

Magnettech ESR5000

- EPR Spectrometer with Scientific Grade Performance

Accessory Manual
Version 006



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1 Introduction

The Magnettech ESR5000 is a state of the art ESR spectrometer with scientific grade performance. It combines a compact size, allowing for a flexible field of applications, with a high field stability and outstanding sensitivity as necessary for scientific examinations. It also comes with versatile options, such as an automated goniometer for measurements of angular dependencies, low temperature measurements, a large collection of specialized sample holders and glassware, and comfortable software for data handling, data post processing, and evaluation.

A huge number of ESR 5000 accessories are available for special applications. This manual provides a short overview of the most common modules in the form of 'quick-start' guides.



Installation, initial commissioning, retrofitting, repairs, adjustments or dismantling of the device must only be carried out by Bruker Service or personnel authorized by Bruker. Damage due to servicing that is not authorized by Bruker is not covered by warranty.

1.1 Overview

The manual enables safe and efficient handling of the Magnettech ESR5000.

This manual is an integral part of the device, and must be kept in close proximity to the device where it is permanently accessible to personnel. In addition, instructions concerning labor protection laws, operator regulations tools and supplies must be available and adhered to.

Before starting any work, personnel must read the manual thoroughly and understand its contents. Compliance with all specified safety and operating instructions, as well as local work safety regulations, are vital to ensure safe operation.

The figures shown in this manual are designed to be general and informative and may not represent the specific Bruker model, component or software/firmware version you are working with. Options and accessories may or may not be illustrated in each figure.

1.2 Intended Use

This device has been designed and constructed solely for the intended use described herein. Intended use also includes compliance with all specifications within this manual. Please read and observe all technical specifications and safety considerations in this manual thoroughly.

The Magnettech ESR5000 is a research-grade Electron Spin Resonance spectrometer intended for studying materials containing unpaired electrons. It is intended to be used by a properly-trained operator in a laboratory environment. The instrument is only intended to be used in conjunction with the glassware, accessories, and software described within this manual.

Contra-Indication

Any use which exceeds or differs from the intended use shall be considered improper use. No claims of any kind for damage will be entertained if such claims result from improper use.

Only use original equipment! When using non-original equipment, an increased risk of accident is to be expected. When using non-original equipment from other manufacturers, any damages resulting from those will void any warranty claims towards Bruker.

In case of accidents of persons or damages to the environment or to other equipment with non-original equipment no liability is accepted by Bruker.

Applications, which are not specified in the technical data, on the specification plate or in the contract of delivery, are not allowed. Operating the measurement device with missing or damaged safety installations is forbidden.

2 Safety

This section provides an overview of all the main safety aspects involved in ensuring optimal personnel protection, as well as safe and smooth operation.

Non-compliance with the action guidelines and safety instructions contained in this manual may result in serious hazards.

The unit housing has been designed for safe operation, and the electrical power cord with a grounding conductor and safety plug.

All work on the unit must be performed by Bruker trained personnel. Opening of unit without the supervision of a Bruker representative will void any and all warranty.

2.1 System Owner's Responsibility

System Owner

The term *system owner* refers to the person who operates the device for trade or commercial purposes, or who surrenders the device to a third party for use/application, and who bears the legal product liability for protecting the user, the personnel or third parties during the operation.

System Owner's Obligations

The device is used in the industrial sector, universities and research laboratories. The system owner of the device must therefore comply with statutory occupational safety requirements.

In addition to the safety instructions in this manual, the safety, accident prevention and environmental protection regulations governing the operating area of the device must be observed.

In this regard, the following requirements should be particularly observed:

- The system owner must obtain information about the applicable occupational safety regulations, and - in the context of risk assessment - must determine any additional dangers resulting from the specific working conditions at the usage location of the device. The system owner must then implement this information in a set of operating instructions governing operation of the device.
- During the complete operating time of the device, the system owner must assess whether the operating instructions issued comply with the current status of regulations and must update the operating instructions if necessary.
- The system owner must clearly lay down and specify responsibilities with respect to installation, operation, troubleshooting, maintenance and cleaning.
- The system owner must ensure that all personnel dealing with the device have read and understood this manual. In addition, the system owner must provide personnel with training and hazard information at regular intervals.
- The system owner must provide the personnel with the necessary protective equipment.
- The system owner must warrant that the device is operated by trained and authorized personnel as well as all other work, such as transportation, mounting, start-up, the installation, maintenance, cleaning, service, repair and shutdown, that is carried out on the device.
- All personnel who work with, or in the close proximity of the device, need to be informed of all safety issues and emergency procedures as outlined in this user manual.

- The system owner must document the information about all safety issues and emergency procedures in a laboratory SOP (Standard Operating Procedure). Routine briefings and briefings for new personnel must take place.
- The system owner must ensure that new personnel are supervised by experienced personnel. It is highly recommended to implement a company training program for new personnel on all aspects of product safety and operation.
- The system owner must ensure that personnel are regularly informed of the potential hazards within the laboratory. This is all personnel that work in the area, but in particular laboratory personnel and external personnel such as cleaning and service personnel.
- The system owner is responsible for taking measures to avoid inherent risks in the handling of dangerous substances, preventing industrial disease, and providing medical first aid in emergencies.
- The system owner is responsible for providing facilities according to the local regulations for the prevention of industrial accidents and generally accepted safety regulations according to the rules of occupational medicine.
- All substances needed for operating and cleaning the device samples, solvents, cleaning agents, gases, etc. have to be handled with care and disposed of appropriately. All hints and warnings on storage containers must be read and adhered to.
- The system owner must ensure that the work area is sufficiently illuminated to avoid reading errors and faulty operation.
- The system owner must ensure that the laboratory is equipped with an oxygen warning device, in case the device is operated with e.g. nitrogen or helium.

Furthermore, the system owner is responsible for ensuring that the device is always in a technically faultless condition. Therefore, the following applies:

- The system owner must ensure that the maintenance intervals described in this manual are observed.
- The system owner must ensure that all (electrical, mechanical, etc.) safety devices are regularly checked to ensure full safety functionality and completeness.

2.2 Personnel Requirements



Only trained Bruker personnel are allowed to install, mount, retrofit, repair, adjust and dismantle the unit!

2.2.1 Qualifications

This manual specifies the personnel qualifications required for the different areas of work, listed below:

Laboratory Personnel

Laboratory personnel are health care professionals, technicians, and assistants staffing a research or health care facility where specimens are grown, tested, or evaluated and the results of such measurements are recorded. Laboratory personnel are able to carry out assigned work and to recognize and prevent possible dangers independently due to their professional training, knowledge and experience as well as profound knowledge of applicable regulations.

The workforce must only consist of persons who can be expected to carry out their work reliably. Persons with impaired reactions due to, for example, the consumption of drugs, alcohol, or medication are prohibited from carrying out work on the device.

When selecting personnel, the age-related and occupation-related regulations governing the location used must be observed.

2.2.2 Unauthorized Persons



WARNING

Risk to life for unauthorized personnel due to hazards in the danger and work zone!

Unauthorized personnel who do not meet the requirements described in this manual will not be familiar with the dangers in the work zone. Therefore, unauthorized persons face the risk of serious injury or death.

- ▶ Unauthorized persons must be kept away from the danger and work zone.
- ▶ If in doubt, address the persons in question and ask them to leave the danger and work zone.
- ▶ Cease work while unauthorized persons are in the danger and work zone.

2.2.3 Instruction

Personnel must receive regular instruction from the owner. The instruction must be documented to facilitate improved verification, see the example form below.

Date	Name	Type of Instruction	Instruction Provided By	Signature

2.3 Personal Protective Equipment

Personal protective equipment is used to protect the personnel from hazards which could affect their safety or health while working.

Personnel must wear personal protective equipment while carrying out the different operations at and with the device as described in this manual.

Depending on the operation, these are:

- Protective Goggles
 - Protect the eyes from injury due to flying object, cold gases, liquids and parts.
- Protective Gloves
 - Protect the hands from injury caused by contact with extremely cold gases, liquids or surfaces.
- Protective Clothes
 - Used to protect the body from injury caused by contact with extremely cold liquids or surfaces and for protection from wounds.

- Safety Shoes
 - Used to protect the feet from injury from falling of heavy objects. An anti-slip sole protects from injury caused by slipping and falling on slippery floor and steps. Only use safety shoes with non-ferromagnetic toe caps.
- Portable Oxygen Monitor and Alarm
 - Used to warn against low oxygen concentrations in surrounding air.

Additional protective equipment may be defined by the head of the laboratory to protect against hazards that are specific to the working area.

2.4 Basic Hazards

2.4.1 General Workplace Hazards

Transport

The device is well prepared for transport. Open the custom-fitting transport case carefully.



CAUTION

Risk of injury or damage from heavy objects

The unit packing crate is quite heavy:

- ▶ To prevent injury to your back, be very careful when lifting the box in or out of the packaging crate.
- ▶ Take care to avoid trapping fingers under the unit box as it is placed.
- ▶ Be sure that any surface on which the unit is temporarily or permanently placed is capable of supporting its weight and will not allow the unit to tip or slide off.

Hazards from Magnetic Fields



WARNING

Risk to life due to strong magnetic fields during operation

Strong magnetic fields may cause serious injuries or death and significant damage to property.

- ▶ People with heart pacemakers must be kept away from the device. The functionality of the heart pacemaker could be compromised.
- ▶ Persons with metal implants must be kept away from the device. Implants may heat up or be subject to magnetic attraction.
- ▶ Do not use ferromagnetic tools or items within the vicinity of the device.

In order to ensure user's health and safety the system owner needs to control or rather authorize access to the EPR-system in accordance with national and local regulations.

From an EMF-perspective, the magnet exhibits a very good shielding with a minimal magnetic stray field of < 0.2 mT (2 Gauss) outside the housing of the instrument. Therefore, the *group of people at particular risk* (persons with active or passive implants, people wearing medical devices or pregnant women) are at neglectable risk when working in the vicinity of the **operational** instrument. Nevertheless, all persons working with the system should be informed about the existence of the magnetic field during **operation**.

Sample materials



CAUTION

Risk of equipment damage due to improper sample placement.

Improper placement of sample materials may lead to mechanical damage of samples and equipment.

- ▶ Place the sample materials in the intended position.

Storage

Warning notice: Disconnect from the power supply when storing the equipment for a longer period.

Other hazards



CAUTION

Accident hazard from bright light

Peering into the lighting system's irradiation may result in temporary blinding of the eyes due to the bright light.

- ▶ Do not look into the ray of the light.
- ▶ Only use the unit fully mounted as described in the manual.
- ▶ Switch off the equipment before maintenance work.

3 Packaging, Storage and Transportation

3.1 Inspection at Delivery

Upon receipt, immediately inspect the delivery for completeness and transport damage.

Proceed as follows in the event of apparent external transport damage:

- Do not accept the delivery or accept only under reservation.
- Note the extent of the damage on the transport documentation or the shipper's delivery note.
- Initiate complaint procedure and document the condition of the transport damage with high quality photos (sharp, unshaken, everything legible).



Issue a complaint in respect to each defect immediately following detection. Damage compensation claims can only be asserted within the applicable complaint deadlines.

3.2 Packaging

About Packaging

The individual packages are packaged in accordance with anticipated transport conditions. Only environmentally friendly materials have been used in the packaging.

The packaging is intended to protect the individual components from transport damage, corrosion and other damage prior to assembly. The components should stay packed until just prior to installation.

Handling Packaging Materials

Keep the original container and packing assembly, at least as long as the warranty is valid, in case the unit has to be returned to the factory. When the packaging material is no longer needed dispose of it in accordance with the relevant applicable legal requirements and local regulations.

3.3 Storage & Transportation

Climatic Conditions for Storage & Transportation

- Store the packages according to the climatic conditions defined in IEC 60721-3-1, classification level 1K21.
- For the transportation, it is recommended to comply with the climatic conditions defined in IEC 60721-3-2, classification level 2K11.



Under certain circumstances, storage instructions may be affixed to packages that extend the requirements specified here. Comply with these accordingly.

4 Accessories

A huge number of accessories for special applications are available. Here is a short overview over the most common modules in form of quick installation guides.

NOTICE

Please read all safety instructions before installing and operating any accessories.

4.1 Temperature Controller TCH04 (Module E8000109)

The Temperature Controller TCH04 is made for heating and cooling of samples in a nitrogen gas flow.



Figure 4.1: Temperature Controller TCH04



Power off the system before plugging in and removing any of the cables. Otherwise, there is a risk of electrically damaging the spectrometer's mainboard as well as the accessory unit itself.

4.1.1 Technical Specifications

The temperature ranges are specified for the temperature at sample position.

Set-up specified for:

- Guidance Tube F-120 / 1.5T - E8000200
- Sample tube P 165 / 3 - E8000300



Be careful while operating 50 µl capillaries closed with sealing kit. At temperatures above 40 °C the sealing will melt.

General Information

Data	Value	Unit
Length	450	mm
Width	700	mm
Height	810	mm
Weight	17	kg

Table 4.1: Dimensions: Table

Data	Value	Unit
Length	Diameter = 388	mm
Width	---	mm
Height	600	mm
Weight	11.3	kg

Table 4.2: Dimensions: Bottle

Data	Value	Unit
Length	Diameter = 135	mm
Width	---	mm
Height	340 (with resonator dewar)	mm
Weight	1.7	kg

Table 4.3: Dimensions: Heater

Data	Value	Unit
Length	115	mm
Width	115	mm
Height	602	mm
Weight	0.7	kg

Table 4.4: Dimensions: Evaporator

Data	Value	Unit
Length	192	mm
Width	230	mm
Height	133	mm
Weight	6.8	kg

Table 4.5: Dimensions: E-Box

Data	Value	Unit
Length	Diameter = 80	mm
Width	---	mm
Height	120 (with exhaust steam attachment)	mm
Weight	0.3	kg

Table 4.6: Dimensions: Sample Holder Top

Connection Values

Data	Value	Unit
Voltage	115/230 [12/6]	VAC [A]
Real Power	380	W
Frequency	47 – 63	Hz

Table 4.7: Electrical Connection Values

Environmental Conditions

Data	Value	Unit
Temperature Range	10-30	°C
Relative humidity at 31 °C maximum	< 80	%
Decreasing linear to relative humidity at 40 °C max	< 50	%
Temperature Stability	+/- 1K target temperature at stable room temperature.	

Table 4.8: Environmental Conditions

Data	Value	Unit
Temperature Range	-180 to +200	°C
Heating and Cooling Times		
- Time from minimum to maximum	1200	seconds
- Time from maximum to minimum	800	seconds
Stability for entire temperature range	± 0.25	K
Accuracy of temperature at sample position	± 1	K

Table 4.9: Temperature at Sample Position

Control Mode

Data	Value
Maximum overshooting	$\pm 25\%$ of throw
Maximum of transient effects	3 cycles of oscillations

*Table 4.10: Control Mode***4.1.2 Installation**

The TC complete package (E8000109) comes with following accessories:

- Table size rack with electronic box and connection cables (700 x 450 x 800 mm)
- TCH04 with
 - E8000206 Sample Holder Top
 - E8000207 Sample Holder Bottom
 - E8000208 Resonator Dewar
 - E8000200 Guidance Tube
 - Heating unit (Heater)
 - Evaporation unit (Evaporator)
 - Mounting plate and screws
 - Protection cap
 - Nitrogen can with 1x O-ring and 2x clamps
- 2x wrench 13er
- 1x screwdriver hexagon socket screw 3 mm, 1x with 4 mm
- Mini level
- Power supply cable
- Spectrometer bus cable
- Tube for Nitrogen



Figure 4.2: The Complete Temperature Controller Package

Setup

- Put the spectrometer on the table size rack and use the mini level and wrenches to adjust the table. Afterwards, turn the spectrometer to the side and adjust the mounting plate on the bottom side with the 3 screws with use of the screwdriver for hexagon socket screws 3 mm.



Figure 4.3: Align the Spectrometer on the Table

- Align the spectrometer on the table. The mounting plate should fit in the center of the hole.



Figure 4.4: The Spectrometer Mounting Plate

- Use the wider nitrogen tube for connecting backside of the spectrometer with the electronic box. Also, connect the front side with the smaller tube. It is used to flush the resonator with nitrogen gas to avoid oxidation processes at high temperature and formation of condensation water outside the resonator Dewar at low temperatures. Nitrogen gas is directly taken from the can. A peristaltic pump inside the electronic box is used for purging the cavity with inert gas.



Figure 4.5: The Nitrogen Tube Connection

- Mount the heating unit by sticking the resonator Dewar into the designed hole around the temperature sensor. The glass has a mark at one of the two sides. This comes from the glass blowing. Turn this side to the bottom. This step is only necessary if the glass was packed separately.



Figure 4.6: Mounting the Heating Unit

- Remove the original spectrometer sample holder.



Figure 4.7: Removing the Sample Holder

- Carefully insert the heating unit with resonator Dewar from the bottom side into the spectrometer system and fix it with the 2 screws with aid of the screwdriver 4 mm. Easiest way is laying on the floor to mount the unit.



Figure 4.8: Inserting the Heating Unit with the Resonator Dewar

- Carefully insert the sample holder bottom into resonator Dewar. It will fall down to the desired position directly over the temperature sensor on the glass taper. It is used to fix the position of the guidance tube later.
- Mount the new sample holder top out of the delivery package.



Figure 4.9: Mounting the Sample Holder Top

- Insert the guidance tube into the sample holder top. First unscrew the Teflon inside of the holder and afterwards fix it to mount the guidance tube. Cover it with the protection cap to avoid problems with condensation.

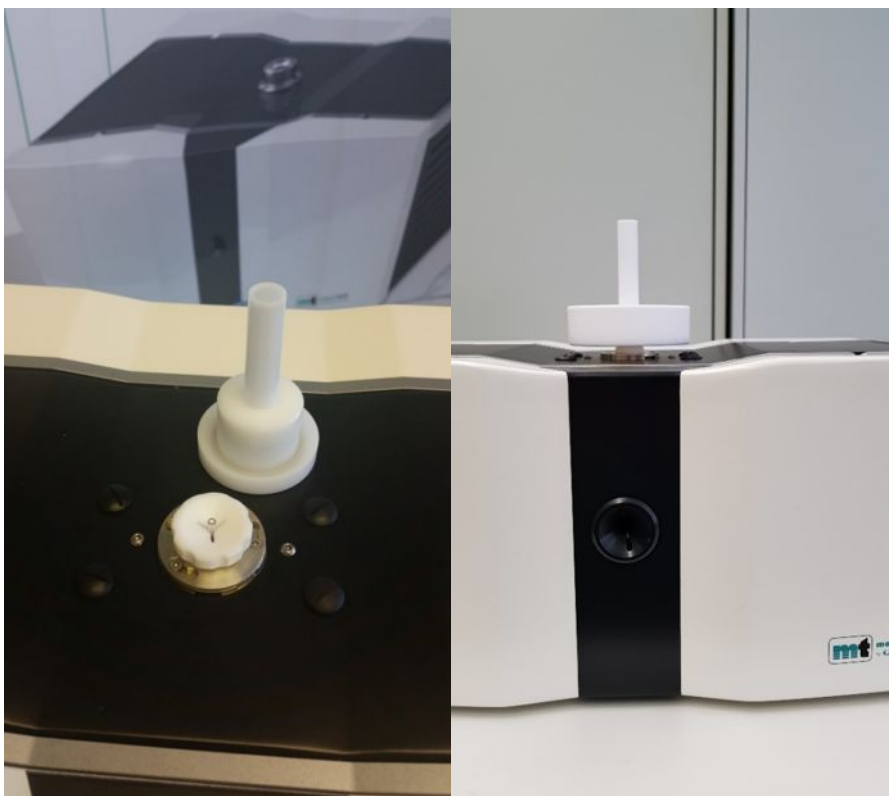


Figure 4.10: Inserting the Guidance Tube

- Use the delivered cleaning brush to clean the riser pipe of the evaporation unit (indicated with red arrows in the figure).

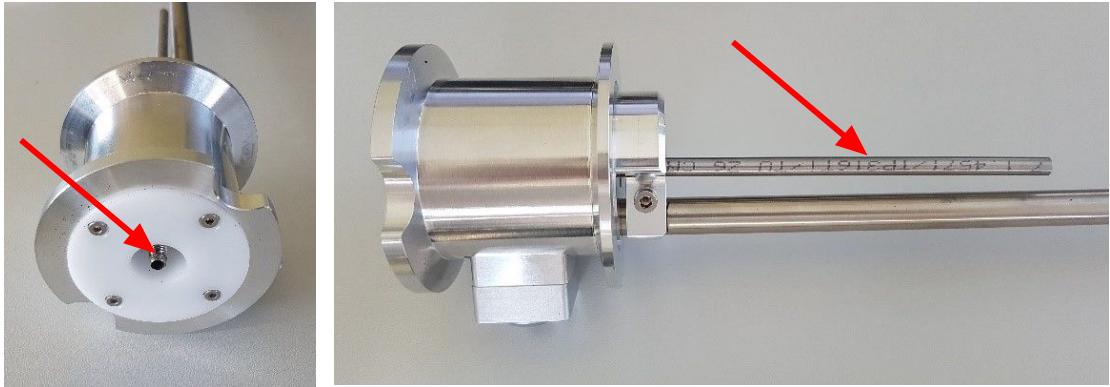


Figure 4.11: Use the Cleaning Brush to Clean the Riser Pipe

CAUTION



Risk of injury due to condensed water residues.

Residues of condensed water could harm the device and also your health.

- Use the delivered cleaning brush to clean the red marked riser pipe of the evaporation unit each time before inserting the evaporation unit into the nitrogen can.

Wear appropriate protection equipment (closed shoes, cryogen protection gloves and safety glasses) when performing the following procedure.

- Place the O-ring fitting on the liquid nitrogen can and slowly insert the evaporation unit into the can. Use the C-clamp to secure the evaporation unit to the nitrogen can. Place the nitrogen can directly beneath the heating unit and place the O-ring fitting on top of the evaporation unit. Extend the bottom of the heating unit and align with the evaporation unit. Use the C-clamp to secure the evaporation unit to the heating unit. Connect the two cables labeled *Evaporator* and *Heater* from the electronic box to the respective units. Be careful not to mix the connection of the cables.



Figure 4.12: Inserting the Evaporation Unit and Connecting to the Heating Unit

- Connect the smaller Nitrogen tube to the Evaporator.



Figure 4.13: Connecting the Nitrogen Tube to the Evaporator

- Connect the heating unit and evaporation unit power to the electronic unit.



Figure 4.14: Connecting the Electronic Unit



Power off the system before plugging in and removing any of the cables. Otherwise, there is a risk of electrically damaging of the spectrometer's mainboard as well as the accessory unit itself.

Turning off the Temperature Controller TCH04

After usage switch off the electronic box. Remove the clamp between the evaporator and the heating unit. Raise the bottom part of the heating unit to separate it from the evaporator. Pull the nitrogen can out. Remove the clamp connecting the evaporator and the can. Pull the evaporator out of the can. Let the evaporator unit dry before next use or use a hair dryer (not heat gun) to dry it. The Dewar and the heating unit can stay mounted to the spectrometer. Flush the Dewar with dry air or nitrogen gas to remove any remaining moisture.

Exchanging the Nitrogen Can

A full nitrogen can will last around one working day. If the can is empty, a message in the ESRstudio will occur. The measurement is aborted. Please act as follows:

- Switch off the electronic box.
- Remove the clamp between heating and evaporation unit.
- Pull the nitrogen can out.
- Remove the second clamp and pull the evaporation unit out of the nitrogen can.
- Fill up the can.
- Make sure to dry the evaporation unit and flush the Dewar with dry air or nitrogen gas before reassembling the system.
- Re-assemble the system.



The nitrogen can may be pressurized! Please be careful while removing the evaporation unit!

4.1.3 Software Handling

- Use **Temperature Series** in the **Recipe Editor** tab.

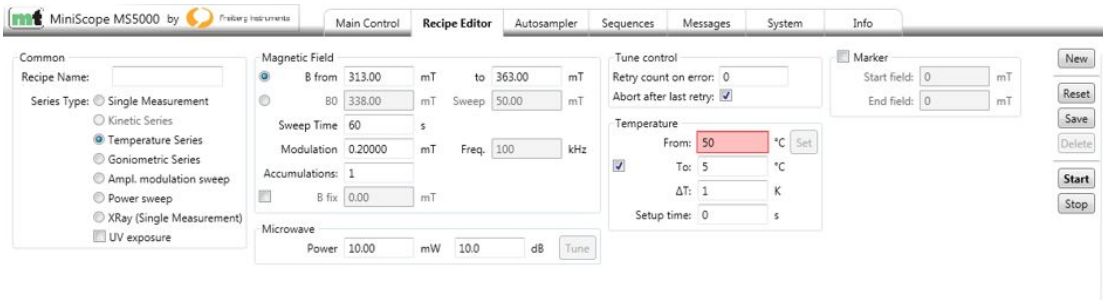


Figure 4.15: Software Handling

- Select the desired temperature in °C in the red marked window and push the **Set**-button to heat the system up. Available temperature is from room temperature up to +60 °C.
- Start your measurement recipe.
- Temperature series are selectable by setting the start and end temperature and the step ΔT in K. **Setup time** is a time during which the system stays at the chosen temperature before the acquisition starts.
- Advanced users may use the **Sequences** tab to define complex measurement sequences.
- To record the temperature profile, use the diagram in the slider **System** and **Device state** with the setting **Temporator.Temperature [°C]**.

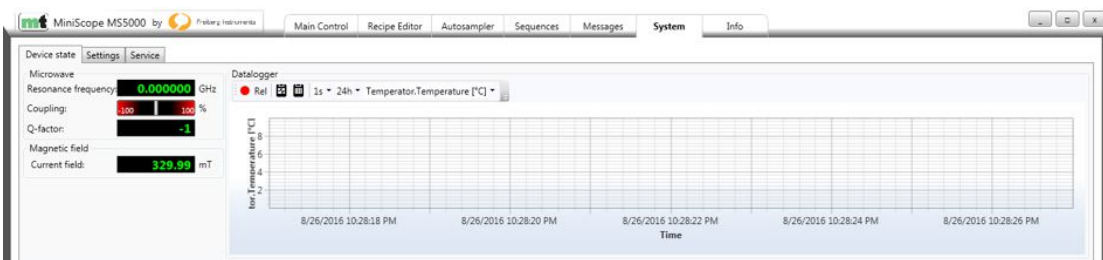


Figure 4.16: Device State

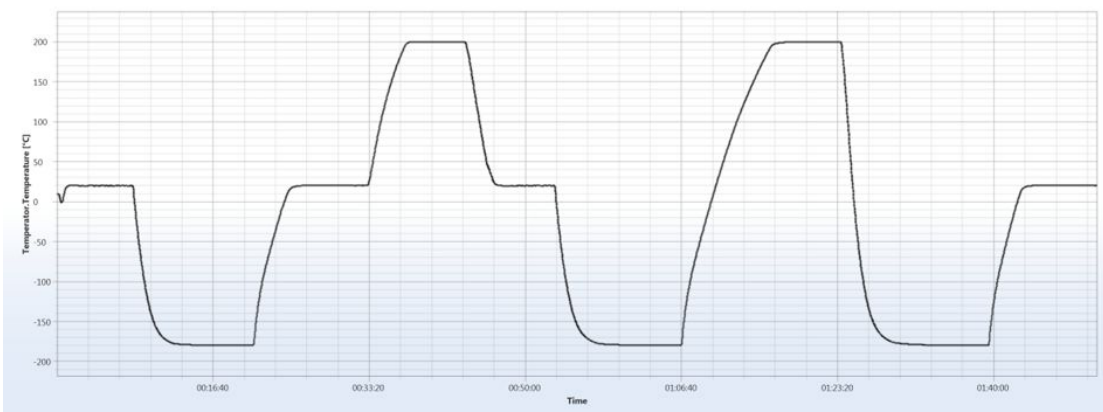


Figure 4.17: Temporator Temperature Graph

4.2 Autosampler for Solids and Powders (Module E8000121)

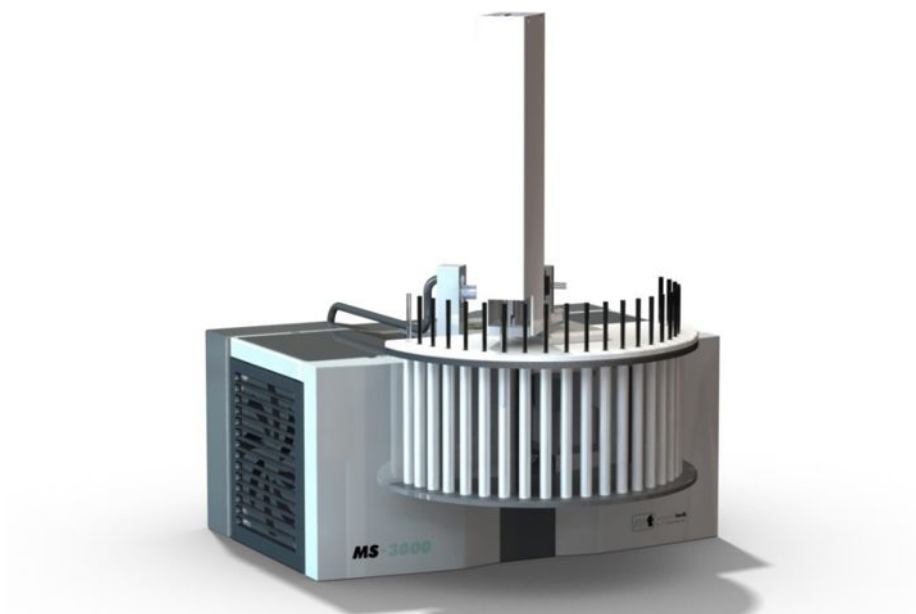


Figure 4.18: Autosampler for Solids and Powders



Power off the system before plugging in and removing any of the cables. Otherwise, there is a risk of electrically damaging the spectrometer's mainboard as well as the accessory unit itself.

4.2.1 Installation

The Autosampler for solids and powders comes with a transport case containing with following accessories:

- Grabbing unit
- Carousel
- Set of 26 glass tubes fitting to your samples with grips
- Tool for centering
- Spectrometer bus cable



Figure 4.19: Autosampler for Solids and Powders Parts



Important: There is a transport lock. Remove it before the first use and secure it again for transport.



Figure 4.20: Removing the Transport Lock

Setup

- Place the spectrometer on top of a table. Remove the 4 plastic screws and the inside of the holder.

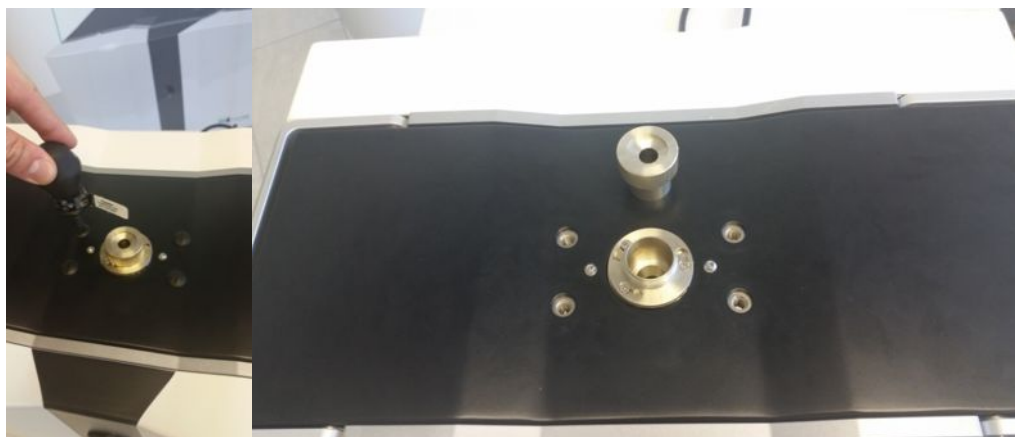


Figure 4.21: Removing the Inside of the Holder

- Mount the Autosampler with the 4 screws on top of the spectrometer, connect the spectrometer Bus cable on the backside, remove the tube holder by loosening the 4 inner screws and using the centering tool to align the setup centrally. Afterwards tighten all screws and replace the centering tool from the tube holder.



Figure 4.22: Mounting the Autosampler

- Mount the carousel with the sample tubes only by clipping over the 2 pins.



Figure 4.23: Mounting the Carousel

Teaching

The Autosampler comes pre-installed. Nevertheless, due to mechanical tolerances, a readjustment may be necessary. Two positions are critical: handover position of slot 1 and handover to the ESR spectrometer.

Therefore, please follow the instructions from the manual or contact a member of our support team.

For height adjustment of the sample tube in the spectrometer, screw or unscrew the inner part of the tube holder.

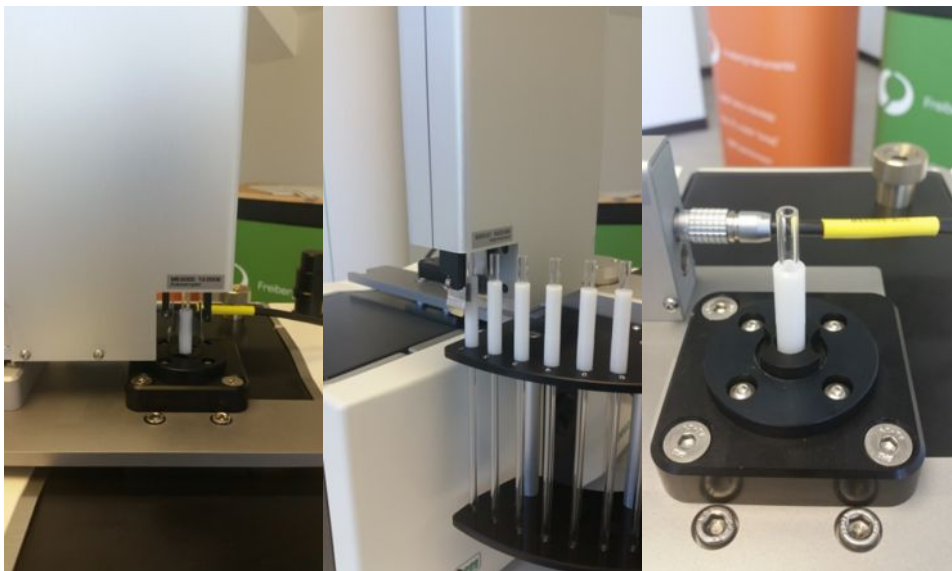


Figure 4.24: Sample Tube Height Adjustment

Combination with a Goniometer

A combined usage of autosampler and goniometer is possible.

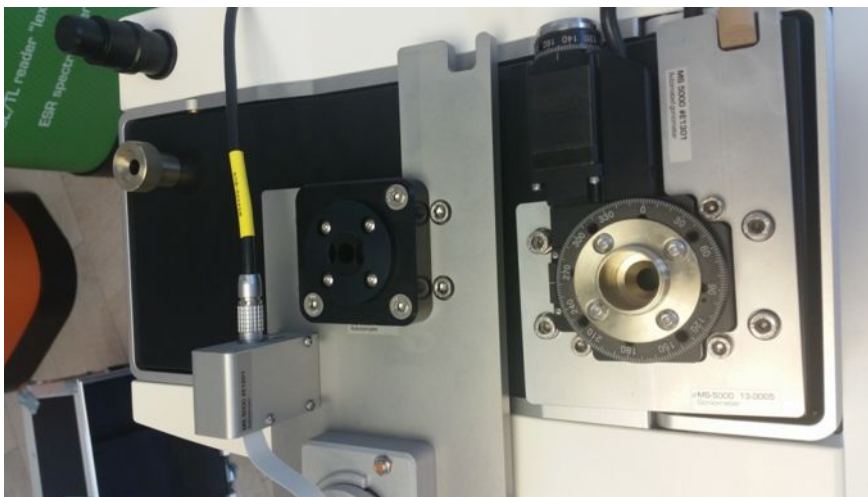


Figure 4.25: Combination with a Goniometer

Therefore, please act as follows:

- Loosen the 3 screws connecting the goniometer to the ground plate.



Figure 4.26: Loosen the Screws Connecting the Goniometer

- Turn the ground plate and remove the PCB by loosening the 5 screws.



Figure 4.27: Removing the PCB

- Remove the tube holder from the ground plate of the autosampler, dismount it from the spectrometer (if necessary) and turn it to the side. Mount the goniometer (screwed from the front side).



Figure 4.28: Removing the Tube Holder from the Ground Plate

- Mount the PCB on the backside of the ground plate with the 5 screws and cord grip.

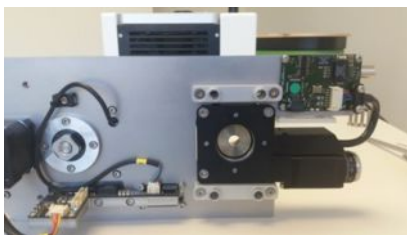


Figure 4.29: Mounting the PCB on the Back of the Ground Plate

- Mount the setup to the spectrometer and center it like described before. Connect both autosampler and goniometer to the spectrometer with help of the spectrometer bus cables.



Figure 4.30: Mounting the Autosampler and Goniometer to the Spectrometer

4.2.2 Software Handling

Use the tab **Autosampler**. The first window on the left is the control unit for the autosampler. Use it to load or unload a sample and start your recipe manually.

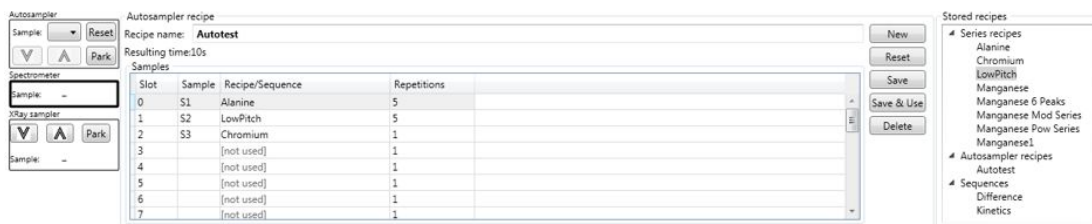


Figure 4.31: Autosampler Software Handling

In case of desired automated handling use the menu in the middle. Pull pre-defined recipes from the list on the right to the slot of your sample, name the sample, define the number of repetitions, save and use the new autosampler recipe. Advanced users may also use the sequence editor.

4.3 Automated Goniometer (Module E8000127)

The automated goniometer is intended for doing angular dependent measurements, especially on crystals or powders. Furthermore, it is used to increase the accuracy in case of performing measurements at same sample with different angles and doing an averaging.

The goniometer is able to turn from 0 to 360° with a minimum step width of 0.1°.

Combination with autosampler is possible as well.

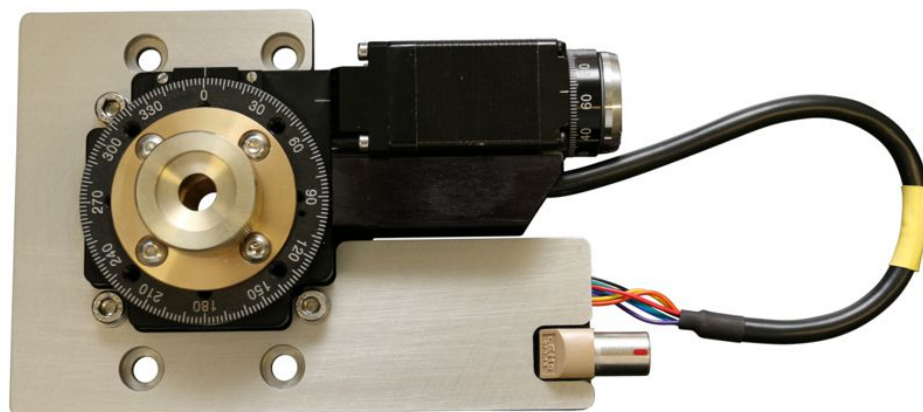


Figure 4.32: Automated Goniometer



Power off the system before plugging in and removing any of the cables. Otherwise, there is a risk of electrically damaging of the spectrometer's mainboard as well as the accessory unit itself.

4.3.1 Technical Specifications

Automated Goniometer Module E8000127

- Fully automated angular rotation of sample.
- Step size 0.1 to 180°.
- Each measurement of a new spectrum starts with an automated readjustment of the spectrometer for best measurement performance.
- Special adaption for Autosampler (available on request).

4.3.2 Installation

The Automated Goniometer comes with following accessories:

- Goniometer unit with 4 screws
- Spectrometer bus cable
- Tool for centering



Figure 4.33: Automated Goniometer Parts

Setup

- Place the spectrometer on top of a table. Remove the 4 plastic screws and the inside of the holder.



Figure 4.34: Removing the Inside of the Holder

- Mount the Goniometer with the 4 screws on top of the spectrometer, connect the spectrometer bus cable on the backside, remove the tube holder by loosening the 4 inner screws and use the centering tool to align the setup centrally. Afterwards tighten all screws and replace the centering tool from the tube holder.



Figure 4.35: Mounting the Goniometer on Top of the Spectrometer

Combination with an Autosampler

A combined usage of autosampler and goniometer is possible.

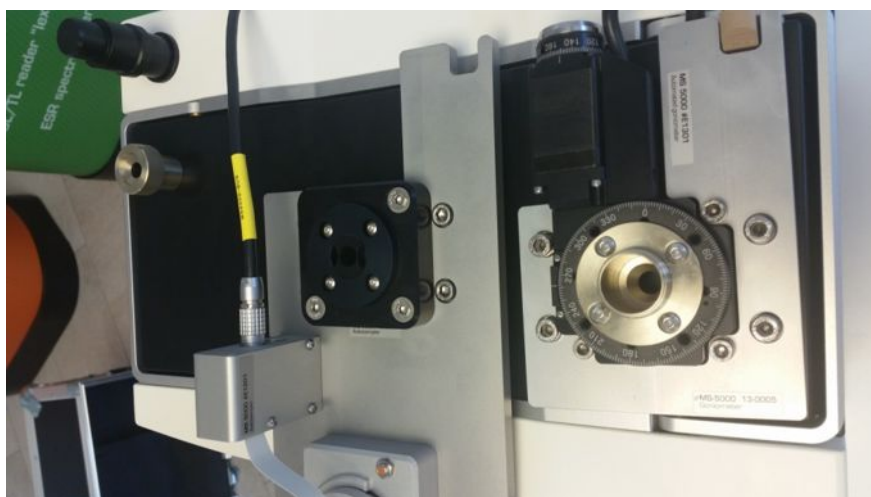


Figure 4.36: Combination with a Goniometer

Therefore, please act as follows:

- Loosen the 3 screws connecting the goniometer to the ground plate.



Figure 4.37: Loosen the Screws Connecting the Goniometer

- Turn the ground plate and remove the PCB by loosening the 5 screws.



Figure 4.38: Removing the PCB

- Remove the tube holder from the ground plate of the autosampler, dismount it from the spectrometer (if necessary) and turn it to the side. Mount the goniometer (screwed from the front side).



Figure 4.39: Removing the Tube Holder from the Ground Plate

- Mount the PCB on the backside of the ground plate with the 5 screws and cord grip.

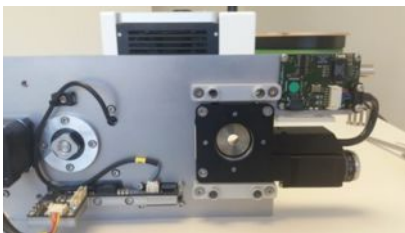


Figure 4.40: Mounting the PCB on the Back of the Ground Plate

- Mount the setup to the spectrometer and center it like described before. Connect both autosampler and goniometer to the spectrometer with help of the spectrometer bus cables.

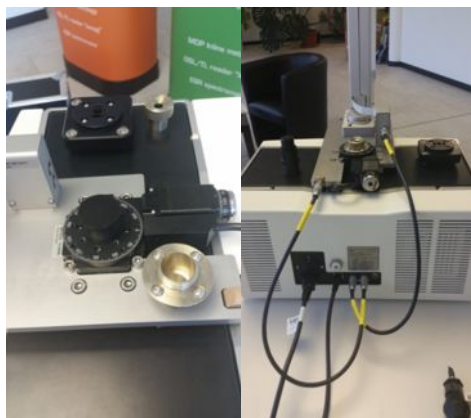


Figure 4.41: Mounting the Autosampler and Goniometer to the Spectrometer

4.3.3 Software Handling

Select **Recipe editor** and **Goniometer Series**. Select your parameter and on the right-hand side an angle range and the step width.

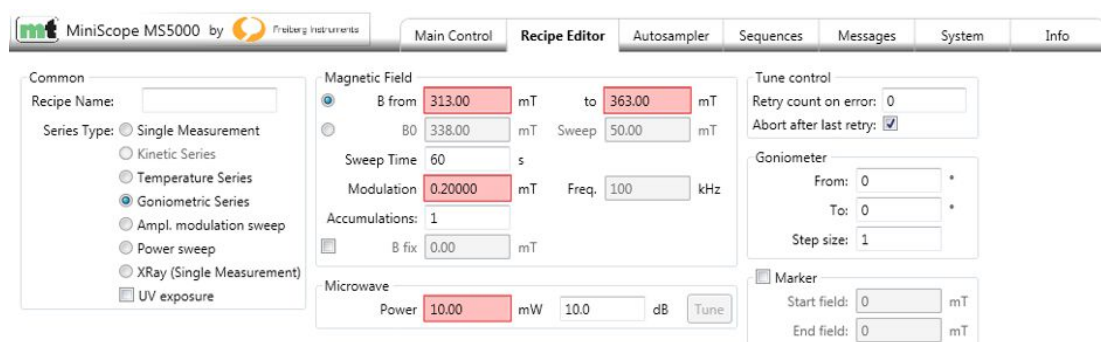


Figure 4.42: Goniometer Software Handling

4.4 Bio-Temperature Controller BTC01 (Module E8000286)

The Bio-Temperature Controller (BTC) is intended for temperature stabilization of biological systems like tissue samples.



Figure 4.43: Bio-Temperature Controller BTC01



Power off the system before plugging in and removing any of the cables. Otherwise, there is a risk of electrically damaging of the spectrometer's mainboard as well as the accessory unit itself.

4.4.1 Technical Specifications

- Requires Small Rack (Module E8000156)
- Temperature range: Room temperature to +60 °C [+85 °C on request]
- Accuracy: Better than ± 0.1 K
- Stability: Better than ± 0.25 K

4.4.2 Installation

The BTC comes with a transport case containing the following accessories:

- E8000286 BTC01 (Room temperature up to 60/85 °C)
 - Mounting plate with 3 screws
 - Power supply and cable
 - Spectrometer bus cable
- E8000156 Small rack



Figure 4.44: Bio-Temperature Controller BTC01 Parts

Setup

- Place the small rack on a table and the spectrometer on top. The pedestals should fit the slots.



Figure 4.45: Placing the Small Rack on a Table

- Install the mounting plate with the 3 screws with help of the hex-wrench. Do not tighten too much. If necessary, remove the front plate. It is only fixed with magnets.



Figure 4.46: Installing the Mounting Plate

- Align the BTC from the backside of the spectrometer by a slight turning. Loosen the screws if necessary and tighten them at the end after the BTC is in position. Connect the bus cable and the power supply.



Figure 4.47: Mounting the BTC on the Backside of the Spectrometer

4.4.3 Software Handling

- Use **Temperature Series** in the **Recipe Editor** tab.

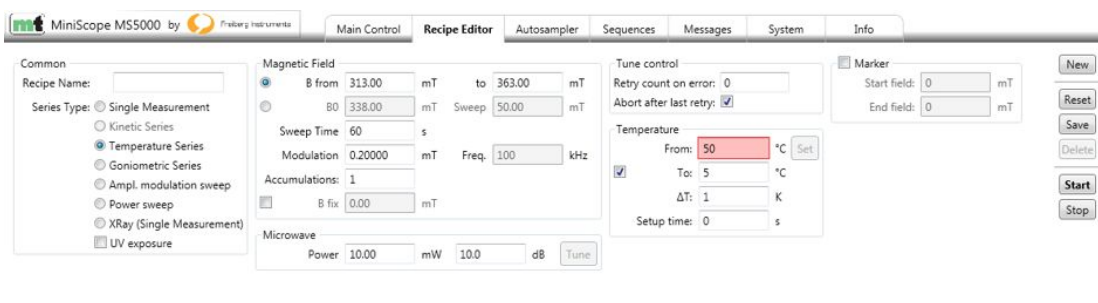


Figure 4.48: Software Handling

- Select the desired temperature in °C in the red marked window and push the **Set**-button to heat the system up. Available temperature is from room temperature up to +60 °C.
- Start your measurement recipe.
- Temperature series are selectable by setting the start and end temperature and the step ΔT in K. **Setup time** is a time during which the system stays at the chosen temperature before the acquisition starts.
- Advanced users may use the **Sequences** tab to define complex measurement sequences.
- To record the temperature profile, use the diagram in the slider **System** and **Device state** with the setting **Temperatur.Temperature [°C]**.

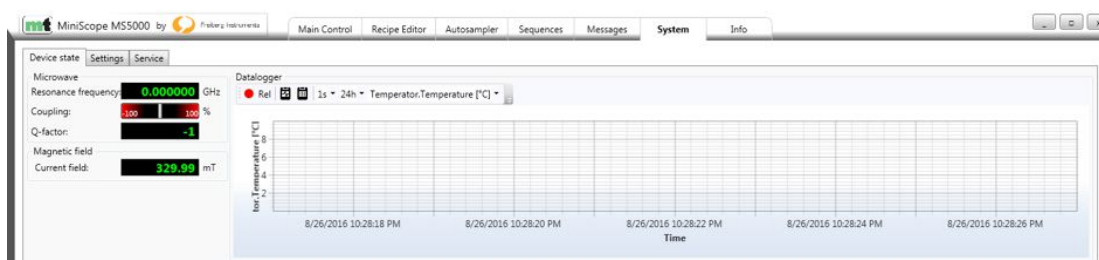


Figure 4.49: Device State

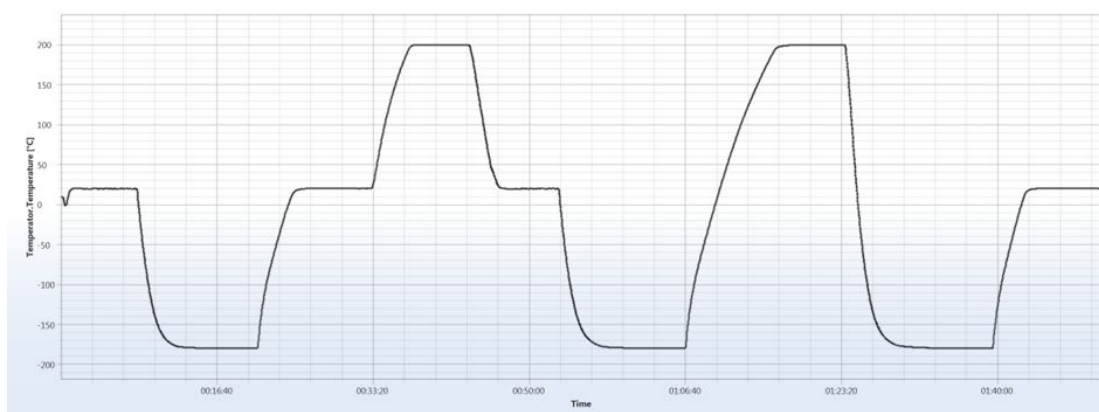


Figure 4.50: Temperature Graph

4.5 Bio-Temperature Controller BTC03 (Module E8000106)

The fully integrated BTC03 (Module E8000106) is intended for sample annealing, especially for biological samples.



Figure 4.51: Bio-Temperature Controller BTC03 (Module E8000106)



Power off the system before plugging in and removing any of the cables. Otherwise, there is a risk of electrically damaging the spectrometer's mainboard as well as the accessory unit itself.

4.5.1 Technical Specifications

The BTC03 is intended for sample annealing especially for biological samples.

- Temperature range: 0 °C to +40 °C
- Accuracy: Better than ± 0.5 K
- Stability: Better than ± 0.25 K
- Requires Small Rack (Module E8000156)

4.5.2 Installation

Scope of delivery

The BTC03 comes with a transport case containing the following accessories:

- BTC03 (0 °C up to 40 °C)
 - Mounting plate with 3 screws
 - Power supply and cable
 - ESR5000 Bus cable
 - Resonator dewar with sample holder for BTC03
 - Tube 4 mm outer diameter
 - Tube 8 mm outer diameter
 - Tube adapter from 8 mm to 4 mm
- Small rack.



Figure 4.52: The BTC03 with Transport Case and Accessories

Setup

- Place the small rack on a table and the ESR5000 on top. The pedestals should fit the slots.



Figure 4.53: The BTC03 with Rack

- Install the mounting plate with the 3 screws with help of the hex-wrench. Do not tighten too much. If necessary, remove the front plate. It is only attached with magnets.

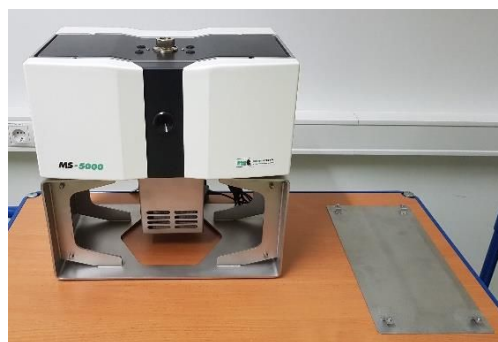


Figure 4.54: Installing the Mounting Plate

- Insert the BTC from the backside of the spectrometer by a slight turning. Be careful with the temperature sensor on top of the BTC. This sensor must be in vertical position like in the picture below. Connect the bus cable, the power supply and the nitrogen tube.



Figure 4.55: Temperature Sensor

- Insert the glass dewar on the top of the ESR5000. The temperature sensor must be inside of the dewar directly below sample position.



Figure 4.56: Placement of the Temperature Sensor Inside the Dewar

4.5.3 Software Handling

- Use **Temperature Series** in the **Recipe Editor** tab.

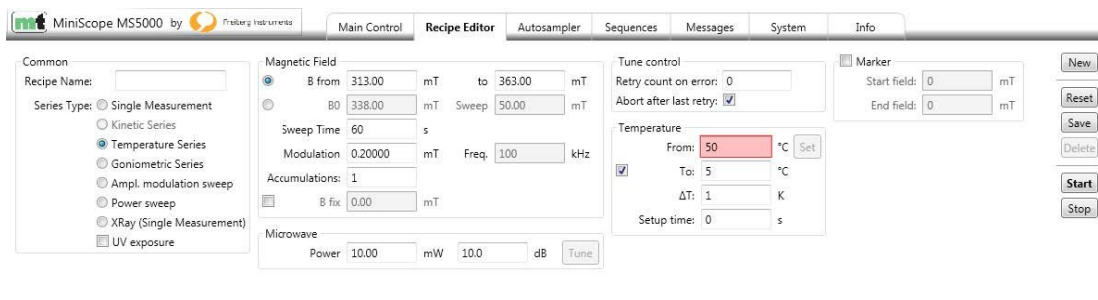


Figure 4.57: Temperature Series in the Recipe Editor

- Select the desired temperature in °C in the red marked window and push the **Set**-button to heat the system up.
- Start your measurement recipe. Temperature series are selectable by setting the start and end temperature and the step width in K. **Setup time** is a time during which the system stays at the chosen temperature before the acquisition starts.
- Advanced users may use the **Sequences** tab to define measurement sequences.
- To record the temperature profile, use the diagram in the tab **System** and **Device state** with the setting **Temporator.Temperature [°C]**.

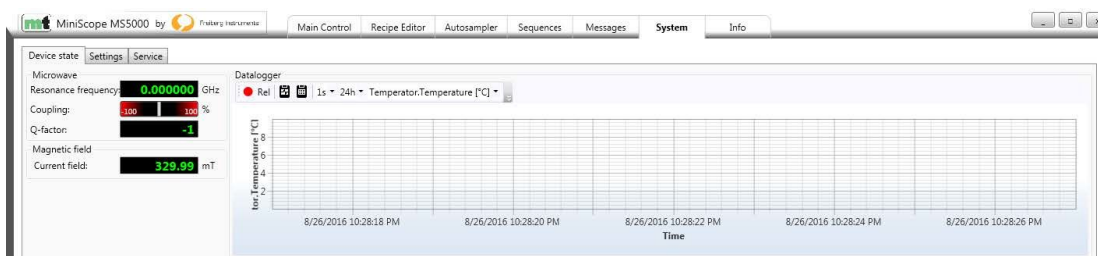


Figure 4.58: Recording the Temperature Profile

4.6 Optical Stimulation Unit - Integrated (Module E8000130)

The optical stimulation unit is made for sample illumination. This may catalyze chemical reactions or influence the sample behavior in general.

The lamp is directly controlled and triggered by the ESRstudio software.

Following operation modes are possible:

- Lamp is switched on at measurement start.
- Lead time between measurement start and switching on of the lamp.

Users are fully free to select lead and illumination time as well as the power output as desired.

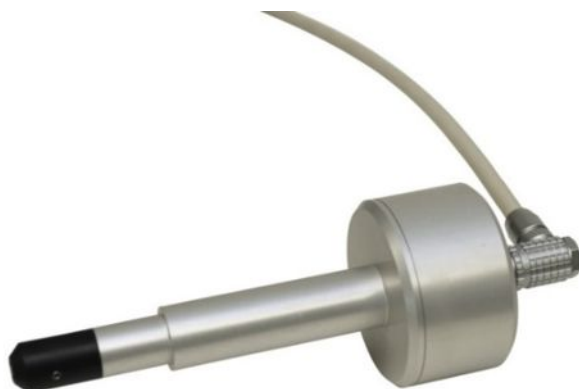


Figure 4.59: Optical Stimulation Unit – Integrated



Power off the system before plugging in and removing any of the cables. Otherwise, there is a risk of electrically damaging of the spectrometer's mainboard as well as the accessory unit itself.

CAUTION



Accident hazard from bright light

Peering into the lighting system's irradiation may result in temporary blinding of the eyes due to the bright light.

- ▶ Do not look into the ray of the light.
- ▶ Only use the unit fully mounted as described in the manual.
- ▶ Switch off the equipment before maintenance work.

4.6.1 Technical Specifications

The default setting is as follows:

- Wavelength: 365 nm
- Power output: 400 mW

Other wavelengths on request, following are available:

Wavelength [nm]	Power output [mW]
462	300
523	100
590	80
625	200
850	400

Table 4.11: Available Wavelengths

4.6.2 Installation

The Optical Stimulation Unit comes with following accessories:

- Optical Stimulation (OS) unit
- Spectrometer bus cable (long)
- Holder and positioning system



Figure 4.60: Optical Stimulation Unit – Integrated Parts

Setup

Remove the front cowl with the tool from the delivery package. Insert the metal housing and the OS unit. Fix it with the screw. Connect the unit with the spectrometer bus cable to one of the 3 plugs at the backside of the system.



Figure 4.61: Optical Stimulation Unit Installation

4.6.3 Software Handling

Start the ESRstudio and log in as **Advanced**. Switch to tab **System** and **Settings**. Search for the field **Optical unit**, select **BusDevice**, leave the COM-Port empty and type in the maximum power (optional, specified in the measurement capability). **Save** and **Initialize** the system.

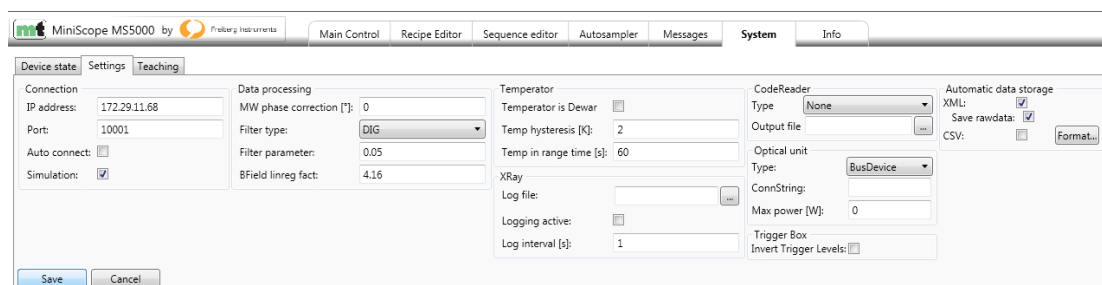


Figure 4.62: Optical Stimulation Unit Software Handling

Recipe editor

Choose **Optical sample irradiation** in the list on the right-hand side and define the power (from 0 to 100%) as well as possibly a time shift for illumination start after measurement start (Lead Time) and the illumination time (Exposure Time).

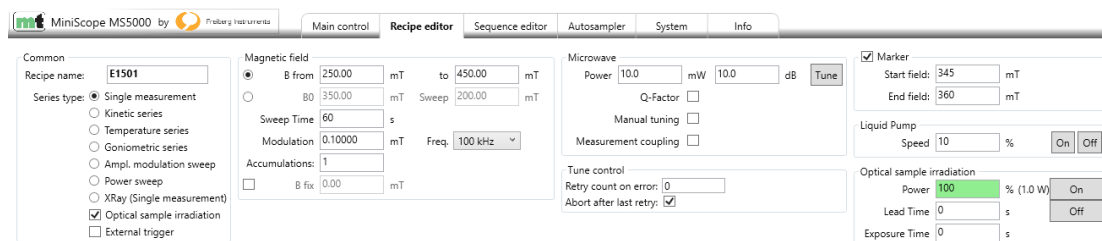


Figure 4.63: Optical Stimulation Unit Recipe Editor

For example, select a sweep time of 60 s, lead time of 10 s and exposure time of 20 s. The measurement will start and after 10 s the lamp will be switched on for 20 s.

The power selection is in %. If the maximum power is inserted in the settings tab, we will calculate the illumination power.



The displayed power is the optical power output of the lamp, not the power at sample position! This is not calculable because the modulation sticks are in the optical path, therefore the power at sample's position is strongly depending on the geometry of the sample tube.

For manual switching on or off the light source, use the **On** and **Off** button. Do not switch on the lamp while not mounted to avoid injuries.

Optical sample irradiation

Power	100	% (1.0 W)	On
Lead Time	0	s	Off
Exposure Time	0	s	

Keep an eye on the status of the lamp displayed in the menu at the bottom:

UV inactive

4.7 Optical Stimulation Unit Hamamatsu (Module E8000131)

Hamamatsu lamps are made for sample illumination. This may catalyze chemical reactions or influence the sample behavior in general.

The lamp is directly controlled by the ESRstudio software but triggering runs through the computer. This may cause latencies. Optionally, you may use the trigger box (E8000149) to avoid this.



Figure 4.64: Optical Stimulation Unit – External

Following operation modes are possible:

- Hamamatsu lamp is switched on at measurement start.
- Lead time between measurement start and switching on of the lamp.

Users are fully free to select lead and illumination time as well as the power output as desired.

Remember that the Hamamatsu lamp needs a warm up time not controllable by the ESRstudio software. An additional independent controlling software for the lamp is provided on a CD with the lamp.



⚠ CAUTION

Accident hazard from bright light

Peering into the lighting system's irradiation may result in temporary blinding of the eyes due to the bright light.

- ▶ Do not look into the ray of the light.
- ▶ Only use the unit fully mounted as described in the manual.
- ▶ Switch off the equipment before maintenance work.

4.7.1 Technical Specifications

Since we offer different wavelength ranges, technical data belong to the chosen lamp type. Find an excerpt of technical data here:

SELECTION GUIDE			
Type No.	Radiant wavelength range ^①		Lamp for maintenance ^②
L9566-01A / L9588-01A	[365 nm type]	300 nm to 450 nm	L10852
L9566-02A / L9588-02A	[250 nm band enhanced type]	240 nm to 400 nm	L10852
L9566-03 / L9588-03	[Visible light type]	400 nm to 700 nm	L8253
L9566-04 / L9588-04	[Total reflection type]	300 nm to 800 nm	L8253
L9566-06A / L9588-06A	[365 nm wide band type]	240 nm to 550 nm	L10852

NOTE: ① Major radiant wavelengths. Various optical filters (sold separately) can also be attached. Refer to the radiant spectral distribution graph for details on the spectral distribution.
 ② L10852 is 200 W super-quiet mercury-xenon lamp. L8253 is 150 W super-quiet xenon lamp with ozone-free bulb.

SPECIFICATIONS		
Parameter	L9566	L9588
Light Guide Port	Rear	Operation panel (front)
Lamp Replacement Position	Operation panel (front)	Rear
UV Intensity (Typ.) ^④	4500 mW/cm ² Typ. (at 365 nm, -01A type)	
Radiant Wavelength Range	Refer to the SELECTION GUIDE	
Lamp for Maintenance	Refer to the SELECTION GUIDE	
Lamp Service Life	Refer to the SELECTION GUIDE	
Power Supply Input	100 V ac to 240 V ac (100 V / 200 V auto switching), single phase 47 Hz to 63 Hz	
Power Consumption	280 VA Typ.	
Weight	Approx. 6.4 kg	
Operating Ambient Temperature	+5 °C to +35 °C	
Storage Ambient Temperature	-10 °C to +70 °C	
Operating and Storage Humidity	Less than 80 % (no condensation)	

NOTE: ④ UV Irradiance (at 365 nm) is measured in the center at a point 10 mm away from the output end of the A10014-35-0110 light guide (sold separately) with E5147-04, by using the Hamamatsu C6080-13 UV power meter.

Figure 4.65: Optical Stimulation Unit – External Technical Data

4.7.2 Installation

The components for Hamamatsu lamp come packed separately. Following is included:

- Lamp body
- Light bulb
- Light guide
- Mechanical adapter for light guide
- PC adapter cable
- Gender changer
- Set of mini Allen keys
- Power supply cable for Hamamatsu lamp
- Holder for sample positioning



Figure 4.66: Optical Stimulation Unit – External Parts

Setup

- Unpack the boxes.
- Mount the light bulb. Open the backside of the lamp body, remove the tape, open the counter nut, insert the light bulb (only touch with gloves!), fix the counter nut and close the cover.



Figure 4.67: Optical Stimulation Unit – External Setup

- Mount the light guide. Loosen the screw, remove it, and connect the input side of the light guide with the lamp body. Fix it with the screw.



Figure 4.68: Mounting the Light Guide

- Remove the front cap of the spectrometer using the tool from the spectrometer's delivery package. Insert from module E8000140 the metal ring only.



Figure 4.69: Removing the Front Cap of the Spectrometer

- Mount the mechanical adapter for the light guide. Normally this is done before delivery. If not, measure 40.5 mm like displayed in the picture and use the Allen key to fix the position of the adapter. Remove the cap of the light guide output side.



Figure 4.70: Mounting the Mechanical Adapter for the Light Guide

- Carefully insert the light guide including the adapter into module E8000140 and fix it with the screw.



Figure 4.71: Inserting the Light Guide into the Module

- Now connect the lamp with power supply and the PC with the ESRstudio installation. Therefore, use the gender changer and the RS232-USB-adapter cable. Switch the lamp on using the main plug and, in addition, push the **On** button on the right-hand side of the front display. The green LED shines after warming up and the lamp is ready for use.



Figure 4.72: Connecting the Lamp to the Power Supply and PC

4.7.3 Software Handling

- Install the drivers for the RS232-USB-Adapter. Normally Windows does this automatically, otherwise use the drivers from the enclosed CD. Afterwards, check the **Device Manager** in the **Control Panel** to get information about the used COM-Port.
- Start the ESRstudio and log in as Advanced. Switch to tab **System** and **Settings**. Search for the field **Optical unit**, select **HamamatsuLamp**, type in the COM-Port from the **Device Manager** and the maximum power (optional, specified in the data sheet). **Save** and **Initialize** the system.

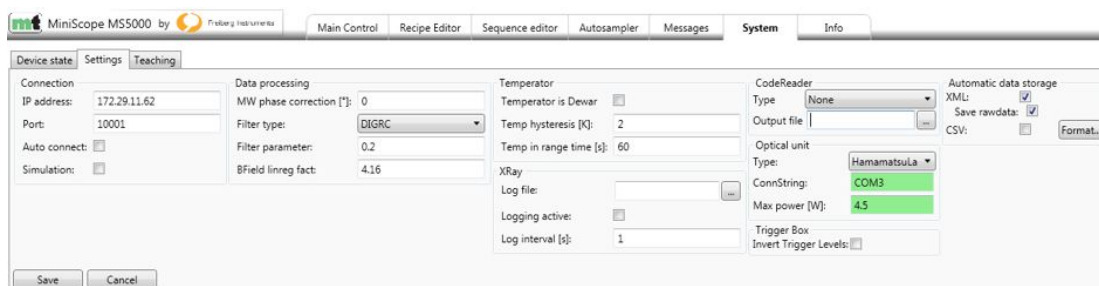
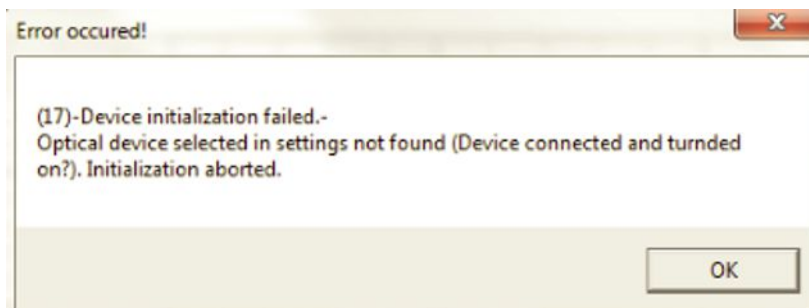


Figure 4.73: Light Guide Software Handling

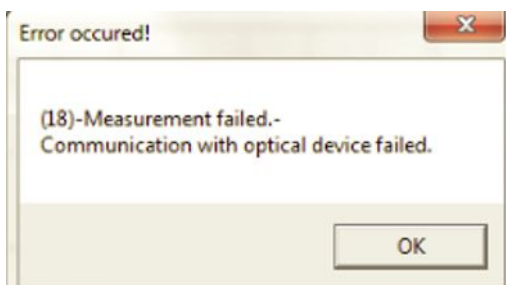


Remember to preheat the lamp.

- If the Hamamatsu lamp is selected in the settings, the spectrometer is only able to initialize if the lamp is powered. Otherwise an error message occurs. If you like to operate the spectrometer without Hamamatsu lamp, change settings to **BusDevice**, **Save** and **Initialize** or switch the lamp on and use recipes without **Optical sample irradiation**.



Do not switch off the lamp after successful initialization if a recipe with **Optical sample irradiation** is selected. Otherwise the system will stop before starting the measurement with following error message:



Recipe editor

Choose **Optical sample irradiation** in the list on the right-hand side and define the power (from 0 to 100%) as well as possibly a time shift for illumination start after measurement start (Lead Time) and the illumination time (Exposure Time).

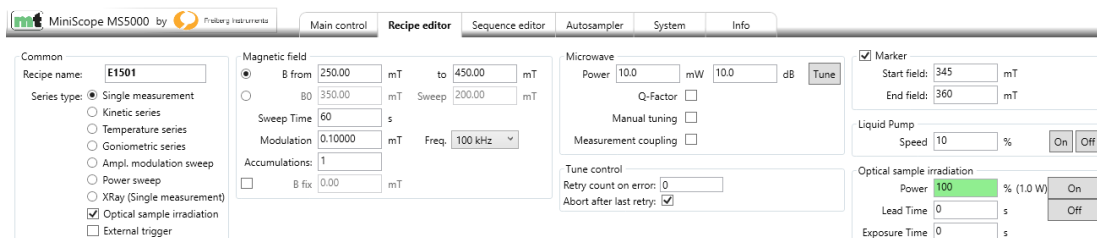


Figure 4.74: Optical Stimulation Unit Recipe Editor

For example, select a sweep time of 60 s, lead time of 10 s and exposure time of 20 s. The measurement will start and after 10 s the lamp will be switched on for 20 s.

The power selection is in %. If the maximum power is inserted in the settings tab, we will calculate the illumination power.



The displayed power is the optical power output of the lamp, not the power at sample position! This is not calculable because the modulation sticks are in the optical path, therefore the power at sample's position is strongly depending on the geometry of the sample tube.

For manual switching on or off the light source, use the **On** and **Off** button. Do not switch on the lamp while not mounted to avoid injuries.

Optical sample irradiation		
Power	100	% (1.0 W) On
Lead Time	0	s Off
Exposure Time	0	s

Keep an eye on the status of the lamp displayed in the menu at the bottom:

UV inactive

4.8 Continuous Flow System (Module E8000128)

The Continuous Flow System (E8000128) is made for measurements with liquids. There are 2 operation modes:

- Measure while the system continuously is pumping new material into the flat cell.
- Pumping a defined volume of the material into the flat cell and measuring after pumping has stopped.

The second mode is recommended since the continuous flow causes instabilities.

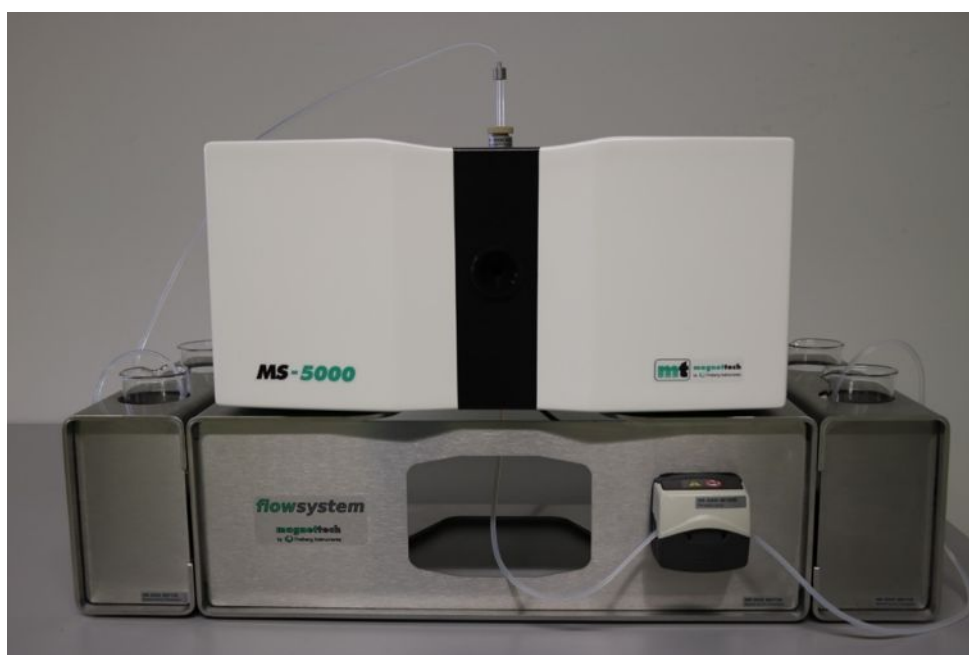


Figure 4.75: Flow System (Module E8000128)



Power off the system before plugging in and removing any of the cables. Otherwise, there is a risk of electrically damaging of the spectrometer's mainboard as well as the accessory unit itself.

4.8.1 Technical Specifications

Pumping velocity is selectable between 1 and 100%. Maximum throughput is 340 ml/min depending on tube diameter. We recommend using pump rates below 10% to avoid air bubbles inside the tube. For mode b, a volume input of 2 ml for normal tube lengths guarantees that the sample fully fills the flat cell cuvette. But this is not absolute but depends on tube length, inner diameter and used flat cell as well.

Keep in mind that the tubes are wear parts. Control the abrasion of the tube inside the peristaltic pump weekly or daily with continuous operation to avoid leakage.

The beakers at the pump's side are used for sample, the ones at the other side for "waste" after the measurement. The pump has a direction, so please do not mix the tube thoroughfare inside of the peristaltic pump. Connect the sample entry at the bottom side of the spectrometer and flat cell and the outlet on top to reduce air inside the flat cell cuvette. Regularly check the adapters for leakage

The Continuous Flow System features:

- Single channel flow system.
- Peristaltic pump (E8000124) offers constant flow or pulsed injection.
- Stable rack (E8000147) of massive steel including 4 glass beakers.
- Flat cell cuvette (E8000237) included, as well as SH-P holder (E8000308) and holder for flat cells (E8000235).
- Tube connector for flat cell cuvette and 15 meter flexible precision tube (E8000293).
- External irradiation unit (optional).

4.8.2 Installation

The delivery package of a Flow System E8000128 contains as follows:

- E8000147 Special rack for Flow System consisting of:
 - E8000124 Peristaltic pump
 - Central rack with electronic board
 - 2 side racks for sample beakers
 - 4 glass beakers with 100 ml volume each
 - Spectrometer bus cable (short)
- E8000124 (or E8000237) Flat cell (flow through)
- E8000308 SH-P directional sample holder
- E8000235 Fixed holder for flat cell cuvettes
- E8000293 Flexible precision tube (15 meter)
- 2 pieces of Tube-Flat-Cell-Adapter

The content usually is packed separately but you'll see everything in the picture and the installation description. All components are available as spare parts. The adapters usually come with a prepared connected sample tube in a recommended length as a benchmark.



Figure 4.76: Continuous Flow System Parts

Setup

- Unpack the contents.
- Place the mid part of the rack on a horizontal table. The total weight will exceed 50 kg. Mount both parts for storing the 4 beakers and insert them. Place the spectrometer on top, keep an eye on the fitting of the 4 feeds to the belonging deepening.



Figure 4.77: Placing the Rack on a Table

- Remove the original sample holder by loosening the 3 screws with the screwdriver of the delivery package of the spectrometer and insert the new SH-P holder (E8000308) including the fixed holder for flat cell cuvettes (E8000235).



Figure 4.78: Removing the Sample Holder

- Use the prepared 2 pieces of tube or cut 2 new ones. The first should reach from the beaker on the left through the peristaltic pump to the bottom of the flat cell, the second from top of the cell to the beakers on the left. Connect them with the adapters. Use the small, bronze colored at bottom and the bigger silver one on top side. Connecting the tube with the adapter needs some time and practice.



Figure 4.79: Preparing the Tube Pieces

- Mount the flat cell cuvette E8000236. Therefore, take the fixed holder for flat cell cuvettes (E8000235) and loosen the knurled screw on the top side. Carefully insert the flat cell from the bottom side. Adjust the flat in the same direction as the nose of the fixed holder. Leave the knurled screw loosened. The adjustment is done later. Insert the flat cell into the holder.

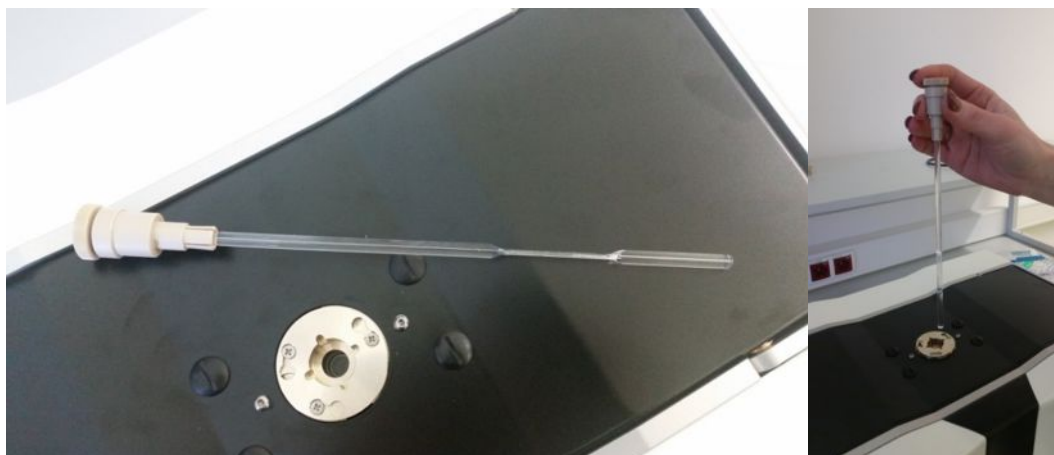


Figure 4.80: Mounting the Flat Cell Cuvette

- Insert the flat cell as deep as the bottom part pokes out of the spectrometer. Connect the longer precision tube with the bronze-colored adapter to the flat cell. You'll need some force. But be careful not to destroy the glass. Afterwards pull the flat cell carefully to the top and keep an eye on getting the adapter into the glass protection tube of the sample holder. Now connect the smaller tube with the other adapter on top side of the flat cell.

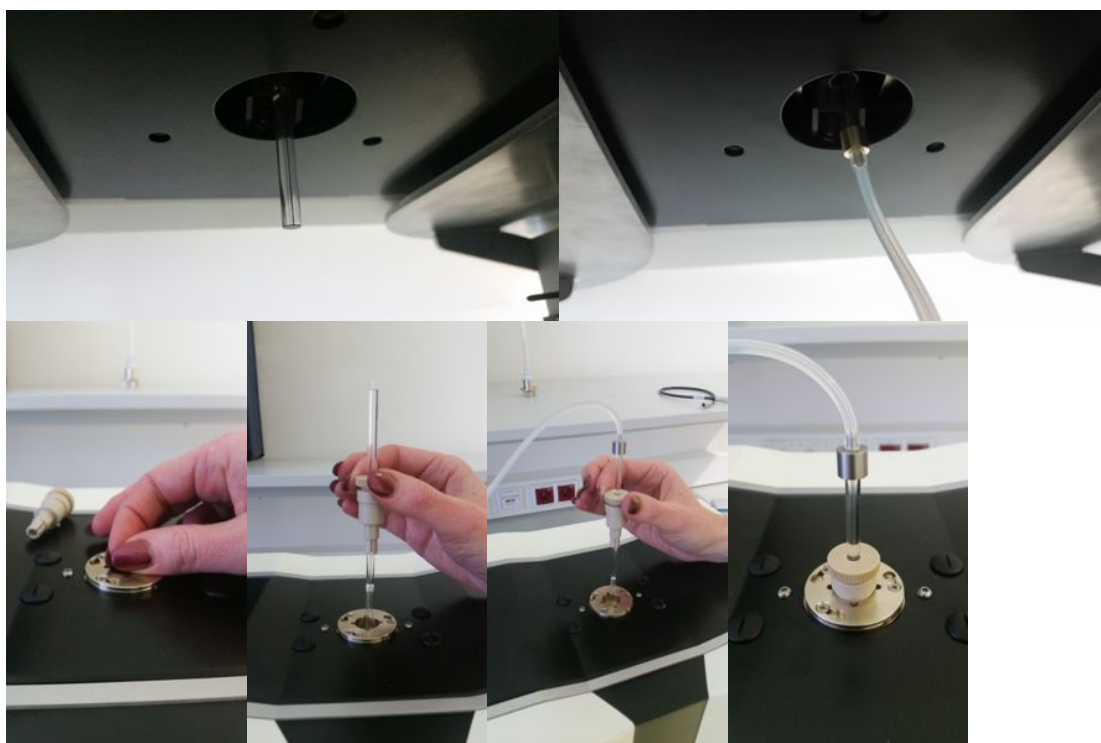


Figure 4.81: Inserting the Flat Cell

- Now adjustment the flat cell. For height adjustment use one of the dimensions from the following construction drawing. Also, check the parallelism of nose and flat again. Having finished adjustment, fix the knurled screw of the fixed holder. If you want to, remove the front insert of the spectrometer with the tool from the delivery package and check if the flat is in the right position. You may change orientation in 4 steps by 90° each to vary the position of the sample in the magnetic field. This will influence the measurement result.

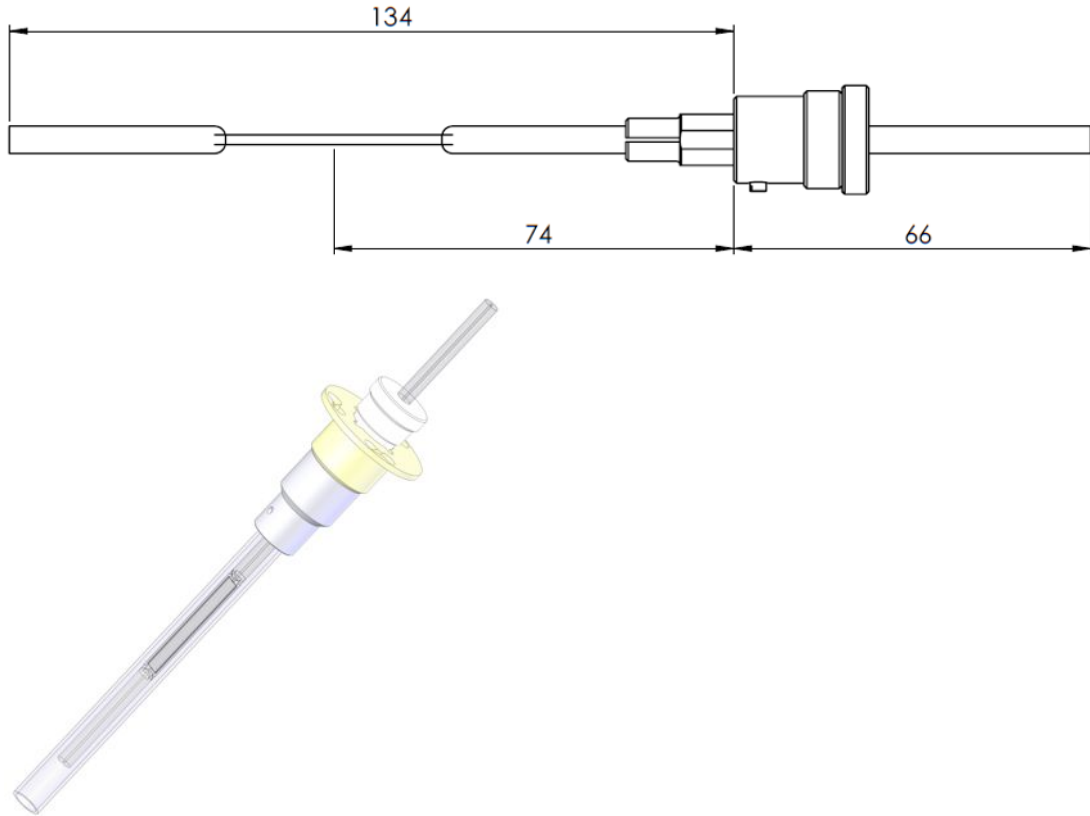


Figure 4.82: Adjusting the Flat Cell

- Connect the precision tube from the top with one of the beakers on the left-hand side. Use the hose clamps. Connect the bottom side with the peristaltic pump. Therefore, open the cover, carefully place the tube inside and close the cover. Always connect the pump output side with the flat cell cuvette. Use the hose clamps to adjust the tube at the beakers on the right. In general, liquid is pumped from the right-hand side beaker through the pump to the flat cell and wasted to the beaker at the left.

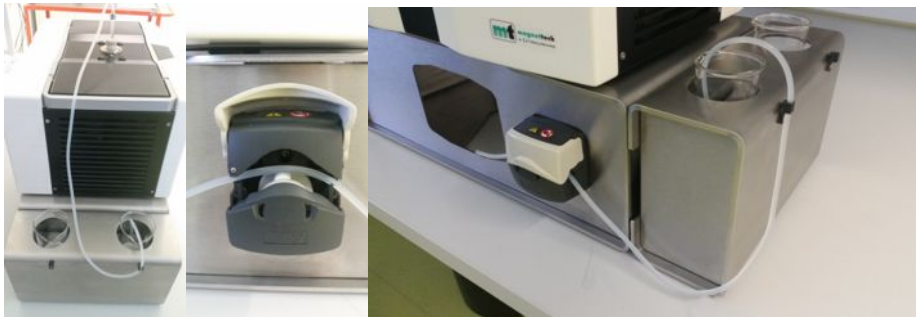


Figure 4.83: Connecting the Precision Tube

- Connect the electronic board in the back of the special rack E8000147 with the spectrometer. Use the bus cable (short). The plugs are equal, all will work. Connect the unit with power supply and Ethernet to the computer. Power the spectrometer, start the ESRstudio software and push the **Initialize**-button.



Figure 4.84: Connecting the Electronic Board

4.8.3 Software Handling

There are two possibilities to handle the Flow System. The easiest way is the recipe editor. The other way, for advanced users, is to switch to the sequence editor.

Recipe editor

After connecting with a Flow System, a new entry called **Liquid pump** is available. Choose a desired speed in percent and switch the pump **On** or **Off**.

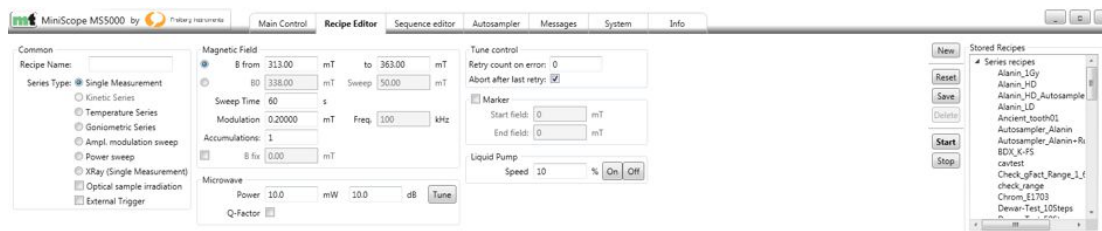


Figure 4.85: Continuous Flow System Recipe Editor

As mentioned in the operation hints, there are 2 possibilities to operate:

- Turn the pump on and measure in continuous flow (this may be unstable!) by starting the desired recipe. This mode is NOT recommended!
- Turn the pump on for a while until the sample is inside the flat cell cuvette (you may check the liquid flow out of the waste tube) and stop it afterwards. Now start your desired recipe.

Sequence editor

More comfortable but also more complex is working with the sequence editor. Three base steps are available:

LiquidPump.Enable Starts the pump with a desired speed in %.

le:

LiquidPump.Disable Ends pumping.

LiquidPump.Volume Use this to pump a defined volume. Select speed and desired volume. Speed less than 10% and volume of 2 ml are recommended to avoid bubbling and fill the whole flat cell cuvette with sample.

Step data			
Comment:			
Name	Value	Parent step parameter name	
Speed [%]	10		
Volumen [ml]	0.1		

Combine these base steps with others for starting measurements to fully automate your sample handling. Keep in mind that the beakers only contain a maximum volume of 100 ml. The flow system will not recognize whether the beaker is empty.

To clean your system after usage, pump air at maximum speed until no liquid leaves the waste tube anymore.

4.9 SH-P Holder for Flat Cell Cuvettes and Tissue Cells

The SH-P holder is made for mounting a variety of flat and tissue cells inside the spectrometer. In addition to the mechanical holder as itself (E8000308), different supports are available for different types of glassware. Most common is the fixed holder for flat cell cuvettes (E8000235).

An orientation of the sample is possible in 4 steps of 90° each. This will influence the measurement results.



Figure 4.86: SH-P Holder for Flat Cell Cuvettes and Tissue Cells

4.9.1 Installation

To change the sample holder:

Remove the 3 screws using the screwdriver from the delivery package of the spectrometer. Remove the original holder and replace it with the new SH-P holder (E8000308). Tighten the screws.

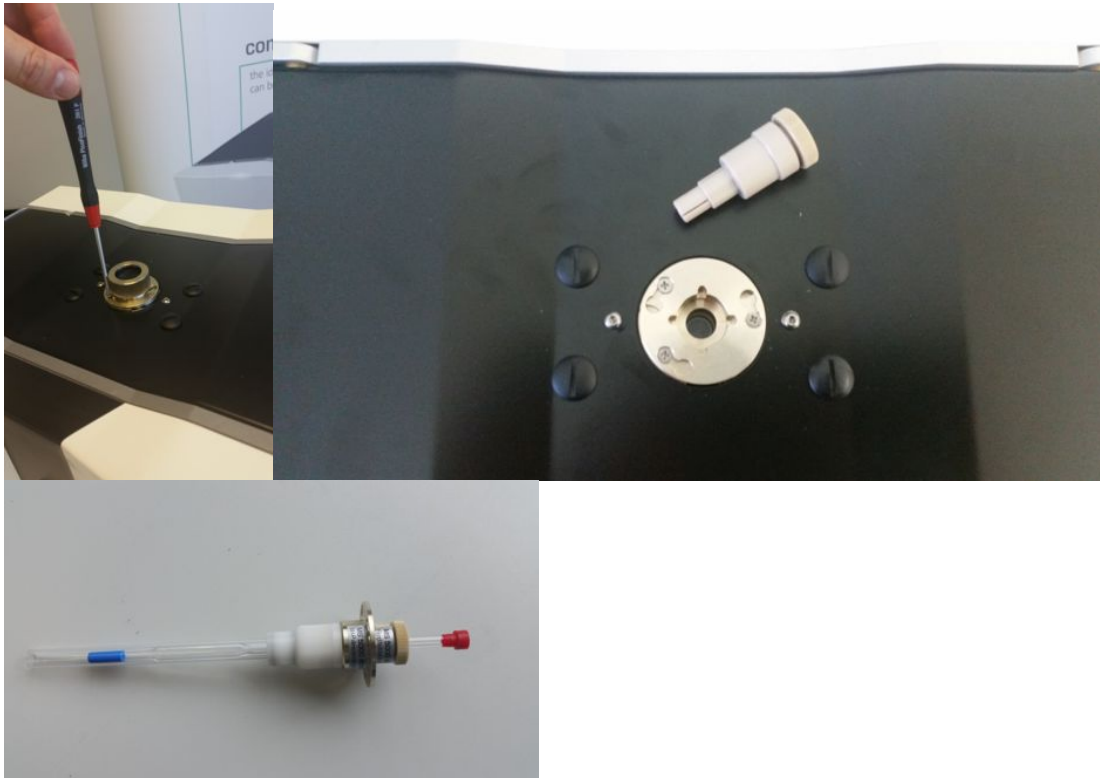


Figure 4.87: Sample Holder Installation

Mounting the glassware

To mount the glassware, loosen the counter nut of the fixed holder, insert the glassware from the bottom side, pay attention to the parallelism of flat and nose of the fixed holder, do the height adjustment after the drawings in the chapters below and fix the counter nut. Insert the fixed holder with the mounted glassware into the SH-P holder (E8000308) in the desired orientation.



Figure 4.88: Mounting the Glassware

4.9.2 Flat Cell Cuvette (Standard)

The Flat Cell Standard is designed for investigation of liquids. Advantage compared to work with capillaries or normal glass tubes is that due to the small sample volume coupling is more stable. Therefore, use the flat cell for substances which are not measurable with other sample setups.



Figure 4.89: Flat Cell Cuvette (Standard)

The Flat Cell Standard comes with a transport case and a set of 5 closing caps 4 (E8000229) and 5 mm (E8000230) each to close the top and bottom.

Setup

Mount the system as described in the pictures below. Use the dimensions of the drawings.

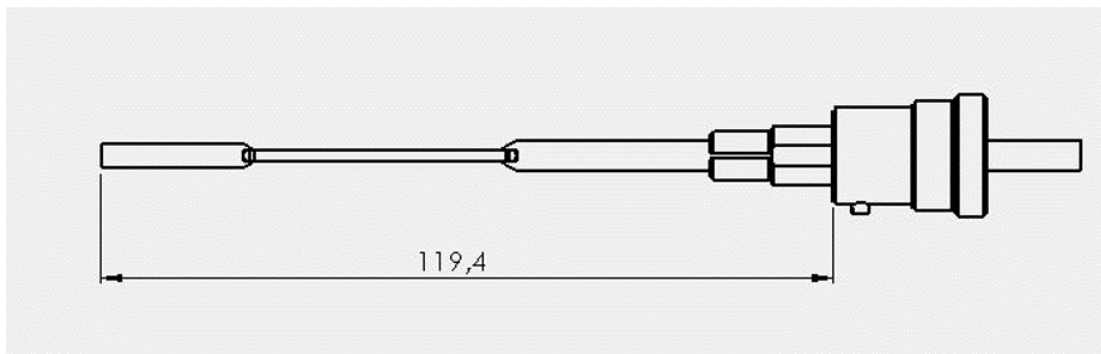




Figure 4.90: Flat Cell Cuvette Installation

4.9.3 Flat Cell Cuvette (Flow Through)

The flat cell Flow Through is specially designed for flow systems and measurements in flow through mode. Therefore, you may use our own designed Flow System E8000128 or connect the flat cell with your own precision tube and pump system.

For easy connection, we separately offer tube-flat-cell-adapters, precision tube (E8000293) or a peristaltic pump (E8000124).

The flat cell Flow Through comes in a transport case. The adapters in the picture are optional.



Figure 4.91: Flat Cell Cuvette (Flow through)

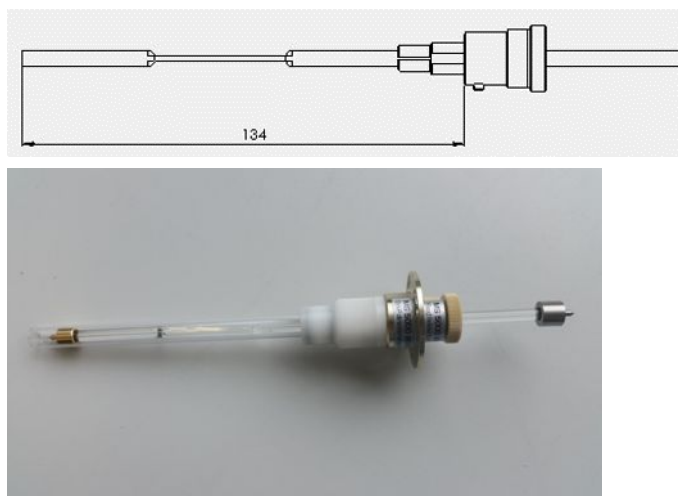
Setup

Figure 4.92: Flat Cell Cuvette (Flow through) Setup

4.9.4 Flat Cell Cuvette (FZKV 200-7x0.3)

The flat cell cuvette FZKV is made for higher sample volumes and is designed for standard process as well as for flow through application.

The flat cell cuvette FZKV comes with a transport case and a set of 2 caps made from glass (not in the picture).



Figure 4.93: Flat Cell Cuvette

Setup

Mount the system as shown in the picture below.



Figure 4.94: Flat Cell Cuvette Setup

4.9.5 Special Cuvette for Dosimetry Test Strips

The special cuvette FFZK is designed for investigation of dosimetry test strips mainly in the field of Alanine application.

The special cuvette FFZK comes with a transport case.



Figure 4.95: Special Cuvette for Dosimetry Test Strips

Setup

Mount it in the same way as the other cells. Keep an eye on positioning the strip in the center of the cavity.

4.9.6 Tissue Cell

The tissue cell is designed for handling of viscous samples. Place the sample in the sample well and use the cover slide and the clamps to fix it.

The tissue cell comes with a set of 2 clamps (E8000242) and a cover slide (E8000241).



Figure 4.96: Tissue Cell Cuvette

Setup

Mount the system as described in the pictures below. Use the dimensions of the drawings.

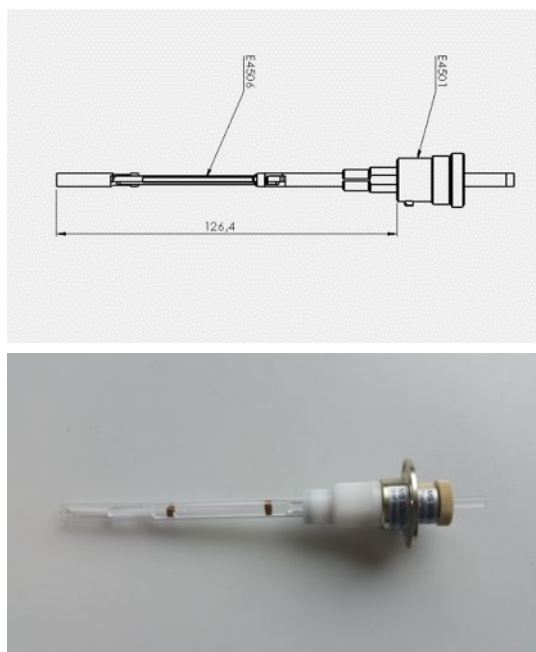


Figure 4.97: Tissue Cell Cuvette Setup

4.9.7 Software Handling

There is no special recipe or settings for working with SH-P holders and glassware. Use the standard recipes and procedures.

4.10 Liquid Nitrogen Dewar (Module E8000210)

The liquid nitrogen Dewar is the easiest way to perform low temperature measurements with the spectrometer system.



Figure 4.98: Liquid Nitrogen Dewar

4.10.1 Technical Specifications

Dewar and holder fit to the device to manually cool the samples.

Module E8000210: Dewars for spectrometer of the line Magnettech ESR5000

- Volume 50 ml
- Outer finger: Outer diameter 10.5 mm, length 178 mm
- Inner finger: Inner diameter 5.4 mm, length 105 mm
- Storage time 2 hours

Sample tube E8000306 & E8000307 are recommended for low-temperature applications.



Figure 4.99: Dewar for Manually Cooling the Samples

Module E8000205: Holder

The holder fits to the device with adjustable height for a fixed temperature Dewar suitable for Magnetech ESR5000 .



Figure 4.100: Dewar Holder

4.10.2 Installation

- Remove the sample holder and replace it with the holder for Dewar (E8000205).



Figure 4.101: Removing the Sample Holder

- Put a small amount of cotton wool at the bottom of the Dewar finger. This helps reducing the bubbling of the liquid nitrogen which disturbs the measurement.
- Put the Dewar carefully into the holder and use the thread for height adjustment of the sample.
- Remove the front cowl with the tool from the delivery accessories.

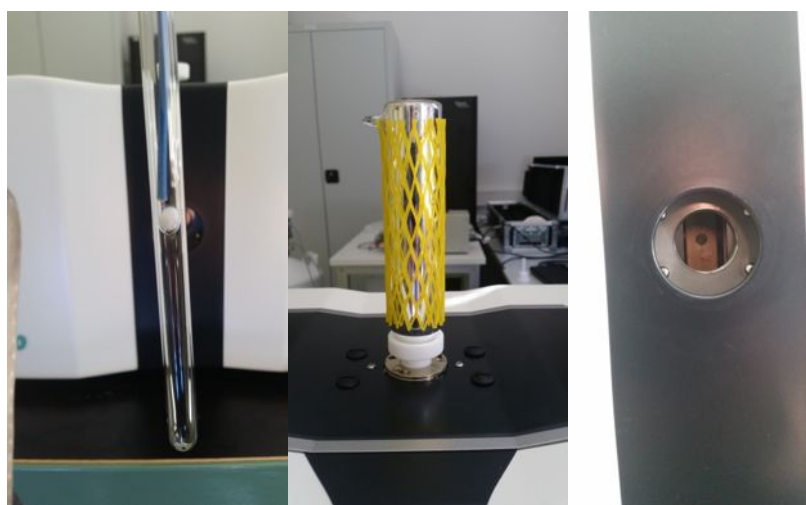


Figure 4.102: Inserting the Dewar

- Remove the Dewar and carefully fill in the liquid nitrogen.

Do not spill the nitrogen over the spectrometer. Do not fill in the nitrogen while the Dewar is mounted.



Figure 4.103: Filling the Liquid Nitrogen

4.10.3 Sample Handling

Introducing sample or nitrogen into the Dewar is related to the desired application and the sample material. The following methods are possible:

- Investigation of solid samples which have a high amount of water which results in tuning problems at room temperature, e.g. grains of wheat. Place them directly into the Dewar's finger and fill up with nitrogen to freeze the water and take the measurement.
- Some researchers directly put their liquid samples into the nitrogen with an injection. The sample will drop down through gravity.
- Use normal glass tubes or capillaries from the delivery package for liquid samples. They are temperature stable. Close capillaries on both sides to avoid direct contact between sample and nitrogen and use some cotton wool to close the top side of the normal glass tubes.
- For advanced and frequent users, we offer special glass tubes in 250 mm length in outer diameter 3 and 4 mm (Module E8000306 and E8000307). Through their length they are easily changeable even in a fully filled Dewar.

Module E8000306: Sample Tube P 250/3

- Length: 250 mm
- Outer diameter: 3 mm
- Wall thickness: 0.4-0.5 mm

Ideal for low temperature measurements, e.g. with Dewar E8000210.

Module E8000307: Sample Tube P 250/4

- Length: 250 mm
- Outer diameter: 4 mm
- Wall thickness: 0.4-0.6 mm

Ideal for low temperature measurements, e.g. with Dewar E8000210.

The nitrogen boils causing bubbling around the sample in the Dewar's finger. This may influence your measurement result as well as cause the sample to float. To avoid this, use an EPR inactive and nonmagnetic weight to hold the sample down.



Figure 4.104: Weighing Down the Sample

Module E8000287: Vessel for Flushing Resonator

To avoid problems with condensed water outside the Dewar's finger, which influences the stability and possibility of tuning, use the vessel for flushing the resonator.

Liquid nitrogen evaporates, flushing the resonator and preventing the condensation of water. This is an optional accessory for the fixed temperature Dewar.



Figure 4.105: Vessel for Flushing Resonator

4.10.4 Software Handling

Use the same recipes like without the nitrogen Dewar.

Before using the Dewar, please log in as **Advanced** and check on **Temperator is Dewar** in the settings file. This will lead to a better coupling between sample and microwave cavity. **Save** and **initialize** the system. Remove the checkmark after the measurements with the Dewar have finished to save the measurement time.

Figure 4.106: Liquid Nitrogen Dewar Software Settings

If you do quantifications, please remember to manually adjust the sample's temperature to 77 K (-196 °C).

Figure 4.107: Comparing Spin Results

4.11 Holder for Standards (Module E8000140)



Figure 4.108: Holder for Standards

4.11.1 Installation

- Remove the front cowl with the tool from the delivery package.
- Insert the metal housing and the plastic interior.
- Fix it with the screw.
- Carefully insert the standard sample.



Figure 4.109: Standards Holder Installation

The module E8000140 is useful for the following standard sample:

- Manganese (Mn^{2+} in ZnS)
- Chromium (Cr^{3+} in MgO)

Depending on which standard sample was purchased, the tube can have different marks for sample positioning:

- A mechanical stopper for usage with E8000140. Do not remove or shift it, this may lead to mechanical damage of the spectrometer. If the mechanical stopper is not attached to the tube of the standard sample, it is included in the holder for standard samples (E8000140) or the standard sample itself. If this is the case, attach the mechanical stopper with an Allen key to the tube of the standard sample, and make sure that the distance from the bottom of the tube to the beginning of the stopper is 142.75 mm (see figure "Inserting the Sample" below). It may not exceed this value, otherwise the modulation sticks can be damaged.

- A black ring on the glass as height adjustment marker for usage with the normal sample holder on top of the spectrometer. If no back marking is present, the standard sample should be positioned as outlined in chapter 6 of the user manual.



Figure 4.110: Manganese Standard (top) and Chromium Calibration Sample (center and bottom)

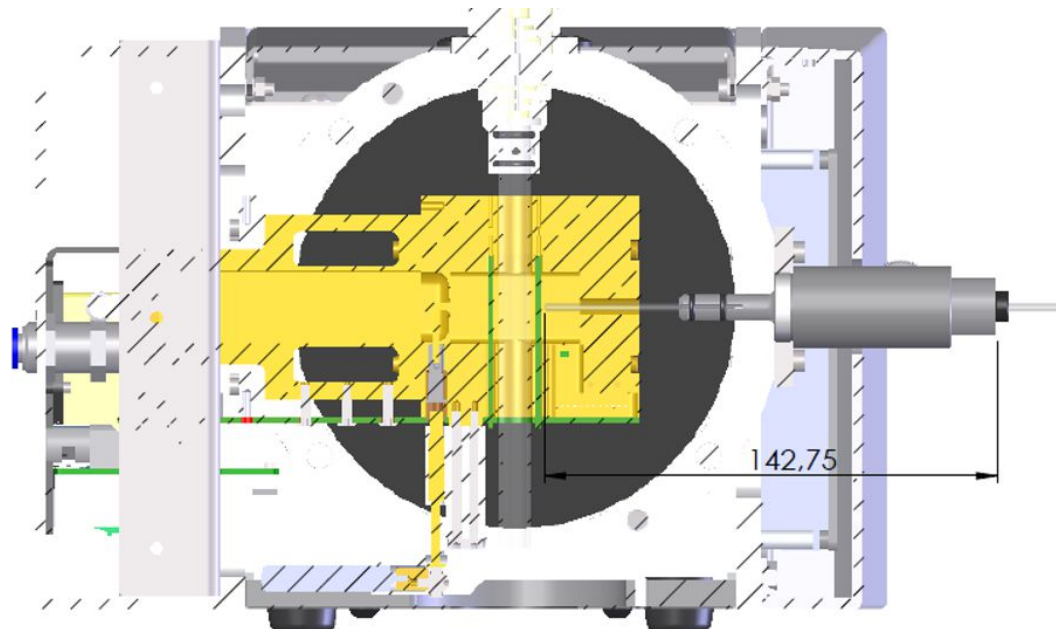


Figure 4.111: Inserting the Sample

4.11.2 Software Handling

The ESRstudio software comes with predefined recipes for Manganese and Chromium. Please keep in mind, that the signals of the samples measured in E8000140 will be 180° phase shifted and from extremely smaller amplitude compared with measurements in normal sample position. This is due to physics.

4.12 Integrated Reference Ruby Crystal (Module E8000141)



Figure 4.112: Integrated Reference Ruby Crystal

4.12.1 Installation

- Remove the front cowl with the tool from the delivery package.
- Insert the reference holder with the ruby crystal.
- Insert the outer 2 screws (marked in the picture) and rotate the inner part of the holder with the delivered socket wrench until the signal in the spectrum matches the desired position.



Figure 4.113: Reference Ruby Crystal Installation

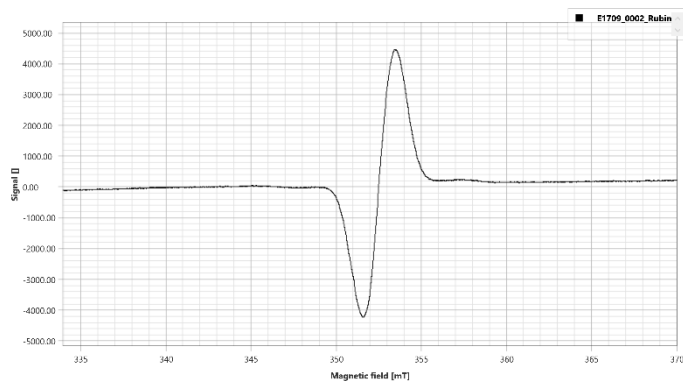


Figure 4.114: Signal in the Desired Position

- Tighten the 2 screws and mount the security cap. The inner part is easily removable for cleaning without changing the position.
- After installation of the ruby crystal use the delivered front cowl to prevent adjusting of the calibrated crystal. Push the front cowl inside the hole of the ESR5000 and tighten the screw in the middle.

4.12.2 Software Handling

In the recipe tab there is a window **Marker**. Choose a start and end of the magnetic field range where the software is searching for the ruby signal. Since the signal is reciprocal (due to position in the cavity), this is the only way to analyse the signal amplitude automatically.

☒ Marker

Start field: mT

End field: mT

Use the **Output file** function in the **Main control** tab for automated analysis of the ruby amplitude and position.

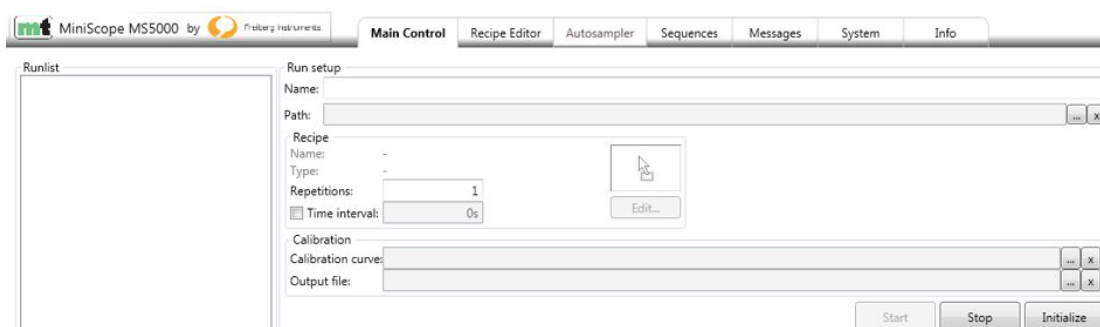


Figure 4.115: Main Control Output File

4.13 Trigger Box (Module E8000149)

The trigger box is intended for triggering external devices like illumination systems without delay times.



Figure 4.116: Trigger Box



Power off the system before plugging in and removing any of the cables. Otherwise, there is a risk of electrically damaging the spectrometer's mainboard as well as the accessory unit itself.

4.13.1 Installation

The Trigger box comes with following accessories:

- Trigger box
- MS5000 bus cable (short)



Figure 4.117: Trigger Box



The customer is required to provide their own BNC cable.

Setup

Switch off the ESR5000 and connect the trigger box to one of the 3 plugs at the backside of the ESR5000 using the ESR5000 bus cable (short). Connect your external device to the trigger box using the BNC cable. Power the spectrometer and initialize.

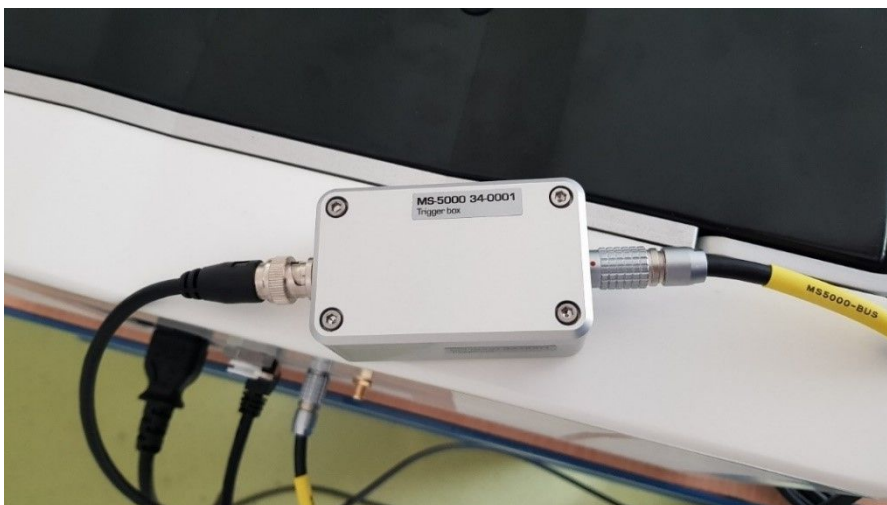


Figure 4.118: Trigger Box Connected

Working Principle

The trigger box sends an +3.3 V trigger signal to your device using open collector principle. The signal is invertible by the software. The normal state is “ground” and switches to **3.3 V** if the trigger is activated. Inversion means that normal state is **3.3 V** and switching to **ground** in case of activating the trigger.

4.13.2 Software Handling

Signal Inversion

Switch to tab **System** and **Settings** to invert the trigger signal, if required. Push **Save** and **initialize** to confirm changes.

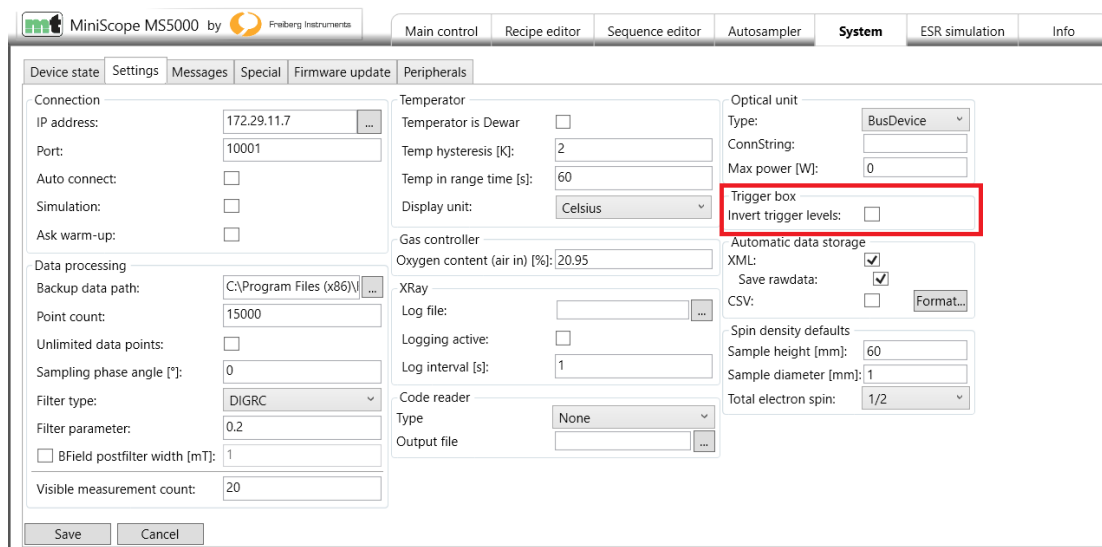


Figure 4.119: Trigger Box Signal Inversion

Recipe Editor

- Activate the **External trigger** in tab **Recipe editor**.

Figure 4.120: Recipe Editor External Trigger

- Select **Lead time** (delay time for trigger start after measurement start) and **On time** (time the trigger is active).
- Press **Save** and then **Start** to start the recipe.

4.14 Clime Reader (Module E8000292)

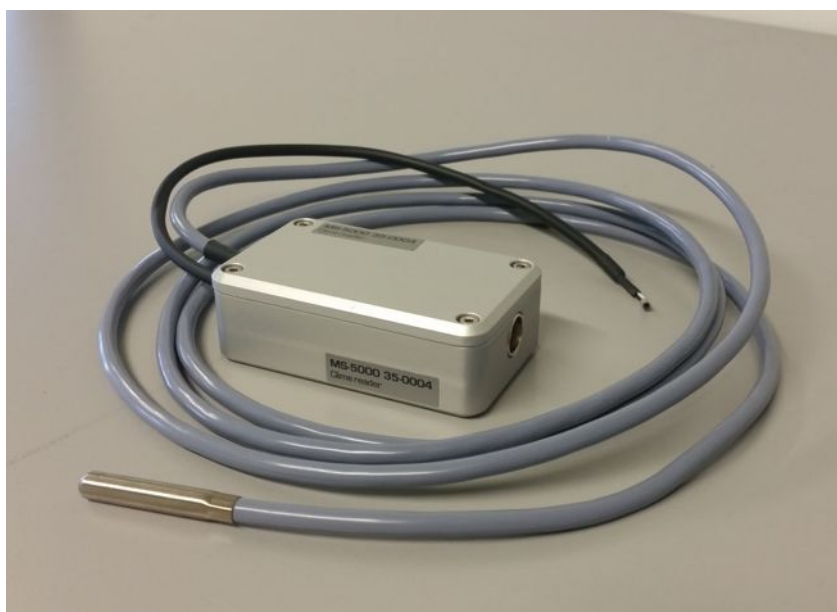


Figure 4.121: Clime Reader



Power off the system before plugging in and removing any of the cables. Otherwise, there is a risk of electrically damaging of the spectrometer's mainboard as well as the accessory unit itself.

4.14.1 Technical Specifications

The clime reader box is made for recording of environmental parameters. The data is directly saved in the raw data files. So, for example, a correlation between humidity and room temperature to EPR signal's amplitude is possible.

Following sensors are available:

- Humidity sensor
- Temperature sensor 1
- Temperature sensor 2

4.14.2 Installation

The Clime reader comes with following accessories:

- Clime reader box with 2 different temperature sensors (mounted with cables) and an internal humidity sensor.
- Spectrometer bus cable (short).

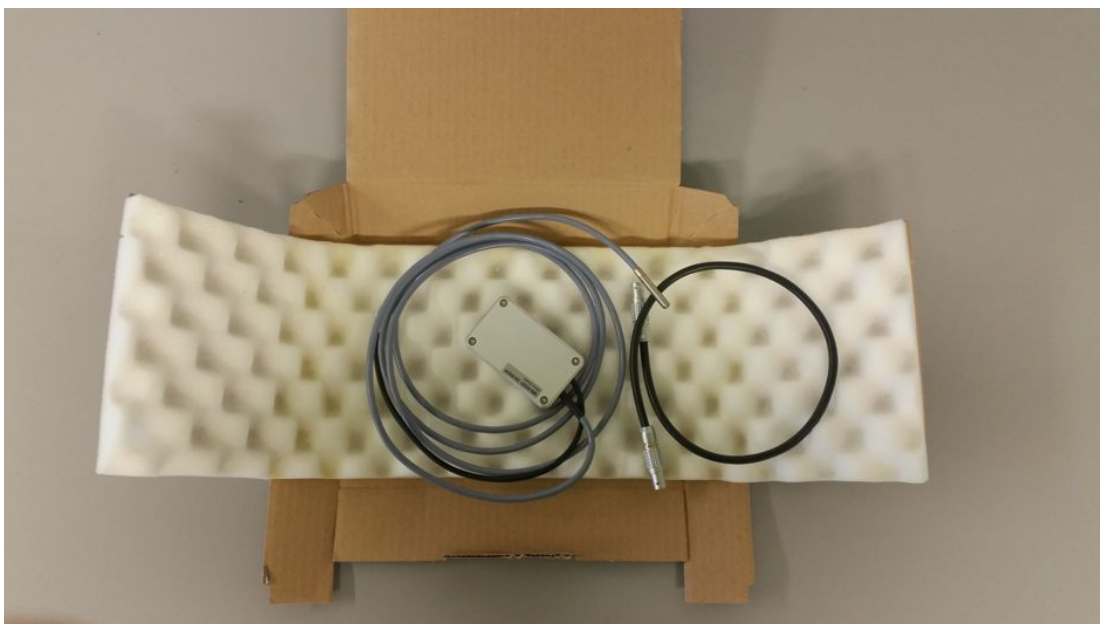


Figure 4.122: Clime Reader Parts

Setup

- Switch off the spectrometer and connect the clime reader box to one of the 3 plugs at the backside of the unit using the spectrometer bus cable.
- Power the spectrometer and initialize.

The humidity sensor is incorporated into the housing at the backside of the box. The clime reader has in addition 2 temperature sensors. For example, you may use one to record the room temperature and the other one to detect the temperature at the top side of the sample holder.



Figure 4.123: Connecting the Clime Reader Box

4.14.3 Software Handling

Record

At the beginning of a measurement, the current value of each sensor is automatically written into the raw data files. You may use the outdesc-file export as well.

Data-logger

The tab **System** and **Device state** offers the possibility to continuously record the values of each sensor to show trends. Choose the desired sensor and push the **Record**-button.

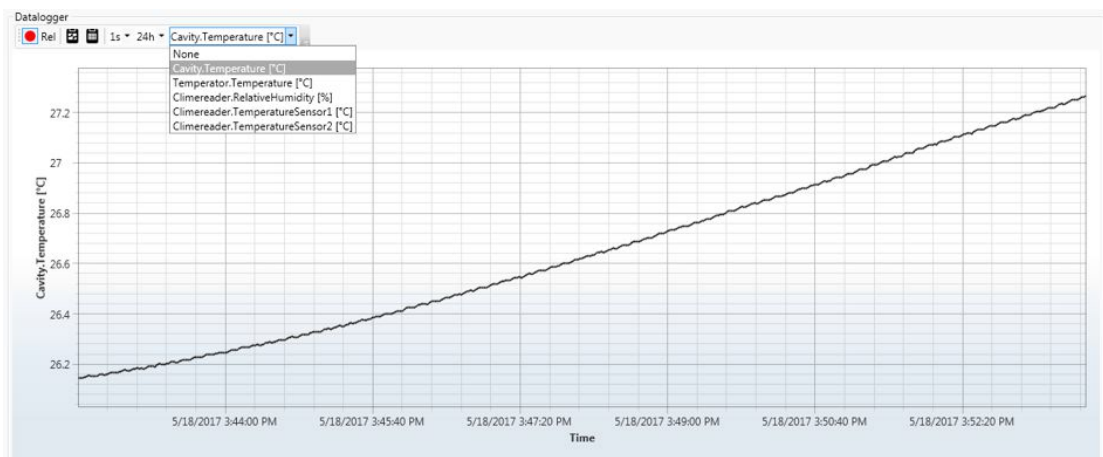


Figure 4.124: Data Logger

4.15 Special Rack for Horizontal Orientation



Figure 4.125: Special Rack for Horizontal Orientation

4.15.1 Installation

The special rack for horizontal orientation comes with a set of 4 screws and 4 new special pedestals.



Figure 4.126: Special Rack for Horizontal Orientation Parts

Setup

- Remove the original pedestals.
- Tip the system onto the side and loosen the 4 screws to remove the floor plate. Pay attention to the buried cable.
- Replace the original pedestals with the new ones, fix them with a wrench and mount the floor plate. Do not squeeze the cables.

Now position the rack and the spectrometer horizontal and place the spectrometer in the right position. The pedestals should match the slots. Due to the weight of the spectrometer, ideally do this with another person. Now fix it with the 4 screws. The easiest way is to position the rack over an edge like displayed in the picture.



Figure 4.127: Positioning the Rack and Spectrometer

After fixing of all screws, tip over the rack to the backside and connect the cables from the back.

4.15.2 Application

Intended application is work with tissue. The special rack helps to mount samples in a horizontal way. An SH-P holder (Module E8000308 and E8000235) and a tissue cell (Module E8000239 or E8000240) are required.

4.16 Standard Samples

We offer a wide range of different types of standard samples for various applications. Keep in mind that they all are intended for a special aim.

4.16.1 Manganese Standard (currently unavailable)

Manganese (Mn^{2+} in ZnS) is designed as field standard to control and calibrate the magnetic field axis. It is a powder coming in a capillary in a 3 mm sample tube and with a certificate containing information about the magnetic field position and distances between each of the 6 main peaks. It is applicable with the holder and positioning system for standards (Module E8000140) to mount it from the front side of the spectrometer.

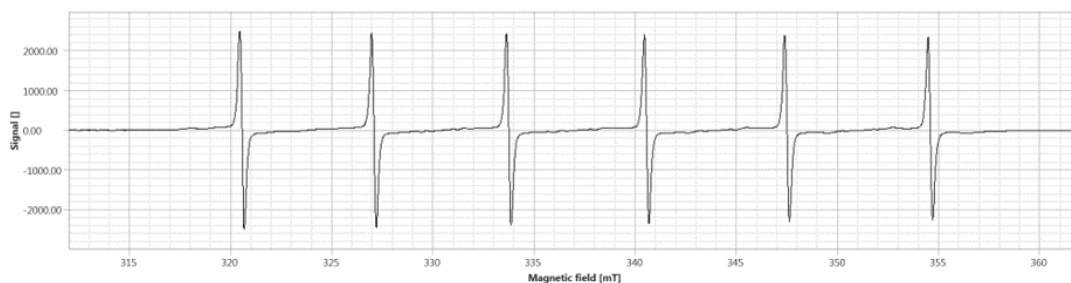


Figure 4.128: Manganese Standard Sample

4.16.2 Chromium Standard (Module E3005336)

Chromium (Cr^{3+} in MgO – Module E3005336) is also designed as a magnetic field standard. The set-up of the sample tube is the same as for the Manganese sample, but in contrast to Manganese, it only shows one main peak at $g = 1.98$. Due to usage of 3 mm sample tube, it matches with Module E8000140.

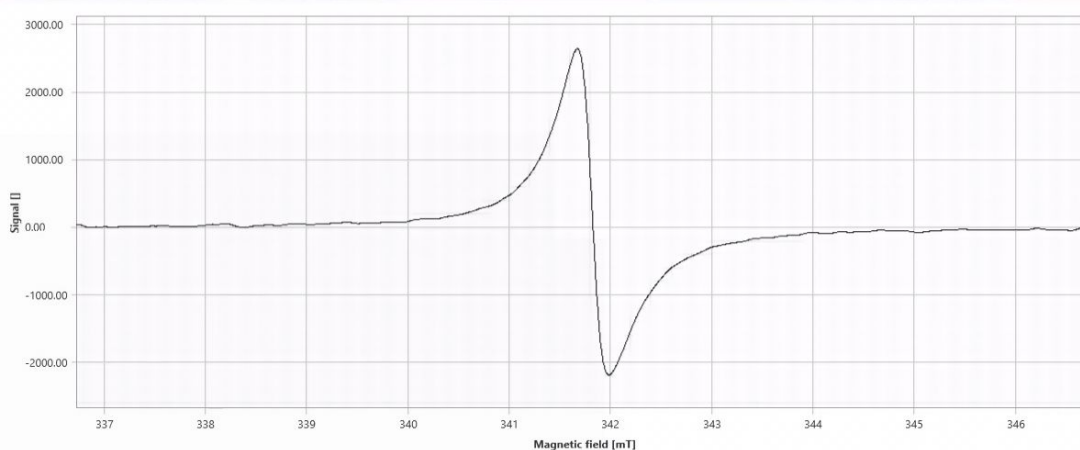


Figure 4.129: Chromium Standard Sample

4.16.3 Ruby Standard (Module E8000141)

Ruby (Al_2O_3) is a single crystal whose magnetic field position is strongly angular dependent. We offer the ruby in mounted on a rotatable front insert, therefore the user may freely select its field position which helps them not to overlie the reference spectrum with the spectrum of the analyzing material. Read more about module E8000141 in [Integrated Reference Ruby Crystal \(Module E8000141\)](#) [79] and about e.g. the Alanine application in Concentration Determination.

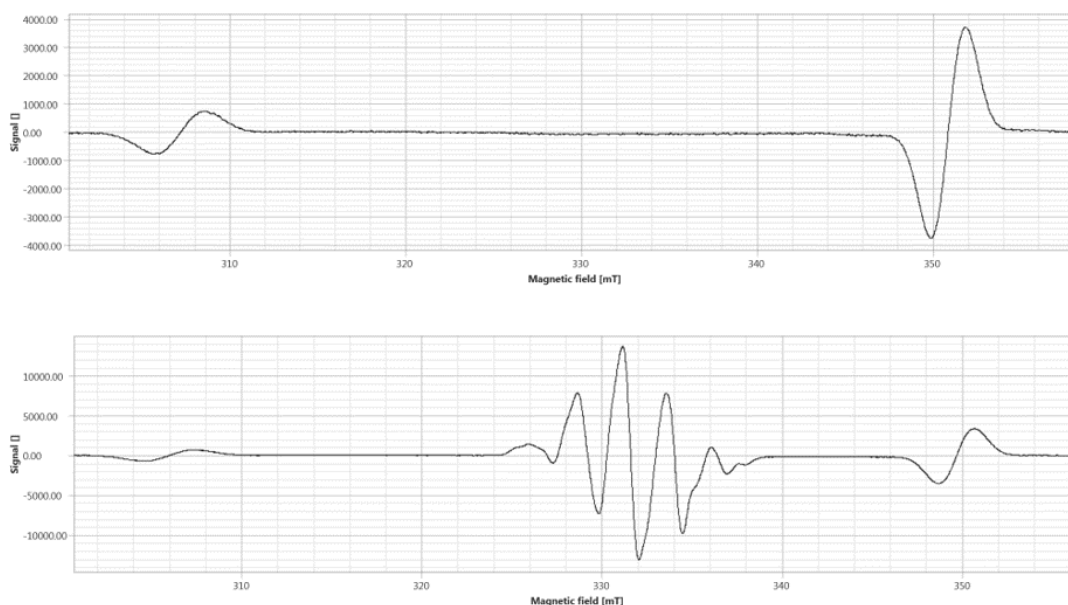


Figure 4.130: Ruby Standard Sample

4.16.4 Alanine Spin Calibration Sample (E3005324)

Irradiated alanine (E3005324) is a suitable reference for relative quantification and recalibration of SpinCount. It is calibrated at the factory and labeled with the number of spins present in the sample.

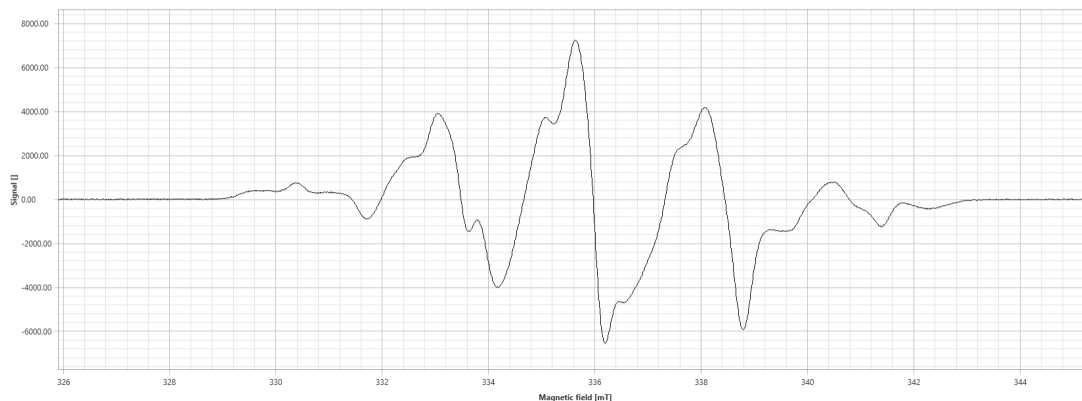


Figure 4.131: Alanine Spin Calibration Sample

4.17 Sample Tubes

We offer a wide range of different sample tubes with outer diameter of 3 to 6 mm and length of 165 mm. In addition, for usage inside the liquid nitrogen Dewar (module E8000210) the catalogue contains sample tubes in 250 mm length (module E8000306-E8000307). Find more information in our product catalogue.

The raw material is synthetic quartz glass. All tubes come with a defined length and wall thickness. The table below shows the typical specifications:

Module	Length [mm]	Outer Diameter [mm]	Wall Thickness [mm]	Typical Inner Diameter [mm]
E8000300	165	3	0.4 ± 0.1	2.2 ± 0.2
E8000301	165	4	0.7 ± 0.1	2.6 ± 0.2
E8000302	165	5	0.7 ± 0.1	3.6 ± 0.2
E8000303	165	6	0.5 ± 0.1	5.0 ± 0.2

Table 4.12: Typical Sample Tube Specifications

4.18 Analog Signal Output Module (Module E8000105)

The Analog Signal Output module is used with external equipment to perform transient measurements.



Figure 4.132: Analog Signal Output Module

4.18.1 Technical Specifications

The Analog Signal Output module for external equipment, used to perform transient measurements, is intended for directly detecting the raw signal (I channel) using an oscilloscope. It is e.g. useful for rapid passage measurements. The module offers an additional connector at the backside of the equipment.



Figure 4.133: Connectors for Analog Signal Output

4.18.2 Installation

- Connect the Analog Signal Output to an oscilloscope using a BNC cable.



Figure 4.134: Connecting the Analog Signal Output to an Oscilloscope

- Use the oscilloscope to detect the signals.

4.19 Dielectric Resonator Ring (Module E8000157)

The Dielectric Resonator Ring provides the ability to detect very low radiation doses with alanine dosimeters.

With an inner diameter of 5 mm it is compatible with Alanine sample tubes with outer diameters smaller than 5 mm. The dielectric resonator ring is compatible with alanine sample tube E8000168.

4.19.1 Installation

- Remove the standard sample holder by loosening the counter nut and removing the three red marked Phillips screws.
- Turn the sample holder to the right and lift it up.

- Remove the black front cover by turning it to the left to get access to the measurement chamber. The center position is visible through the little hole in the third figure below.

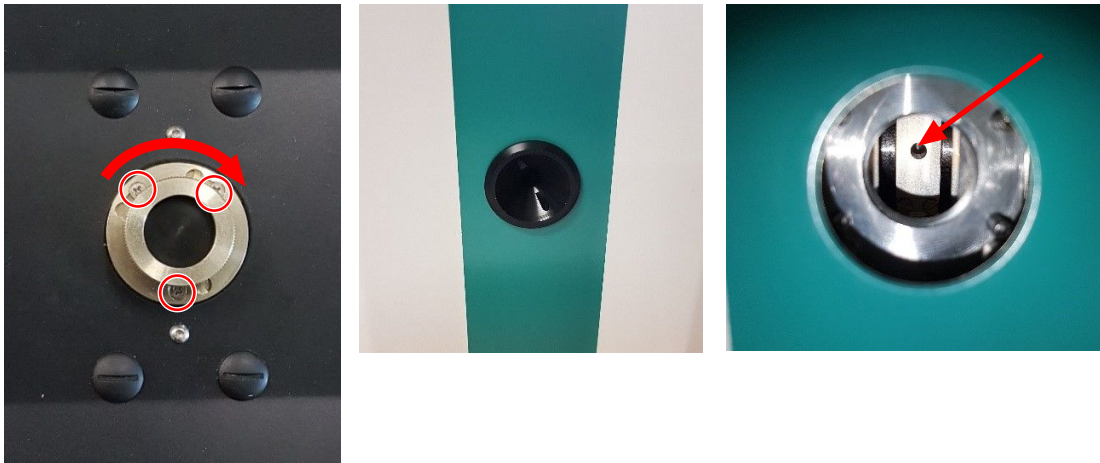


Figure 4.135: Removing the Standard Sample Holder

- Insert the special holder for Alanine pellets (E8000166).
- Turn it to the left and tighten the three screws.
- Remove the counter nut and the sample holder and put the dielectric resonator ring (E8000157) into the holder.
- Check that the ring lies on it's side and centered in the cavity using the little hole in the front.

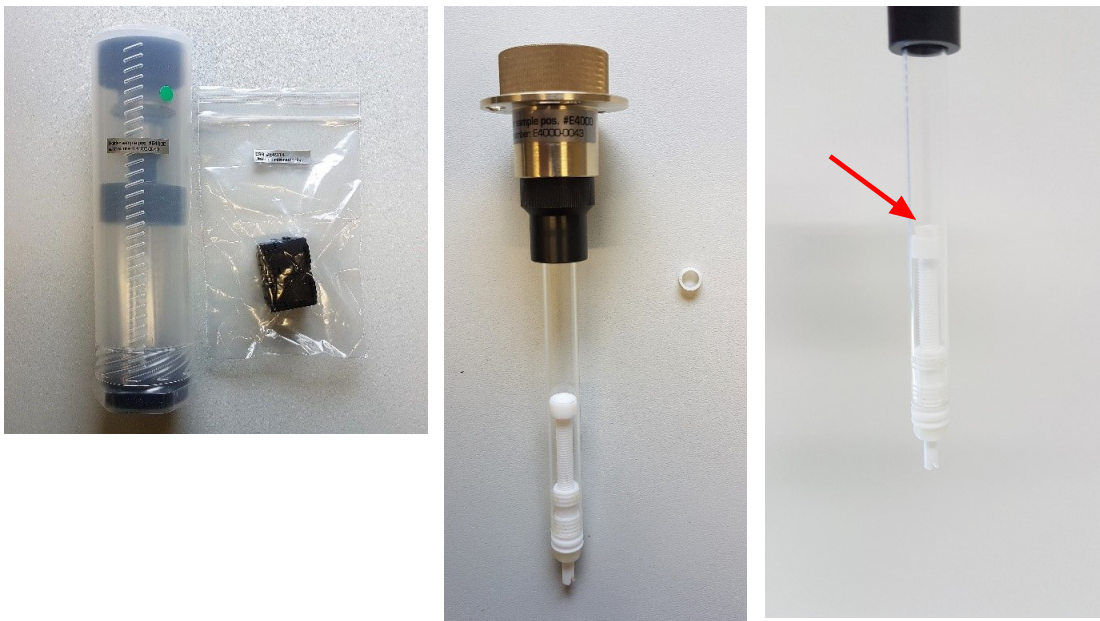


Figure 4.136: Inserting the Special Holder for Alanine Pellets

- Be careful while mounting the sample. The ring must enclose the guidance tube and also your sample.

4.19.2 Effects of the Dielectric Resonator Ring

The signal of this 10 Gy alanine sample is nearly doubled while using the dielectric resonator ring.

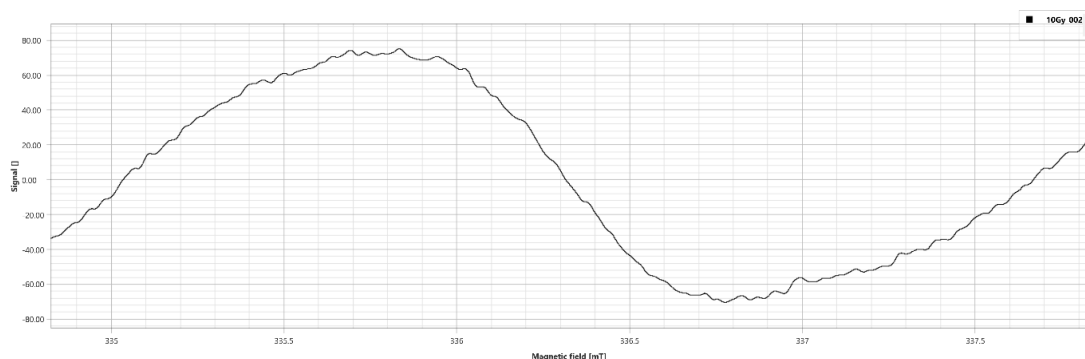


Figure 4.137: 10 Gy Alanine Pellet Spectrum without Dielectric Resonator Ring

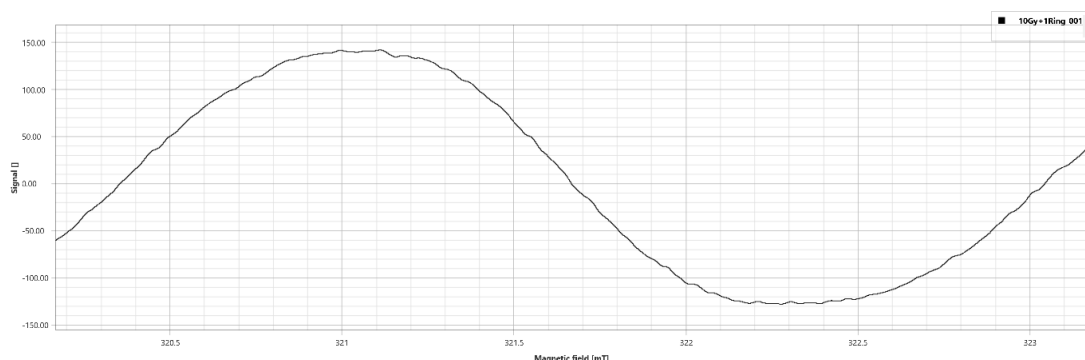


Figure 4.138: 10 Gy Alanine Pellet Spectrum with a Dielectric Resonator Ring

4.20 High Temperature Controller (Module E8000116)

The High Temperature Controller (HTC) is intended for heating samples up to 450 °C using a gas flow. The gas can be dry nitrogen gas or dry/oil-free compressed air from the home network or pressurized gas cylinders. Nitrogen gas is advisable. In case of compressed air sufficient filtering must be ensured to avoid any contamination that may result in unwanted background signals.

The specified temperature ranges apply for the temperature at the sample position centered inside the cavity.

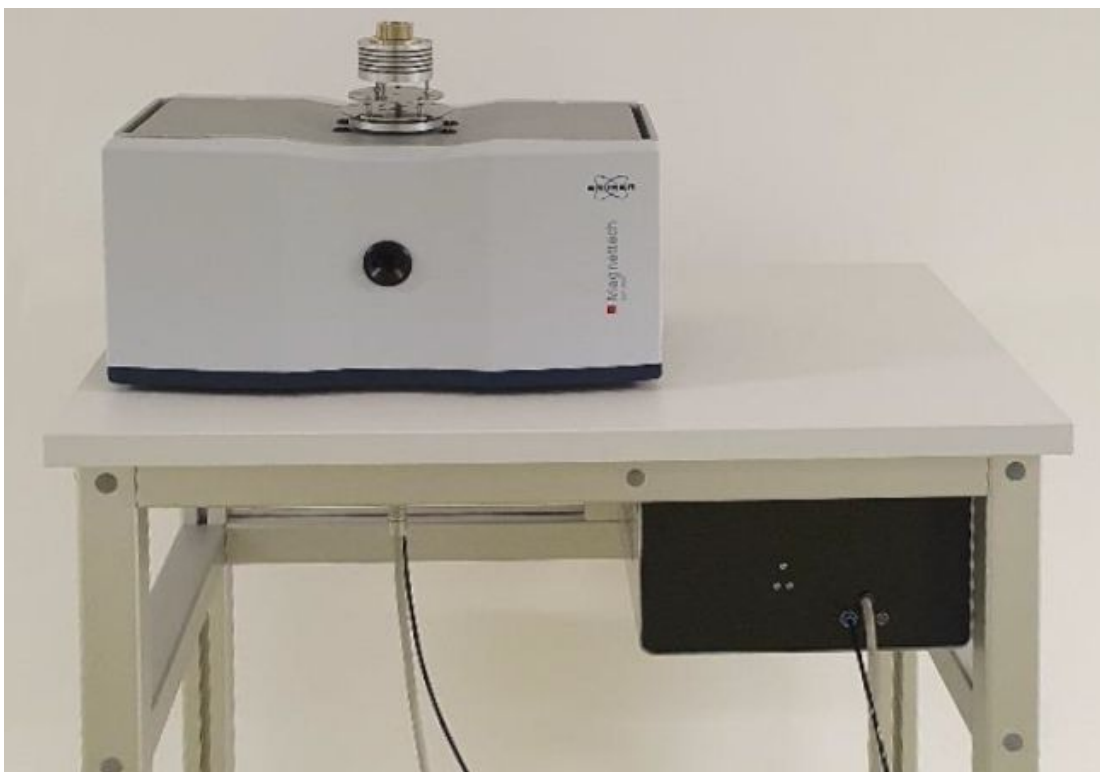


Figure 4.139: The ESR5000 and High Temperature Controller



Power off the system before plugging in and removing any of the cables. Otherwise, there is a risk of electrically damaging the spectrometer's mainboard as well as the accessory unit itself.

4.20.1 Technical Specifications

General Information

Data	Value	Unit
Length	450	mm
Width	700	mm
Height	810	mm
Weight	17	kg

Table 4.13: Dimensions: Table

Data	Value	Unit
Length	192	mm
Width	230	mm
Height	133	mm
Weight	6.8	kg

Table 4.14: Dimensions: E-Box

The specified temperature ranges apply for the temperature at the sample position centered inside the cavity.

Set-up specified for:

- Sample tube P 165 / 3 - E8000300

Temperature at Sample Position

Data	Value	Unit
Minimum Temperature	50	°C
Maximum Temperature	450	°C
Temperature Stability	± 5	K
Accuracy of temperature at sample position	± 5	K

Table 4.15: Temperature at Sample Position

Control Mode

Data	Value
Overshooting of the target temperature	Maximum ± 25% of change/minimum 20 K
Maximum of transient effects	3 cycles of oscillations

Table 4.16: Control Mode

Operation Mode

Sample Heating Mode is the main mode for using the High Temperature Controller. In that mode the high temperature controller is used to heat an inert gas like dry nitrogen or dry/oil-free compressed air. The gas flow is then used to heat up a sample up to 450 °C.

Furnace Gas Mode is a special operation mode to analyze hot gas coming directly from a furnace. For that mode the high-temperature furnace gas is directly fed to the resonator Dewar using a ½" (DN15) connection thread.

4.20.2 Installation

The HTC (module E8000116) comes with following accessories packed in a custom-fit transport case:

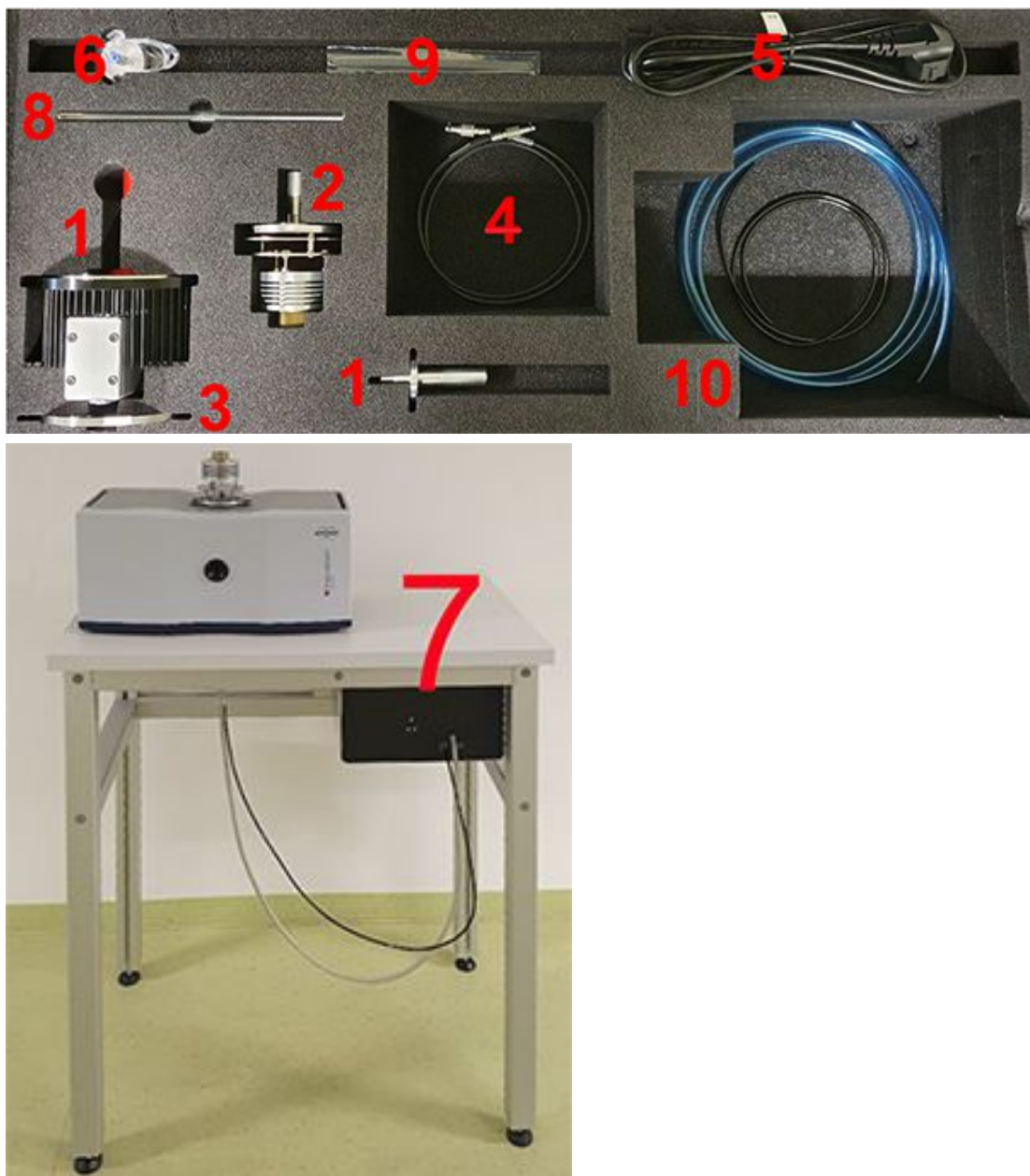


Figure 4.140: High Temperature Controller Accessories

1	Heating Unit with aluminum pipe for centering for resonator Dewar	6	Pressurized Gas Adapter (4 mm diameter)
2	Heat Dissipation Unit	7	Table with preinstalled Electronic Box
3	Mounting Plate	8	Resonator Dewar
4	ESR5000 Bus Cable	9	P165/3 Sample Tube
5	Power Supply Cable	10	Tubes (4 mm and 8 mm outer diameter)

Setup

- Place the ESR5000 on the table and align it above the hole. Use the hex wrenches and the delivered screws to adjust the mounting plate [3] through the hole on the bottom. The mounting plate should be centered on the hole.



Figure 4.141: Centering the Mounting Plate on the Hole of the Table

- Connect the electronic box and the spectrometer with the ESR5000 bus cable [4] and plug in the power supply cable of the ESR5000 device and the electronic box [5].

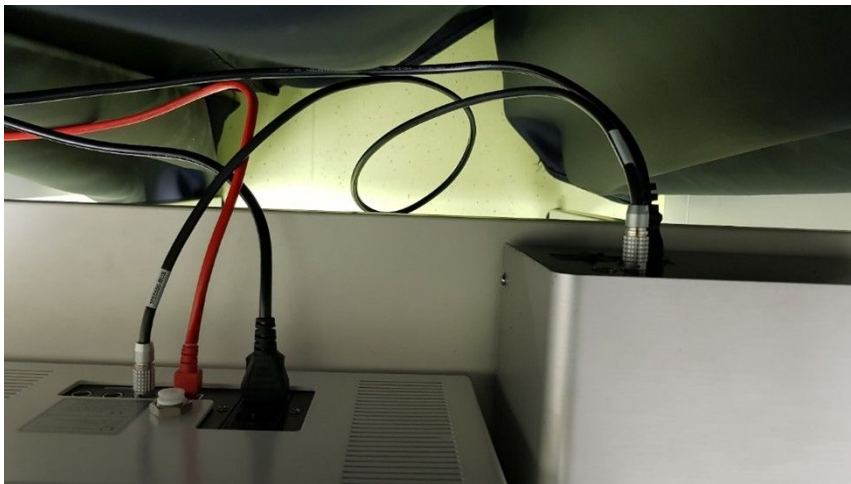


Figure 4.142: Electronic Box Connections

- Prepare the heating unit [1] by removing the red transport lock. Be careful with the integrated temperature sensor. Mount the resonator Dewar [8] into the designed hole around the temperature sensor. The glass has a mark on one of the two sides. This comes from the glass blowing. Please insert this side first into the hole of the heating unit. An O-ring inside the heating unit keeps the resonator Dewar in position. Put the delivered aluminum pipe [1] exactly at the position where the transport lock was mounted and use screws to fix it.

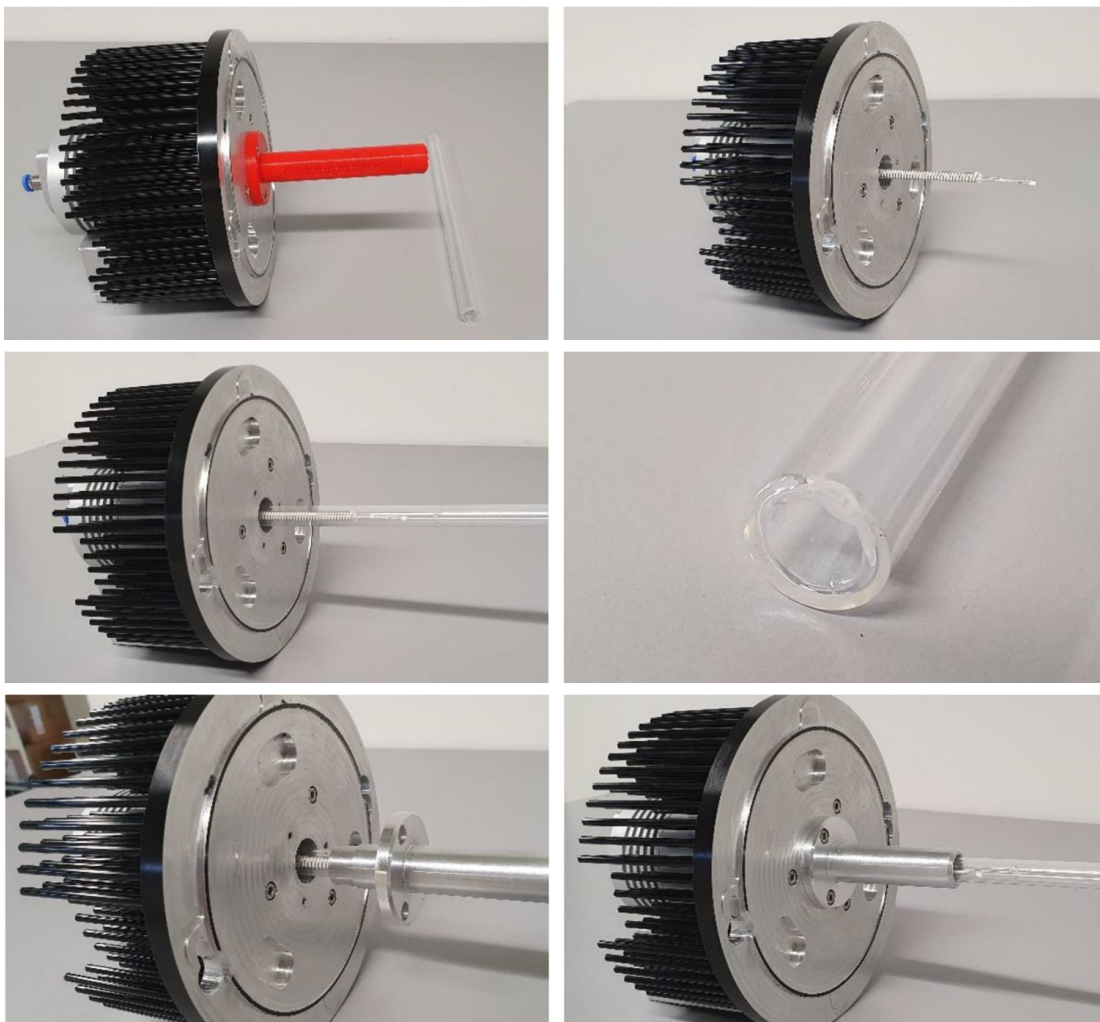


Figure 4.143: Mounting the Heating Unit

- Remove the original ESR5000 sample holder.



Figure 4.144: Removing the ESR5000 Sample Holder

- Carefully insert the heating unit with the resonator Dewar from the bottom side into the ESR5000 system and attach it with the 2 screws (figure below, left and middle). The easiest way is lay on the floor to mount the unit. Mount the adapter for 4 mm tubes on the bottom of the heating unit (figure below right).



To prevent breaking the resonator Dewar, do not tilt the heating unit while mounting it.

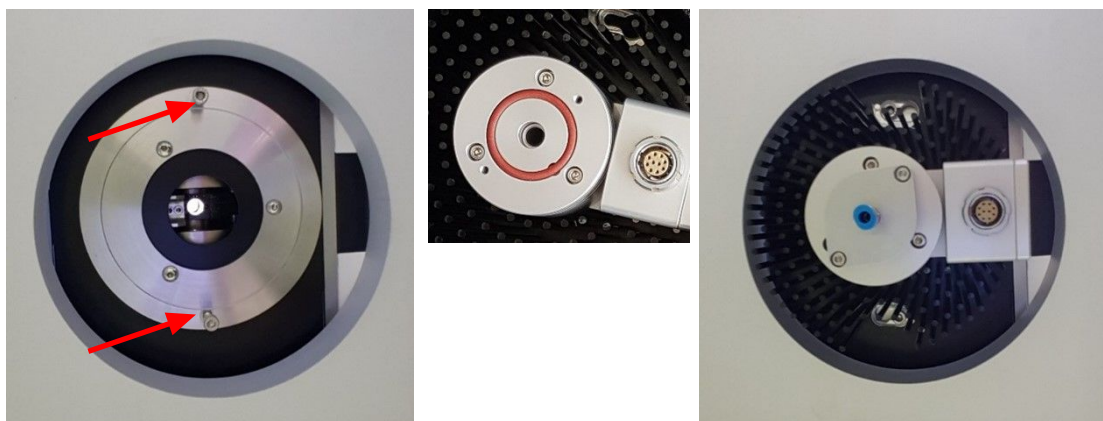


Figure 4.145: Inserting the Heating Unit into the ESR5000 System

- Connect the air pressure tube and the data cable from the electronic box to the heating unit. The flow rate of the pressured air is adjusted to 6 liter/minute. This value should not be changed, otherwise the displayed temperature in the software will differ from the real sample temperature.



Figure 4.146: Connecting the Pressurized Gas Tube and the Data Cable

- Before mounting the Heat Dissipation Unit [2] you must first step by step disassemble the 2 parts. Be careful not to lose the required screws.



Figure 4.147: Heat Dissipation Unit



Figure 4.148: Disassembled Heat Dissipation Unit

- Afterwards mount the bottom plate to the ESR5000 using the normal 3 holder screws and assemble the Heat Dissipation Unit by executing the steps of disassembling in the reversed sequence.



Figure 4.149: Mounting the Bottom Plate

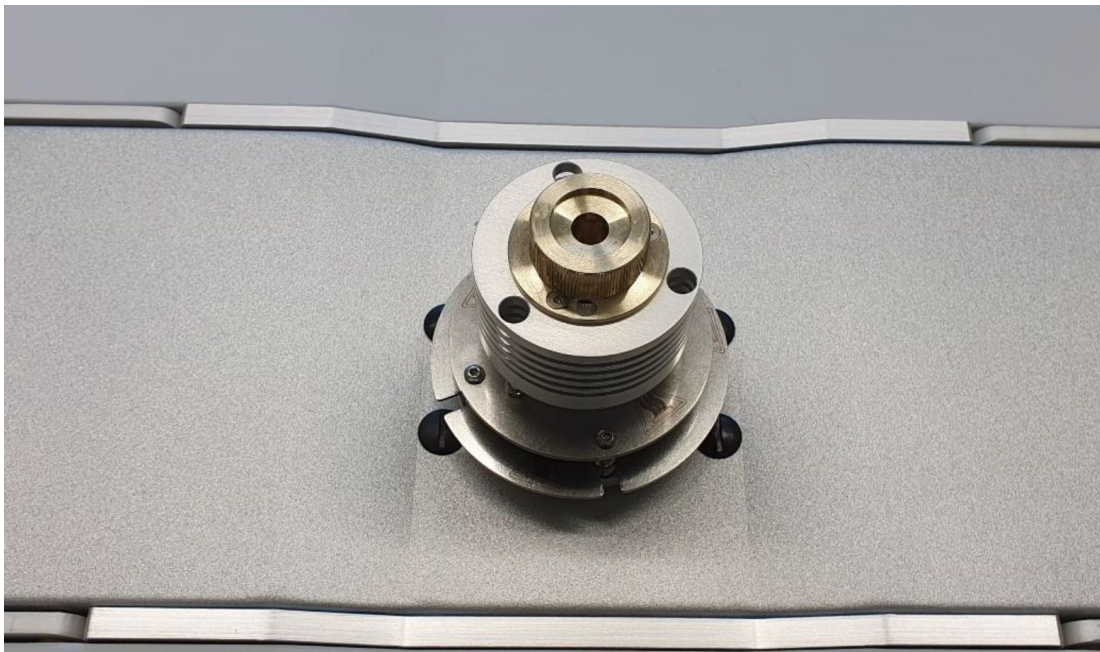


Figure 4.150: Heat Dissipation Unit After Being Properly Installed

- Remove the counter nut to mount the delivered 3 mm sample tube. Insert the tube into the normal standard holder for 3 mm tubes (E8000212). Height adjustment is analog to adjustment of normal samples inside the spectrometer. Pay attention on the three bumps inside the resonator Dewar while inserting the sample tube. Do not apply too much force when meeting a resistance while inserting the tube, otherwise the resonator Dewar can be damaged.

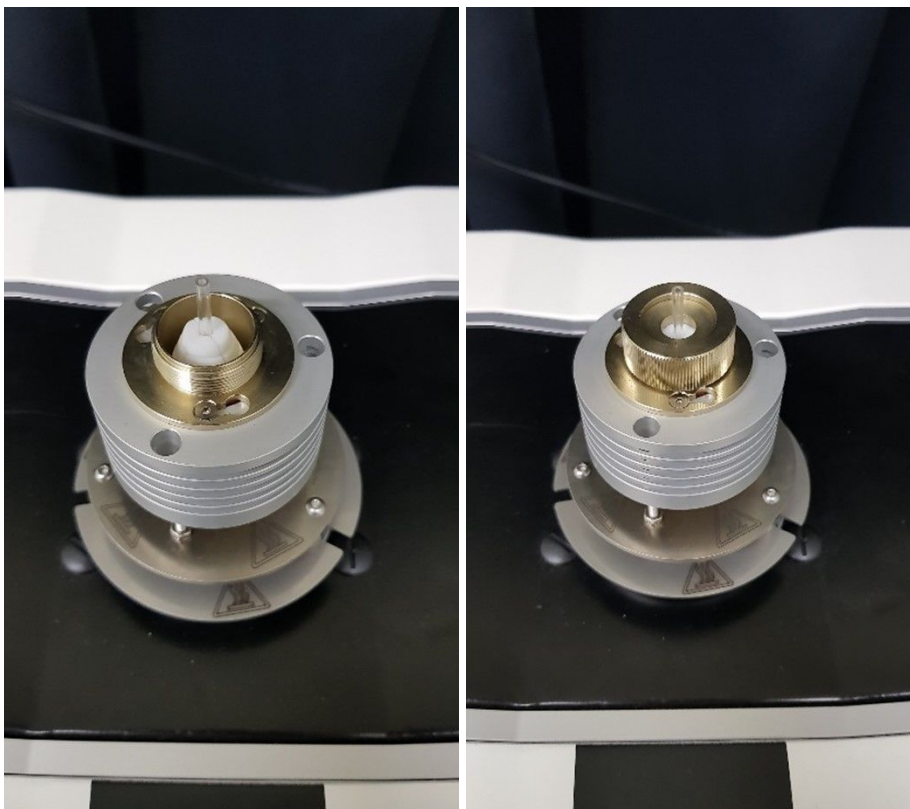


Figure 4.151: Mounting the Sample Tube

- Connect the tube transporting gas (8 mm outer diameter) to the back of the Electronic Box and start the gas flow.

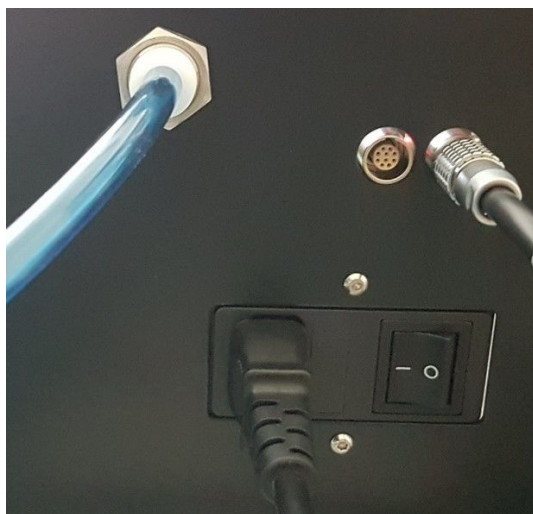


Figure 4.152: The Gas Tube Connection to the Electronic Box (left) from the Gas Flow Regulator (right)

The system is now ready for measurements.

4.20.3 Operation and Safety

The High Temperature Controller (HTC) is intended for sample heating up to 450 °C. The system heats up pressurized gas to anneal the sample in a P165/3 mm sample tube.

Safety Instructions

The HTC has two modes of operation:

- Mode A: Sample Heating
- Mode B: Furnace Gas

CAUTION



Risk of injury from hot surfaces

For both modes, all parts of the unit (heating unit below the spectrometer as well as heat dissipation unit above) will become very hot!

- ▶ Avoid touching the parts.
- ▶ Wear protective gloves if contact cannot be avoided.



For heater protection two fuses are installed that switch off the heater to prevent overheating. If the fuses blow, they must be exchanged by a service engineer.

Mode A: Sample heating

CAUTION



Risk of damage and contamination

Using the unit without gas flow may result in irreversible damage.

- ▶ Always operate the unit with gas flow on.
- ▶ If using pressurized air ensure that enough filters are used in the air circulation to avoid oil contamination of the resonator.

Mode B: Furnace Gas

CAUTION



Risk of damage due to extreme temperatures

Operating the unit with temperatures above 450 °C can destroy the unit, as well as the entire spectrometer.

- ▶ Do not operate the unit above 450 °C.
- ▶ Ensure that the furnace gas does not contain (even invisible) sooty particles. These particles will block the gas flow and lead to overheating.

Long-time operation at high temperatures can result in the buildup of a background signal that can overlap with the sample signal.

The main source of the background signal is the cavity itself due to aging effects, which is accelerated at high temperatures. The aging process of the resonator might be significantly slowed down by keeping the resonator at lower temperatures by flushing it with nitrogen gas from the lab network or using the flushing vessel (E8000287). The baseline correction algorithm can be used for spectra showing baseline distortions.

The background signal can also arise from impurities in the compressed air if it is used as the heating gas. These impurities will contaminate the resonator Dewar, and can be removed by carefully cleaning the resonator Dewar using an ultrasonic bath with addition of citric acid.

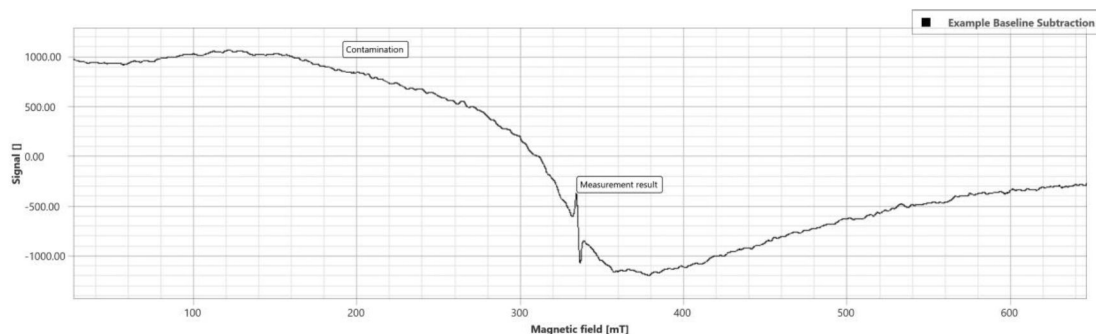


Figure 4.153: Measurement Result

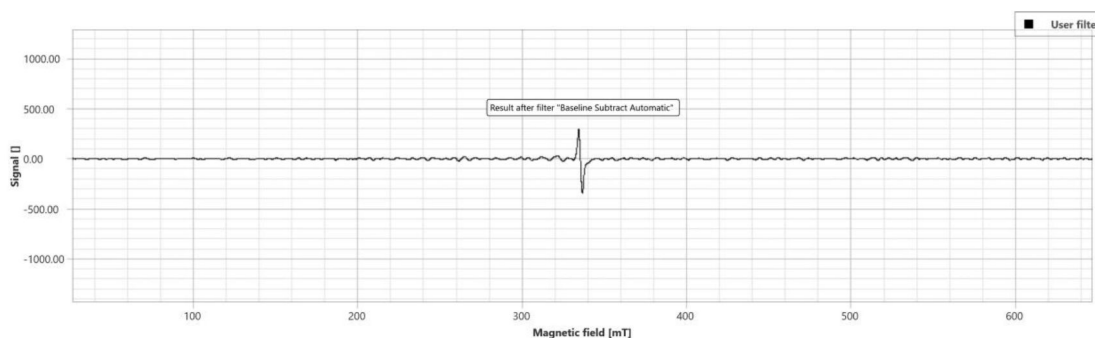


Figure 4.154: Measurement Result after Filter Baseline Subtract Automatic

Refer to the ESR5000 Spectrometer User Manual for more information.

Switching off the unit

After usage:

- Use the software to cool the unit down to 50 °C.
- Switch off the electronic box, you may leave the set-up assembled.
- Turn off the gas flow.

CAUTION



Risk of damage due to electrical charge

Inserting or removing electrical cables when system is running may result in damage to the unit or spectrometer.

- ▶ Power-off the system before removing or inserting electrical cables.

4.20.4 Software Handling

- Select **Recipe Editor | Temperature Series**.

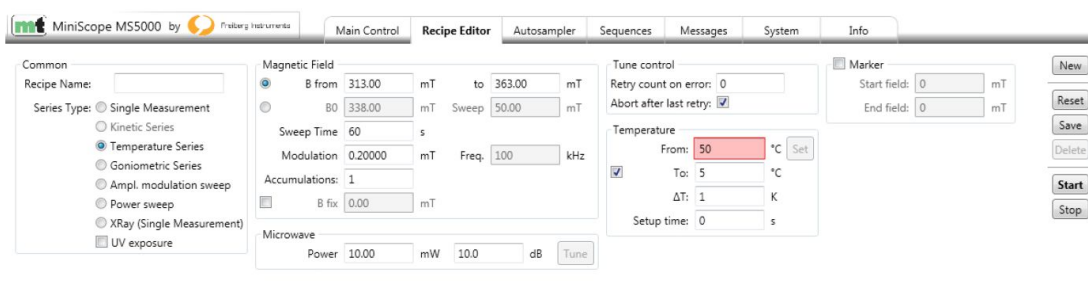


Figure 4.155: The Recipe Editor

- Select the desired temperature in °C in the red marked window and push the **Set**-button to heat up the system. The available temperature range is from 50 up to +450 °C.
- Start your measurement recipe.
- Temperature series can be run by first activating the checkbox left to the **To** field. Then the start and end temperature (in °C) and the step width (in K) must be selected. **Setup time** is the time for holding the chosen temperature before the acquisition starts.

- Advanced users may use the **Sequences** tab to define measurement sequences.
- To record the temperature profile, use the diagram in the tab **System** and **Device state** with the setting **Temperatör.Temperature [°C]**.

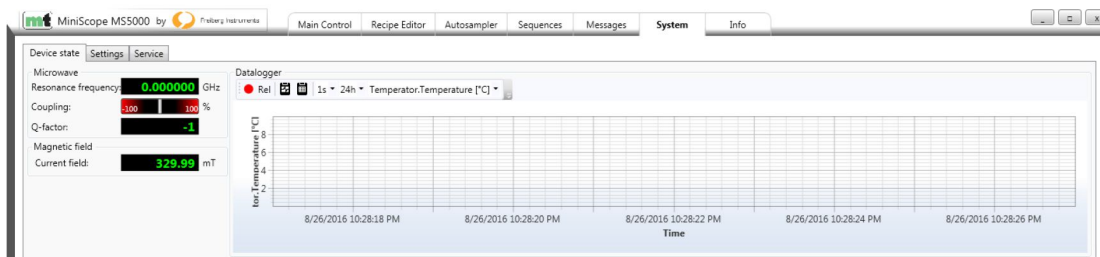


Figure 4.156: The System Tab

For additional information, refer to the ESR5000 User Manual or contact Bruker.

4.21 ESR Electrochemical Cell (Module E8000244)

The ESR cell for electrochemical reactions is designed for generation of radicals in solutions by a potential gradient.



Figure 4.157: ESR Electrochemical Cell

You can find more information about the working principle of an ESR electrochemical cell in the literature.

The following figure shows the components of an ESR electrochemical cell:

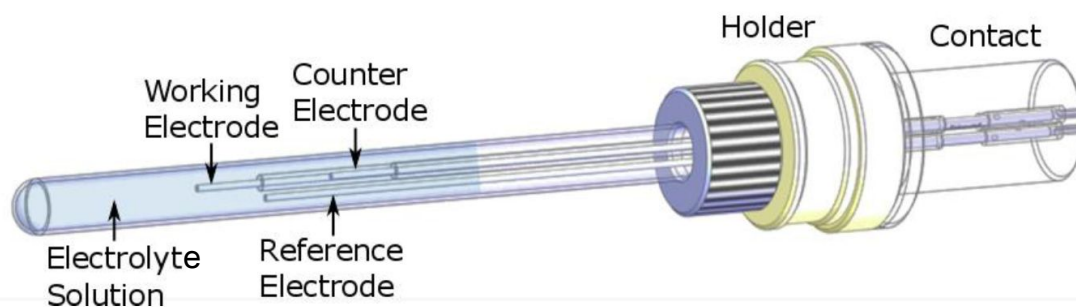


Figure 4.158: Drawing of an ESR Electrochemical Cell Components

4.21.1 Technical Specifications

The ESR cell consists of three main components:

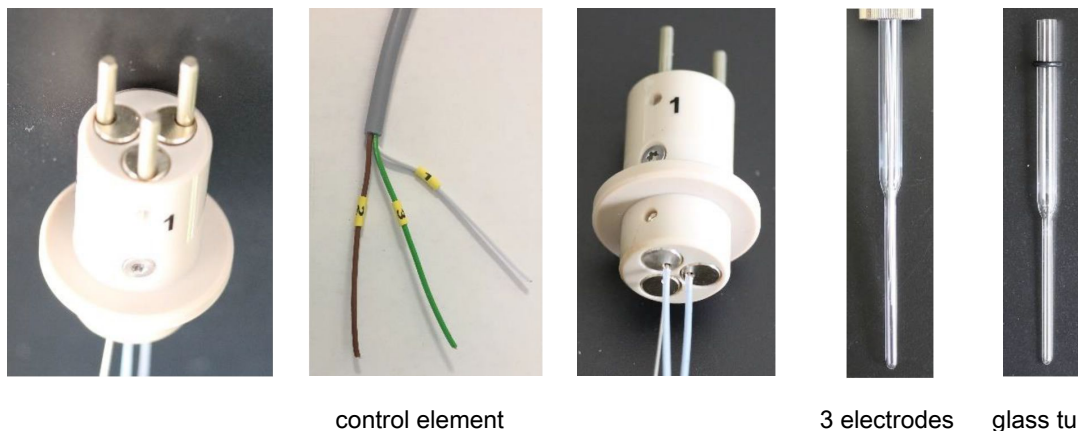


Figure 4.159: Components of the ESR Electrochemical Cell

The control element is capped with a nut to secure the connection point between the electrodes and the cables. There are notches in the nut to define which cable connects to which electrode.

There are three electrodes of different lengths:

- The reference electrode is the shortest one and made of silver. The reference electrode is not in the resonator during the measurements.
- The counter and working electrode have longer lengths and are made of platinum.

All electrodes are adjustable in length.

The glass tube is the container for the electrolyte solution.

4.21.2 Installation

Setup

- Disassemble the cell:



Figure 4.160: Disassembling the Cell

- Fill the tube with electrolyte solution so that all electrodes are immersed in the liquid.
- Make sure that the O-ring between the inner union nut and the tube is seated correctly:

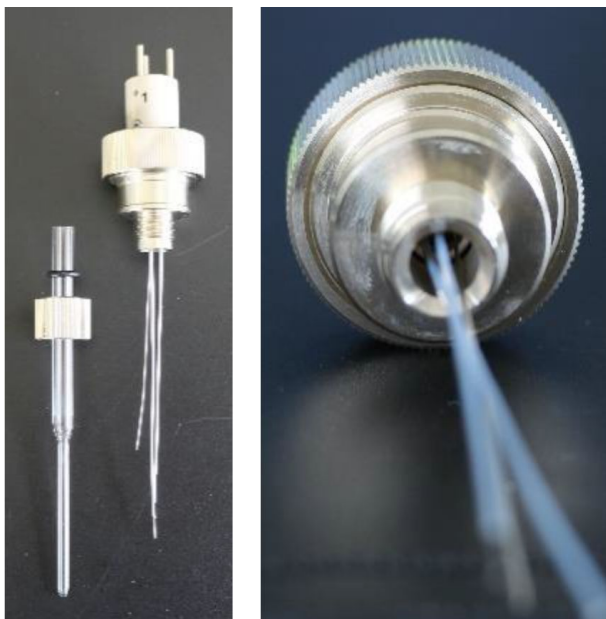


Figure 4.161: The O-Ring between Inner Union and Tube

- Tighten the inner union nut.
- Remove the standard sample holder and replace it with the electrochemical cell:



Figure 4.162: Replacing Sample Holder with ESR Cell

- Connect the cell's cables in the correct positions:
 - 1 = Working Electrode (long, from platinum, insulated)
 - 2 = Counter Electrode (middle, from platinum, insulated)
 - 3 = Reference Electrode (short, from silver)
- Adjust the height of the longest electrode that it is exactly in the middle of the cavity.

Length adjustment and replacement of the electrodes

The cell holder is not adjustable in height.

The electrodes may require replacement due to wear and tear.

To replace the electrodes:

- Open the cap:



Figure 4.163: Opening the Cap

- Remove the holder with the electrodes.

- Loosen the red marked screw at the bottom of the holder:



Figure 4.164: Removing the Holder with the Electrodes

- Pull out the electrodes.

- Replace them with new ones:

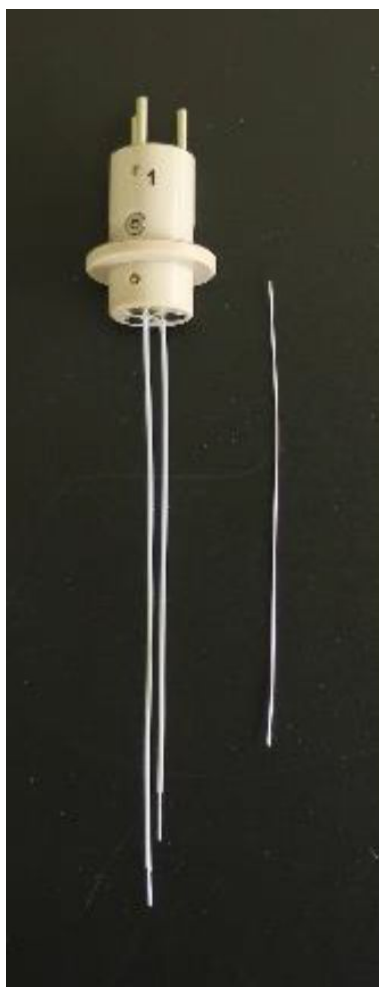


Figure 4.165: Replacing the Electrodes

- Mount the electrodes in the holder by pushing them into the intended slot.
- Adjust the electrodes correctly and tighten the screw.
- Mount the holder carefully onto the glass tube.
- Tighten the holder with the cap.
- Make sure that the electrodes are positioned correctly.

4.21.3 Sample Handling



- ▶ Handle the filled cell with care.
- ▶ Clean the outside of the tube before inserting it into the resonator.
- ▶ Avoid contaminating the cavity with liquids, otherwise unwanted EPR signals may arise.

Tuning the resonator with aqueous solutions is not possible. The electrochemical cell is designed to work with organic solvents such as DMSO, DMF, acetonitrile, etc.

4.21.4 Software Handling

There are no special ESRStudio recipes or steps required for operating the ESR cell for electrochemical reactions.

5 Replacement of Parts



Only trained Bruker personnel are allowed to mount, retrofit, repair, adjust and dismantle the unit!

Loss of Guarantee

The use of non-approved spare parts will invalidate the manufacturer's guarantee.

Purchase spare parts from authorized dealers or directly from the manufacturer. See Contact for manufacturer's address.

Replacement of the Power Cord

A replacement power cord must be tested and comply with the appropriate requirements in IEC 60227/IEC 60245 or ANSI/UL 817 and CSA C22 No. 1.

5.1 Returning the Unit for Repair

If the Bruker Hotline diagnoses an instrument failure that requires a part to be returned for repair, please follow the procedure listed here:

1. Contact your local Bruker office to start the repair process (see Contact). Repair is always handled by your local Bruker office. Their reply will contain all necessary information for the subsequent repair process steps.
2. They will provide you with details on the shipping address, and also in most cases a "Return Merchandise Authorization" number (RMA number) that allows references to the repair case. Always refer to this RMA number in case of questions.
3. Send the defective part to the local Bruker office and include the following documents:
 - RMA sheet (if RMA number was assigned).
 - Signed Equipment Clearance Form (BBIOF002/H152631). The Equipment Clearance Form will be sent to you as part of step 1 (see above) with information about the returned part (part number, serial number, your contact details) already filled in.
4. Attach the relevant papers to the *outside* of the packaging, for instance in a transparent polybag.



The unit should be returned using the original container and packing assembly. If this packaging is no longer available, contact your local Bruker office for further instructions.

6 Contact

Manufacturer

Bruker BioSpin GmbH
Rudolf-Plank-Str. 23
D-76275 Ettlingen
Germany

Contact form: <https://www.bruker.com/en/services/service/magnetic-resonance.html>

Website: <http://www.bruker.com>

WEEE DE43181702

Bruker BioSpin Hotlines

Contact our Bruker BioSpin service centers.

Bruker BioSpin provides dedicated hotlines and service centers, so that our specialists can respond as quickly as possible to all your service requests, applications questions, software or technical needs.

Please select the service center or hotline you wish to contact from our list available at:

<https://www.bruker.com/en/services/support.html>

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