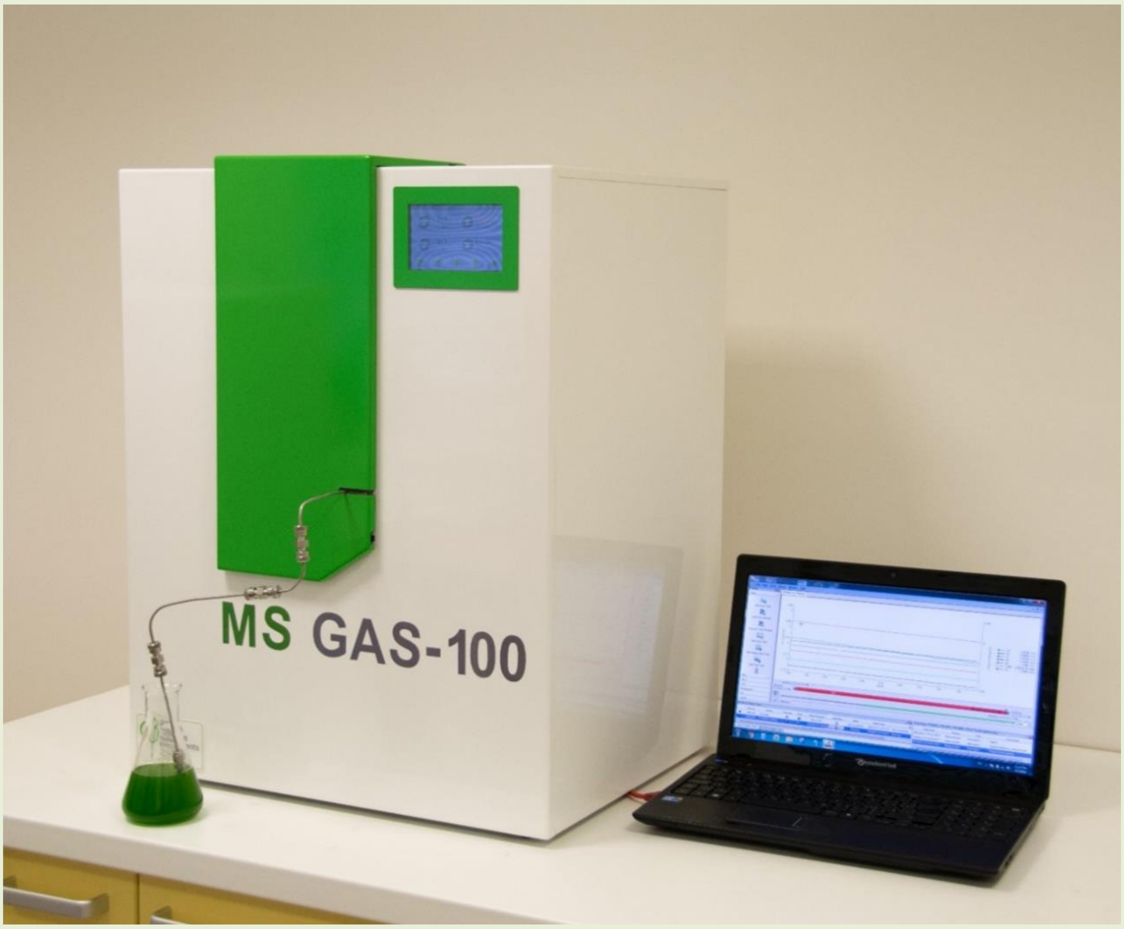
MS GAS-100

Mass Spectrometry Gas Analyzer

Instruction Manual

Please read this manual before operating this product



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The contents of this manual have been verified to correspond to the specifications of the device. However, deviations cannot be ruled out. Therefore, a complete correspondence between the manual and the real device cannot be guaranteed. The information in this manual is regularly checked, and corrections may be made in subsequent versions.

The visualizations shown in this manual are only illustrative.

This manual is an integral part of the purchase and delivery of equipment and its accessories and both Parties must abide by it.

1 TABLE OF CONTENTS

2 WARNINGS AND SAFETY PRECAUTIONS

PLEASE READ THE FOLLOWING INSTRUCTIONS CAREFULLY BEFORE TURNING THE MS Analyzer MS GAS-100 ON:

- Remove all packaging and transport protectors before connecting the MS GAS-100 to the power supply.
- Use only cables supplied by the manufacturer.
- Keep the device dry outside and avoid working in a high humidity environment!
- The manufacturer is not responsible for any damage due to improper operation!

GENERAL ELECTRICAL SAFETY GUIDELINES:

- Perform a routine check of the devices and their wiring.
- Replace worn or damaged cords immediately.
- Use appropriate electrical extension cords/power bars and do not overload them.
- Place the device on a flat and firm surface. Keep away from wet floors and counters.
- Avoid touching the device, socket outlets, or switches if your hands are wet.
- Do not perform any alterations to the electrical parts of the device or its components.

SAFETY PRECAUTIONS – INDIVIDUAL COMPONENTS OF MASS ANALYZER MS GAS-100:

- MPT 200 Digital Pirani Cold Cathode
    - **Strong magnetic field in the vicinity of the sensor head!**
    - **Danger of death for persons with cardiac pacemakers when the magnetic unit is disassembled.**
        - Maintenance personnel with active body aids (e.g. cardiac pacemarkers) should maintain a safe distance of at least 10 cm from the magnetic unit of the transmitter.
        - Disassembled magnetic unit must be kept away from computers, data storage media and other electronic components.

- Pumping system
    - *Diaphragm backing pump MVP 015*
        - **Risk of burns!** In continuous operation of the pump in the high pressure range, temperature can arise, which can cause burns from contact with hot parts.
        - Reduce the actual rotation speed to nominal speed (n_N = 1800 min^{-1}), when the intake pressure is permanently > 100 hPa (mBa).
    - *Turbomolecular pump HiPace 80 SplitFlow*

        - **Danger to health by hazardous substances during maintenance or installation!** Depending on the process vacuum pumps, components or operating fluids can be contaminated by toxic, reactive or radioactive substances. Wear adequate protective equipment during maintenance and repairs or in case of reinstallation.
        - **Risk of injury through hot surfaces!** Vacuum pump can become hot during operation. Allow the pump to cool before maintenance and repairs. If necessary wear protective gloves (directive EN 420).
        - **Risk of injury through sharp edges!** Rotor and stator disks of turbopumps have very sharp edges. Before any working wait for the complete standstill of the pump. Do not rich in the high vacuum flange. If necessary wear protective gloves (directive EN 420).

- Stirling cooler SC-UD08
    - **The unit is designed for use in ambient temperatures between 0 – 35 °C.** Internal damage could result from operating the module outside this temperature range.
    - **The cold side of the unit should be maintained between -100 °C and -20 °C.** Internal damage could result from operating the module outside this temperature range.
    - The Stirling cooler uses a **high pressure gas** inside, please contact PSI (Photon Systems Instruments) if you intend to scrap it.
    - **Placing the Stirling cooler into fire or excessive heat could cause expansion of the contained gas and explosion.**

Symbol	**Description**
⚠	Important information, read carefully
ℹ	Complementary and additional information

3 GENERAL DESCRIPTION

Compact benchtop gas analyzer with mass spectrometry detection **MS GAS-100** is designed for complex analysis of gases and volatiles including isotopes, solvents, and volatile organics.
The modular inlet system of **MS GAS-100** with membrane probe useful in a gaseous and liquid environment or needle valve and needle input for strictly gaseous samples allows determining of dissolved gases in different liquids like water solutions, cell suspensions, etc and also to perform various measurements in normal air conditions as well as in defined atmospheres (**Figure 1**).

Highly sensitive residual gas analyzer and the capability of real-time continual measurements enables to use the **MS GAS-100** in various biotechnological and bioenergetic applications like gas exchange studies of different plants, monitoring of nitrogen-fixing species (e.g. ethylene, N_2), measurements of biofuel productions (H_2, ethanol, hydrocarbons), analyses of isotopic distribution with or without labeled elements. Sensitive mass detection and effective water elimination based on unique cooler water freezing trap allow determining low concentrations of volatile water/air pollutants in environmental studies, to analyze general gas pollutants (CH_4, H_2S, NO_x, SO_2, CS_2, CO, …), to measure various volatile organics and solvents (benzene, toluene, acetone, …), to perform microbial gas exchange monitoring in cell suspensions, etc.

Figure 1: *MS GAS-100/F connected with PBR FMT 150*

This manual contains technical information about the standard version of **MS GAS-100**, a description of instrumentation delivered with the device, and step by step instructions for successful analyzes of gaseous compounds.

4 MS GAS TECHNICAL DESCRIPTION

MS GAS for determination and quantification of volatiles is equipped with a mass spectrometer based on open ion source, electron ionization of samples, two independent filaments, and a single quadrupole mass analyzer with variable mass ranges of 1-100, 1-200 and 1-300 amu. Two types of detectors, Faraday with sensitivity lower than 10 ppm and Secondary electron multiplier (SEM) with sensitivity lower than 100 ppb, are available for the instrument.

The highly effective vacuum pumping system is presented by the combination of a diaphragm backing pump and a turbomolecular pump with SplitFlow. Gas analyzer **MS GAS** is equipped with a high vacuum pressure sensor to determine total pressure in the vacuum chamber and safety inlet pressure sensor for mass spectrometer protection.

The thermostat heating element performs vacuum chamber baking for cleanup from undesirable impurities inside of the mass spectrometer.

Electronically controlled Stirling cooler ensures highly efficient suppression of water molecules background and significantly enhances ion source lifetime. The unique freezing system allows continuous weeks-long measurements. Moreover, water trap temperature can be defined by the user, which enables monitoring special volatile compounds.

Modular inlet options include permeable membrane probe, needle valve with manual control and needle input. Membrane probe inlet allows small levels of the dissolved species to pass through it, this type of inlet is suitable for liquid samples as well as for analysis in gaseous environments. The needle valve and the needle input are designed for direct measurements of volatiles in gaseous samples.

The intuitive setting of the device is available via an integrated TouchScreen including a manual mode with possibilities to change the temperature of the heating/cooling system and to open/close inlet valve, split-flow, and safety valve. The original firmware is also enhanced with automatic functions of valve opening/closing dependent on the signal from the safety pressure sensor after the device is turned on. Mass spectrometer tuning and acquisition of measured data are performed by software Quadera permitting user-defined protocol writing for numerous determinations of desired analytes.

4.1 STANDARD CONFIGURATION OF MS GAS-100 WITH SEM DETECTOR

The standard version of gas Analyzer **MS GAS-100 with SEM detection** consists of the following components:

1) Pumping system: Diaphragm backing pump MVP 015 – 2 DC combined with turbomolecular pump HiPace 80 SplitFlow for achieving high vacuum (HV) $\pm 10^{-6}$ Pa ($\pm 10^{-8}$ mBa)
2) Residual gas analyzer PrismaPlusTM QMG 220 M1 based on open ion source with electron impact, two independent filaments from yttriated iridium, Mass Spec with defined mass range and following detectors:
    a) Single quadrupole mass analyzer with mass range: 1-100 amu
    b) Farraday cup (sensitivity <10 ppm)
    c) Continuous secondary electron multiplier (sensitivity <100 ppb)
3) Pressure sensors:
    a) High vacuum (HV) pressure sensor to determine total pressure in vacuum chamber MPT 200 Digital Pirani Cold Cathode
    b) Medium vacuum (MV) pressure sensor to determine capillary inlet pressure PPT Digital Pirani
4) Solenoid valves:
    a) Inlet valve
    b) Split flow valve
    c) Safety valve
5) Thermostat heating element which can increase the temperature up to 90°C
6) Electronically controlled Stirling cooler SC-UD08 with adjustable temperature up to -80 °C
7) Mini PC (Raspberry pi_RBI) with touchScreen integrated into the body of the instrument
8) Original PSI firmware with manual and automatic mode
9) Notebook with Quadera software for device tuning and acquisition of analytical data

A possible version of MS GAS-100 inputs is membrane probes of different lengths, manual needle valve, and needle input with an adapter for 1/8” tube (**Figure 2**).

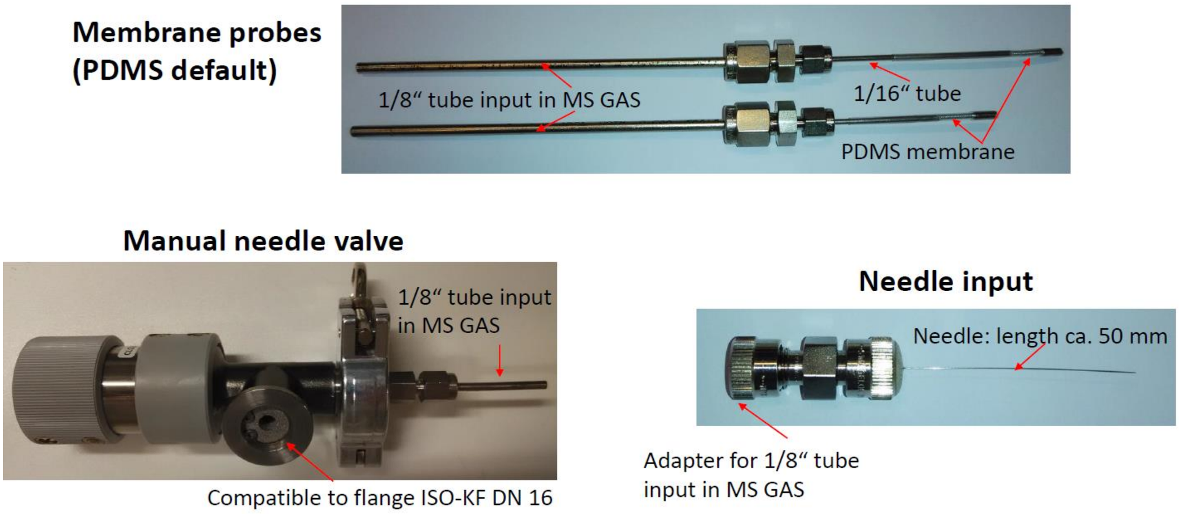


***Figure 2**: Possible MS GAS Inputs*

⚠	Check the contents of the package and compare it with the enclosed standard package list (see below).

4.2 LIST OF STANDARD MS GAS-100 COMPONENTS (FIGURE 3)

1. Gas Analyzer MS GAS-100 with integrated TouchScreen and power cord
2. Notebook and power supply with power cord
3. Ethernet cord _RED for connection of notebook to the MS GAS-100
4. Ethernet cord _GREY for connection of MS GAS-100 to the internet
5. Short membrane probe (polydimethylsiloxane, PDMS) – total length 15 cm
6. Long membrane probe (polydimethylsiloxane, PDMS) – total length 30 cm
7. Polydimethylsiloxane (PDMS) membrane (3 m)
8. UT 1/8” x 1/8” connector – 1 piece
9. CD with Quadera Software for Mass Spectrometer tuning and data acquisition
10. Filter_RITTAL
11. Replacement Fuses

***Figure 3**: Standard components*

4.3 OPTIONAL ACCESSORIES/COMPONENTS

4.3.1 NEEDLE INPUT

The needle (approx. 50mm length, 0.1mm OD) is fixed in UT 1/8“ x 1/8“ connector for easy connection into MS GAS inlet (**Figure 2**).

4.3.2 NEEDLE VALVE KIT

Gas dosing valve with separate shut-off valve and manual control, adapter KF 16 x 1/8, a stainless tube of 1/8“ OD and length 10 cm, clamping ring, elastomer seal and stainless O-ring (**Figure 4**).

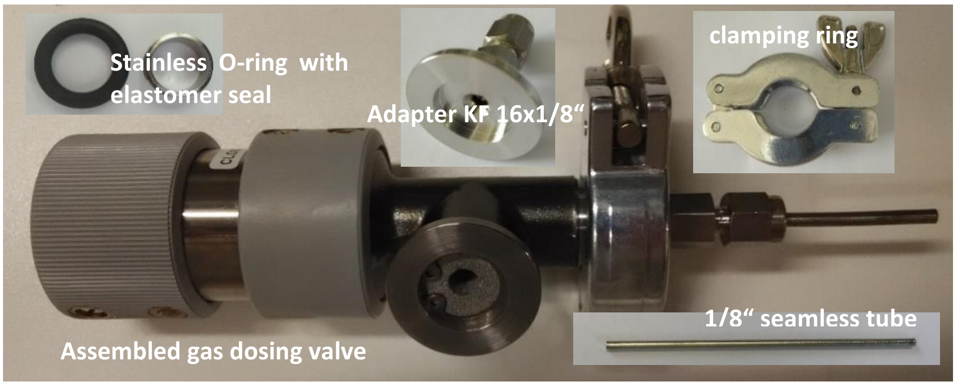


***Figure 4**: Needle valve kit*

4.3.3 MEMBRANE PROBE KIT

Small push spring – 10 pcs, stainless end cap – 10 pcs, the membrane of length 10 m (polydimethylsiloxane, PDMS), a seamless tube of 1/8“ OD and length 0.5 m – 2 pcs, a seamless tube of 1/16“ OD and length 0.5 m – 2 pcs, coupling reduction 1/8“ x 1/16“ OD with ferrules (Let-Lock) – 5 pcs, capillary cutter (**Figure 5**).

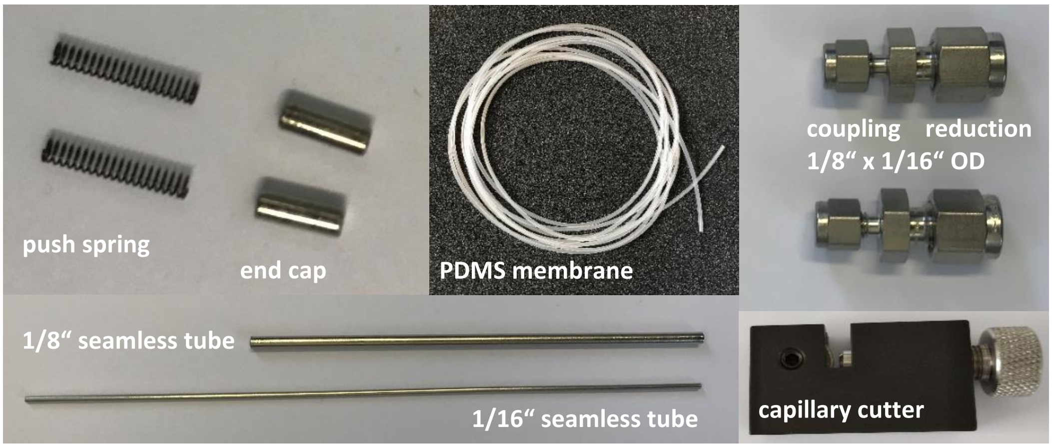


Figure 5: *Membrane probe kit components*

4.3.4 SPECIAL COMPONENTS KIT

Set contains special reductions and spare parts for replacement at inlet part before inlet valve: filter element - 1/4“ OD VCR x VCR, Ni gasket – 5 pcs, UT reduction 1/4“ x 1/8“OD – 3 pcs, UT reduction 1/8“ x 1/16“OD – 3 pcs.

4.3.5 SET FOR MODULAR INLET PREPARATION

UT adapter 1/8“x1/8“ OD – 5 pcs, seamless tube 1/8“ OD, length 0.5 m – 5 pcs, capillary cutter (**Figure 6**).

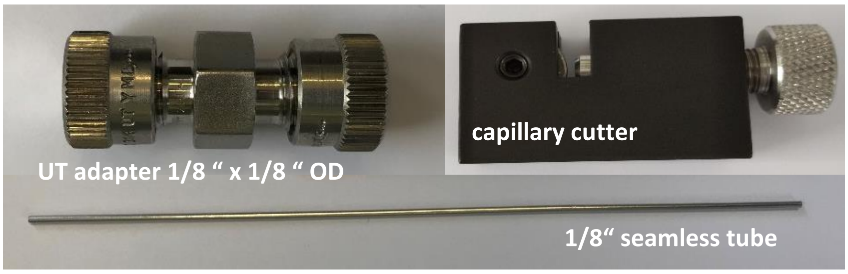


Figure 6: *Components in the set for modular inlet preparation*

4.3.6 PLANT CHAMBER KIT

Airtight chamber – maximum height of plant 20 cm, reduction for smaller plants – maximum height 10 cm, pressure relief valve, integrated pressure valves for replacement of the atmosphere, without light source (**Figure 7**). A manual needle valve is used as the sampling port (**Chapter 4.3.2** , **Figure 4**) – needle valve is not included in the chamber kit.

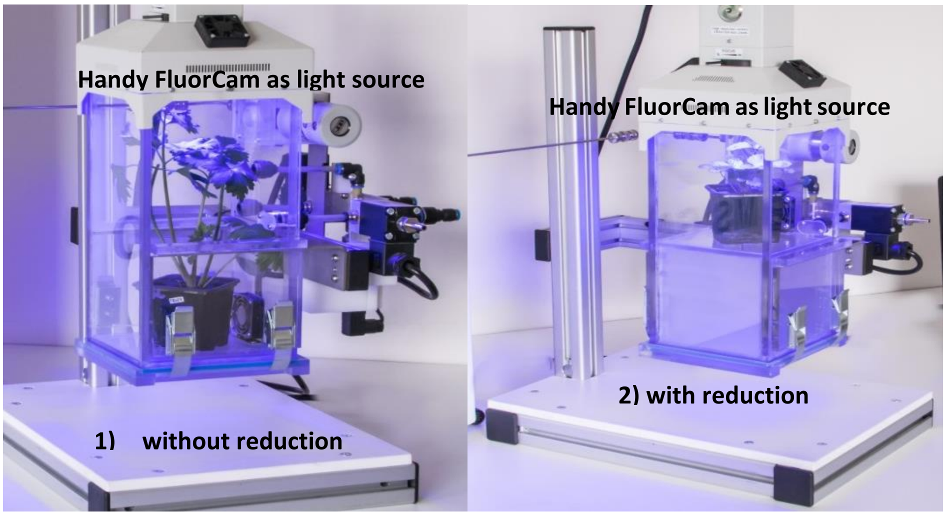


Figure 7: *Plant chamber 1) plant up to 20 cm 2) plant up to 10 cm – PSI Handy FluorCam is not a part of Plant chamber*

4.4 FRONT VIEW OF MS GAS-100 (FIGURE 8)

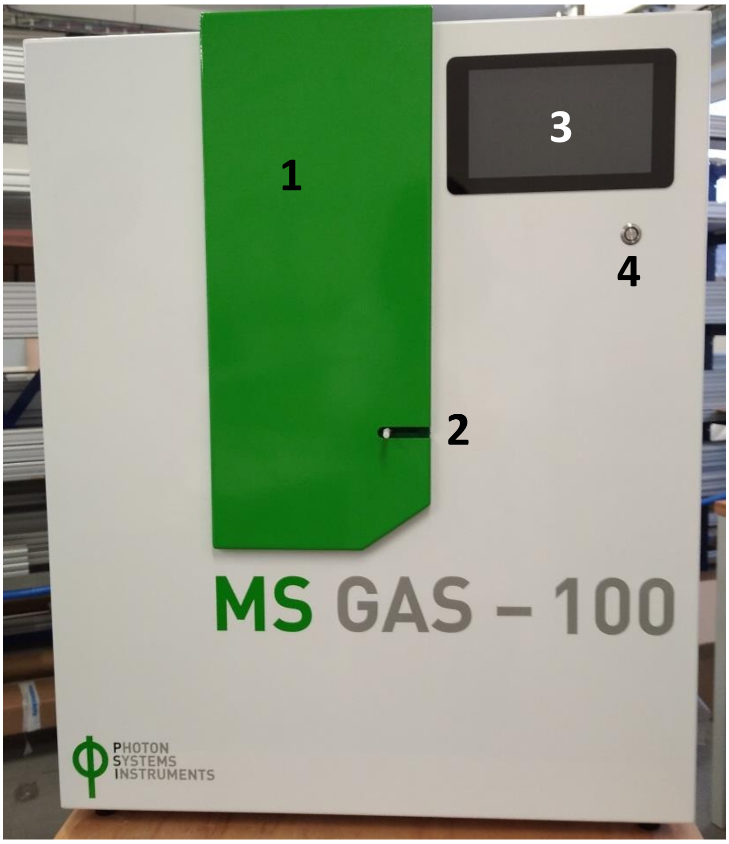


Figure 8 *shows the device front side including door (1) with external inlet system (2), TouchScreen RBI (3) integrated into the top right corner and the button (4) for switch the system ON/OFF*

4.4.1 DEVICE DOOR

The instrument door (**Figure 8**) is used to close the Gas Analyzer area and internal fixed part of the capillary inlet system during operation. The door switch is controlled by touchscreen functions (**Chapter 4.4.3.1**).

4.4.2 INLET SYSTEM

The Inlet system is divided into two parts. The first internal inlet part is fixed inside of **MS GAS-100** and protected by the device door (**Figure 8**). Internal inlet ensures the connection between the external inlet part, Stirling cooling unit, and mass spectrometer. The second external inlet part in outside of **MS GAS-100** could be constituted by modular tubes of different lengths with various endings dependent on actual measurement requirements (membrane probe, needle valve, needle input – **Figure 9**).

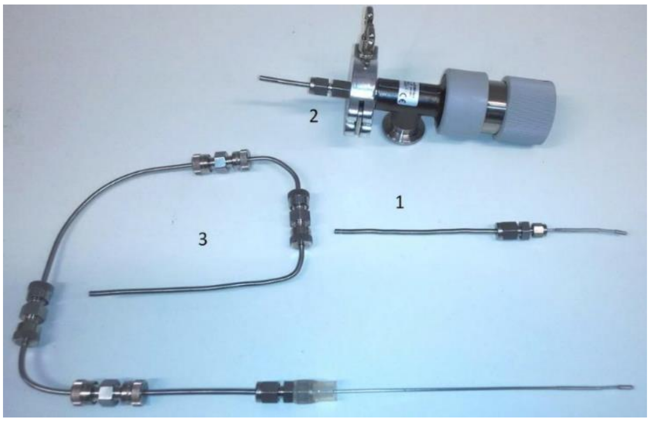
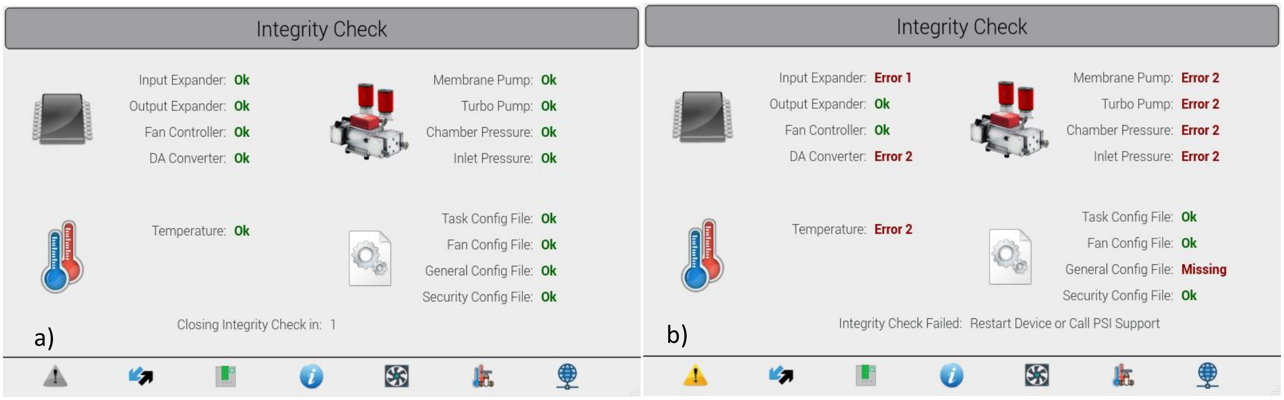


***Figure 9**: 1) simple short membrane probe 2) needle valve 3) modular inlet with long membrane probe*

4.4.3 INTEGRATED TOUCHSCREEN AND DEVICE FIRMWARE

The device is controlled via the touchscreen with two-mode possibilities. After the device is switched on by the button on the front side (**Figure 8**) the fundamental initialization steps start:

- Integrated control PC is loaded
- Defined self-test of the individual components starts (**Figure 10**)

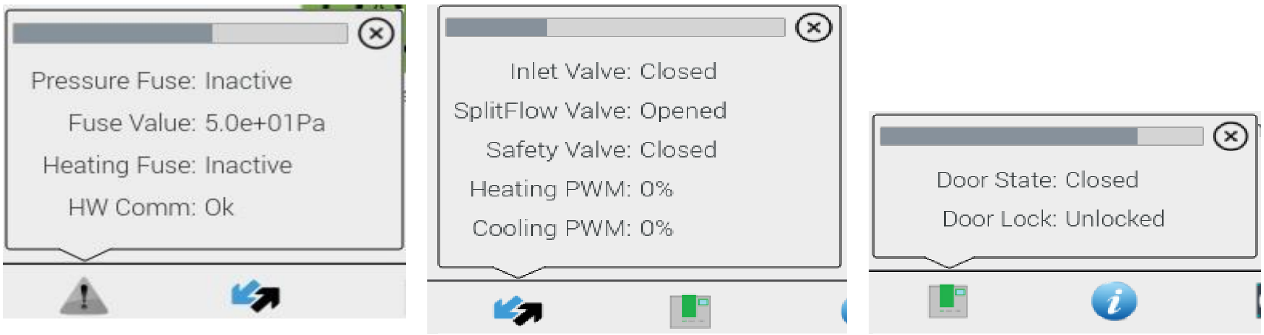


Input Expander: Ok
Output Expander: Ok
Fan Controller: Ok
DA Converter: Ok
Membrane Pump: Ok
Turbo Pump: Ok
Chamber Pressure: Ok
Inlet Pressure: Ok
Temperature: Ok
Task Config File: Ok
Fan Config File: Ok
General Config File: Ok
Security Config File: Ok
Closing Integrity Check in: 1

Input Expander: Error 1
Output Expander: Ok
Fan Controller: Ok
DA Converter: Error 2
Membrane Pump: Error 2
Turbo Pump: Error 2
Chamber Pressure: Error 2
Inlet Pressure: Error 2
Temperature: Error 2
Task Config File: Ok
Fan Config File: Ok
General Config File: Missing
Security Config File: Ok
Integrity Check Failed: Restart Device or Call PSI Support

***Figure 10:** a)Self-test b) Self-test after the device start - functions of important parts of the device are canceled due to error*

Below the black line in each window are information icons summarizing the status of several important device components in real-time. The description of the icons is shown in **Figure 11** and **Figure 12**.



Pressure Fuse: Inactive
Fuse Value: 5.0e+01Pa
Heating Fuse: Inactive
HW Comm: Ok

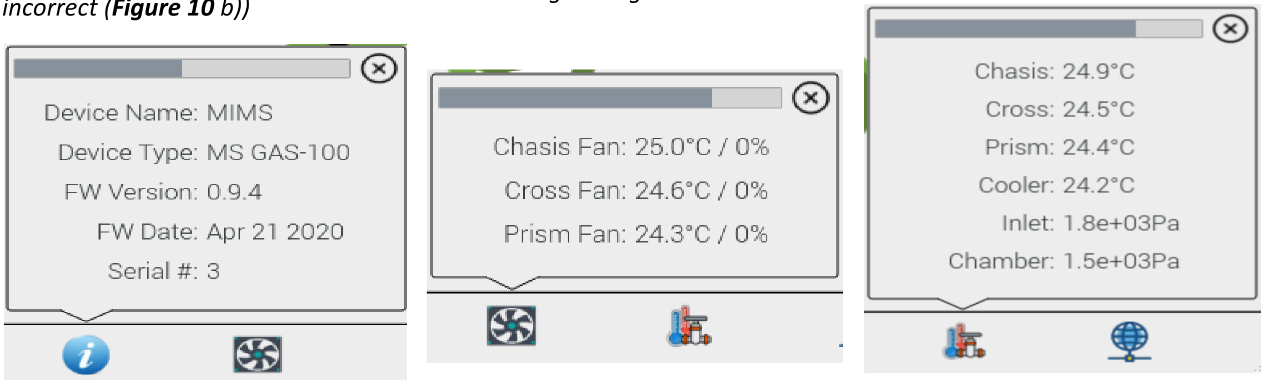
Inlet Valve: Closed
SplitFlow Valve: Opened
Safety Valve: Closed
Heating PWM: 0%
Cooling PWM: 0%

Door State: Closed
Door Lock: Unlocked

*a) Pressure and heating fuse_ icon turns in yellow – device status is incorrect (**Figure 10** b))*

b) Status of individual valves, heating and cooling setting

c) Door and lock state



Device Name: MIMS
Device Type: MS GAS-100
FW Version: 0.9.4
FW Date: Apr 21 2020
Serial #: 3

Chasis Fan: 25.0°C / 0%
Cross Fan: 24.6°C / 0%
Prism Fan: 24.3°C / 0%

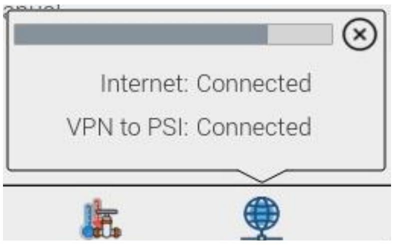
Chasis: 24.9°C
Cross: 24.5°C
Prism: 24.4°C
Cooler: 24.2°C
Inlet: 1.8e+03Pa
Chamber: 1.5e+03Pa

d) Device name and firmware information

e) Fans function

f) Temperature and pressure values measured at monitored parts inside the device

***Figure 11**: Information icons with description*

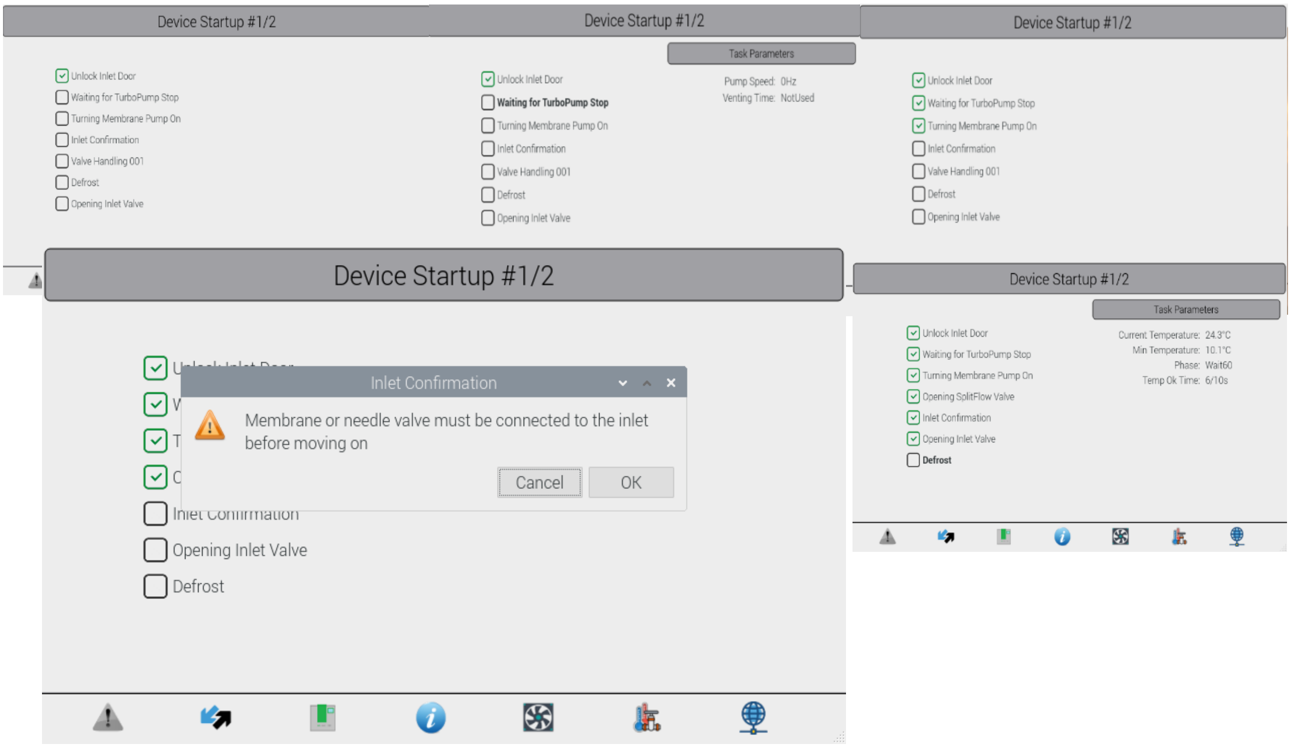


Internet: Connected
VPN to PSI: Connected

***Figure 12**: Internet and PSI VPN connection status*

If the integrity test is passed without any errors, the start-up sequences including the start of both diaphragm backing pump and turbopump begin. The pumps are responsible for the evacuation of the internal part of tubes, ion source, and the mass analyzer. Just before the pump start the **Inlet Confirmation table** about membrane probe and/or needle input (valve) connection appears on the touchscreen (**Figure 13**). After confirmation, the Startup 1/2 smoothly goes through the second Startup 2/2.

⚠	Check the correct connection of external inlet – membrane probe, needle valve, and/or needle input using UT adapter 1/8'' x 1/8'' (**Figure 14**). The incorrect connection can damage the MS GAS pumps.
⚠	Make sure that the diaphragm of the membrane probe is not leaked. If the diaphragm is damaged, replace it! Then you can confirm the Inlet Conformation table (**Figure 13**).



Device Startup #1/2

☑ Unlock Inlet Door
☐ Waiting for TurboPump Stop
☐ Turning Membrane Pump On
☐ Inlet Confirmation
☐ Valve Handling 001
☐ Defrost
☐ Opening Inlet Valve

Device Startup #1/2

☑ Unlock Inlet Door
☐ **Waiting for TurboPump Stop**
☐ Turning Membrane Pump On
☐ Inlet Confirmation
☐ Valve Handling 001
☐ Defrost
☐ Opening Inlet Valve

Task Parameters
Pump Speed: 0Hz
Venting Time: NotUsed

Device Startup #1/2

☑ Unlock Inlet Door
☑ Waiting for TurboPump Stop
☑ Turning Membrane Pump On
☐ Inlet Confirmation
☐ Valve Handling 001
☐ Defrost
☐ Opening Inlet Valve

Device Startup #1/2

☐ Inlet Confirmation
☐ Opening Inlet Valve
☐ Defrost

Inlet Confirmation

Membrane or needle valve must be connected to the inlet before moving on

Device Startup #1/2

☑ Unlock Inlet Door
☑ Waiting for TurboPump Stop
☑ Turning Membrane Pump On
☑ Opening SplitFlow Valve
☑ Inlet Confirmation
☑ Opening Inlet Valve
☐ **Defrost**

Task Parameters
Current Temperature: 24.3°C
Min Temperature: 10.1°C
Phase: Wait60
Temp Ok Time: 6/10s

Figure 13: *Startup 1/2 sequence including the Confirmation of probe and/or needle connection to the external inlet of MS GAS-100*

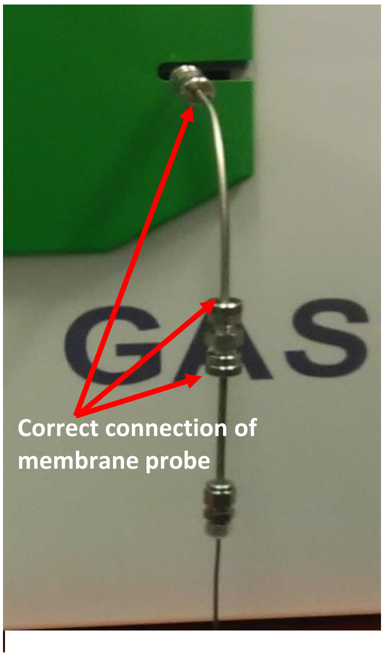


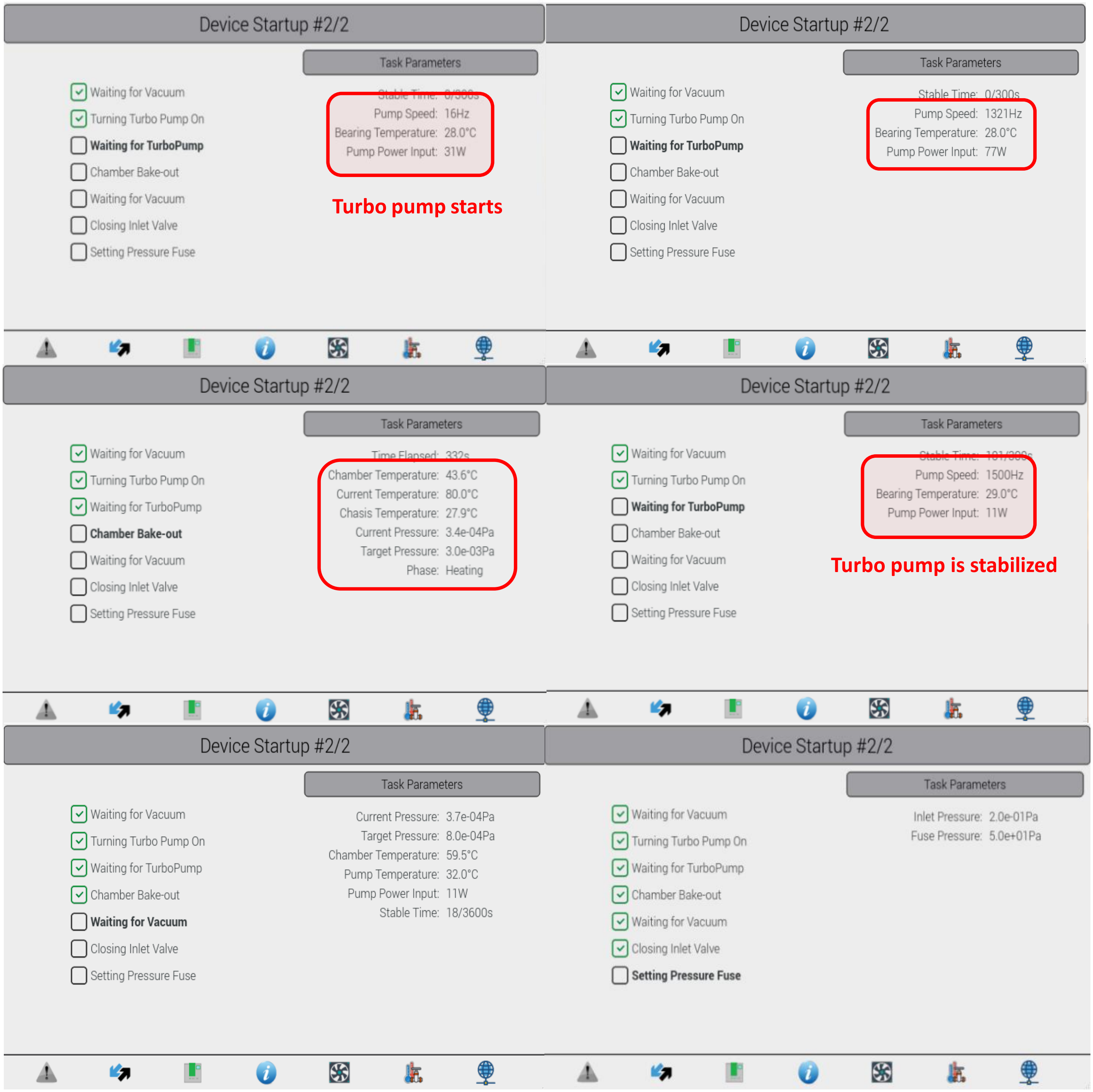
Figure 14: *The membrane probe connection*

In Startup 2/2 membrane and turbomolecular pumps start working and evacuate the inside of the MS GAS-100. In this process, it is very important to monitor the start of the turbopump. The pump must reach a speed of 1500 Hz and the power of the pump has to be reduced from a maximum value of 77 W to approximately 10-20 W (**Figure 15**). If the pump can't reach these values, the system is leaking and the pump will stop. A common cause of leakage is a damage of diaphragm on the membrane probe (**Chapter 5.2.1**) or improper connection of the external inlet to the internal pipe (**Figure 14**, **Figure 42**).

During the evacuation of the device, the inlet valve and the split flow valve are opened (see information icon **Figure 16** a)).

As soon as the turbopump reaches the speed of 1500 Hz and at the same time the pressure inside the device is low enough (controlled by setting the value of the pressure fuse), the ion chamber starts to bake. The incorporated heating element bakes the ion chamber (cross) up to 80 ° C for 10 minutes to clean it after the device has been switched off for a long time. The temperature increasement in the cross can be monitored using the information icon (**Figure 11** icon f) and **Figure 16** b))

Baking is followed by cooling of the ion chamber using a cross fan **Figure 16** c)) to a temperature of about 30 ° C. Then the device waits for a vacuum suitable for further work and stabilization of both pumps. The pressure fuse is checked before switching to the **Idle Screen** (**Figure 18**).



☑ Waiting for Vacuum
☑ Turning Turbo Pump On
☐ **Waiting for TurboPump**
☐ Chamber Bake-out
☐ Waiting for Vacuum
☐ Closing Inlet Valve
☐ Setting Pressure Fuse

Stable Time: 0/300s
Pump Speed: 16Hz
Bearing Temperature: 28.0°C
Pump Power Input: 31W

Turbo pump starts

☑ Waiting for Vacuum
☑ Turning Turbo Pump On
☐ **Waiting for TurboPump**
☐ Chamber Bake-out
☐ Waiting for Vacuum
☐ Closing Inlet Valve
☐ Setting Pressure Fuse

Stable Time: 0/300s
Pump Speed: 1321Hz
Bearing Temperature: 28.0°C
Pump Power Input: 77W

☑ Waiting for Vacuum
☑ Turning Turbo Pump On
☑ Waiting for TurboPump
☐ **Chamber Bake-out**
☐ Waiting for Vacuum
☐ Closing Inlet Valve
☐ Setting Pressure Fuse

Time Elapsed: 332s
Chamber Temperature: 43.6°C
Current Temperature: 80.0°C
Chasis Temperature: 27.9°C
Current Pressure: 3.4e-04Pa
Target Pressure: 3.0e-03Pa
Phase: Heating

☑ Waiting for Vacuum
☑ Turning Turbo Pump On
☐ **Waiting for TurboPump**
☐ Chamber Bake-out
☐ Waiting for Vacuum
☐ Closing Inlet Valve
☐ Setting Pressure Fuse

Stable Time: 101/300s
Pump Speed: 1500Hz
Bearing Temperature: 29.0°C
Pump Power Input: 11W

Turbo pump is stabilized

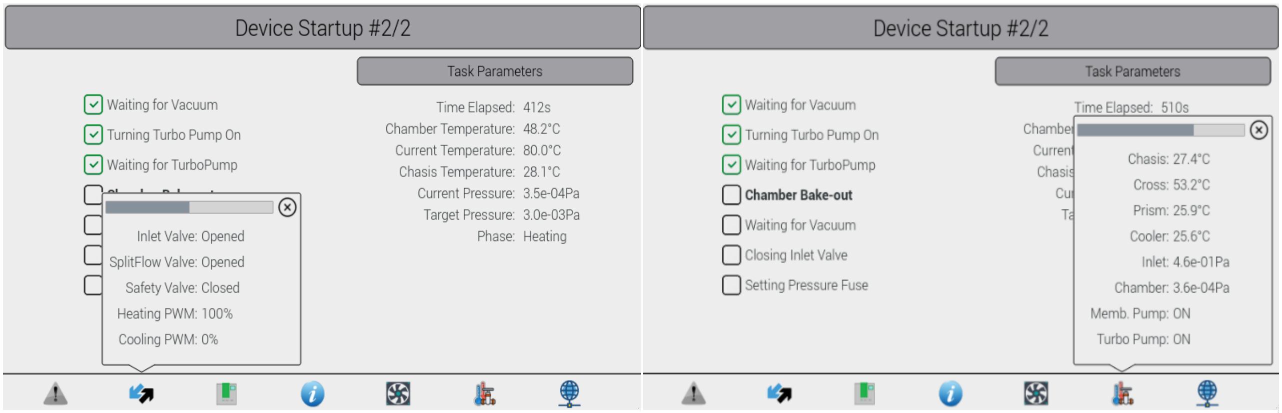
☑ Waiting for Vacuum
☑ Turning Turbo Pump On
☑ Waiting for TurboPump
☑ Chamber Bake-out
☐ **Waiting for Vacuum**
☐ Closing Inlet Valve
☐ Setting Pressure Fuse

Current Pressure: 3.7e-04Pa
Target Pressure: 8.0e-04Pa
Chamber Temperature: 59.5°C
Pump Temperature: 32.0°C
Pump Power Input: 11W
Stable Time: 18/3600s

☑ Waiting for Vacuum
☑ Turning Turbo Pump On
☑ Waiting for TurboPump
☑ Chamber Bake-out
☑ Waiting for Vacuum
☑ Closing Inlet Valve
☐ **Setting Pressure Fuse**

Inlet Pressure: 2.0e-01Pa
Fuse Pressure: 5.0e+01Pa

Figure 15: *Startup 2/2*



Device Startup #2/2

Task Parameters

☑ Waiting for Vacuum
☑ Turning Turbo Pump On
☑ Waiting for TurboPump
☐
☐
☐
☐

Inlet Valve: Opened
SplitFlow Valve: Opened
Safety Valve: Closed
Heating PWM: 100%
Cooling PWM: 0%

Time Elapsed: 412s
Chamber Temperature: 48.2°C
Current Temperature: 80.0°C
Chasis Temperature: 28.1°C
Current Pressure: 3.5e-04Pa
Target Pressure: 3.0e-03Pa
Phase: Heating

Device Startup #2/2

Task Parameters

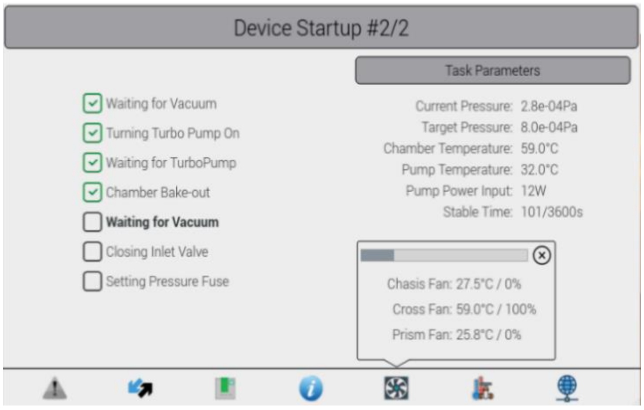
☑ Waiting for Vacuum
☑ Turning Turbo Pump On
☑ Waiting for TurboPump
☐ **Chamber Bake-out**
☐ Waiting for Vacuum
☐ Closing Inlet Valve
☐ Setting Pressure Fuse

Time Elapsed: 510s

Chasis: 27.4°C
Cross: 53.2°C
Prism: 25.9°C
Cooler: 25.6°C
Inlet: 4.6e-01Pa
Chamber: 3.6e-04Pa
Memb. Pump: ON
Turbo Pump: ON

a) *State of valves*

b)*: Information about temperature value in ion chamber (CROSS) during the bake-out*



Device Startup #2/2

Task Parameters

☑ Waiting for Vacuum
☑ Turning Turbo Pump On
☑ Waiting for TurboPump
☑ Chamber Bake-out
☐ **Waiting for Vacuum**
☐ Closing Inlet Valve
☐ Setting Pressure Fuse

Current Pressure: 2.8e-04Pa
Target Pressure: 8.0e-04Pa
Chamber Temperature: 59.0°C
Pump Temperature: 32.0°C
Pump Power Input: 12W
Stable Time: 101/3600s

Chasis Fan: 27.5°C / 0%
Cross Fan: 59.0°C / 100%
Prism Fan: 25.8°C / 0%

c)*: Cross fan works at 100%*

Figure 16*: Startup 2/2 infomation icons*

4.4.3.1 Screen Saver

When the touch screen is not used for about 10 minutes, the **Screen Saver** is available (**Figure 17**).

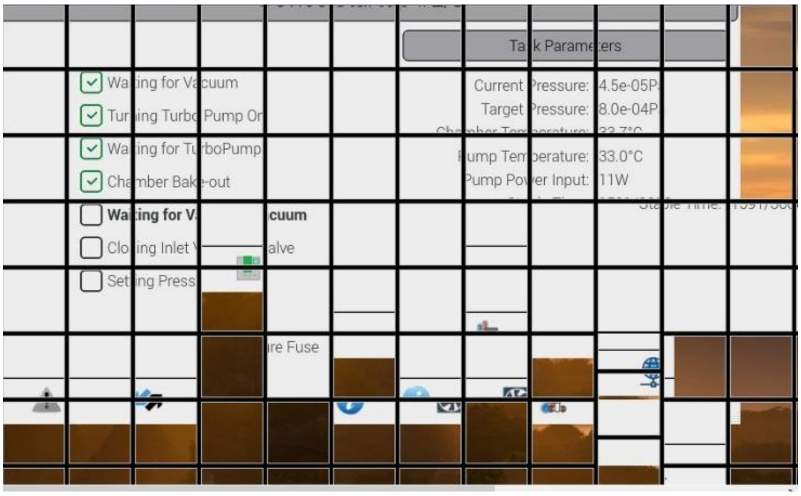


Figure 17*: Screen Saver*

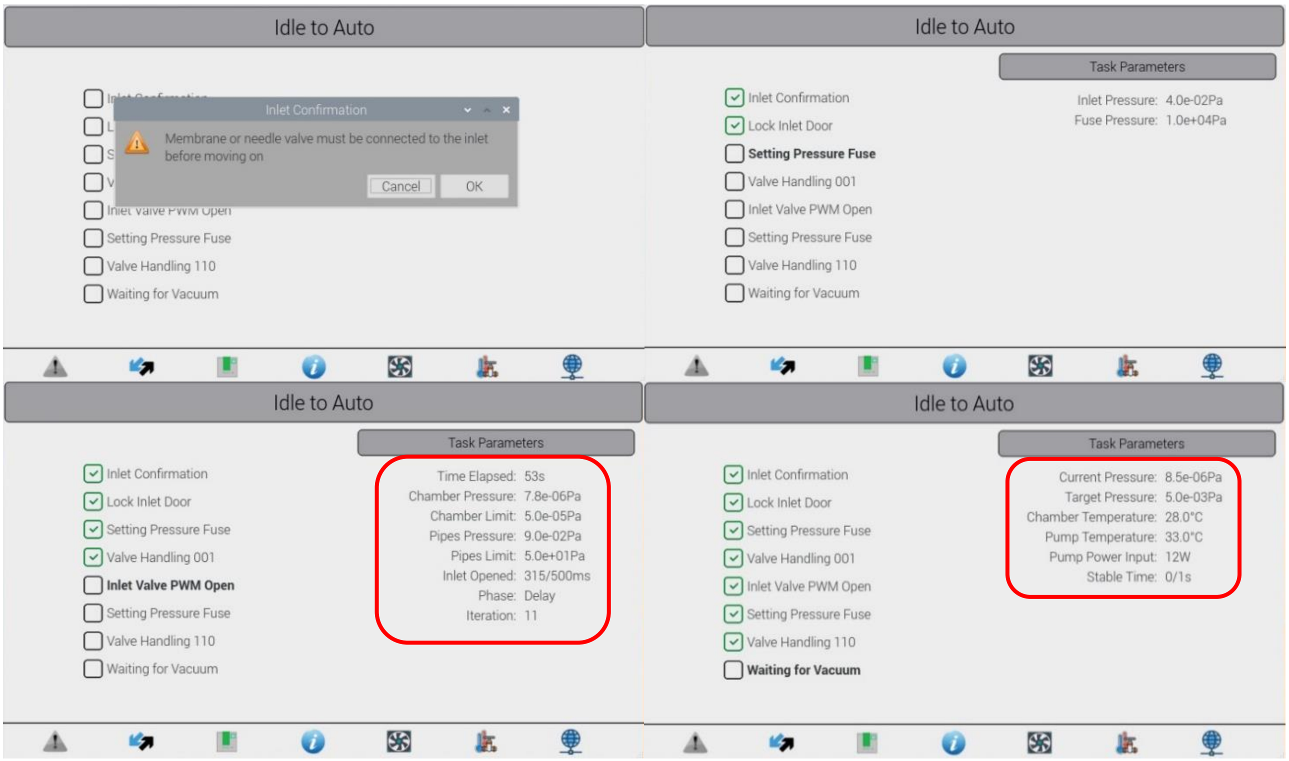
4.4.3.2 *Automatic Mode*

Press the icon **Automatic** (**Figure 18**). The switch to auto mode is possible after confirmation of a membrane probe or needle input (valve) installation at the end of the external inlet part (**Figure 14** and **Figure 19**).

The next automatic functions are responsible for all the necessary steps during inlet system evacuation. Split flow valve and inlet valve are gradually opened/closed until the target pressure values for vacuum are reached in inlet capillaries (**Figure 19**, red frames). After the vacuum stabilization and the setting of pressure protection (pressure fuse), the device is switched into the Automatic mode (**Figure 20**). The device is prepared for measurement with the opened inlet valve, opened safety valve, and closed split-flow valve chamber (**Figure 20**, information table). Two green icons with the same symbol labeled as Inlet and Chamber indicate good pressure values inside the system at the inlet and in the ion chamber (**Figure 20**, red frame). When the set pressure fuse value is exceeded, all valves are closed automatically and the Inlet pressure icon turns red (**Figure 31**).

Figure 18*: Idle screen with the auto and manual mode*

⚠	Check the correct connection of external inlet – membrane probe, needle valve, and/or needle input using UT adapter 1/8’’x1/8’’ (**Figure 14**, **Figure 42**). The incorrect connection can damage the MS GAS pumps.
⚠	Make sure that the diaphragm of the membrane probe is not leaked. If the diaphragm is damaged, replace it! Then you can confirm the Inlet Conformation table (**Figure 19**).



Membrane or needle valve must be connected to the inlet before moving on

☐ Inlet Valve PWM Open
☐ Setting Pressure Fuse
☐ Valve Handling 110
☐ Waiting for Vacuum

☑ Inlet Confirmation
☑ Lock Inlet Door
☐ **Setting Pressure Fuse**
☐ Valve Handling 001
☐ Inlet Valve PWM Open
☐ Setting Pressure Fuse
☐ Valve Handling 110
☐ Waiting for Vacuum

Inlet Pressure: 4.0e-02Pa
Fuse Pressure: 1.0e+04Pa

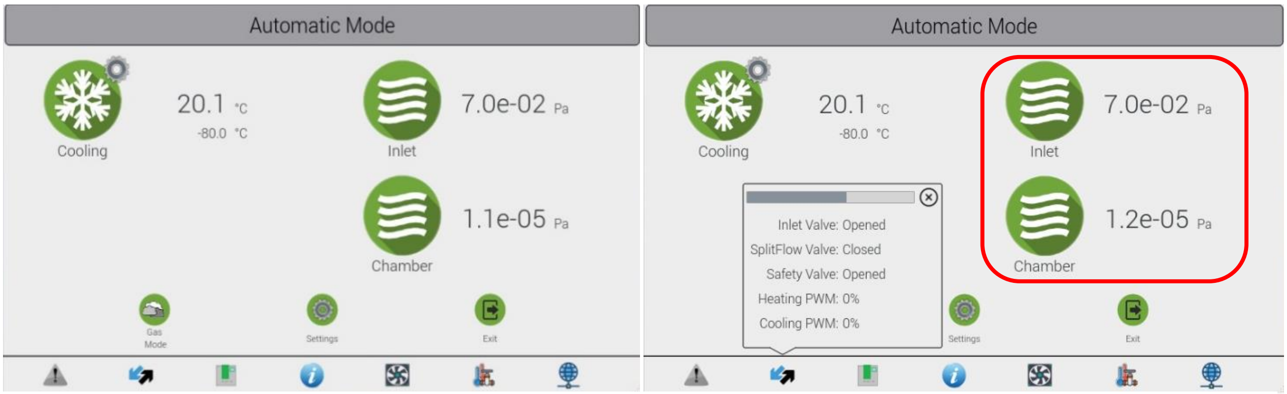
☑ Inlet Confirmation
☑ Lock Inlet Door
☑ Setting Pressure Fuse
☑ Valve Handling 001
☐ **Inlet Valve PWM Open**
☐ Setting Pressure Fuse
☐ Valve Handling 110
☐ Waiting for Vacuum

Time Elapsed: 53s
Chamber Pressure: 7.8e-06Pa
Chamber Limit: 5.0e-05Pa
Pipes Pressure: 9.0e-02Pa
Pipes Limit: 5.0e+01Pa
Inlet Opened: 315/500ms
Phase: Delay
Iteration: 11

☑ Inlet Confirmation
☑ Lock Inlet Door
☑ Setting Pressure Fuse
☑ Valve Handling 001
☑ Inlet Valve PWM Open
☑ Setting Pressure Fuse
☑ Valve Handling 110
☐ **Waiting for Vacuum**

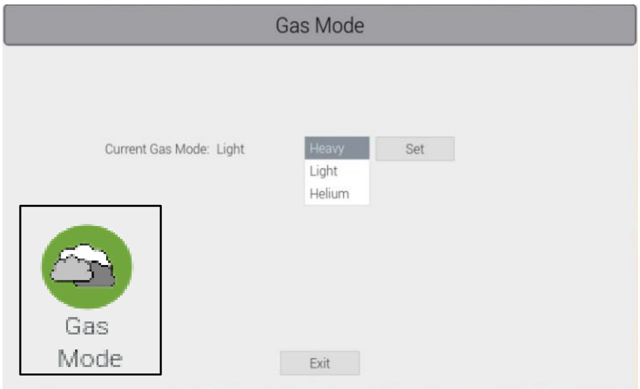
Current Pressure: 8.5e-06Pa
Target Pressure: 5.0e-03Pa
Chamber Temperature: 28.0°C
Pump Temperature: 33.0°C
Pump Power Input: 12W
Stable Time: 0/1s

Figure 19*: Sequence of operation steps of system for switching to Auto mode*



Inlet Valve: Opened
SplitFlow Valve: Closed
Safety Valve: Opened
Heating PWM: 0%
Cooling PWM: 0%

***Figure 20**: Automatic mode screen and information about opened/closed valves and pressure values (red frame)*

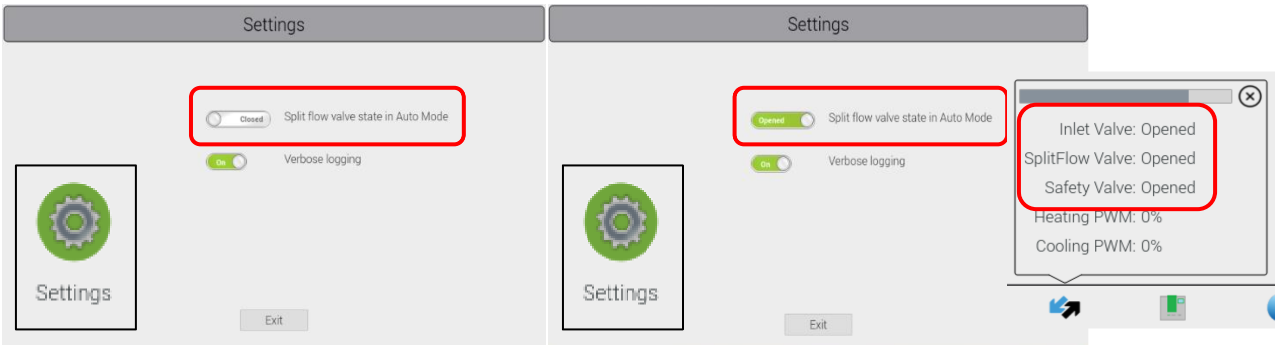


Current Gas Mode: Light

***Figure 21**: Gas mode*

In Automatic mode, there is no possibility of viewing additional turbopump parameters, the user is able only to control and change turbopump gas mode. (**Figure 21**).

If the observed sample is too concentrated, it is sometimes useful to divide it to avoid damaging the mass analyzer. The sample can be divided using an open split-flow valve (see the icon **Setting** - **Figure 22**). The information about the open split-flow valve is available in the valve state information icon (**Figure 22**).



Closed — Split flow valve state in Auto Mode
On — Verbose logging

Opened — Split flow valve state in Auto Mode
On — Verbose logging

Inlet Valve: Opened
SplitFlow Valve: Opened
Safety Valve: Opened
Heating PWM: 0%
Cooling PWM: 0%

***Figure 22**: Split Flow Valve opened/closed and Information icon of valve status*

The automatic mode is designed for direct measurements and easy use of the device. Only one parameter can be changed in auto mode – **cooling temperature** when the liquid samples are determined.

A **Stirling cooler** was integrated into the device inlet to perform dissolved gases analysis in liquid samples. The Stirling cooler function in the device is to protect the mass analyzer before excess water from the liquid sample. The cooler setting for different freezing temperatures can also be used in automatic mode. It is generally recommended to set the freezing temperature to -80 °C - the first desorption temperature of the water, for the efficient removal of water molecules from the system.

The temperature setting is shown in **Figure 23**. The cooling temperature setting table is available by clicking on the symbol next to the snowflake icon (red arrow in **Figure 23**). The temperature setting range is 0 to -80 °C. An incorrectly set value will cause the number field to turn red. After confirming the correct value in the table, the smaller number below the current temperature on the cooler will change. Touching the snowflake icon changes the color of the icon to blue. The integrated sensor monitors the temperature decrease due to the function of the Stirling cooler (red frame in **Figure 23**).

Measurement of aqueous samples – freeze the internal inlet at -80 °C (**Figure 23**).

After the cooling is stopped, automatic **defrosting sequences** follow. Frozen water thaws very slowly from the surface of the capillary system. The inlet valve and safety valve are closed and the split-flow valve is opened to protect the mass analyzer from large amounts of desublimed water. Defrosting the system is a long-term process because the temperature inside the pipes must reach at least 16° C. When the system is tempered again to the target temperature, the automatic evacuation of the inlets begins by gradually opening/closing the inlet valve and the split flow valve concerning the set pressure limits. The screen will eventually switch back to automatic mode (**Figure 25** and **Figure 20**).

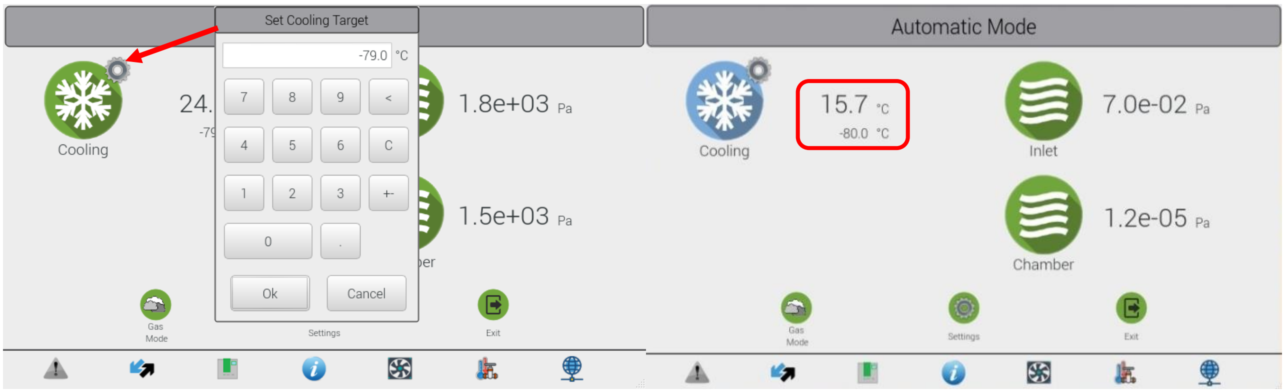
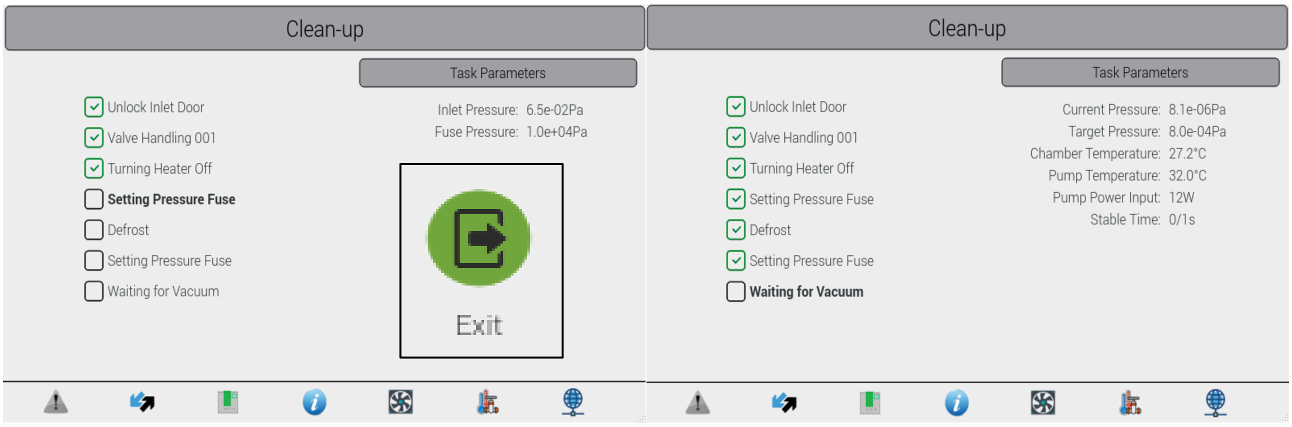


***Figure 23** Setting of cooling temperature, the Stirling cooler is switched ON (blue icon), the value of temperature decreases (red frame)*

The **Exit** icon allows switching from automatic mode to the main screen (Idle Mode, **Figure 18**). The switching process is based on a new sequence of Clean-up controls. The automatic steps check the pressure ratio in the ion chamber and the capillary system of the device and also monitor the temperature of the Stirling cooler and the water defrosting (**Figure 24**).


Clean-up

Task Parameters

- ☑ Unlock Inlet Door
- ☑ Valve Handling 001
- ☑ Turning Heater Off
- ☐ **Setting Pressure Fuse**
- ☐ Defrost
- ☐ Setting Pressure Fuse
- ☐ Waiting for Vacuum

Inlet Pressure: 6.5e-02Pa
Fuse Pressure: 1.0e+04Pa

Clean-up

Task Parameters

- ☑ Unlock Inlet Door
- ☑ Valve Handling 001
- ☑ Turning Heater Off
- ☑ Setting Pressure Fuse
- ☑ Defrost
- ☑ Setting Pressure Fuse
- ☐ **Waiting for Vacuum**

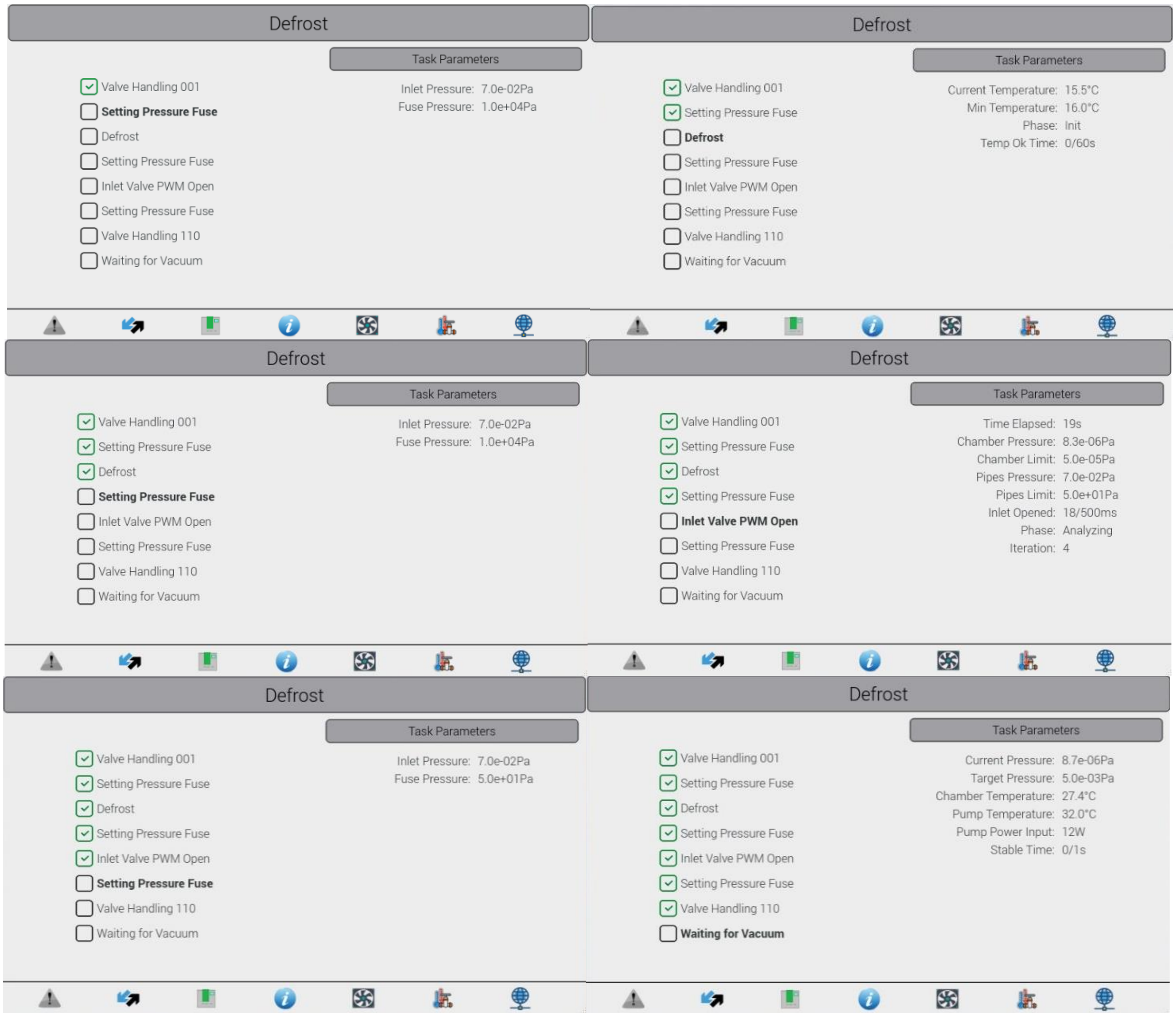
Current Pressure: 8.1e-06Pa
Target Pressure: 8.0e-04Pa
Chamber Temperature: 27.2°C
Pump Temperature: 32.0°C
Pump Power Input: 12W
Stable Time: 0/1s

***Figure 24** Clean-up*

4.4.3.1 Manual Mode

After selecting the **manual mode** (**Figure 18**), the **pressure fuse** (**Figure 26**) is set and the screen switches to the main screen of the manual mode (**Figure 26**). Compared to the automatic mode, manual mode contains more variable functions.

Manual mode allows the setting of cooling and heating temperature defined by the user. The setting and control of the **Stirling cooler** are the same as in automatic mode (**Chapter 4.4.3.2**).



Defrost

Task Parameters

☑ Valve Handling 001
☐ **Setting Pressure Fuse**
☐ Defrost
☐ Setting Pressure Fuse
☐ Inlet Valve PWM Open
☐ Setting Pressure Fuse
☐ Valve Handling 110
☐ Waiting for Vacuum

Inlet Pressure: 7.0e-02Pa
Fuse Pressure: 1.0e+04Pa

Defrost

Task Parameters

☑ Valve Handling 001
☑ Setting Pressure Fuse
☐ **Defrost**
☐ Setting Pressure Fuse
☐ Inlet Valve PWM Open
☐ Setting Pressure Fuse
☐ Valve Handling 110
☐ Waiting for Vacuum

Current Temperature: 15.5°C
Min Temperature: 16.0°C
Phase: Init
Temp Ok Time: 0/60s

Defrost

Task Parameters

☑ Valve Handling 001
☑ Setting Pressure Fuse
☑ Defrost
☐ **Setting Pressure Fuse**
☐ Inlet Valve PWM Open
☐ Setting Pressure Fuse
☐ Valve Handling 110
☐ Waiting for Vacuum

Inlet Pressure: 7.0e-02Pa
Fuse Pressure: 1.0e+04Pa

Defrost

Task Parameters

☑ Valve Handling 001
☑ Setting Pressure Fuse
☑ Defrost
☑ Setting Pressure Fuse
☐ **Inlet Valve PWM Open**
☐ Setting Pressure Fuse
☐ Valve Handling 110
☐ Waiting for Vacuum

Time Elapsed: 19s
Chamber Pressure: 8.3e-06Pa
Chamber Limit: 5.0e-05Pa
Pipes Pressure: 7.0e-02Pa
Pipes Limit: 5.0e+01Pa
Inlet Opened: 18/500ms
Phase: Analyzing
Iteration: 4

Defrost

Task Parameters

☑ Valve Handling 001
☑ Setting Pressure Fuse
☑ Defrost
☑ Setting Pressure Fuse
☑ Inlet Valve PWM Open
☐ **Setting Pressure Fuse**
☐ Valve Handling 110
☐ Waiting for Vacuum

Inlet Pressure: 7.0e-02Pa
Fuse Pressure: 5.0e+01Pa

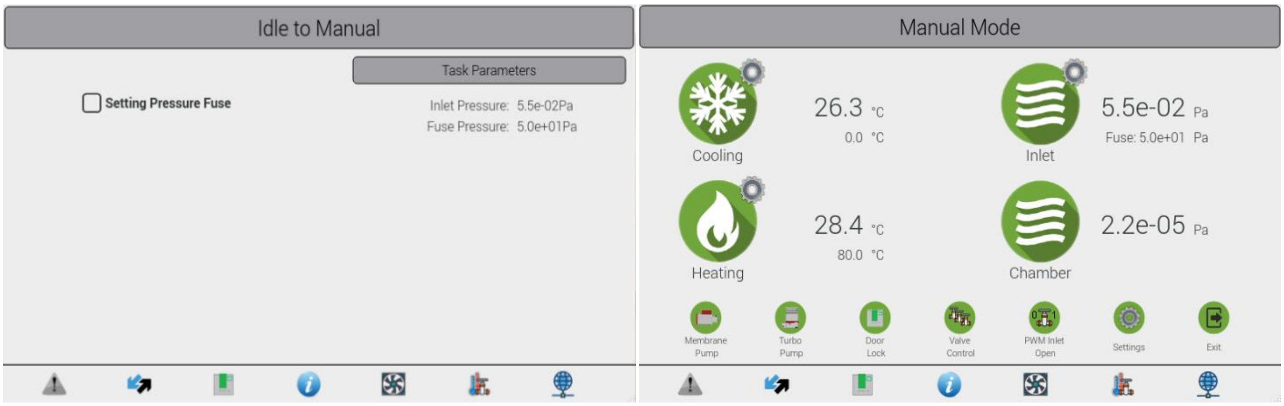
Defrost

Task Parameters

☑ Valve Handling 001
☑ Setting Pressure Fuse
☑ Defrost
☑ Setting Pressure Fuse
☑ Inlet Valve PWM Open
☑ Setting Pressure Fuse
☑ Valve Handling 110
☐ **Waiting for Vacuum**

Current Pressure: 8.7e-06Pa
Target Pressure: 5.0e-03Pa
Chamber Temperature: 27.4°C
Pump Temperature: 32.0°C
Pump Power Input: 12W
Stable Time: 0/1s

Figure 25*: System defrosting and evacuation before next operations in Automatic mode*



Idle to Manual

Task Parameters

☐ **Setting Pressure Fuse**

Inlet Pressure: 5.5e-02Pa
Fuse Pressure: 5.0e+01Pa

Manual Mode

Cooling 26.3 °C 0.0 °C
Inlet 5.5e-02 Pa Fuse: 5.0e+01 Pa
Heating 28.4 °C 80.0 °C
Chamber 2.2e-05 Pa

Figure 26*: Pressure fuse setting and the switching to the main screen of Manual mode*

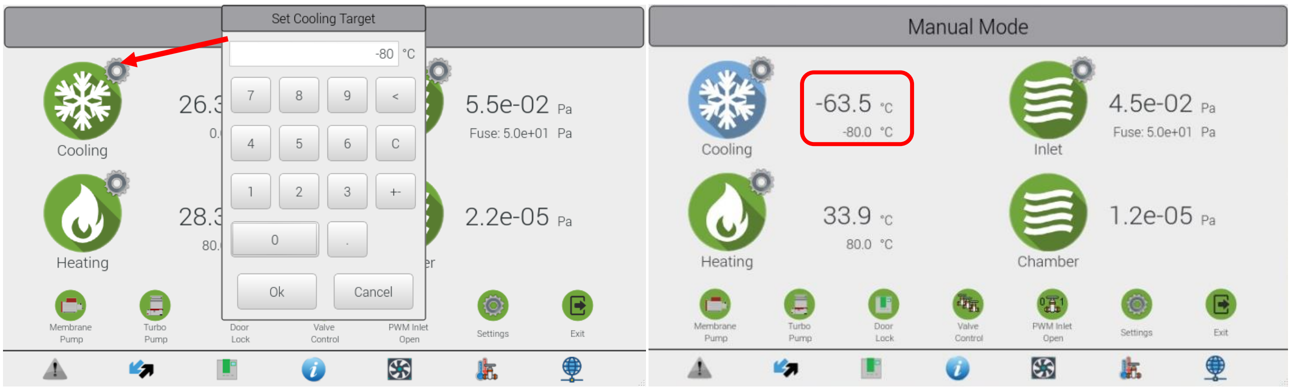


__Figure 27__: Setting of cooler temperature _Blue icon_ Stirling cooler is currently working

In the manual mode the ion chamber can be cleaned by baking it out at higher temperatures. The **heating** settings and control are the same as for cooling (**Figure 28** and **Chapter 4.4.3.2**). The ion chamber can be baked up to 90 °C. If an incorrect value is given in the temperature setting table, the field will turn red (**Figure 28**). The correct function of the heating element is indicated by the red color of the heating icon and the gradually increasing temperature (**Figure 29**, red frame).

In Automatic mode, the ionization of analyzed gases and their determination by the mass analyzer are realized only under laboratory temperature. The second function of the controlled heating in manual mode is that the measurement can be performed under higher temperatures in the ion source chamber. The heating in the mass chamber can increase the kinetics of ions and shorten measurement.

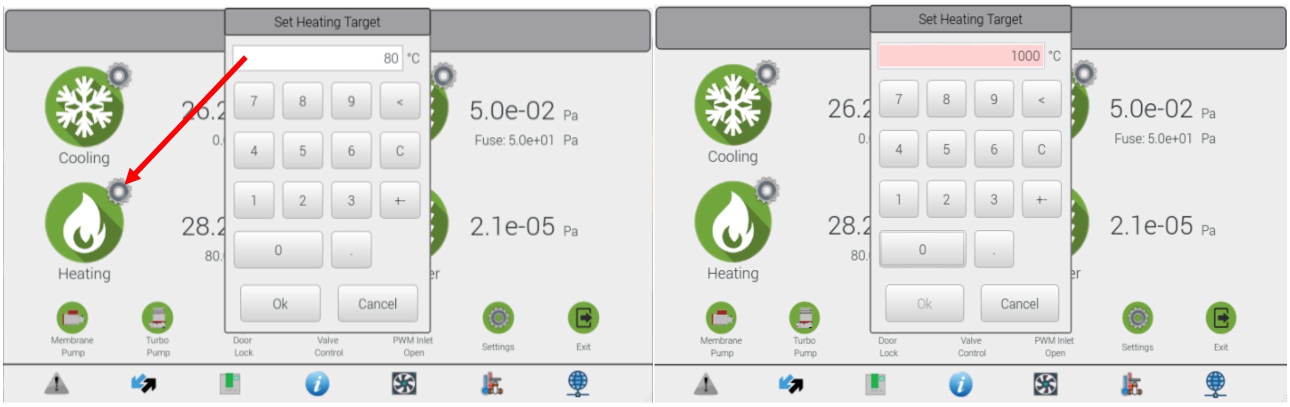


__Figure 28__: Setting of heating temperature, the wrong value of set temperature

__Figure 29__: The heating is ON

Any values of the **pressure fuse** can be set (**Figure 30**). The pressure fuse serves primarily to protect the mass analyzer from high-pressure impact, which can destroy very expensive parts of the device. The value of maximum pressure deviation in inlet capillaries with open valves is 200 Pa (2 mBa) which doesn't damage the Mass Spectrometer function. If the pressure overruns preset fuse value (**Figure 31**), all device valves are automatically closed. Protection can be default by touching the symbol next to Inlet icon. The setting and control of the fuse is the same as for heating and cooling. A smaller number indicates the set value of the pressure fuse, a larger number indicates the current pressure in the capillary inlet (**Figure 30**, red frame).

There are also some cases when the pressure fuse has to be set on higher than recommended values (for example 10,000 Pa), especially after analysis of liquid samples. When measuring liquid samples, cooling to -80 °C must be used. After stopping the cooling, the device starts defrosting the inlet pipe. For water samples, the pressure inside the inlet capillaries is increased above the set pressure protection due to the gradual desublimation of the frozen water. For complete defrosting of the system, it is necessary to open the split flow valve (other valves are closed, **Figure 37** d)), so that it is possible to effectively achieve the gradual removal of water in small quantities. A too low value of the pressure fuse would cause closing this valve. After reopening the valve, a large amount of liquid water could damage or destroy the diaphragm backing pump and mainly turbomolecular pump.

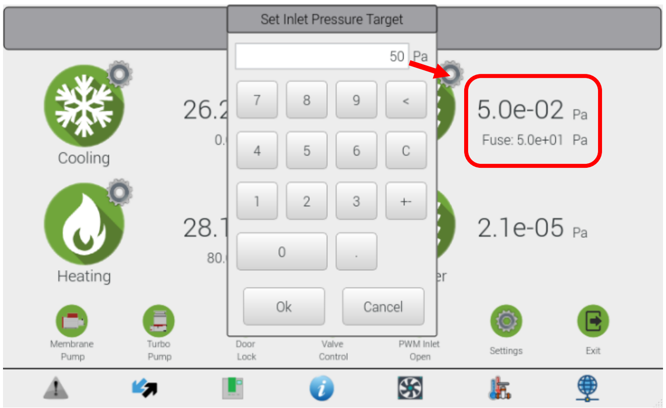


***Figure 30**: The setting of pressure fuse*

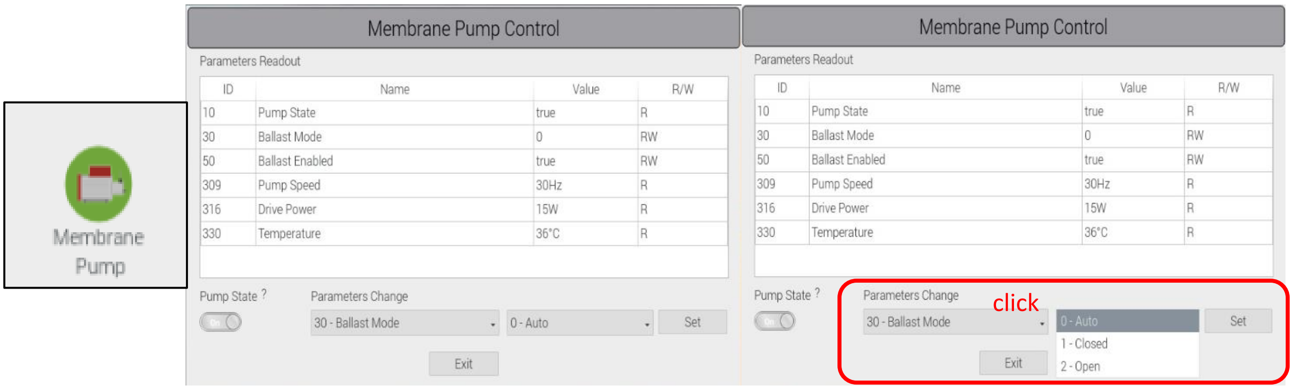
ℹ	The recommended values of the pressure fuse are about 10 - 100 Pa (0.1-1 mBa).
⚠	The maximum pressure fuse is 200 Pa (2 mBa). Higher pressure than 200 Pa (2 mBa) damages or destroys the MS GAS-100 .
⚠	Before the fuse value setting over the recommended values make sure your MS GAS-100 is not in the measurement process - the filament and SEM detector must be switched OFF (**Chapter 6.2.3**)!

***Figure 31**: Red Inlet icon_ set fuse limit has been exceeded*

Several characteristics of the **diaphragm and turbomolecular pumps** are available in manual mode. The control of some important pump parameters is recommended for good pumps running evaluation.

The most important parameter of the **diaphragm backing pump** is the **Ballast Valve** function. The ballast valve opens and closes automatically and is controlled by the pump itself. However, valve status can also be changed manually to open/close at all times. Changing the valve state to opened is useful for regenerating the membranes inside the pump after long-term measurement of liquid samples and/or when the pressure in the ion chamber is not capable to reach the recommended value (**Chapter 5.1 , Chapter 7**).

After clicking on the Membrane pump icon, an information table with the pump parameters is available. Touching in the parameter change window allows the setting of the selected parameter (**Figure 32**).



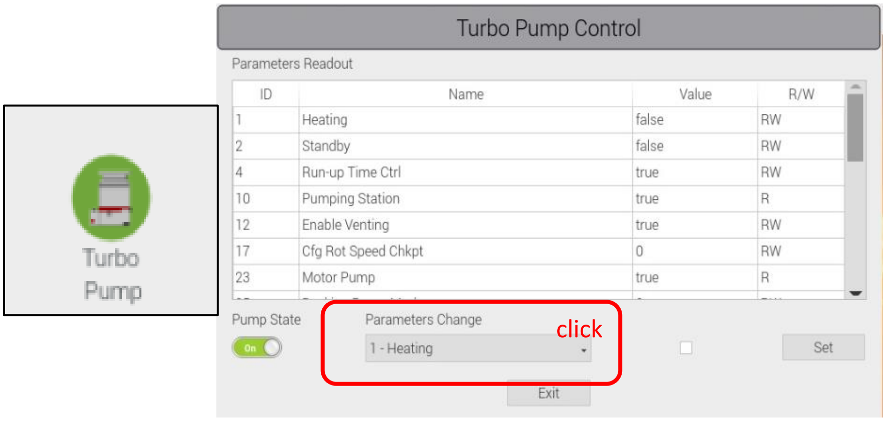
ID	Name	Value	R/W
10	Pump State	true	R
30	Ballast Mode	0	RW
50	Ballast Enabled	true	RW
309	Pump Speed	30Hz	R
316	Drive Power	15W	R
330	Temperature	36°C	R

30 - Ballast Mode
0 - Auto
1 - Closed
2 - Open

__Figure 32__: Membrane pump icon, list of parameters and the setting of changed parameter

The **Turbo Pump** icon opens a table with selected turbomolecular pump parameters (**Figure 33**). Individual parameters of the pump can be displayed by clicking on the Change parameters field (**Figure 33** and **Figure 34**). It is generally not recommended to change most of the factory-set items.

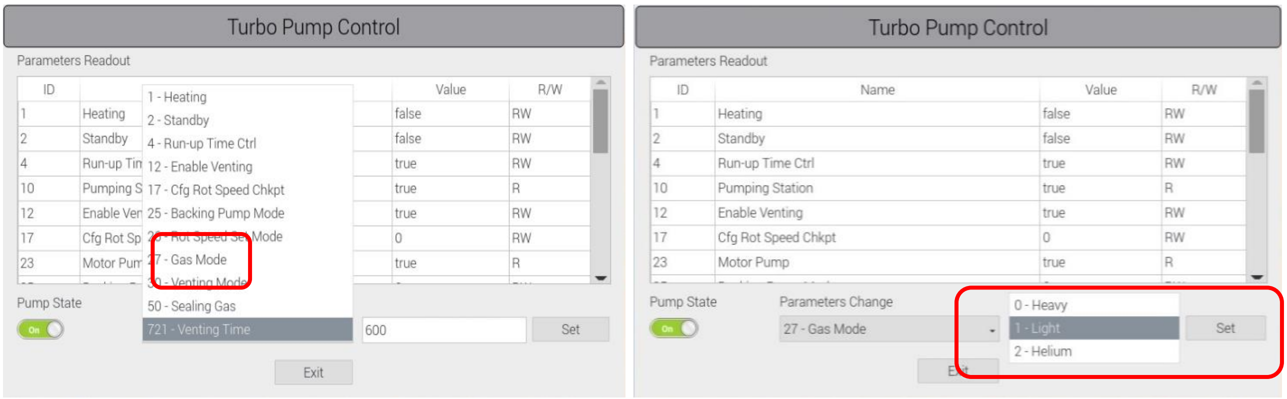
The **Gas Mode** in turbopump parameter list is especially important for the measurement (**Figure 34**). The choice of helium, light, or heavy gas affects the pumping speed of the turbopump. The selection is made concerning the majority gaseous component of measured sample. If the dominant gas in the measured sample is air, the mode is set to **1-light** on the pump, because the major component of air is nitrogen with low molecular weight. However, for measuring for example in argon atmosphere it is necessary to set the pump gas mode to **2-heavy**. The helium mode is used only during the pipe leakage test.



ID	Name	Value	R/W
1	Heating	false	RW
2	Standby	false	RW
4	Run-up Time Ctrl	true	RW
10	Pumping Station	true	R
12	Enable Venting	true	RW
17	Cfg Rot Speed Chkpt	0	RW
23	Motor Pump	true	R

1 - Heating

__Figure 33__: The parameters of turbopump and their change



ID	Name	Value	R/W
1	Heating	false	RW
2	Standby	false	RW
4	Run-up Time Ctrl	true	RW
10	Pumping Station	true	R
12	Enable Venting	true	RW
17	Cfg Rot Speed Chkpt	0	RW
23	Motor Pump	true	R

Parameters Change: 27 - Gas Mode

Figure 34*: List of parameters and selection of Gas Mode*

The **door** function (**Figure 8**) is to separate the external and internal inlet of the device. The external inlet can be variably changed, while the internal inlet is a fixed part inside the device. Just behind the door, there is a pocket for a small part of the inlet containing the changeable filter unit. The special filter protects the internal inlet part, ion chamber, and mass analyzer from impurities contained in the samples (**Figure 35** a)). You can control the door lock also from the manual mode using the door icon (**Figure 35** b))

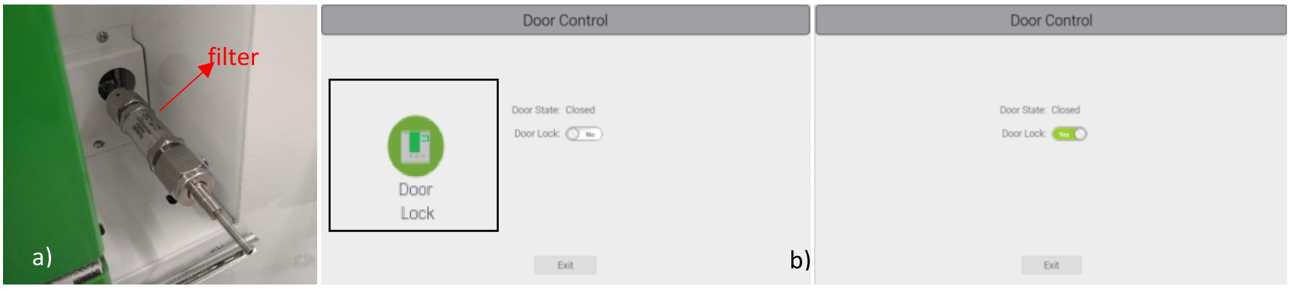
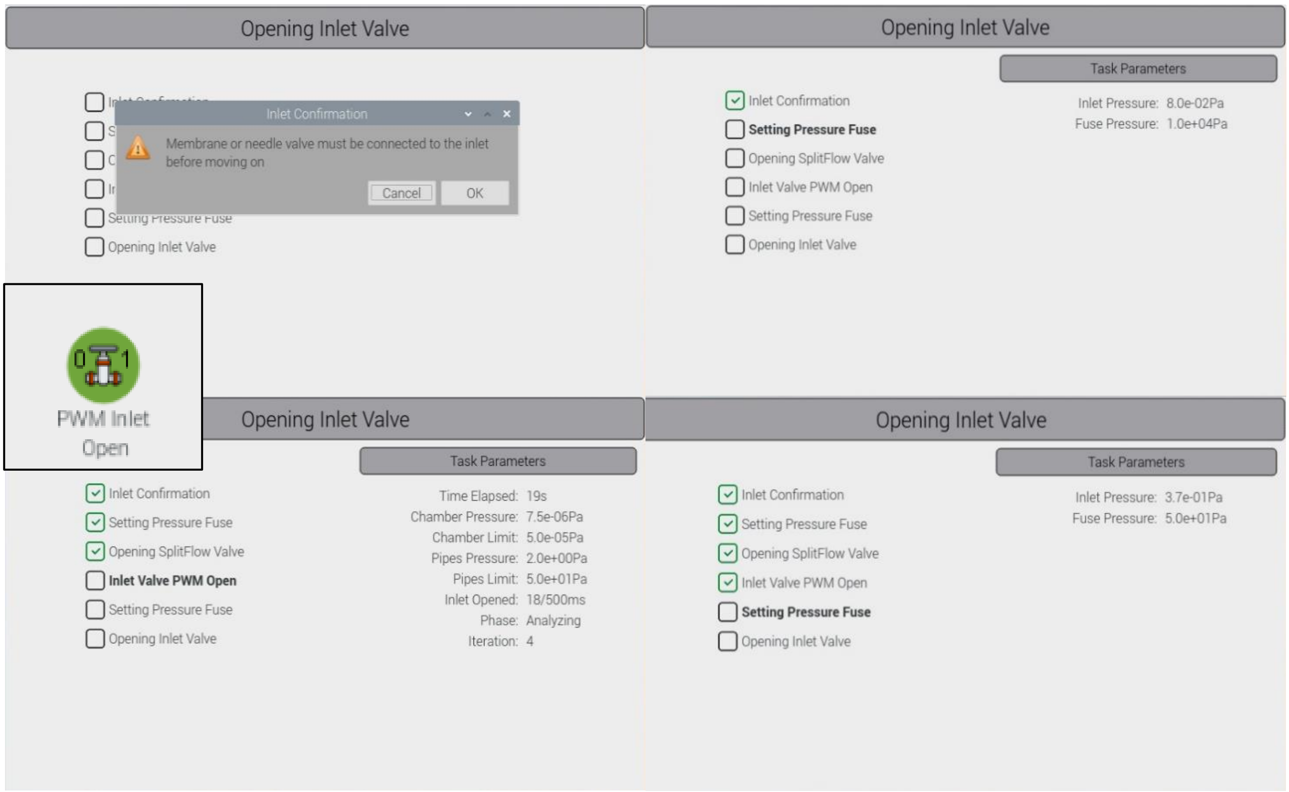


Figure 35*: a) Inlet filter; b) Door control*

Before the switch from Idle mode to Manual mode, it is not required the confirmation of a membrane probe or needle (valve) connection as in the case of automatic mode (**Chapter 4.4.3.2**). The reason is that after the switching in manual mode the external inlet of the device is not automatically evacuated. The evacuation is started only manually by touching on the icon **PWM Inlet Open**. After touching on the icon the sequence of automatic process is available. At the beginning of the evacuation, the confirmation of a membrane probe and or needle input (valve) is required (**Figure 36**).

⚠	Check the correct connection of external inlet – membrane probe, needle valve, and/or needle input using UT adapter 1/8’’x1/8’’ (**Figure 14**, **Figure 42**). The incorrect connection can damage the MS GAS pumps.
⚠	Make sure that the diaphragm of the membrane probe is not leaked. If the diaphragm is damaged, replace it! Then you can confirm the Inlet Confirmation table (**Figure 36**).



Membrane or needle valve must be connected to the inlet before moving on

☐ Setting Pressure Fuse
☐ Opening Inlet Valve

☑ Inlet Confirmation
☐ **Setting Pressure Fuse**
☐ Opening SplitFlow Valve
☐ Inlet Valve PWM Open
☐ Setting Pressure Fuse
☐ Opening Inlet Valve

Inlet Pressure: 8.0e-02Pa
Fuse Pressure: 1.0e+04Pa

☑ Inlet Confirmation
☑ Setting Pressure Fuse
☑ Opening SplitFlow Valve
☐ **Inlet Valve PWM Open**
☐ Setting Pressure Fuse
☐ Opening Inlet Valve

Time Elapsed: 19s
Chamber Pressure: 7.5e-06Pa
Chamber Limit: 5.0e-05Pa
Pipes Pressure: 2.0e+00Pa
Pipes Limit: 5.0e+01Pa
Inlet Opened: 18/500ms
Phase: Analyzing
Iteration: 4

☑ Inlet Confirmation
☑ Setting Pressure Fuse
☑ Opening SplitFlow Valve
☑ Inlet Valve PWM Open
☐ **Setting Pressure Fuse**
☐ Opening Inlet Valve

Inlet Pressure: 3.7e-01Pa
Fuse Pressure: 5.0e+01Pa

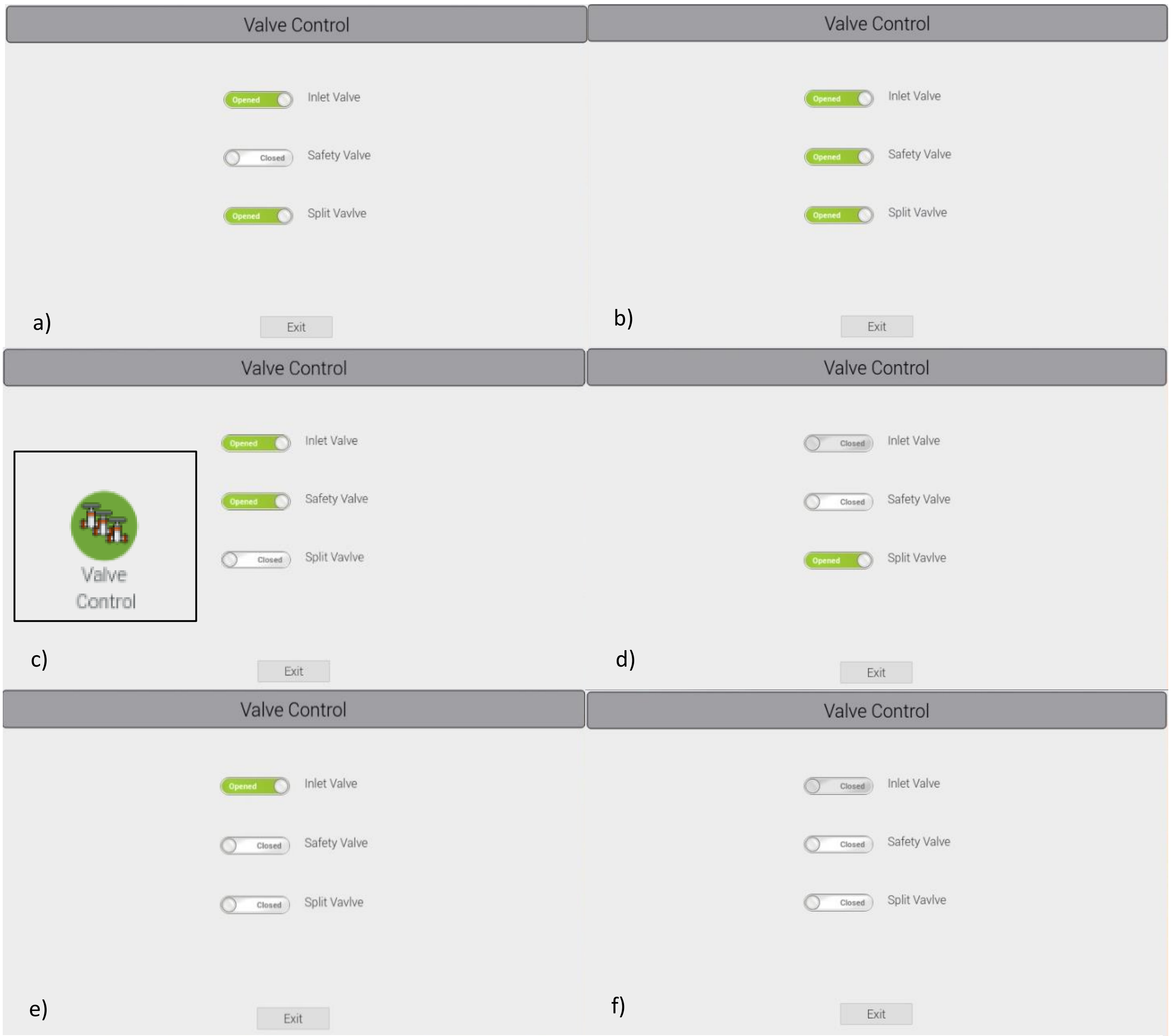
Figure 36*: Evacuation sequence*

In the manual mode, the state of valves is controlled by the **Valve Control** icon (**Figure 37**). Just after the evacuation of the device, the inlet valve and the split flow valve are opened, but the safety valve stays closed (**Figure 37** a)). Valve Control enables to open/close all protective valves as it is shown in **Figure 37**. The possibility of manually valves opening/closing is highly useful for many operations with MS GAS-100 including external inlet part exchanges.

For valve operations, it is necessary to know when it is possible to open/close which valve:

- The opening of the **inlet valve** is always conditioned by the connection of an external inlet to the MS GAS-100 input 1/8 "pipe (**Chapter 5.2**). The external inlet can thus only be changed if **the inlet valve is closed**.
- The **split flow valve** must always be **opened when defrosting** the system. The inlet valve and the safety valve must be closed at the same time.
- During device evacuation, the inlet and split flow valves are opened/closed automatically and the safety valve stays closed during this process. After the complete evacuation of the stainless-steel capillaries inside the device and the external inlet, both valves remain open while the safety valve stays closed in manual mode.
  A safety valve must be opened before starting the measurement of the samples. The ion source and Mass Spec inside the device are not protected after opening the **safety valve**, thus it can **only be opened if** we are sure that **the evacuation of the system is perfect and the external inlet is not damaged**.

⚠	Improper opening of the protecting valves will destroy the device.



a)
Valve Control
Opened Inlet Valve
Closed Safety Valve
Opened Split Vavlve

b)
Valve Control
Opened Inlet Valve
Opened Safety Valve
Opened Split Vavlve

c)
Valve Control
Opened Inlet Valve
Opened Safety Valve
Closed Split Vavlve

d)
Valve Control
Closed Inlet Valve
Closed Safety Valve
Opened Split Vavlve

e)
Valve Control
Opened Inlet Valve
Closed Safety Valve
Closed Split Vavlve

f)
Valve Control
Closed Inlet Valve
Closed Safety Valve
Closed Split Vavlve

***Figure 37**: Valves status*

Icon **Exit** switches the instrument from the manual mode back to the Idle mode (**Figure 18**). Following auto sequence controls default parameters of the device, mainly Stirling cooler temperature and vacuum values, and starts many automatic steps (opening/closing of valves, auto sublimation of capillary inlet, etc.) before switching to the Main Screen (**Figure 38**).

The state of valves is monitored in the information valve icon. Before switching to the Idle Mode inlet valve and safety valve are automatically closed while the split-flow valve is opened (**Figure 39**).

☑ Unlock Inlet Door
☑ Valve Handling 001
☑ Turning Heater Off
☐ **Setting Pressure Fuse**
☐ Defrost
☐ Setting Pressure Fuse
☐ Waiting for Vacuum

Inlet Pressure: 5.0e-02Pa
Fuse Pressure: 1.0e+04Pa

☑ Unlock Inlet Door
☑ Valve Handling 001
☑ Turning Heater Off
☑ Setting Pressure Fuse
☐ **Defrost**
☐ Setting Pressure Fuse
☐ Waiting for Vacuum

Current Temperature: -33.2°C
Min Temperature: 16.5°C
Phase: Defrost
Temp Ok Time: 0/60s

☑ Unlock Inlet Door
☑ Valve Handling 001
☑ Turning Heater Off
☑ Setting Pressure Fuse
☑ Defrost
☐ **Setting Pressure Fuse**
☐ Waiting for Vacuum

Inlet Pressure: 4.5e-02Pa
Fuse Pressure: 5.0e+01Pa

☑ Unlock Inlet Door
☑ Valve Handling 001
☑ Turning Heater Off
☑ Setting Pressure Fuse
☑ Defrost
☑ Setting Pressure Fuse
☐ **Waiting for Vacuum**

Current Pressure: 7.9e-06Pa
Target Pressure: 8.0e-04Pa
Chamber Temperature: 28.1°C
Pump Temperature: 34.0°C
Pump Power Input: 12W
Stable Time: 0/1s

Figure 38: *Clean-up sequence*

☑ Unlock Inlet Door
☑ Valve Handling 001
☑ Turning Heater Off

Current Temperature: -31.6°C
Min Temperature: 16.6°C
Phase: Defrost
Temp Ok Time: 0/60s

Inlet Valve: Closed
SplitFlow Valve: Opened
Safety Valve: Closed
Heating PWM: 0%
Cooling PWM: 0%

Figure 39: *Information about valves status*

4.5 DESCRIPTION OF MS GAS-100 REAR PANEL

The rear panel houses connectors for all connecting cables (**Figure 40**).

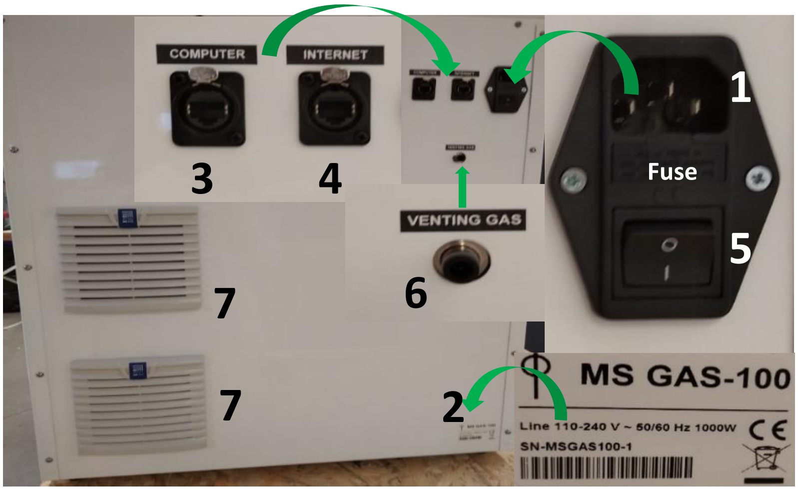


Figure 40*: MS GAS-100 rear panel:.* ***1)*** *Power supply connector* ***2)*** *MS GAS-100 label with serial number.* ***3)*** *Ethernet connector* ***4)*** *Firmware communication connector* ***5)*** *ON/OFF switch* ***6)*** *Input for Venting Gas* ***7)*** *Fans*

4.5.1 POWER SUPPLY CONNECTOR

The power connector is used to connect the MS GAS-100 to an electrical outlet using a power cord. The power cord is included in the MS GAS-100 package (**Figure 3**, point 1)

4.5.2 IDENTIFICATION LABEL WITH SERIAL NUMBER

Each MS GAS-100 device is assigned by the serial number after the final testing. The serial number is marked on the label (**Figure 40**, point 2).

4.5.3 ETHERNET COMMUNICATION CONNECTOR

Ethernet communication cable is provided as a part of the **MS GAS-100** package (**Figure 3**, point 3) for connecting the device with the notebook (**Figure 3**, point 2) via ethernet communication connector (**Figure 40**, point 3). The software Quadera (**Chapter 6.2**) installed in the connected notebook controls then all operations with the Mass Spec, mainly its calibration and tuning on selected weights of gases and volatiles, and the acquisition of measured data.

4.5.4 INTERNET COMMUNICATION CONNECTOR

The MS GAS-100 can be connected on the internet via an INTERNET communication connector (**Figure 40**, point 4). The grey Ethernet communication cable is in the basic package of the MS GAS-100 (**Figure 3**, point 4). The router inside the device enables the remote control of the firmware by PSI service support.

4.5.5 THE ON/OFF SWITCH

The switch is used to switch the machine off and on, respectively to apply electrical voltage to the source of the control computer inside the device.

4.5.6 VENTING GAS

The inlet labeled VENTING GAS (**Figure 40**, point 6) is used for external gas supply during the shutdown process. Inside the MS GAS-100, the supply line is terminated by an automatic venting valve for controlled aeration of the turbomolecular pump and other parts of the system. The pump is usually aerated with laboratory air, but the universal connector on the outer end of the aeration input allows the device to be connected to any gas from a gas cylinder or other source.

⚠	The input for venting gas must be opened all the time during the aeration of the system. If the system is not completely aerated during switching off, the next start can damage the turbomolecular pump.

4.5.7 VENTILATORS

RITTAL ventilators are situated on the rear side of the device (**Figure 40**, point 7). Their function is to keep the temperature at a stable range inside the device. It is necessary to keep their internal filters clean. In the basic package with MS GAS-100 you received two spare filters for their replacement (**Figure 3**, point 11).

5 INSTALLATION OF THE DEVICE

5.1 *FIRST OPERATIONS WITH MS GAS-100*

To install the device properly, it is necessary to follow the proper sequence of the assembly instructions as described below:

- Gas Analyzer should be placed on a flat, firm, and dry surface.
- All physical manipulation with Gas Analyzer **MS GAS-100** should be done with installed **locking screw** situated on the bottom of the device. Locking screw (**Figure 41**) fixes diaphragm backing pump to device frame and its wrong installation or device manipulation without tight screw causes serious damages of diaphragm backing pump function or its fixing.

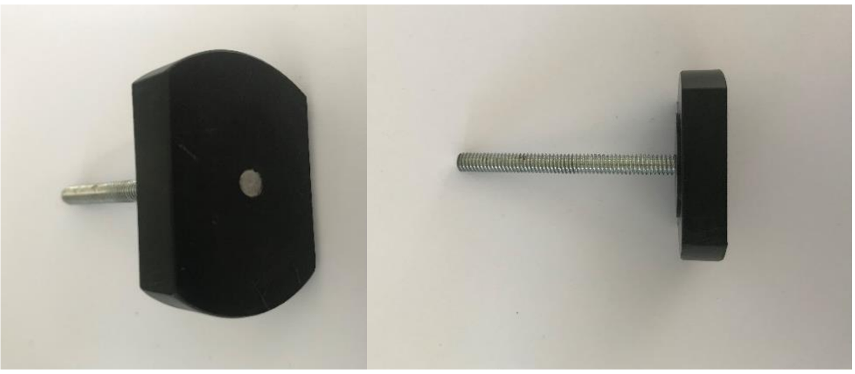


***Figure 41**: Locking screw.*

- Before any other operations with Gas Analyzer MS GAS-100, the **locking screw has to be taken off**. The device switch ON with a tight locking screw produces characteristic vibrations of diaphragm backing pump with a disagreeable strong sound. To remove the screw, tilt the device so that the base is visible from below and then unscrew the locking screw (screw has a black head, **Figure 41**).

ℹ	Locking screw length is about 7 cm, device weight is approx. 70 kg

- Make sure that the device is switched off – the **I/O switch** situated on the device rear side has to be in position 0 (**Figure 40**, point 5).
- Plug the **power cord** (**Figure 3**, point 1) of the MS GAS-100 into the connector on the device rear side placed above the I/O switch (**Figure 40**, point 1).
- Prepare a **membrane probe** (**Figure 3**, point 5) and make sure that the membrane is not damaged. If the surface of the membrane is damaged (there is a visible hole), you need to change it (**Chapter 5.2.1.1**)
- Take out the **protective cap** from the **pipe on the front side of the device** (**Figure 8**, point 2). Connect the pipe with one of the two screw of the **UT adapter 1/8'' x 1/8'' OD** (**Figure 42**). Do not remove completely the screw from UT adapter, just loosen it, put the 1/8'' OD tube of external inlet into the hole of the adapter screw (it is important to hear a weak click sound) and tighten it by hand.
- Connect the membrane probe with the second free screws of **UT adapter 1/8'' x 1/8'' OD** (**Figure 3**, point 8). The operation must be carried out in the same way as the connecting of external device inlet (**Figure 42**).

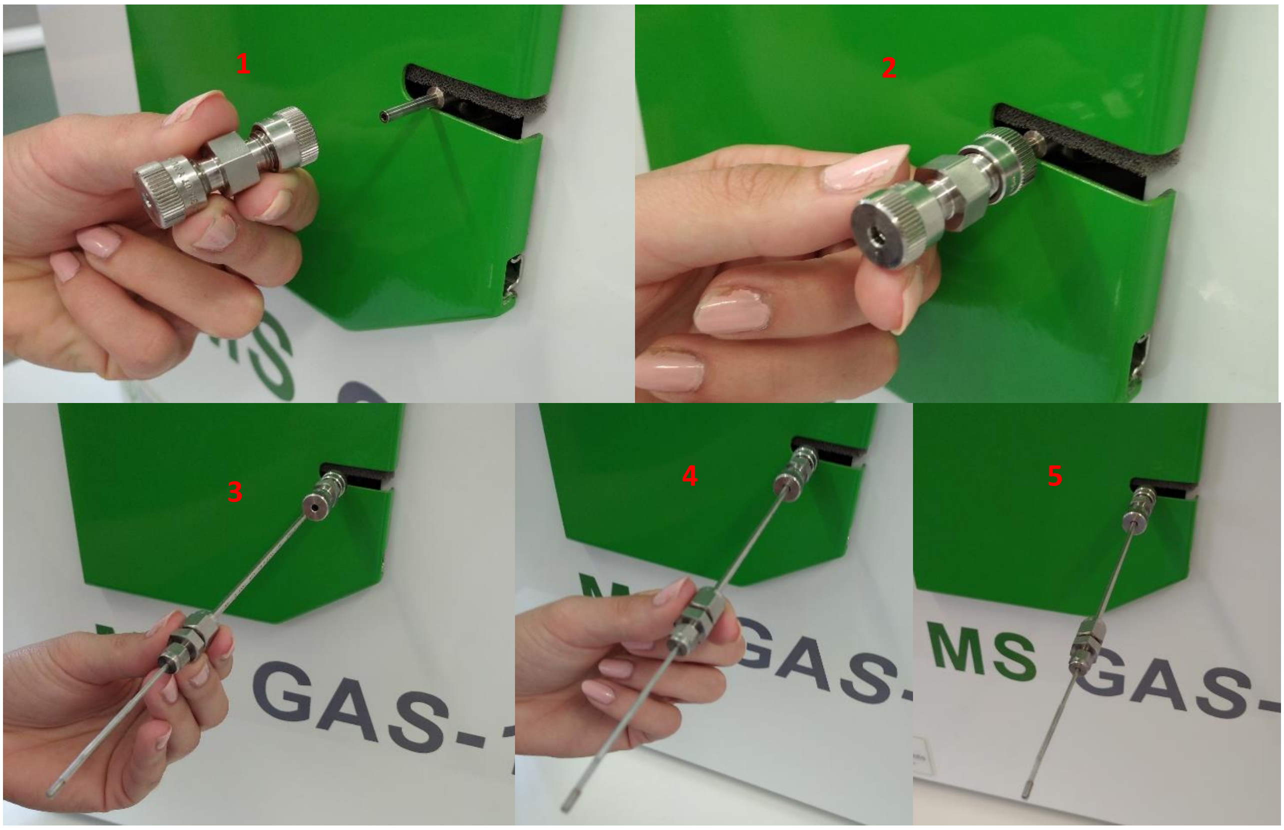


***Figure 42**: Membrane probe connection to MS GAS-100*

⚠	Check the correct connection of external inlet – membrane probe, needle valve, and/or needle input using UT adapter 1/8’’x1/8’’ (**Figure 42**, **Figure 47**, **Figure 48**). The incorrect connection can damage the MS GAS pumps.

- Connect the instrument to a 110/230 V outlet and flip the switch on the rear side to position 1 (**Figure 40**, point 5).
- Switch on the button under the touch screen (**Figure 8**, point 4) holding it for 3s. PC operating system starts – basic PC main screen with fundamental tests (**Figure 10**) is available finely. The vacuum system starts to evacuate ion source and a mass analyzer. All information about the instrument state related to time is visible on the touchscreen – description in **Chapter 4.4.3** .
- The device is prepared for followed operations after the main screen with choosing either AUTOMATIC or MANUAL MODE appears on the touchscreen.

⚠	For the perfect function of MS GAS-100 let evacuate the device for at least 24 hours before switching to selected mode.
ℹ	The recommended value of the vacuum pressure in the ion chamber is for other work at the range: $5.10^{-6} – 7.10^{-6}$ Pa ($5.10^{-8} – 7.10^{-8}$ mBar).

- For vacuum control switch to Manual mode or Automatic mode - the number next to Chamber Icon indicates the actual value of the vacuum in the ion chamber and mass analyzer (**Figure 20**, **Figure 26**). The evacuation process can be more effective and faster if the vacuum chamber was baked before 80 °C - 90 °C. The heating of cross chamber walls causes gas molecules to release from the wall surface, then the time of evacuation is shorter. The baking can be monitored using MID analysis with water molecule measurement as the mass 18 (the setting and start of MID protocol is described in **Chapter 6.2.6.2**). The ion chamber is automatically baked after the device start from the state of shut down (**Figure 15**). In automatic mode, the ion source chamber cannot be baked, while in the manual mode the baking is controlled by the user (**Figure 28**, **Figure 29**).
- The communication with Mass Analyzer inside the device is provided by Quadera control software installed in the notebook (**Figure 3**, point 2). Connect the MS GAS-100 with the notebook using the RED ethernet cable (**Figure 3**, point 3) and the COMPUTER ethernet connector on the rear side of the device (**Figure 40**, point 3). The active connection between the Mass Spec and the Quadera software is described in **Chapter 6.2.1** .
- The second ethernet communication connector (**Figure 40**, point 4) is used for PSI service support. Put the GREY ethernet cable (**Figure 3**, point 4) into the INTERNET connector and write us for the help. The remote communication with the device is useful mainly for device firmware update.
- The device is now ready for the measurement of samples.

5.2 EXTERNAL INLET PART

Before the start of gas analyses, the external part of the inlet should be installed with the followed evacuation of capillaries.

The installation of the external inlet (membrane probe, needle valve, or needle input) has to be performed strictly with the **closed Inlet Valve** (**Figure 37**, d) or f)). Just after the correct connection of the external inlet (**Figure 42**), the device can be completely evacuated. In the Manual mode the closing of the inlet valve is controlled by the user (**Figure 37**, d) or f)). Before switching in the Automatic mode the Inlet Confirmation table for external inlet connection is available (**Figure 19**).

Three types of external inlet are used depending on sample characteristics and properties.

5.2.1 MEMBRANE PROBE

The membrane probe is a fundamental external inlet. The probe is composed of the 1/8” tube, Let-Lok reduction adapter 1/8”x1/16” OD, 1/16” tube, small push spring, metal end piece (cap), and membrane of length about 2 – 3 cm. The 1/16” and 1/8” tubes can be of different lengths (**Figure 5**).

5.2.1.1 MS GAS-100 Basic Set

The basic MS GAS kit contains two membrane probes with different lengths of measuring part (1/16"tube) (**Figure 3**, points 5 and 6).

Due to repeated use of the membrane probe, the membrane surface may be damaged from time to time (there is a hole) and must be replaced. If a damaged diaphragm is used, the machine won't be evacuated.

The basic kit also contains 3 m of the spare membrane (**Figure 3**, point 7). Cut a piece of a new membrane (about 2-3 cm), insert the end cap from the original probe into one end of the membrane, and second membrane end insert a small spring. Then pull the membrane end with the spring over the surface of a 1/16 ”(thinner) pipe. The pipe would move the spring on the side of the metal end cap. The small spring inside the diaphragm must be next to the end cap and lightly touch the 1/16 '' tube. (**Figure 43**)

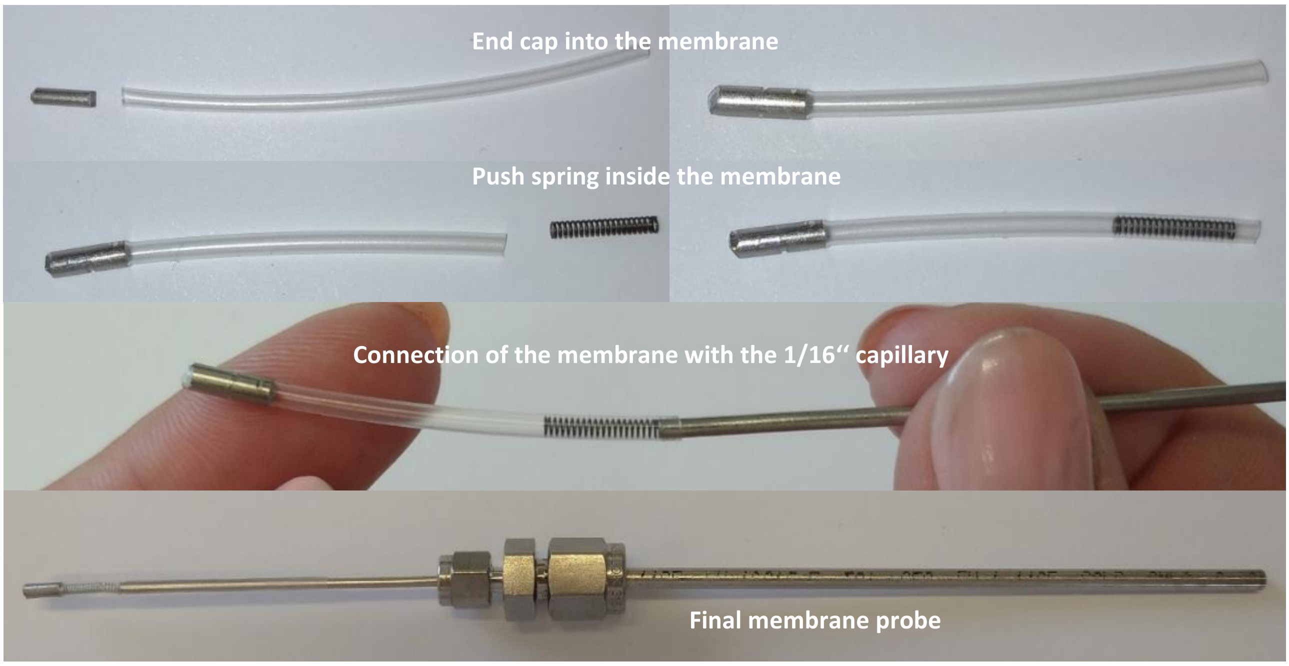


Figure 43*: The damaged membrane replacement*

5.2.1.2 Construction of a New Membrane Probe

All standard components for membrane probe preparation are part of the MS GAS-100 spare parts kit (**Figure 5**). Cut the required length of both pipes by 1/8 ”and 1/16” OD (The cutting procedure is the same for both pipes.) and insert the respective ends into the Let-Lok 1/8 ”x 1/16” OD adapter (**Figure 44**). Tighten both adapter nuts by hand first. Then fasten the assembly to the vice and tighten the nuts by a wrench by 3/4 turn. The next steps are identical to the membrane replacement in **Chapter 5.2.1.1** .

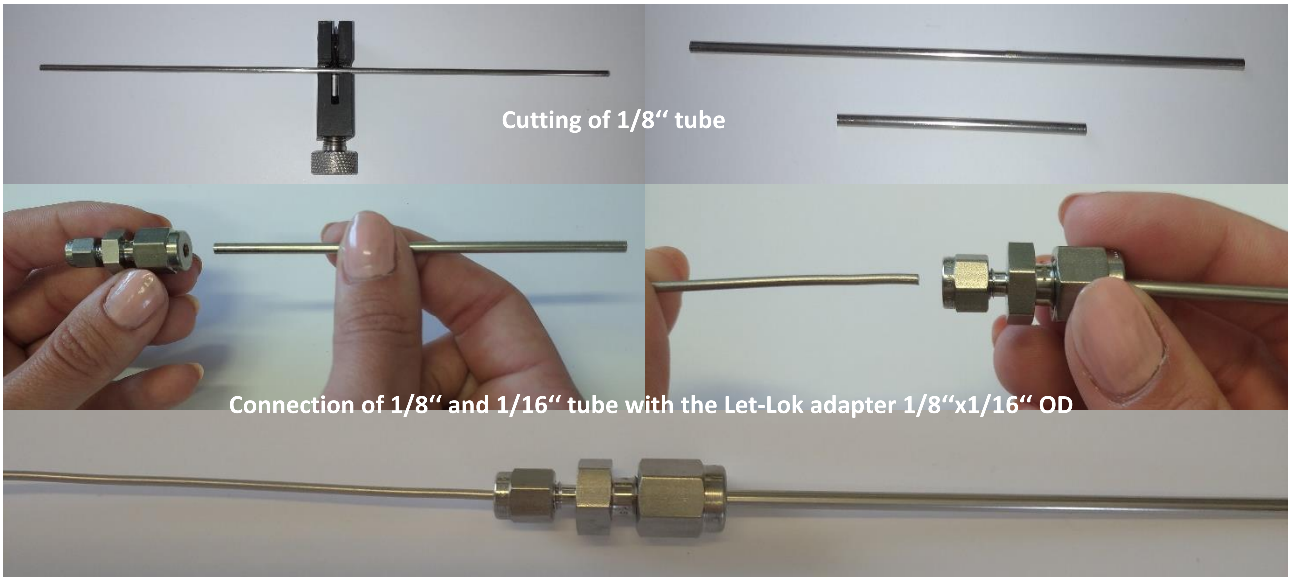


Figure 44*: Completion of the membrane probe using a spare part kit*

The MS GAS-100 with prepared membrane probe is primarily intended for measuring liquid samples, but can also be used for analyses in a gaseous environment. The connection of the membrane probe to the MS GAS-100 is described in **Chapter 5.1** , **Figure 42**.

5.2.2 NEEDLE VALVE

The needle valve set includes special components for needle valve completion. The kit contains two parts. The first part consists of the proper needle valve with manual regulation of the gas flow, the second part is formed by the flange with a stainless O-ring, an elastomer seal, and a 1/8" metal capillary for needle valve connection to the external inlet of MS GAS-100. The procedure of the described parts connection is shown in **Figure 45**.

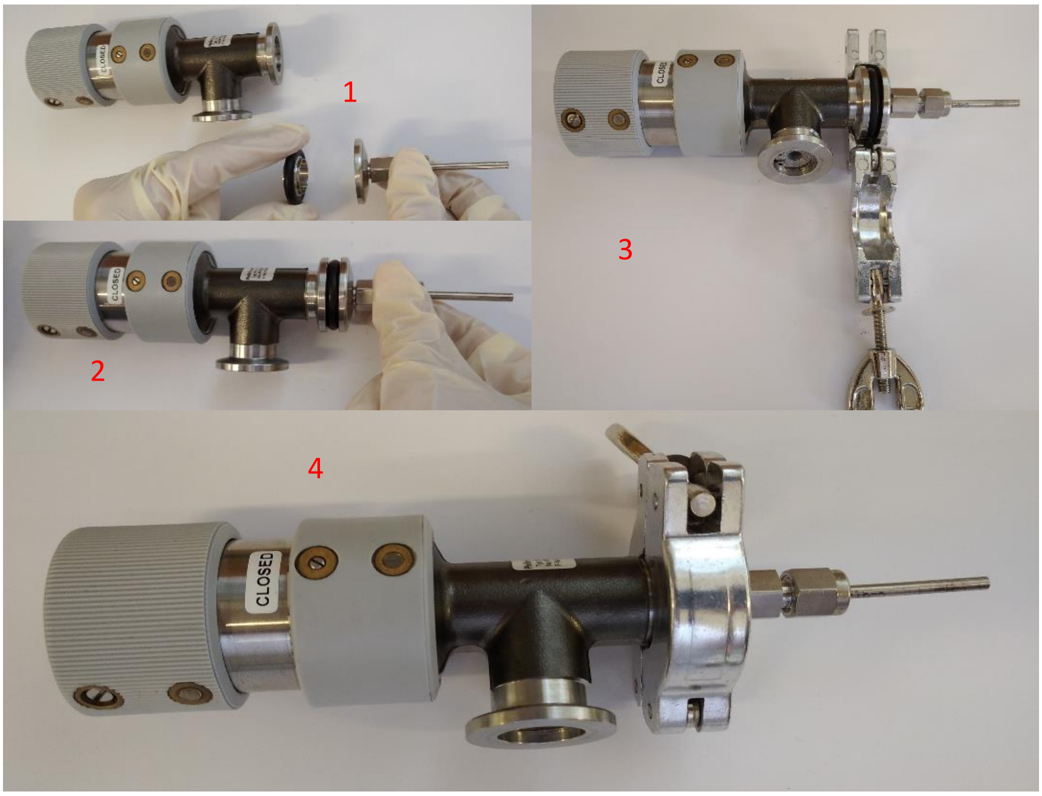


***Figure 45**: Completion of needle valve*

The sample volume depends on the opening degree of the valve. First, open the needle valve by OPEN-CLOSE port. Second, turn by the manual counterpart of the needle valve to obtain the pressure value 4 – 5.10^{-3} Pa (4 – 5.10^{-5} mBa) in the ion chamber of the gas analyzer (**Figure 46**).

The needle valve with manual regulation serves for direct inlet of strictly gaseous samples.

⚠	Open the first part of the manual needle valve very slowly because of pressure impact!
ℹ	Look at the counter of needle valve – the optimal pressure for MS analyzer use should be obtained around the value 110.
⚠	The pressure in the ion chamber (**Figure 26**) must not be higher than 3.10^{-3} Pa (3.10^{-5} mBa).

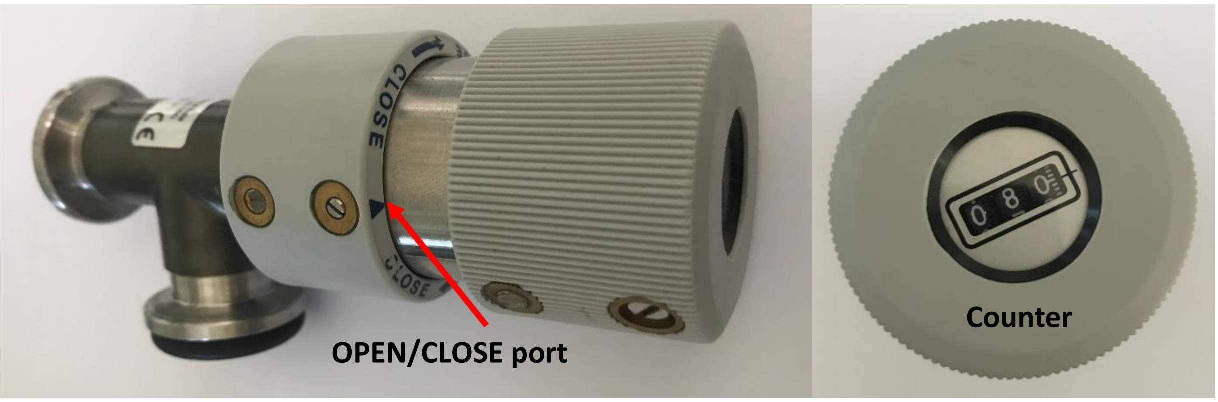


***Figure 46**: Needle valve_OPEN/CLOSE port and the manual counter*

The connection of complete needle valve is similar as in the case of membrane probe. The connection procedure is shown in **Figure 47**.

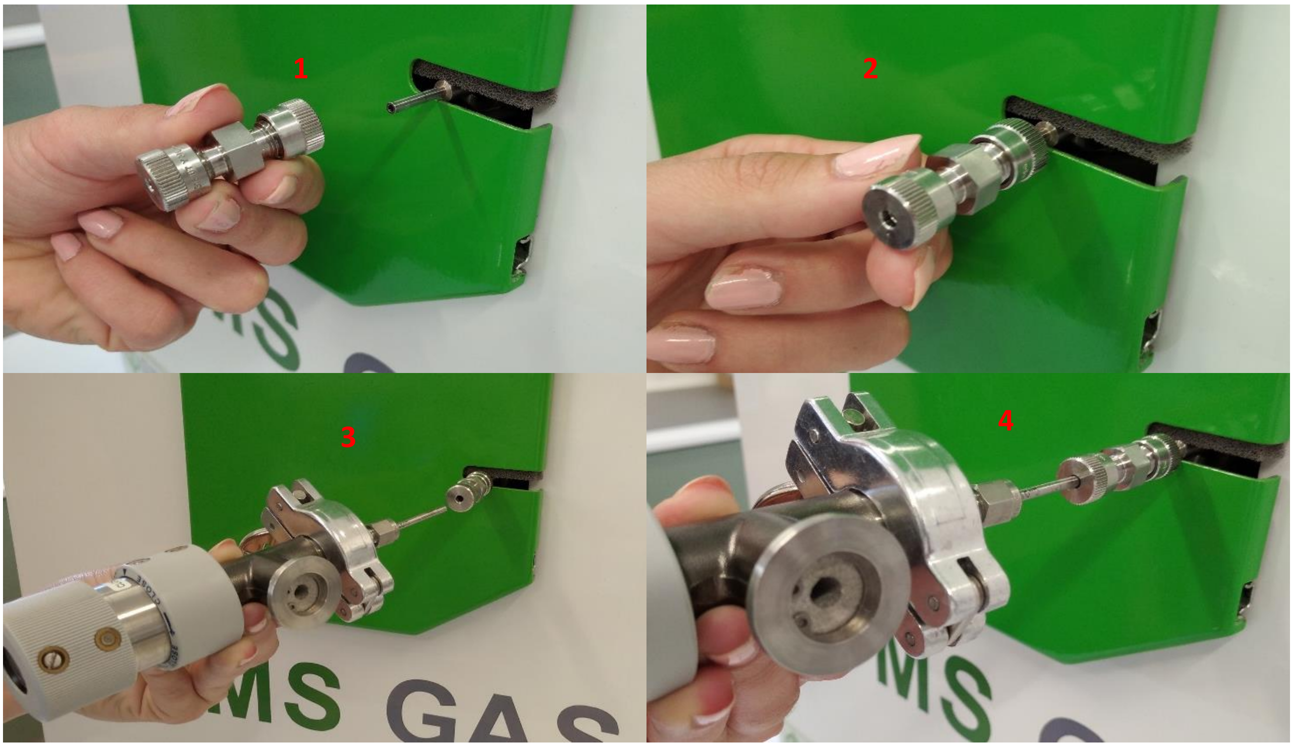


***Figure 47**: Needle valve connection to the device*

5.2.3 NEEDLE INPUT

The needle input (**Figure 2**) serves as well as the manual needle valve (**Chapter 5.2.2**) for direct inlet of only gaseous samples and is designed for direct connection to the MS GAS-100 via the integrated UT adapter 1/8'' x 1/8'' OD.

Connect the needle input using the free screw of **UT adapter 1/8'' x 1/8'' OD**. Do not remove completely the screw from UT adapter, just loosen it, insert the free inlet pipe of the MS GAS-100 into the hole of the adapter (it is important to hear a weak click sound) and tighten it by hand (**Figure 48**).

***Figure 48**: Procedure of needle input connection to MS GAS-100*

5.3 OTHER OPTIONAL FEATURES AND ACCESSORIES

5.3.1 SET FOR MODULAR INLET PREPARATION

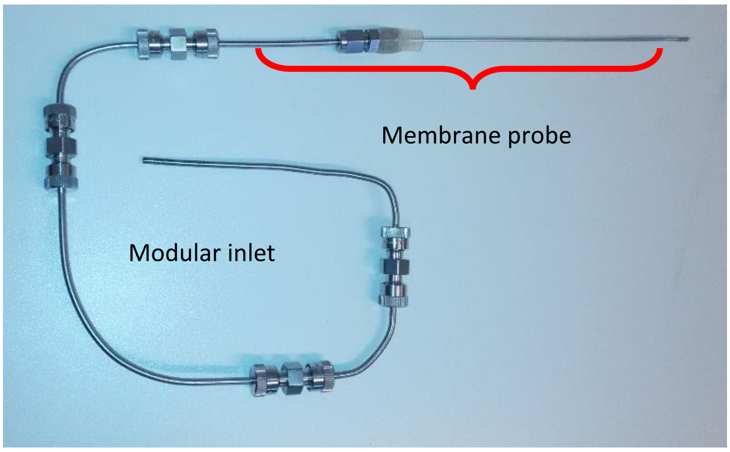


***Figure 49**: External modular inlet ended by a membrane probe*

The set includes special coupling UT adapters 1/8“x1/8“ OD and 1/8“ seam-less pipes for preparation of modular inlet (**Figure 6**).

The inlet with modular function allows the connection of the gas analyzer with other devices. The 1/8‘‘ pipes can be cut on different lengths and bent. An example of the MS GAS-100 connection with the Photobioreactor FMT 150 via external modular inlet is shown in **Figure 1**. Using these special vacuum adapters doesn't affect the final signal on the Mass Spec (**Figure 49**).

5.3.2 SPECIAL COMPONENTS KIT

The set contains special reducers and spare parts. The most important is the **filter union** (**Figure 35** a)), which is located just behind the MS GAS-100 door. The filter protects the components of the device inside the instrument from contaminated samples. The filter element must be replaced, in particular, when the detection of gases and volatile substances is very difficult in comparison with previous analyses.

For all device components, a special type of VCR vacuum connection is used. The manipulation with the VCR connector and the special Ni gasket is shown in **Figure 50**.

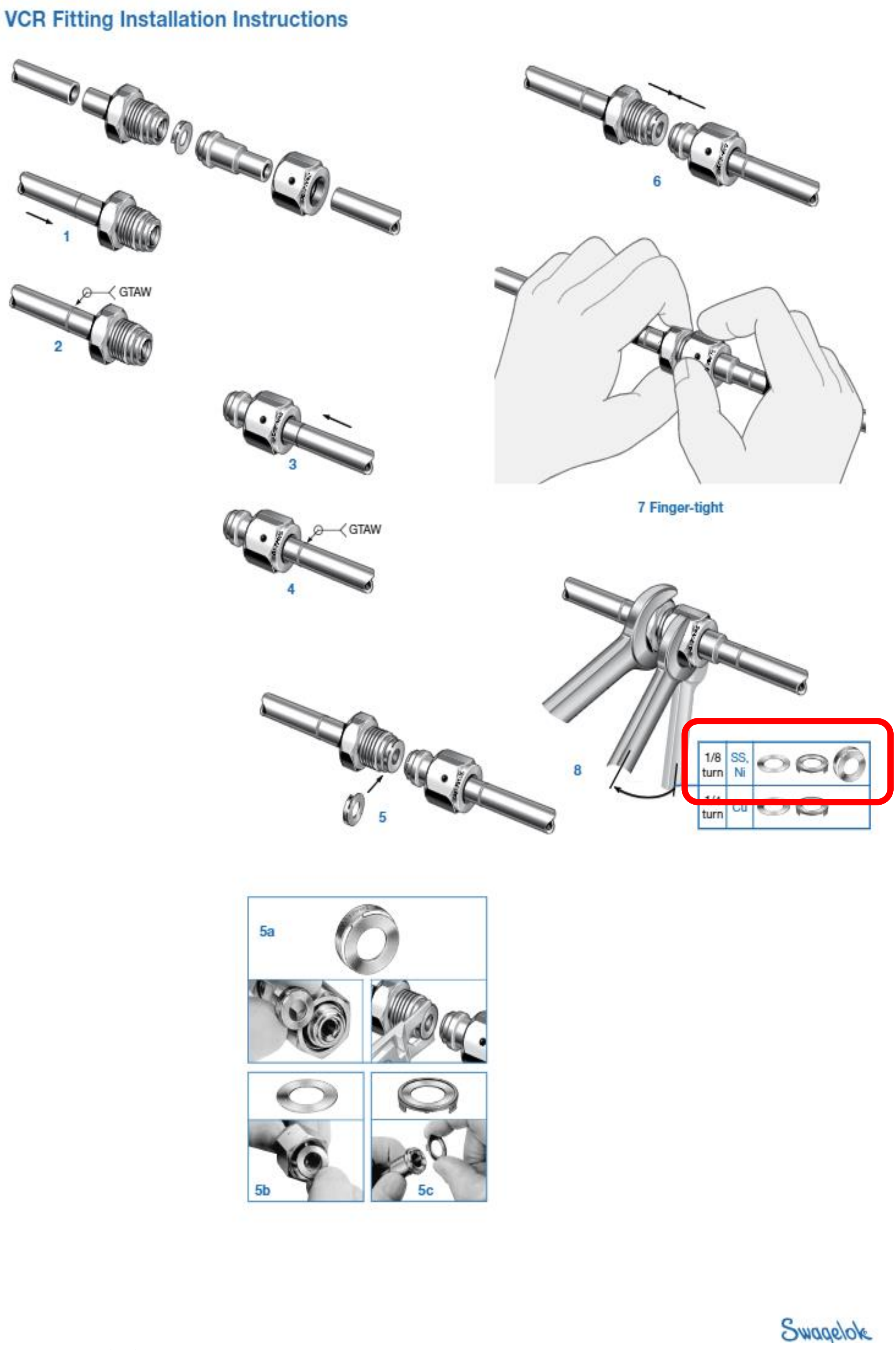


Figure 50*: VCR fitting (de)installation_original document from Swagelok*

5.3.3 PLANT CHAMBER

The airtight chamber can be used for long-term monitoring of gases and volatile compounds, for monitoring gas exchange in plants during photosynthesis and for many other applications. The chamber is made from special plexiglass with a high permeability to the light. The chamber is equipped with a pressure relief valve as well as valves that can replace the atmosphere in the chamber during measurement. A manual needle valve is used as the sampling port (**Chapter 4.3.6** , **Figure 7**).

The chamber is primarily intended for plant analysis. Plants can reach two defined heights thanks to the offered classic bottom and one bottom with reduction. The handling of the chamber is very simple and its parameters can be adjusted according to the customer's ideas.

The **Figure 51** shows an example of the gas exchange measurement with celery inside of the plant chamber.

Figure 51*: The connection of the Plan chamber with MS GAS-100 1) via modular inlet (Chapter 5.3.1) 2) sample input via needle valve (Chapter 5.2.2)*

6 USER'S GUIDE FOR MEASUREMENTS OF GASES

6.1 INTRODUCTION TO MASS SPECTROMETRY

6.1.1 ION SOURCES

Before the actual analysis of the gas in the mass filter, it is necessary to ionize the gas molecules. Ionization occurs by electron impact (electron beam bombardment) in the ion source.

In an ion source, electrons are emitted from an electrically heated cathode (filament). The electrons are accelerated by the voltage applied between the anode and cathode. Neutral gas molecules that are present in the formation space between the anode and cathode are ionized by collisions between electrons and form single and multiple positive or negative ions (**Figure 52**). The energy of the colliding ions has a significant effect on the number and type of ions that are formed.

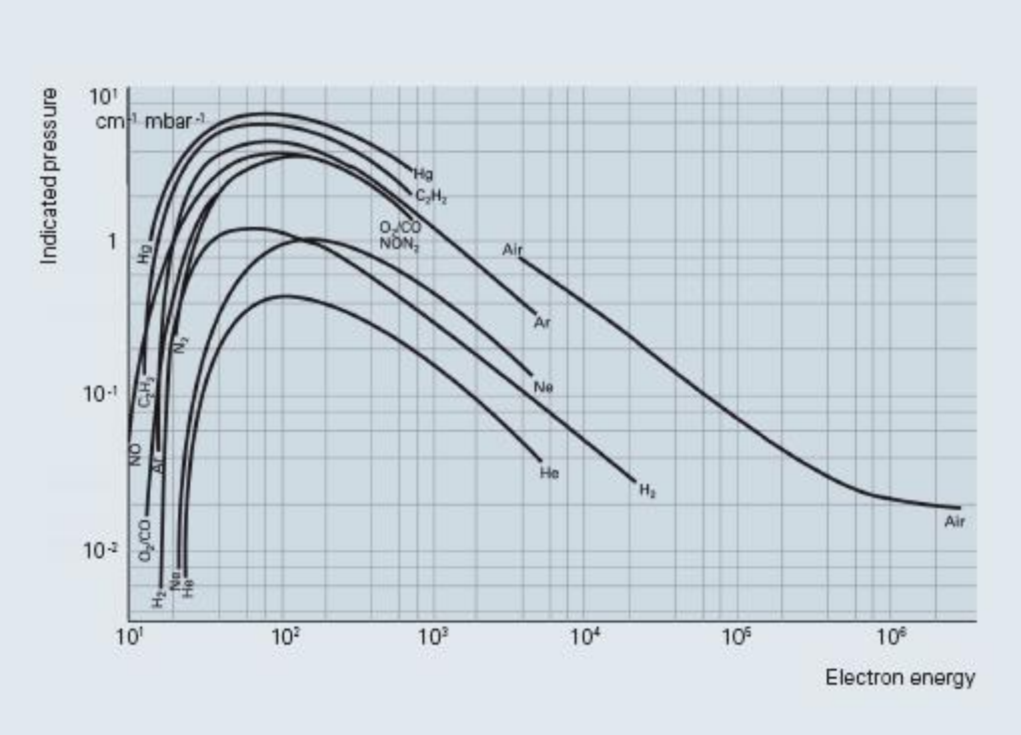


***Figure 52:** Ion density as a function of electron energy (Source: Pupp / Hartmann, Vakuumtechnik, Grundlagen und Anwendungen, Hanser Verlag)*

The ionization of neutral particles already takes place with minimum electron energy between 10 and 30 eV (appearance potential). The number of ions formed increases rapidly with increasing electron energy (accelerating voltage), reaches a maximum at 50 to 150 eV depending on the type of gas, and slowly decreases while the energy is constantly increasing. Because the yield in ions, and thus the sensitivity of the mass spectrometer, should be as high as possible, electron energies between 70 and 100 eV are usually used (**Figure 52**).

The ionic current IK + of the gaseous component K can be calculated from the following relation (**Formula 1**):

Formula 1: Ion current $I_{K+} = i\_ \cdot l_e \cdot s \cdot p_K$ where:

i_- = electron current (emission current), in [A]

l_e = mean path length of the electrons, in [cm]

S = differential ionization effect cross section K, in [1 /(cm · mBar)]

p_K = partial pressure of the gas component K, in [mBar]

Many types of ions are formed when ionizing complex molecules. In addition to single- and multiple-charge molecule ions (ABC^+, ABC^{++}) fractal ions also occur:

- $ABC^+ + 2e^-$
- $ABC^{++} + 3e^-$
- $AB^+ + C + 2e^-$
- $BC^+ + A + 2e^-$
- $A^+ + BC + 2e^-$
- $C^+ + AB + 2e^-$
- $B^+ + A + C + 2e^-$

In addition to these types, it is also possible for recombination ions, such as AC^+, to form. The occurrence and relative frequency of individual types of ions are characteristic of a certain type of molecule and serve as an important aid in identifying the molecule, and thus as an aid in qualitative gas analysis. **Figure 53** shows the fragment ion distribution (cracking pattern or fractal pattern) of the simple molecule CO_2, recorded at 70 eV of electron energy.

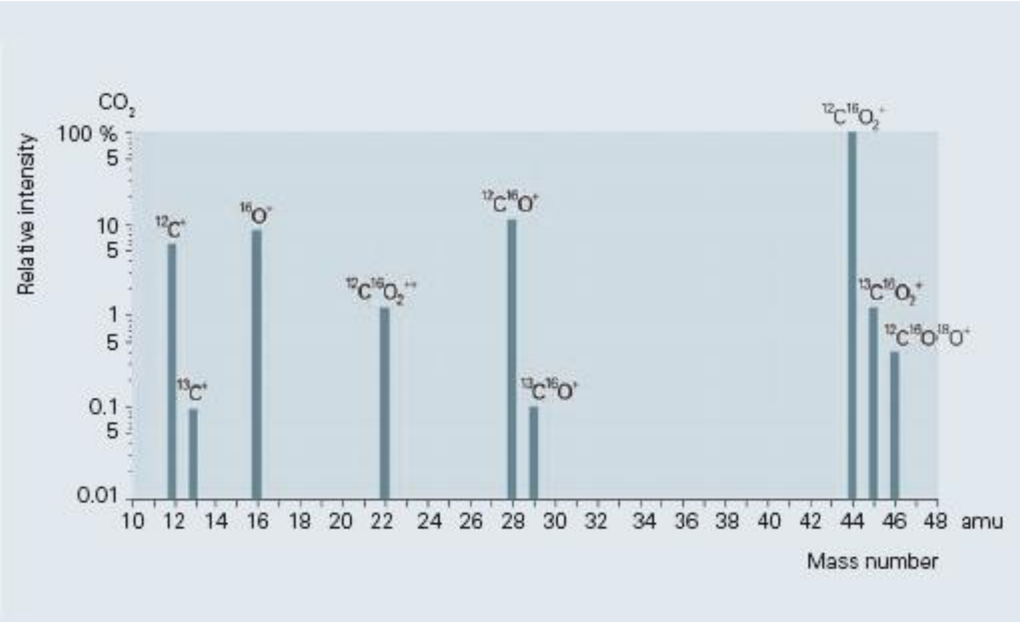
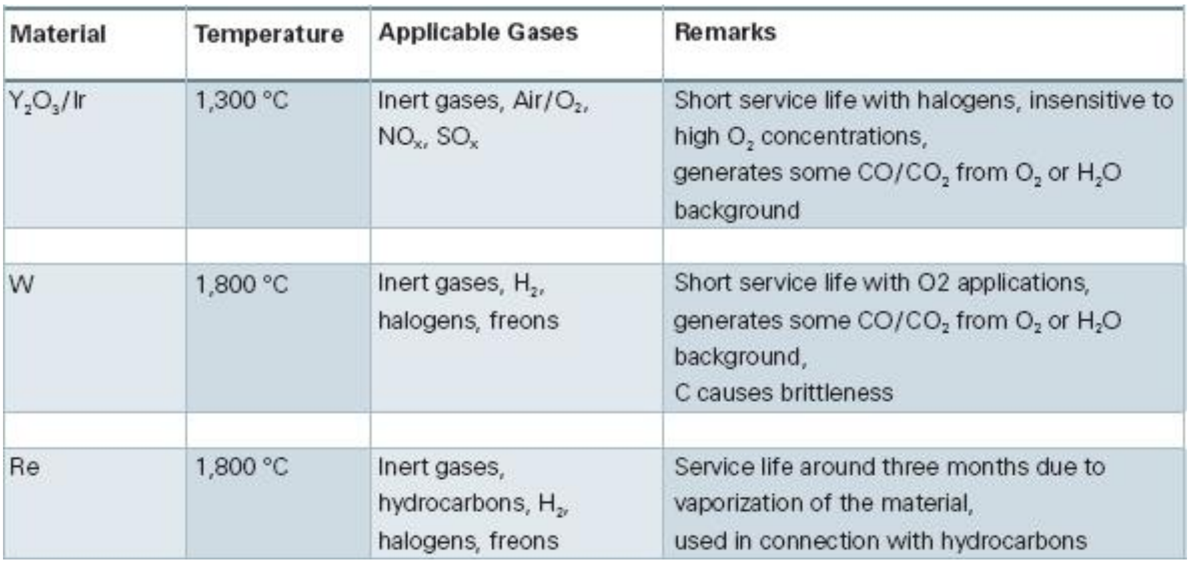


Figure 53: *Fractal ion distribution of CO_2*

The selection of an ion source and optimal filament material is based upon the requirements imposed by the measurement task. Applications often impose contradictory requirements on the ion source. To achieve optimal results, the ion source must be matched to the task at hand. This has resulted in the development of different types of ion sources that can almost all be equipped with cathodes made of rhenium (Re), tungsten, or yttriated iridium (Y_2O_3/ Ir).

T-cathodes are preferred in the UHV range or where the vapor pressure of Re could have a disturbing effect (**Table 1**). However the brittleness of tungsten cathodes due to the tungsten /carbon/ oxygen cycle must be taken into account; i.e. due to the formation of W_2C. Yttriated iridium is increasingly being used today instead of the pure metal cathodes that were used in the past. The advantages offered by these cathodes are significantly lower operating temperature and relative insensitivity to air inrush. Consequently, the preferred fields of implementation for these cathodes are analysis of temperature-sensitive substances, e.g. metal-organic compounds, or analysis of contaminants in gas mixtures containing a high concentration of oxygen.

The various ion sources are described in **Chapter 6.1.2** based on their attributes and fields of application. What all ions have in common is that they can be electrically biased up to 150 V. This avoids signal background due to ESD (electron stimulated desorption) ions.



Material	Temperature	Applicable Gases	Remarks
Y_2O_3/Ir	1,300 °C	Inert gases, Air/O_2, NO_x, SO_x	Short service life with halogens, insensitive to high O_2 concentrations, generates some CO/CO_2 from O_2 or H_2O background
W	1,800 °C	Inert gases, H_2, halogens, freons	Short service life with O2 applications, generates some CO/CO_2 from O_2 or H_2O background, C causes brittleness
Re	1,800 °C	Inert gases, hydrocarbons, H_2, halogens, freons	Service life around three months due to vaporization of the material, used in connection with hydrocarbons

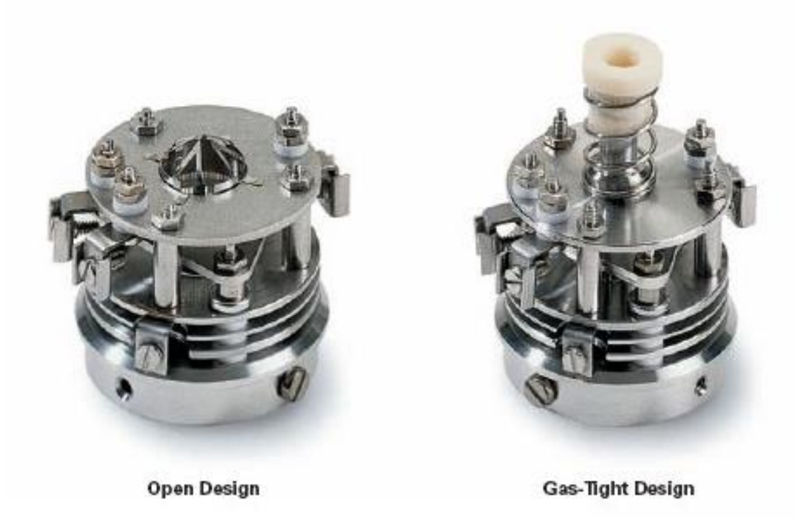
Table 1: *Filament materials and their employment*

6.1.2 STANDARD PRISMA ION SOURCE

The PrismaPlus and/or PrismaPro mass spectrometers from Pfeiffer Vacuum are equipped with a robust and highly sensitive ion source especially suitable for residual gas analysis. Ion source has two cathodes, thus affording particularly secure operation. Both an open version as well as a gas-tight version with gas inlet in the axial direction are available.

All ion sources described here ionize using electron collision (EI = electron impact). The ion sources can be categorized into two groups:

- Open ion sources are used if the process gas is to be analyzed and additional pressure reduction is not required
- Closed ion sources are used in analytical applications, for example, to require only small volumes of gas or to increase sensitivity relative to the substrate of the vacuum system. Closed ion sources are used in combination with a differentially pumped system (**Figure 54**) to analyze higher-pressure gases.



Open Design

Gas-Tight Design

Figure 54: *PrismaPlus™ ion source*

6.1.3 IONS DETECTION – QUADRUPOLE MASS FILTER

The filter system of a quadrupole mass spectrometer consists of four parallel rods arranged in the form of a square. Each pair of opposite rods in **Figure 55**, designated (+) or (−), is connected with the other. Voltage is applied between the two pairs of rods.

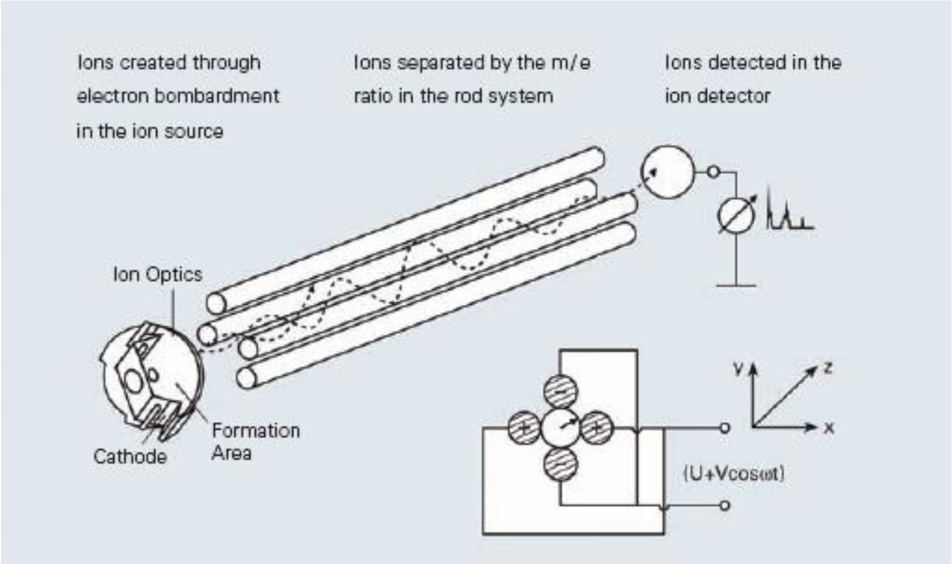


Figure 55: *Operating principle of a quadrupole mass spectrometer*

At this point, only a brief phenomenological description of the operating principle will be provided. Ideal quadrupole fields require rods that have a hyperbolic profile. In actual practice, however, round rods are used, with the rod radius being equal to 1.144 times the field radius r_0. An electrical quadrupole field is formed between the rods. Ions of varying mass are shot axially into the rod system at approximately equal energy and move through the rod system at uniform velocity. The applied quadrupole field deflects the ions in the X and Y directions, causing them to describe helical trajectories through the mass filter.

Formula 2: *Quadrupole deflection voltage*

$$U_1 = U + V \cdot \cos \omega t$$

To solve the movement equations, the dimensionless variables are introduced to obtain Mathieu's differential equations. Their solutions yield the stable area with oscillation amplitudes of less than r_0 beneath the triangle formed by the two solubility curves in **Figure 56**. The values a_p = 0.23699 and q_p = 0.706 apply for the apex of the triangle. All solutions outside result in increasing oscillation amplitudes and thus in the neutralization of the ions on the rods of the quadrupole filter.

Dividing the two equations by one another yields: This is the pitch of the so-called load line of the mass filter (**Figure 56**).

From **Figure 56**, it can be seen that: $\frac{a}{q} = 2\,\frac{U}{V}$

- All ions whose parameters **a** and **q** (**Formula 3** and **Formula 4**) are located in the triangle above the load lines will reach the detector
- The ions will only reach the detector under-voltage conditions: $\frac{U}{V} = \frac{a_p}{2 \cdot q_p}$ < 0,1678.

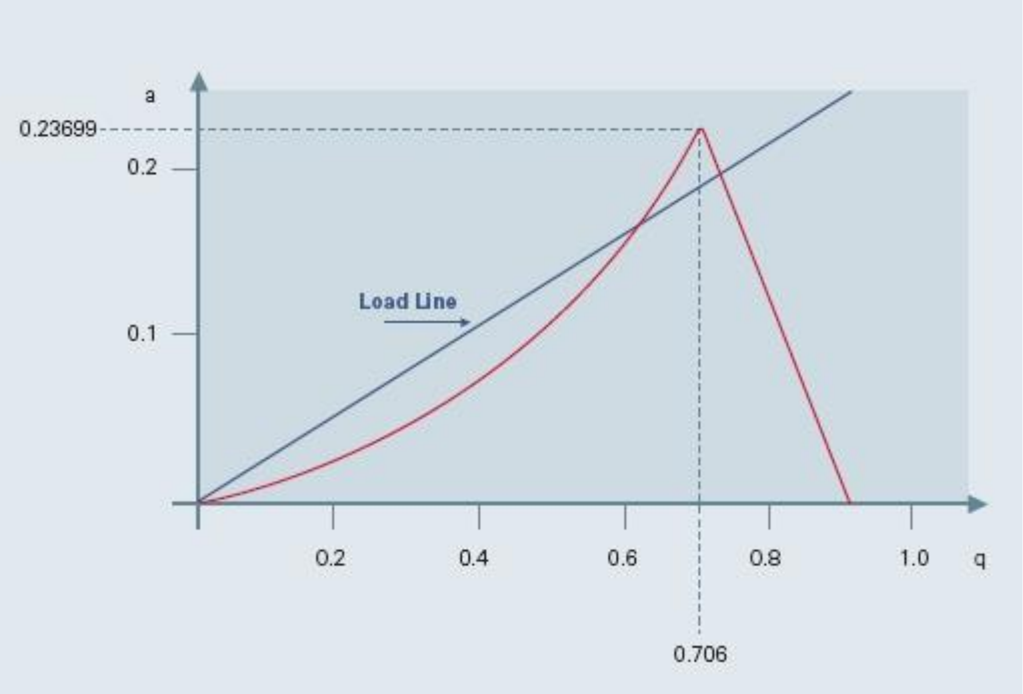


Figure 56: *Stability diagram of a quadrupole filter*

Formula 3: *Stability parameter* ***a***

$$a = \frac{8 \cdot e \cdot U}{m \cdot r_0^2 \cdot \omega^2}$$

Formula 4: *Stability parameter* ***q***

$$q = \frac{4 \cdot e \cdot V}{m \cdot r_0^2 \cdot \omega^2}$$

Introducing the ratio between the atomic mass unit 1 amu = 1.66055. 10^{-27} kg and the elementary charge and multiplying it by the dimensionless mass number M (**Formula 5**) of the corresponding ion yields the following conditions for *U* and *V* for the apex of the stability triangle (**Figure 56**).

Formula 5*: Elementary charge multiplied by the dimensionless mass number M:*

$$e = 1{,}602 \cdot 10^{-19}\,\mathrm{A \cdot s}\,\frac{m_{amu}}{e} = 1{,}0365 \cdot 10^{-8}\,\frac{kg}{A \cdot s}$$

Formula 6: *Stability condition* **U**

$$U = 1.2122 \cdot 10^{-8}\,\frac{kg}{A \cdot s} \cdot M \cdot r_0^2 \cdot f^2$$

and

Formula 7: *Stability condition* **V**

$$V = 7.2226 \cdot 10^{-8}\,\frac{kg}{A \cdot s} \cdot M \cdot r_0^2 \cdot f^2$$

With the DC voltage de-energized, *U* = 0, all trajectories of the ions where $q < 0.905$ will be stable; according to Formula 6, these will all be masses where the condition from **Formula 8** is done.

Formula 8*: High-pass condition*

$$M > \frac{1.0801 \cdot 10^{7} \cdot V}{r_0^2 \cdot f^2} \cdot \frac{A \cdot s}{kg}$$

The filter thus acts as a high pass. As HF amplitude (*V*) increases, ever-heavier types of ions become unstable, starting with the light masses, and are thus sorted out. This operating mode produces an integral spectrum.

The shot conditions are crucial for the transmission of ions through the filter. Ions parallel to the rod system must be shot within the following diameter (**Formula 9**).

Formula 9: *Shot orifice*

$$D = \frac{1}{2} \cdot r_0 \cdot \frac{M}{\Delta M}$$

The maximum shot angle must satisfy the condition from **Formula 10**.

Formula 10: *Shot angle*

$$tg\psi < 11.85 \cdot \frac{r_0^2}{L^2}$$

And the energy must be as uniform as possible.

For the amplitudes of the unstable ions to become large enough to strike the rods, where they are neutralized, these ions must perform a minimum number of oscillations in the separating field. The following equation (**Formula 11**) applies to the maximum acceleration voltage in the Z direction.

Formula 11: *Maximum acceleration voltage U_{zmax}*

$$U_{zmax} \approx 4.2 \cdot 10^{-6} \frac{kg}{A \cdot s} \cdot L^2 \cdot f^2 \cdot M \cdot \frac{\Delta M}{M}$$

In practical operation, the ratio *U/V* is activated as a function of the mass number in such a manner that the actual resolution *ΔM/M*, does not remain constant, but that instead the line width *ΔM* remains constant. This means that resolution increases proportionally to the mass number. Due to **Formula 7** (*V* is proportional to *M*), the quadrupole (as opposed to the sector field mass spectrometer) produces a linear mass scale.

One point of major significance for a QMS is the required HF power. If *C* is used to designate the entire capacity of the system and *Q* to designate the factor quality of the power circuit, the required HF power will increase with high powers off and r_0. An enlargement of field radius r_0 will lessen the occurring relative mechanical tolerances, thus resulting in improved behavior. Essentially, it is advantageous to select f_0 and r_0 as large as possible. However, there are limits in this regard due to the associated increase in HF power (**Formula 12**). While extending the rod system permits a lower operating frequency, the size of a production unit should not exceed certain dimensions.

Formula 12: *HF power*

$$N_{HF} \approx \frac{C}{Q} \cdot M^2 \cdot f^5 \cdot r_0^4$$

The required mass range and desired resolution are governed by the dimensions of the filter and the selection of the operating frequency. Devices with 6, 8, and 16 mm rod diameters and appropriately matched electronics are available to satisfy the respective requirements.

What follows is a brief digression on the relationship between resolution and mechanical precision. Let us consider a quadrupole mass filter that works at the apex of the stability diagram; i.e. that works at high resolution (**Formula 13** and **Formula 14**).

Formula 13 *applies for the DC amplitude*

$$U = 1{,}2122 \cdot 10^{-8} \frac{kg}{A \cdot s} \cdot M \cdot r_0^2 \cdot f^2$$

Formula 14 *applies for the AC amplitude.*

$$V = 7{,}2226 \cdot 10^{-8} \frac{kg}{A \cdot s} \cdot M \cdot r_0^2 \cdot f^2$$

Here, *M* designates the mass of the ion, r_0 the field radius, and *f* the frequency at which the filter is operated. We are making the idealized assumption that both voltages *U* and *V*, as well as frequency *f*, can be set and maintained "as precisely as desired".

What follows from this is **Formula 15** (c_k is a constant), and following differentiation and division by *M*, the filter scatter caused by r_0 is expressed by
Formula 16.

Formula 15*: Mass of the ion*

$$M = c_k \cdot \frac{1}{r_0^2}$$

Formula 16: *Scatter*

$$\frac{dM}{M} = \frac{-2 \cdot dr_0}{r_0}$$

Let us assume that the field radius r_0 changes by Δr_0 = 0.03 mm over the length of the mass filter. Now let us consider the effect of this change to the scatter *ΔM/M* for two mass filters of different sizes. For optimal transmission, the resolution set on the spectrometer (we select: *ΔM/M* = 1/100) must be greater than the scatter generated by the fluctuation of r_0 (**Formula 17**).

Formula 17*: Mass Spec resolution*

$$\frac{\Delta M}{M} > \frac{-2 \cdot \Delta r_0}{r_0}$$

For a filter (a) having a field radius of 3 mm, this results in *ΔM/M* = 0.02, i.e. a contradiction; and for a filter (b) having a field radius of 12 mm, this results in *ΔM/M* = 0.005, i.e. coincidence with the above condition. In other words: If a resolution of *ΔM/M* = 0.01 has been set for both filters, most of the ions will not be able to pass through the filter in case (a). In the case of the larger filter (b), all ions will be able to pass through the filter, since the set resolution is greater than the scatter.

Although this simplified error calculation does not nearly take into account all of the effects that can contribute to transmission, it does teach several fundamental relationships:

The field radius must be maintained significantly better than 1% over the entire length of the filter, depending on the selected mass range. Fluctuations in the field radius will lead to transmission losses

The larger the dimensions of the rod system are selected, the lower the influence of the absolute mechanical tolerances will be

The higher the mass range, and if differentiation between adjacent masses is still to be made, the stricter will be the requirements relating to the relative accuracy of the mass filter. In this simple picture, the required relative accuracy is scaled linearly with the mass range

6.1.4 SUMMARY

A quadrupole mass filter is a dynamic mass filter for positive and negative ions. The mass scale is linear to the applied amplitude of the HF voltage. Mass resolution can be conveniently and electrically set using the ratio between the DC voltage *U* and the high-frequency voltage amplitude *V*. Due to their small dimensions and lightweight, quadrupole mass spectrometers are suitable both as pure residual gas analyzers and, in higher-quality design, as sensors for gas analysis.

6.2 QUADERA_SOFTWARE OPERATION

6.2.1 CONNECTION AND STARTING OF THE NOTEBOOK

Communication with the mass analyzer inside the device, its tuning, and visualization of measured data is provided by the Quadera control software installed in the notebook (**Figure 3**, point 2). The Mass Spec is connected to the notebook via a RED Ethernet cable (**Figure 3**, point 3) and a COMPUTER Ethernet connector on the back of the device (**Figure 40**, point 3).

The **PASSWORD** for your laptop operating system is **mims2021**. The active connection between Mass Spec and Quadera software is described in the **Chapter 6.2.2** .

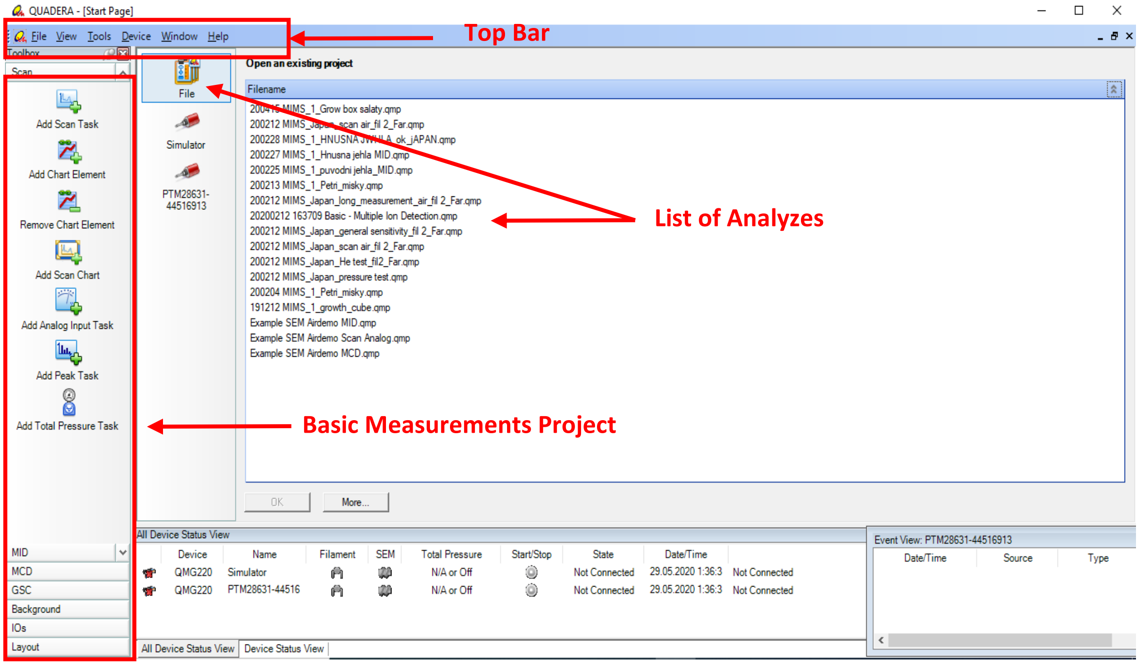
6.2.2 STARTING THE SOFTWARE

Figure 57*: Quadera icon*

Quadera software is Inficon's licensed software for controlling the Mass Spec Prisma Plus from Pfeiffer Vacuum, the most important component of the MS GAS-100 gas analyzer.

The basic set of the new device includes a laptop with Quadera software installed, as well as a CD with Quadera and a registration key (**Figure 3**, point 2 and 9). The software can be installed on any other PC.

The Quadera software is started by clicking on the icon on the computer desktop (**Figure 57**). The **Start Page** opens (**Figure 58**).



Open an existing project

Filename
200415 MIMS_1_Grow box salaty.qmp
200212 MIMS_Japan_scan air_fil 2_Far.qmp
200228 MIMS_1_HNUSNA JEHLA_ok_jAPAN.qmp
200227 MIMS_1_Hnusna jehla MID.qmp
200225 MIMS_1_puvodni jehla_MID.qmp
200213 MIMS_1_Petri_misky.qmp
200212 MIMS_Japan_long_measurement_air_fil 2_Far.qmp
20200212 163709 Basic - Multiple Ion Detection.qmp
200212 MIMS_Japan_general sensitivity_fil 2_Far.qmp
200212 MIMS_Japan_scan air_fil 2_Far.qmp
200212 MIMS_Japan_He test_fil2_Far.qmp
200212 MIMS_Japan_pressure test.qmp
200204 MIMS_1_Petri_misky.qmp
191212 MIMS_1_growth_cube.qmp
Example SEM Airdemo MID.qmp
Example SEM Airdemo Scan Analog.qmp
Example SEM Airdemo MCD.qmp

All Device Status View

Device	Name	Filament	SEM	Total Pressure	Start/Stop	State	Date/Time	
QMG220	Simulator			N/A or Off		Not Connected	29.05.2020 1:36:3	Not Connected
QMG220	PTM28631-44516			N/A or Off		Not Connected	29.05.2020 1:36:3	Not Connected

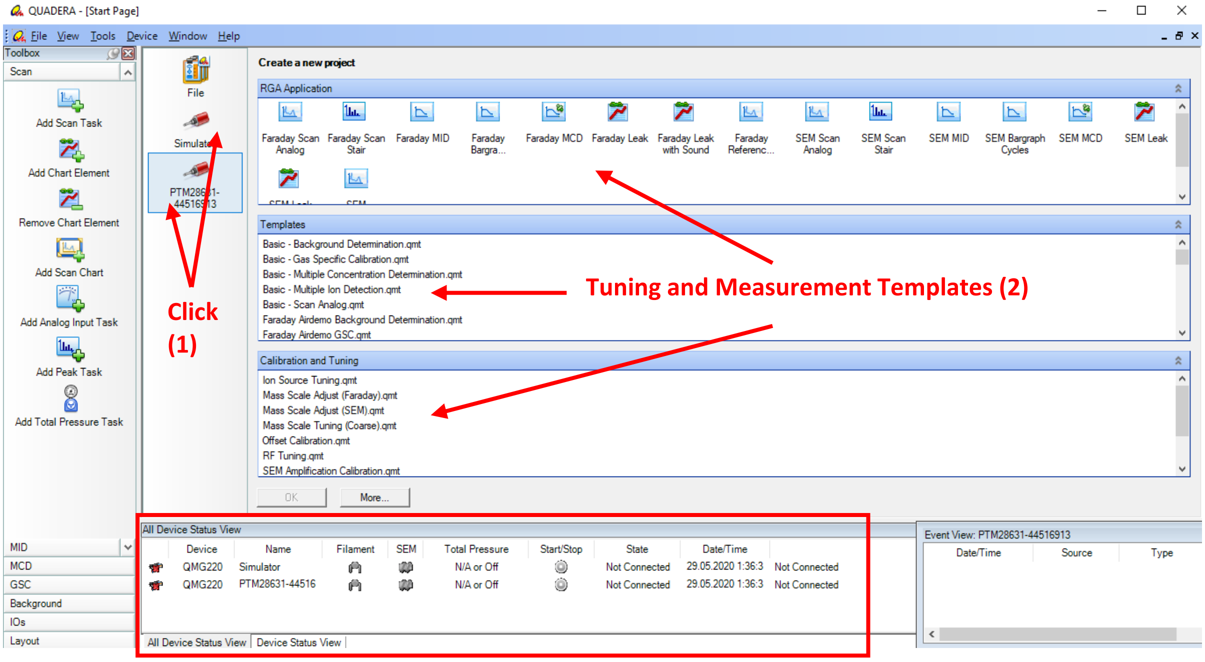
***Figure 58**: Start page of Quadera*

The top bar hides the main features of the Quadera setting. There are function for Quadera window displaying (**View** and **Window** tab) and some **Tools** – the most important is the **Spectra Library**. The basic measurement protocols are summarized on the left side of the Start Page. Next to the protocol bar is a **File** icon, a **Simulato**r icon, and a **Mass Spec** icon (**Figure 58**).

Clicking on the **File** icon opens a list of performed analyses (**Figure 58**). The **Simulator** is a tool of the Quadera software for training mass analyzer settings. The user can try almost all the features of Mass Spec without running it.

When the Quadera has been connected to a mass analyzer unit in the past, an icon with the registration number of this **Mass Spec** is available (**Figure 59**, point 1). Click on this icon and the list of performed analyses is replaced by predefined protocols and templates with different types of Mass Spec tuning and many measurement variations (**Figure 59**, point 2). The menu is the same as the simulator icon menu.

On the bottom the status of connected device is shown (**Figure 59**, red frame). All devices can be shown, which were in the past connected to the software (All Device Status View). Only the status of currently connected Mass Spec can be display also (Device Status View).



Templates
Basic - Background Determination.qmt
Basic - Gas Specific Calibration.qmt
Basic - Multiple Concentration Determination.qmt
Basic - Multiple Ion Detection.qmt
Basic - Scan Analog.qmt
Faraday Airdemo Background Determination.qmt
Faraday Airdemo GSC.qmt

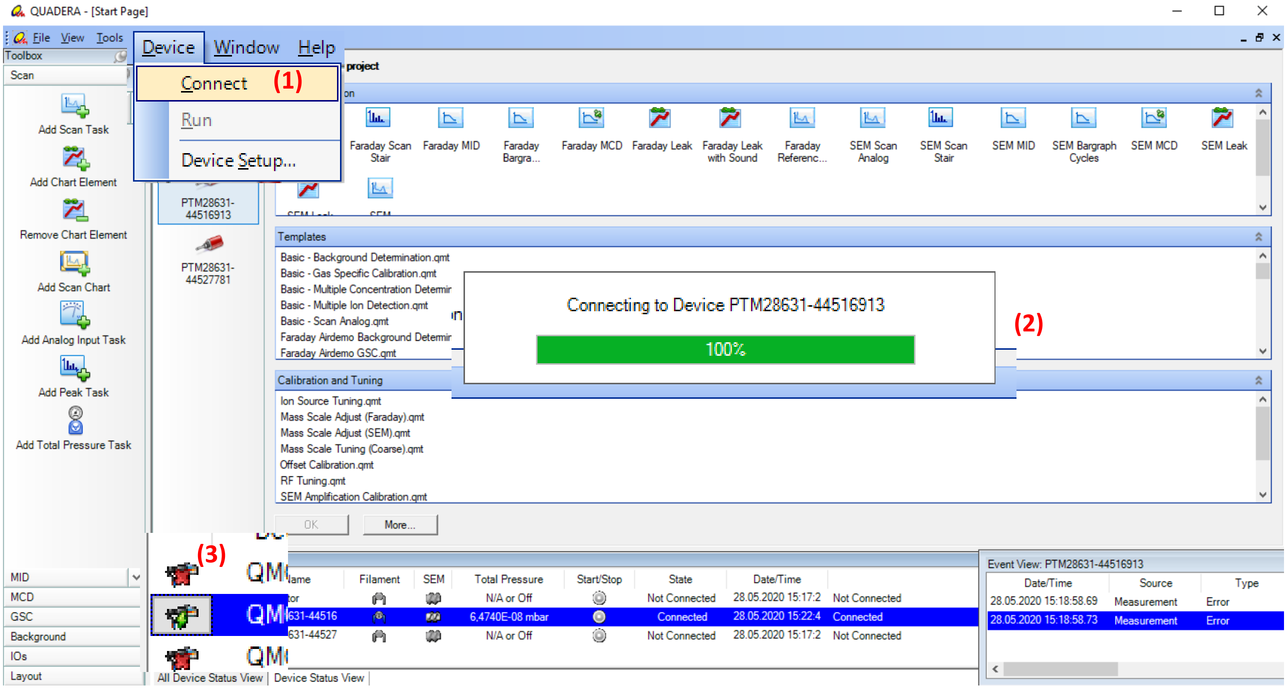
Calibration and Tuning
Ion Source Tuning.qmt
Mass Scale Adjust (Faraday).qmt
Mass Scale Adjust (SEM).qmt
Mass Scale Tuning (Coarse).qmt
Offset Calibration.qmt
RF Tuning.qmt
SEM Amplification Calibration.qmt

All Device Status View

Device	Name	Filament	SEM	Total Pressure	Start/Stop	State	Date/Time	
QMG220	Simulator			N/A or Off		Not Connected	29.05.2020 1:36:3	Not Connected
QMG220	PTM28631-44516			N/A or Off		Not Connected	29.05.2020 1:36:3	Not Connected

***Figure 59**: Tuning and measurement protocols after the click on Stimulator and/or Mass Spec icon*

For an active connection between Mass Spec and PC click on the tab **Device** in the top menu of Quadera software and after that click on the tab **Connect** (**Figure 60**, point 1). The same procedure is done by clicking on the **red icon** of the Mass Spec in the **All Device status View** frame (**Figure 60**, point 3). If the software reads the IP address of the Mass Spec without any problem the MS GAS-100 is quickly connected (**Figure 60**, point 2) and the Mass Spec icon became from red to green (**Figure 60**, point 3).



Connecting to Device PTM28631-44516913

100%

ame	Filament	SEM	Total Pressure	Start/Stop	State	Date/Time	
tor			N/A or Off	○	Not Connected	28.05.2020 15:17:2	Not Connected
631-44516			6,4740E-08 mbar	○	Connected	28.05.2020 15:22:4	Connected
631-44527			N/A or Off	○	Not Connected	28.05.2020 15:17:2	Not Connected

Event View: PTM28631-44516913

Date/Time	Source	Type
28.05.2020 15:18:58.69	Measurement	Error
28.05.2020 15:18:58.73	Measurement	Error

***Figure 60**: Direct connection of Quadera to the Mass Spec_step 1, 2 and 3*

If the device is not connected quickly use the tab **Device Setup** (**Figure 61**, point 1). In the open window click on **Search** (**Figure 61**, point 2) and followed window choose the Mass Spec server (**Figure 61**, point 3). Labeled server number verify by click on icon **Add** (**Figure 61**, point 4). After click on **OK** (**Figure 61**, point 5) the MS GAS-100 connects to the Quadera software and the device icon on the bottom become green (**Figure 61**,point 6). The communication device control software Quadera is restored, the device is prepared for measurement.

It is possible, that the Mass Spec server is not found immediately. The reason is that the initiation of the Mass Spec server takes a few minutes after device switch on, respectively device connection on PC by ethernet cable. When the connection of Mass Spec with PC is not established, repeat the described procedure steps (**Figure 61**).

6.2.3 PREPARATION OF FILAMENT AND SEM DETECTOR FOR MEASUREMENT

Before the start of gases analyses the filament and the detector have to be prepared for measurement.

Two filaments are contained in the opened ion source. The filament is made from yttriated irridium, the highly stable material. For the correct function of the filament it is necessary to switch the filament for at least 30 minutes before the start of any analysis (**Figure 62**).

⚠	**Before any manipulation with the ion source (filaments) the valves of MS GAS-100 must be in given states** (**Chapters 4.4.3.2** and **4.4.3.1** , **Figure 37**): **Inlet valve – opened** (after the evacuation of Inlet) for all type of calibration and analyses, except the Background Determination (**Chapter 6.2.5.7**) **Split Flow valve - opened/closed** – it depends on the concentration of the sample **Safety Valve - opened**

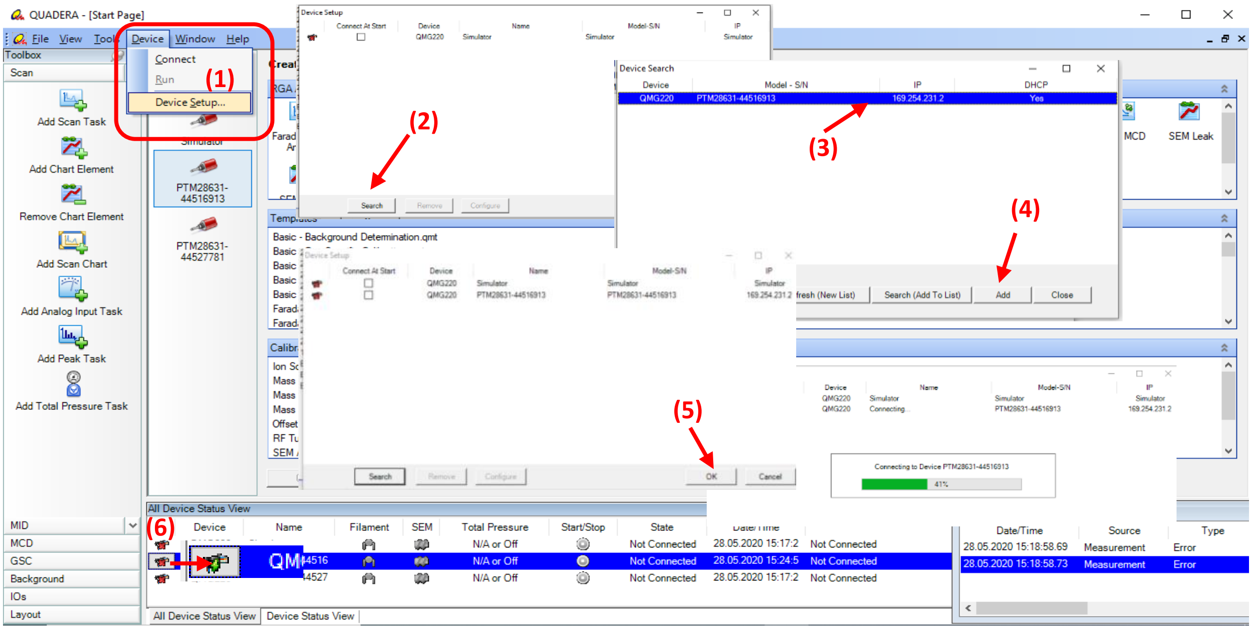
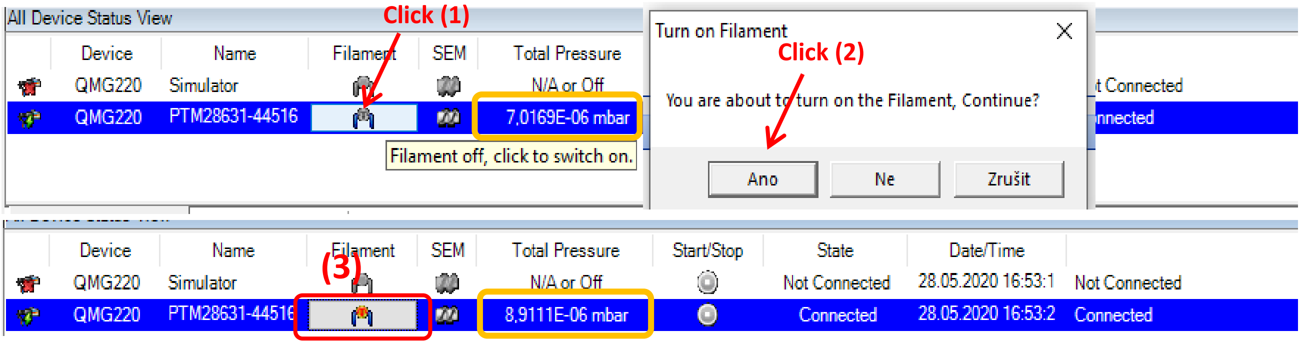


Figure 61: *Connection of device via Device Setup*



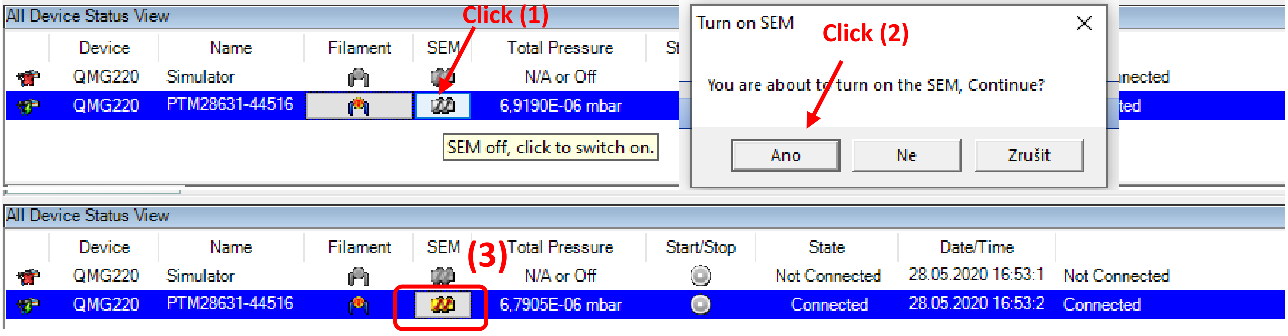
Filament off, click to switch on.

You are about to turn on the Filament, Continue?

Figure 62: *The filament switch ON*

Immediately after switching on the filament, the pressure inside the ion chamber increases for a short time (see **Figure 63**., pressure values in orange frames).

Similar steps are used also while turning on and working with the SEM detector. The Faraday cup detector is turned on and working all the time (**Figure 63**).



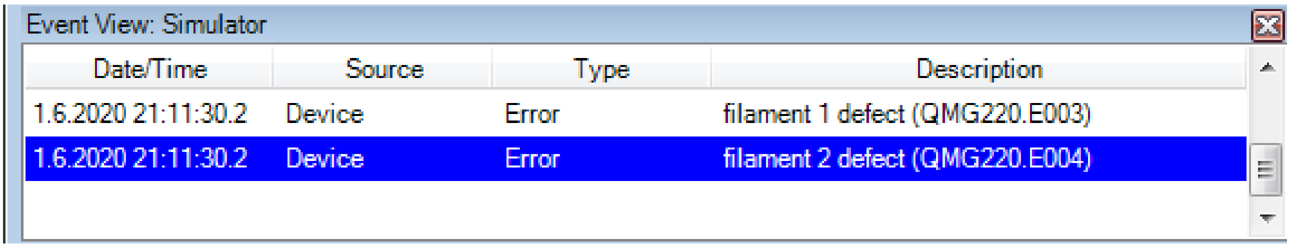
SEM off, click to switch on.

You are about to turn on the SEM, Continue?

Figure 63: *The SEM detector turns ON*

Both, the filament as well as the SEM detector, are switched OFF by clicking on appropriate icons. The icon color changes from red (filament) respectively yellow (SEM) to grey. It is important, that the safety valve can be closed only after switching off both the filament and the SEM detector.

⚠	Warning! If the device valves are opened/closed incorrectly during a measurement (i. e. the filament is switched ON), the filament might be damaged seriously (**Figure 64**).


Event View: Simulator

Date/Time	Source	Type	Description
1.6.2020 21:11:30.2	Device	Error	filament 1 defect (QMG220.E003)
1.6.2020 21:11:30.2	Device	Error	filament 2 defect (QMG220.E004)

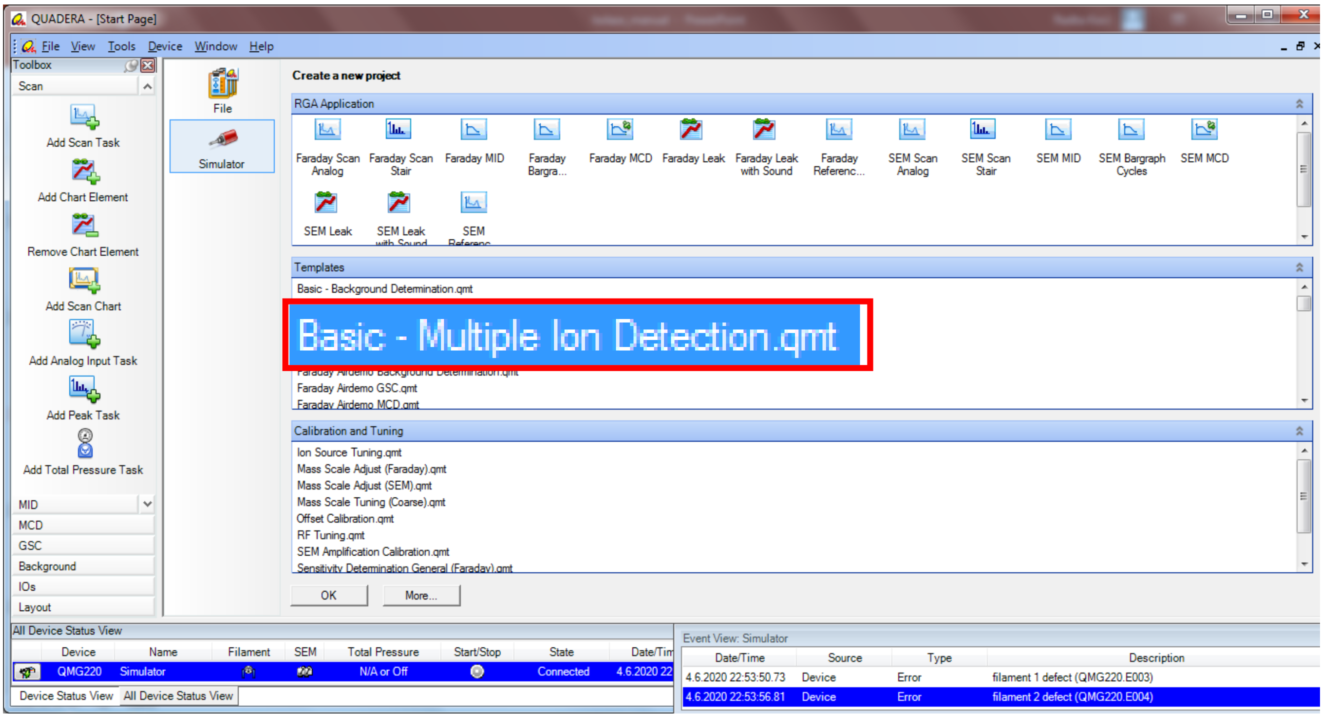
***Figure 64**: Detection of filament defect by Quadera*

6.2.4 GENERAL PROTOCOL SETTINGS AND CONTROL

Before using any tuning protocol and/or measurement project template read this part about parameter settings and graphics possibilities offered by Quadera software. All tuning, as well as measurement protocols, are controlled by the same or very similar systematic steps.

6.2.4.1 Parameters Setting and Protocol Control

Double-click any template (***.qmt file**) from Calibration and Tuning or Templates section (**Figure 65**). The working area window opens (**Figure 66**). The bar on the left side of the opened window contains five basic measurement projects: Scan, Multiple Ion Detection (MID), Multiple Concentration Detection (MCD), Gas Specific Concentration (GSC) and Background (**Figure 66**, red frame).



All Device Status View

	Device	Name	Filament	SEM	Total Pressure	Start/Stop	State	Date/Tim
	QMG220	Simulator			N/A or Off	○	Connected	4.6.2020 22

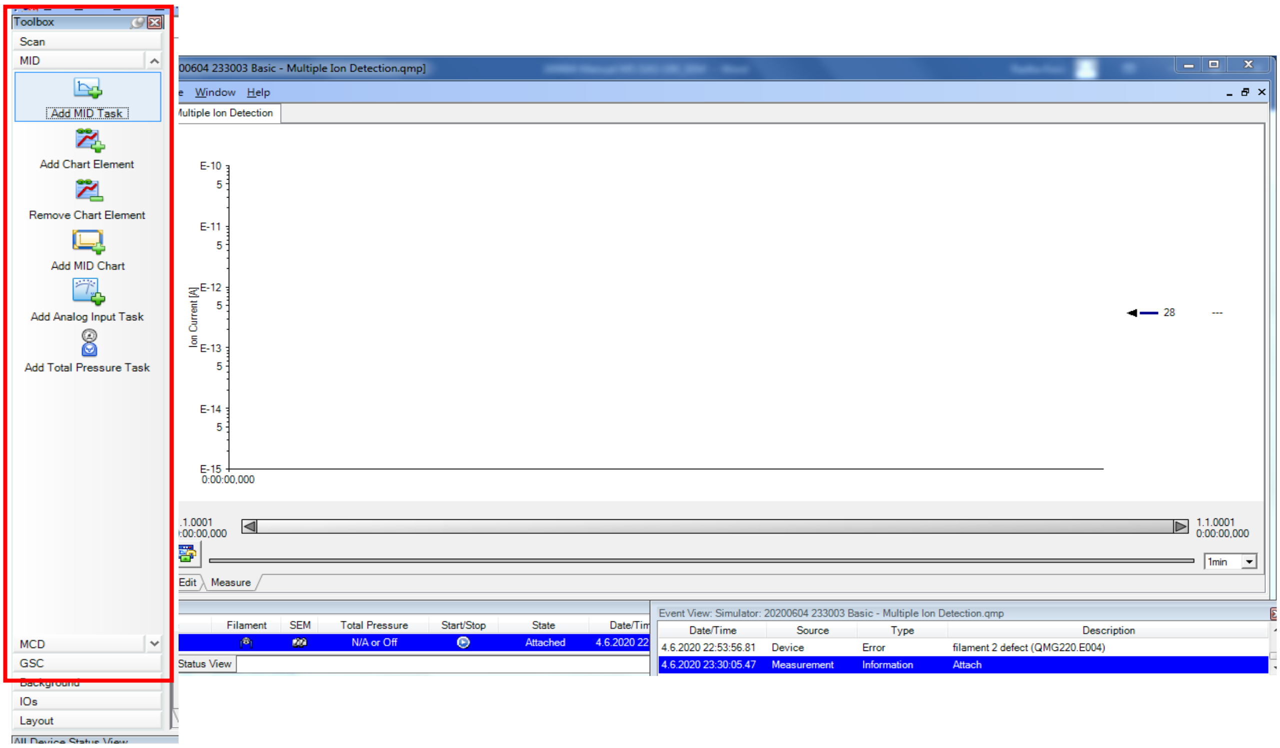
Event View: Simulator

Date/Time	Source	Type	Description
4.6.2020 22:53:50.73	Device	Error	filament 1 defect (QMG220.E003)
4.6.2020 22:53:56.81	Device	Error	filament 2 defect (QMG220.E004)

***Figure 65**:MID measurement project start*

Every measurement project includes a group of tasks (**Figure 67**). MID tasks and MCD tasks are related to recipe Editor. The Editor table is opened by click on the **Edit** tab (**Figure 68**, point 1). If the protocol allows, new tables for the setting off other masses and measurement parameters are added by clicking and dragging the right mouse button from the **MID task** tab usually (**Figure 68**, point 2). Parameters in the table can be changed easily by clicking on the table row. For measurement with MS GAS-100 set mainly the Task name, Mass, Dwell (between two next masses) and Detector type (**Figure 69**).

In the Editor table the task for determination of total pressure in ion chamber can be also inserted simultanously with MID task (**Figure 70**).



Filament	SEM	Total Pressure	Start/Stop	State	Date/Tim
		N/A or Off		Attached	4.6.2020 22

Event View: Simulator: 20200604 233003 Basic - Multiple Ion Detection.qmp

Date/Time	Source	Type	Description
4.6.2020 22:53:56.81	Device	Error	filament 2 defect (QMG220.E004)
4.6.2020 23:30:05.47	Measurement	Information	Attach

***Figure 66**: Working area of the MID project*

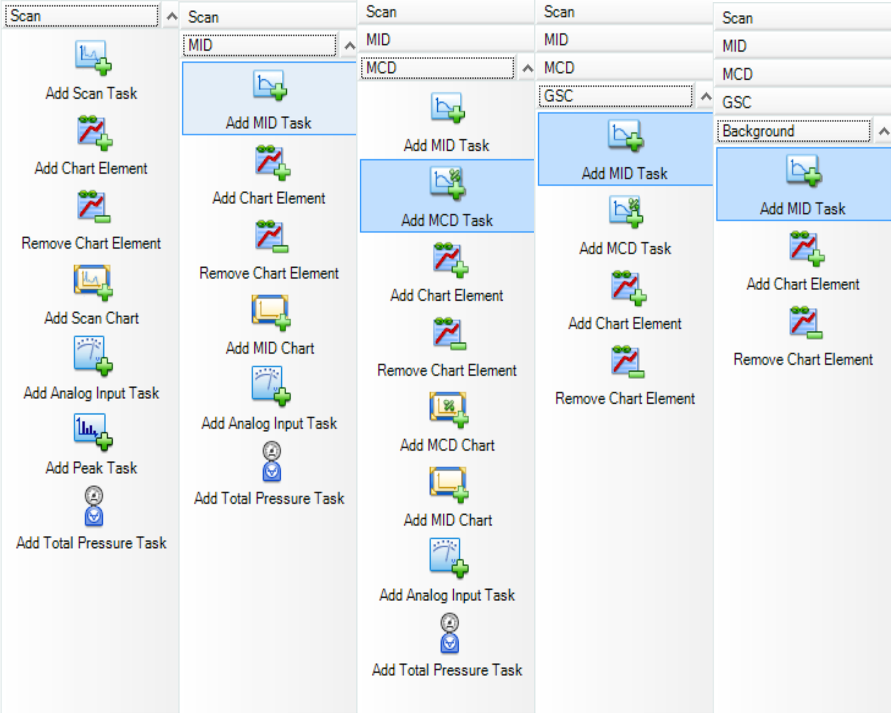
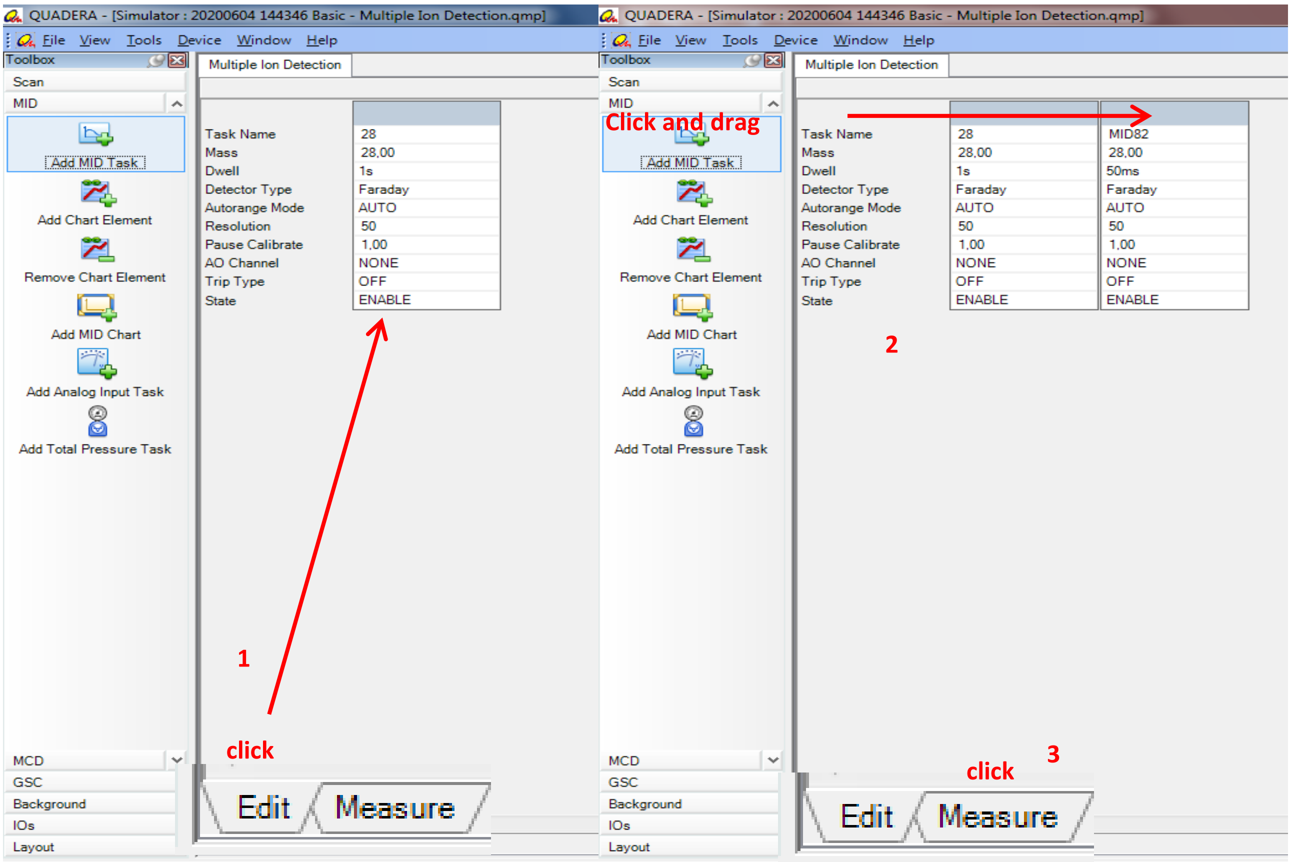
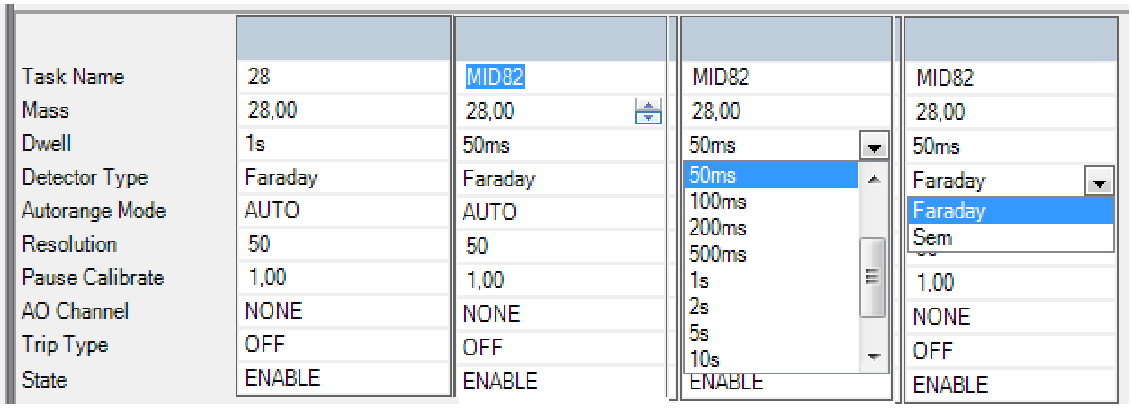


***Figure 67**: Tasks included in basic measurement projects_left bar*



Task Name	28	MID82
Mass	28,00	28,00
Dwell	1s	50ms
Detector Type	Faraday	Faraday
Autorange Mode	AUTO	AUTO
Resolution	50	50
Pause Calibrate	1,00	1,00
AO Channel	NONE	NONE
Trip Type	OFF	OFF
State	ENABLE	ENABLE

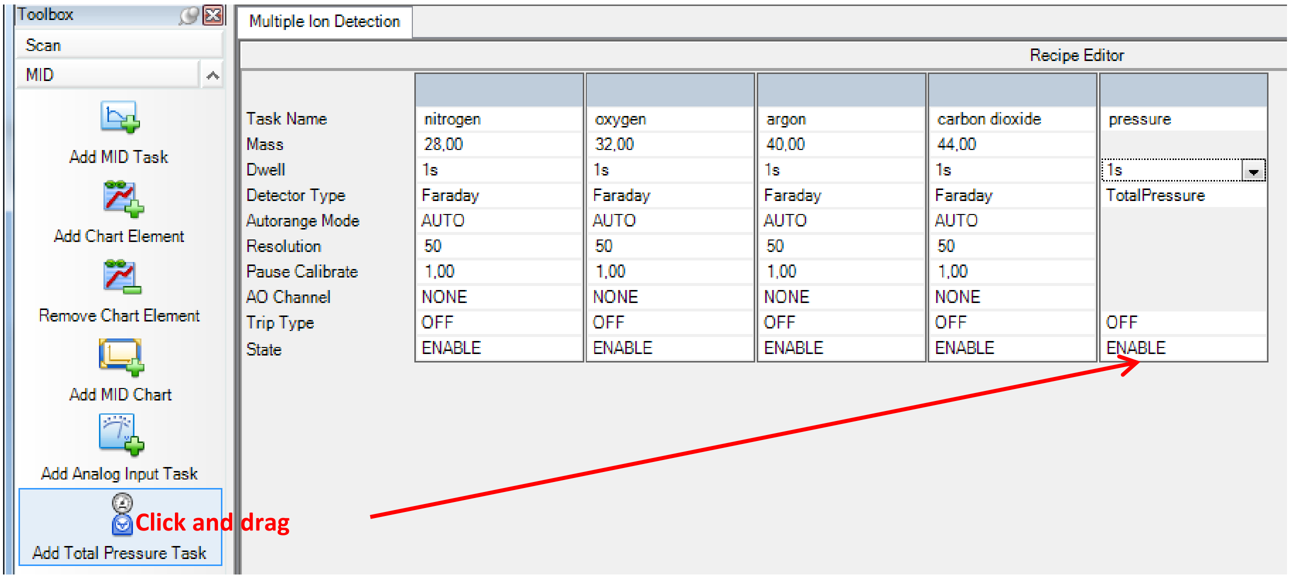
Figure 68: *Parameter editing: 1) editor table opened 2) new mass inserted 3) returing on the working area window*



Task Name	28	MID82	MID82	MID82
Mass	28,00	28,00	28,00	28,00
Dwell	1s	50ms	50ms	50ms
Detector Type	Faraday	Faraday		Faraday
Autorange Mode	AUTO	AUTO		
Resolution	50	50		
Pause Calibrate	1,00	1,00		1,00
AO Channel	NONE	NONE		NONE
Trip Type	OFF	OFF		OFF
State	ENABLE	ENABLE	ENABLE	ENABLE

Figure 69: *Setting of different parameters in Editor table*

	The compounds must be set in the editor table **from the lowest to the highest** mass values.
	The SEM detector must not be used in combination with a Faraday cup during one measuring cycle: - measure the desired compounds (masses) using a Farraday detector - select masses with an ion current signal **lower than 10^{-10} A** and use them for SEM measurements..



Task Name	nitrogen	oxygen	argon	carbon dioxide	pressure
Mass	28,00	32,00	40,00	44,00	
Dwell	1s	1s	1s	1s	1s
Detector Type	Faraday	Faraday	Faraday	Faraday	TotalPressure
Autorange Mode	AUTO	AUTO	AUTO	AUTO	
Resolution	50	50	50	50	
Pause Calibrate	1,00	1,00	1,00	1,00	
AO Channel	NONE	NONE	NONE	NONE	
Trip Type	OFF	OFF	OFF	OFF	OFF
State	ENABLE	ENABLE	ENABLE	ENABLE	ENABLE

***Figure 70**: MID complete setting*

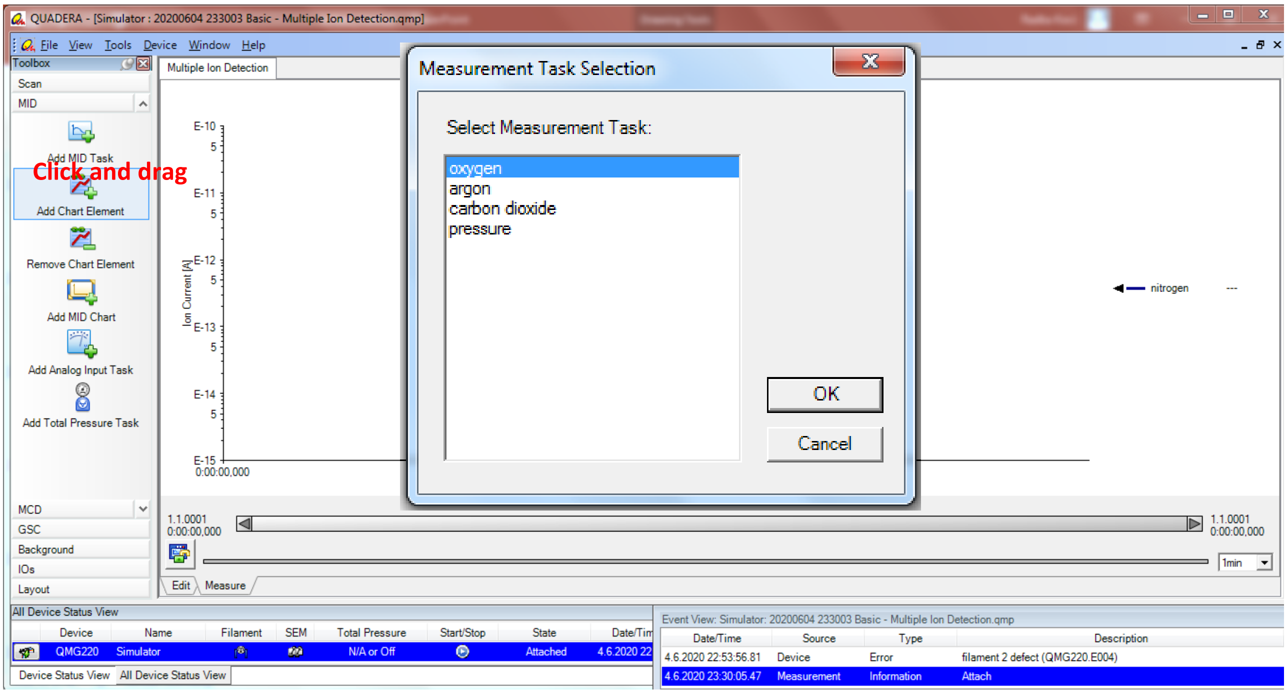
Click on the tab next to the **Edit** (**Tune, Calibrate, Measure** - the name of the tab depends on the selected measurement or tuning protocol;**Figure 68**, point 3). An empty graph will appear with only one parameter in the legend set as the default.

Click and drag the **Add Chart Element** icon (left taskbar) to insert each selected compound (mass) into the chart (**Figure 71**). To remove an element from the chart use the **Remove Chart Element** icon in the same way.

Selected gases can be displayed on the main/secondary Y-axis. The measured compounds are usually displayed on the Y-1 axis, while the total pressure expressed in mBa uses the Y-2 axis. The measured compounds are characterized by different colors of signals in the legend. Right-click on the **colored line** of the measured gas in the legend (**Figure 72**, point 1). The table with the option to select the axis and colors opens (**Figure 72**, points 2 and 3).

Another setting concerns the **X-axis**, which is expressed as a time-axis and/or mass-axis for almost of measurements and calibrations. For the **time-axis** (MID and MCD projects), there are two time unit options. Right-click on the X axis. Time can be expressed as absolute time (real time) and/or relative time (how many minutes and hours the experiment runs - **Figure 72**,point 4).

After completing the parameter settings and configuration of the graph, start the measurement using the **Start/Stop** tab (**Figure 72**). To stop the analysis, click on the same Start / Stop tab again.



Measurement Task Selection

Select Measurement Task:

oxygen
argon
carbon dioxide
pressure

All Device Status View

Device	Name	Filament	SEM	Total Pressure	Start/Stop	State	Date/Tim
QMG220	Simulator			N/A or Off		Attached	4.6.2020 22

Device Status View | All Device Status View

Event View: Simulator: 20200604 233003 Basic - Multiple Ion Detection.qmp

Date/Time	Source	Type	Description
4.6.2020 22:53:56.81	Device	Error	filament 2 defect (QMG220.E004)
4.6.2020 23:30:05.47	Measurement	Information	Attach

***Figure 71**: Configuration of analyzed masses in chart*

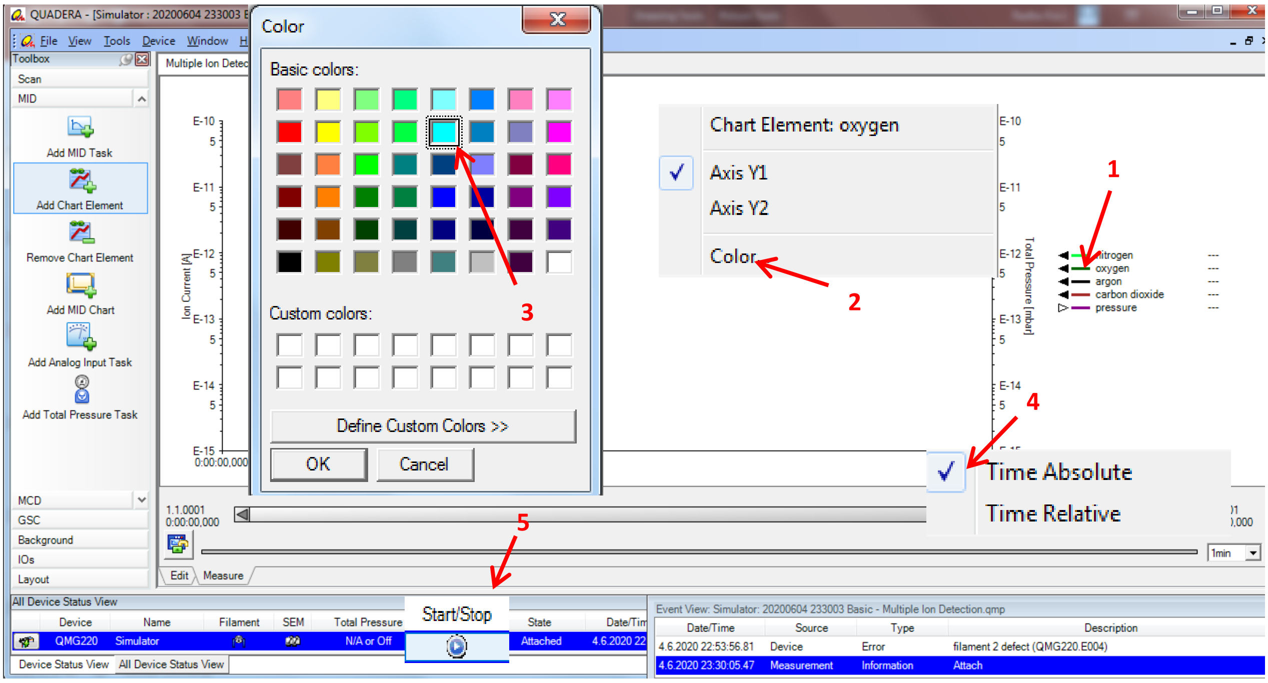


Figure 72: Chart measured elements configuration

During the measurement, the graph shows the measured signals in the default logarithmic format on the Y-axis. Right-click the cursor on the Y-axis. A table with a selection of logarithmic or linear scales appears (**Figure 73**, point 1). For X-axis, you can select which time period for the time-axis is to be monitored in the graph (**Figure 73**, point 2).

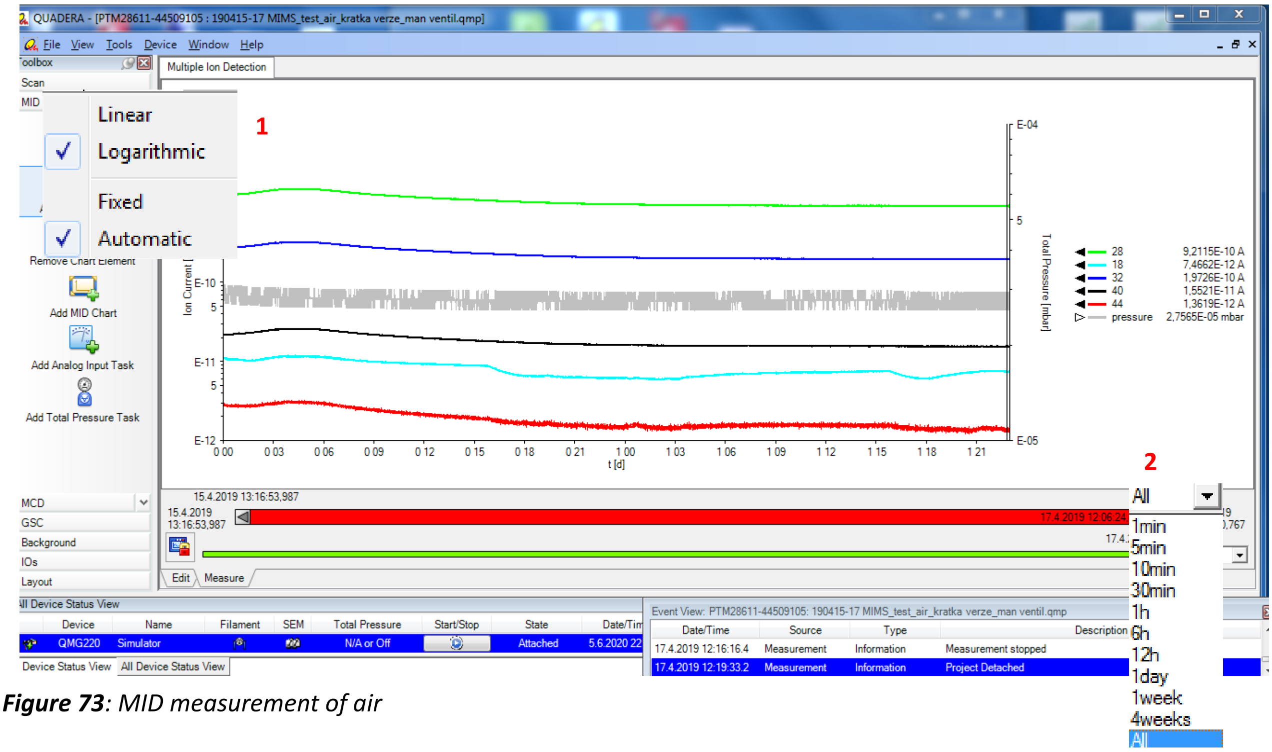


Figure 73: MID measurement of air

After right-clicking on the working area, a table with many functions for editing the chart is shown (**Figure 74**). There are included the function for chart zoom, the displaying of chart legend, manual background subtraction and/or conversion of row data expressed as ion current to partial pressure (**Figure 74**, red frame). The functions can be used in most of projects except Ion Source Tuning protocol (**Chapter 6.2.5.1**), Mass Scale Calibration (Coarse) (**Chapter 6.2.5.3**), and special projects as RF generator tuning (**Chapter 6.2.5.4**). Measured data can be displayed in the chart as a trend analysis, bargraph or the table with numeric values (**Figure 75**).

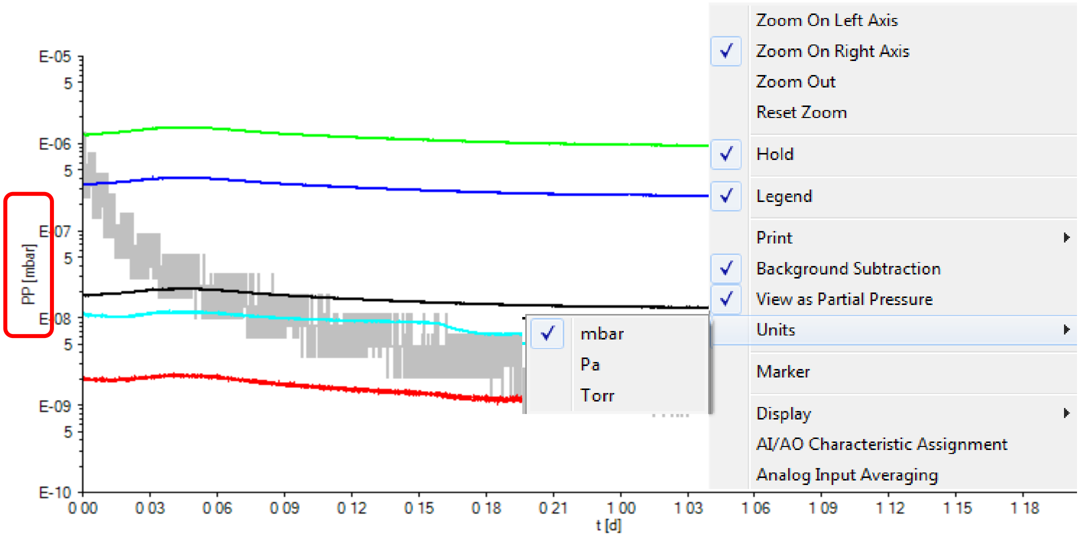
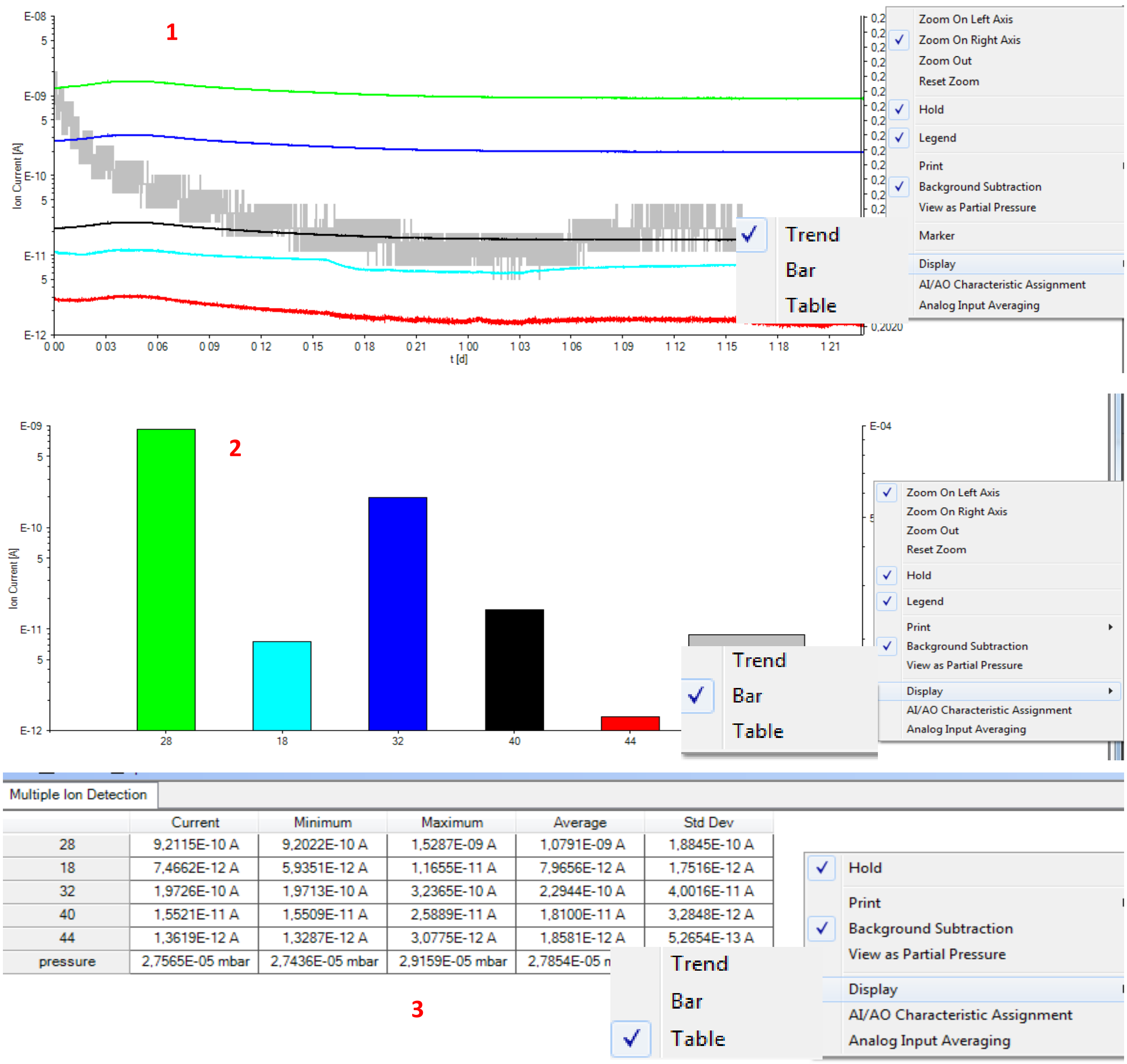


***Figure 74**: Chart functions table*



	Current	Minimum	Maximum	Average	Std Dev
28	9,2115E-10 A	9,2022E-10 A	1,5287E-09 A	1,0791E-09 A	1,8845E-10 A
18	7,4662E-12 A	5,9351E-12 A	1,1655E-11 A	7,9656E-12 A	1,7516E-12 A
32	1,9726E-10 A	1,9713E-10 A	3,2365E-10 A	2,2944E-10 A	4,0016E-11 A
40	1,5521E-11 A	1,5509E-11 A	2,5889E-11 A	1,8100E-11 A	3,2848E-12 A
44	1,3619E-12 A	1,3287E-12 A	3,0775E-12 A	1,8581E-12 A	5,2654E-13 A
pressure	2,7565E-05 mbar	2,7436E-05 mbar	2,9159E-05 mbar	2,7854E-05 n	

***Figure 75**: Different forms of the same chart: 1) Trend analysis, 2) Bargraph, 3) Table with measured values*

6.2.4.2 *Exporting Data*

Final data can be exported only from the measurement protocol. Open the **File** in the top bar and click on **Export** row. Click on **ASCII** tab (**Figure 76**)**.** ASCII format will export the displayed data as the text-only. You may open and further process these data in any spreadsheet program (e.g. Excel). The **Export Options** window used for selecting measuring data opens up. Select the required measurement tasks in the **Measure Tasks** area. Select the required data in the **Data Range** area. **All** exports all data of the measurement project, Selection exports only the displayed data in the selected section (scan chart, MID chart etc.), Cycles exports the selected cycles of the measurement project. Click OK (**Figure 77**, point 1). The **ASCII-Export data** as window used for exporting measuring data opens up. Select a suitable name and path and then click **Save** (**Figure 77**, point 2). The measurement project will be exported.

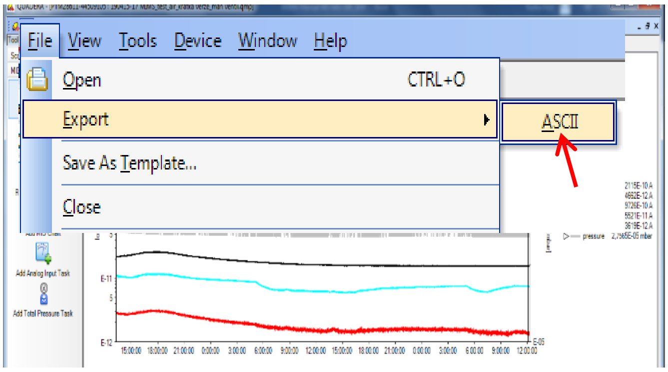
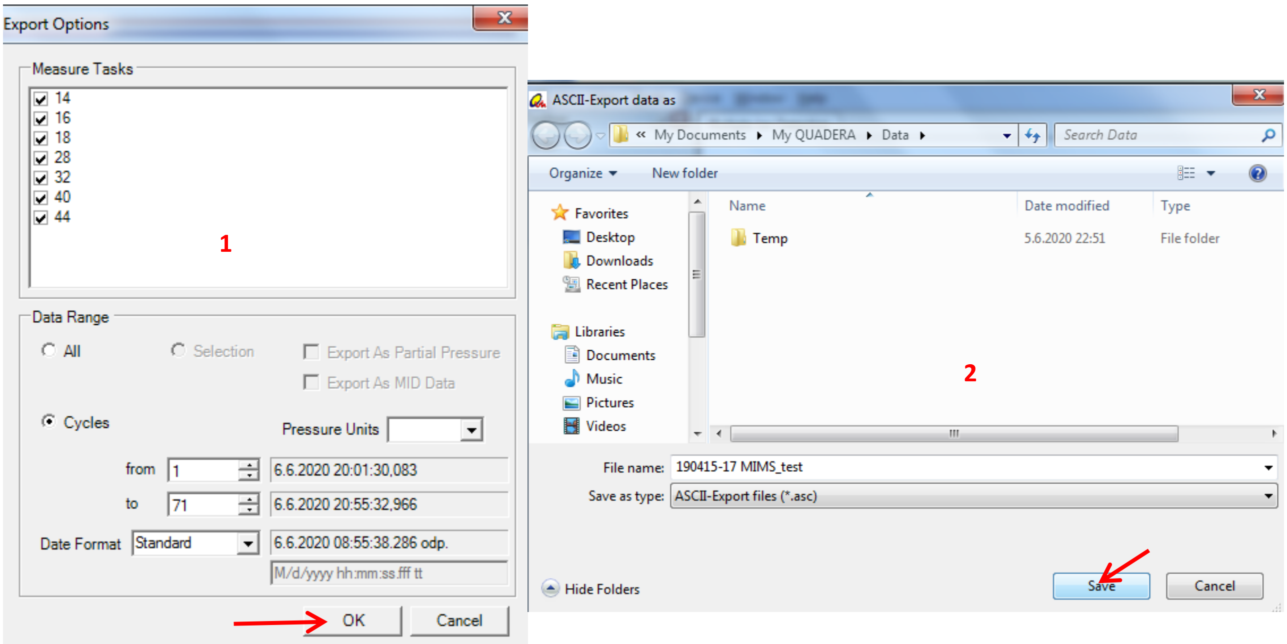


Figure 76*: Data export start*

	If you have not selected a section of a measurement project the export submenu item is disabled.



☑ 14
☑ 16
☑ 18
☑ 28
☑ 32
☑ 40
☑ 44

○ All ○ Selection ☐ Export As Partial Pressure ☐ Export As MID Data

● Cycles Pressure Units

from 1 6.6.2020 20:01:30,083
to 71 6.6.2020 20:55:32,966
Date Format Standard 6.6.2020 08:55:38.286 odp.
M/d/yyyy hh:mm:ss.fff tt

Temp 5.6.2020 22:51 File folder

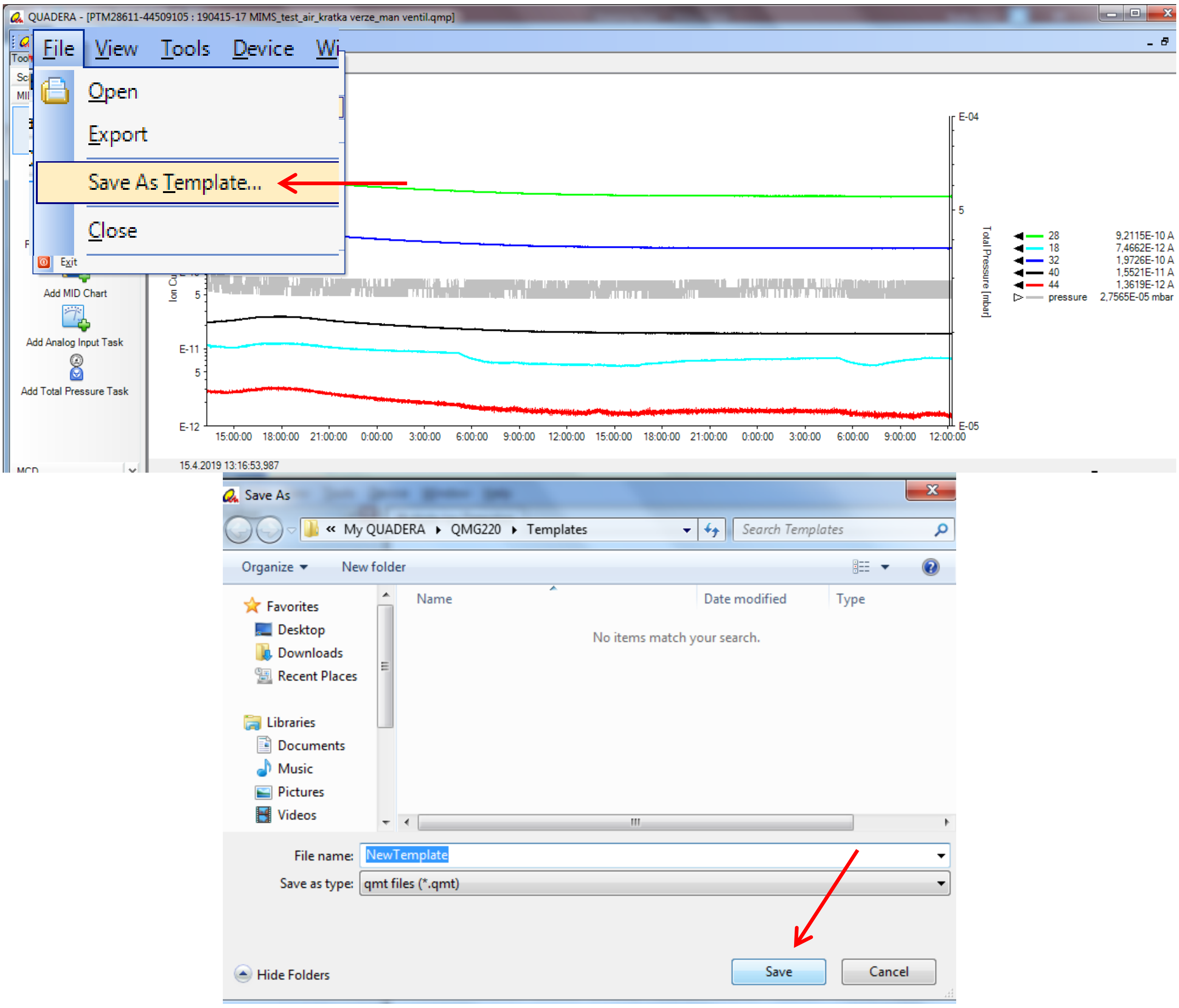
File name: 190415-17 MIMS_test
Save as type: ASCII-Export files (*.asc)

Figure 77*: Data Export – 1) data configuration 2) data storage*

Delimiters, the date and time format, and decimal separators (period and comma) are country specific. These settings can be adjusted in the operating system: **Windows Start menu > Control Panel > Date, Time, Language, and Regional Options**. The settings for the operating system will also be used by Quadera.

6.2.4.3 *Safe a Template of Measurement Project*

The template retains the settings of the measurement method predefined by the user. In the top bar, click on **File**. Select **Save As Template**. Select a suitable name, the path will be automatically offered in the **Template** section. Click **Save** and your method with all predefined parameters will be saved in the Template section on the Quadera main screen (**Figure 78**).



File name: NewTemplate
Save as type: qmt files (*.qmt)

Figure 78*: Creation of the measurement project template*

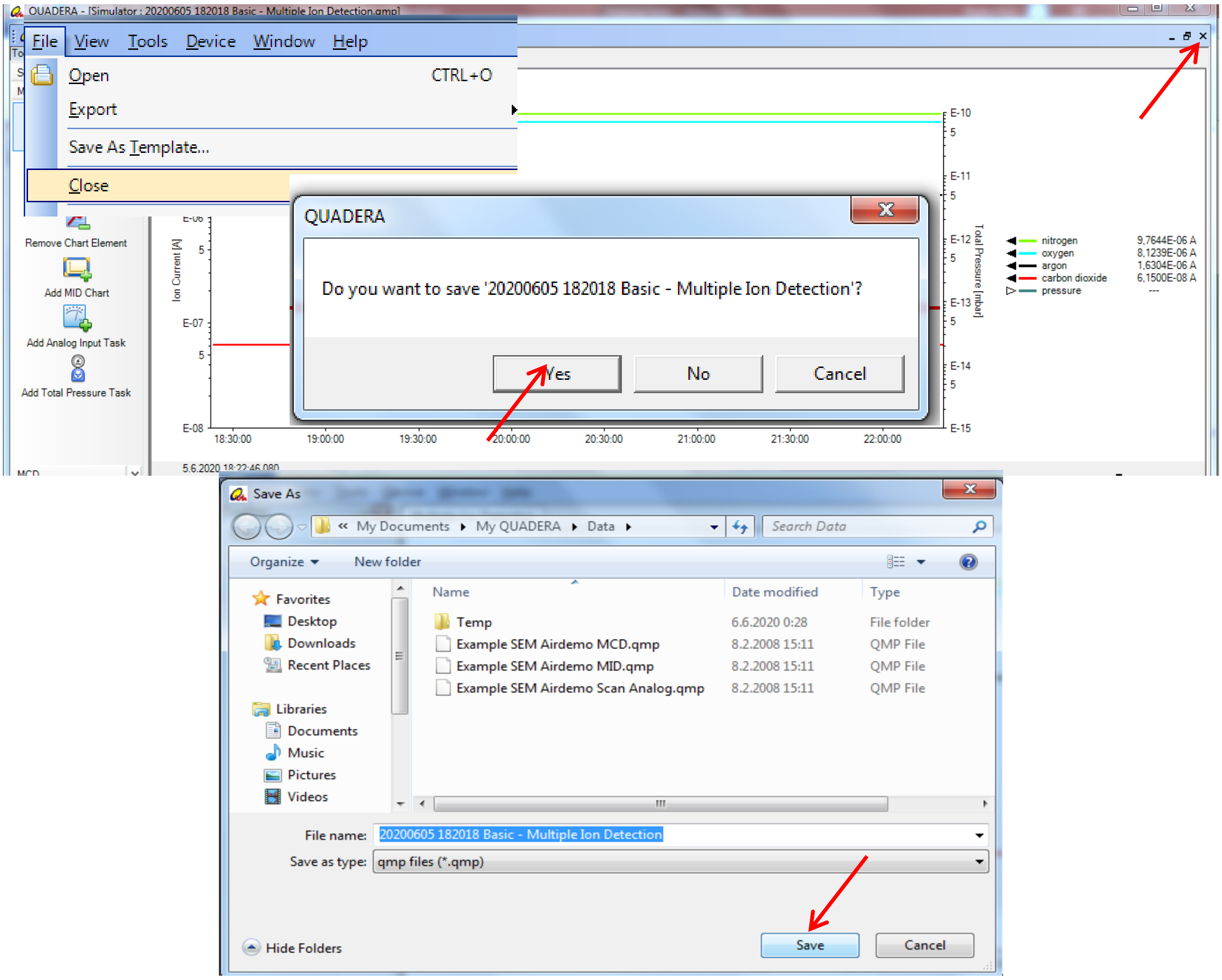
The file extension for Template of measurement projects is ***.qmt**.

6.2.4.4 Close and Save a Measurement Project

In the **File** menu, click **Close** or click on the close symbol **X** in upper right corner. You are requested if you want to save the measurement project. Quadera suggests an automatically generated file name that is based on date/time of the measurement and the used template. Click on **Yes** and the **Save A**s window used for saving measuring data opens up. Select a suitable name and path and then click **Save** (**Figure 79**). The measurement project will be saved and closed.

The file extension for measurement projects is ***.qmp**.

ℹ	**No** (recommended option): the measurement project will be closed without saving the data (**Figure 79**).
ℹ	**Cancel**: the action is cancelled. You will return to the measurement project without saving the data (**Figure 79**).



Do you want to save '20200605 182018 Basic - Multiple Ion Detection'?

Name	Date modified	Type
Temp	6.6.2020 0:28	File folder
Example SEM Airdemo MCD.qmp	8.2.2008 15:11	QMP File
Example SEM Airdemo MID.qmp	8.2.2008 15:11	QMP File
Example SEM Airdemo Scan Analog.qmp	8.2.2008 15:11	QMP File

File name: 20200605 182018 Basic - Multiple Ion Detection
Save as type: qmp files (*.qmp)

***Figure 79**: Save the measurement project*

6.2.5 CALIBRATIONS

Calibration or tuning of the instrument depends on the measurement frequency. It is usually performed once a month or in special cases, especially after a long shutdown, replacement of a damaged measurement filament, or repair of any part of the Mass Spec. The calibration measurements result in the optimal function of the ion source to detect most compounds within the analyzer's range, including ions (fragments) near the limit of detection.

In Quadera, devices are calibrated by using calibration projects, which are based on normal measurement projects. Calibration projects are called via the start page from the Calibration and Tuning section.

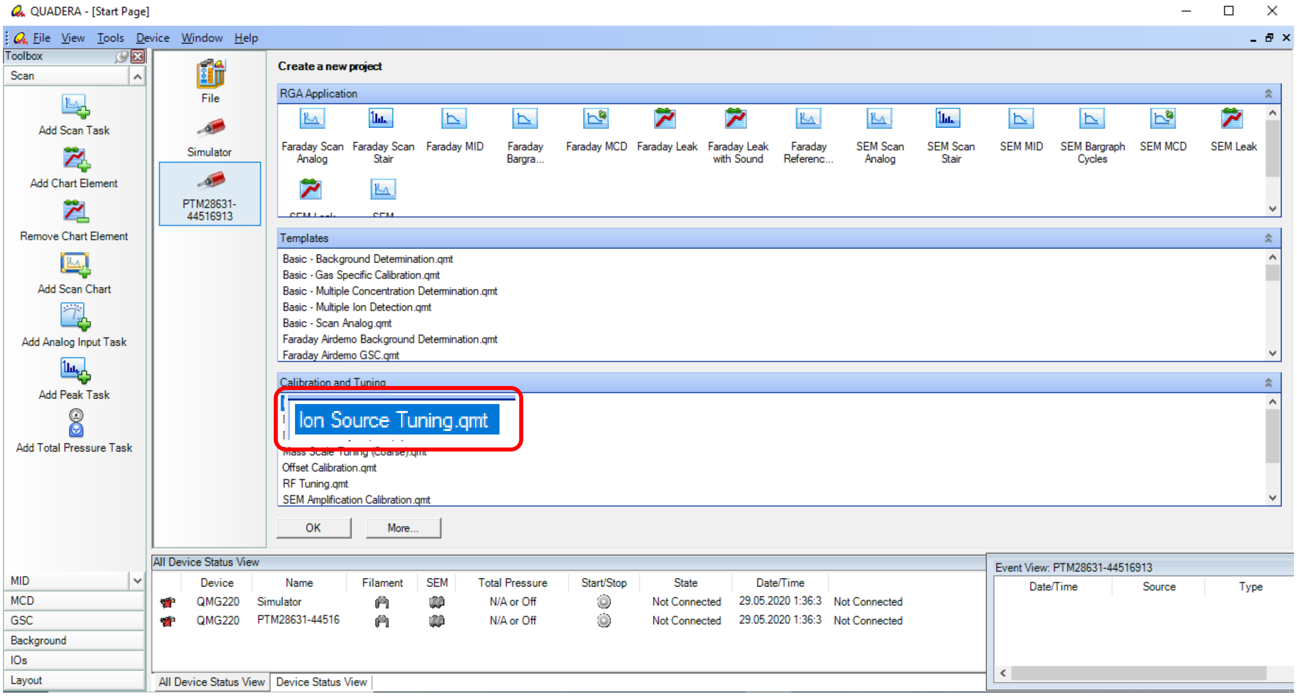
Quadera provides basic templates for each calibration project, such as gas specific calibration and background determination. These templates are intended to create user-specific calibration project templates, for ab initio calibration projects.

Saving the measurement data of a calibration procedure is usually not required. However, saving the calibration project as a template may be useful for future applications (**Chapter 6.2.4.3)**.

6.2.5.1 Ion Source Tuning

This calibration project allows optimizing the performance of the ion source. It is recommended to do ion source tuning after a long shutdown, replacement of a damaged filament or after repairing any part of the Mass Spec.

In the Calibration and Tuning section of the Quadera Start page click on the **Ion Source Tuning** (**Figure 80**). The protocol for Mass Spec tuning opens.



Device	Name	Filament	SEM	Total Pressure	Start/Stop	State	Date/Time	
QMG220	Simulator			N/A or Off		Not Connected	29.05.2020 1:36:3	Not Connected
QMG220	PTM28631-44516			N/A or Off		Not Connected	29.05.2020 1:36:3	Not Connected

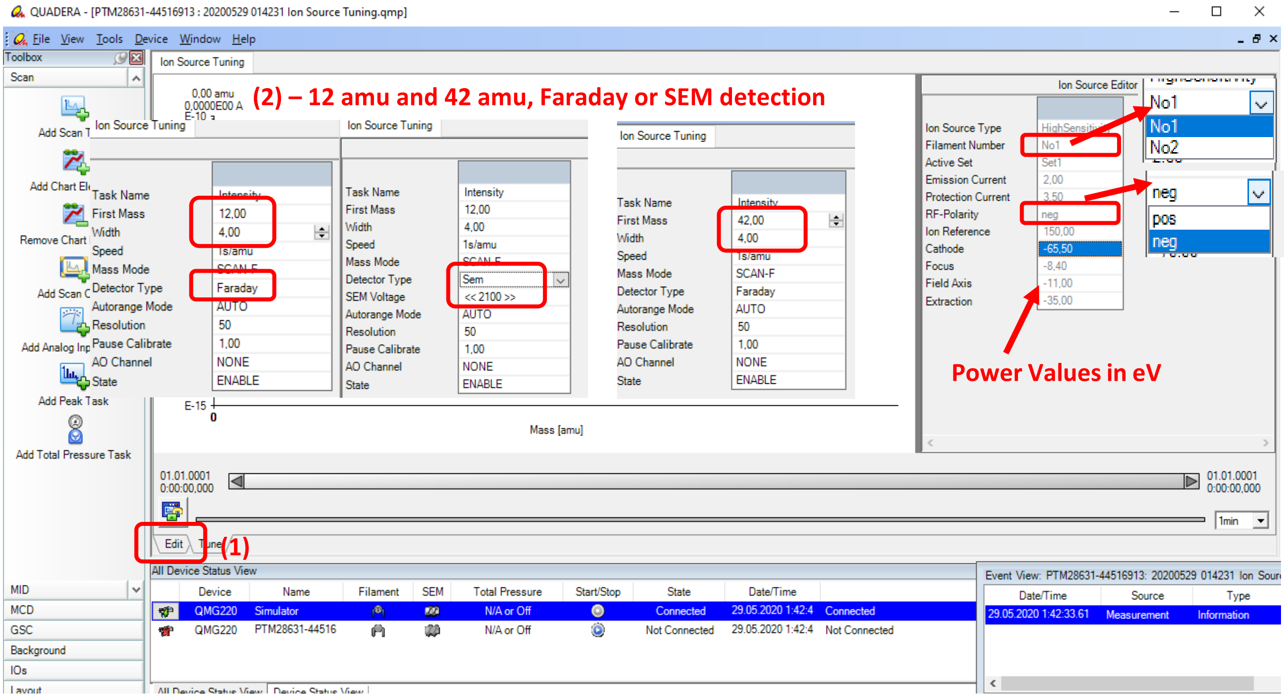
***Figure 80**: Start of Ion Source Tuning protocol*

Consider one or two peaks of a scan-analog measurement to tune the ion source (ion source optimization). Use the **Editor** (**Figure 81**, points 1 and 2) to define scanned masses, frequency of scanning, and the type of the detector (Faraday, SEM). Typically, two masses included in the desired measurement range are scanned. For air as calibration gas, the scan of masses **12 amu** (nitrogen atom) and **42 amu** (carbon dioxide) with set **width 4** are usually used.

Change parameters in **Ion Source Editor** to get an optimal statistical distribution of the peak (Gauss curve) with low noise and maximum signal amount (**Figure 81**, the table on the right side, see Gauss curves in **Figure 82**). Select one of the **filament** (the open ion source contains two independent filaments) and **polarity** of the rod system. The measurement under negative RF polarity is usually more stable. **Protection Current** has to be set at 3.5 A. Optimize the ion source voltages: **Cathode** for best sensitivity, **Field Axis** for useful resolution and peak shape, **Extract** and **Focus** alternately for maximum peak height – default settings is noted below.

Default settings:

Emission Current:	2 mA
Protection Current:	3.5 A
RF-Polarity:	pos
Ion Reference:	150 V
Cathode:	-75 V
Focus:	-5 V
Field Axis:	-7 V
Extract:	-50 V



Task Name	Intensity
First Mass	12,00
Width	4,00
Speed	1s/amu
Mass Mode	SCAN-F
Detector Type	Faraday
Autorange Mode	AUTO
Resolution	50
Pause Calibrate	1,00
AO Channel	NONE
State	ENABLE

Task Name	Intensity
First Mass	12,00
Width	4,00
Speed	1s/amu
Mass Mode	SCAN-F
Detector Type	Sem
SEM Voltage	<< 2100 >>
Autorange Mode	AUTO
Resolution	50
Pause Calibrate	1,00
AO Channel	NONE
State	ENABLE

Task Name	Intensity
First Mass	42,00
Width	4,00
Speed	1s/amu
Mass Mode	SCAN-F
Detector Type	Faraday
Autorange Mode	AUTO
Resolution	50
Pause Calibrate	1,00
AO Channel	NONE
State	ENABLE

Ion Source Editor

Ion Source Type	HighSensitivity
Filament Number	No1
Active Set	Set1
Emission Current	2,00
Protection Current	3,50
RF-Polarity	neg
Ion Reference	150,00
Cathode	-65,50
Focus	-8,40
Field Axis	-11,00
Extraction	-35,00

Device	Name	Filament	SEM	Total Pressure	Start/Stop	State	Date/Time	
QMG220	Simulator			N/A or Off		Connected	29.05.2020 1:42:4	Connected
QMG220	PTM28631-44516			N/A or Off		Not Connected	29.05.2020 1:42:4	Not Connected

Event View: PTM28631-44516913: 20200529 014231 Ion Source

Date/Time	Source	Type
29.05.2020 1:42:33.61	Measurement	Information

***Figure 81**: Basic tuning protocol*

The scans will be recorded when you turn back by the **Tune** tab and **start** the calibration project (**Figure 82**, points 1 and 2). The currently set ion source parameters are used. They are displayed in the working area. Tune the ion source while the measurement is running; optimize the peak shape and intensity by changing the ion source parameters. Changed parameters are forwarded from the calibration project to the device and accepted instantly. Stop the measurement by click on the Start/Stop tab (**Figure 82**, point 2).

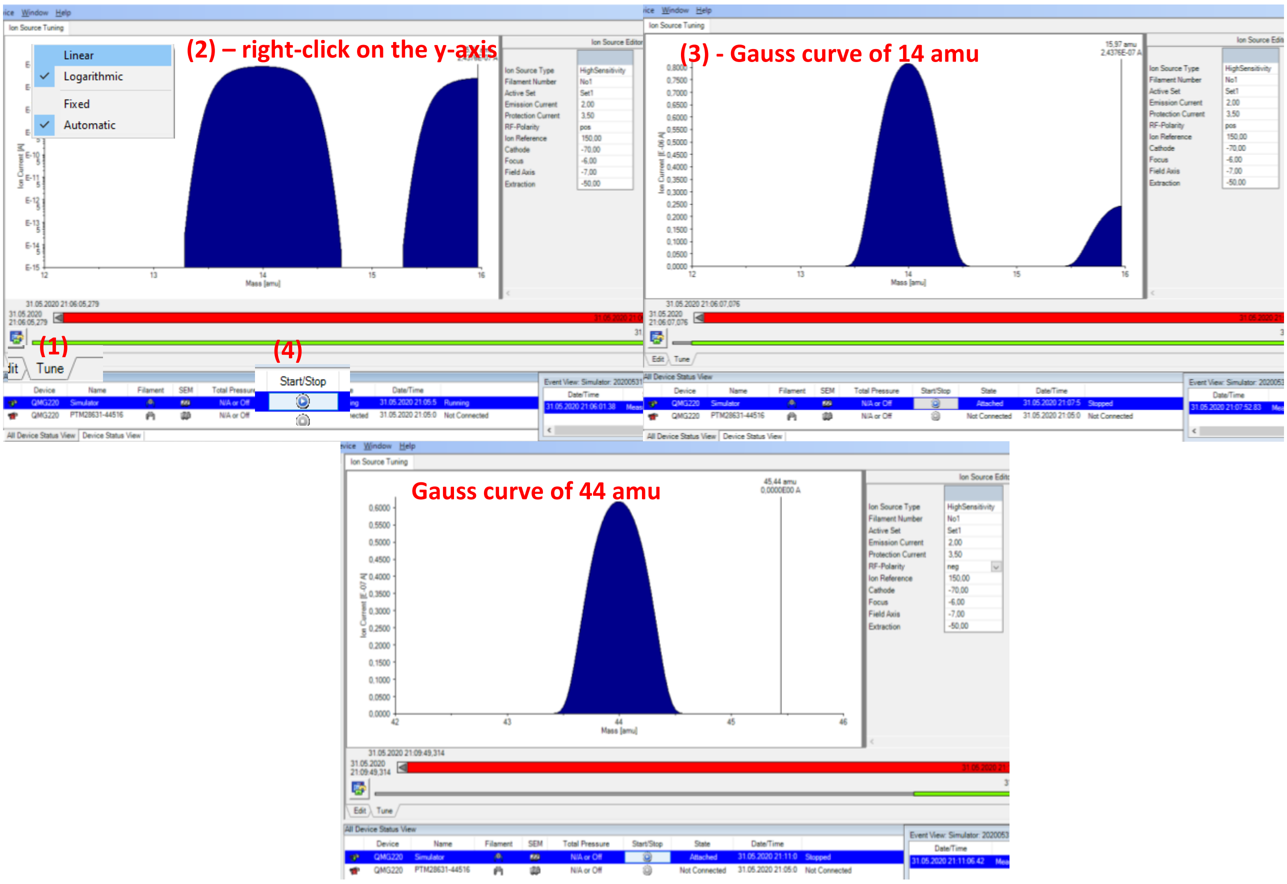


***Figure 82**: Optimal tuning for masses 14 amu and 44 amu_(1) returning to working area, (2) logarithmic scale_X-axis, (3) linear scale_X-axis (4) Start/Stop of the tuning*

6.2.5.2 *Offset Calibration*

The offset is caused by a drift of the measuring amplifier and can be compensated by a correction measurement. The offset determination is usually necessary after ion source tuning (**Chapter 6.2.5.1**).

The measurement of the offset is performed by using the **Offset Calibration** template (**Figure 83**, point 1) in the Calibration and Tuning section. The offset of the electronic circuit of the measuring amplifier is determined for each measuring range at a user-defined mass number (**Figure 83**, point 2). After the measurement start (**Figure 83**, point 3) the values of ion current intensity are automatically stored in the device and will remain after the power has been cut.

The measured intensity will be applied to future measurement projects by subtracting it from the results of the corresponding measurement tasks. You cannot switch on and off the offset values for saved data. If you wish to erase the saved values, there is a delete button in the Offset Calibration template in the Calibration and Tuning section (**Figure 83**, point 4).

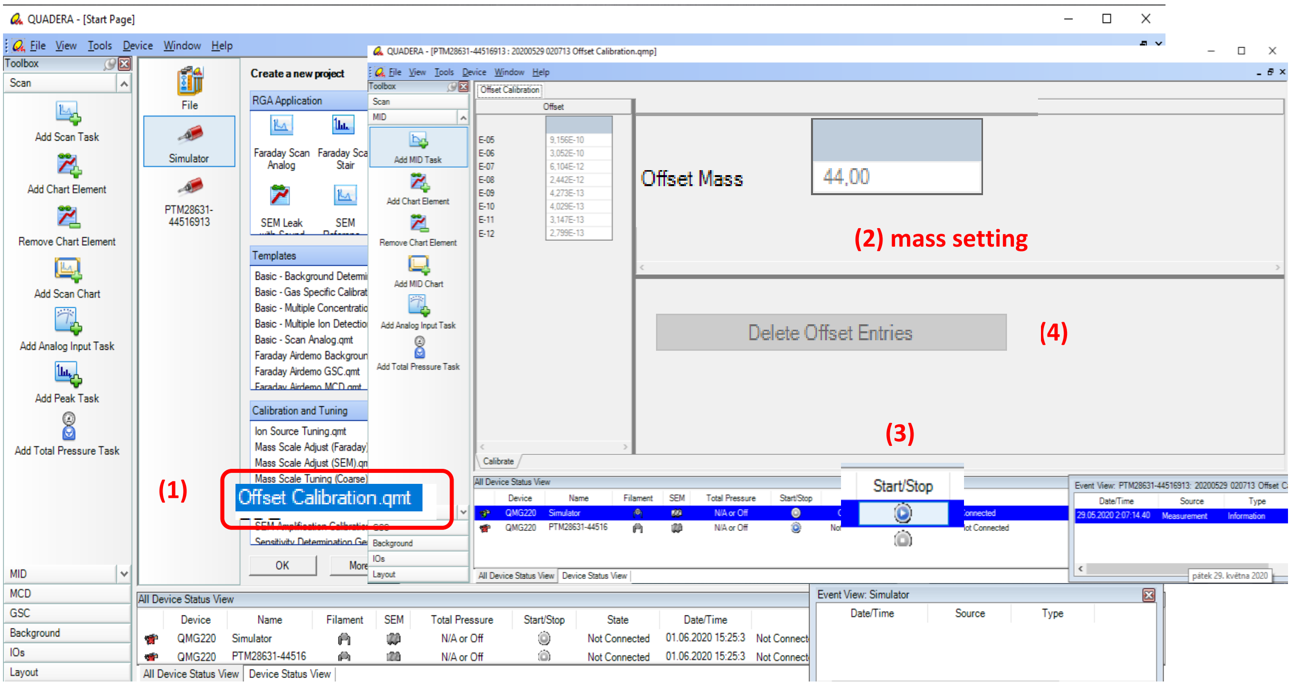


Figure 83*:Offset calibration*

6.2.5.3 *Mass Scale Tuning*

Gas components are represented by integer mass numbers. Typically, the measuring apparatus causes small deviations between the nominal mass number and the actual position of the peak maximum. The correct position of the peak maximum is usually very close to the theoretical value, e.g. at 13.95 instead of 14.00. However, an exact measurement requires measuring the peak real maximum. To achieve the best accuracy in measurements, the exact position of the peak maximum has to be determined by calibrating the mass scale.

It is preferred that the two scans cover a mass range as wide as possible. The first peak should appear at lowest mass number possible, in opposite the second peak on highest mass number.

For tuning the use of special calibration gases is recommended, but it is also possible to use the laboratory air when calibration gases are not available.

When the air is used as calibration gas, the typically tuned masses are 14 amu and 44 amu Recommended setting in recipe editor (**Figure 84**, point 3):

1. Mass: 12 amu , Width 4 amu
2. Mass: 42 amu, Width 4 amu

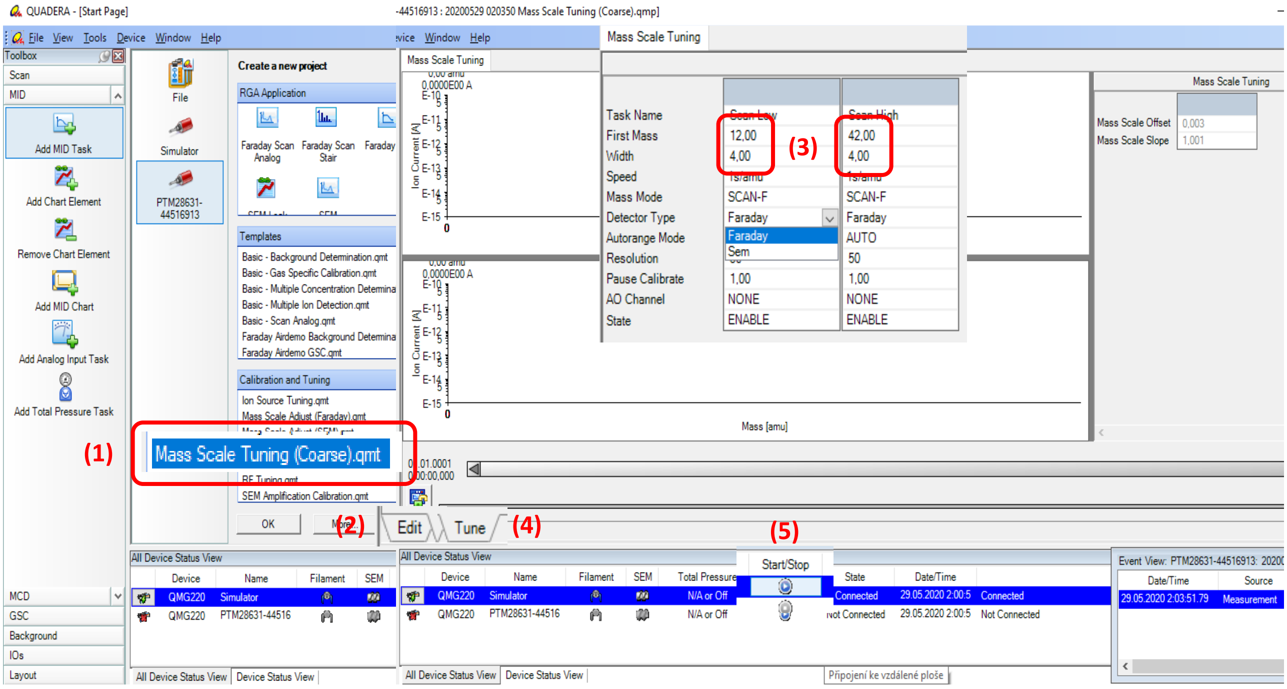
Mass Coarse Tuning

Two parameters are used to shift and to shrink or stretch the mass scale.

Click on **Mass Scale Tuning (Coarse).qmt** (**Figure 84**, point 1). This way you create a new calibration project based on the selected template. You can modify it according to your needs by using the recipe editor (**Figure 84**, point 2). Set desired mass values, scan speed of mass analyzer and detection method (**Figure 84**, point 3). Return by clicking on **tab Tune** (**Figure 84**, point 4) and **Start** the tuning (**Figure 84**, point 5). The device now receives the parameters for the current measurement task. You can adjust the chart layout while the measurement is running (**Figure 85**).

Adjust the actual peak positions to the nominal mass numbers: change the parameters in the section of the calibration project (Mass Scale Tuning editor) while the measurement is running. You can observe the effect of modified parameters during the next measurement task and iteratively tune the mass scale (**Figure 85**, red frame):

- **Mass Scale Offset** shifts the mass scale,
- **Mass Scale Slope** shrinks and stretches the mass scale.



Task Name	Scan Low	Scan High
First Mass	12,00	42,00
Width	4,00	4,00
Speed	1s/amu	1s/amu
Mass Mode	SCAN-F	SCAN-F
Detector Type	Faraday Faraday Sem	Faraday
Autorange Mode		AUTO
Resolution		50
Pause Calibrate	1,00	1,00
AO Channel	NONE	NONE
State	ENABLE	ENABLE

Mass Scale Offset	0,003
Mass Scale Slope	1,001

Figure 84*: Start of Mass Scale Tuning (Coarse)*

A particle that moves through the 4 rods of the QMS must keep a stabilized trajectory parallel to the rods to get out at the other side and hit the SEM. There are two stability parameters which are a combination of the AC-DC voltage and the electromagnetic properties of the particle. Each mass has its stability distribution inside the QMS. Stability distribution was calculated by Matthews RG and hence called Stability Diagram (**Figure 56**).

In **Figure 56** ***q*** & ***a*** are the stability parameters. The red triangle is the differential solution for the diagram per arbitrary mass. The Apex is the infinite resolution of that mass and the Load line is the AC-DC combinations voltages that are selected by the user.

The coarse mass tuning will change the slope of the load line and its offset from the axis origin point. This will not affect the load line itself. As can be seen from this step enough doesn't insure us to get a high resolution for peaks of higher masses. To get a good resolution for the whole domain we will need to curve the linear load line. Intuitively this is done by the fine mass tuning and looks like the dashed line in **Figure 86**.

Mass Scale Tuning

Mass Scale Offset 0,003

Mass Scale Slope 1,001

Device	Name	Filament	SEM	Total Pressure	Start/Stop	State	Date/Time	
QMG220	Simulator			N/A or Off		Attached	31.05.2020 21:28:2	Stopped

Date/Time	Source	Type
31.05.2020 21:28:27.48	Measurement	Information

Figure 85: *Tuning of Offset and Slope on maximum intensities of choosing mass values*

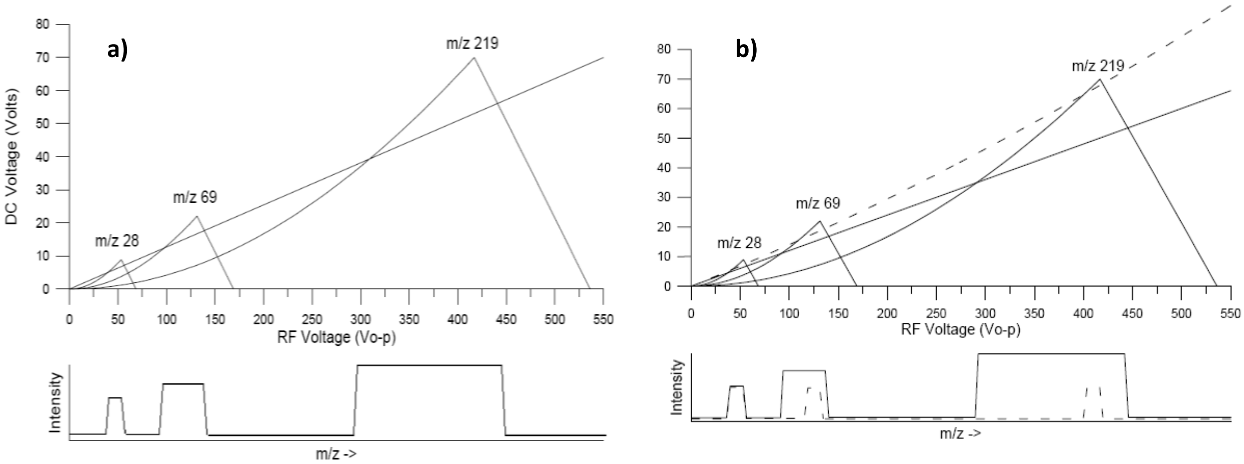


Figure 86: *The slope of the load line*

Mass Scale Adjust (Faraday, SEM)

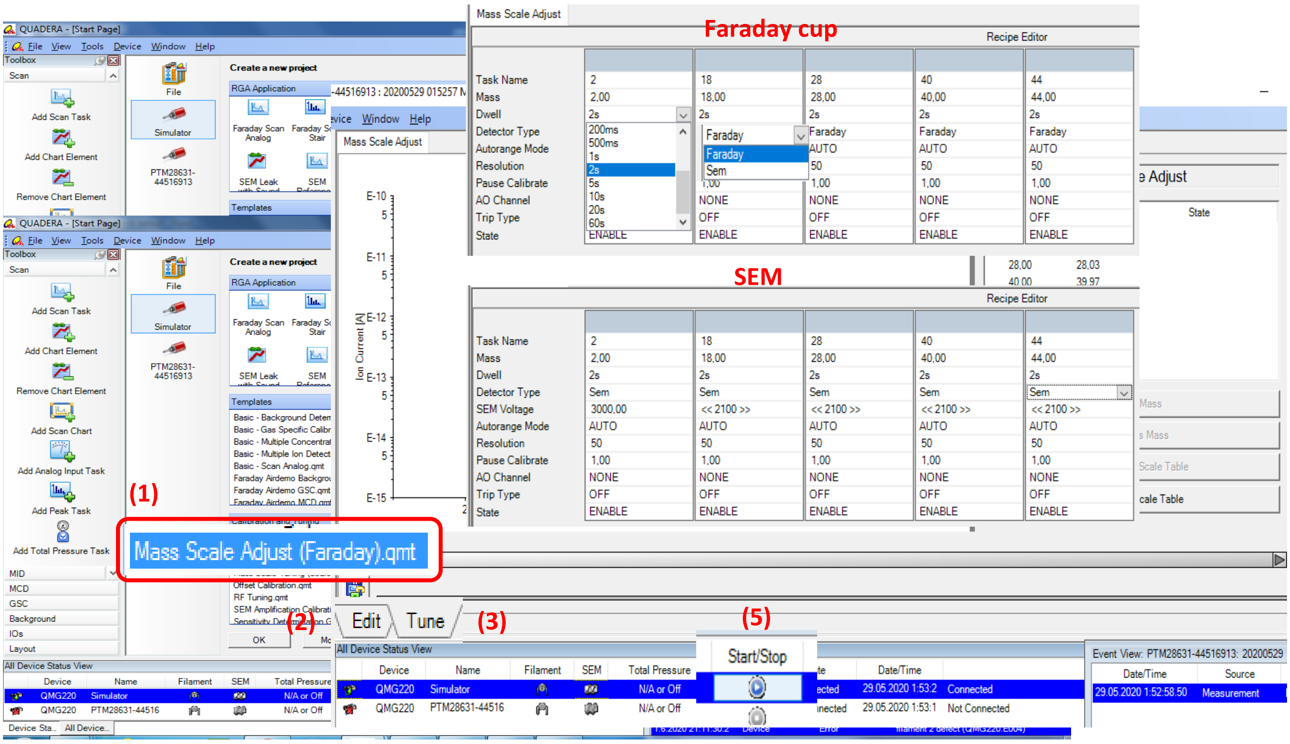
The nominal mass is the integer value of the actual mass measured by Mass Spec. In the adjust calibration the detector searches for a new peak near the nominal mass and in turn will change the actual value. The calibration must be done typically after any changes with an influence on the physical properties of the semi-permeable membrane in the membrane probe.

The template **Mass Scale Adjust** (**Figure 87**, point 1); is provided that automates the determination of measured mass numbers and can be configured through the **Recipe Editor** (**Figure 87**, points 2 and 3). The tuning; the decision to save or discard mass scale tune entries is still left to the user.

Mass Scale Adjust uses the following controls (**Figure 87**, point 3, **Figure 88**):

- **Update Mass List** – Adds new masses from the recipe into the mass adjust list. To update the mass adjust list, masses must first be entered into the recipe as measurement tasks.
- **Remove Mass** – Removes a selected mass from the mass adjust list. This action does not affect the masses (measurement tasks) in the recipe.
- **Apply to Mass Scale Table** – Downloads the changes made in mass-scale adjust to the device mass scale table. This action commits and saves the changes determined by Quadera (and the device) into the device.
- **Reset Mass Scale Table** – Resets all actual masses in the mass adjust list to their respective reference mass.
- **State column** – The following states can occur when running mass scale adjust:
  - **Ok**: The calibration has been successfully performed; the exact mass numbers are shown in the column **Actual Mass**.

- **Mass number too low**: The entered mass number is too far to the left of the peak
- **Mass number too high**: The entered mass number is too far to the right of the peak
- **Peak Intensity below threshold**: Intensity lower than the level defined by the Threshold parameter.
- **Peak Intensity too high**: Intensity equal to or higher than full-scale range.
- **Peak width too narrow**: The peak is too narrow to determine a maximum.
- **Mass number too low; intensity tail too high**: Intensity is too constant to determine a maximum.



Faraday cup – Recipe Editor

Task Name	2	18	28	40	44
Mass	2,00	18,00	28,00	40,00	44,00
Dwell	2s	2s	2s	2s	2s
Detector Type		Faraday	Faraday	Faraday	Faraday
Autorange Mode			AUTO	AUTO	AUTO
Resolution			50	50	50
Pause Calibrate		1,00	1,00	1,00	1,00
AO Channel		NONE	NONE	NONE	NONE
Trip Type		OFF	OFF	OFF	OFF
State	ENABLE	ENABLE	ENABLE	ENABLE	ENABLE

SEM – Recipe Editor

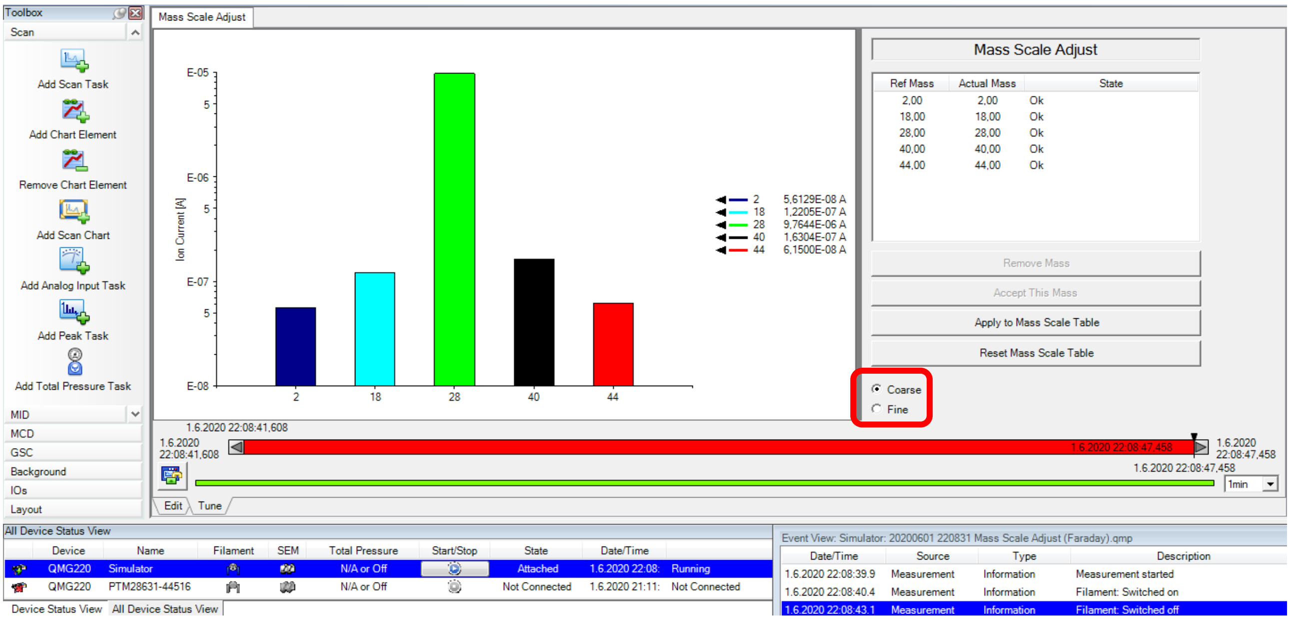
Task Name	2	18	28	40	44
Mass	2,00	18,00	28,00	40,00	44,00
Dwell	2s	2s	2s	2s	2s
Detector Type	Sem	Sem	Sem	Sem	Sem
SEM Voltage	3000,00	<< 2100 >>	<< 2100 >>	<< 2100 >>	<< 2100 >>
Autorange Mode	AUTO	AUTO	AUTO	AUTO	AUTO
Resolution	50	50	50	50	50
Pause Calibrate	1,00	1,00	1,00	1,00	1,00
AO Channel	NONE	NONE	NONE	NONE	NONE
Trip Type	OFF	OFF	OFF	OFF	OFF
State	ENABLE	ENABLE	ENABLE	ENABLE	ENABLE

All Device Status View

Device	Name	Filament	SEM	Total Pressure	Start/Stop	State	Date/Time	
QMG220	Simulator			N/A or Off		Connected	29.05.2020 1:53:2	Connected
QMG220	PTM28631-44516			N/A or Off		Not Connected	29.05.2020 1:53:1	Not Connected

Figure 87: Mass Scale Adjust Editor

The mode **Coarse** of the calibration is recommended. It expresses the distance between the actual and nominal mass (**Figure 88**, red frame).



2	5,6129E-08 A
18	1,2205E-07 A
28	9,7644E-06 A
40	1,6304E-07 A
44	6,1500E-08 A

Mass Scale Adjust

Ref Mass	Actual Mass	State
2,00	2,00	Ok
18,00	18,00	Ok
28,00	28,00	Ok
40,00	40,00	Ok
44,00	44,00	Ok

● Coarse
○ Fine

All Device Status View

Device	Name	Filament	SEM	Total Pressure	Start/Stop	State	Date/Time	
QMG220	Simulator			N/A or Off		Attached	1.6.2020 22:08:	Running
QMG220	PTM28631-44516			N/A or Off		Not Connected	1.6.2020 21:11:	Not Connected

Device Status View | All Device Status View

Event View: Simulator: 20200601 220831 Mass Scale Adjust (Faraday).qmp

Date/Time	Source	Type	Description
1.6.2020 22:08:39.9	Measurement	Information	Measurement started
1.6.2020 22:08:40.4	Measurement	Information	Filament: Switched on
1.6.2020 22:08:43.1	Measurement	Information	Filament: Switched off

Figure 88: Mass Scale Adjust Table

6.2.5.4 RF Generator Tuning

The high-frequency voltage of the RF generator is applied to the rod pairs of the quadrupole analyzer (QMG) to separate ions. The output resonant circuit of the generator has to be matched to the QMG rod system when commissioning the device and/or masses near the upper mass scale limit are measured.

The calibration project in Quadera is used to display and check the tuning voltage (**Figure 89**). Other operations with the RF generator are performed only by the PSI service.

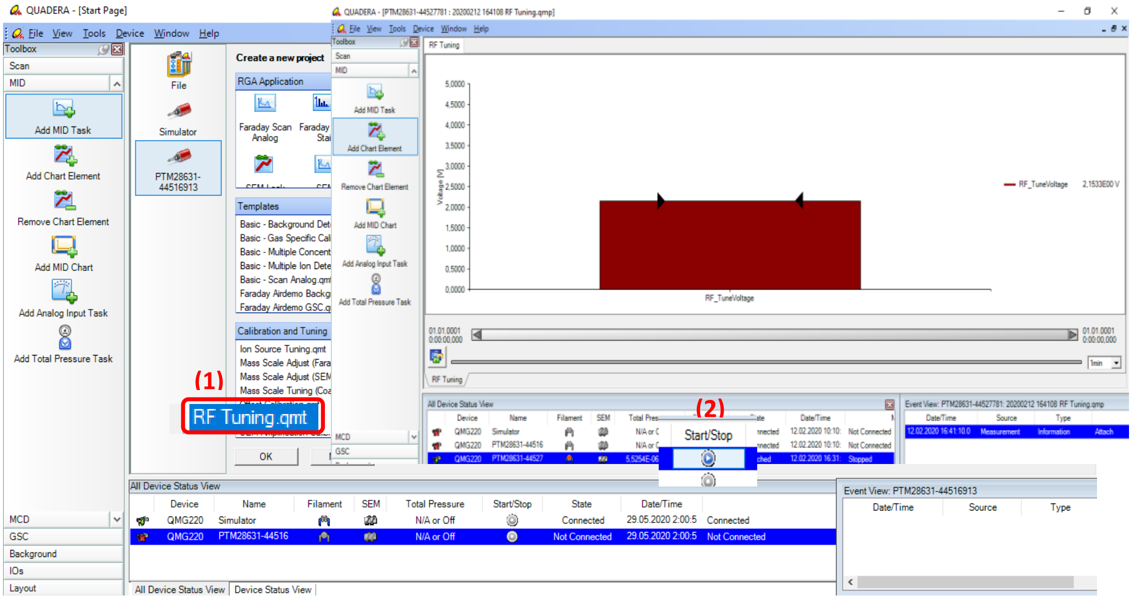
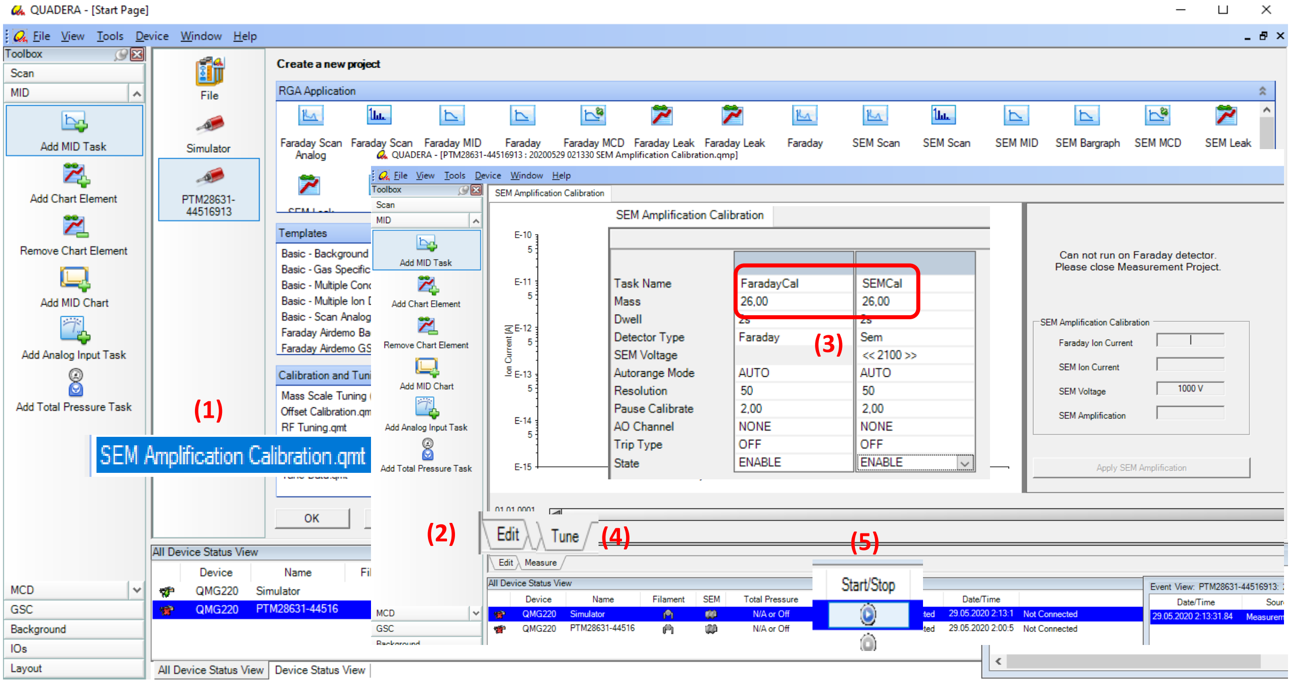


Figure 89: *RF tuning*

6.2.5.5 SEM Amplification Calibration

Calibration of the SEM detector signal multiplication is always performed for the selected chemical compound, which is in the analyzed sample under limit detection of Faraday cup.

Click on the **SEM Amplification Calibration** project in the section of Calibration and Tuning (**Figure 90**, point 1). Mass configuration of a compound is applied via recipe **Editor** (**Figure 90**, points 2 and 3). After turn use the **Tune tab** (**Figure 90**, point 4) and **Start** the measurement (**Figure 90**, point 5).



SEM Amplification Calibration

Task Name	FaradayCal	SEMCal
Mass	26,00	26,00
Dwell	2s	2s
Detector Type	Faraday	Sem
SEM Voltage		<< 2100 >>
Autorange Mode	AUTO	AUTO
Resolution	50	50
Pause Calibrate	2,00	2,00
AO Channel	NONE	NONE
Trip Type	OFF	OFF
State	ENABLE	ENABLE

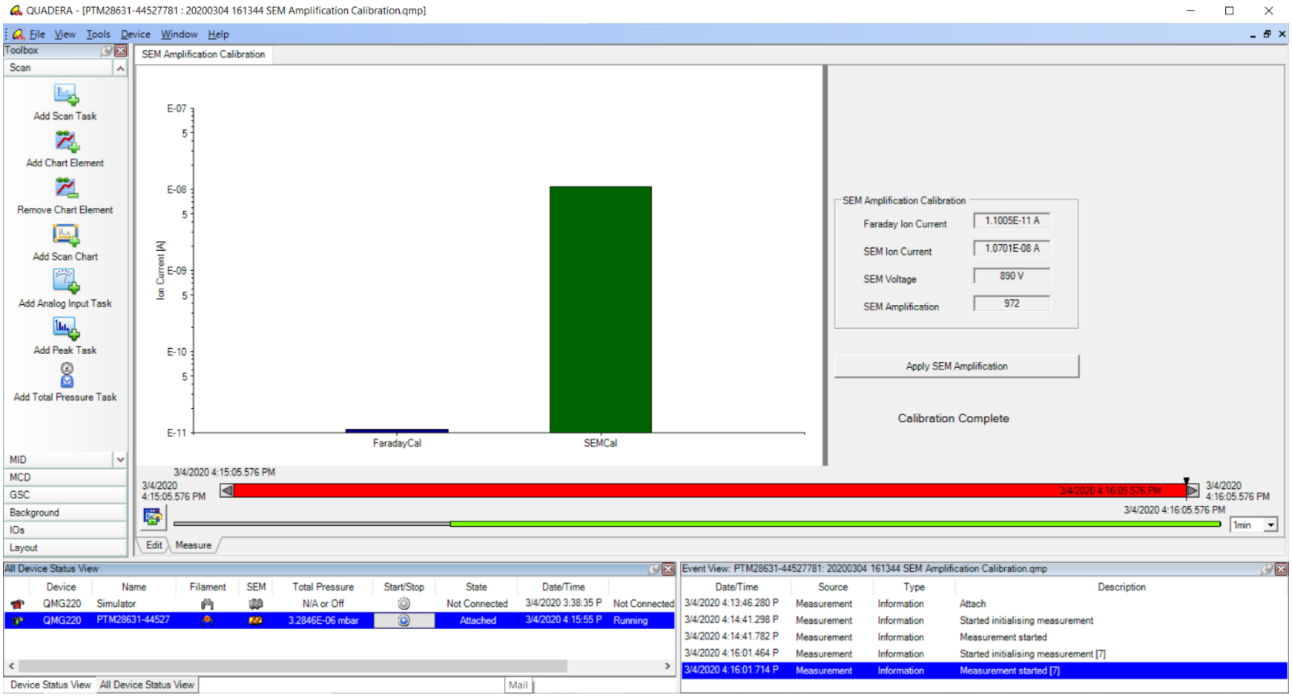
Can not run on Faraday detector. Please close Measurement Project.

SEM Amplification Calibration
Faraday Ion Current:
SEM Ion Current:
SEM Voltage: 1000 V
SEM Amplification:

Figure 90 *SEM Amplification conditions*

The selected mass (compound) is detected by the Faraday detector. The signal from Faraday is compared with the gradually increasing signal (ion current in amps) on the SEM detector. The signal growing is due to the automatic application of increasing voltage values on SEM. When the intensity of the ion current signal on the SEM detector reaches about a 1000-fold increase compared with the Faraday response, the multiplier calibration is finished (**Figure 91**).

Gas concentration is expressed as ion current in amps. **Before use of SEM, the ion current value of the measured gas (selected mass) must not exceed ±10^{-11} A on Faraday detector.** Amplification of the SEM on the selected mass with a higher ion current signal will cause serious damage to the SEM detector.



SEM Amplification Calibration
Faraday Ion Current 1.1005E-11 A
SEM Ion Current 1.0701E-08 A
SEM Voltage 890 V
SEM Amplification 972

Calibration Complete

All Device Status View

	Device	Name	Filament	SEM	Total Pressure	Start/Stop	State	Date/Time	
	QMG220	Simulator			N/A or Off	○	Not Connected	3/4/2020 3:38:35 P	Not Connected
	QMG220	PTM28631-44527			3.2846E-06 mbar	○	Attached	3/4/2020 4:15:55 P	Running

Event View: PTM28631-44527781: 20200304 161344 SEM Amplification Calibration.qmp

Date/Time	Source	Type	Description
3/4/2020 4:13:46.280 P	Measurement	Information	Attach
3/4/2020 4:14:41.298 P	Measurement	Information	Started initialising measurement
3/4/2020 4:14:41.782 P	Measurement	Information	Measurement started
3/4/2020 4:16:01.464 P	Measurement	Information	Started initialising measurement [7]
3/4/2020 4:16:01.714 P	Measurement	Information	Measurement started [7]

***Figure 91**: Final amplification of SEM*

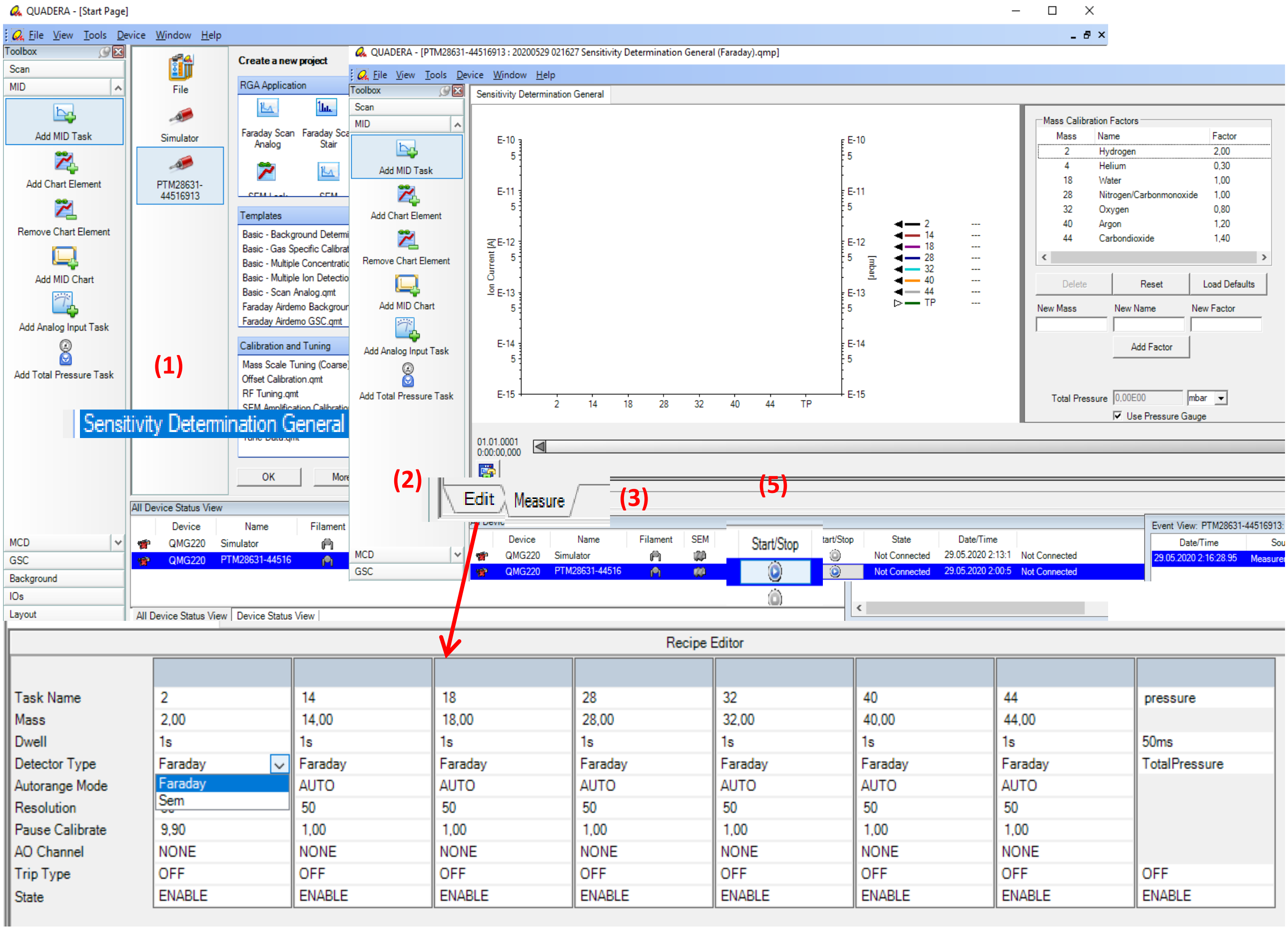
6.2.5.6 Sensitivity Determination

The sensitivity must be determined for the device to display partial pressure.

Before the measurement of sensitivity, the Offset Calibration (**Chapter 6.2.5.2**) is required.

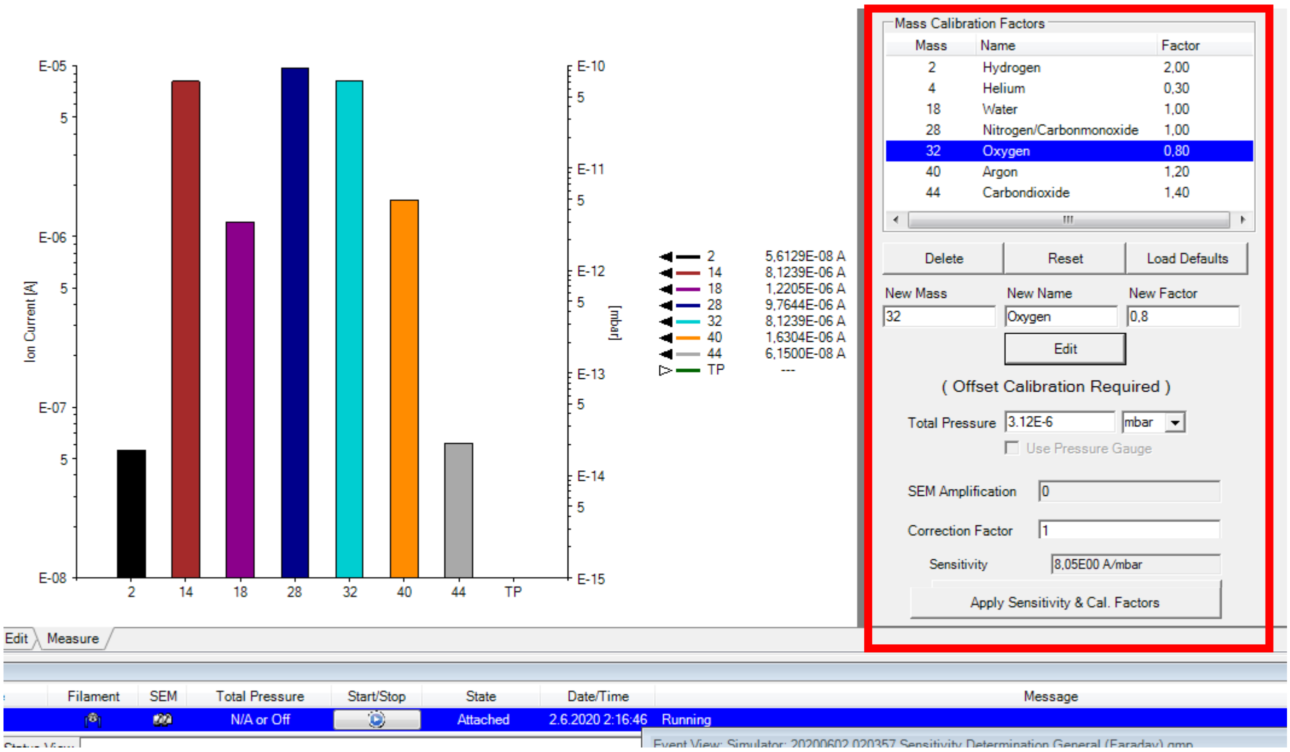
Also, each selected mass you want to include in the sensitivity measurement must have a calibration factor specified. For calibration factor determination see the **chapter 6.2.5.8** .

To determine the sensitivity, run the **Sensitivity Determination** measurement project (**Figure 92**, point 1). Set all selected masses via recipe **Editor** (**Figure 92**, point 2). If the calibration factors of measured gases are saved in the firmware device from previous analyses (**Chapter 6.2.6.3**), the calibration factor table fills automatically (**Figure 93**, red frame). Turn back using the **Measure** tab (**Figure 92**, point 3). Enter the **total pressure** manually if no gauge is attached (**Figure 92**, point 4) and run the measurement project (**Figure 92**, point 5). The device sensitivity can then be saved to its firmware (**Figure 93**, red frame).



Task Name	2	14	18	28	32	40	44	pressure
Mass	2,00	14,00	18,00	28,00	32,00	40,00	44,00	
Dwell	1s	1s	1s	1s	1s	1s	1s	50ms
Detector Type	Faraday	Faraday	Faraday	Faraday	Faraday	Faraday	Faraday	TotalPressure
Autorange Mode	Faraday	AUTO	AUTO	AUTO	AUTO	AUTO	AUTO	
Resolution	Sem	50	50	50	50	50	50	
Pause Calibrate	9,90	1,00	1,00	1,00	1,00	1,00	1,00	
AO Channel	NONE	NONE	NONE	NONE	NONE	NONE	NONE	
Trip Type	OFF	OFF	OFF	OFF	OFF	OFF	OFF	OFF
State	ENABLE	ENABLE	ENABLE	ENABLE	ENABLE	ENABLE	ENABLE	ENABLE

***Figure 92**: Setting of parameters for device sensitivity determination*



2	5,6129E-08 A
14	8,1239E-06 A
18	1,2205E-06 A
28	9,7644E-06 A
32	8,1239E-06 A
40	1,6304E-06 A
44	6,1500E-08 A
TP	---

Mass Calibration Factors

Mass	Name	Factor
2	Hydrogen	2,00
4	Helium	0,30
18	Water	1,00
28	Nitrogen/Carbonmonoxide	1,00
32	Oxygen	0,80
40	Argon	1,20
44	Carbondioxide	1,40

Delete Reset Load Defaults

New Mass: 32
New Name: Oxygen
New Factor: 0,8

Edit

(Offset Calibration Required)

Total Pressure: 3.12E-6 mbar
☐ Use Pressure Gauge
SEM Amplification: 0
Correction Factor: 1
Sensitivity: 8,05E00 A/mbar

Apply Sensitivity & Cal. Factors

Filament	SEM	Total Pressure	Start/Stop	State	Date/Time	Message
		N/A or Off		Attached	2.6.2020 2:16:46	Running

Event View: Simulator: 20200602 020357 Sensitivity Determination General (Faraday).qmp

***Figure 93**:Determined device sensitivity*

6.2.5.7 Background Determination

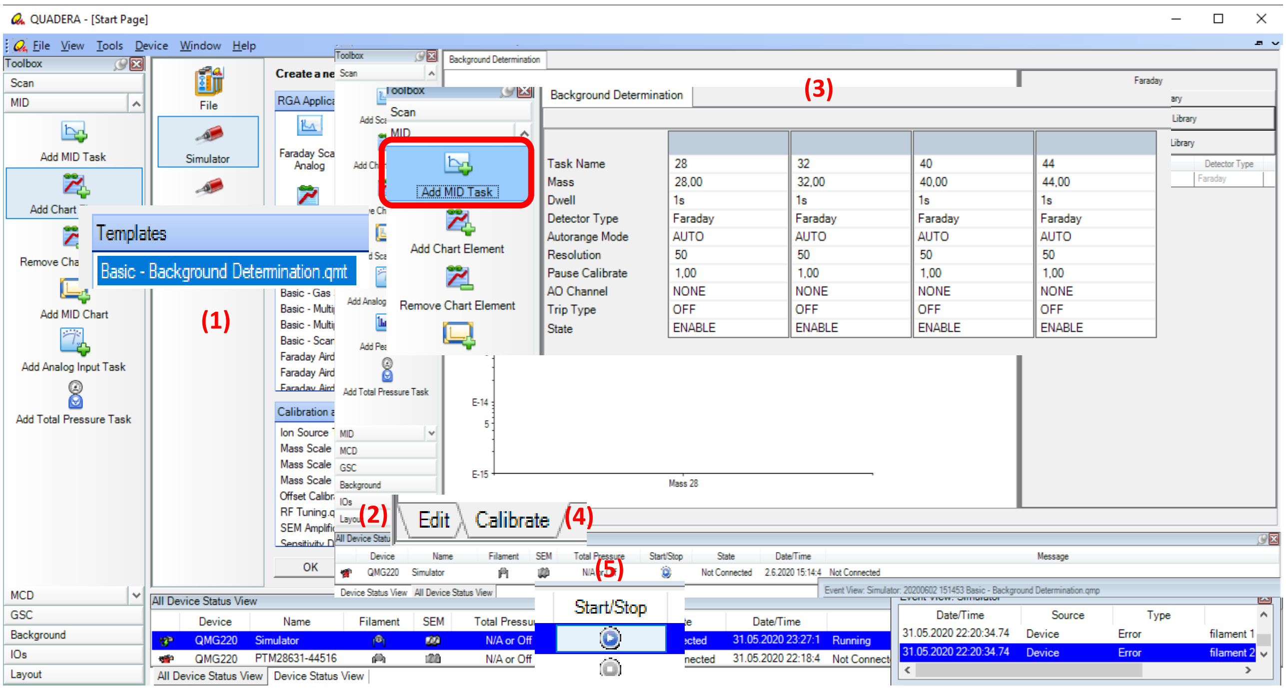
The mass spectrometer background is caused by residual gases. These gases are present in every analysis chamber and do not originate from the measuring sample. The background is mainly constant if the measuring apparatus is well-conditioned. The background determination identifies the existing residual gases. The detected ion currents are subtracted from subsequent measurement projects. The result will show the true composition of the measured gas.

Residual gases are measured either with the gas inlet closed (the Inlet Valve must be closed, **Figure 37**) or by using a zero gas, i.e. a pure gas that is fed into the ion chamber to reach the same pressure as expected for future measurements.

A MID measurement (**Chapter 6.2.6.2** , **Figure 95** – left red frame) is used to determine the background. You can determine the mass spectrometer background for each detector type separately and store the results in the Background Library. The stored values will be used in future MID and MCD measurement projects (**Chapters 6.2.6.2** and **6.2.6.3**) where they are subtracted from the measured signals.

⚠	Always pump down the chamber to final vacuum before performing the background determination, or let in a suitable zero gas. Otherwise, serious measurement errors may result.
⚠	Before starting the measurement, check the correct calibration of the mass spectrometer components. If calibration is necessary, perform the following preparatory measurements: Calibrate the mass scale (**Chapter 6.2.5.3**) Determine the offset. (**Chapter 6.2.5.2**)

After applying a suitable zero gas into the vacuum chamber, or pumping the chamber down to the final vacuum with a closed inlet valve, create a new calibration project. Use the protocol **Background Determination.qmt** (**Figure 94**, point 1) in the Template list. Edit the parameters (**Figure 94**, points 2, and 3) and return to calibration (**Figure 94**, point 4). The measurement tasks must not contain mass numbers where peaks of the zero gas appear. After click on the Start/Stop tab the measurement is running automatically (**Figure 94**, point 5). Check the measured values and click on **Apply to Background Library** to forward the background measurement to the Background Library (**Figure 95**, right red frame).



Task Name	28	32	40	44
Mass	28,00	32,00	40,00	44,00
Dwell	1s	1s	1s	1s
Detector Type	Faraday	Faraday	Faraday	Faraday
Autorange Mode	AUTO	AUTO	AUTO	AUTO
Resolution	50	50	50	50
Pause Calibrate	1,00	1,00	1,00	1,00
AO Channel	NONE	NONE	NONE	NONE
Trip Type	OFF	OFF	OFF	OFF
State	ENABLE	ENABLE	ENABLE	ENABLE

Device	Name	Filament	SEM	Total Pressure	Start/Stop	State	Date/Time	Message
QMG220	Simulator			N/A or Off		Not Connected	2.6.2020 15:14:4	Not Connected

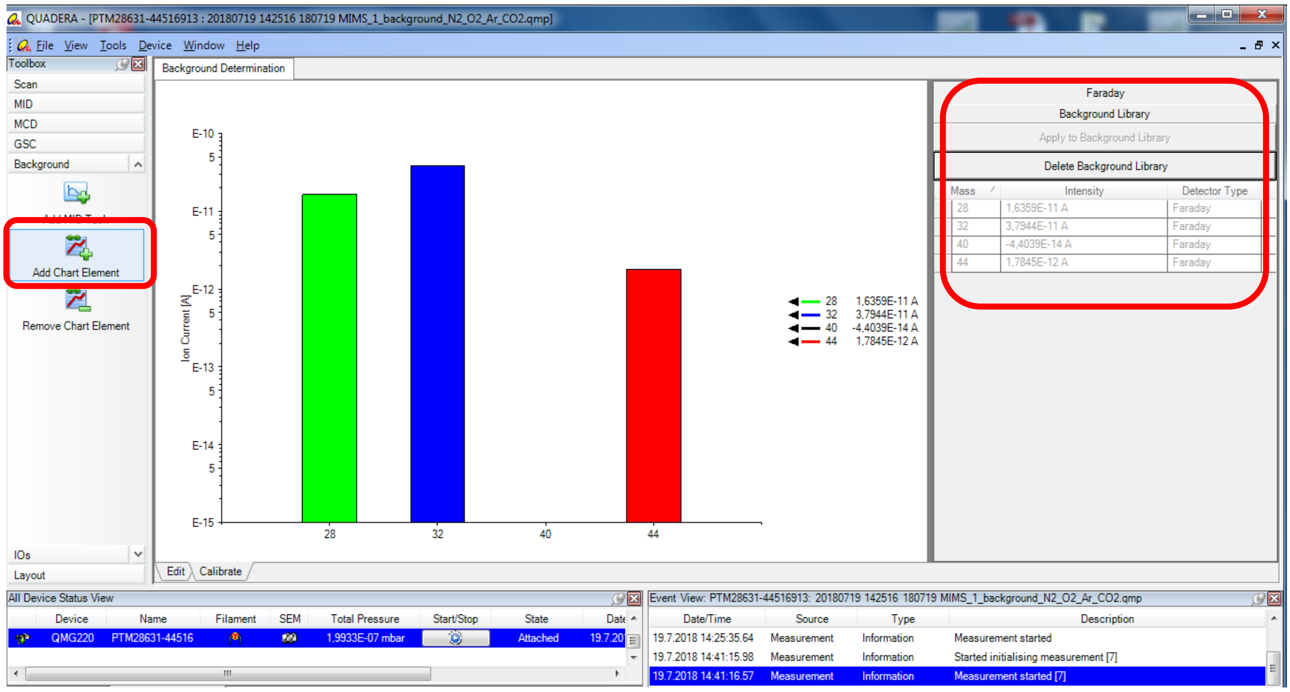
Event View: Simulator: 20200602 151453 Basic - Background Determination.qmp

Date/Time	Source	Type	
31.05.2020 22:20:34.74	Device	Error	filament 1
31.05.2020 22:20:34.74	Device	Error	filament 2

Figure 94: Background Determination project start

	New background measurements (mass numbers) are added to the Background Library, current measuring values replace existing data sets in the Background Library.

The data sets in the Background Library will be subtracted in MID and MCD measurements (**Chapters 6.2.6.2** and **6.2.6.3**). If you wish to skip this subtraction then reset the data sets in the Background Library (Delete Background Library button). Background Subtraction can also be disabled, without resetting the Background Library, from the context menu when running a Measurement Project (**Figure 95**).



Mass	Intensity	Detector Type
28	1,6359E-11 A	Faraday
32	3,7944E-11 A	Faraday
40	-4,4039E-14 A	Faraday
44	1,7845E-12 A	Faraday

Device	Name	Filament	SEM	Total Pressure	Start/Stop	State	Date
QMG220	PTM28631-44516			1,9933E-07 mbar		Attached	19.7.20

Event View: PTM28631-44516913: 20180719 142516 180719 MIMS_1_background_N2_O2_Ar_CO2.qmp

Date/Time	Source	Type	Description
19.7.2018 14:25:35.64	Measurement	Information	Measurement started
19.7.2018 14:41:15.98	Measurement	Information	Started initialising measurement [7]
19.7.2018 14:41:16.57	Measurement	Information	Measurement started [7]

Figure 95*: Background Determination with Inlet Valve closed*

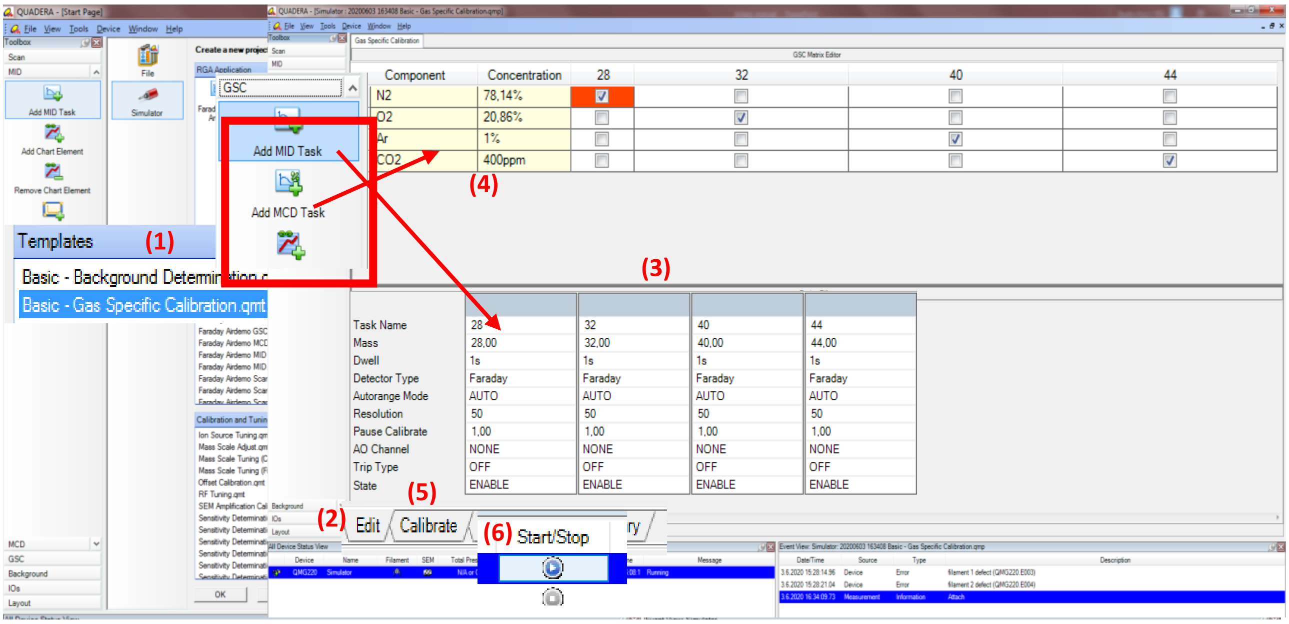
6.2.5.8 Gas Specific Calibration (GSC)

A Multiple Concentration Detection (MCD, **Chapter 6.2.6.3**) measurement project uses a matrix of calibration factors (analysis matrix) to convert ion currents into gas concentrations. The calibration factors are not universally valid. They have to be quantified by a separate calibration measurement **Gas Specific Calibration** using known gases. Calibration gases are selected according to the components in the measurement project whose concentrations are to be measured.

⚠	Perform the following preparatory measurements before the gas specific calibration: Calibrate the mass scale (**Chapter 6.2.5.3**) Determine the offset (**Chapter 6.2.5.2**) Determine the background (**Chapter 6.2.5.7**).
⚠	Gas mixtures that contain components with overlapping masses are not suited for use as calibration gas. The same mass number must not be assigned to several components.

Create a MID measurement project using a click on **Gas Specific Calibration** in Template section (**Figure 96**, point 1). The recipe parameters include the mass numbers of the calibration gas components and can be modified in the **Editor** (**Figure 96**, points 2 and 3). The GSC Matrix editor specifies the concentration of each calibration gas component and its contribution to the related mass number (**Figure 96**, point 4). The gas specific calibration factors are required for the actual MCD measurement project. They are determined in the calibration project (**GSC Result View)** and saved in the **MCD Calibration Factor Library** (**Chapter 6.2.6.3**). Return by Calibrate tab (**Figure 96**, point 5) and **Start** the measurement (**Figure 96**, point 6).

ℹ	Use an identical name for the gas component in an MCD project. Otherwise, Quadera cannot refer to this component. For example, Quadera does not equate O_2 and Oxygen.

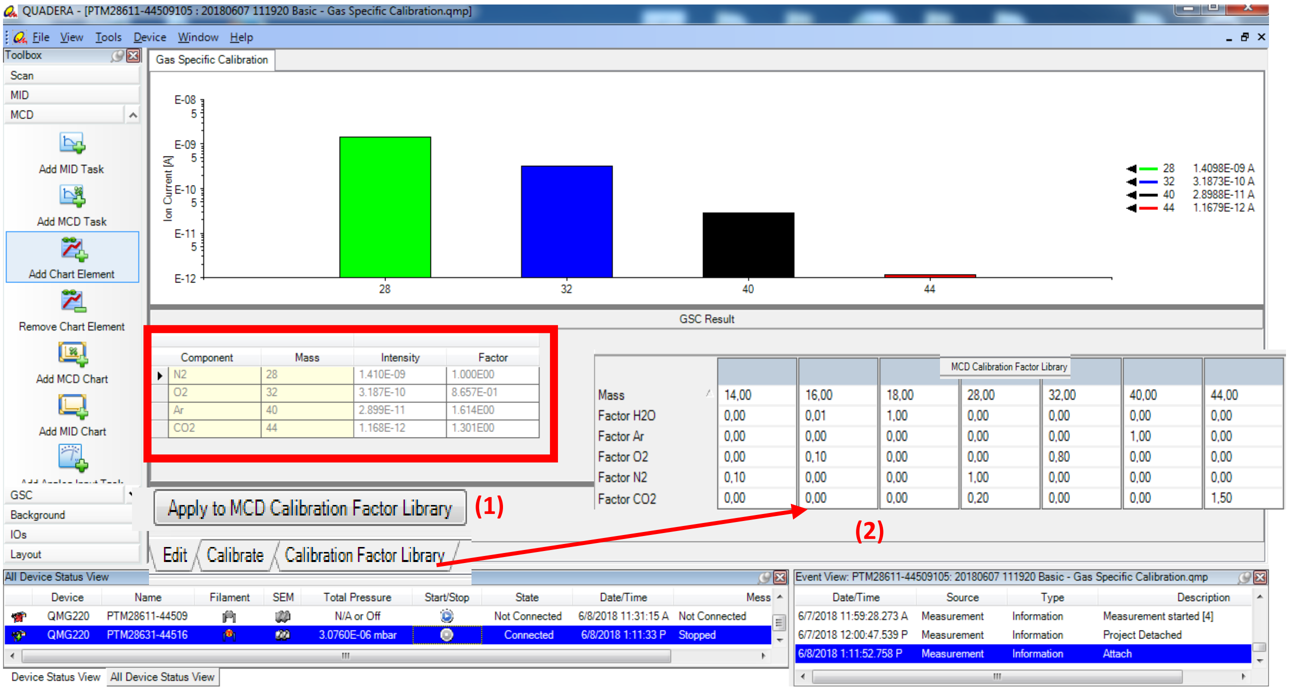


Component	Concentration	28	32	40	44
N2	78,14%	☑	☐	☐	☐
O2	20,86%	☐	☑	☐	☐
Ar	1%	☐	☐	☑	☐
CO2	400ppm	☐	☐	☐	☑

Task Name	28	32	40	44
Mass	28,00	32,00	40,00	44,00
Dwell	1s	1s	1s	1s
Detector Type	Faraday	Faraday	Faraday	Faraday
Autorange Mode	AUTO	AUTO	AUTO	AUTO
Resolution	50	50	50	50
Pause Calibrate	1,00	1,00	1,00	1,00
AO Channel	NONE	NONE	NONE	NONE
Trip Type	OFF	OFF	OFF	OFF
State	ENABLE	ENABLE	ENABLE	ENABLE

Date/Time	Source	Type	Description
3.6.2020 15:28:14.96	Device	Error	filament 1 defect (QMG220.E003)
3.6.2020 15:28:21.04	Device	Error	filament 2 defect (QMG220.E004)
3.6.2020 16:34:09.73	Measurement	Information	Attach

***Figure 96**: Setting of selected masses in GSC recipe editor*



Component	Mass	Intensity	Factor
N2	28	1.410E-09	1.000E00
O2	32	3.187E-10	8.657E-01
Ar	40	2.899E-11	1.614E00
CO2	44	1.168E-12	1.301E00

Mass	14,00	16,00	18,00	28,00	32,00	40,00	44,00
Factor H2O	0,00	0,01	1,00	0,00	0,00	0,00	0,00
Factor Ar	0,00	0,00	0,00	0,00	0,00	1,00	0,00
Factor O2	0,00	0,10	0,00	0,00	0,80	0,00	0,00
Factor N2	0,10	0,00	0,00	1,00	0,00	0,00	0,00
Factor CO2	0,00	0,00	0,00	0,20	0,00	0,00	1,50

All Device Status View

Device	Name	Filament	SEM	Total Pressure	Start/Stop	State	Date/Time	Mess
QMG220	PTM28611-44509			N/A or Off		Not Connected	6/8/2018 11:31:15 A	Not Connected
QMG220	PTM28631-44516			3.0760E-06 mbar		Connected	6/8/2018 1:11:33 P	Stopped

Event View: PTM28611-44509105: 20180607 111920 Basic - Gas Specific Calibration.qmp

Date/Time	Source	Type	Description
6/7/2018 11:59:28.273 A	Measurement	Information	Measurement started [4]
6/7/2018 12:00:47.539 P	Measurement	Information	Project Detached
6/8/2018 1:11:52.758 P	Measurement	Information	Attach

***Figure 97**: Final Calibration Factors*

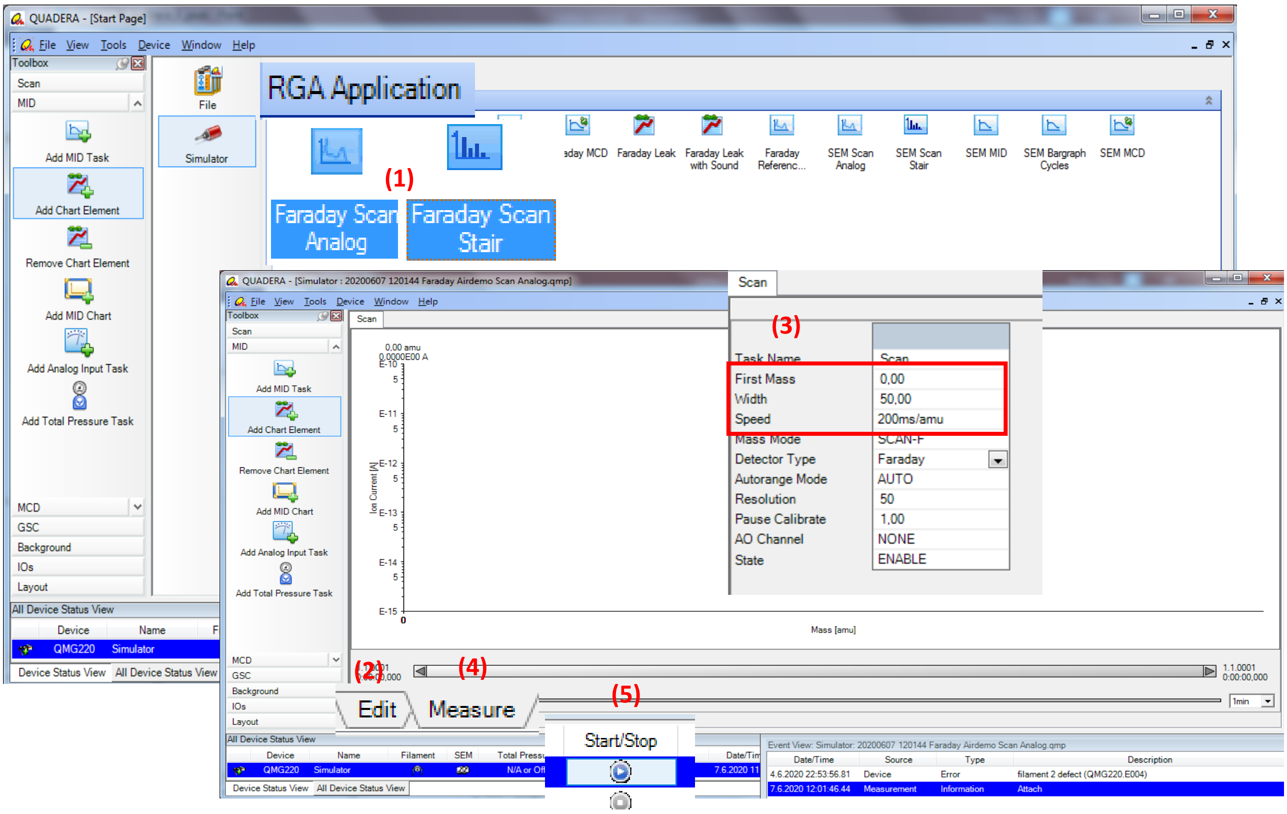
In the **GSC Result View**, check the measured values and the calculated calibration factors (**Figure 97**, red frame). Click **Apply to MCD Calibration Factor Library** to save the calibration factors in the MCD calibration factor library (**Figure 97**, point 1). New calibration factors are added to the MCD Calibration Factor Library, current values replace existing data (**Figure 97**, point 2). When required for future applications save the calibration project as template (**Chapter 6.2.4.3**).

6.2.6 RGA APPLICATION SECTION_BASIC PROJECTS

6.2.6.1 Scan of the Mass Range

In a scan measurement, the ion current is measured as a function of ion weight **m** to ion charge **e** (**m/e**). The scan quickly allows to know a qualitative composition of the gas and/or volatiles in samples. Using scan project the qualitative analysis is performed, the majority content or significant fractions in entire range of mass analyzer can be identified. The scan graphs are displayed in analog and/or stairs mode.

Double-click on the selected protocol (**Faraday Scan Analog, Faraday Scan Stair** or SEM Scan Analog, SEM Scan Stairs, **Figure 98**, point 1), the window with working area opens. In the recipe **Editor** set the scaned mass range and speed of scanning (**Figure 98**, points 2 and 3, red frames). Turn by **Measure** tab (**Figure 98**, point 4) and **Start** the measurement (**Figure 98**, point 5).



Task Name	Scan
First Mass	0,00
Width	50,00
Speed	200ms/amu
Mass Mode	SCAN-F
Detector Type	Faraday
Autorange Mode	AUTO
Resolution	50
Pause Calibrate	1,00
AO Channel	NONE
State	ENABLE

Event View: Simulator: 20200607 120144 Faraday Airdemo Scan Analog.qmp

Date/Time	Source	Type	Description
4.6.2020 22:53:56.81	Device	Error	filament 2 defect (QMG220.E004)
7.6.2020 12:01:46.44	Measurement	Information	Attach

***Figure 98**: Scan mode setting*

The MassSpec scans the sample contents at selected mass range. The graph displayes the signal in analog or stairs mode (**Figure 99**). Quadera also provides a scan-analog measurement project template that displays a 3D graph of analog scans .

⚠	3D uses system resources and should be used for only short periods of time, if it is possible.

However, in the scan mode, it is relatively difficult to derive the quantitative composition of the gas in % or ppm. Information over time is only available from a series of scans, so long-term measurements lead to an extremely large amount of data.

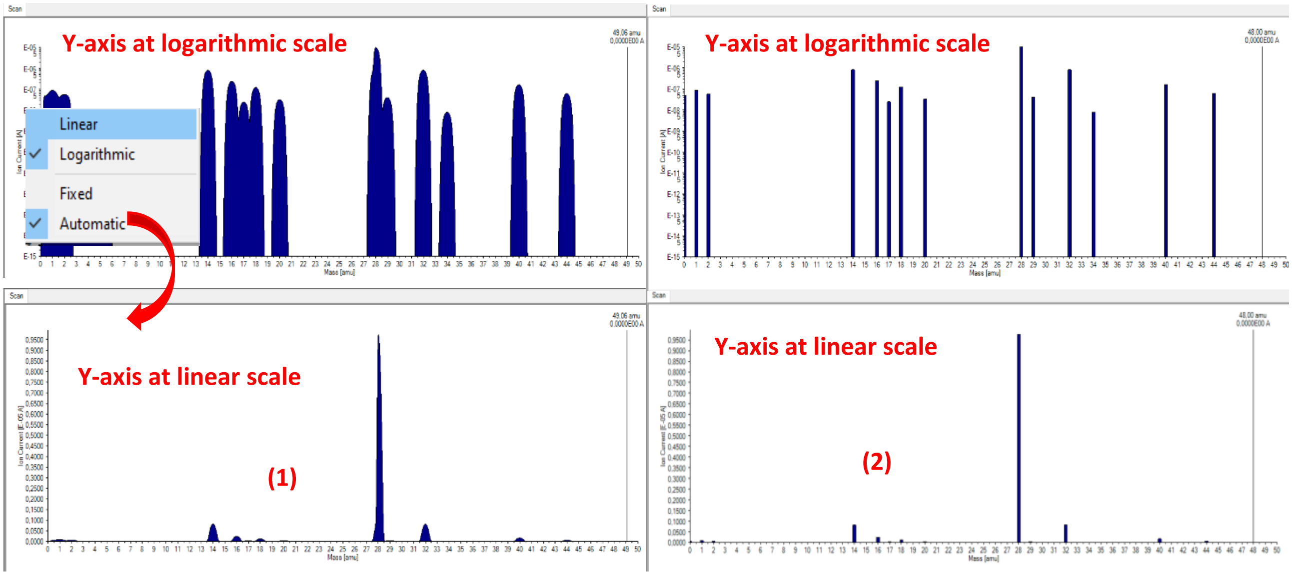


Figure 99*: Scan chart: 1) analog mode 2) stairs mode*

6.2.6.2 Multiple Ion Detection (MID)

The MID measurement was taken as the general example for explicate the parameters setting and control of predefinied protocols in **Chapter 6.2.4** .

In an **MID** measurement (**Multiple Ion Detection**) you specify distinct m/e values for which the ion current has to be measured. Only the specified mass numbers are scanned in this case. Each of these mass numbers is represented by a separate measurement task for which all parameters can be set (**Figure 65**,**Figure 66**, **Figure 70**). Changes in the gas composition with time can be recorded and displayed easily (**Figure 73**). An additional analysis of the measuring data allows to convert the results into concentrations given in %, ppm, etc (MCD measurement, **Chapter 6.2.6.3**).

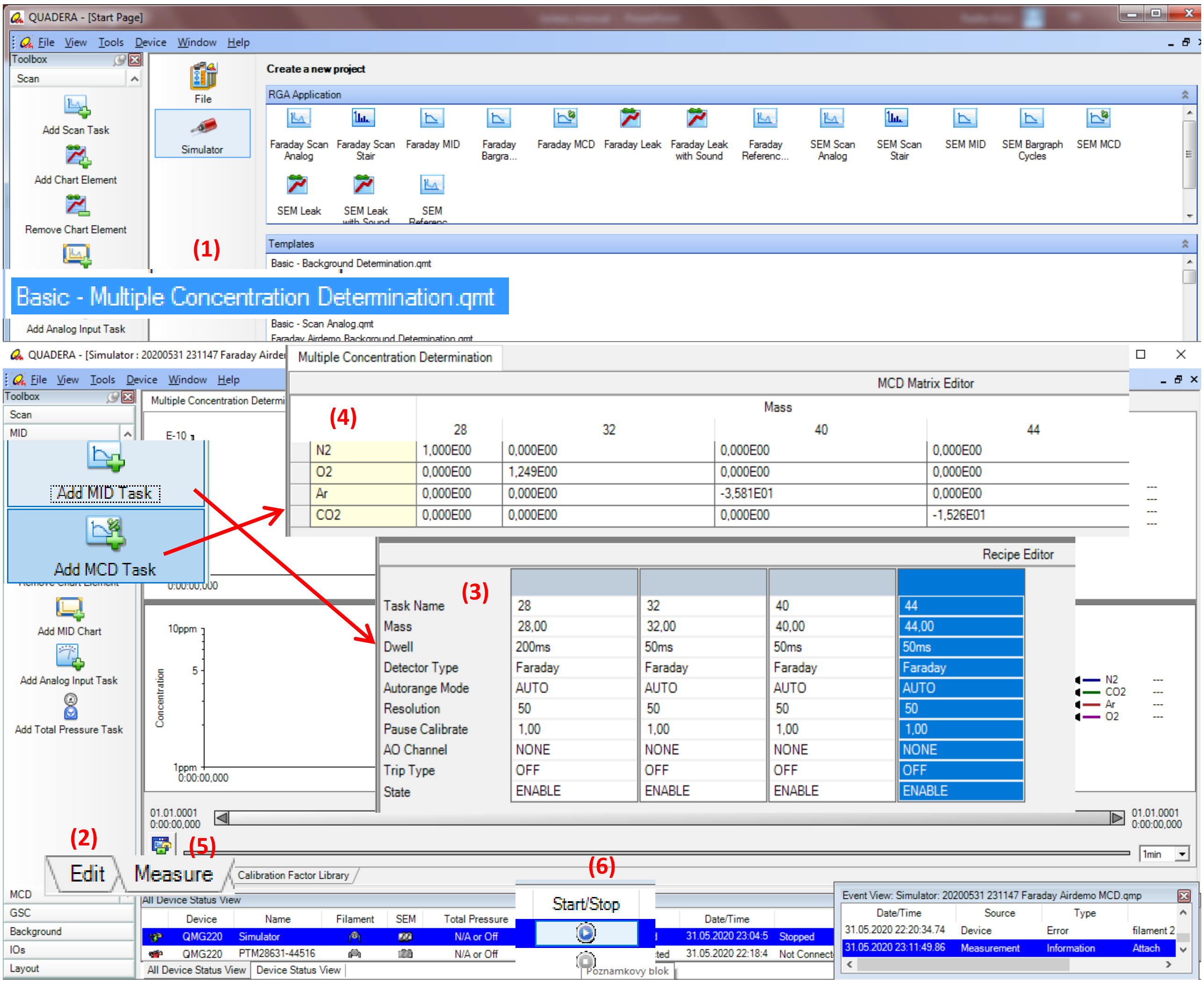
MID Measurement data can be displayed in amps or partial pressure. To display in partial pressure, one must first run the Offset Calibration measurement (**Chapter 6.2.5.2**), calibrate the SEM Amplification (**Chapter 6.2.5.5**), then determine the device Sensitivity (**Chapter 6.2.5.6**) and at last set the partial pressure units (**Figure 75**).

6.2.6.3 Multiple Concentration Determination (MCD)

The **MCD** measurement (**Multiple Concentration Determination**) is based on a MID measurement project (**Chapter 6.2.6.2**) and includes additional evaluation to measure concentrations. A matrix of calibration factors (analysis matrix) is used to convert the measured ion currents into concentrations of the individual gas components. The calibration factors are not universally valid. They must be quantified by a separate measurement using calibration gases, i.e. one or more GSC measurements (**Chapter 6.2.5.8**). The actual MCD measurement and the calibration are closely connected.

⚠	Perform the following preparatory measurements before starting the measurement: Calibrate the mass scale (**Chapter 6.2.5.3**) Determine the offset (**Chapter 6.2.5.2**) Determine the background (**Chapter 6.2.5.7**) Determine the gas specific calibration factors (**Chapter 6.2.5.8**)

Double-click on the **MCD** measurement project in the Templates list (**Figure 100**, point 1). A new MCD project window will appear. Click the **Edit** tab (**Figure 100**, point 2). Two tables are opened to modify MID parameters (**Figure 100**, point 3 and **Figure 101**, **Chapter 6.2.6.2**) and MCD matrix with specific calibration factors downloaded from the GSC project (**Figure 100**, point 4 and **Chapter 6.2.5.8**). Return by **Measure** tab (**Figure 100**, point 5) and **Start** the measurment (**Figure 100**, point 6). Two graphs appear. The first graph shows a normal MID measurement with the ion current signal of individual gases. The calculated set gas concentrations based on the matrix and specific calibration factors are included in the second graph (**Figure 102**).



MCD Matrix Editor

	Mass 28	32	40	44
N2	1,000E00	0,000E00	0,000E00	0,000E00
O2	0,000E00	1,249E00	0,000E00	0,000E00
Ar	0,000E00	0,000E00	-3,581E01	0,000E00
CO2	0,000E00	0,000E00	0,000E00	-1,526E01

Recipe Editor

Task Name	28	32	40	44
Mass	28,00	32,00	40,00	44,00
Dwell	200ms	50ms	50ms	50ms
Detector Type	Faraday	Faraday	Faraday	Faraday
Autorange Mode	AUTO	AUTO	AUTO	AUTO
Resolution	50	50	50	50
Pause Calibrate	1,00	1,00	1,00	1,00
AO Channel	NONE	NONE	NONE	NONE
Trip Type	OFF	OFF	OFF	OFF
State	ENABLE	ENABLE	ENABLE	ENABLE

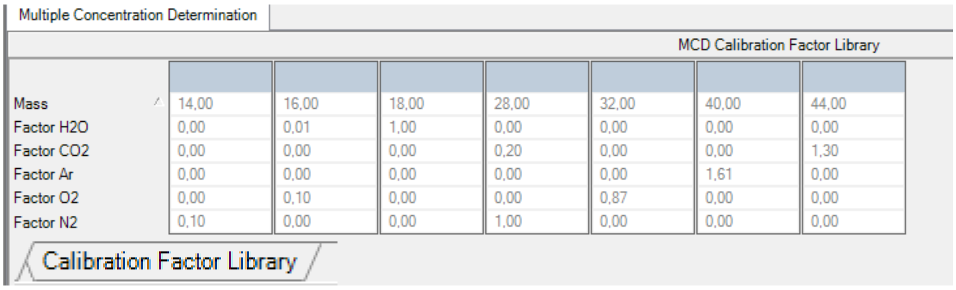
All Device Status View

Device	Name	Filament	SEM	Total Pressure	Date/Time	
QMG220	Simulator			N/A or Off	31.05.2020 23:04:5	Stopped
QMG220	PTM28631-44516			N/A or Off	31.05.2020 22:18:4	Not Connect

Event View

Date/Time	Source	Type	
31.05.2020 22:20:34.74	Device	Error	filament 2
31.05.2020 23:11:49.86	Measurement	Information	Attach

***Figure 100**: MCD protocol setting*



Mass	14,00	16,00	18,00	28,00	32,00	40,00	44,00
Factor H2O	0,00	0,01	1,00	0,00	0,00	0,00	0,00
Factor CO2	0,00	0,00	0,00	0,20	0,00	0,00	1,30
Factor Ar	0,00	0,00	0,00	0,00	0,00	1,61	0,00
Factor O2	0,00	0,10	0,00	0,00	0,87	0,00	0,00
Factor N2	0,10	0,00	0,00	1,00	0,00	0,00	0,00

***Figure 101**: Example of specific calibration factors for several masses*

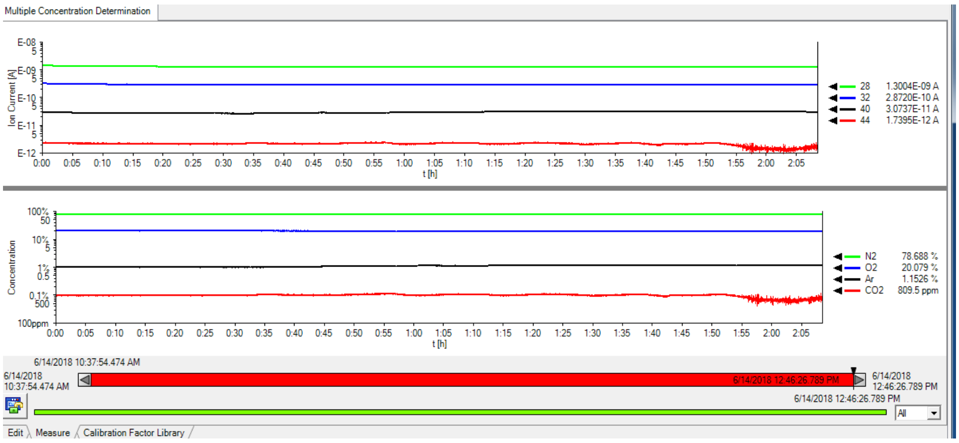
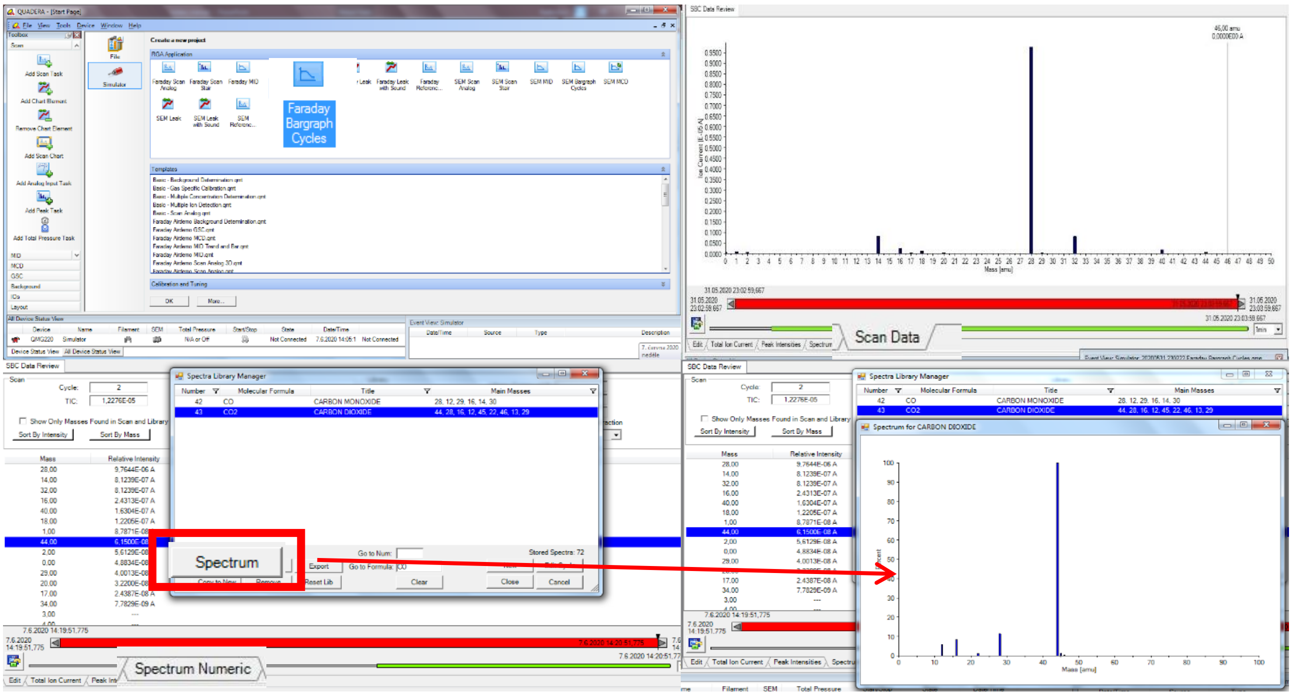


***Figure 102**: MCD measurement of control athmosphere in Plant chamber (**Figure 7**) with lettuce*

6.2.6.4 *Special Measurement Protocols*

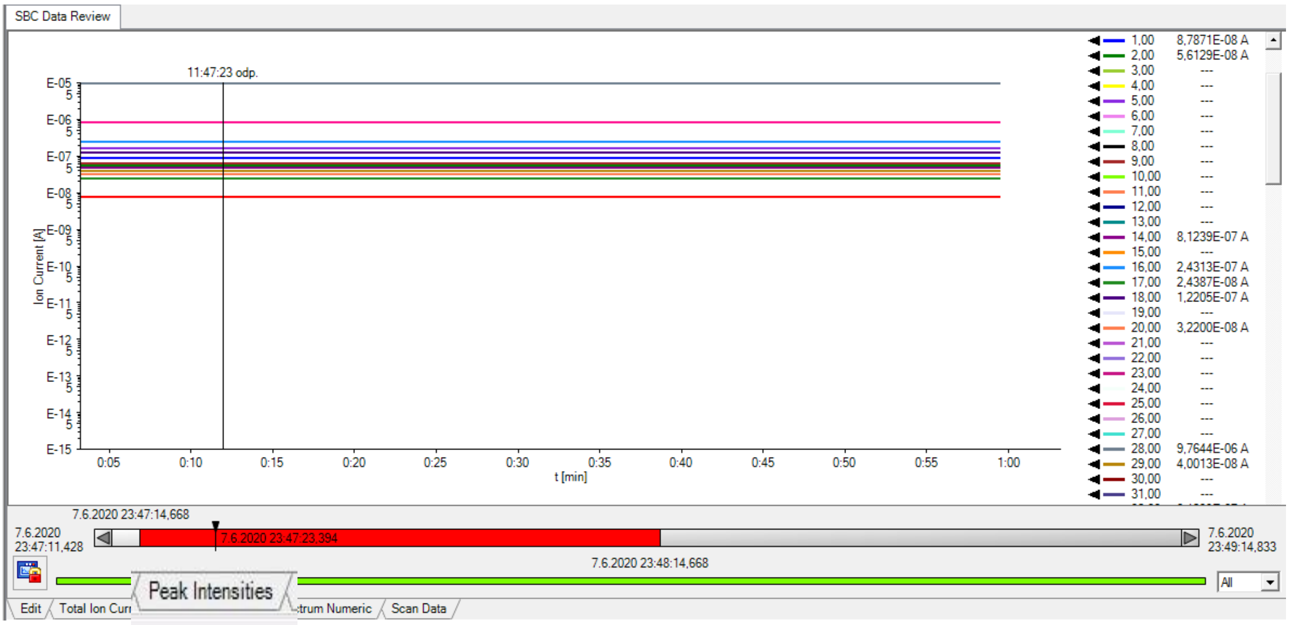
Quadera software allows to display the scan graph in other special modes:

- **Bargraph Cycles -** a separate measurement project can be used to plot analog scans against time. The basic graph is a mass scan at the selected mass range. The scan can then be related to the timeline. The project also works closely with the spectrum library stored in the Quadera software. Theoretical mass spectra can be compared with real sample spectra and used for the identification of unknown components (**Figure 103**, **Figure 104**).



Number	Molecular Formula	Title	Main Masses
42	CO	CARBON MONOXIDE	28, 12, 29, 16, 14, 30
43	CO2	CARBON DIOXIDE	44, 28, 16, 12, 45, 22, 46, 13, 29

***Figure 103**: Bargraph scan and relationship with Spectrum Library*



1,00	8,7871E-08 A
2,00	5,6129E-08 A
3,00	---
4,00	---
5,00	---
6,00	---
7,00	---
8,00	---
9,00	---
10,00	---
11,00	---
12,00	---
13,00	---
14,00	8,1239E-07 A
15,00	---
16,00	2,4313E-07 A
17,00	2,4387E-08 A
18,00	1,2205E-07 A
19,00	---
20,00	3,2200E-08 A
21,00	---
22,00	---
23,00	---
24,00	---
25,00	---
26,00	---
27,00	---
28,00	9,7644E-06 A
29,00	4,0013E-08 A
30,00	---
31,00	---

***Figure 104**: Analog scan as function of time*

- **Reference Spectra Substraction** is a measurement project to eliminate the influence of the environment on the analyzed sample. Measure the gas spectrum of the analytical space and store it as a reference spectrum. The spectrum of the sample is then determined in the same analytical space as the reference spectrum. The automatic subtsraction of the reference spectrum from the sample spectrum helps to obtain a real spectrum of components and fragments present in the sample (**Figure 105**).

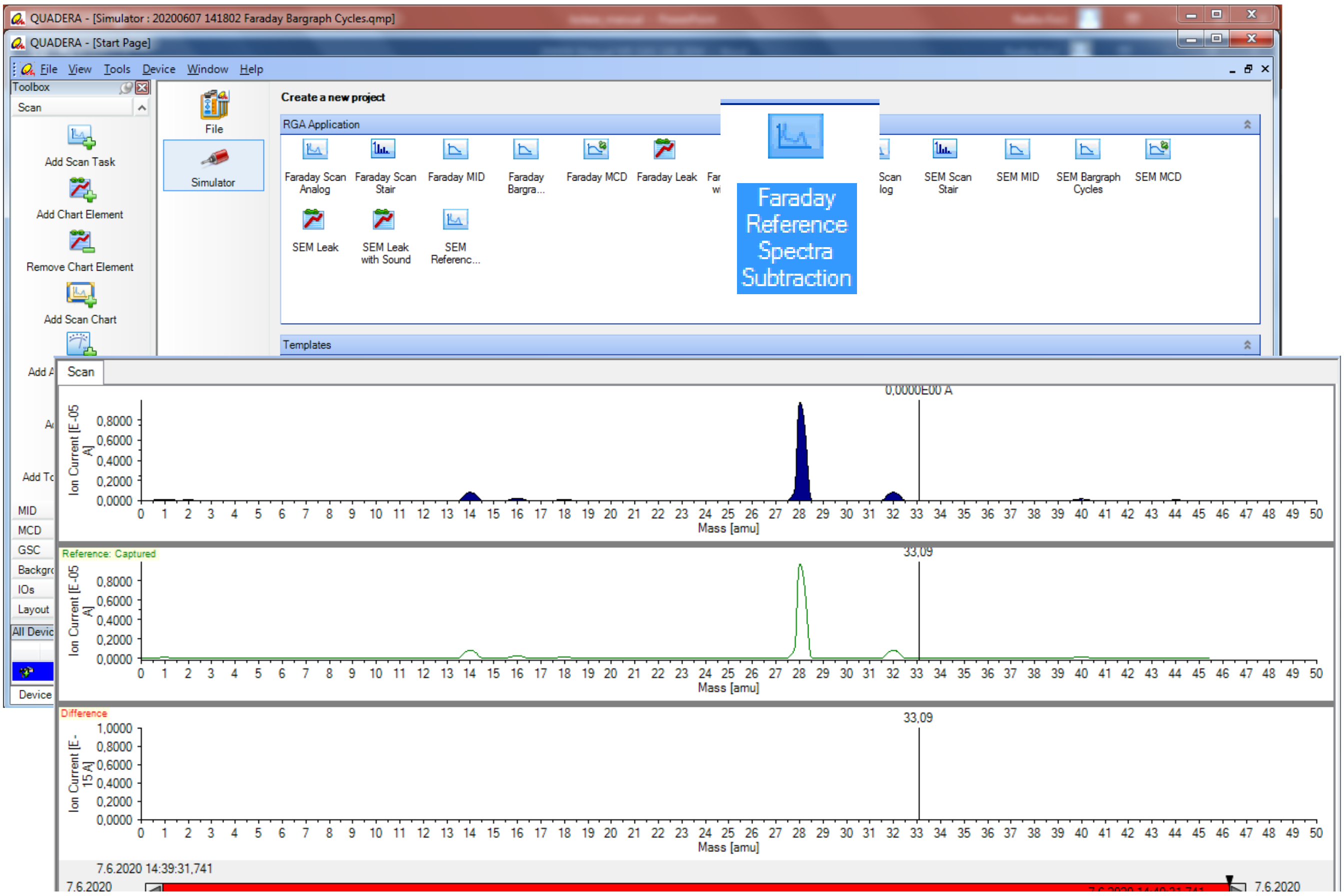


Figure 105: *In the Figure the reference spectrum is identical as the spectrum of the sample, the difference is zero*

- **Leak test** is a protocol for the monitoring of very small mass molecule - mostly helium. The measurement monitors the penetration of helium or, conversely, its escape into/from closed systems. The test is used if we need the product or its part to be as gas-tight as possible (**Figure 106**).

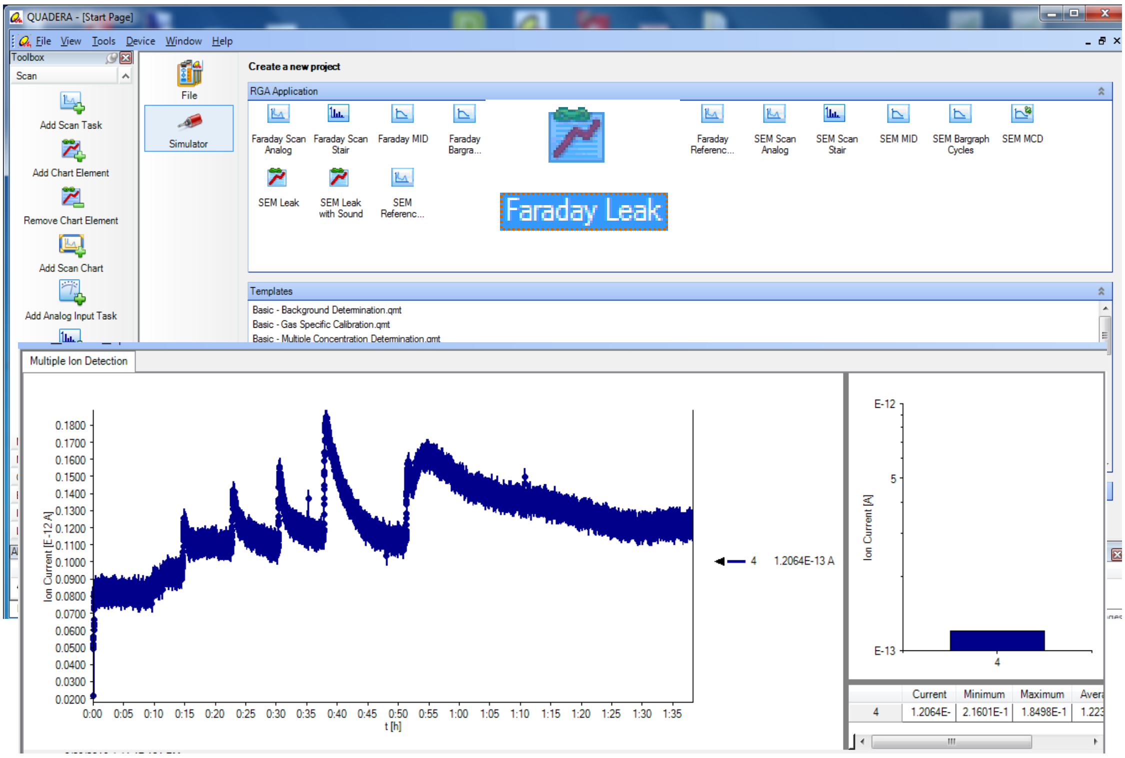
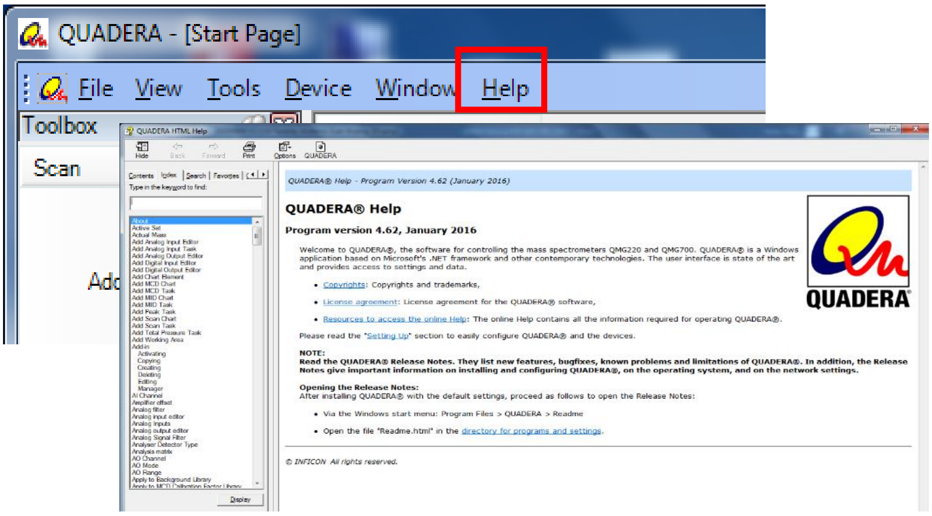


Figure 106: *Helium leak test*

6.2.7 QUADERA HELP

The software Quadera contains a large help section for more detailed information (**Figure 107**).



QUADERA® Help - Program Version 4.62 (January 2016)

QUADERA® Help

Program version 4.62, January 2016

Welcome to QUADERA®, the software for controlling the mass spectrometers QMG220 and QMG700. QUADERA® is a Windows application based on Microsoft's .NET framework and other contemporary technologies. The user interface is state of the art and provides access to settings and data.

- Copyrights: Copyrights and trademarks,
- License agreement: License agreement for the QUADERA® software,
- Resources to access the online Help: The online Help contains all the information required for operating QUADERA®.

Please read the "Setting Up" section to easily configure QUADERA® and the devices.

NOTE:
Read the QUADERA® Release Notes. They list new features, bugfixes, known problems and limitations of QUADERA®. In addition, the Release Notes give important information on installing and configuring QUADERA®, on the operating system, and on the network settings.

Opening the Release Notes:
After installing QUADERA® with the default settings, proceed as follows to open the Release Notes:

- Via the Windows start menu: Program Files > QUADERA > Readme
- Open the file "Readme.html" in the directory for programs and settings.

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***Figure 107**: Quadera help*

6.3 TURNING OFF THE DEVICE

Shutting down of the MS GAS-100 is based on a series of very important steps.

⚠	The sequence of the shut down steps must not be changed due to serious damage of the MS GAS-100 system.

After stopping the MS GAS-100 measurement, the SEM detector and the filament must be turned off by clicking on the component icons (**Chapter 6.2.3**). You can then close the Quadera software and disconnect the notebook from the device.

Switching off the device itself starts by switching off the button below the Touch Screen (**Figure 8**, point 4). It must be held for about 3 seconds. The touch screen switches off, but it takes about 2 minutes to switch off the Raspberry (RBI) system's backup batteries. The RBI system also controls all other device components, such as pumps, pressure gauges, valves inlcuding the venting valve, Mass Spec or Stirling cooler. After switching off the RBI system, all components are switched off also.

The pump system stops the evacuation of the device. The membrane pump is switched off immediately, while the turbo pump has a certain inertia. It is necessary to wait until the speed of the turbopump is reduced from 1500 Hz to approx. 700 Hz, so that the pump and thus the whole system start to be vented by an automatic valve. At the end of aeration, the pump usually emits a characteristic sound. After turbopump complete aeration flip the switch I/O on the rear side to position 0. The device is shut down.

⚠	You must wait until the turbopump have been completely aerated and without any sound.
ℹ	The process takes approx.5-10 min.

7 DEVICE SERVICE

Several MS GAS-100 components must be serviced at certain intervals in order for the device to operate properly. It is necessary to observe the following maintenance of the device:

- Replacement of the operating fluid reservoir with capillary rods in the **turbomolecular pump** every four years. Contact PSI service.
  Noise from the turbopump means serious damage to the bearings and then replacement is also recommended. Contact PSI service.

- Regeneration of the **diaphragm pump** - if the pump membranes are damp after long-term measurement with membrane probe in liquid sample, open the ballast valve and let it open for 10 hours (**Figure 32**). The device must be switched ON. The pump membranes are usually regenerated by this process.
  If the pump function is limited always, the membranes inside the pump must be replaced. Contact PSI service.

- Cleaning of the **filter in the Mass Spec electronic** head - to prevent overheating, clean the filtr every four years. Contact PSI service.
  If the electronics overheat, the Quadera software will display a warning. Contact PSI service.

- Replacement of damaged filaments – if one of filaments is damaged, switch on the second filament in Ion Source Tuning left table (**Chapter 6.2.5.1** , **Figure 81**). If both filaments are damaged (Quadera software displays the filament defect, **Figure 64**), contact PSI service.

- Replacement of the **filter unit** (**Figure 35** a) connected to the MS GAS-100 input via a VCR connection (VCR fitting instalation instruction **Figure 50**) is necessary when the ion current signal of the air compounds (or calibration gas) falls below the quantification limit. If the Mass Spec function is calibrated and without any problems, but the gas signals are low, the filter unit is contaminated.

⚠	The filter unit replacement must be performed when the MS GAS-100 is not under measurement mode due to filament and SEM damage and with the inlet valve closed for do not damage pumping system and Mass Spec..

- Replacement of the **RITTAL filter** if the impurities are visible on the filter surface. The Rittal filtr is located under Rittal fan cover on the rear side of the MS GAS-100 (**Figure 40**, point 7). Open the fan cover and replace the filtr with a new piece from the spare parts (**Figure 3**, point 11).

8 TECHNICAL SPECIFICATION

Measuring Principle:

Online measurements of gases, volatile organics and/or solvents

Mass analyzer:

Residual gas analyzer (RGA) PrismaPlus (Pfeiffer Vacuum, Asslar, Germany)

Mass range available: 1-100 amu

Ion source :

Open or closed version

Two independent filaments (material: yttriated iridium)

Electron impact ionization method

Detectors:

Response time < 20 seconds

Farraday cup ……………………………………………………………… sensitivity < 10 ppm

Continuous secondary electron multiplier (C-SEM) ……… sensitivity < 100 ppb

Vacuum system:

Turbomolecular pump HiPace 80 SplitFlow (Pfeiffer Vacuum, Asslar, Germany)

Diaphragm backing pump MVP 015 – 2 DC (Pfeiffer Vacuum, Asslar, Germany)

Inlet options:

Membrane probe (Polydimethylsiloxane, PDMS)

Needle Input

Heating system:

Thermostat heating element 2x100 W - maximal attainable temperature 90 °C

Cooling system:

Integrated cooler water freezing trap SC-UD08 - minimal attainable temperature -80 °C

Electronic control

Pressure sensors:

High vacuum pressure sensor MPT 200 Digital Pirani Cold cathode for measurement of total pressure in mass spec chamber

Inlet pressure sensor PPT AR 200 Digital Pirani for protection of mass spectrometer

Raspberry (RBI) mini-computer with Touch Screen:

System control and actual readings

BIOS:

Upgradeable firmware

Communication port:

Ethernet TCP/IP intervals

External PC:

Notebook with software Quadera 4.62 for Mass Spec tuning and acquisition of measured data

Dimensions:

54.5 x 72 x 45.5 cm

Total weight:

aprox. 70 kg

Electrical:

110 - 230 V AC

9 WARRANTY TERMS AND CONDITIONS

- This Limited Warranty applies only to the MS GAS -100, MS GAS-200 and MS GAS-300. It is valid for one year from the date of shipment.
- If at any time within this warranty period the instrument does not work as warranted, return it and the manufacturer will repair or replace it with no charge. The customer is responsible for shipping and insurance charges (for the full product value) to PSI. The manufacturer is responsible for shipping and insurance on return of the instrument to the customer.
- No warranty will apply to any instrument that has been (i) modified, altered, or repaired by persons unauthorized by the manufacturer; (ii) subjected to misuse, negligence, or accident; (iii) connected, installed, adjusted, or used otherwise than in accordance with the instructions supplied by the manufacturer.
- The warranty is return-to-base only, and does not include on-site repair charges such as labor, travel, or other expenses associated with the repair or installation of replacement parts at the customer's site.
- The manufacturer repairs or replaces faulty instruments as quickly as possible.
- The manufacturer will keep spare parts or their adequate substitutes for a period of at least five years.
- Returned instruments must be packaged sufficiently so as not to assume any transit damage. If damage is caused due to insufficient packaging, the instrument will be treated as an out-of-warranty repair and charged as such.
- PSI also offers out-of-warranty repairs. These are usually returned to the customer on a cash-on-delivery basis.
- *Wear & Tear Items* (such as sealing, tubing, padding, etc.) are excluded from this warranty. The term *Wear & Tear* denotes the damage that naturally and inevitably occurs as a result of normal use or aging even when an item is used competently and with care and proper maintenance.

10 TROUBLESHOOTING AND CUSTOMER SUPPORT

In case of troubles and for customer support, please, write to **support@psi.cz** or contact your local distributor.