

# **Operating Manual**

## **GC-APPI Inferface for:**

Q-Exactive<sup>™</sup> MS, Exactive<sup>™</sup> MS, and LTQ Orbitrap<sup>™</sup> Series MS

P/N: MC510000, P/N: MC511000, P/N: MC512000

**Revision D** 



### **GC-APPI-Interface**

© 2016 MasCom Technologies GmbH Original Operating Instructions

The manual describes the installation and operation of the GC-APPI-Interface for the Exactive and LTQ Orbitrap series MS systems.

**Revision History:** 

Revision A: published in November 2016 Revision B: published in January 2017 Revision C: published in April 2017 Revision D: published in March 2018

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#### Safety Instructions

Please read all of these instructions carefully before starting the installation of the GC-APPI-Interface and follow all recommendations.

- 1) Set the mass spectrometer to "off" mode.
- 2) Cool down the heated capillary of the mass spectrometer
- When removing the Ion Max housing with the ESI sprayer be aware that the heated capillary may still be hot.
- 4) **Caution**: don't squeeze your fingers when closing the cover of the interface
- 5) Please use nitrile gloves, or similar ones, to ensure your own safety and avoid contamination.
- 6) Always unplug the heater cable before detaching the interface from the mass spectrometer. Never connect the interface heater cables when the APPI interface is not attached to the mass spectrometer. There is a high risk of overheating because the fan is not running.

#### Caution: hot surface with high risk to get burned!

During operation, the Interface housing may warm up to 65 deg. C. When the interface is opened, the heater is automatically turned off but the source may still be very hot (up to 330 deg. C, depending on the operating temperature).



We also recommend watching the GC-APPI-Interface installation video to familiarize yourself with the procedure. This video has been provided on a USB stick with the interface. Alternatively, you can download it using the following link: <u>http://www.mascom-</u>

bremen.de/html/en/downloads/gc-appiinformation









#### Important information

Any changes or modification to the GC-APPI interface, undertaken without our consent, may endanger the functionality and/or safety, and will result in the termination of the declaration of conformity.

#### Tools required for source switching

Changing from ESI to GC-APPI on the Exactive or LTQ Orbitrap series instruments only requires a few tools:



Figure A: Special tool for removing the transfer tube



**Figure B:** Screwdriver for removing the spray cone and for tighten the plug on the APPI lamp



**Figure C:** 5/16-wrench for the dopant gas connection

Tools used for the GC column connection on the GC injector are described in the GC manual.



#### Installation of the GC-APPI Interface

Before installing the GC-APPI-Interface you have to remove the ESI sprayer. Make sure the mass spectrometer is set to "Off", and the heated capillary is set to room temperature (see Figure 1).

**Caution:** Removing the ESI sprayer while the instrument is on or in standby will load the default Tune file and eventually start the capillary heating.

Instrument Cor	htrol
Scan paramet	ters 🎗
History	$\rightarrow$
Scan range	150.0 to 2,000.0 m/z
Fragmentation	None
Resolution	35,000
Polarity	Positive
Microscans	1
Lock masses	Off

#### Figure 1: Instrument in "Off" mode

Disconnect the waste hose from the bottom of the lon Max source, as well as all gas, syringe, and electrical connections.



Figure 2: Instrument with ESI sprayer

Carefully remove the Ion Max source from the mass spectrometer interface and put it aside in a safe place.

Remove the Thermo ion sweep cone as shown in Figure 3, which is the removable spray shield that sits just over the capillary. Using a small flat-head screwdriver, loosen the set screws on the sides of the spray shield.





**GC-APPI** Interface

**Caution:** The *ion sweep cone* may be hot. *Be sure to cool down the heated capillary*.



Figure 3: Sweep gas cone of the Ion Max source

Sheath gas flow rate	20	
Aux gas flow rate	0	
Sweep gas flow rate	0	
Spray voltage (  kV  )	3.50	
Spray current (µA)		
Capillary temp. (°C)	35	
S-lens RF level	32.0	

Figure 4: Capillary Temperature setting

After removing the ESI sprayer, you can access to the transfer tube of the mass spectrometer. Remove the transfer tube with the special tool



Figure 5: Special tool for transfer tube removal





**GC-APPI Interface** 

Figure 6: Standard (A) and GC-APPI (B) transfer tubes

(see Figure 5) or with the 5/16 wrench as described in the operating manual of the mass spectrometer.

**Caution:** Exchanging the transfer tubes at high temperature is difficult and may damage them.

Replace the standard transfer tube (A) with the GC-APPI transfer tube (B) (see Figure 6), and place the Vespel disk on to the transfer tube (Figure 7). Make sure the disk is clean and free of dust.



Figure 7: GC-APPI transfer tube with Vespel disk

Bring the closed GC-APPI Interface in front of the Mass Spectrometer and arrange the cable for the APPI lamp close to its final position. Mount the interface with the ion source quick connectors.



Figure 8: Mounting of the GC-APPI-Interface

Open the interface and connect the cable to the APPI plug (see arrow on Figure 8).





Figure 9: Transfer line connection to APPI source

Next, attach the transfer line to the APPI-source.

Place the connector in front of the transfer line (Figure 10) and after inserting the GC column place the graphite ferrule with the conical side towards the transfer line.



Figure 10: Transfer line with connector



Figure 11: Transfer line with connector and ferrule

Attach the transfer line with connector and ferrule to the APPI source and only slightly tighten the connector (see Figure 11).





Figure 12: Transfer line connection to APPI source

Adjust the GC column before finally tighten the connector. It just has to protrude out of the inner source (see Figure 13a and b) by about 0.5 to 1 mm (check with a flashlight).





Figure 13a and b: GC column inside APPI source

Finally tighten the transfer line connector and press the transfer line against the connector before fixing it with the clamp (see Figure 15).





Figure 14: Transfer line in its final position



Figure 15: Transfer line fixing with clamp

Now insert the APPI lamp into the lamp holder and tighten the two screws to fix the lamp in its final position.



Figure 16: Positioning of the APPI lamp





**GC-APPI Interface** 

Figure 17: Fixing APPI lamp in its final position

The lamp safety switch must have contact to the opposite screw on the lamp holder.



Figure 18: Contact of safety switch of APPI lamp on lamp holder

Make sure the opposite screw activates the safety switch (see Figures 18 and 19).



Figure 19: APPI lamp with safety switch

When the lamp is inserted and fixed, connect the cable to the lamp and fix the plug with the screws.



Figure 20: Connecting the APPI lamp cable.





Figure 21: Fixing APPI cable.

Close the cover of the interface and make sure the protection shield completely covers the transfer line. When the cover is closed, check if the fan is running (air stream underneath the interface).



Figure 22: Closed APPI interface with dopant gas line connected

In case you want to use an optional dopant gas, remove the blind plug and connect the dopant gas line as seen in Figure 22. If you do not want to use any dopant gas, please check if the blind plug is properly tighten.

When everything is attached to the mass spectrometer, you can plug in the power line of





**GC-APPI** Interface

Figure 23: APPI source heater connection on Trace 1310 GC



**Figure 24:** APPI source heater connection to external power supply

the GC-APPI interface either into the GC (on Trace 1310 GC or Trace GC Ultra, see Figure 23), or into the optional power supply (P/N MC900010) for any other GC (see Figure 24). The operation of the external power supply will be described in the next section.



**Caution**: do not connect the heater cable without the interface attached to the mass spectrometer!

Please note: when connecting the heater cable for the first time, it will only be recognized by the GC if the cover is closed.

### **Operation of the GC-APPI Interface**

## Setting up the Instrument Configuration for Thermo GC Systems

Close all Xcalibur programs and open the Instrument Configuration. Make sure the GC devices are installed and select the Thermo Focus, Trace 2000, Trace Ultra or Trace 1310 GC from the list of GC devices.



Thermo Foundation Instrument Configuration	X
Device Types : Gas Chromatograph	
Available Devices:	Configured Devices: TRACE 1300 Series GC
Add >>	Configure
Done	Help

Figure 25: Xcalibur Instrument Configuration

If you are using an autosampler, please select it from the list too. Check that the communication of the GC (and autosampler) is correct and close the Instrument Configuration panel.

Start Xcalibur and wait for all instruments to initialize and to become "Ready for download".

#### Tune the MS

Turn on the MS and tune the mass spectrometer in APPI mode. Make sure the APPI lamp is on.

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Figure 26: MS tune when APPI lamp is off

If the APPI lamp is off, the noise level will be very high (see Figure 26).

When using a dopant gas you can tune the MS using the dopant gas' molecular ion (i.e. m/z 92 for toluene). Without a dopant gas the easiest way to tune the MS is to select a column-bleed ion like m/z 207 or similar.

Once the tuning is optimized, save the Tune file.



APPI source	
Sheath gas flow rate	8
Aux gas flow rate	0
Sweep gas flow rate	0
Discharge voltage (  kV  )	
Discharge current (µA)	10.00
Capillary temp. (°C)	280
S-lens RF level	32.0
Vaporizer temp (°C)	30
Ionization	APPI
Source Auto-Defaults	
Apply Help	Hot link

Figure 27: Example of MS tuning in APPI mode

#### Setup the Instrument Method

The following is valid when using the Thermo Scientific Trace 1310 GC. For any other GC, we refer to page 16 of this manual.





Open the Instrument Method editor from the Xcalibur Home page. In the editor, you will see the different instruments.

Select the Trace 1310 GC and setup the desired GC method. On the Aux Temperatures tab you have to enter the temperature of the transfer line and the temperature of the APPI source (listed as Aux. Heater 2, see Figure 29). Both temperatures should not differ a lot from each other. Typical temperatures are between 225 and 300 deg. C.

Please note:

the maximum temperature of the APPI source is 330 deg. C.



Switch to the MS to select or setup the appropriate MS method with the corresponding MS Tune file. If an autosampler is in use, also setup the right autosampler method.

p_toluene_ptv_15	m_col.meth - Thermo Xo	alibur Instrument Set	up	
File TRACE 1300	Help			
	X 8			
	Oven S/SL (front)	PTV (back) Aux.	Temperatures	Run Table
Exactive Plus - Orbitrap MS	Transfer Line 1: Aux. Heater 2:	✓ 270 °C   ✓ 270 °C		
TRACE 1300 Series GC				
TriPlus RSH Autosampler				

Figure 29: Aux Temperature for Trace 1310 GC in Instrument Method Setup

Save the Instrument Method and setup a sequence file.

#### Setup the Sequence File

From the Xcalibur Home page, select the Sequence editor to setup one or more measurements. For details, we refer to the standard MS operation manual.

#### **GC Setup**

We refer to the GC Manual, for setting up the GC and connecting the Helium.

#### Running a GC sample

When all instrument devices are "Ready for download", you can start the sample or sequence measurement from the sequence setup.



Acquisition Upti	ions		User: Thermo	
Instrument	Start Instrument			
ſ	Change Instruments In Use		Name Process	23
	Instrument	In Use	Start Instrument	
Start When Instrument N Start Up Shut Down Programs Pre Acquisi Post Acquis	Exactive Plus - Orbitrap MS TRACE 1300 Series GC TriPlus RSH Autosampler	Yes Yes Yes	Yes	
Run Sync		Cancel	Help	]

Figure 30: "Change Instruments in Use" of Xcalibur

Once you have set up the GC and autosampler as new devices in the instrument method, Xcalibur will ask to "Change the Instruments in Use". Figure 30 shows the setup for using an autosampler as start device. Without an autosampler no "Start Instrument" has to be selected, which will force the instrument devices to wait for the contact closure (see Figure 31).

nstrument	In Use	Start Instrument
Exactive Plus - Orbitrap MS IRACE 1300 Series GC friPlus RSH Autosampler	Yes Yes	

Figure 31: "Change Instruments in Use" without Autosampler

After clicking on OK, the instrument method will be downloaded into each device and when everything is ready, the autosampler will start with the injection. Without an autosampler the instruments will be pending with showing the message ,Waiting for contact closure'. This contact closure will be performed when pressing the start button on the GC after a manual sample injection. Make sure the check box 'Start When Ready' is activated (see red arrow on Figure 30).



#### **Final Remarks**

When everything is in place and the communication cables connected, close Xcalibur before switching on the GC and optionally the autosampler. After starting Xcalibur, all devices will be initialized and finally "ready for download" as shown below.



Figure 34 Xcalibur Home Page showing all devices "Ready for Download"

## Setup the Instrument Method when using a Trace GC Ultra

Setting up the GC and instrument method will be identical to the procedure using the Trace 1310 GC with one exception. The APPI source heater might not be available as Aux Heater and must be





Figure 32: External power supply for APPI source heating

set and controlled through the external power supply (see Figure 32). The control of the power supply is done by the 3 buttons on the front of the device (see Figure 33).

After turning the power on (switch on the back side of the power supply), the actual temperature



Figure 33: Control Panel of power supply

will be shown on the display. For setting the temperature, toggle with the "P" button until the display shows "SP". After releasing the "P" button, the display (after a few seconds) will show the set temperature. With the arrow up or down button, you can select the desired temperature.



**Caution**: never change any of the maximum temperature settings of the power supply. The factory setting for the maximum temperature is 330 deg. C. Increasing the maximum temperature is a misuse and may damage your system!

#### Switching back to ESI operation



Before starting with the dismantling of the GC-APPI interface turn off the mass spectrometer, the source heating (unplug the cable) and the transfer line heating.

#### Wait until everything is cooled down!

When all parts are at room temperature, you can uninstall the GC-APPI interface and re-connect the ESI onto the mass spectrometer. Do not forget to



exchange the transfer tube and the sweep gas cone.

#### **Final Disposal**

In case you want to finally dispose the GC-APPI interface, you can return it to us with a corresponding declaration of decontamination (form can be downloaded on our web site).



#### Troubleshooting

When the vacuum is not in the range of 10e-1 mBar, the position of the interface to the MS might be wrong and needs to be re-adjusted.

For that, you have to loosen the knurled screw



Figure 35 Knurled screw, which fixes the interface adjustment

(see Figure 35) and adjust the distance of the interface with the two Allen screws (see Figure 36) by watching the vacuum readback of the mass spectrometer. Turning the screws out will bring the interface closer to the MS and result in better vacuum.



Figure 36 Allen screws for interface adjustment



### Spare Part List

The following spare parts are available:

Part No.	<b>Description</b>
MC502240 –	$MgF_2$ Window
MC505000 -	PID-Lamp, 10.6 eV Kr
MC502610 –	Vespel Disk
MC502350 –	Gold Gasket for MgF2 Window
SG-073111 –	Graphite Ferrule for Transfer Line Connector ID 0.32 (pack of 10)
SG-073113 –	Graphite Ferrule for Transfer Line Connector ID 0.45 (pack of 10)
MC501730 –	Transfer Tube short (QExactive and Exactive Plus)
MC502220 –	Transfer Tube Long for all other systems
MC502450 –	Filter Cartridge, Triple Filter F301



#### **Optional Items for GC-APPI**

The following items are available as option:

#### Pneumatic Table for GC or HPLC

P/N: MC600200

- work surface size 60 x 80 cm
- lift able columns
- adjustable height from 76 to 121 cm
- integrated 3 IEC power plugs
- table on caster wheels for easy transport





## **EU-Konformitätserklärung**

**EU-Declaration of Conformity** 

Name des Herstellers: Name of manufacturer MasCom Technologies

Adresse des Herstellers: Address of manufacturer Sophie-Germain-Str. 4 28201 Bremen Germany

Der Hersteller erklärt hiermit, dass das Produkt The manufacturer herewith declares that the following product

## GC-APPI-Interface

Part-Nummer: MC510000, MC511000 und MC512000

Part number: MC510000, MC511000 and MC512000

alle anwendbaren Anforderungen folgender EG-Richtlinien erfüllt: fulfills all applicable provisions of the following EC Directives:

Richtlinie 2006/42/EG Directive 2006/42/EC Richtlinie 2004/108/EG Directive 2004/108/EC

Die folgenden harmonisierten Normen wurden angewandet: the following harmonized standards were applied:

EN ISO 12100:2010

Schutzanforderungen der Richtlinie 2006/95/EG gemäß Anhang I Nr. 1.5.1 der Richtlinie 2006/42/EG Safety objectives of Directive 2006/95/EC according to Annex I No. 1.5.1 of Directive 2006/42/EC

Bremen, Germany, 10. December 2016

MasCom

Geschäftsführer: General Manager

(Dr. Helmut Münster)