, and must not be released into the work area.

A determination must be made by the user in advance whether a release of toxic gases is to be expected during the measurement. In such cases, appropriate safety precautions are absolutely essential!

If the measurement requires the use of special reaction gases in the sample chamber, a safety check of the potential hazard from the gas or gas mixture is imperative. Here, the following points are particularly important:

1. Are explosive gas mixtures of explosive compounds evolved from the sample when oxygen (air) is introduced?
2. Is it certain that no toxic compounds can evolve from the gases or gas mixtures at the selected application temperatures? In this regard, a check should also be made for possible reactions between the purge gas in the balance chamber and the reaction gas in the sample chamber!
3. Can corrosive effects from the gases on lines and seals in the system or peripheral equipment (valves, manometer, flow meter) be ruled out? If not, under certain conditions, leaks are to be expected!
4. What secondary effects do the gases or gas mixtures have on accessories in the system which may not necessarily cause leaks but could considerably increase wear and possibly lead to total failure of the system?

Attention!

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 regarding the possibility of using various gases can be found in the respective technical literature or can be requested from the gas manufacturers or retailers.

Below is a list of purge gases and the properties limiting their application. No claim is made as to the entirety of this list.

Measurements under helium may only be carried out up to a sample temperature of 800°C!

Helium (He)	Chemically inert (inert gas) No technical safety limitations
Argon (Ar)	Chemically inert (inert gas) Asphyxiating No technical safety limitations
Nitrogen (N_2)	Substantially inert Asphyxiating No technical safety limitations
Air	Oxidizing No technical safety limitations, but can react with sample Application range above room temperature. Maximum application temperature dependent on furnace material, sample holder type, crucible material.
Oxygen (O_2)	Flammable (Must not come into contact with fats and oils!) No technical safety limitations, but strong reactions with sample possible. Application range above room temperature. Maximum application temperature dependent on furnace material, sample holder type, crucible material.
Carbon dioxide (CO_2)	Non-toxic (MAK value 5000 vpm), Non-flammable Can be used above room temperature.
Hydrogen (H_2)	Flammable Explosion hazard with the introduction of oxygen! Use not allowed due to technical safety reasons!
Ammonia (NH_3)	Toxic (MAK value 50 vpm) Flammable Explosion hazard with the introduction of oxygen! Possible corrosive effect on seals!

Carbon monoxide (CO)	Toxic (MAK value 30 vpm) Flammable Explosion hazard with the introduction of oxygen! Use not allowed due to technical safety reasons!
Hydrogen sulfide (H_2S)	Toxic (MAK value 10 vpm), Flammable Corrosive Use not allowed due to technical safety reasons!
Other reducing gases or gas mixtures	Due to technical safety reasons the use of these is not possible. Explosive compounds can form if contact with oxygen or air occurs. Under certain circumstances, however, gas mixtures with inert gases are possible where there are no technical safety concerns and compliance with the analytical conditions is maintained. The manufacturers of gases and gas mixtures can provide the necessary information.
Chlorine (Cl_2)	Extremely toxic (MAK value 0.5 vpm) Non-flammable itself, but accelerates burning, corrosive, caustic Use not allowed due to technical safety reasons!
Hydrogen Chloride (HCl)	Toxic (MAK value 5 vpm) Non-flammable, corrosive, caustic Use not allowed due to technical safety reasons!
Sulfur dioxide (SO_2)	Toxic (MAK value 2 vpm), non-flammable, corrosive Use not allowed due to technical safety reasons!
Hydrogen sulfide (H_2S)	Toxic (MAK value 10 vpm) Flammable, corrosive Use not allowed due to technical safety reasons!
Fluorine (F_2)	Extremely toxic (MAK value 0.1 vpm) Flame accelerant, corrosive, caustic Use not allowed due to technical safety reasons!

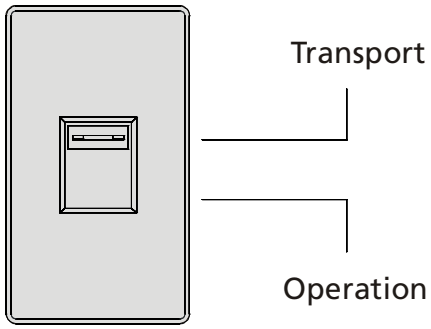
Hydrogen fluoride (HF)	Extremely toxic (MAK value 3 vpm) Extremely caustic, corrosive Use not allowed due to technical safety reasons!
Gaseous hydrocarbon	can form explosive gas mixtures with air With standard instrument installation, use not allowed due to technical safety reasons!

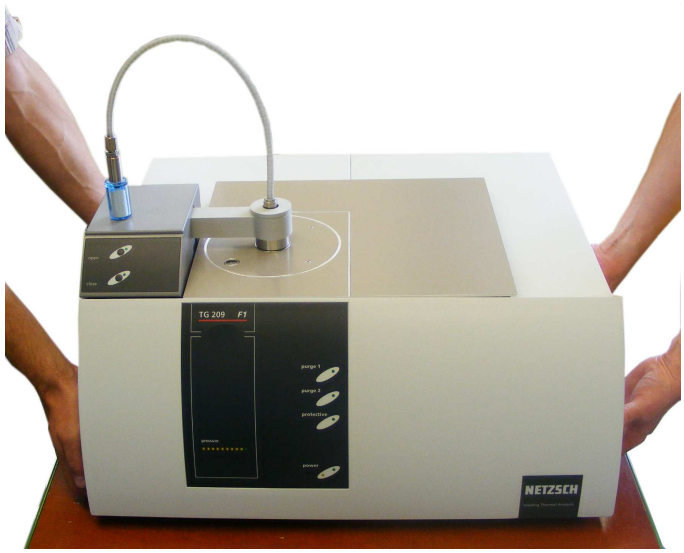
MAK value 2007

When these gases or gas mixtures are expelled from the apparatus, it is imperative that they be drawn into an exhaust hood. After completion of the measurement, the measuring unit must be sufficiently purged with inert gas.

Transport

- During transport the TG 209 *F1* is secured with a transport safety device – selected via the switch.
- The switch is placed at the left rear side of the instrument.
- For setting the switch use a small screw driver.

<table>
<tr>
<td> 
</td>
<td>• For the transport remove the sample holder and set the switch to position T (Transport).

ATTENTION!
If the sample holder is not removed, the lowering furnace could damage the arrested sample holder!</td>
</tr>
<tr>
<td> 
</td>
<td>ATTENTION!

Two persons are required in order to lift up and transport the instrument. Touch the instrument on the recessed grips at the bottom side as shown in the figure to lift it upwards (see picture).</td>
</tr>
</table>

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!

Libra®

TG 209 *F1*

Chapter V

Appendix

Contents

Technical data

Weighing System	
weighing range:	2000 mg
resolution:	0.1 µg
sample weight:	max. 2 g (with crucible)
Heating System	
temperature range:	20 ... 1100°C
heating rates:	0.001 ... 200 K/min
cooling rates:	12 min from 1100°C to 100°C
Sample Atmosphere	
vacuum:	10^{-2} mbar
static/dynamic:	inert gas, reactive gas* (*non-toxic, non-flammable, non-explodable)
mass flow controller:	(5 - 250) ml/min N_2, resolution: (1 ± 1) ml/min N_2
Sample Crucible	
outer diameter:	max. 9 mm
capacity:	max. 0.35 ml
material:	Al, Pt, Ag, Au, Al_2O_3,fused silica, graphite, ZrO_2 other materials upon request
Computer System	
	Operating system MS-WINDOWS XP Professional, MS-WINDOWS Vista Business/Enterprise, MS-WINDOWS 7 Professional/Enterprise/Ultimate USB Interface, Windows-compatible printer
Dimensions in mm (w x d x h) /Weight (net) in kg	
	660 x 530 x 360 / 37
Power Supply	
	230 V (115 V); 50 Hz or 60 Hz; < 1000 W
Operational Conditions	
	Ambient temperature 20°C +/- 5°C Relative air humidity 60% +/- 20% Atmospheric pressure 1013 hPa +/- 30 hPa

Technical data subject to change

Adapter for coupling systems

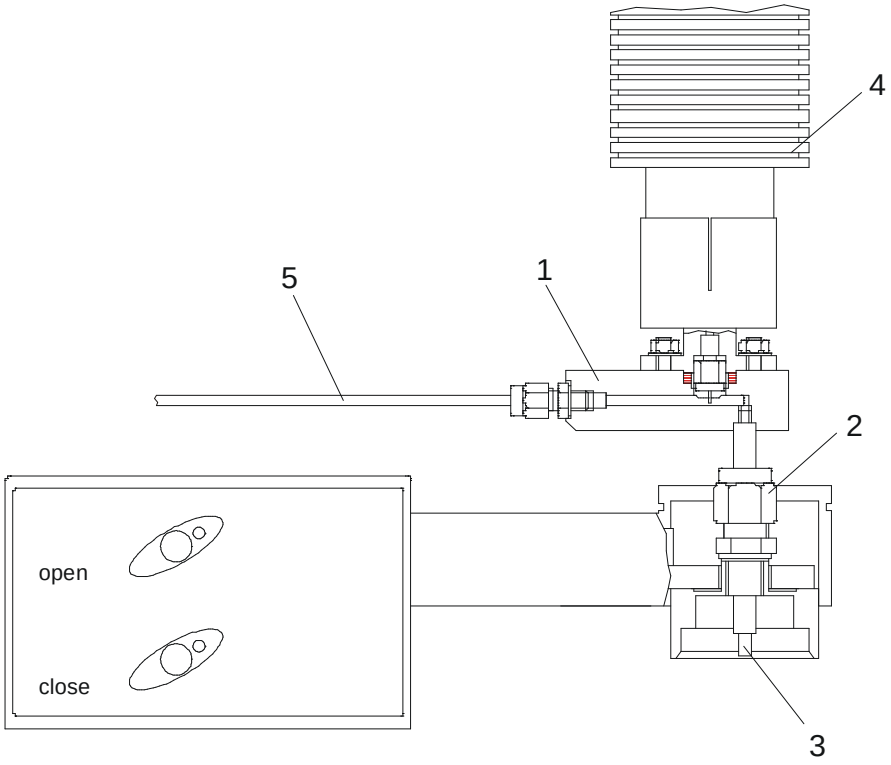
Appropriateness

coupling of the TG 209 *F1* with FT-IR Tensor and/or QMS Aëolos.

Order number: 6.220.10-43.5

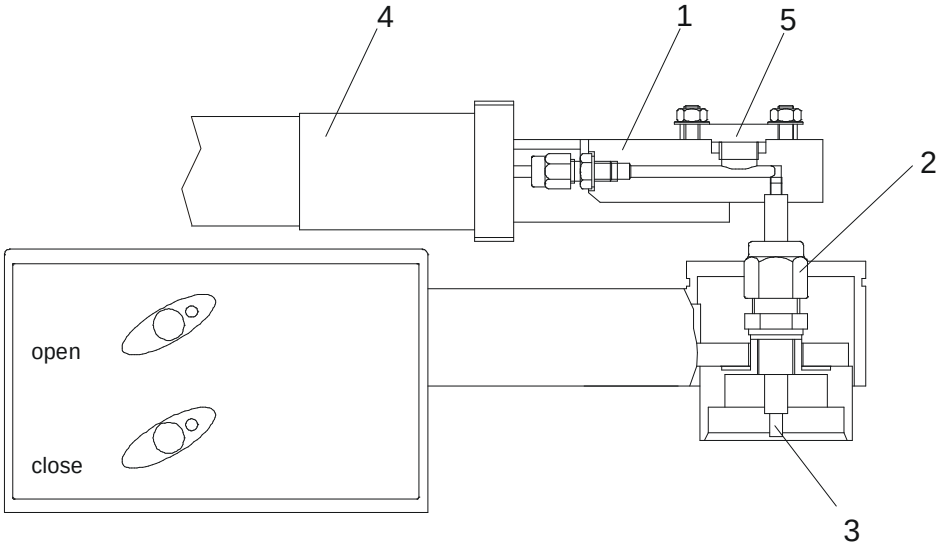
The adapter is simultaneously used at the same time for FT-IR and QMS coupling.

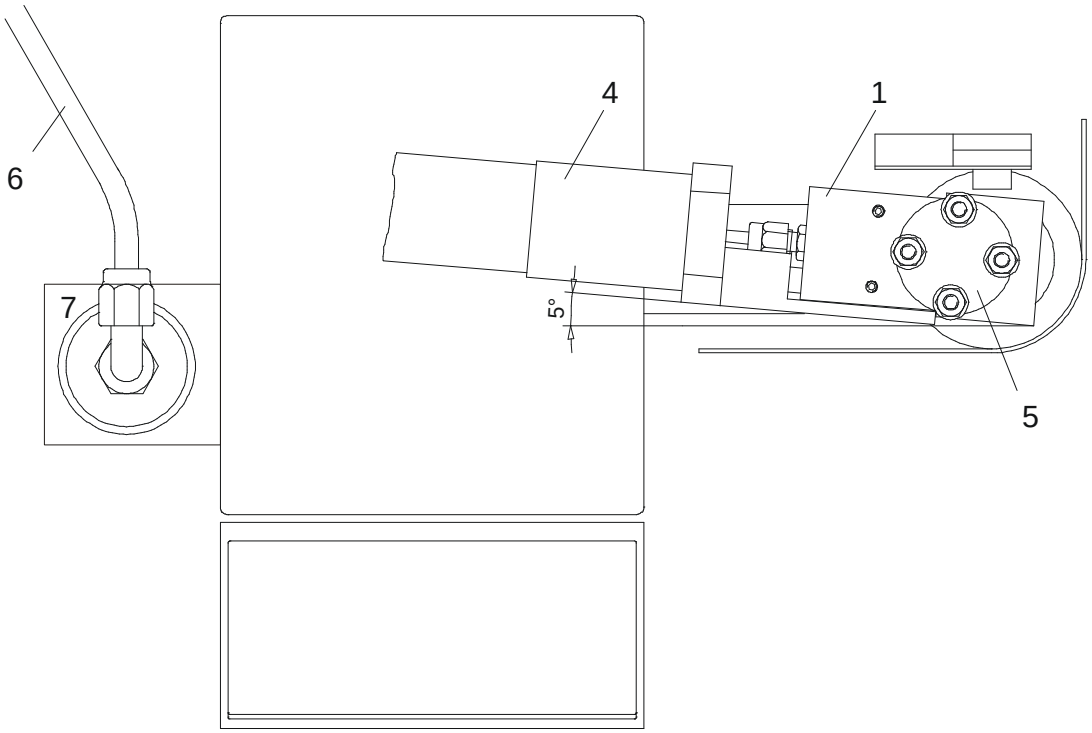
QMS-coupling



The adapter head (1) is attached by the screwing (2) on the cover of the TG 209 *F1*. The gas tube (3) can be unscrewed for cleaning. The transfer line (4) is fixed upright to the gas flow on the adapter head. The excess gas is dissipated via the gas outlet (5). The adapter can be heated up to 300°C.

FT-IR-coupling





The adapter head (1) is attached by the screwing (2) on the cover of the TG 209 *F1*. The gas tube (3) can be unscrewed for cleaning. The transfer line (4) is screwed on the end of the adapter head. The connection to the QMS is closed with a cover (5). The gas outlet tube (6) is guided from the outlet of the FT-IR cell back to the gas outlet of the TG 209 *F1*. The adapter head must be posed a little bit angular (about 5°), so that the filter (7) is not touched by the transfer line when the cover of the TG 209 *F1* is moved.

Attention!

The adapter may be heated only up to 230°C because of the FT-IR transfer line.

FT-IR and QMS-coupling (simultaneous mode)

Additionally to the FT-IR configuration the transfer line to the QMS is mounted.
At simultaneous mode the FT-IR transfer line is used as a bypass for the QMS.

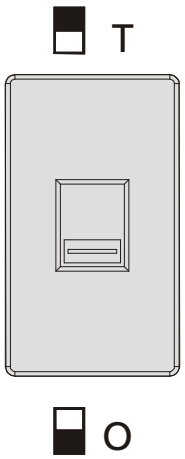
Attention!

The adapter may be heated only up to 230°C because of the FT-IR transfer line.

Transportation lock

- During transport the TG 209 *F1* is secured with a transport safety device – selected via the switch.
- The switch is placed at the left rear side of the instrument.
- For setting the switch use a small screw driver.

Normal operation (O)

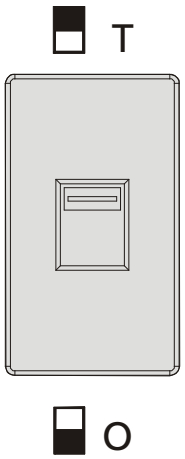


Operation

- For normal operation insert the sample holder and set the switch to position O (Operation).

Transport (T)

Transport



- For the transport remove the sample holder and set the switch to position T (Transport).

If the sample holder is not removed, the lowering furnace could damage the arrested sample holder!

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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.