



Installation and Operations Manual

SICRIT® GC/SPME Module



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This manual must be to be stored carefully and must be at hand to any user of the described system. In addition to this manual, Plasmion GmbH provides further installation documentation e.g.:

- *Installation Manual for SICRIT® Interface TX3*
- *Hardware and Operations Manual for SICRIT® SC-30X Ionization Set*

Please check for further and updated versions of this manual on www.plasmion.de



Attention!

Please read and understand this manual before operating the described system. In case you discover obvious errors or contradictions for your product, contact the manufacturer before operating the system.

The content of this document has been checked thoroughly and is considered to be reliable. However, Plasmion GmbH does not take any responsibility for damages of foreign or own products and instruments resulting from improper use. Plasmion GmbH is not liable for consecutive damages resulting from integration and/or operation of its products in/with other systems. If the system is used in any manner not specified by Plasmion GmbH, the protection of the system could be impaired. Plasmion GmbH is not responsible for ignoring the outlined safety guidelines or the misuse of this system.

The technology and application of the system described in this manual is covered by patents and patent applications and is used under license.

All trademarks are property of their respective owners.

Declaration of conformity

The products outlined in this manual are engineered and build according to the requirements of electrical safety and health protection as outlined in the EC low voltage directive and electromagnetical compliance (EMC) directive. Any change or modification of any of the referred products, not verified by Plasmion GmbH, voids this declaration.

Plasmion GmbH certifies that the

SICRIT® GC/SPME Module

is designed and build to meet the EU Regulation No. 2014/35/EU (low voltage directive) and the Guideline 2014/30/EU (EMC Directive). The product fulfills the following safety requirements and safety standards for electrical measurement, control and laboratory use:

IEC/EN 61010-1:2010

IEC/EN 61010-2-010:2014

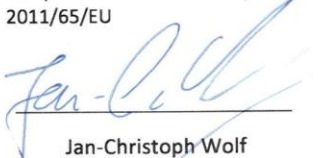
The product fulfills the following directives for electromagnetical compliance of electrical measurement, control and laboratory use:

IEC/EN 61326-1:2012, Class A

CISPR 11/EN 55011:2009

The product is compliant with RoHS-Guideline 2011/65/EU.



<p style="text-align: right;">Plasmion Simple Smart Sensitive</p>	
<p>EG Konformitätserklärung EC Declaration of conformity</p>	
<p>Name des Herstellers: manufacturer's name</p>	<p>Plasmion GmbH</p>
<p>Adresse des Herstellers manufacturer's address</p>	<p>Am Mittleren Moos 48 86167 Augsburg Germany</p>
<p>Der Hersteller erklärt, dass das Produkt The manufacturer declares that the following product</p>	
<p>Name des Produkts: product name</p>	<p>SICRIT GC/SPME Module</p>
<p>mit den folgenden EG Richtlinien und harmonisierten Standards übereinstimmt: is in conformity with the following EC Directives and harmonized standards</p>	
<p>Niederspannungsrichtlinie Low Voltage Directive 2014/35/EU</p>	<p>EN 61010-1:2010 EN 61010-2-010:2014</p>
<p>EMV-Richtlinie EMC Directive 2014/30/EU</p>	<p>IEC/EN 61326-1:2012, Klasse /class A CISPR 11/EN 55011:2009</p>
<p>Ergänzende Informationen: Complementary information</p>	<p>Das Produkt hält die RoHS-Bestimmungen ein The product is in conformity with RoHS Directive 2011/65/EU</p>
<p>Augsburg, Germany, 15.10.2018</p>	<p> Jan-Christoph Wolf Geschäftsführer Executive Director</p>

Safety Instructions

The following safety labels on the product and within this manual indicate safety risks and necessary precautions that arise during installation or from operating the products.





	<p>[Attention!], marks possible dangers to your safety and health.</p>
	<p>[Dangerous Voltage!], indicates parts and situations where there is the risk of exposure to dangerous electrical voltages.</p>
	<p>[Attention Hot Surface!], indicates potentially hot surfaces that might cause burning injuries if touched without protective gear.</p>
	<p>[Note], marks important information or advice, not related to safety issues.</p>

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1. Intended Use of the SICRIT® GC/SPME Module

The system described is intended for use only in laboratory and/or R&D environment. If the system is used in a way not specified by the manufacturer, misused or modified causing an infringement of the safety measures, Plasmion GmbH refuses any liability for consecutive damages in any form.

1.1 The SICRIT® Technology

Soft Ionization by Chemical Reaction In Transfer (SICRIT®) is a flow through ionization technique to be coupled with LC mass spectrometers featuring an API. Inside the ion source a cold plasma is used for ionization of the analytes passing through. This enables direct gas phase measurements as well as coupling with chromatographic systems as GC or HPLC. While the former one can be conducted with the GC/SPME Module as explained in this manual, the latter requires additional coupling modules.

1.2 The SICRIT® GC/SPME Module

The GC-SPME Module is designed for direct analysis of SPME fibers (SPME-SICRIT®-MS) as well as for coupling of the SICRIT® Ion sources with gas chromatographs (GC-SICRIT®-MS). Therefore, the module is directly connected to the SICRIT® Ion source, which is mounted onto the mass spectrometer (MS) inlet. Further details regarding installation of the ion source and mounting of the SICRIT® MS Interfaces are provided in the respective manuals. The SICRIT GC-SPME Module is empowered and controlled via the SICRIT® Control unit.

The module features a heating chamber for conditioning and desorption of SPME fibers, which can be injected either manually, using a 24 gauge manual fiber holder, or using a PAL® Autosampler. For coupling with GC, the GC column is introduced into the heated module where the analyte is transferred into the ion source. In both operation modes carrier gas (typically N₂) passes through the heating chamber and is then, together with the analyte, drawn into the SICRIT® Ion source and the MS inlet, respectively.

2. Technical Data

SICRIT® GC/SPME Module



Dimensions	110 x 50 x 50 mm
Weight	0.5 kg
Supply Voltage	Supply by SICRIT® Control unit SC-30: 24 VDC, 100 W
Electrical Connectors	1.4 m cable with plug for SC-30 Control unit
IP-Code	IP20
Protective Class	III
Operation Conditions	5 °C to 25 °C surface temperature 0 bis 80% RH (non condensating)
Maximum Operation Temperature	250°C (long term), 320°C (short term)
Storage Conditions	-5 °C to 50 °C 0 to 80% RH (non condensating)
Possible Carrier Gases	Air, N ₂ , CO ₂ , Dopants* Flow < 2.5 L/min *contact the manufacturer for further information

3. Setup and Operation Modes of the SICRIT® GC/SPME Module

The SICRIT® GC/SPME Module is intended for use in combination with GC devices (GC-SICRIT®-MS) as well as desorption unit for direct SPME-SICRIT®-MS measurements (Figure 1). The construction allows for mounting in different orientations, depending on one's requirements.

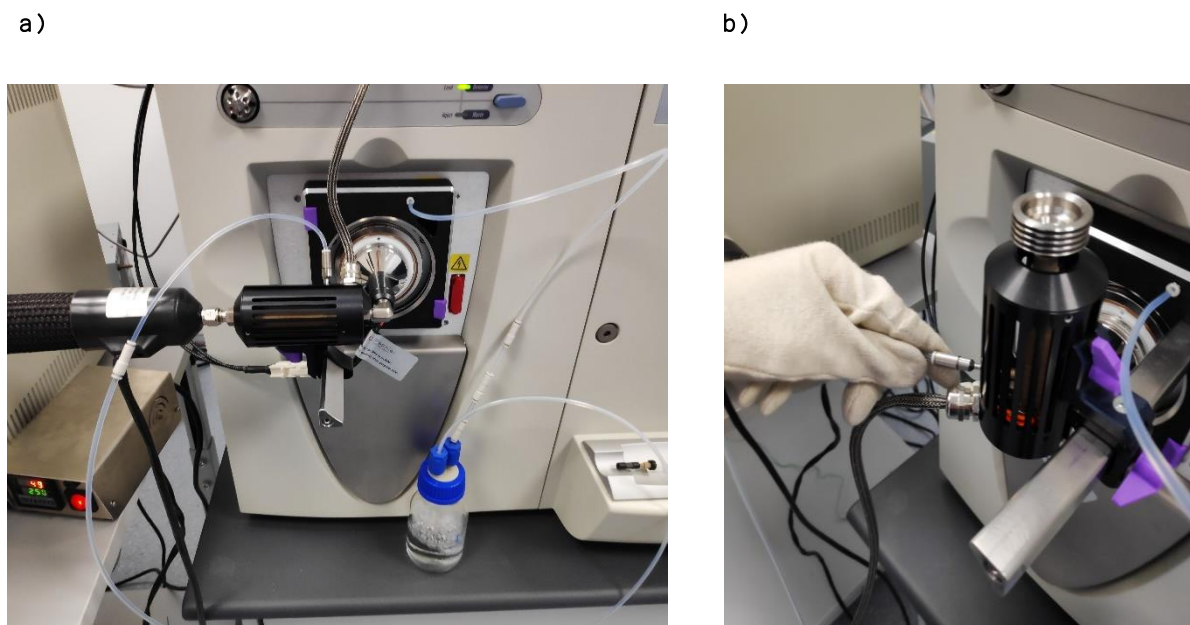


Figure 1: Installation of the GC/SPME Module in horizontal (a) for GC-SICRIT®-MS or in vertical (b) orientation for SPME-SICRIT®-MS.

For GC-MS measurements by means of SICRIT® Technology, the module acts as heating chamber where the analyte is transferred out of the GC column into the SICRIT® Ion source (see Figure 1 a).

For direct SPME-MS measurements, the module can be equipped with a PAL® Autosampler needle guide (Art.-Nr. 04-02-03), where the SPME fiber holder is held in position during conditioning and desorption steps (see Figure 1 b).

Inside the heating chamber of the module there is a GC liner modified with a Swagelok tube fitting (Art.-Nr. 04-02-02) for connection with the SICRIT® Ion source. This allows for transfer of the analyte out of the GC column or the SPME fiber, respectively, into the MS by the heated carrier gas, passing the ionization core inside the ion source.

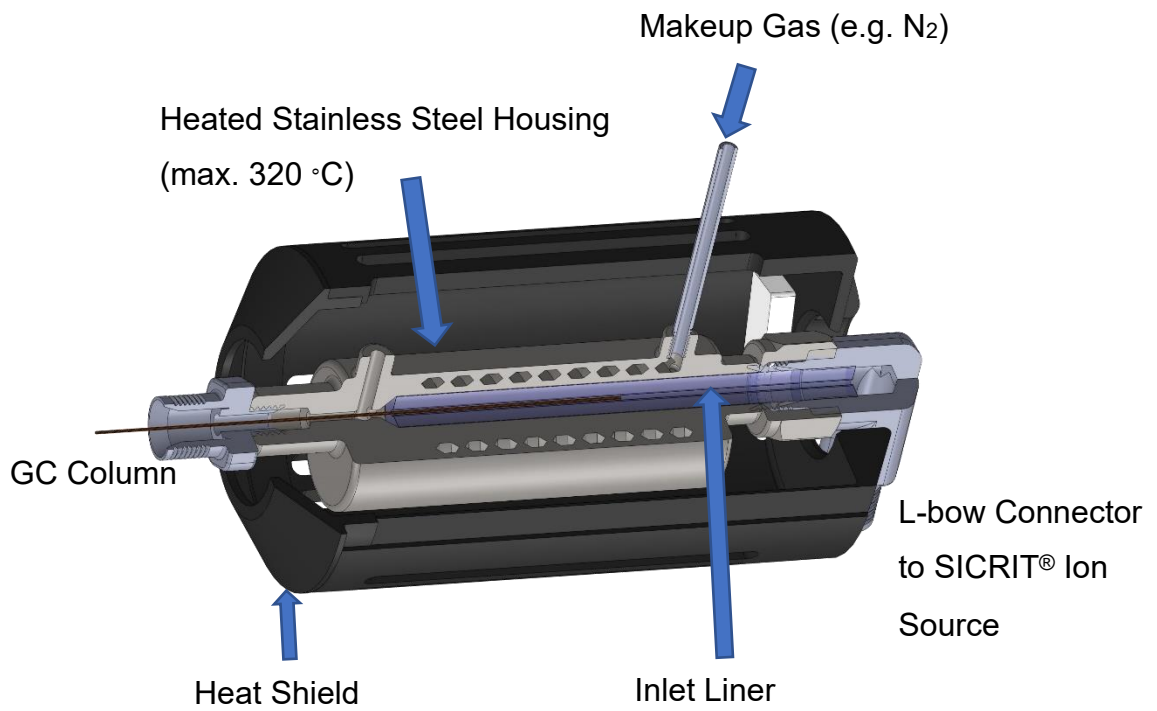


Figure 2: Components of the SICRIT® GC/SPME Module.

4. Installation and Operation of the SICRIT® GC/SPME Module

The SICRIT® GC/SPME Module is installed by mounting it on the respective SICRIT® MS-Interface, using the linear mounting rail included in the Module Kit. Each interface features two mounts to fix the rails for horizontal (GC application) or vertical (SPME application) orientation of the module (see Figure 3).

The following installation procedure is described exemplarily for a Thermo Fisher system and the corresponding SICRIT Interface TX2. However, it applies similarly for other systems and interfaces

4.1 Installation prerequisites

Before mounting the SICRIT® GC/SPME Module to your MS, install your specific SICRIT® MS-Interface and the SICRIT Ion source following the steps in the provided manuals:

- *Installation Manual for SICRIT® Interface*
- *Hardware and Operations Manual for SICRIT® SC-30X Ionization Set*

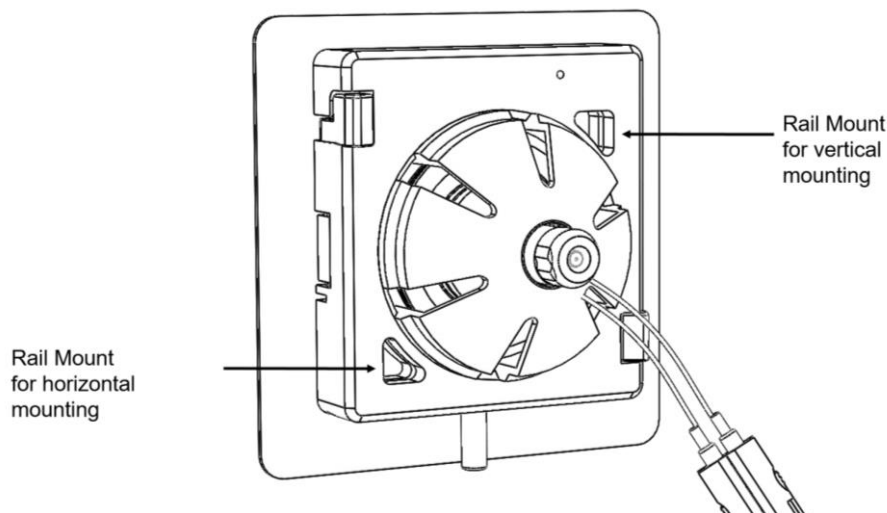


Figure 3: System with SICRIT® Interface TX2 ready for installation of the GC/SPME Module.

4.2 Initial operation of the SICRIT® GC/SPME Module:

The GC/SPME Module consists of a stainless-steel heating chamber surrounded by an aluminum housing. The carrier gas passes a spiral coil inside the heating chamber. To remove contaminations due to the manufacturing process, the heating chamber is thermal pre-treated by the manufacturer. This process can result in blooming patterns on the chamber surface.

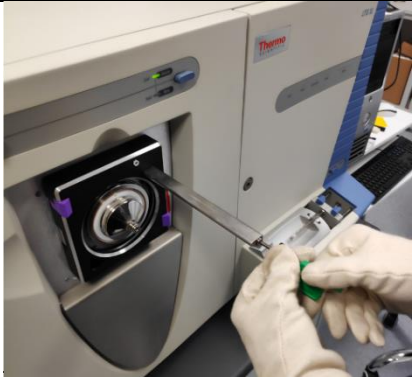
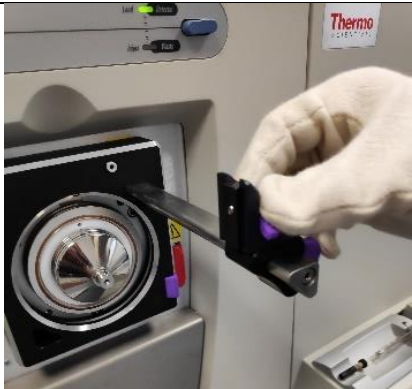
Before using the GC-SPME Module in analytical experiments, the module should be heated before insertion of the GC liner to remove deposited residues.




4.3 Mounting the SICRIT® GC/SPME Module in vertical position for SPME desorption measurements

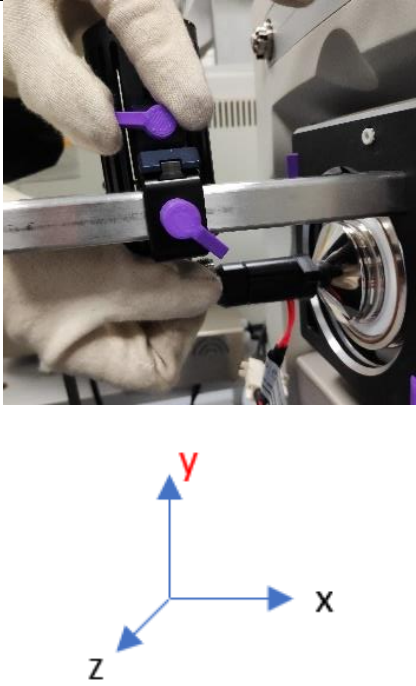


Generally, the mounting rail system allows for easy and flexible alignment of add-on modules for use with the SICRIT® ionization technology. A lock lever system enables fine adjustment of the modules in x, y, and z-direction.

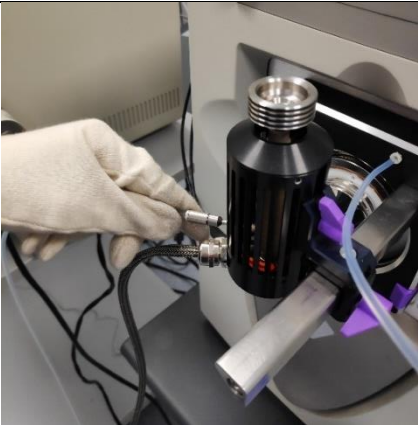
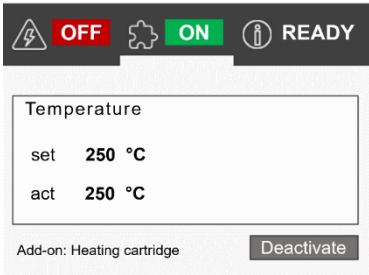
During the first installation of the system, some adjustments must be performed to meet the individual space and orientation requirements. These pre-settings can then be used for future installations.



Please follow the step by step instructions below:

1		<p>Insert the rail of the mounting holder system in the designated rail mount at the top right of the interface and secure it with the provided Torx tool.</p>
2		<p>Place the slider with lock lever on the rail. Assemble the GC/SPME Module with the holder system and make an initial rough adjustment. Slide the module on the rail and move it in place. Do not lock it yet!</p>

<p>3</p>		<p>Insert the provided inlet liner with the attached Swagelok L-bow connector (see Figure 2) from below into the module. Watch out to hold it in place since it might slide out and get damaged.</p> <p>Align the system and connect the Swagelok L-bow with the ion source.</p>
		<p>During vertical installation of the GC/SPME Module carefully insert the inlet liner to avoid mechanical stress on the glass and hold the liner tight until the tube fitting is fixed on the ion source.</p>
		<p>Only tighten the Swagelok connection hand tight to avoid mechanical stress on the inlet liner and the ion source.</p>

<p>4</p>		<p>Carefully tighten the purple levers and fix the y-positioning. You need to determine the y-position only at the first installation.</p> <p>Make sure all purple levers are locked hand tight before operating the GC/SPME Module.</p>
<p>5</p>		<p>Connect the GC/SPME Module cable with the SICRIT® Control unit.</p>
<p>6</p>		<p>When you switch the power on the SICRIT® SC-30 Control unit the next tab is activated. The display shows the actual temperature of the GC/SPME Module.</p>




<p>7</p>		<p>Connect the carrier gas with the module using the tube fitting inlet (Ø 3.2 mm). Gas supply can be provided by the aux gas supply of the MS using the one-touch fitting included in the SICRIT® Interface kit.</p> <p>To improve the ionization efficiency a gas humidifying system should be installed in closed sampling systems. The maximum humidity is 90% RH at 25 °C</p> <p>The delivered humidifier setup contains a wash bottle equipped with a 3.2 mm inlet tube (MS Interlock -> wash bottle) and a 4 mm outlet tube (wash bottle -> module). Please check for correct installation.</p> <p>Ensure that no droplets are transported into the source and that the humidity does not exceed the specified limits (see data sheet).</p> <p>Choose a carrier gas flow which is about 25% higher (usually 2 L/min) than the flow which is drawn in by the vacuum of the MS inlet. Excess carrier gas passes off the module's gas outlet port.</p>
<p>8</p>		<p>Choose the desired set temperature following your SPME fiber desorption protocol in the module tab of the SICRIT® Control unit and activate it to heat up the module.</p>

	<p>Attention!</p> <p>Avoid bending the cables. Do not place the cable over sharp edges or hot surfaces. Avoid strain on the cables and use strain relief measures.</p>
	<p>If you do not know the flow rate of your MS, the rate may be measured using a flow meter directly connected to the SICRIT® Ion source. If you need further technical assistance, contact Plasmion or its respective sales agent.</p>

4.4 SPME-SICRIT®-MS

For MS analysis of SPME fibers follow your established conditioning, enrichment and desorption protocol. The SICRIT® GC/SPME Module is designed for a **maximum operation temperature of 320 °C**. Please refer to the datasheets of your fiber supplier, to check the specifications and optimum temperature conditions.

The analyte is desorbed inside the module's heating chamber and transferred to the SICRIT® Ion source, where the analyte is ionized. This setup enables direct online SPME-MS analysis with high sensitivity.

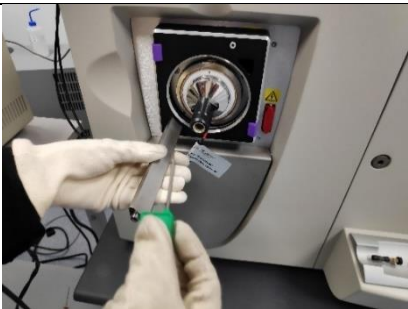
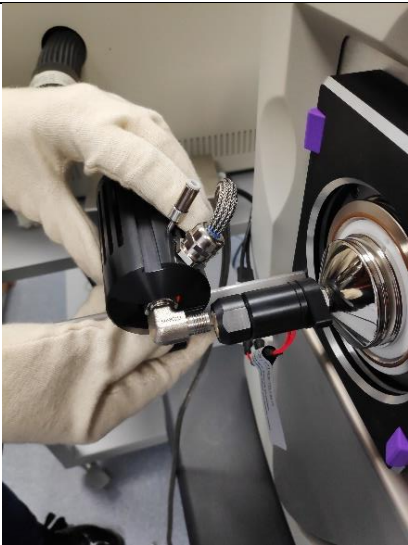

	<p>The PI heating control of the GC/SPME Module is designed for fast desorption ramps. By heating straight to maximum operation temperature (rt to 320 °C) there is the possibility of a short overheating. The system should reach stable temperature within 10 min. For best performance, set a value 10% below the desired value and increase it to the desired final temperature when the set value is reached.</p>
	<p>If you do not operate the GC/SPME Module continuously with heated carrier gas, the module must be flushed with heated carrier gas (250 °C) before every operation to remove residues and ensure a defined background atmosphere.</p>
	<p>Avoid direct contact to the surfaces of the GC/SPME module. The surface may be hot! After operation, let cool down the system before deinstallation of the module. Take special care when removing the liner, which can be very hot! after operation. Make sure to wear suitable protective gear (e.g. woolen gloves) when disassembling the module.</p>

4.5 Mounting the SICRIT® GC/SPME Module in horizontal position for GC-measurements

Generally, the mounting rail system allows for easy and flexible alignment of add-on modules for use with the SICRIT® ionization technology. A lock lever system enables fine adjustment of the modules in x, y, and z-direction.

During the first installation of the system, some adjustments must be performed to meet the individual space and orientation requirements. These pre-settings can then be used for future installations.

Please follow the step by step instructions below:


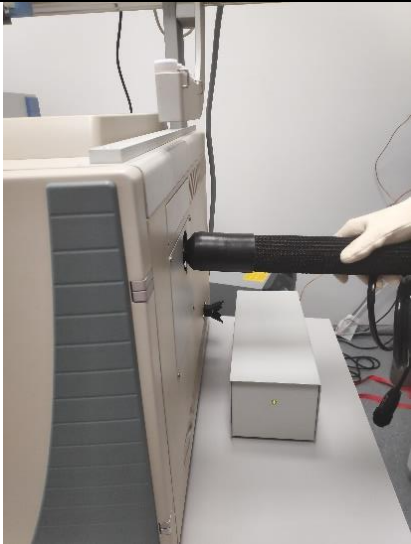
1		<p>Insert the rail of the mounting holder system in the designated rail mount at the bottom left of the interface and secure it with the provided Torx tool.</p>
2		<p>Place the slider with lock lever on the rail. Assemble the GC/SPME Module with the holder system and make an initial rough adjustment.</p> <p>Slide the module on the rail and move it in place. Do not lock it yet!</p> <p>Insert the provided liner with the attached Swagelok L-bow connector (see Figure 2) into the module.</p> <p>Watch out to hold it in place since it might slide out and get damaged</p> <p>Align the system and connect the Swagelok L-bow with the Ion source.</p>
		<p>During horizontal installation of the GC/SPME module carefully insert the inlet liner to avoid mechanical stress on the glass and hold the liner tight until the tube fitting is fixed on the ion source.</p>



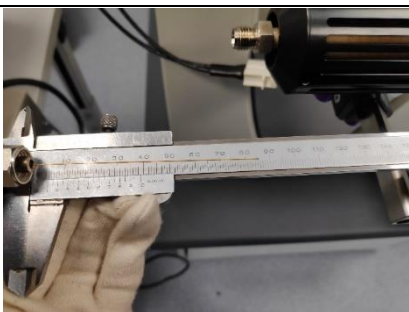
		<p>Only tighten the Swagelok connection hand tight to avoid mechanical stress on the Inlet liner and the Ion source.</p>
<p>3</p>		<p>Connect the carrier gas with the module using the tube fitting inlet (Ø 3.2 mm). Gas supply can be provided by the aux gas supply of the MS using the one-touch fitting included in the SICRIT® Interface kit.</p> <p>To improve the ionization efficiency a gas humidifying system should be installed in closed sampling systems. The maximum humidity is 90% RH at 25 °C</p> <p>The delivered humidifier setup contains a wash bottle equipped with a 3.2 mm inlet tube (MS Interlock -> wash bottle) and a 4 mm outlet tube (wash bottle -> module). Please check for correct installation.</p> <p>Ensure that no droplets are transported into the source and that the humidity does not exceed the specified limits (see data sheet).</p> <p>Choose a carrier gas flow which is about 25% higher (usually 2 L/min) than the flow which is drawn in by the vacuum of the MS inlet. Excess carrier gas passes off the module's gas outlet port.</p>



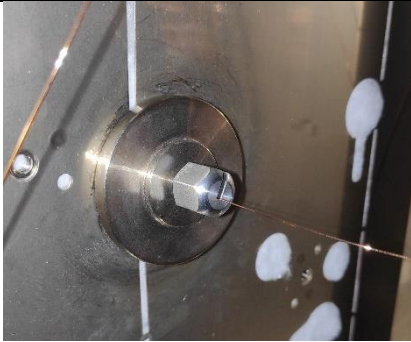

4.6 Connecting the SICRIT® GC Transferline to the SICRIT® Ion source

To connect your GC instrument to your LC-MS instrument you need to install a heated transferline.

Please follow the step by step instructions below:

1		<p>Prepare your GC to insert the transferline into the GC oven. Choose the right or left side depending on what fits best.</p>
2		<p>Insert the transferline into the GC oven. Make sure that the inlet of the transferline is placed as deep as possible into the oven to avoid cold spots.</p>

<p>3</p>		<p>Fix the transferline with the mounting disk. Depending on the length of your transferline you might need to install further mounting devices on the outside to avoid mechanical stress.</p>
<p>4</p>		<p>Unwind about 50 – 100 cm (depending on the transferline length) of the GC column. Pierce the column end through a 5 mm GC Septum. Move the septum down the column according to your transferline length + about 15 cm.</p> <p>To avoid blocking of the column cut 1–2 cm of the column end.</p>
<p>5</p>		<p>Feed the column carefully through the transferline until it sticks out of the end.</p> <p>To determine the later position of the column in the GC-SPME Module adjust the septum in the GC in the position that the column sticks out about 9 cm.</p>

<p>6</p>		<p>Retract the column back in the transferline to avoid damage during rough adjustments and positioning of the GC and transferline.</p> <p>After rough adjustment of the set up: position the GC column carefully into the GC/SPME Module. Make sure it is inserted into the modified Inlet liner.</p>
<p>7</p>		<p>Move the transferline along the GC column. Lock the Swagelok fitting hand tight Don't use any tools!</p>
<p>8</p>		<p>Push the GC column through the transferline until you reach the previously marked position by the septum and fix it with the fixing nut.</p>
<p>9</p>		<p>Your setup is complete.</p>

4.7 GC-SICRIT®-MS measurements

For GC-MS analysis follow your established GC protocol. For heating of the SICRIT® GC Transferline, Plasmion provides external heating controllers or specific connectors to use the auxiliary heating zone of the GC instrument. In case of external heating, please only use the delivered heating controller. Before starting your GC-MS measurements, please read the instructions given in the provided manual:

- *Short User Guide Heating Controller for SICRIT® GC Transferline*

Instructions for Thermo GC instruments providing auxiliary temperature zones:

For Thermo GC instruments Plasmion provides transferlines with connectors to use the GC auxiliary temperature zone for heating.

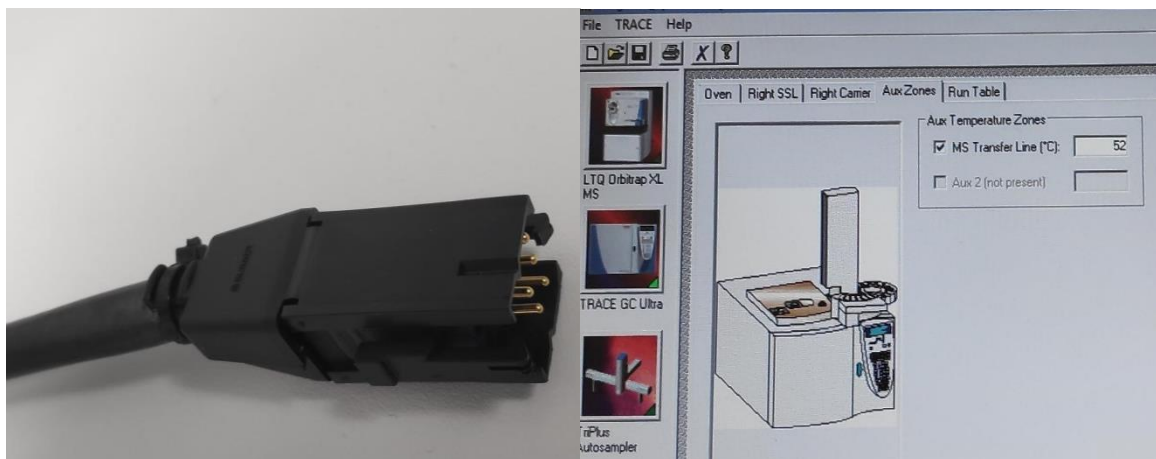
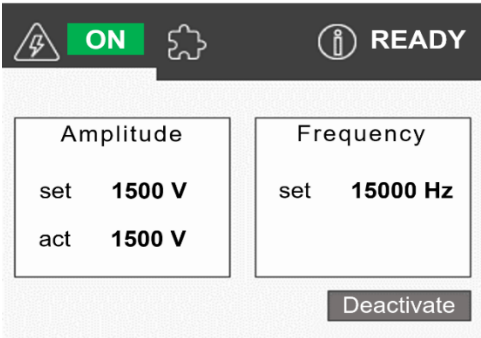
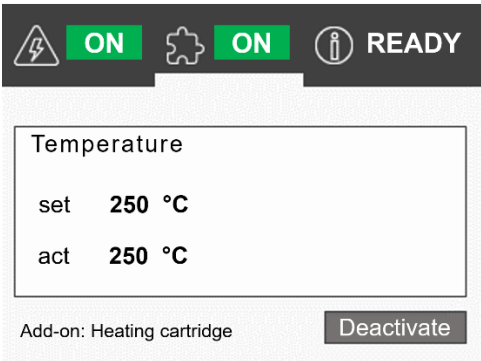
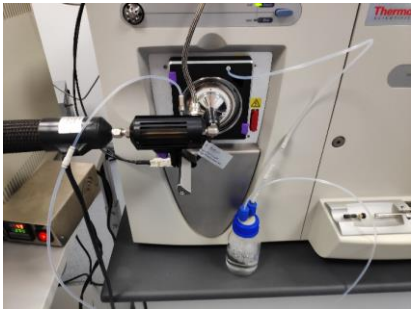



Figure 4: Connector for heating of the transferline by GC instrument (left), Excalibur software with settings for Aux Temperature Zones (right)

- Plug in the connector of the transferline into the respective socket on the right side of the GC instrument
- Press the AUX button of the keypad
- Choose *Temperature Zone* in the display menu and press the ENTER button
- Now the actual temperature of the transferline (shown as MS X-Line) is displayed and the set value can be adjusted using the keypad or using Excalibur MS Software.

<p>1</p>		<p>To prepare a GC-MS run turn on the voltage on your SICRIT® Control unit to start the plasma. Furthermore, turn on the gas flows, the heatings of the GC oven and inlet, the transferline and the GC/SPME Module</p>
<p>2</p>		<p>Wait until the transferline and the GC/SPME Module reach their final temperature and the temperatures are stabilized.</p>
<p>3</p>		<p>Make sure that a tiny excess of nitrogen flows out of the GC/SPME Module. Now all preparations are done. You can start your GC-MS runs.</p>

	<p>Before first use for analytical GC-MS measurements, please bake out the GC Transferline at the maximum operation temperature for > 30 min.</p>
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5 Service and Maintenance

The SICRIT® GC/SPME Module does not require any service or maintenance in routine operation. The only replaceable part is the modified GC liner inside the heating chamber, which must be replaced in case of contaminations, as you would usually do it, when using a liner in the GC injection port.



Do not open the module! In case of technical question or problems, please contact Plasmion or its respective sales agent.

5.1 Cleaning and decontamination

In an unplugged state, the surface of the module may be cleaned with a humid cloth. To remove contaminations a 50:50 methanol:water mixture can be used. If you use other solvents check the persistence of the anodized aluminum surfaces against these cleaning agents.

Any contact of the inner cables and components with liquids must be avoided! Before next operation, the module must be completely dry.

In case of chemical contaminations visible in your measurements you should clean or replace the inlet liner. Inlet liners with Swagelok tube fittings can be purchased as spare parts from the Plasmion (support@plasmion.de) or your respective sales partner.

5.2 Maintenance and service of the SICRIT® GC/SPME Module

The operational status of the module can be checked using the display of the control unit. After plugging in the cable of the module and turning on the control unit, the actual temperature of the module should be displayed and the temperature should be adjustable by the rotary encoder. If there is no or a lost connection the display will show [--] for set and actual value.

If there is an overheating of the system, the control unit will stop operation and display a warning.

Correct heating of the module should show a response of the actual temperature value within 20 seconds in the display of the control unit.

Do not operate the system if the housing shows obvious damages.

In case of unrealistic actual temperature values or fluctuations in the displayed values without heating of the module, there is a defect of the module or the control unit. Please contact Plasmion or its respective sales agent for further advice.

In case of abnormal heating of the module there is a defect of the controller. Please do not further use the module and contact Plasmion or its respective sales agent.

In case of noticeable overpressure of the carrier gas flow at the GC liner outlet, there maybe a clogging of the heating chamber due to high particle load in the carrier gas supply. If you do not get the contamination removed by manual cleaning, contact Plasmion or its respective sales agent.

6 Risk Avoidance or Residual Safety Risks

Regularly check the casing and cables for damages.

Ensure that access to the system is restricted for any unauthorized or untrained personnel.

Visually check the contact pins of the connectors for changes and damages.

Check if all connections are engaged before operating the system.

Never use the system without the connected ion source.

7 Operation with Potentially Harmful Substances

The risks of operation and handling of harmful or toxic substances that can be analyzed with the SICRIT® Ion Source fall to the operator. Stick to all safety guidelines and take all necessary precautions. Ensure that the substances introduced do not damage the system. The materials used in the SICRIT® Ion Source are PEEK, stainless steel and ceramics. Consider also the durability at higher temperatures.

The device itself does not contain harmful substances.

For recycling of the system, please contact the manufacturer.

8 Troubleshooting the SICRIT® GC-SPME Module

In the following section problems are discussed that might occur during operation of the system. If you do not feel confident to solve the problems after this brief troubleshooting guide, please contact Plasmion or its respective sales agent for further advice.



Please support us in the further development of the devices by sending us a short description of the error, its occurrence and/or the solution via email to support@plasmion.de we appreciate your efforts! We are further happy to receive feedback on the handling or operation, since we are always eager to improve our customers' experience.

8.1 Problem description: The TIC is spiking unexpectedly

Error: Fast spiking TIC (vertical flanks) signals indicate water droplets have been drawn into the system that change the ionization efficiency drastically.

- Make sure the distance of the humidifier and the source is large enough. If necessary, interate a small filter (e.g. paper).
- Make sure the temperature of the humidifier is lower than the gas temperature anywhere in the system to avoid condensation and subsequent droplet formation.

8.2 Problem: Error Message: THERMOFUSE TRIPPED

The GC/SPME Module is equipped with a resetting thermofuse to prevent the module from unintended overheating in case of hard- or software error. Tripping the fuse will result in switching off the heating and showing the error message in the display.

- Check, if the GC/SPME Module is freely positioned and the air can circulate. Don't cover the housing with thermal insulation material.
- If the thermofuse has been tripped, wait >1 hour before heating again for resetting of the fuse.

9 Spare Parts

Description	Part No.
Inlet liner	04-02-02
PAL needle guide	04-02-03
GC connector	04-02-04
Heated SICRIT® GC Transferline	05-01-00