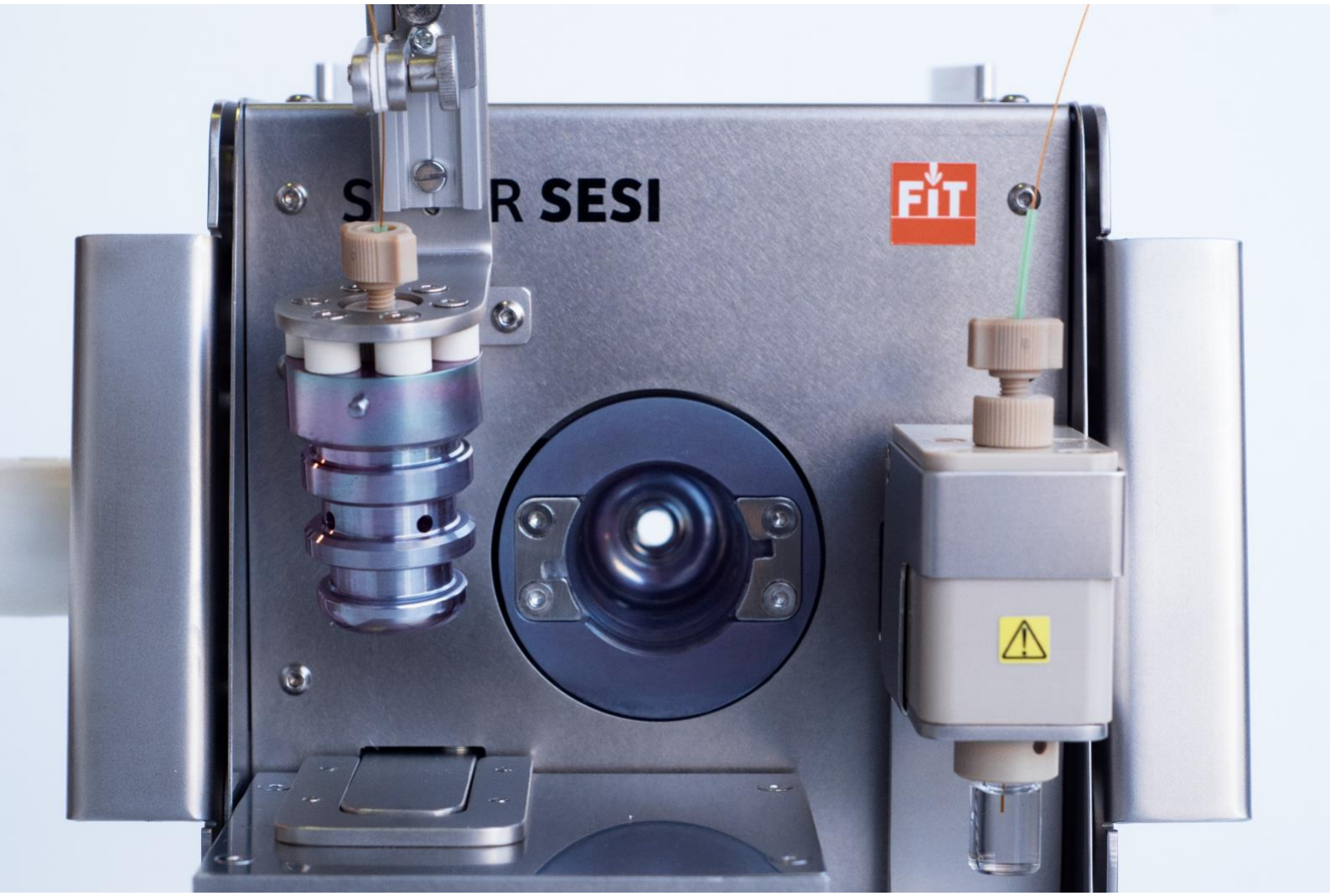
SUPER SESI

Airborne Molecules & Metabolites Analysis

User Manual

Revision 2.1 -February 2020



FOSSILIONTECH

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Revision 1.0 (product release) April 2017
Revision 2.0 Update after product improvements February 2019
Revision 2.1 Correction of typos February 2020

For Research Use Only. Not for use in diagnostic procedures.

Table of Contents

Preface

This *User Manual* describes how to install, remove, use and maintain the SUPER SESI source developed and commercialized by Fossil Ion Technology.

If you would like to suggest a change in this document, or if you need further assistance, please email us at info@fossiliontech.com. You can also contact us through our online contact form: http://www.fossiliontech.com/contact-us/

Related Documentation

The SUPER SESI source works in tandem with the mass spectrometers of Thermo Fisher Scientific. Please read the corresponding instruction manual of the mass spectrometer and observe its indications when using the SUPER SESI.

Compatible Mass Spectrometers

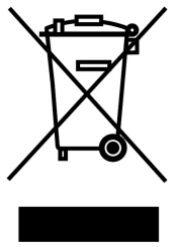
SUPER SESI is compatible with the following mass spectrometers:

- Orbitrap Q-Exactive Series
- Orbitrap Exactive series
- LTQ Series
- LCQ Fleet Series
- Orbitrap LTQ
- Orbitrap Elite

We are working to increase the number of compatible mass spectrometers. If you have instrument compatibility questions, please contact us via email: info@fossiliontech.com You can also contact us through the contact form in Fossil Ion Technology web page: http://www.fossiliontech.com/contact-us/

WEEE compliance: Directive 2002/96/EC

This product complies with the European Union's Waste Electrical & Electronic Equipment (WEEE) Directive 2002/96/EC. It is marked with the following symbol:



If this product is located in Europe and you want to discard it, please send an email request to info@fossiliontech.com with the following information:

- Product specification
- Number of product pieces and estimated total weight and volume
- Pick-up address and contact person (please include contact information)
- Declaration of decontamination, stating that all hazardous fluids or materials have been removed from the product.

Fossil Ion Technology shall contact you and arrange a pick-up service at the most convenient time to recycle your product at no cost for you. Please note that this recycling program is not for biological hazard products and contaminated products. You must treat these types of products as biohazard waste and dispose of them in accordance with your local regulations. SUPER SESI is specifically designed solely for the purposes of research and development.

EMC Compliance: Directive EMC 2014/30/EC

SUPER SESI meets the requirements of the directive EMC 2014/30/EC. It has been tested, and it is in accordance with the norm IEC 61326-1.

Low Voltage Safety Compliance: Directive LVD 2014/35/EC

SUPER SESI meets the requirements of the directive LVD 2014/35/EC. It has been tested, and it is in accordance with the norm IEC 61010-1.

Notices and symbols used in this manual

Please make sure that you understand the special notices, symbols, and caution labels in this guide. Most of the special notices and cautions appear in boxes; those pertaining to safety also have corresponding symbols. Some symbols are also marked on the SUPER SESI source itself and can appear in color or in black and white. Some safety and notices used in this user manual include the following:

CAUTION NOTE highlights hazards to humans, property, or the environment. Each CAUTION warning is accompanied with the corresponding CAUTION symbol.

IMPORTANT NOTE highlights information to prevent invalid test results, damage to software or loss of data.

INTERESTING NOTE *highlights information of general interest*

TIPS & TRICKS *highlights key information that can be useful to complete of a task or to improve the quality of the results.*

Contacting us

If you need further assistance, please contact us.

Address: *Fossil Ion Technology S.L.: Calle la Gitanilla No. 17, Nave 22; ZIP 29004, Malaga, Spain.*

You can also contact us via email: info@fossiliontech.com, or through the contact form in our Technology web page: http://www.fossiliontech.com/contact-us/

General safety precautions and important notes

CAUTION Do not perform any servicing that is not contained in this manual. In order to avoid injury or damage to the instrument, **do not perform any servicing that is not specified in this manual unless you are qualified to do so**.

CAUTION Some parts of the SUPER SESI are heated for optimum performance. The sample line and the ionization chamber are heated by electric resistors. The electrospray probe can be heated by thermal conduction. These parts may cause burns if touched. **Allow heated components to cool before touching them, and prevent other persons to touch them**.

CAUTION The tip of the electrospray silica capillary is very sharp and small. It can puncture the skin, and it can be harmful to the eye. **Handle it with care and wear safety glasses when handling the silica capillary.**

CAUTION The recommended electrospray liquid is high purity water with 0.1% formic acid, but other solvents can also be used, including flammable solvents such as methanol or ethanol. Use care when handling these solvents. Also, **be cautious when handling the system in the presence of flammable materials**. The SUPER SESI could produce small glow discharges that could ignite flammable atmospheres.

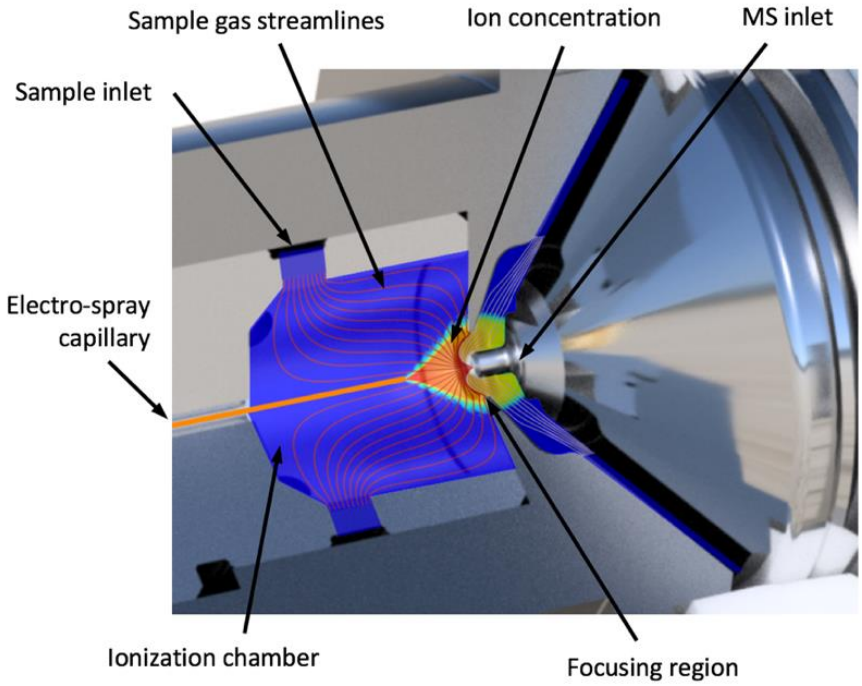
CAUTION The SUPER SESI uses the high voltage that is provided by the Mass Spectrometer. The SUPER SESI incorporates three high resistors (1 GOhm each) to reduce the currents down to 0.03mA in the event that an electrode is exposed to ground or to a person. This current is well below the hazardous threshold and the threshold that can be felt by a human. Nevertheless, **to prevent damage to the instrument, the electrodes should not be touched when the voltage is applied**.

IMPORTANT NOTE The SUPER SESI is a very sensitive instrument. It is very vulnerable to contamination, which can be observed as background signals. Even if no chemically hazardous substances are used, **always use gloves when handling the SUPER SESI to avoid contaminating it.**

SUPER SESI, general description

Principle of operation

A nano-electrospray creates the ions that are used to charge the molecules of interest, which are introduced though the sample line. The molecules of interest and the charging ions react at ambient pressure to form analyte ions. These ions are then passed to the mass spectrometer for analysis.



Ions are not fragmented, enabling for MS-MS analysis and reliable analyte identification.

An accelerated flow is used to radially confine the ions and focus them to the mass inlet, without perturbing the ionization region. The fast response of the ionizer enables kinetic studies, which eases to identify specific responses to controlled stimuli, and to differentiate confounding variables from real discoveries.

SUPER SESI incorporates all the components and controls in one single unit. It is ready to connect with the MS and start analyzing. Very little sample preparation and virtually no laboratory consumables are required. SUPER SESI is designed to easily replace the capillary when required. A nano-ampere meter is used to monitor the stability of the electrospray.

Intended use

SUPER SESI is designed to be coupled with a mass spectrometer and to be used by trained personnel in a laboratory environment. The purpose of SUPER SESI is to enable analysis of VOCs at trace concentration. It is not intended for use with concentration of substances that could be harmful or corrosive.

CAUTION The safety of this equipment could be compromised if it is not used in the manner specified in this manual.

Complete system: SUPER SESI

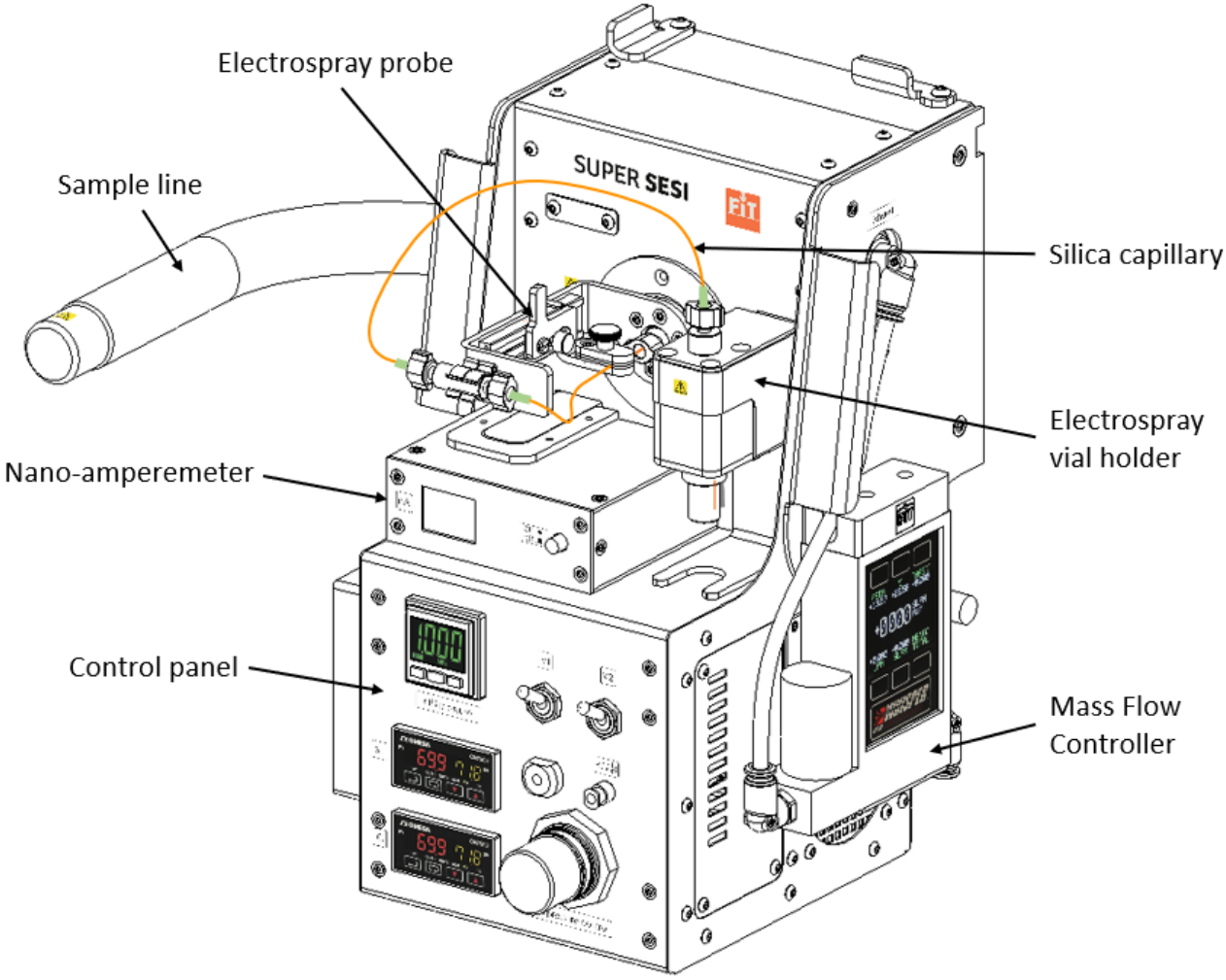


Figure 1. SUPER SESI front view.

Components: Main Body

The main body incorporates the ionization chamber, the sample line, all the controls, and the coupling with the mass spectrometer.

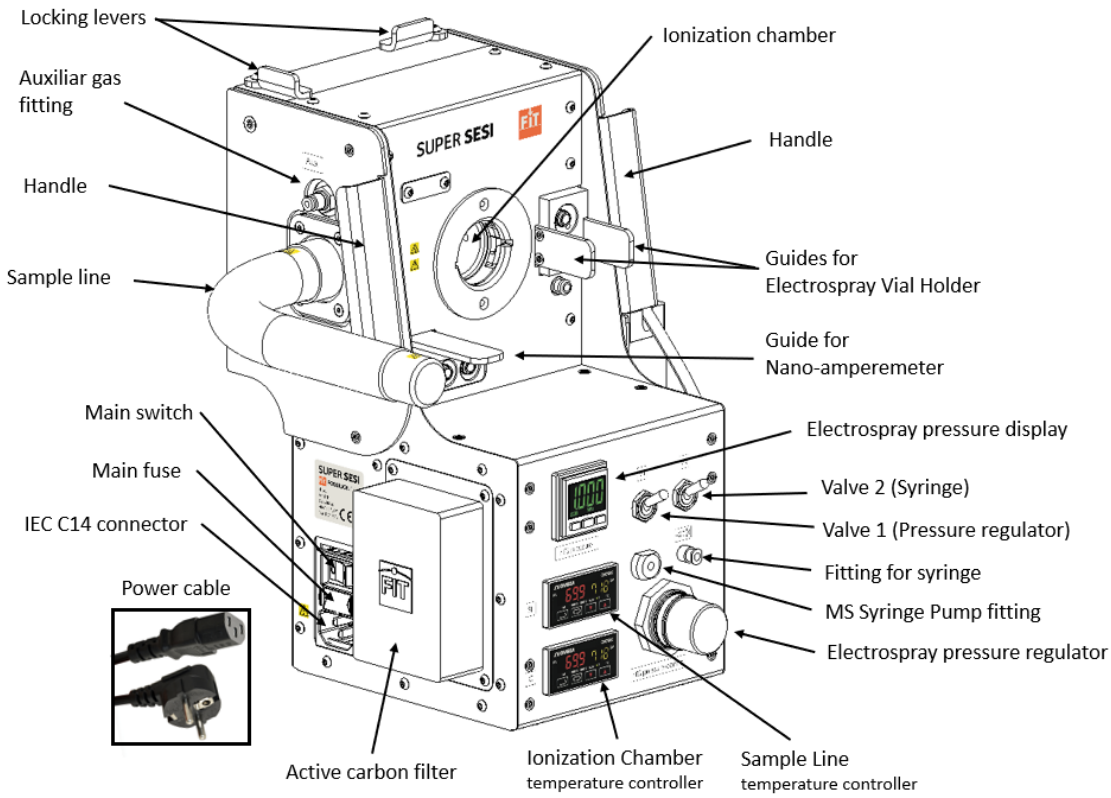


Figure 2. Main body, front view.

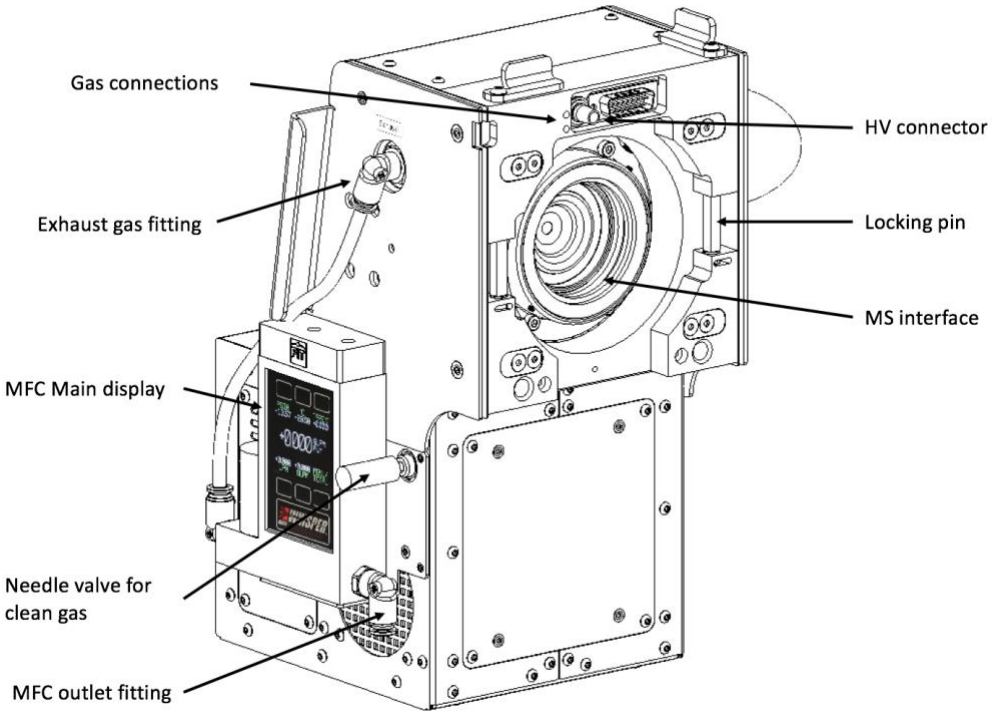


Figure 3. Main body, rear view.

Components: Electrospray vial holder

The electrospray vial holder incorporates all the connections and fittings required to produce the electrospray. It can be seamlessly coupled and decoupled with the main body to ease operation and cleaning procedures.

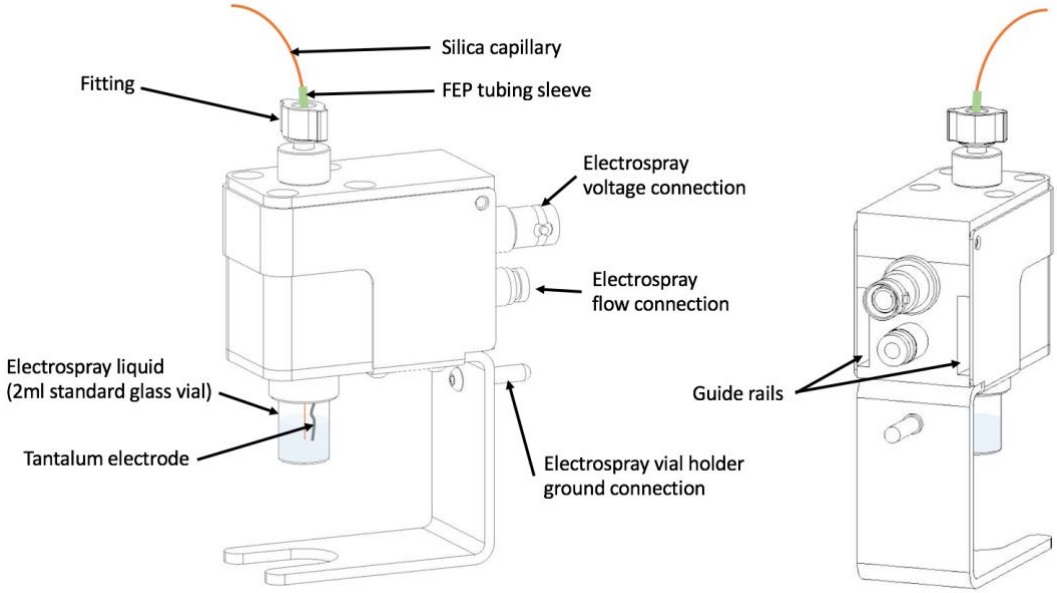


Figure 4. Electrospray vial holder

Components: Electrospray Probe

The electrospray probe houses the tip of the silica capillary, and incorporates a positioning system that allows repeatable and optimum conditions. It closes the ionization chamber and guides the sample gas smoothly to the electrospray tip to minimize convective evaporation at the meniscus of the spray, thereby improving the stability of the spray at temperatures close to the boiling point of the electrospray liquid.

The handgrip is thermally insulated with ceramic spacers to ensure that the parts in contact with the sample gas can be maintained at high temperature while the parts in contact with the user remain at safe temperatures.

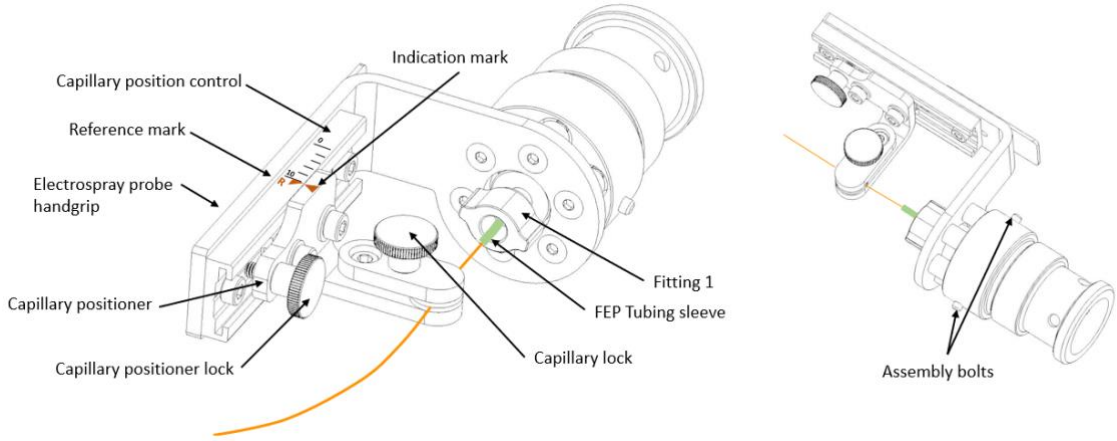


Figure 5. Electrospray probe.*The figure may differ from the latest design.

Components: Sample inlet protection cover

The inlet of the sample line is protected with a Teflon cover to prevent contamination when it is not being used and to protect the user from high temperature surfaces. This cover must be removed in normal usage.

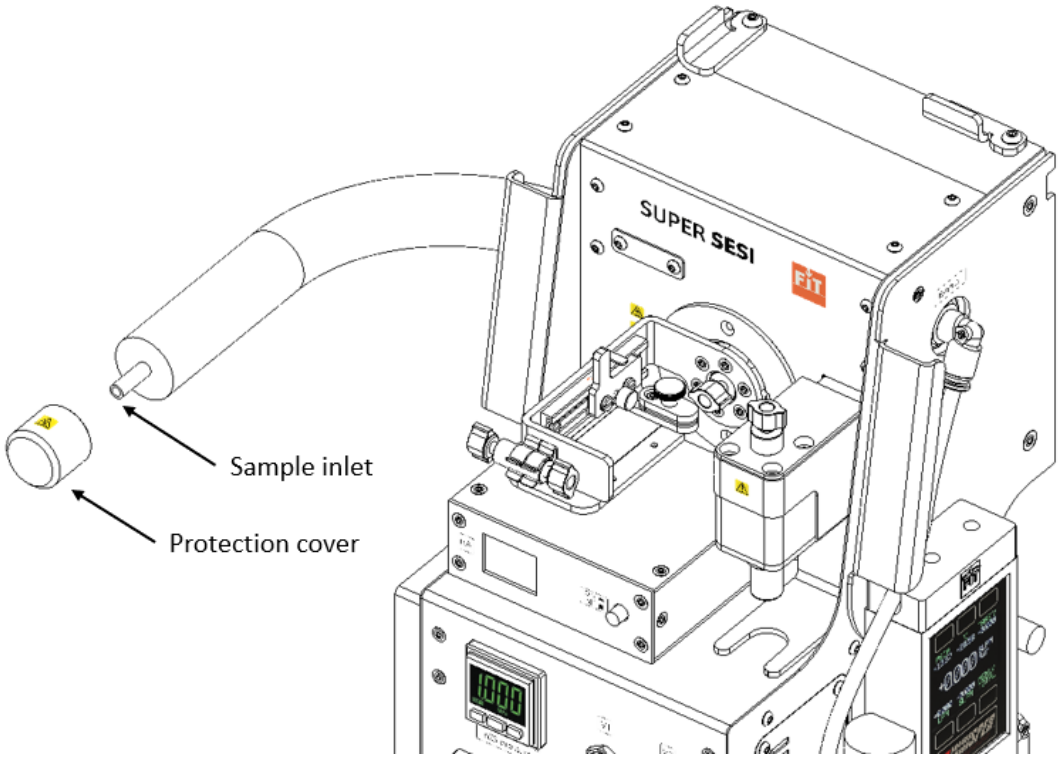


Figure 6. Sample inlet protection cover.

Components: Nano-amperemeter

The nano-amperemeter provides a measurement of the electrospray current. This parameter and the current measured by the mass spectrometer are used to evaluate the stability of the electrospray. The nano-amperemeter is not externally powered. Instead, it uses rechargeable batteries to avoid any leaked currents. It can be easily connected with the main body, and with the charging unit when the batteries need to be recharged.

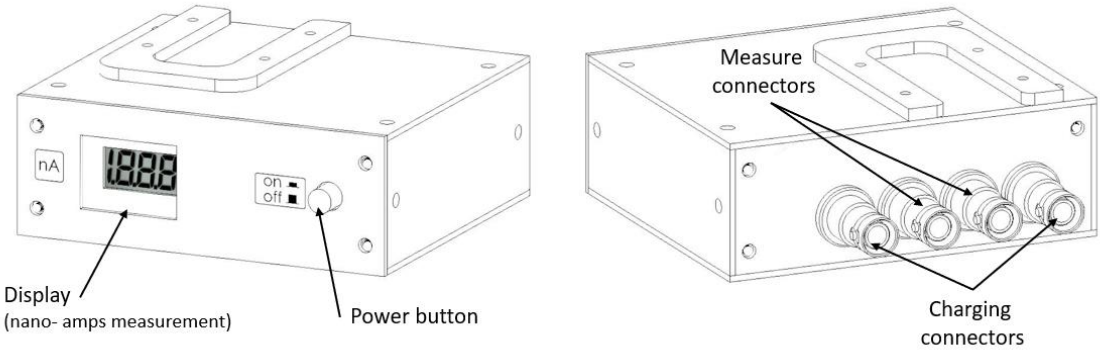


Figure 7. Nano-amperemeter

Components: nano-amperemeter charging unit

This unit charges the nano-amperemeter while they are connected. A Status LED indicate if the nano-amperemeter batteries are charging (red light) or fully charged (green light).

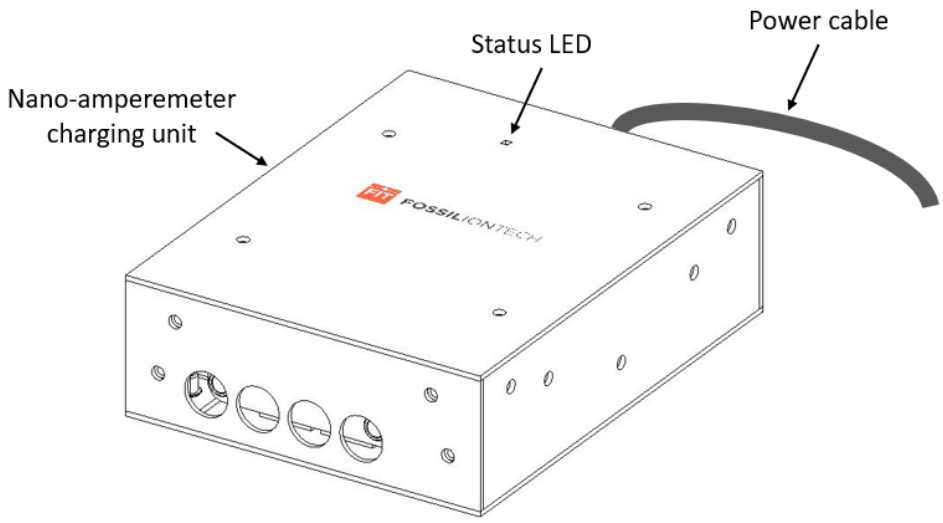


Figure 8. Nano-amperemeter charging unit

Symbols used in the SUPER SESI

Label Name	*Label design*	*Location*	*Purpose*
Identification label	SUPER SESI FIT FOSSILIONTECH Model: Serial No: 220-240V AC 50/60 Hz, 250VA Fuse: F, 1A-250V CE	Besides power connector	Identification.
Contact us	Do not open. Opening this panel could result in warranty void. Contact us if you need technical assistance: Fossil Ion Technology S.L., C/ La Gitanilla 17, nave 22, 29004-Málaga, Spain. e-mail: info@fossiliontech.com Web-page: www.fossiliontech.com	Rear panel	Contact info. for technical assistance.
General warning	⚠	Front panel	Identify potential risks.
Hot surface warning	♨	Near ionization chamber & sample line	Identify potential hot surface.
High voltage warning	⚡	Near power input	Identify power voltage.
Ruler	0 10	Electrospray probe	To position the tip of the electrospray
Aux gas outlet identifier	Aux	Near aux gas	Identify aux gas outlet

Label Name	*Label design*	*Location*	*Purpose*
NanoAmp units	nA	Near nanoAmp display	Identify nanoAmp units
NanoAmp on/off	on off	Near nanoAmp on/off button	Identify button
nES pressure display	nES pressure	Near nESI pressure display	Identify display
Ionization chamber temperature control identifier	IC	Near ionization chamber temperature controller	Identify controller
Sample line temperature control identifier	SL	Near sample line temperature controller	Identify controller
Valve V1 identifier	V1	Near valve V1	Identify valve V1
Valve V2 identifier	V2	Near valve V2	Identify valve V2
Syringe inlet identifier	1	Near syringe inlet	Identify syringe inlet
Syringe pump steam cleaning inlet	2	Near syringe inlet	Identify syringe inlet
nES pressure control identifier	nES pressure control	Near Pressure control	Identify pressure control
Exhaust identifier	Exhaust	Near exhaust outlet	Identify exhaust outlet

Technical specifications

Power:	220-240 V, 50-60 Hz, 250VA
Dimensions:	207x200x291 mm
Weight:	10Kg
Max. temperature of sample line:	180ºC
Max. temperature of ionization chamber:	130ºC
Protection level:	IP20

Installation

Installation requirements

The SUPER SESI is an ion source for mass spectrometry analysis. In order to function, it must be coupled with a compatible mass spectrometer. It requires an electric power receptacle 230 V ac, ±10%, frequency of 50/60 Hz, single phase. The SUPER SESI must be powered with the same ground connection, and the same phase as the mass spectrometer.

A small table located near the working area is required to place the source and the different components during initial installation and routine operation.

SUPER SESI is designed to operate under the following conditions:

- Indoor use; altitude up to 2 000 m; temperature 5°C to 40°C; maximum relative humidity 80% for temperatures up to 31°C, decreasing linearly to 50% at 40°C;
- TRANSIENT OVERVOLTAGES up to the levels of OVERVOLTAGE CATEGORY ll;
- applicable POLLUTION DEGREE of the intended environment (POLLUTION DEGREE 2).

IMPORTANT NOTE SUPER SESI does not incorporate circuit breakers or residual current breakers. It only incorporates protection overcurrent fuses (1A). The user must ensure that the electrical power installation for SUPER SESI provides the required electrical protections, in accordance with local legislation.

Unboxing the SUPER SESI

The SUPER SESI is packed with the nano-amperemeter, the vial holder, the electrospray probe and the Mass Flow Controller in their working position. In order to protect the SUPER SESI from contamination during shipping, the MS interface is covered with a stainless steel cover. To get the SUPER SESI out of its original packaging, grab the Handles and lift the SUPER SESI. Remove the parafilm before using the SUPER SESI.

CAUTION Do not grab the sample line to lift the SUPER SESI.

IMPORTANT NOTE Make sure that no Parafilm is left on the SUPER SESI before you start using it. In particular, make sure that all Parafilm is removed before you start the SUPER SESI heaters.

Along with the SUPER SESI, you will also find the following: a power cord, a syringe and the battery charger for the nano-amperemeter.

IMPORTANT NOTE Keeping the original packaging to store the SUPER SESI is strongly advisable to prevent contamination of your SUPER SESI when it is not being used. The SUPER SESI should be transported in its original package.

Coupling the SUPER SESI and the Mass Spectrometer

Before coupling the SUPER SESI to the Mass Spectrometer, the original Ion source and the ion sweep cone must be removed. Remove the Thermo ion source housing and the ion sweep cone from the mass spectrometer following the instructions indicated in the Operating Manual of the instrument. Once these components are removed, the inlet of your mass spectrometer should have the appearance of the figure 9:

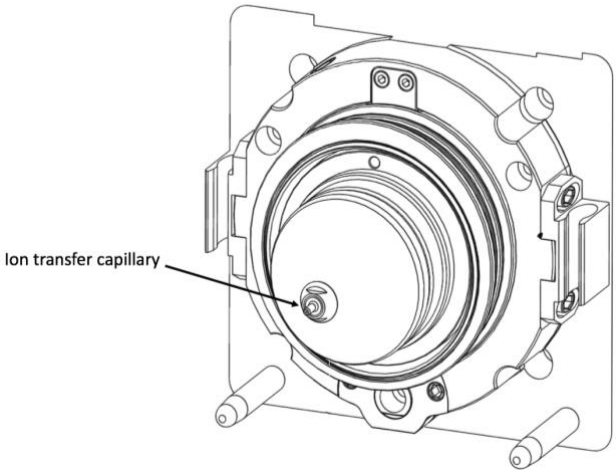


Figure 9. Inlet of the mass spectrometer

CAUTION Hot Surface. The ion transfer capillary typically operates at 250 to 400 ºC. Be careful not to touch the capillary or the cone while it is hot. Before removing the sweep cone, reduce the capillary temperature by setting the capillary temperature to 25 ºC, and wait for the ion transfer capillary. Allow the ion transfer capillary to cool to room temperature for approximately 60 minutes before you touch or remove either component.

Make sure that the SUPER SESI locking levers are placed in the open position. See figure below:

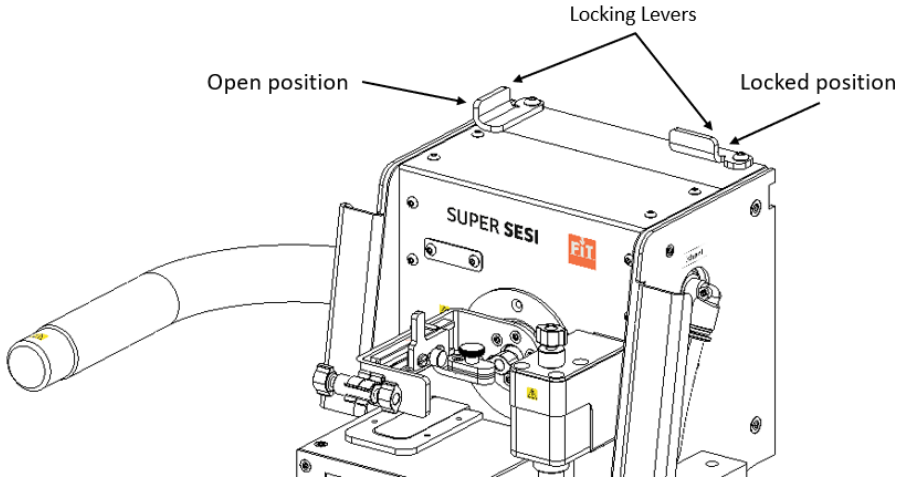


Figure 10. SUPER SESI Locking Levers in open position.

Install the SUPER SESI on the mass spectrometer in the same way as the original Thermo ion source. For this: Carefully align the two guide pin holes on the rear of the SUPER SESI with the ion source housing guide pins on the mass spectrometer; Carefully press the SUPER SESI onto the ion source mount using the handles; Rotate the SUPER SESI locking levers 90 degrees to lock the SUPER SESI onto the ion source mount assembly.

CAUTION Use the SUPER SESI handles to lift the SUPER SESI and to press it against the Mass Spectrometer. Do not grab the sample line or other parts to lift the SUPER SESI. Doing so could damage the sample line.

Finally, connect the power cable from the IEC C14 connector in SUPER SESI to the wall power receptacle.

IMPORTANT NOTE the SUPER SESI and the mass spectrometer must be connected to the same line, and they must share a common ground and a common phase.

Changing the electrospray liquid

CAUTION During this operation, the electrospray voltage must be set to zero and the pressure of the electrospray vial holder must be set to zero.

If the spray voltage is on, set it to zero. See section 'Stopping the electrospray´ for more details. If the MS is acquiring, set the MS to stand-by mode. Refer to the user manual of your mass spectrometer for this. If the vial holder is pressurized, depressurize it. For this, make sure that the syringe is not connected to the syringe port and open the syringe valve 2 (up position).

IMPORTANT NOTE It is very important to make sure that the electrospray liquid and the vial holder are free from contamination. Always use gloves when manipulating them, and make sure that all surfaces are very clean and free from any contamination that could affect your measurements. If the vial holder or the electrospray liquid is contaminated, the background of your measurements will be affected until you clean the vial or change to a new liquid.

If required, the electrospray vial holder can be removed before changing the electrospray liquid. For this, take the vial holder, and pull gently towards you. Before removing the vial holder, make sure that the silica capillary is not simultaneously attached to the vial holder and the electrospray probe.

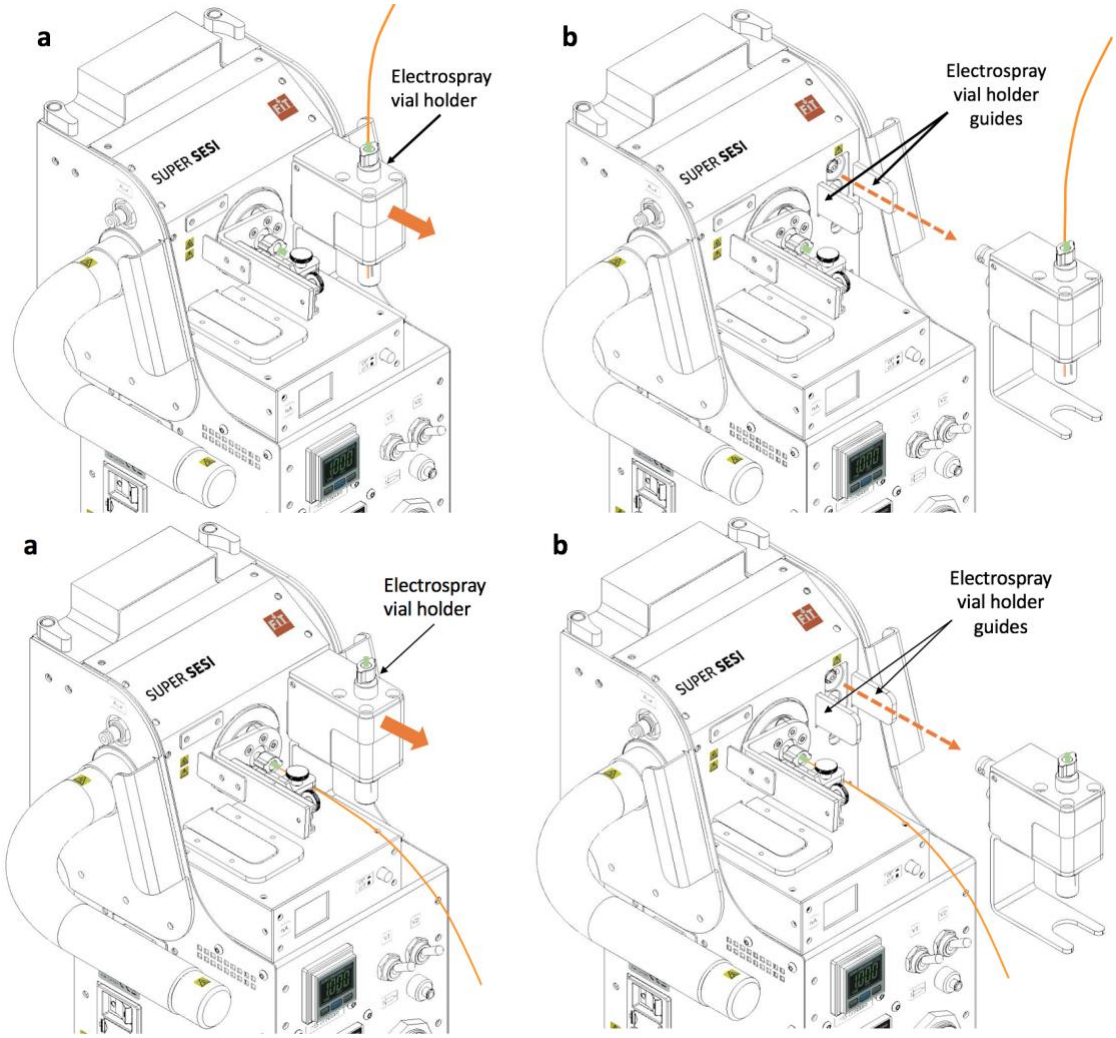


Figure 11. Removing Vial Holder. *The figure may differ from the latest design.

Use a horizontal and clean surface to place and store the Vial Holder. Always place the Vial Holder vertically as shown in figure 12.

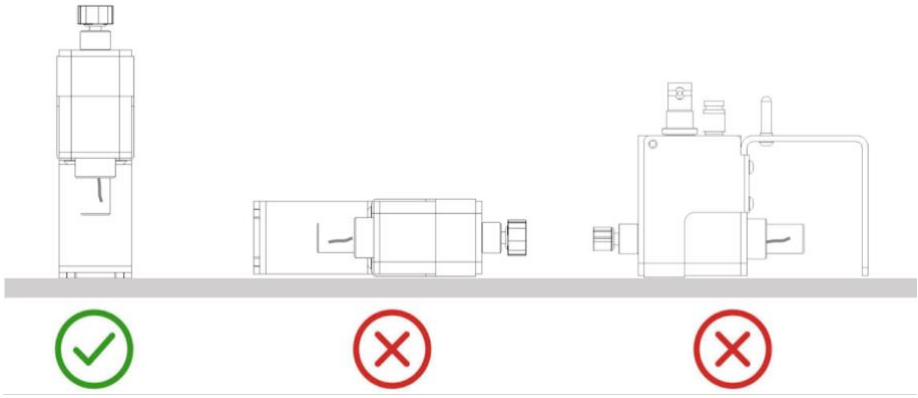


Figure 12. Storing the Vial Holder.

In order to change the liquid, with the voltage set to zero and the vial holder depressurized, unscrew the vial using your fingers, gently pull the vial down, and the remove it making sure that the tantalum electrode and the silica capillary are not bended.

IMPORTANT NOTE Be careful not to bend the Tantalum Electrode and the Silica Capillary when removing the glass vial from the Vial Holder.

CAUTION. The vial is made of glass. If broken, sharp edges may by produced. Be careful when handling the vial, specially when screwing, unscrewing and tightening it.

TIPS & TRICKS *you can clean the tantalum electrode and the silica capillary by gently wiping it with a Kimwipe soaked with new charging liquid.*

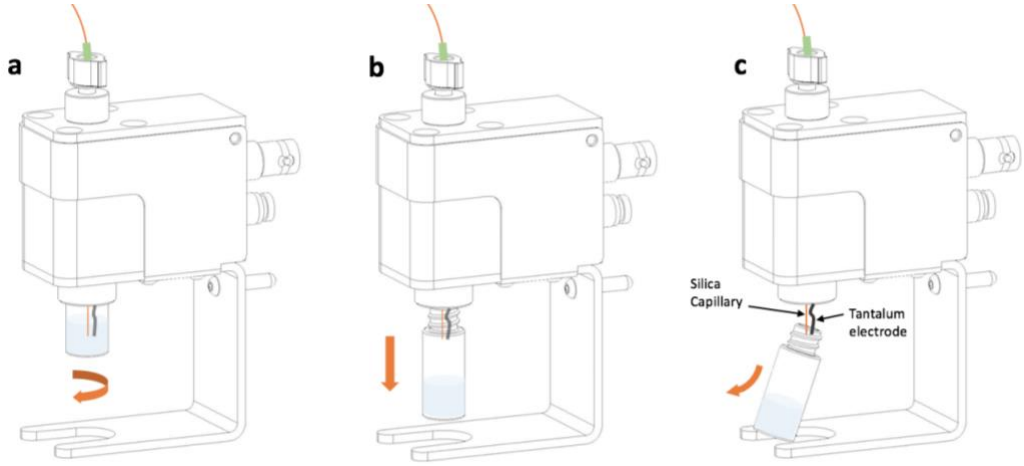


Figure 13. Changing the electrospray liquid

Clean the vial or use a new clean vial with new liquid. The maximum level of liquid shall be 3/4 parts of the total glass vial capacity.

IMPORTANT NOTE Use a solution of 0.1% formic acid in H_2O. Use only high purity (HPLC grade) solvents and reagents.

Introduce the Tantalum Electrode and the Silica Capillary through the bore of the new vial. Be careful of not to bend them. Slide the vial up to the top, and screw it until it is gas tight.

TIPS & TRICKS *In order to check that the vial is gas tight, close the valve 1 (down position), open the valve 2 (up position), pressurize the vial with the syringe, and close the valve 2 (down position). If the vial is properly tightened, the pressure should be stable*

CAUTION. The vial must be finger tight. Do not use tools to tighten or loose the vial. Doing so could damage the vial and the vial holder.

Changing the electrospray silica capillary

CAUTION During this operation, the electrospray voltage must be set to zero, the pressure of the electrospray vial holder must be set to zero, and the temperature of the ionization chamber must be set below 40ºC. Once the ionization chamber set temperature is changed, wait for at least 30 min until it cools down.

If the spray is on, set the electrospray voltage to zero. See section 'Stopping the electrospray´ for more details.

If the MS is acquiring, set the MS to stand-by mode. Refer to the user manual of your mass spectrometer for this.

If the vial holder is pressurized, depressurize it. For this, make sure that the syringe is not connected to the syringe port and open the syringe valve 2 (up position).

If the ionization chamber temperature is high, set it to a safe value and wait for it to cool down.

IMPORTANT NOTE It is very important to make sure that the silica capillary, the electrospray probe and the electrospray vial holder are free from contamination. Always use gloves when manipulating them, and make sure that all surfaces are very clean and free from any contamination that could affect your measurements. If these parts get contaminated by accidental contact with the skin or with other contaminated surface, the background of your measurements will be affected until you clean them.

Once the system is cooled, the voltage is switched off, and the vial holder is depressurized, unlock the electrospray probe, gently pull it out from the ionization chamber, and put it on the maintenance position.

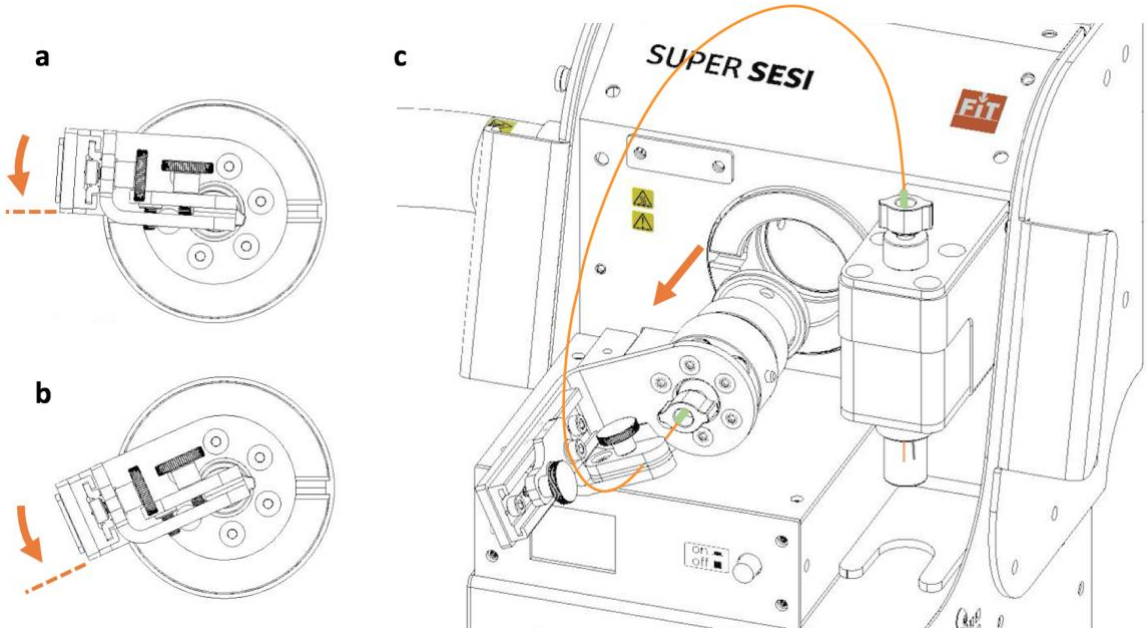


Figure 14. Unlocking the electrospray probe. *The figure may differ from the latest design.

TIPS & TRICKS *In order to reduce the pressure needed to pressurize the vial holder and create the Electrospray, we recommend to use a sharp silica capillary (20um id) as short as possible*, and an extension of silica capillary (50um id) unto the vial holder. For this, we provide a Union fitting that is attached to the Electrospray Probe Handgrip, so the two capillaries described previously can be connected at this point.*

** This length must be long enough to reach the Fitting union (Figure 15).*

In order to remove the old silica capillary, loosen the Capillary lock, the Upchurch fitting 1 (electrospray probe) and the Union fitting (if installed), then pull the capillary gently to remove it from both ends.

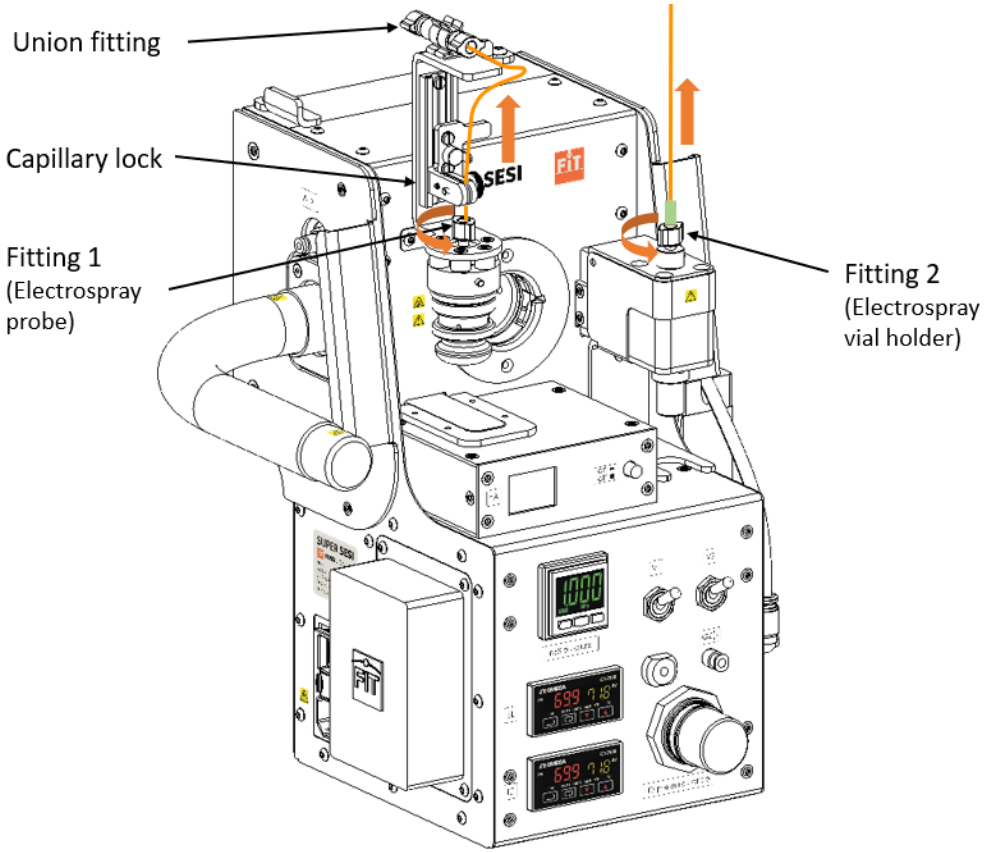


Figure 15. Loosing the capillary lock and the Upchurch fittings.

IMPORTANT NOTE The capillary is extremely thin and sharp. It may get clogged in the presence of aerosols. The tip might be damaged if it touches other hard materials. Refer to the instructions provided with the silica capillaries for handling.

The new 20 µm capillary has to be first inserted and locked in the electrospray probe. For this, first make sure that the Capillary lock, the Upchurch fitting 1 are loosen, then you will need to insert the unsharpened end of the capillary through the Teflon tube (used to seal the silica capillary with the Upchurch fitting 1) accessing from the open side of the electrospray probe (see Figure 16). Introduce it all the way through until it is visible from the Upchurch fitting 1.

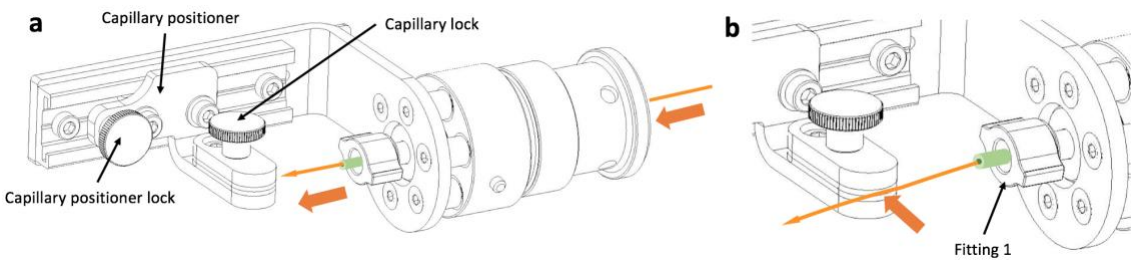


Figure 16. Inserting the silica capillary in the electrospray probe. *The figure may differ from the latest design.

***TIPS & TRICKS**. Take your time to introduce the silica capillary through the Teflon capillary. The tip of the silica capillary is sharpened and it is very fragile. Make sure that all movements are very gentle and that the tip of the silica capillary does not touch any metal or PEEK parts.*

IMPORTANT NOTE The Teflon capillary must be clean and free from any solid residue that could get attached to the tip of the silica capillary. This would affect the stability of the electrospray. If you are not sure whether there might be some residues in your Teflon capillary, unscrew the fitting 1, clean the Teflon capillary with high purity solvents, or replace it with a new one, and put it back in the electrospray probe.

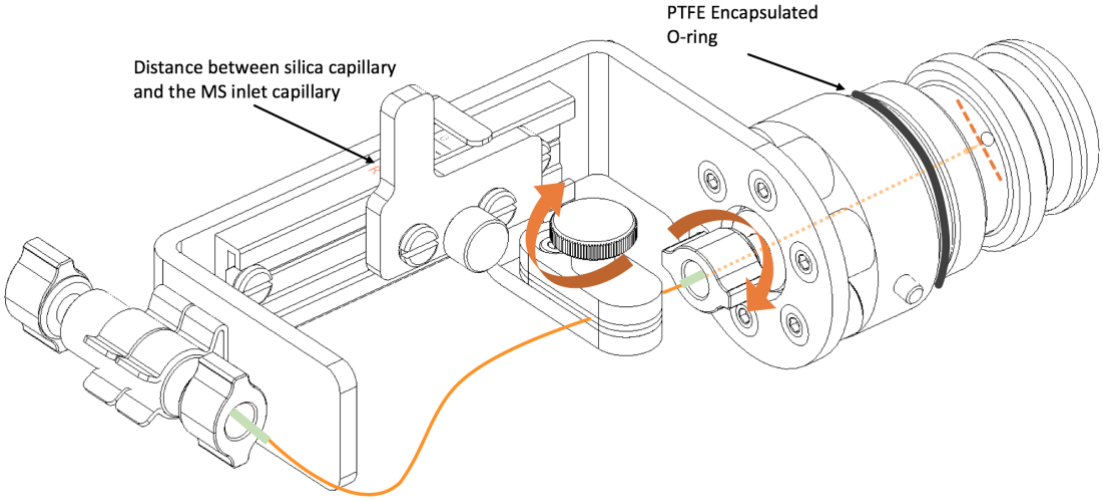


Figure 17. Positioning the silica capillary in the electrospray probe.

In order to reference the position of the silica capillary, unlock the capillary positioner, align the reference sign of the capillary positioner and the electrospray probe handgrip, and lock it in this position. Then unlock the capillary lock and the Upchurch fitting 1 (they should be unlocked for the installation of the silica capillary), slide the silica capillary through the capillary lock, and align it about the plane defined by the orifices of the electrospray probe, as shown in the figure 17.

Once the silica capillary is positioned, tighten the capillary lock to fasten the capillary, and gently tighten the Upchurch fitting 1.

***TIPS & TRICKS**. The fitting 1 should be sufficiently tight to maintain a sealed passage, but sufficiently loosen to allow for the silica capillary to slide through the Teflon capillary. To adjust the tightness of this fitting, unlock the capillary positioner lock and slide the capillary positioner. You should note some friction, but the silica capillary should be able to slide without buckling.*

Finally, insert the electrospray probe in the ionization chamber and lock it. Insert the flat end of the silica capillary into the vial holder and lock the fitting-2.

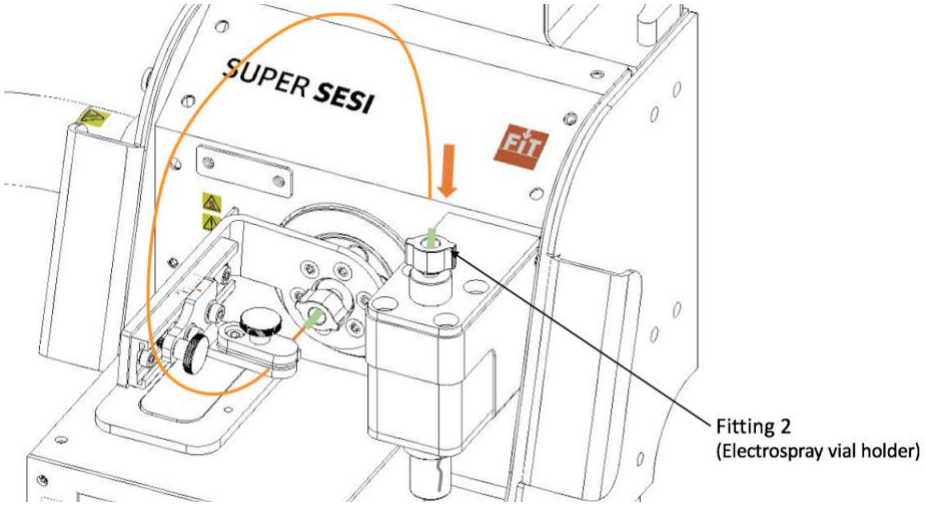


Figure 18. Inserting the silica capillary. *The figure may differ from the latest design.

IMPORTANT NOTE The back end of the silica capillary must be immersed in the electrospray liquid**.** For optimum performance, use only the sharp silica capillaries provided by FIT.

Operation

Adjusting the temperature

In order to control the temperatures, the SUPER SESI need to be powered, and the main switch must be in the ON position.

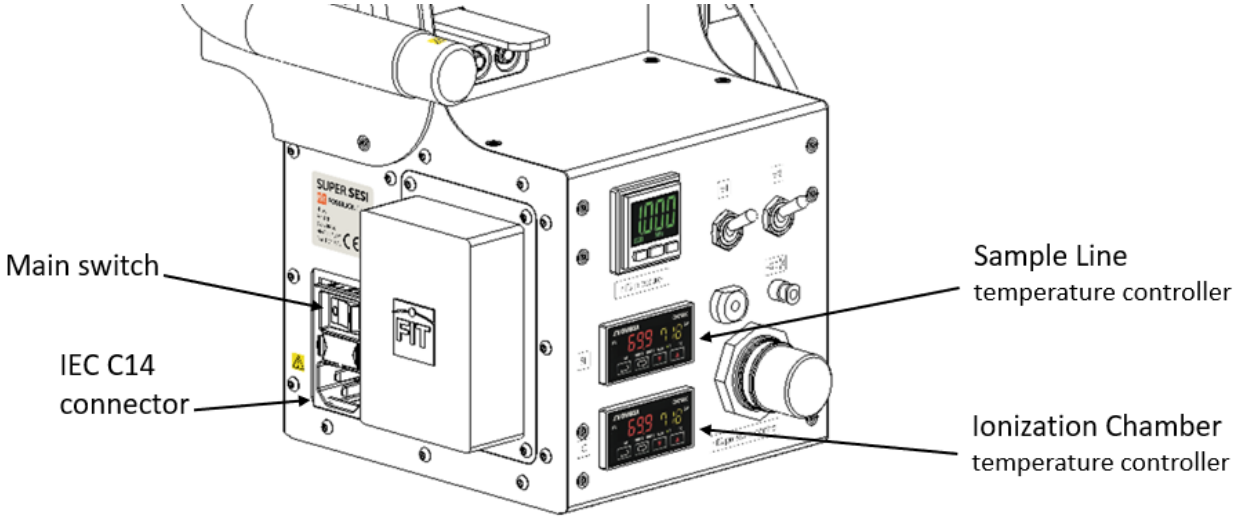


Figure 19. SUPER SESI main switch, IEC-C14 connector, and temperature controllers.

The SUPER SESI incorporates two independent Proportional Integral Derivative (PID) temperature controllers for the Sample Line and the Ionization Chamber. The electrospray probe is heated by the ionization chamber by thermal conduction. In order to change the temperature, follow the instructions below:

1. In the corresponding temperature controller, set the new value using the Up and Down Arrows (Figure 20). The new set point value is displayed at the right part of the controller. The new set value is flickering until the enter key is hit.

2. Press Enter. Once the new set point is entered, the PID changes the power accordingly to reach the new desired temperature.
3. Wait until the Actual Temperature (displayed at the left part of the controller) reach the chosen set point value.

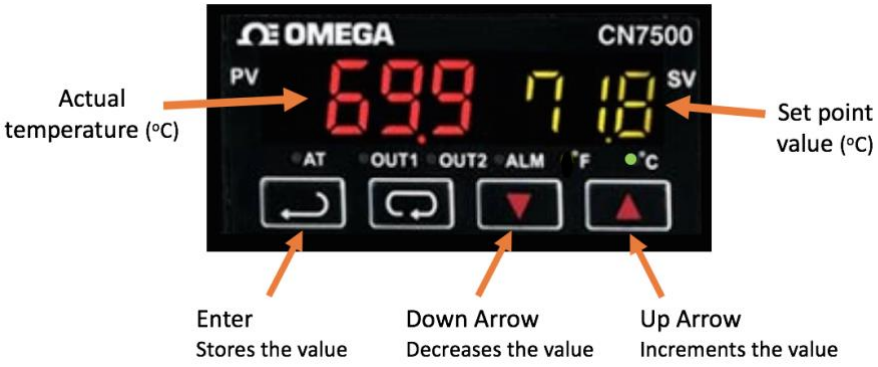


Figure 20. Temperature controller

IMPORTANT NOTE The SUPER SESI has certain thermal inertia. In particular, the electrospray prove might take some extra time to reach the new temperature. Although the reading of the actual temperature might indicate that the new set-point has been reached, reaching a uniform temperature might take some extra time. It is recommended to allow at least 30 min to ensure that the temperature in the ionization chamber and the electrospray probe is uniform when the temperature is changed.

CAUTION Hot Surface. At operating temperatures, the ionization chamber can be very hot. Be careful when setting a high temperature and allow enough time for the temperature to be reduced before touching any surface after reducing the set value to safe temperatures.

CAUTION The temperature control settings are locked to make sure that the SUPER SESI is operated always within safe operation parameters. Do not unlock the temperature control. Doing this could lead to temperature control errors and to serious damage to the instrument.

Adjusting the clean gas flow and the sample gas flow

INTERESTING NOTE *SUPER SESI uses the flows provided by the mass spectrometer. The SHEATH GAS is used to pressurize the vial to create the electrospray. The clean gas at the ionizer is provided by the AUX GAS, and it is used in the interface between the SUPER SESI and the mass spectrometer to focus the ions toward the mass spectrometer inlet. We call it clean gas because is filtered by an activated carbon filter just before the flow enters in the Ionization Chamber.*

Adjusting the flows of SUPER SESI is very important. The Mass Flow Controller (MFC) allows a very fine control of the flows in the ionization region, that results in very stable and optimized signals. The MFC is located at the end of the Exhaust pipe on the right side of SUPER SESI.

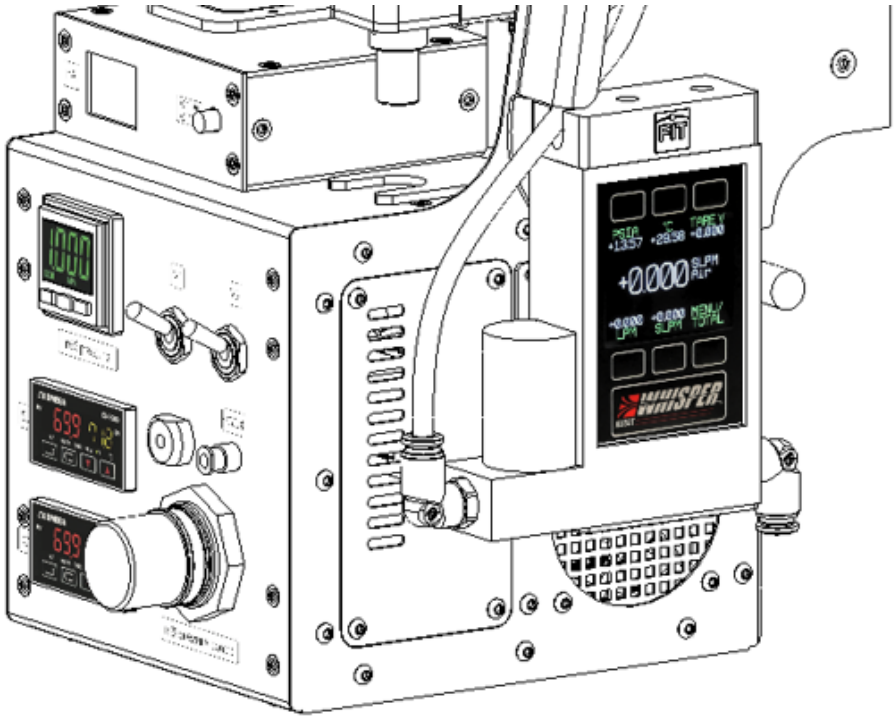


Figure 21. Mass Flow Controller location

The main screen displays live data for all flow parameters simultaneously. Live data is measured 1000 times every second but refreshed more slowly on the display. Press once the button above or below of any of the four flow parameters shown, to highlight its value in the center of the screen. Press the same button again to enter the engineering unit selection menu for that parameter. Press the large button with the Whisper logo to toggle the backlight on and off.

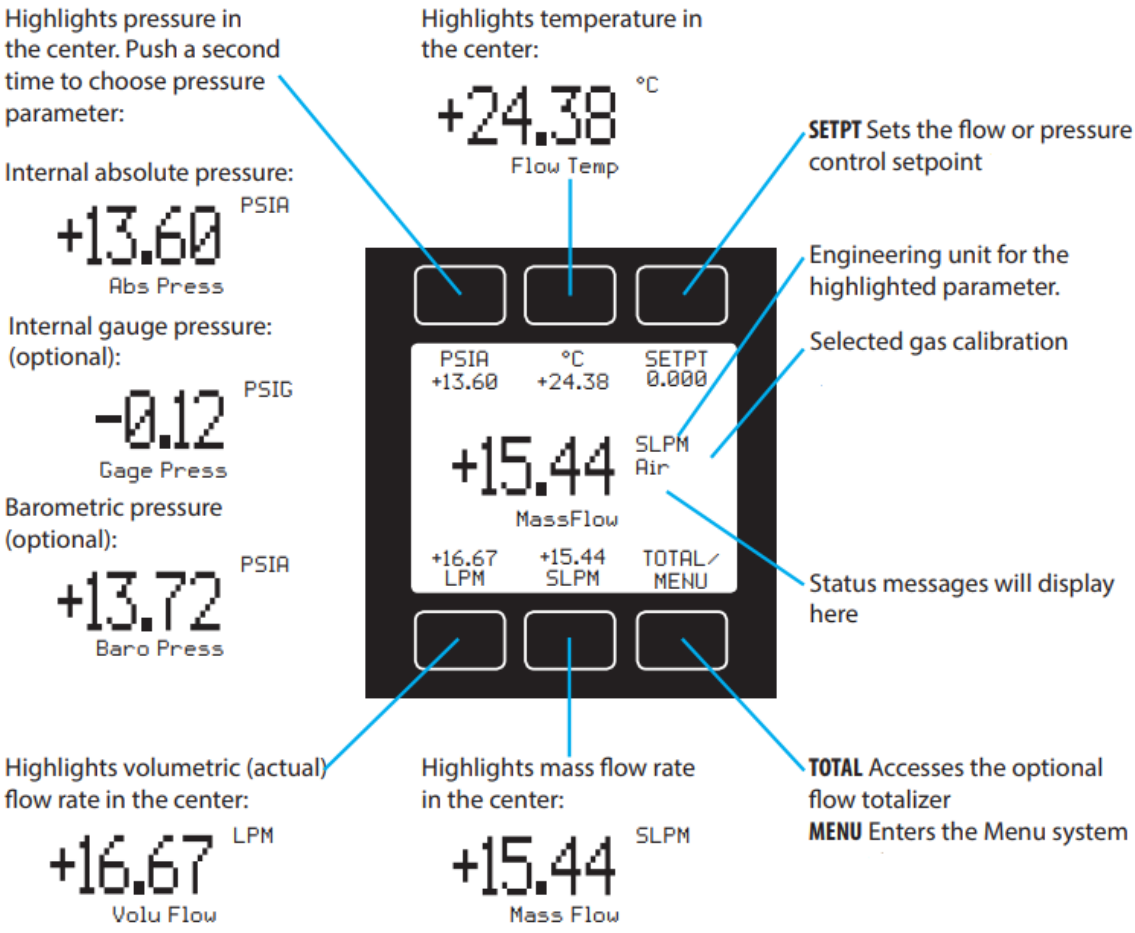


Figure 22. Mass Flow Controller Main Display

Operating the MFC is an easy task. For choosing your setpoint select **SETPT** from the Main Display to select your flow rate. Press **SET**, and the controller immediately adjusts to the new setpoint. You can monitor live reading of flow, pressure and temperature by viewing the screen. Readings are updated in real time.

First step is to adjust the clean gas flow. It is recommended to do it every day since the barometric pressure changes every day, and these changes affect the flow ingested by the MS and the rest of the flow rates. To adjust this gas follow these steps:

1. Provide AUX GAS to the SUPER SESI by the MS control software. Refer to the user manual of your mass spectrometer to set the flows.
2. Make sure that the setpoint at the MFC (right-up value) is higher than the flow value read at the MFC (center value) to ensure that the clean gas is not restricted by the MFC.
3. To fine tune the clean gas, you have to plug with gloved hands the Sample Line inlet. With this inlet closed, part of the clean gas is being ingested by the MS (always the same flow, with small variations depending on barometric pressure changes), and the rest goes to the MFC through the EXHAUST fitting. (The outlet of the MFC can be open to the room or it can be connected to an evacuation exit. Refer to section “Introducing samples and acquiring data” for more information).
   With the Sample Line inlet closed, adjust the clean gas by turning the gold needle valve or the white plastic knob (depend on the version, see Figure 3) until the value at the MFC display is between 0.2 and 0.5 LPM. Once this value is adjusted, uncover the Sample Line inlet. Now, a part of the clean gas is ingested by the MS, another part goes

through the EXHAUST fitting, and a third part goes to the Sample Line, to clean it and avoid entering contamination when no sample is introduced. The clean gas now is adjusted.

For adjusting the Sample Gas Flow, choose your setpoint value at the MFC: it will be the result of the sum of the measured clean gas (step 3) with the sample gas flow that you need to enter the ionization region (this flow depends on the application).

This fine control of the sample gas flow helps with the standardization when introducing samples: always the same sample flow enters the ionization region.

EXAMPLE After obtaining a 0.35 LPM clean gas flow value in step 3 and knowing that my analysis acquisitions require 0.2 LPM at most, I will fix a setpoint of 0.55 (0.35 + 0.2 = 0.55 LPM)

***TIPS & TRICKS**. In order to select an optimum sample gas flow for your application is recommended to try some acquisitions and select the most effective one.*

***INTERESTING NOTE** Taring your Mass Flow Controller is an important practice that ensures that it is providing the most accurate measurements possible. This function gives the flow controller a good zero reference for flow measurements. Taring can also be used to align the internal absolute pressure sensor with the barometric pressure reading.*

*When auto tare is -**ON**- your flow controller automatically tares its flow rate whenever it has a zero setpoint for more than 1.2 seconds. For manual tares, follow these steps:*

1. *Ensure that nothing is flowing through the device, usually by giving the controller a zero setpoint.*
2. ***MENU→TARE→TARE FLOW**. Flow tares should occur at the expected process pressure, as long as there is no flow.*
3. ***MENU→TARE→TARE PRESS**. Absolute pressure tares must be done with the controller open to atmosphere.*

When to tare:

- *After significant changes in temperature or barometric pressure.*
- *After dropping or bumping the flow controller.*
- *After installing the controller in a different orientation.*

Starting the electrospray

***INTERESTING NOTE** Before you can start the electrospray, the SUPER SESI must be properly coupled with the mass spectrometer, the temperatures must be stable, the flows must also be stable and the mass spectrometer must identify the ion source.*

Position the electrospray tip at a distance of 0 to 8 mm from the inlet capillary of the mass spectrometer. For this, unlock the capillary positioner lock, slide the positioner until the positioner mark is at the desired distance, and lock the positioner in the new position. The distance between the tip of the silica capillary and the inlet capillary of the MS is marked in the electrospray probe handgrip.

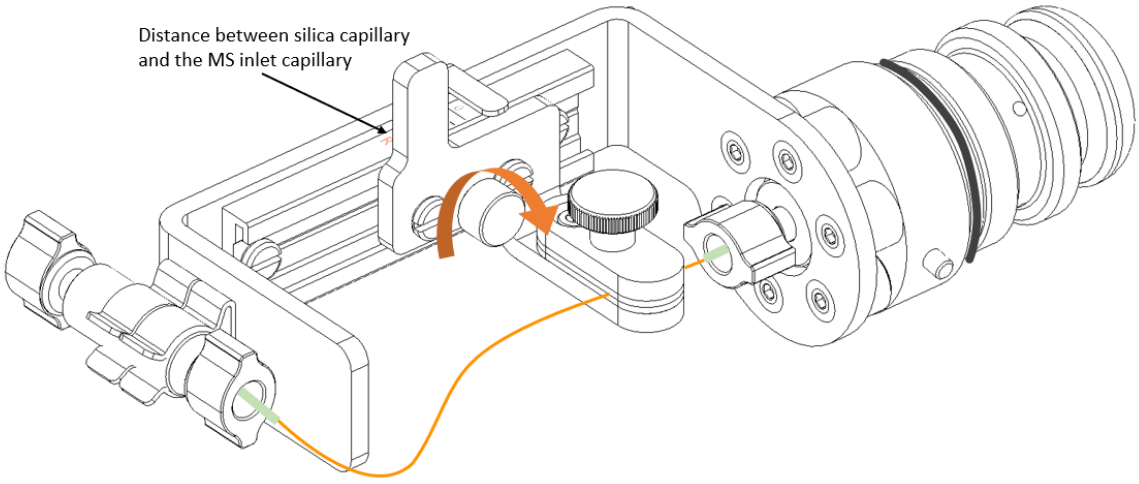


Figure 23. Detail of the positioning marks for the electrospray tip

CAUTION do not pass the zero. Doing so will introduce the silica capillary in the inlet capillary of the mass spectrometer. This can cause the silica capillary to break. Resulting debris could be ingested by the mass spectrometer and damage it.

Rise the ion source voltage. This voltage is provided by the mass spectrometer, and it is controlled by the mass spectrometer software. Refer to the user manual of your mass spectrometer to rise this voltage. The SUPER SESI requires a typical voltage between 2.5 kV and 3.5 kV. The SUPER SESI can be operated in negative and positive modes irrespectively.

INTERESTING NOTE *SUPER SESI uses the flows provided by the mass spectrometer. The SHEATH GAS is used to pressurize the vial to create the electrospray.*

To pressurize the electrospray vial holder first rise the SHEATH GAS on the MS-software to provide a continuous flow into SUPER SESI.

To rise pressure first open the valve 1 (up position), and set the pressure with the pressure regulator. Note that the high-pressure circuit has some inertia. You will see that the pressure display rises slowly when the regulator is actuated until it reaches a plateau.

Once the pressure is raised, the liquid starts to flow through the capillary. It usually takes about one to two minutes for it to reach the tip of the silica capillary. At this point, the electrospray will be formed. You will see a positive reading in your nano-amperemeter and the background spectra in your mass spectrometer.

INTERESTING NOTE *It is strongly advised to verify that the electrospray is stable. The stability of the electrospray can be evaluated by monitoring the intensity of the signals detected by the mass spectrometer, and by visually inspecting the nano-amperemeter. You can fine tune the electrospray voltage, the electrospray pressure, and the position of the tip to improve the stability. The stability of the electrospray can also be affected by the temperature of the ionization chamber. If this temperature is above the boiling point of the liquid, or if it is too close to it, the electrospray might be very unstable. If this is your case, lower it by 5ºC.*

Introducing samples and acquiring data

The data is acquired with your mass spectrometer. To acquire data, refer to the user manual of the mass spectrometer.

Use the inlet of the sample line to introduce the sample. Before starting the analysis, remove the Teflon cover to expose the inlet. For headspace analysis, you will need an auxiliary gas to carry the samples of interest towards the inlet of the sample line.

INTERESTING NOTE *In order to prevent any contamination to be accidentally ingested into the SUPER SESI and the mass spectrometer, samples have to be pushed through the inlet of the sample line. When no sample is being introduced, a fraction of the clean gas (provided by the AUX GAS) cleans the ionization region and passes through the sample line. This gas is outputted through the inlet of the sample line. In addition to prevent any unwanted contamination from entering in the SUPER SESI, this flow helps to desolvate and clean the sample line between measurements. To adjust the clean gas, refer to section "Adjusting the clean gas flow and the sample gas flow".*

CAUTION Hot Surface. At operating temperatures, the sample line can be very hot. Be careful when handling the inlet of the sample line and the protection cover is removed.

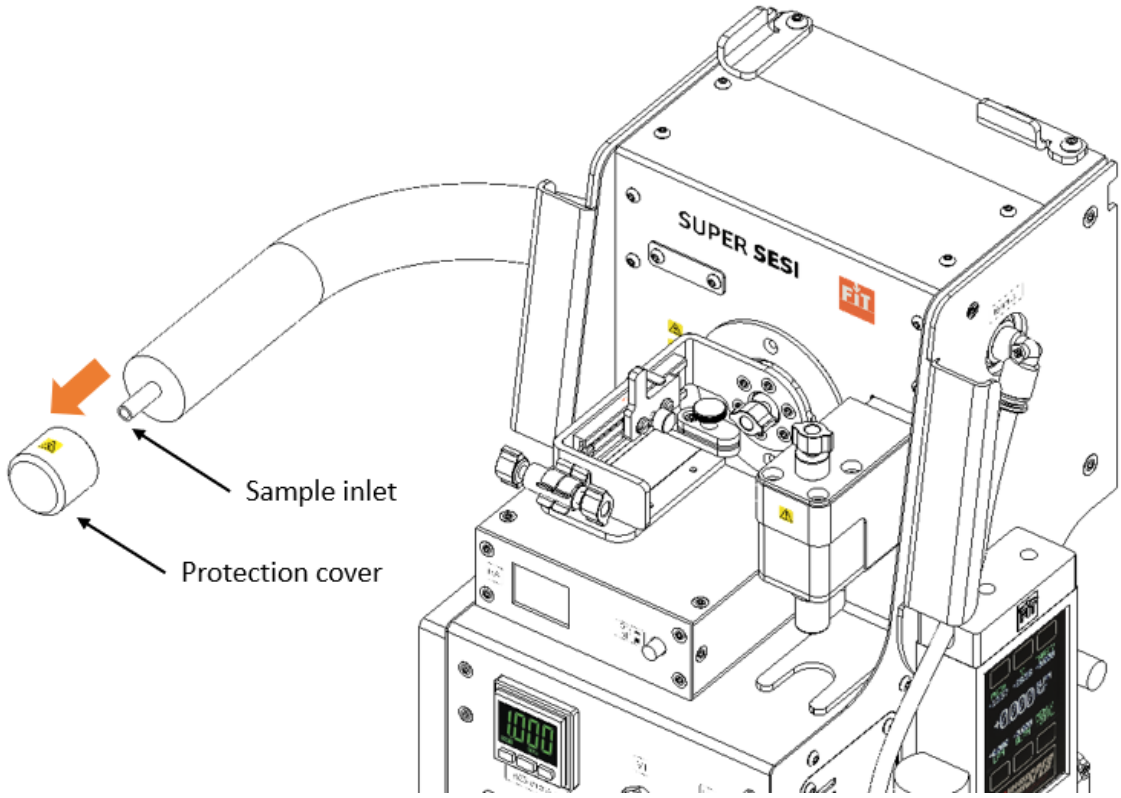


Figure 24. Removing the sample inlet protection cover

If you use hazardous samples, make sure that the fitting at the outlet of the Mass Flow Controller (Figure 3) is connected to a tube and is properly evacuated. Refer to the safety officer of your laboratory for this.

IMPORTANT NOTE If a tube is installed at the outlet of the Mass Flow Controller (Figure 3), please make sure that this tube has the biggest diameter possible*, so the pressure drop of that tube is as low as possible. This minimizes its effect to the overall pressure drop of the system, so that the introduction of samples can be done easily and smoothly.

*Use a proper union fitting reduction to connect this tube with the outlet fitting of the Mass Flow Controller (OD6mm).

Stopping the electrospray

Reduce the pressure of the electrospray to zero. For this, open the valve 1 and reduce the pressure down with the pressure regulator, or just opening the valve 2.

To stop the electrospray, set the voltage to zero. This voltage is controlled by the software of the mass spectrometer (ionization voltage). Refer to the user manual of your mass spectrometer.

To fully stop the electrospray, you can further set the MS to stand-by mode. Refer to the user manual of your mass spectrometer for this. Once the mass spectrometer is set to the stand-by mode, close the valve 1 (down) and open the valve 2 (up) to fully depressurize the electrospray vial holder.

If you plan to stop the SUPER SESI for a long period of time, it is advisable to pull out the liquid from the silica capillary. For this, close the valve 1 (down position), open the valve 2 (up position), create a negative pressure with the syringe, close the valve 2 (down position), and wait for the silica capillary to be emptied. When the silica capillary is emptied, the gas that comes into the electrospray liquid vial through the silica capillary forms visible bubbles in the liquid.

High temperature cleaning cycle

INTERESTING NOTE *In normal operation, the temperature of the ionization chamber is limited by the boiling point of the electrospray liquid. As a result, low volatility species tend to build up in this region, thus increasing background signals. In order to remove this contamination, it is advisable to rise the temperature from time to time so as to evaporate these contaminants and to sweep them away.*

If the spray is on, set the electrospray to zero. See section 'Stopping the electrospray´ for more details.

Pass a continuous stream of gas through the capillary. For this, lift the back end of the silica capillary within the electrospray vial holder so that it is not immersed in the liquid. After this, open the valve 2 (up position) and pressurize the electrospray vial holder with the syringe.

At the MS control software, adjust the AUX GAS to ensure that a fraction of gas is counter-flowing through the sample line, rise the SHEATH GAS, open the valve 1 (up) and close the valve 2 (down) to ensure a continuous supply of gas to the silica capillary. An overpressure of 500mBar is enough to maintain a stable flow of gas.

Set the temperatures of the ionization chamber and the sample line to the maximum allowed by their controllers (130ºC and 180ºC respectively) and let vapors be released for at least 30 minutes.

TIPS & TRICKS *A deep high temperature cleaning cycle can take up to one night.*

Once the cleaning cycle is completed, set the normal operation temperature and wait for about 30 to 60 minutes for the new temperatures to stabilize. In order to return to the original configuration, slide the back end of the silica capillary through the Upchurch fitting 2 and dip it in the liquid.

Removing the SUPER SESI from the MS

If the spray is on, set the electrospray voltage to zero. See section 'Stopping the electrospray´ for more details. If the MS is acquiring, set it to stand-by mode. Refer to the user manual of your mass spectrometer for this.

If the vial holder is pressurized, depressurize it. For this, make sure that the syringe is not connected to the syringe port and open the syringe valve 2 (up position).

Rotate the SUPER SESI locking levers 90 degrees to unlock the SUPER SESI onto the ion source mount assembly.

Carefully pull the SUPER SESI out of the ion source mount. Use the handles.

CAUTION Use the SUPER SESI handles to manipulate, lift or pull the SUPER SESI and to press it against the Mass Spectrometer. Do not grab the sample line or other parts to lift the SUPER SESI. Doing so could damage the sample line.

Turn the main switch off, and disconnect the power cord.

Maintenance

Required materials

It is recommended to have the following materials:

- Clean area to operate
- Ultrasonic cleaner
- N_2 gas supply to dry and clean
- Delicate task wipers (as Kimwipes)
- Cotton tipped applicators
- MeOH, H_2O (HPLC grade) and MeOH (HPLC grade)
- Tweezers and a tray
- Allen wrenches
- MS Syringe pump

Cleaning the vapor path

In the event that the SUPER SESI gets contaminated and the normal high temperature cycle is not sufficient to reduce persistent background contamination, the vapor path can be cleaned with a cleaning liquid. Use a mixture of methanol and water 50-50 HPLC grade.

INTERESTING NOTE *The vapor path consists of those parts that are exposed to the vapors. This includes the sample line, the ionization chamber, and the electrospray probe. These parts are made of Stainless Steel (SAE 316), polished (in the accessible surfaces) and coated with a passivation layer of amorphous silicon further functionalized to provide the most inert surface available.*

The electrospray probe can be cleaned with a swab or a Kimwipe soaked with liquid solvents. Additionally, it can be immersed in an ultrasonic bath for further cleaning. For this, remove the capillary and immerse only the part that is normally in contact with the sample vapors.

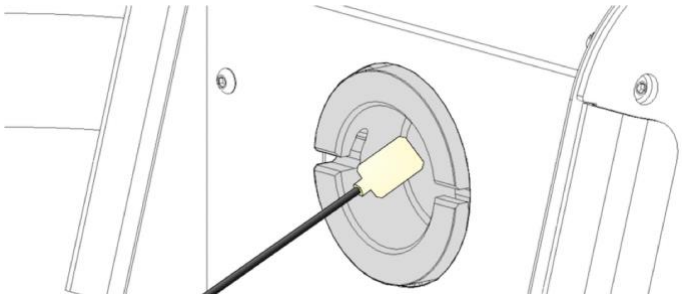


Figure 25. Cleaning the ionization chamber.

IMPORTANT NOTE Do not immerse the electrospray probe handgrip in the bath. This could damage the labels used to indicate the position of the silica capillary. After this process dry the probe with N_2.

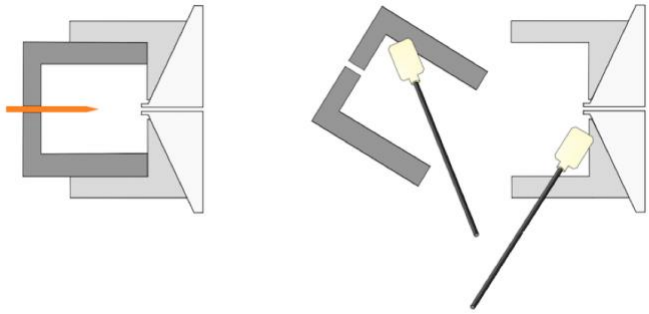


Figure 26. Cleaning the ionization chamber and the electrospray probe

To clean the sample line, remove the electrospray probe from the ionization chamber, introduce two Kimwipes in the ionization chamber, load a syringe of 10 ml with the solvents of choice*, and inject 10 ml of the cleaning liquid through the inlet of the the sample line. The cleaning liquid shall be collected by the Kimwipes in the ionization chamber. Replace the Kimwipe with two new dry ones, and inject 10 ml. Repeat as much as it is required.

To clean the ionization chamber, use a cleaning swab or a Kimwipe soaked with the cleaning liquid.

CAUTION When cleaning the vapor path with cleaning liquid, the SUPER SESI must not be connected to the mass spectrometer. Solvents entering accidentally in the mass spectrometer could severely damage the instrument.

CAUTION When cleaning the vapor path with cleaning liquid, the main power switch must be in OFF position, and the power cord must be disconnected.

CAUTION To introduce the cleaning liquid through the sample line, use a 10ml syringe to ensure that the maximum amount of solvent that can be spilled into the SUPER SESI is below 10 ml. If you spill the liquid or if you suspect that the liquid has been spilled inside the control module of your SUPER SESI, wait for it to dry before continuing. Spilling the liquid inside the control box of the SUPER SESI could damage it.

CAUTION During this operation, the ionization chamber and the sample line must be at room temperature. Wait for at least 30 min until all parts cooled down.

* ***INTERESTING NOTE*** *We recommend to use a sequence of these solvents: MeOH (gradient grade) - H_2O (HPLC) – MeOH (HPLC)*

Charging the Nano-amperemeter

CAUTION During this operation, the electrospray voltage must be set to zero.

The Nano-amperemeter is powered by internal rechargeable batteries. To ensure proper insulation, these batteries are encapsulated and cannot be replaced. When the batteries run out of charge, you will need to recharge them.

Disconnect the Nano-amperemeter from the SUPER SESI by pulling it gently, and connect the nano-amperemeter to its charger until charge is completed once the LED Status changes from red to green light. Once the Nano-amperemeter is charged, return to its original position.

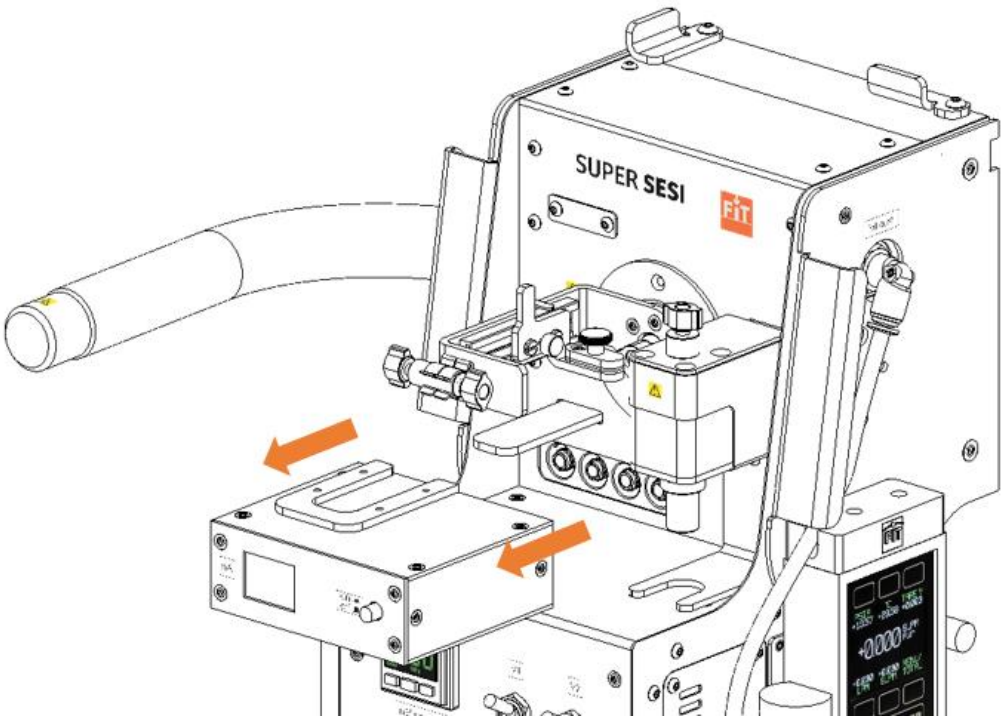


Figure 27. Removing the ampere-meter from the SUPER SESI

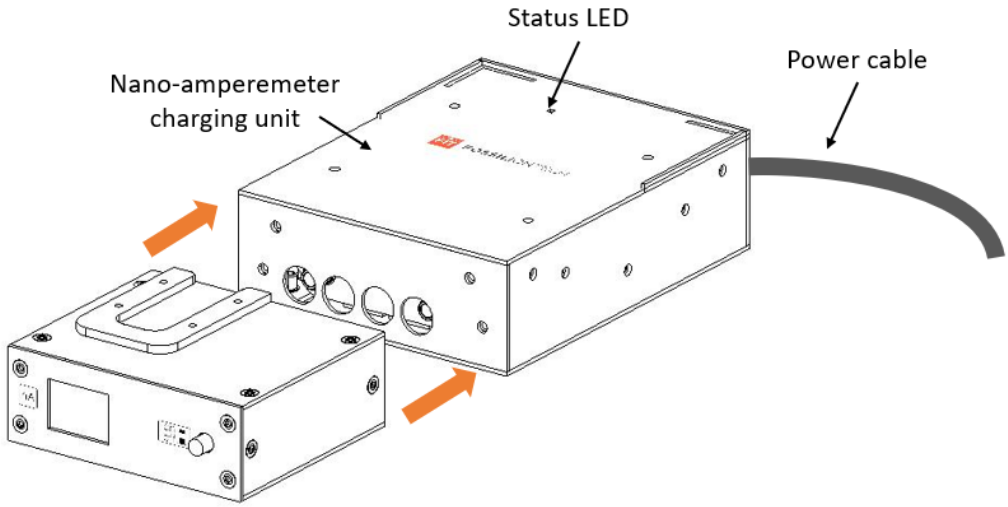


Figure 28. Connecting the nano-amperemeter to the charging unit

Resetting overheating protection relays

The sample line and the ionization chamber are protected against overheating with resettable thermal relays. If the overheating relay is activated, it will shut the corresponding heater down until it is reset by the user. In order to reset them, turn of the main switch, wait until the system cools down, and restart the power. If the problem persists, contact FIT for technical assistance.

Replacing the fuses

In order to change a fuse, first make sure that the power cord is not connected. Open the fuse holder, and replace the fuses. The fuse type is 'Quick Acting F LBC Fuse, 1A, 5x20 mm'.

Trouble-shooting

SUPER SESI is provided with two years of technical service. Please contact us via email or telephone if you need further assistance. In order to allow us to give you a better service, please specify the type of problem and we will get back to you as soon as possible.

Fossil Ion Technology S.L.

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email: info@fossiliontech.com,

SUPER SESI for Thermo, User manual; 2019

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