

SOLIA 990 MicroGC

User manual



Dear user,

Thank you for choosing this SRA Instruments product.

This manual contains all the necessary information for the correct use of your instrument. Should you need further information or if you encounter any problems, please contact our After Sales Service:

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1. Introduction

For reasons of clarity, this manual does not contain all detailed information on all types of coupling. In addition, it cannot describe every possible case concerning installation, use and maintenance.

If you require additional information about this device or if you encounter problems that are not addressed in this manual, you can contact SRA Instruments for assistance.

The content of this manual is not part of any previous or existing agreement, commitment or legal status and does not change these. All the commitments of SRA Instruments are contained in the respective sales contracts, which also contain the only and entire applicable warranty terms. These warranty conditions in the contract are neither extended nor limited by the content of this manual.

2. Safety instructions

Important information

This instrument has been designed for chromatographic analysis of appropriately prepared samples. It must operate using appropriate gases and solvents and within specified maximum ranges for pressure, flows, and temperatures as described in this manual. If the equipment is used in a manner not specified by SRA Instruments, the protection provided by the equipment may be impaired.

Moreover, it is your responsibility to inform SRA Instruments after-sales service if the SOLIA has been used for the analysis of hazardous samples, prior to any instrument service being performed or when an instrument is being returned for repair.

2.1 For your protection

Warnings :

Warning: Shock hazard



Do not replace components while the power cable is plugged in. To avoid injuries, always turn off power before touching them. Install the SOLIA so that access to the power cable is easy. Make sure that you connect the cable to an earth socket, otherwise there is a lethal hazard.

Warning: Hot surfaces



Several parts of the SOLIA work at temperatures high enough to cause severe burns.

These parts include, among others:

- The inlet tube to modules
- The heated membrane filter
- The inlet module
- The column nuts attaching the module to an inlet or a vent

You must be extremely careful to avoid touching these heated surfaces. Columns temperature can be maintained as high as 180 °C. Do not use the instrument if the MicroGC module is disassembled.

Warning: Electrostatic discharge is a threat to electronics



Electrostatic discharge (ESD) can damage the printed circuit boards of the SOLIA. If you must hold an electronic card, wear a grounded wrist strap, and hold it only by its edges.

Warning: Use of gases



Do not use gases that can form an explosive mixture. Avoid using hydrogen as carrier gas or purge gas for your analyses.

Warning concerning the use of hydrogen

When using hydrogen (H₂) as the carrier gas, be aware that hydrogen gas can create a fire or explosion hazard. Be sure that the supply is turned off until all connections are made.

Hydrogen is highly flammable. Leaks, when confined in an enclosed space, may create a fire or explosion hazard.

In any application using hydrogen, leak test all connections, lines, and valves before operating the instrument. Always turn off the hydrogen supply at its source before working on the instrument.

- Hydrogen is combustible over a wide range of concentrations.
At atmospheric pressure, hydrogen is combustible at concentrations from 4 % to 74.2 % by volume.
- Hydrogen has the highest burning velocity of any gas.
- Hydrogen has a very low ignition energy.
- Hydrogen that can expand rapidly from high pressure into the atmosphere can self-ignite.
- Hydrogen burns with a nonluminous flame which can be invisible under bright light.

Warnings related to chemical products

When handling or using chemicals for preparation or use within the MicroGC, all applicable local and national laboratory safety practices must be followed. This includes, but is not limited to, correct use of Personal Protective Equipment, correct use of storage vials, and correct handling of chemicals, as defined in the laboratory's internal safety analysis and standard operating procedures. Failure to adhere to laboratory safety practices could lead to injury or death.

2.2 Safety and regulatory information

This instrument and its accompanying documentation comply with the CE specifications and the safety requirements for electrical equipment for measurement, control, and laboratory use.

This device has been tested and found to comply with the limits required by regulations. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy. If it is not installed and used in accordance with the user manual, it may cause harmful interference to radio communications.

NOTICE: This instrument has been tested per applicable requirements of EMC Directive as required to carry the European Union CE Mark. As such, this equipment may be susceptible to radiation/interference levels or frequencies, which are not within the tested limits.



This symbol confirms that the SOLIA complies with all electrical safety regulations.

2.3 General safety precautions

Follow the following safety practices to ensure safe equipment operation:

- Perform periodic leak checks on all supply lines and pneumatic plumbing.
- Do not allow gas lines to become kinked or punctured.
Place lines away from foot traffic and extreme heat or cold.
- Avoid exposure to potentially dangerous voltages. Disconnect the instrument from all power sources before removing protective panels.
- When it is necessary to use a non-original power cord and plug, make sure the replacement cord adheres to the color coding and polarity described in the manual and all local building safety codes.
- Replace faulty or frayed power cords immediately with the same type and rating.
- Place this instrument in a location with enough ventilation to remove gases and vapors. Make sure there is enough space around the instrument so that it can cool sufficiently.
- Before plugging the instrument in or turning the power on, always make sure that the voltage and fuses are set appropriately for your local power source.
- Do not turn on the instrument if there is a possibility of any kind of electrical damage. Instead, disconnect the power cord and contact SRA Instruments.
- The supplied power cord must be inserted into a power outlet with a protective ground connection. When using an extension cord, make sure that the cord is also properly grounded.
- Do not change any external or internal grounding connections, as this could endanger you or damage the instrument.
- The instrument is properly grounded when shipped. You do not need to make any changes to the electrical connections or to the instrument chassis to ensure safe operation.
- When working with this instrument, follow the regulations for Good Laboratory Practices (GLP). Take care to wear safety glasses and appropriate clothing.
- Do not place containers with flammable liquids on this instrument. Spilling liquid over hot parts may cause fire.
- This instrument may use flammable or explosive gases, such as hydrogen gas under pressure. Before operating the instrument be sure to be familiar with and to follow accurately the operation procedures prescribed for those gases.
- Never try to repair or replace any component that is not described in this manual without the assistance of SRA Instruments. Unauthorized repairs or modifications will result in rejection of warranty claims.
- Always disconnect the AC power cord before attempting any type of maintenance.
- Use the proper tools when working on the instrument to avoid endangering yourself or damaging the instrument.
- Do not attempt to replace the battery or a fuse in this instrument with parts not specified in the manual.
- Damage can result if the instrument is stored under unfavorable conditions for prolonged periods. (For example, damage will occur if stored while subject to heat, water, or other conditions exceeding the allowable operating conditions).
- Do not shut off column flow when the oven temperature is high, since this may damage the column.
- This instrument has been designed and tested in accordance with recognized safety standards and designed for use indoors.

- If the instrument is used in a manner not specified by the manufacturer, the protection provided by the instrument may be impaired.
- Substituting parts or performing any unauthorized modification to the instrument may result in a safety hazard.
- Changes or modifications not expressly approved by the responsible party for compliance could void the user's authority to operate the equipment.

2.4 To begin with

- Check that the operating voltage of the instrument is compatible with the one of your electrical network before switching it on. Otherwise the device may be damaged.
- Use only gases and solvents specified in the operating procedures.
- Do not open the instrument without the permission of SRA Instruments.
- Eliminate from the environment of the instrument: vibrations, magnetic effects, and explosive gases.
- The SOLIA must only be used indoors; it is designed for use at room temperature and under conditions where no condensation can occur. Install the SOLIA on a rigid and stable surface.
- Have your instrument serviced by SRA Instruments.

3. Transport, cleaning, and disposal of the instrument

3.1 Shipping instructions

If your SOLIA must be shipped for any reason, it is very important to follow these additional shipping preparation instructions:

- Place all the vent caps on the back of the instrument.
- Always include the power supply.
- Include, if used and if possible, the inlet filter(s).

3.2 Cleaning

To clean the surface of the SOLIA :

1. Switch off the instrument.
2. Remove the power cord.
3. Put protection plugs on the sample inlet and other gas connections.
4. Use a soft brush (not hard or abrasive) to carefully brush away all dust and dirt.
5. Use a soft, clean cloth dampened with mild detergent to clean the outside of the instrument.
 - Never clean the inside of the instrument.
 - Never use alcohol or thinners to clean the instrument; these chemicals can damage the case.
 - Be careful not to get water on the electronic components.
 - Do not use compressed air to clean the instrument.

3.3 Instrument disposal

When the SOLIA or its parts have reached the end of their useful life, dispose of them in accordance with the environmental regulations that are applicable in your country.

4. Instrument overview

4.1 Presentation

The SOLIA is a modular instrument in which are associated to the maximum 4 analytical channels of 990 MicroGC. Each module can analyze several compounds that are detected by a universal non-destructive detector.

This analyzer is controlled by the Soprane II software: see Appendix I.

Coupling with a Mass Spectrometer is possible thanks to a dedicated heated interface which allows the association of the 2 detectors in series, without loss of performance.



MSD Heated interface

SOLIA 990 MicroGC

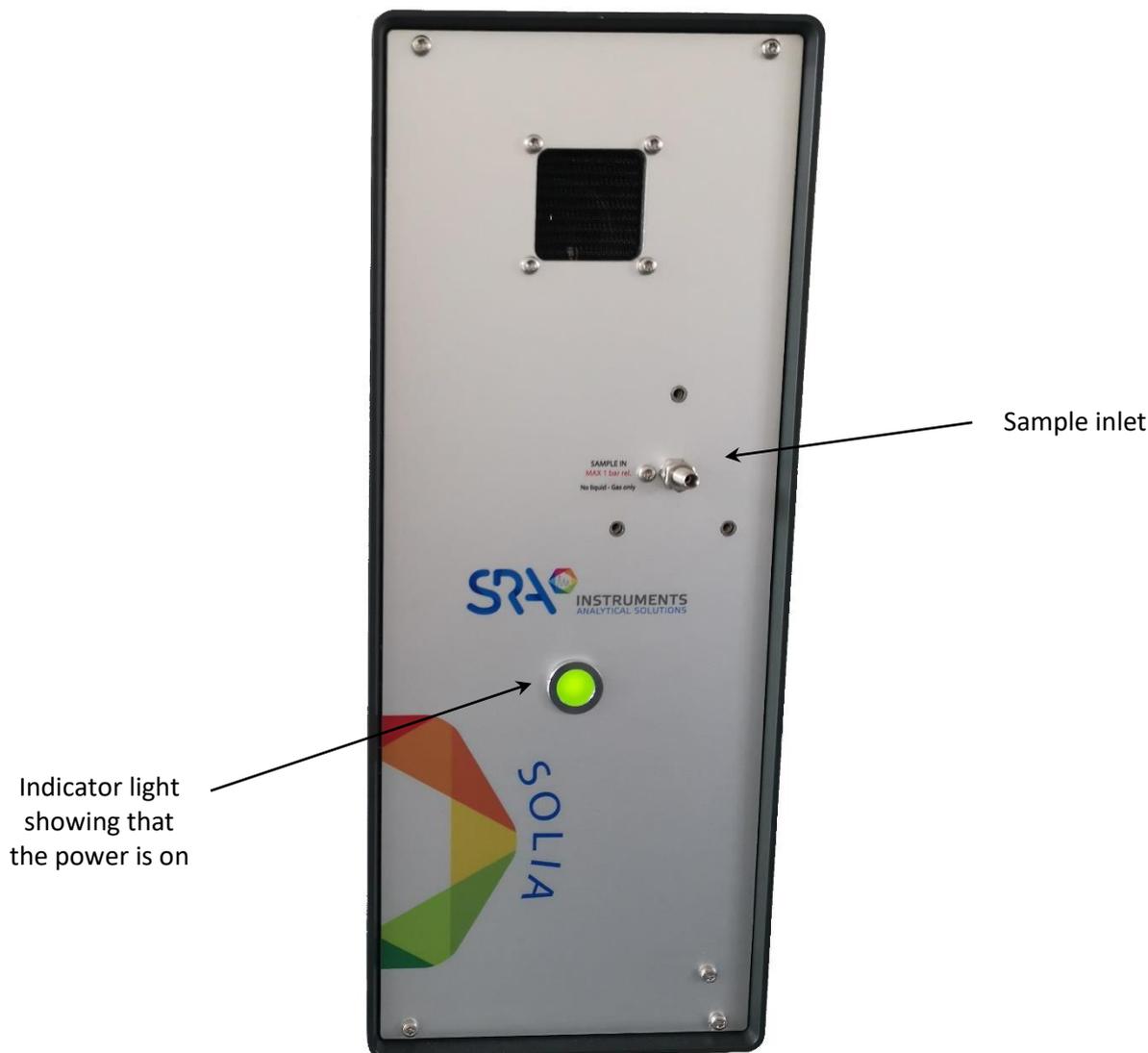
4.2 Operating principle

The SOLIA can be equipped with 1 to 4 independent column channels. Each column channel is a miniaturized and complete GC, including:

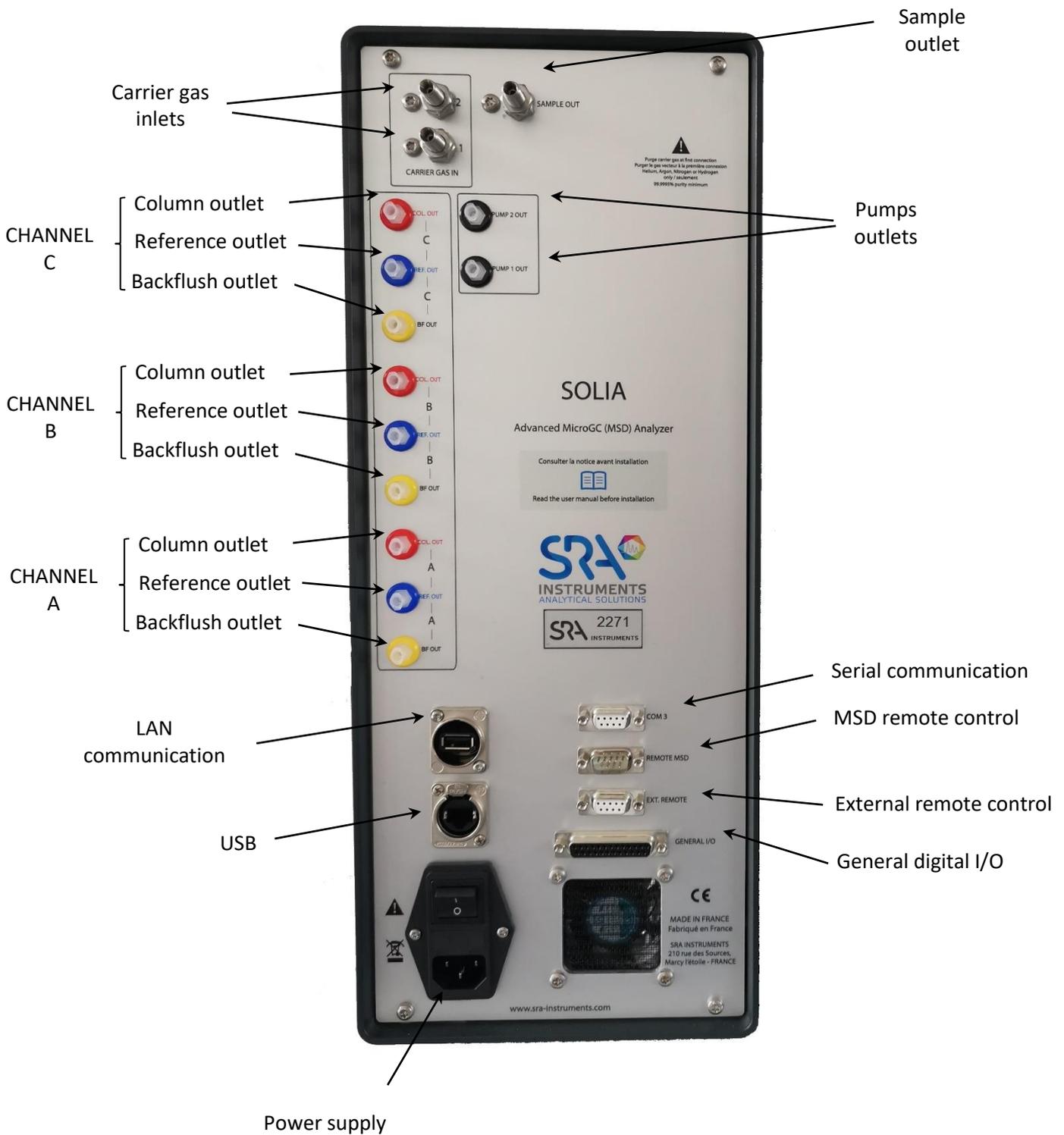
- A micro-machined injector
 - An analytical column of small diameter
 - A micro catharometer (μ TCD)
 - An electronic gas control
- } Module

The analytical channels of the SOLIA can be optionally equipped with a backflush (see chapter 9.5). This has the advantage of protecting the stationary phase of the column from moisture and carbon dioxide. In addition, the analysis times are reduced since late elution compounds, which are therefore of no interest, do not enter the analytical column.

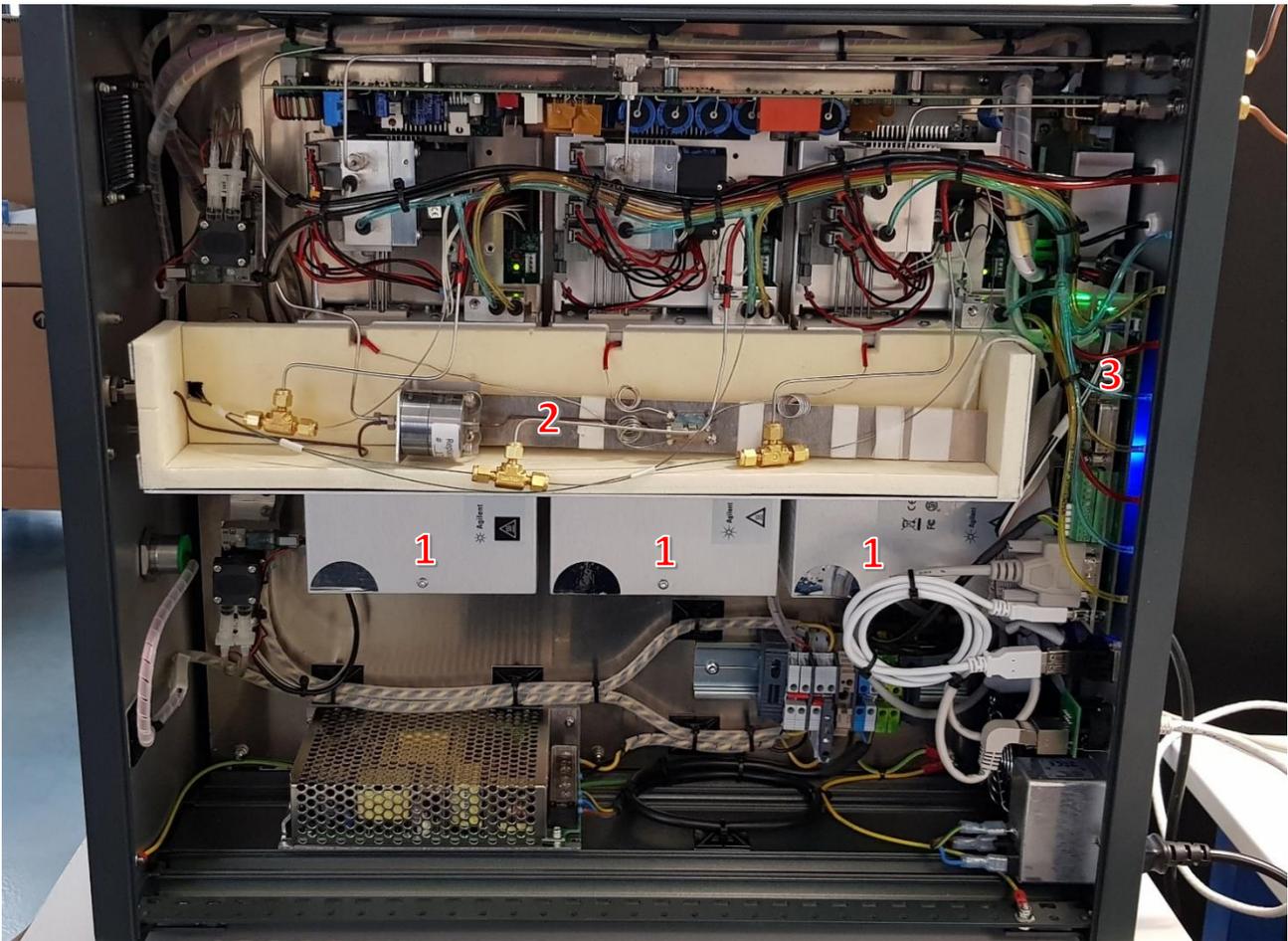
4.3 Front view



4.4 Back view



4.5 Inside view



1: Analytical modules

2: Heated sample distribution manifold

3: MicroGC main board

4.6 Power supply

100-240 Vac ; 50-60 Hz ; 6,3 At

! Only use the power supply provided with your instrument.

4.7 Ambient pressure

The MicroGC automatically shuts down if the ambient pressure is greater than 120 kPa.

4.8 Ambient temperature

The MicroGC automatically shuts down if the ambient temperature exceeds 65 °C.

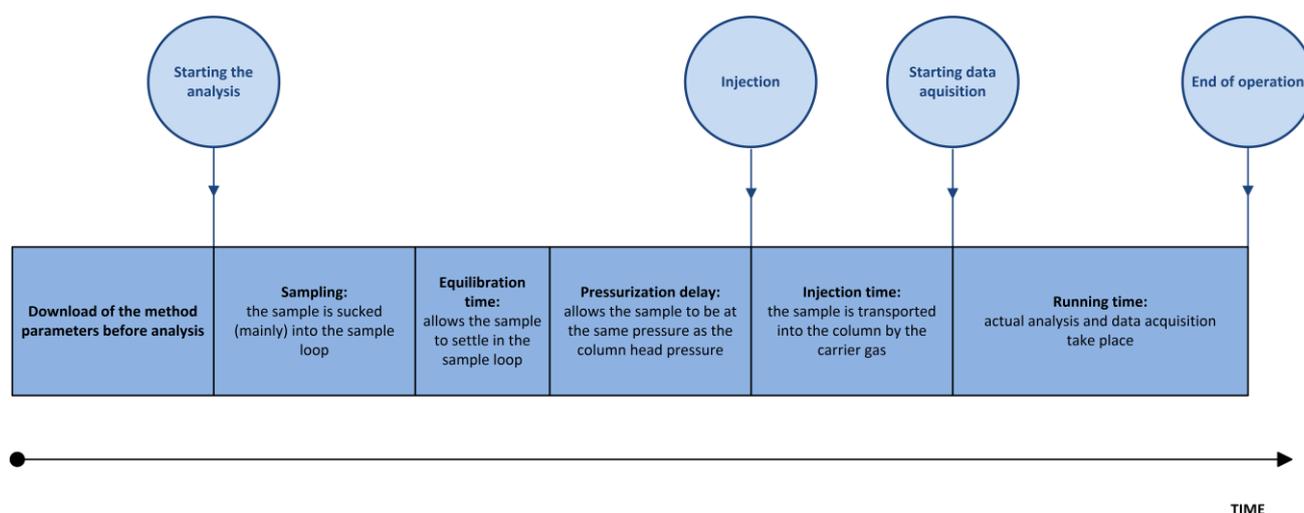
4.9 Maximum operation altitude

Certified up to 2000 meters above sea level.

4.10 MicroGC cycle with constant pressure

The timing diagram below provides an overview of cycle of the MicroGC at constant pressure.

This description is only for one channel. In most cases a dual-channel system is used. When a dual-channel system is used, the sequence is the same, but the timing settings can differ. If the sampling time on channel A and channel B is different, the longest time will be used. The run time can be specified per channel; the data acquisition stops for each channel as soon as the analysis time has elapsed. The total analysis time depends on the longest run time.



5. Advice before installing the instrument

5.1 Pre-installation requirements

Prepare the installation site as described in the installation prerequisites manual, including recommended gas cleaning filters.

5.2 Inspect the shipping packages

The SOLIA will arrive in one large box and one or more smaller cartons. Inspect the cartons carefully for damage or signs of rough handling. Report damage to the carrier and to SRA Instruments.

5.3 Unpack the MicroGC

Unpack the SOLIA and accessories carefully and transfer them to the work area using proper handling techniques. Inspect the instrument and accessories carefully, report any damage to the carrier and SRA Instruments as soon as possible.

Warning: Avoid back strain or injury by following all safety precautions when lifting heavy objects.

! The gas inputs/outputs of the instrument could be protected during transport by protective caps. Before use, remove these caps.

5.4 Provide the necessary tools and accessories for installation

5.4.1 Tools

- 1/8" copper or stainless-steel tubing for carrier gas connection
- 1/8" Swagelok nuts, and ferrules
- 1/16" stainless-steel tubing for sample connection
- 1/16" Swagelok nuts, and front and back ferrules
- Two 7/16" wrenches
- 5/16" wrench
- 9/16" wrench
- 1/4" wrench
- Torx T-20 screwdriver

5.4.2 Accessories

Electronic leak detector (optional).

Note: Do not use a liquid leak detector: liquid can contaminate the analyzer.

5.5 Recommendations before installation

5.5.1 Venting

To optimize the performance of the analyzer and increase its service life, allow sufficient air circulation around the analyzer to dissipate heat and eliminate the releases of potentially toxic, harmful, or flammable carrier gases or samples. If necessary, toxic emissions can be trapped.

Avoid discharging gaseous effluents into an area that may experience pressure fluctuations (wind or releases with variable temperature). Changes in pressure can affect the stability of the baseline and the sensitivity of the analyzer. For releases outside atmospheric pressure (e. g. glove boxes) please contact SRA Instruments to find a suitable solution.

5.5.2 Tubes

- The diameter of the tubes depends on the distance between the gas cylinder and the analyzer and the total flow rate required. The use of 1/8" tube is correct for a line length of less than 5 m. Beyond that, or when several analyzers are connected to the same gas inlet, the use of 1/4" tube is preferable.
- Do not use plastic tubes as the air diffused into the tubes could make the baselines noisy and reduce sensitivity. Metal tubes must be cleaned for MicroGC use. Buy chromatographically or flame-cleaned tubes.
- Do not use sealings: they may contain volatile materials that may contaminate the distribution system.

5.5.3 Optimization of gas purity

To get the best carrier gas quality on your analyzer:

- Use a pressure reducer adapted to your needs.
- Use suitable tubes and ferrules.
- Purge correctly dead volumes before connecting the tube to your analyzer.
- Confirm the absence of leaks with an electronic detector.
- Always send to MicroGC a purge method (with TCD OFF) to purge the dead volumes of the analyzer and column before setting the detector ON.

5.5.4 Carrier gases

The performance of analyses requires the use of a continuous flow of carrier gas at a constant flow rate. SRA instruments recommends the use of "instrument" or "chromatography" grade gases specifically intended for this use.

The SOLIA can be used with helium, nitrogen, argon, and hydrogen.

Specifications concerning the carrier gas:

- Pressure: 550 kPa \pm 10 % (80 psi \pm 10 %)
- Purity: 99.9996 % minimum ; 99.9999 % for trace analysis
- Dehydrated and particle-free

Gas Clean filters are recommended to remove all traces of moisture and oxygen.

Gas Clean filters are filled with nitrogen. If you do not use nitrogen as a carrier gas, flush filters, and gas lines after installing a new filter.

The type of analysis you want to perform will guide you on the type of carrier gas to use. The difference between the relative thermal conductivity of the carrier gas and the species to be analyzed should be as high as possible.

See table below for the different relative thermal conductivities.

Carrier gas	Relative thermal conductivity	Carrier gas	Relative thermal conductivity
Hydrogen	47.1	Ethane	5.8
Helium	37.6	Propane	4.8
Methane	8.9	Argon	4.6
Oxygen	6.8	Carbon dioxide	4.4
Nitrogen	6.6	Butane	4.3
Carbon monoxide	6.4		

5.5.5 Vents

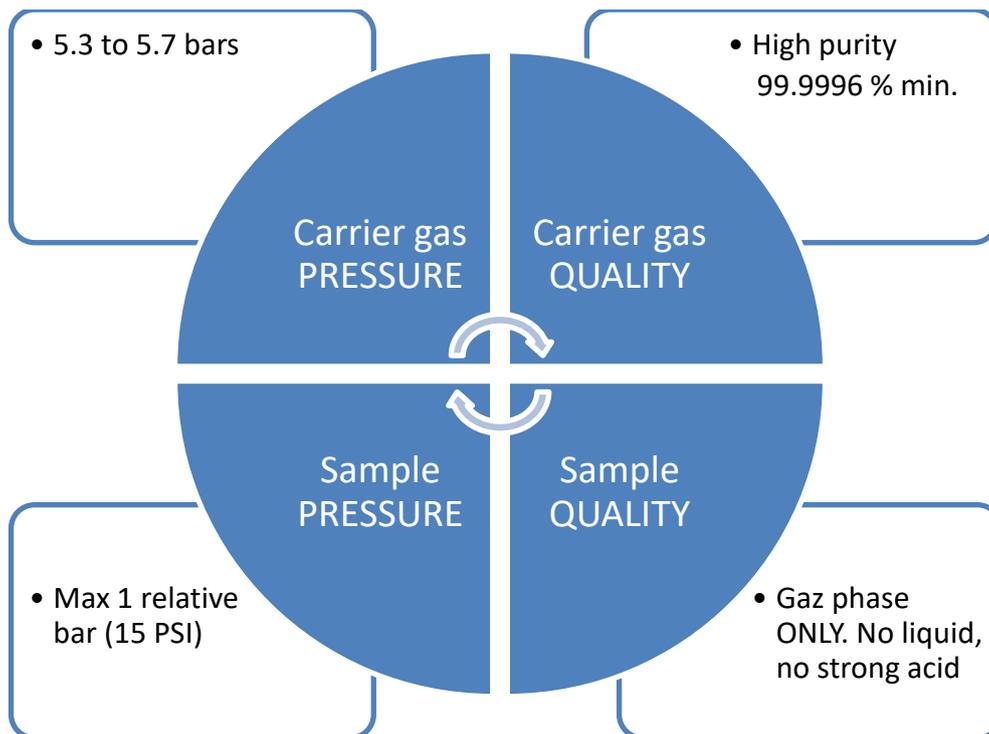
The connection of the vents is also a critical parameter not to be neglected for the analyzer. Keep the outputs at a constant (almost) atmospheric pressure to avoid "peaks" on the TCD signal. Grounding the same vent lines when they are metallic is also recommended.

5.6 The 4 golden rules

MicroGC technology is easy to use. No chemical or analytical knowledge is required for basic use and setup. However, as with any analysis instrument, there are important rules to follow to protect your instrument and its functionality.

These rules can be presented as "the 4 golden rules":

- Carrier gas pressure
- Carrier gas quality
- Sample pressure
- Sample quality



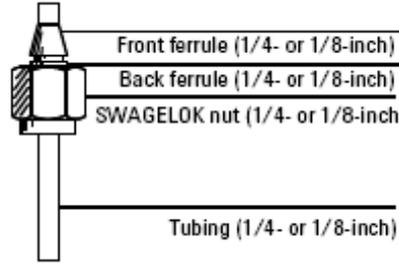
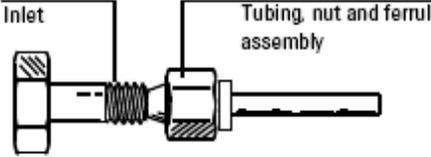
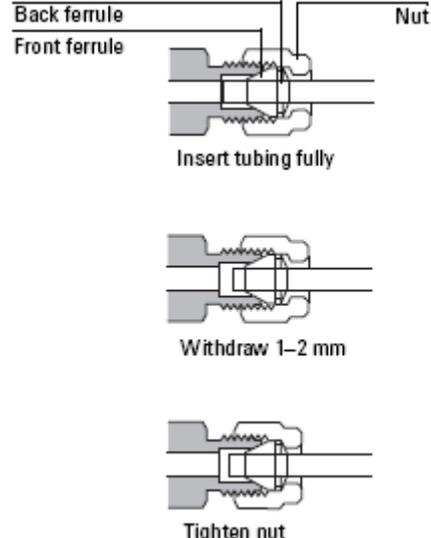
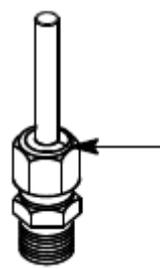
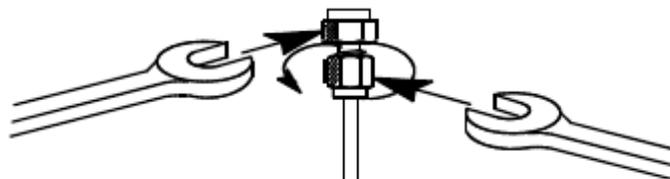
Not respecting these rules highly increases the risk of damaging your instrument. All standard procedures for using MicroGC are derived from these 4 golden rules: the quality of the carrier gas will require a tube purge to ensure this level of quality.

5.7 Swagelok fittings

Pneumatic connections use Swagelok fittings. If you are not familiar with this type of fitting, read the procedure below.

Equipment required:

- 1/8" preconditioned copper tube
- 1/8" Swagelok nut and ferrules
- Two 7/16" wrenches

<p>Insert the 1/8" nut and the 2 ferrules.</p>	
<p>Make sure the ferrule is in contact with the inlet, then secure the nut by hand.</p>	
<p>Insert the tube as far as possible into the female part, then remove it from 1 to 2 mm.</p>	
<p>Mark the position of the nut with a pencil.</p>	
<p>Use two 7/16" wrenches to tighten the nut. Tightening requires 3/4 turn with a 1/8" nut.</p>	

6. Gas samples handling

6.1 Sampling

WARNING

The sample must be clean and dry. Although the internal filter removes many particulate contaminants, samples containing aerosols, excessive amounts of particulate matter, high water concentrations and other contaminants can damage your instrument. The presence of acids (HF, HCl, H₂SO₄ and HNO₃) is prohibited.

The inlet pressure of the sample must be less than 1 relative bar and its temperature must not exceed 100 °C.

6.1.1 Introduction

For a good representation of the sample or standard analyses, the volume of the lines to be scanned up to the analyzer input should be as small as possible ideally. This means that the sample conditioning as well as the selection valve, if present, must be in the immediate proximity of the analyzer. The sample flow rate flowing through the analyzer is also a critical parameter for a good representativity of the analyses, it must be at least 20 mL/min.

As an example, and considering the two criteria above, a sample will take about 1 minute to cover 10 meters of standard 1/8" O.D. stainless steel tube at a flow rate of 25 mL/min.

6.1.2 Sampling modes

You will need suitable mounting equipment to connect the sample to the MicroGC or an accessory.

Sampling and conditioning are essential points for good analysis and correct results. It is important to study this part as well as possible.

Sample with pressure greater than one atmosphere

Samples with a pressure between 0 and 1 bar rel. can be connected directly to the MicroGC. Above 1 bar rel. it is necessary to use a suitable pressure reduction system between the sample and the MicroGC.

Sample available at atmospheric pressure

In this case, the MicroGC suction pumps will allow the sample to circulate for an adjustable time in the loops of the injectors of the analytical modules before injection. Here are some examples of samples at atmospheric pressure:

- Atmospheric air: (ex. Online control of atmospheric pollution)
- Tedlar bag: you can adapt a syringe needle on the analyzer sample inlet, the exiting needle will be put in the septum of the bag which will be presented.
- Container with septum: same possibilities as with the Tedlar bag, but in this case, you can only perform several injections, because after the pressure will decrease.

6.2 Heated sample lines

A heated sample line is always combined with a heated injector. A heated injector with a sample line is optional on one channel and is used to prevent condensation in the sample lines when analyzing condensable samples. Whenever possible, remove moisture from samples introduced into the MicroGC.

The heated sample and injector can be controlled between 30 °C and 110 °C.

6.3 How to connect your sample to the MicroGC

Warning: The metal surfaces of the sample line heater can be very hot. Before connecting a sample line, allow the sample line heater to cool down to ambient temperature.

Front inlet

Connect the sample line to the heated sample inlet on the front of the MicroGC using 1/8-inch female Swagelok fittings.



! Isolate the sample line connected to the MicroGC to avoid damaging the communication cables.

7. Installation

This chapter describes how to install the instrument.

7.1 MicroGC installation

If you are installing the SOLIA MicroGC for the first time, follow the steps as described below. If you are performing a re-installation, see "Long Storage Recovery Procedure" in section 8.4.

7.1.1 Step 1 : Connect the carrier gas

Install gas regulators and set pressures

Carrier gas cylinders should have a two-stage pressure regulator to adjust the carrier gas pressure to 550 kPa \pm 10 % (80 psi \pm 10 %). Set cylinder regulator pressure to match the gas inlet pressure.

Connect carrier gas to the MicroGC

The MicroGC supports the use of helium, nitrogen, argon, and hydrogen. The recommended purity for carrier gas is 99.9996 % minimum. The carrier gas line is connected to the MicroGC via the **CARRIER GAS IN 1** or **CARRIER GAS IN 2** ports on the back panel (see section 4.4). Connect the carrier gas to the desired connector and open the gas flow.

The use of helium as carrier gas with the MicroGC configured for Ar/N₂ will decrease the sensitivity of the detector (about 10 times), invert the peaks, with no further impact.

 **The use of argon as carrier gas with the MicroGC configured for helium will destroy the TCD filaments.**

7.1.2 Step 2 : Install power supply

Connect the power connector to the MicroGC, and then plug the power cord into an appropriate power source. See section 4.6.

Your MicroGC is shipped from the factory with default settings. The following information shows the factory default parameters and settings:

- When the MicroGC is turned on, the system begins the flush cycle procedure. The flush cycle is a 2-minute cycle in which the various valves are activated or deactivated to flush entrapped air from the manifold, valves, and tubing.
- Once the flush cycle is completed, the process that was the last active before the instrument was switched off is activated:
 - All heated zones are set at 30 °C.
 - The detector filaments are set to OFF.

7.1.3 Step 3 : Connect to computer or local network

The SOLIA requires a connection to a computer on which a chromatography software is installed. This connection uses a TCP/IP protocol with Ethernet.

7.1.4 Step 4 : Install the Chromatographic Data System

For further instructions about installation of the chromatographic data system, see the corresponding installation manual.

7.1.5 Step 5 : Assign IP address (optional)

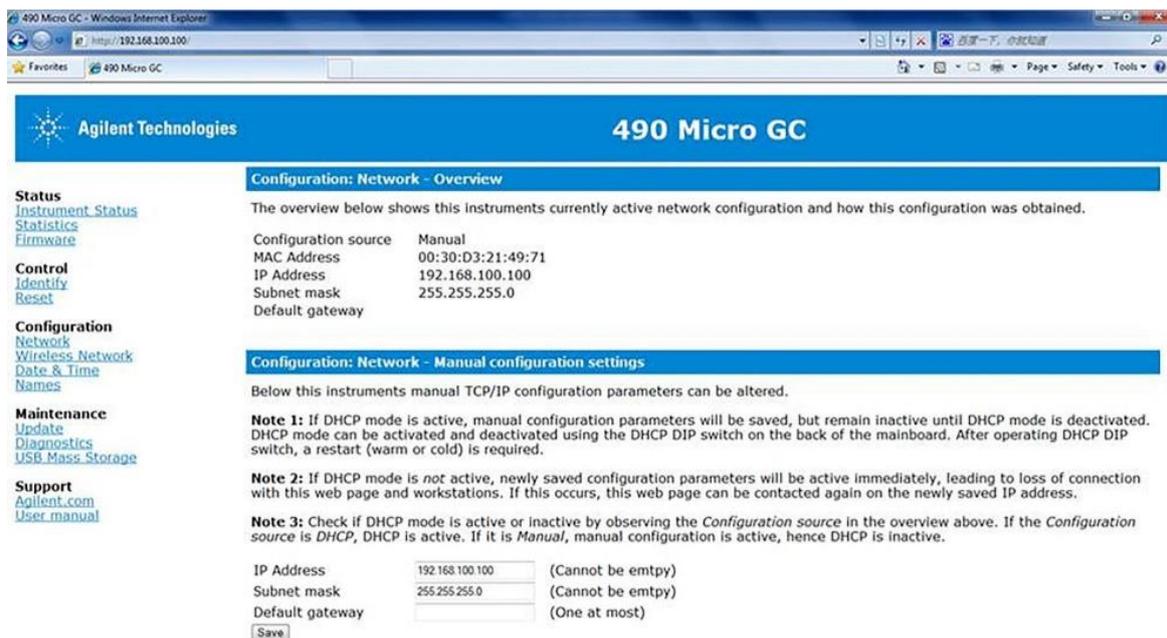
Upon arrival from SRA Instruments, the MicroGC has a static default IP address configured. The active IP address is specified in the table below. The MAC address and serial number of the main board are specified on a sticker on the main board.

Default IP address	10.1.1.101 (or 192.168.100.100 in some cases)
Subnet mask	255.255.255.0
Host name	microgc
Default Gateway	N/A (not used)

1. Change the IP address of your laptop or PC to an address in the same range as the current IP address as the MicroGC.
2. Start up the web browser.
3. Connect to the MicroGC’s website. Type the IP address of the MicroGC in the address field of the web browser.
4. On the web page, click **Network**.
5. Log in as administrator. Use the factory default login and password:
 - Login name: admin
 - Password: agilent



6. In the network webpage, the upper section shows the current IP configuration. Type the **IP Address**, **Subnet mask**, and **Gateway** you want to assign to the MicroGC in the corresponding fields.



7. Click **Save** to save the IP configuration.
8. This IP address is now the active IP address. Communication with the MicroGC will be lost, since the active IP address has changed.
9. Change the IP address of your laptop or PC to an address in the same range as the new IP address of the MicroGC.
10. To reestablish communication, type the new IP address in the web browser address bar.

7.1.6 Step 6 : Complete the configuration of the MicroGC in the chromatography software

If this is not already the case, complete the additional configuration of the MicroGC in the chromatography software used. Ensure that the types of carrier gases correspond to the gas supplied to the MicroGC.

7.2 Restore the factory default IP address

At the factory, the MicroGC (with G3581-65000 main board) is configured with a default static IP address (see table below for settings).

Default IP address	192.168.100.100
Subnet mask	255.255.255.0
Host name	microgc
Default Gateway	N/A (not used)

A reset button on the main board allows you to restore these default IP settings, if necessary. When the IP address settings are not known, this functionality can be used to reconnect the instrument and modify the custom IP settings.



The reset button can be accessed on the main board (see pictures above). To restore the factory default IP address, follow this procedure:

1. Power off the MicroGC.
2. Press and hold the reset button and power on the MicroGC.
3. Release the reset button shortly after powering on the MicroGC (approximately 3 seconds).

Note 1 : When the reset button is released too quickly (less than 1 second), it may result in the IP setting not reverting to its factory defaults.

Note 2 : Holding the reset button too long (more than 10 seconds), will result in an instrument reboot, without restoring the default IP settings.

4. The default IP address is now restored.

8. Use

8.1 Create the test method

At first startup, perform a checkout to make sure the MicroGC is functioning properly.

A test method for each standard column type has been provided in the sections listed in the table below.

! If you ordered a Molsieve column, make sure it is conditioned before use. See Appendix II, section 15.1 for the parameters.

Column type	Table
Molsieve 5A	Table 1 on page 57
CP-Sil 5 CB	Table 2 on page 58
CP Sil 13 CB or 19 CB	Table 3 on page 59
PoraPlot U or Q 10 m	Table 4 on page 60
Hayesep A 40 cm	Table 5 on page 61
COx 1 m and Al ₂ O ₃ /KCl	Table 6 on page 62
MES(NGA) and CP-WAX 52 CB	Table 7 on page 63

Use the chromatography software to define the verification parameters for each MicroGC channel. Apply the verification procedure parameters to the MicroGC and allow the instrument to stabilize at initial operating conditions. Monitor the status of the instrument using the software status display (refer to the corresponding manual for more details)

Each test method has been designed to determine if the instrument channel is functioning properly and includes an example test chromatogram.

8.2 Perform a series of runs

1. Create a short sequence of at least three runs using a test sample and an analysis method.
2. Launch the sequence.
3. After a first run, the results for each channel should become similar to the example chromatograms.

8.3 Shut down the instrument

! The detector can be damaged by improper shut down. If shutting down the instrument for more than a few days, carry out the procedure below.

1. Create a method for all channels with these settings:
 - Filaments switched OFF.
 - Column temperature set at 30 °C.
 - Injector temperature set at 30 °C.
 - Pressure set at 50 kPa.
2. Apply the method to the MicroGC.
3. Wait until the temperature of the columns and injectors are < 50 °C (to protect the column), then switch off the MicroGC.
4. Remove the carrier gas tubing and plug all the vents and carrier gas connections with 1/8-inch brass nuts or plastic caps.

Before using the instrument again, perform the "Long Storage Recovery Procedure" described in the following chapter.

8.4 Long storage recovery procedure

Follow this recovery procedure if your MicroGC has been stored for a long period of time.

1. Remove the 1/8-inch brass nuts and plastic caps from all of the vents and carrier gas connections.
2. Connect the carrier gas tubing and apply pressure to the MicroGC.
3. Wait at least 10 minutes before switching ON the MicroGC.
4. Immediately check if the detector filaments are switched OFF. Switch OFF if necessary.
5. Set the column(s) temperature(s) to the maximum allowed temperature (160 °C or 180 °C depending on the column limit).
6. Condition the MicroGC column, preferably overnight. This will ensure that all the water has been removed from the column module and no damage will occur to the TCD filaments.

8.5 Column conditioning

Follow this procedure to make sure that any water that might be present inside the analytical column is removed before the TCD is switched on.

Also follow this procedure if the MicroGC module has been stored for a long period.

! The detector filaments may be damaged by improper conditioning. Follow this procedure to avoid damaging the detector filaments.

Column conditioning procedure

1. Switch off the TCD filaments in the method.
2. Set the column temperature of the module to the maximum temperature (160°C or 180 °C depending on the column limit). Leave the filaments off.
3. Download this method to the MicroGC.
4. Run the downloaded method to condition the column, preferably overnight.

This will assure you that all the water has been removed from the column and no damage will occur to the TCD filaments.

Co-elution of nitrogen and oxygen in Molsieve columns

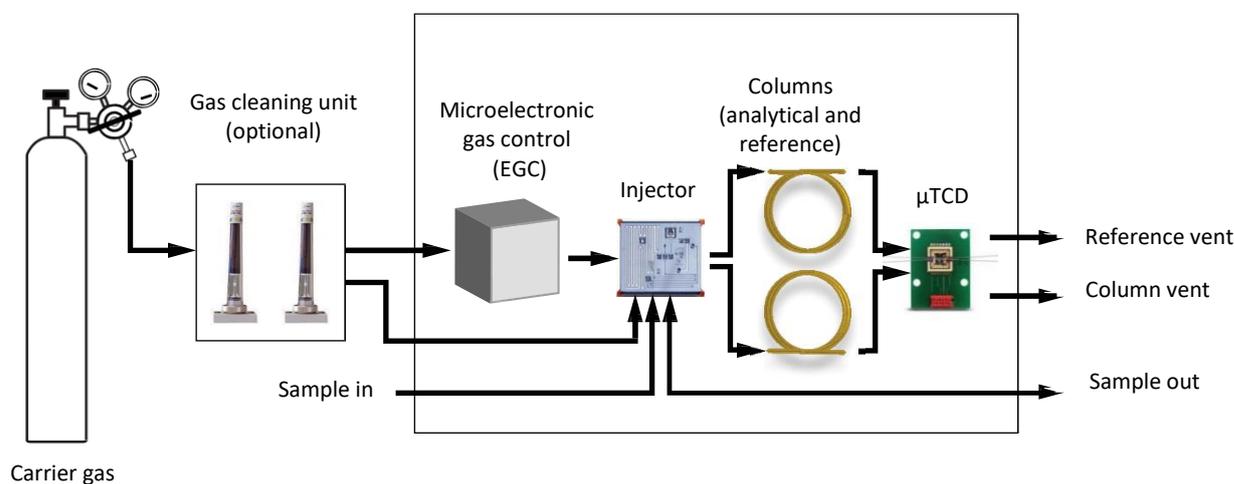
On a properly activated column, nitrogen and oxygen will be well separated. However, in time you will find that these two peaks begin to merge together. This is caused by water and carbon dioxide present in the sample or carrier gas, adsorbing to the stationary phase.

To restore the column efficiency, condition the column, described above, for about an hour. After reconditioning, you can test the column performance by injecting plain air. If you have a proper separation between nitrogen and oxygen again, the column separation power has been restored. If the MicroGC frequency of use is very high, you might consider routinely leaving the oven temperature at 180 °C overnight. The longer the reconditioning period, the better the column performance.

9. MicroGC channels

The instrument contains up to 4 channels. A MicroGC channel contains a gas regulator, an injector, a column, and a TCD detector. See figure below.

This chapter provides a brief analysis of the major components of the MicroGC and the backflush option.



Gas flow diagram

9.1 Electronic pressure control (EPC)

The instrument precisely controls the temperature, the pressure, and the flows electronically during the analysis and between the analyses, without operator intervention.

9.2 Inert sample path

The SOLIA is equipped with an UltimetTM-treated sample path. This deactivation process ensures the integrity of the sample and helps to achieve the best detection limits possible.

The deactivation is applied to tubing running from the sample inlet to the injector.

9.3 Injector

The gaseous sample enters the MicroGC heated manifold. The manifold regulates the temperature of the sample and directs this one towards the injector.

The injector then drives the sample onto the column, while a vacuum pump helps conducting the sample through the system.

9.4 Column

A variety of column configurations are possible on the MicroGC. The columns you require for your specific analyses have been installed at the factory. Other configurations are, of course, possible, but altering the GC channels is a delicate matter that can only be handled by an SRA Instruments technician. The table below shows several standard columns as supplied in the MicroGCs and selected applications. Other columns are available by contacting Agilent Technologies.

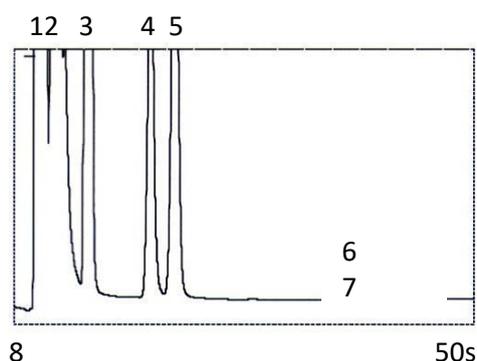
Column/Phase type	Target components
Molsieve 5Å	Permanent gases (N ₂ /O ₂ separation), methane, CO, NO, and so forth. 20 m required for O ₂ -Ar baseline separation). Natural gas and biogas analysis. Optional Retention Time Stability (RTS) configuration.
Hayesep A	Hydrocarbons C ₁ –C ₃ , N ₂ , CO ₂ , air, volatile solvents, natural gas analysis.
CP-Sil 5 CB	Hydrocarbons C ₃ –C ₁₀ , aromatics, organic solvents, natural gas analysis
CP-Sil 19 CB	Hydrocarbons C ₄ –C ₁₀ , high boiling solvents, BTX.
CP-WAX 52 CB	Polar volatile solvents, BTX.
PLOT Al ₂ O ₃ /KCl	Light hydrocarbons C ₁ –C ₅ saturated and unsaturated. Refinery gas analysis
PoraPLOT U	Hydrocarbons C ₁ –C ₆ , halocarbons/freons, anesthetics, H ₂ S, CO ₂ , SO ₂ , volatile solvents. Separation of ethane, ethylene, and acetylene.
PoraPLOT Q	Hydrocarbons C ₁ –C ₆ , halocarbons/freons, anesthetics, H ₂ S, CO ₂ , SO ₂ , volatile solvents. Separation of propylene and propane, coelution of ethylene and acetylene.
CP-COX	CO, CO ₂ , H ₂ , Air (coelution of N ₂ and O ₂), CH ₄ .
CP-Sil 19CB for THT	THT and C ₃ –C ₆ in Natural Gas Matrix.
CP-Sil 13CB for TBM	TBM and C ₃ –C ₆ + in Natural Gas Matrix.
MES NGA	Unique column specially tested for MES in natural gas (1 ppm).

! All columns except the Hayesep A (160 °C) and MES (110 °C) columns can be used up to 180 °C, the maximum temperature of the column oven. Exceeding this temperature will cause the column to lose efficiency instantly and the column module will need replacement. All channels have a built-in protection that prevents a setpoint above the maximum temperature.

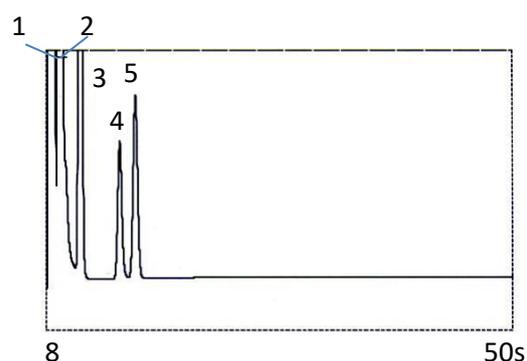
See section 15, Appendix II for more details on the columns.

9.5 Backflush option

The analytical modules of the SOLIA can be optionally equipped with a backflush. This has the advantage of protecting the stationary phase of the column from moisture and carbon dioxide. In addition, the analysis times are reduced since late elution compounds, which are therefore of no interest, do not enter the analytical column.



Natural gas analysis, straight



Natural gas analysis, with backflush

A backflush system always consists of a pre-column and an analytical column. The two columns are coupled at a *pressure point*, which makes it possible to invert the carrier gas flow direction through the pre-column at a preset time, called the *backflush time*. See Figure 2.

The injector, the two columns and the detector are in series.

The sample is injected onto the pre-column where the pre-separation takes place. Injection takes place in normal mode. See Figure 1.

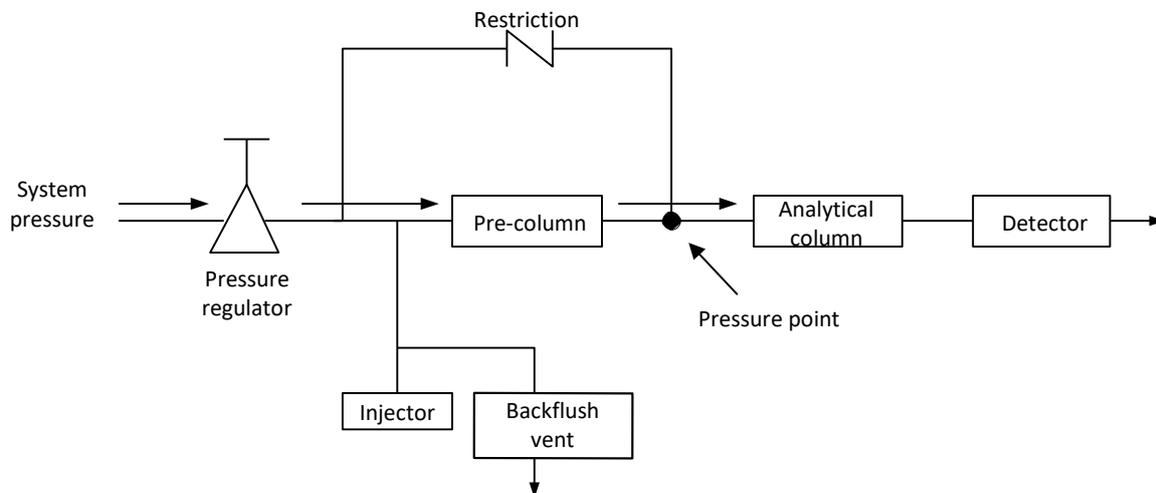


Figure 1

When all compounds to be quantified are transferred to the analytical column, the backflush valve switches (at the backflush time). On the pre-column, the flow inverts and all compounds left on the pre-column now backflush to the vent. On the analytical column the separation continues because there the flow is not inverted. See Figure 2.

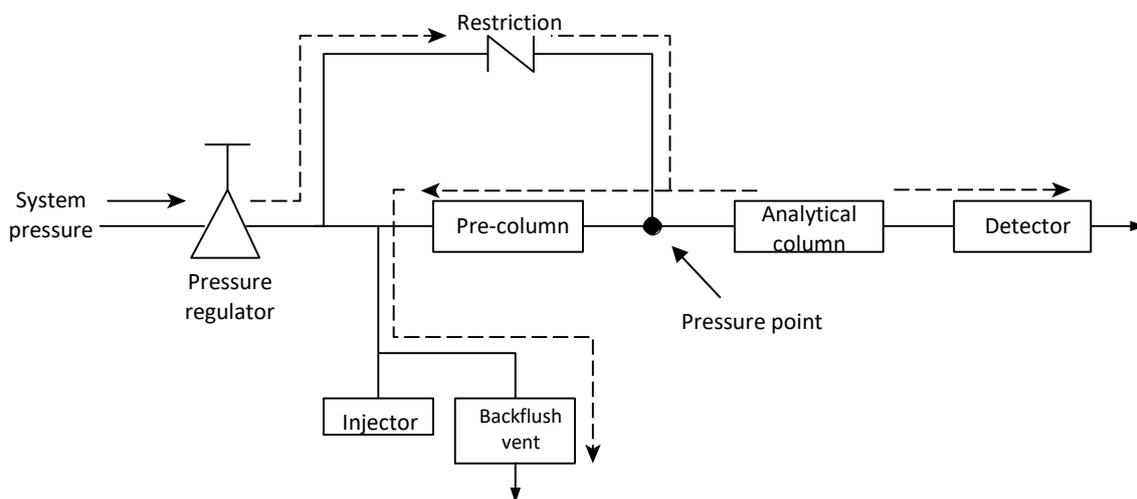


Figure 2

The standby mode is the backflush configuration (if the instrument is equipped with an optional backflush valve).

Backflushing saves the time required to elute high boiling components that are not of interest and ensures that the pre-column will operate under good conditions.

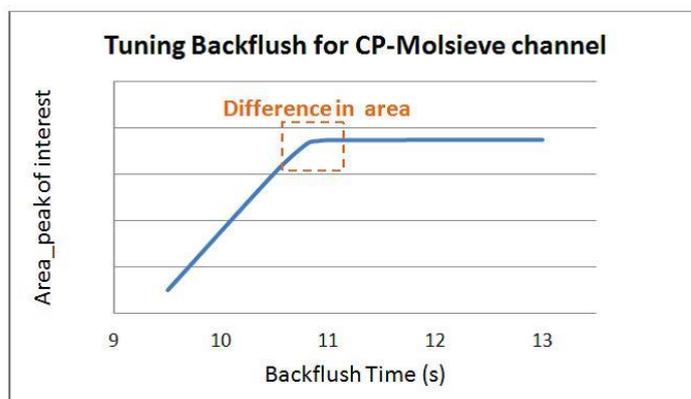
9.5.1 Tuning the backflush time (except on a HayeSep A channel)

Tuning the backflush time is necessary for each new channel. This chapter describes how to tune the backflush time on all channels except HayeSep A.

Tuning procedure for the backflush time

1. Set the backflush time to 0 s and analyze the checkout sample or a specific channel sample. The purpose of this operation is to identify the components of the calibration mixture.
2. Change the backflush time to 10 s and start up. We observe that:
 - When the backflush time is set too early, the peaks of interest are partially or totally backflushed.
 - If the backflush time is set too late, the unwanted components are not backflushed and show up in the chromatogram.
3. Perform runs with different backflush times until there is no huge difference in the peak of interest. To fine tune the backflush time, set smaller steps (for example 0.10 seconds) until you find the optimal backflush time.

The figure below shows an example of tuning the backflush time for the CP-Molsieve 5A channel.



Effect of the backflush time on the peak of interest

9.5.2 To disable backflush

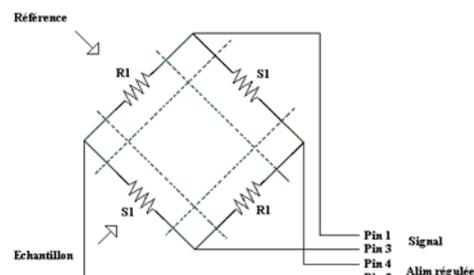
To disable backflushing, set the **Backflush Time** to 0. This puts the system in normal mode during the entire run.

9.6 Detector

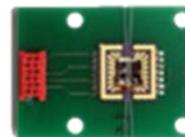
After separation on the column, the sample flows through a thermal conductivity detector (TCD), which is a standard Wheatstone bridge. One way is reserved to the reference gas (pure carrier gas). The second way is reserved to the analytical column flow. This flow is constituted by pure carrier gas or contains molecules from the sample.

When the analytical flow is pure carrier gas, bridge is equilibrated, and signal is zero.

When the analytical flow contains other molecules, the heat capacities of the polyatomic sample molecules cause fluctuations in the filament temperature. The bridge is unbalanced, and the signal is amplified by the electronics to get the chromatogram.



Each module has its own detector with ultra-miniaturized volume (μ TCD).



The μ GC TCD is a universal detector, easy to use. With a response time of 5 ms, the μ TCD performs very fast analysis on capillary columns.

This μ TCD is sensitive and linear. The dynamic response starts from several ppm to 100 % with an excellent linearity (± 10 % on 6 decades).

The μ TCD has 2 different gain scales (standard and high). The software will automatically compensate the signal level. The peak area remains the same in both sensitivities, only the Signal/Noise ratio changes. It is convenient to use only one calibration method for both gains.

WARNING: gas flow in the detector!

It is strictly recommended to purge all the carrier gas lines before powering on the TCD. Air traces in the tubing, module and columns can seriously damage the TCD filaments.

Although each analytical module is equipped with safety systems to impose the detector power dump in absence of carrier gas, please remain careful, in particular when changing the carrier gas cylinders, or when starting the instrument afterwards a long unused period.

If the carrier gas cylinder is empty, safety systems require the pressure regulator and TCD to be switched off.

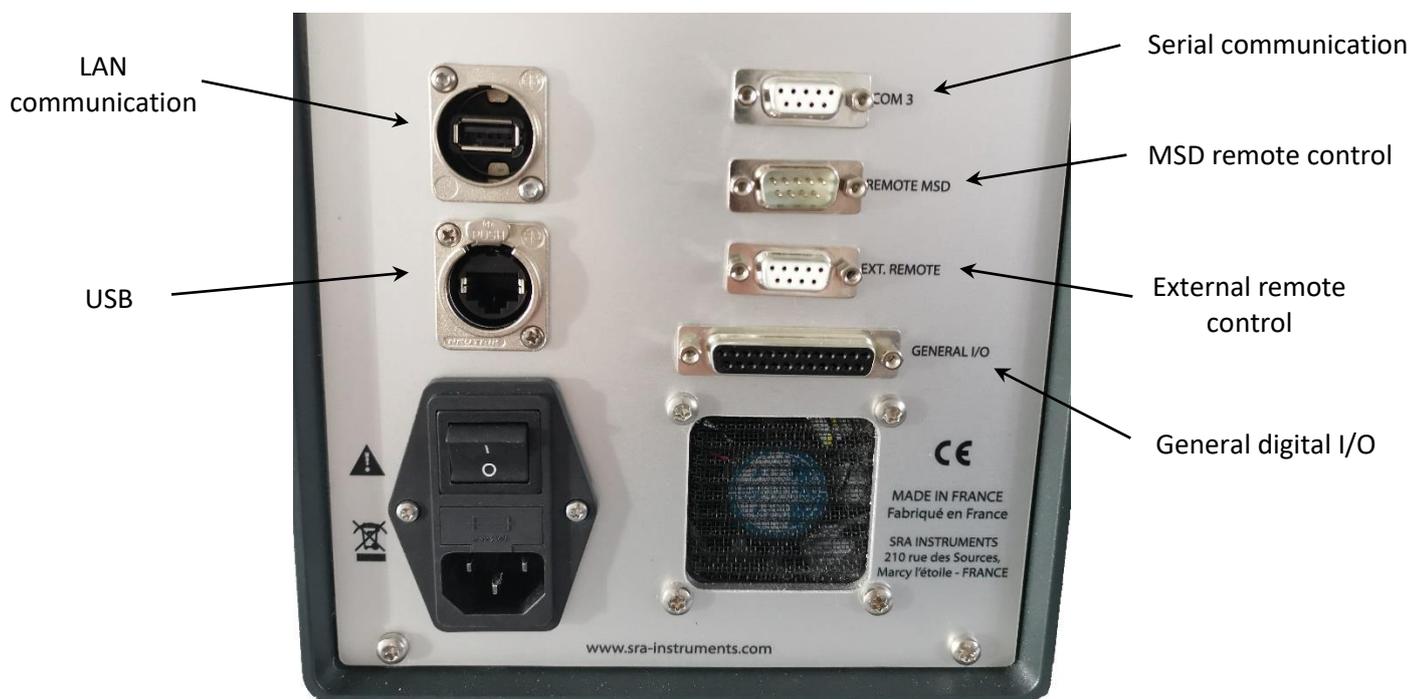
The problem is that when you change cylinders, the air enters the tubes and passes through the TCD, which creates a risk of damaging the detector when you switch it back on again.

To avoid this risk, it is necessary to correctly purge pressure regulator and distribution system from the cylinder to the MicroGC carrier gas inlet. To do this, you must download a « purge » method on the instrument, to purge the internal parts with the new carrier gas, keeping the detector OFF.

10. Communication

This chapter describes the input and output ports accessible in the SOLIA for interfacing with external devices.

10.1 Access to connection ports



10.2 Chromatography software

The SOLIA requires a software for control, peak identification, integration, data analysis, reporting, and so forth. A LAN (Ethernet) connection is required for the control from a computer. The maximum number of MicroGC controlled is limited by your software license. For more information on installation procedures, refer to the dedicated manuals for each software.

10.3 FAQ and Glossary

10.3.1 Frequently Asked Questions

Q: Can I connect the MicroGC to my site network?

A: Yes, if the network works with a standard Ethernet and uses a TCP/IP protocol with UTP cabling.

Q: I use a DHCP server, can I use it to assign an IP address to the MicroGC?

A: If you have a MicroGC with a G3581-65000 main board installed, yes.

Q: How do I assign an IP address to the MicroGC?

A: See paragraph 7.1.5 "Assign IP address".

Q: Are the network settings saved if the MicroGC is restarted, or after a loss of power?

A: Yes, the MicroGC network settings are stored in the flash memory and will not be erased in the event of a power failure.

Q: Can I control my MicroGC anywhere in the world via the Internet?

A: Yes, if your network is designed for this, and has internet access or remote access systems (ports 4900, 4901 and 4902 can be opened).

10.3.2 Glossary of network terms

Crossover cable A cable used to connect two, and **only two**, Ethernet devices directly without the use of a hub or switch.

Domain One of several settings within the TCP/IP configuration that identifies paths used to communicate with Ethernet devices. The Domain is an IP address.

Ethernet address (MAC address) This is a unique identifier that every Ethernet communication device has assigned to it. Typically, the Ethernet address cannot be changed and is the permanent way of identifying a particular hardware device. The Ethernet address consists of 6 pairs of hexadecimal digits.

Gateway This is one of several settings within the TCP/IP configuration that identifies paths used to connect with Ethernet devices on a different subnet. The Gateway is assigned to an IP Address.

Host name The host name is an alternate way to identify a device in an easier way for people. Usually the host name and IP address can be used in an interchangeable way.

IP address This is a unique number for each Ethernet device within the set of connected devices. Two PCs may have identical IP addresses so long as they are not interconnected to each other through the Internet. The IP Address consists of a series of four sets of decimal numbers (between 1 and 255) that provide routing information used by the TCP/IP protocol to establish a reliable connection. Without the IP Address, communications would be bogged down trying to establish connections to Ethernet addresses at unknown locations.

Patch cable A cable that is used to connect Ethernet devices to hubs, switches, or your company network.

Protocol A set of rules that govern how computers send and receive information.

RJ45 connector A telephone jack style connector used for a Universal Twisted Pair (UTP) hardware connection for 10/100Base-T Ethernet connections. RJ45-style connectors are used by the MicroGC.

TCP/IP An international standard protocol used by the Internet. We use this protocol for communication to the MicroGC. You may find several network protocols, such as IPX/SPX and NetBEUI, installed on your computer.

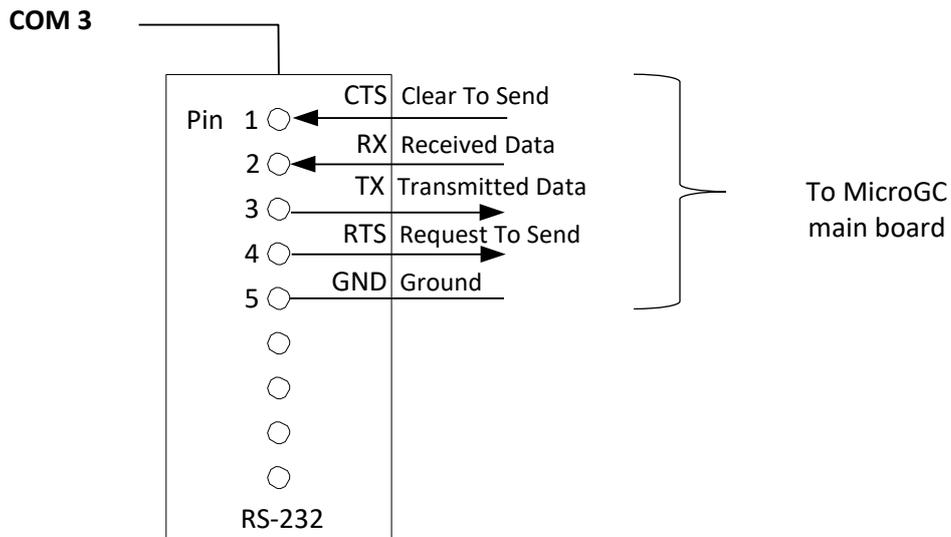
10.4 Pinout of the connectors on the rear panel

10.4.1 Serial port

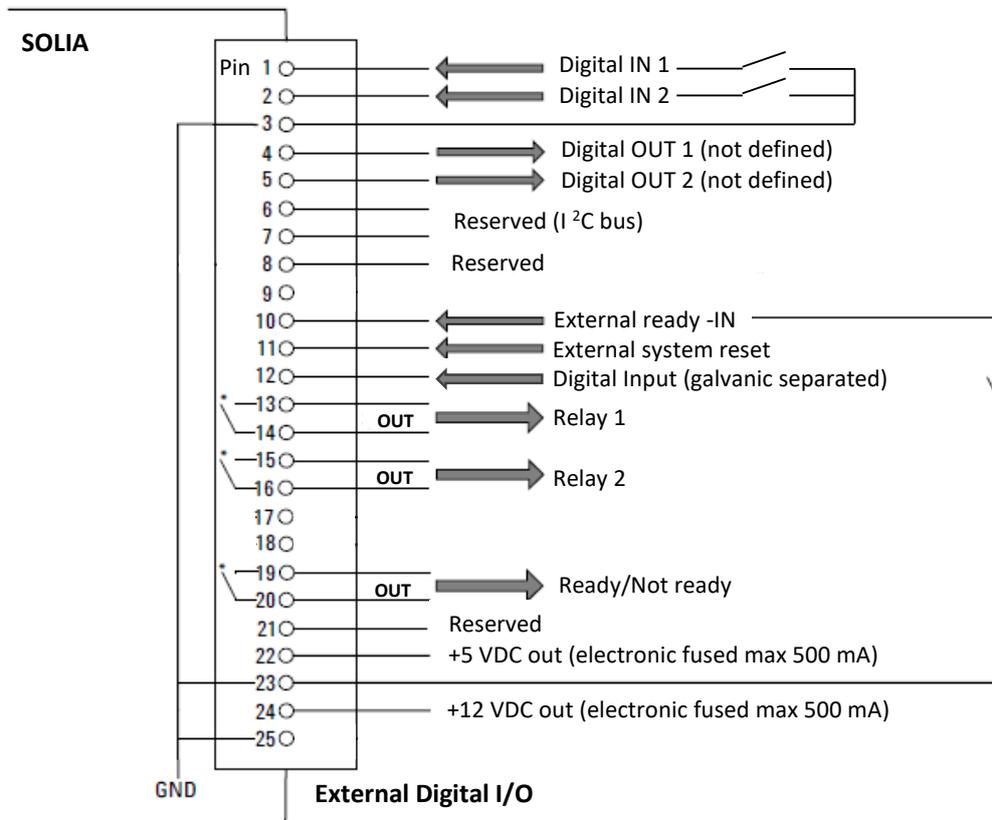
a) COM 3 configuration from MicroGC main board

Pin number	RS232	RS422	RS485 2-wire	RS484 4-wire
1	CTS	RX-	not used	RX-
2	RX	RX+	not used	RX+
3	TX	TX+	Data+	TX+
4	RTS	TX-	Data-	TX-
5	GND	GND	GND	GND

b) COM 3 - DB9 F on SOLIA 990 back panel : RS-232 setting from factory

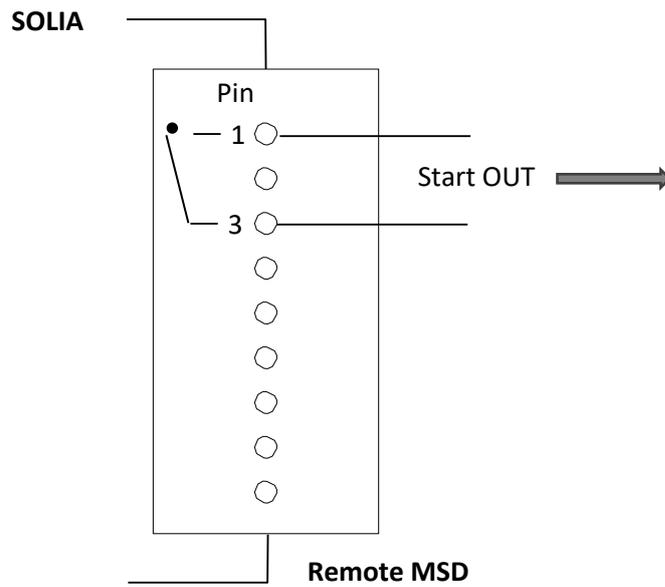


10.4.2 General I/O - DB 25

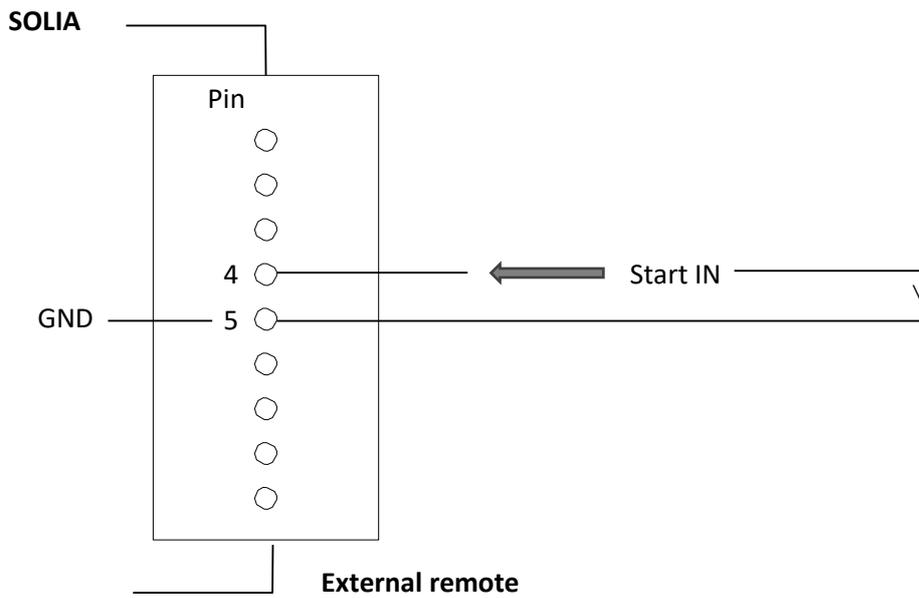


* Relay contacts maximum 24 Volt 1 Ampere

10.4.3 Remote MSD - DB9 M



10.4.4 External remote - DB9 F



11. Errors

11.1 Error handling

During operation, a series of events and error messages are generated, indicating the start or end of certain actions and procedures, as well as small or fatal errors within the instrument. This section describes how the MicroGC reacts to these events or messages.

The following error classes as well as the subsequent actions are available:

Class 0 *Internal event*. These are events indicating a certain procedure has started or finished. In no way do they influence the proper functioning of the instrument.

Class 1 *Advisory fault ; the instrument continues*. These are the least critical consultative errors that do not require immediate action by the user. The current operation can be minimally affected, so it does not need to be stopped. Class 1 error messages indicate certain malfunctions of the instrument. Some errors of this type prevent the instrument from being ready.

Class 2 *Critical errors for logging ; error LED ON*. These errors are critical and the user must be notified immediately (a popup or warning may appear in the data system and the error LED lights). The current operation stops because it could seriously damage the instrument. Corrective action by the user or instrument may be required.

Class 3 *Fatal errors for logging ; instrument shutdown, error LED and buzzer ON*. These are fatal errors for which the user needs immediate warning. The Error LED lights. An instrument shutdown occurs. Corrective action by the user or service is required.

All errors, regardless of class, are available in the data system under the status of the instrument (for troubleshooting). All errors of class 1 or higher are also recorded in the flash memory of the instrument.

Individual numbers identify all errors; these numbers are built using the error class and a number. Events are not numbered.

11.2 Error list

The General Error State is composed of the following items.

The error must be handled as CLNNN in which:

- C = error class (severity)
- L = location
- NNN = error number or event number.

The Error class can be one of the following values:

- 0 = diagnostic error.
- 1 = advisory error.
- 2 = critical error.
- 3 = fatal error.

There are five locations:

- 0 = main board.
- 1 = channel 1.
- 2 = channel 2.

- 3 = channel 3.
- 4 = channel 4.

The table below lists the possible errors.

Error number	Error class	Event/error code	Description	Action needed
1	0	Init passed (event)	End of initialization phase	
2	0	Pressure restored	Pressure restored after Too Low Pressure	
3	0	Start flush cycle	Is a part of the initialization cycle	
4	0	Flush cycle passed	Is a part of the initialization cycle	
5	0	TCD calibrating	Automatic generation after method activation or download.	TCD off and temp. control to default
6	1	Too low pressure	Pressure drops below 35 kPa	Check gas supply
7	1	Pressure fault	Pressure state not ready after 5 minutes	Check gas supply or replace manifold
8	1	Low battery 1	Battery 1 low power (portable Micro GC only)	Recharge battery
9	1	Low battery 2	Battery 2 low power (portable Micro GC only)	Recharge battery
10	2	Sample line sensor fault	Sample line temperature sensor error	Heater turned off
11	2	Sample line temperature fault	Temperature not reached within 35 min (heater error)	Replace sample line heater
12	2	Injector temperature fault	Temperature not reached within 35 min (heater error)	Replace module
13	2	Column temperature fault	Temperature not reached within 35 min (heater error)	Replace module
14	1	TCD Temperature limit activated	Hardware protection activated	
15	0	EDS logging error	Unable to update EDS log	Call service
16	1	Low power supply	Voltage < 10 Volt	Recharge battery
17	2	Injector sensor fault	Injector temperature sensor error	Replace module

Error number	Error class	Event/error code	Description	Action needed
18	2	Column temperature sensor fault	Column temperature sensor error	Replace module
19	2	TCD control error	TCD voltage not or incorrectly set	Call service
20	2	TCD calibration failed	Any error during TCD calibration	Replace module□ or TCD controller board
21	2	Hardware reset	Instrument reset request from WS	
22	2	Pressure too high	Pressure > 450 kPa for at least 2 minutes	Replace manifold
23	3	Initialization error	During initialize	Call service
24	3	Internal communication error	During/after initialization, between MPU and IOC/IOE	Call service
25	3	Instrument EDS incorrect	Instrument Electronic Data sheet incorrect	Call service
26	3	EDS incorrect	Electronic Data sheet incorrect	Call service
27	3	Internal power failure	During/after initialization, internal supplies	Call service
28	0	Flush cycle aborted	Flush cycle stopped before completion	
29	0	GC module changed	Changing a channel (controller or module) and restarting the instrument	
30	0	TCD Gain calibrated	End TCD Gain calibration	
31	0	TCD Offset calibrated	End of Offset calibration	
32	0	Null String	Not used	
33	0	ADC reading out of range	Analog Digital Control out of range	
34	0	EDS Analytical Module incorrect	Electronic Data Sheet Analytical Module incorrect	
35	0	EDS Config checksum incorrect	Electronic Data Sheet Configuration checksum incorrect	

Error number	Error class	Event/error code	Description	Action needed
36	0	EDS Logbook checksum incorrect	Electronic Data Sheet Logbook checksum incorrect	
37	0	EDS Protected checksum incorrect	Electronic Data Sheet Protected checksum incorrect.	
38	0	EDS C.C. Config checksum incorrect	Electronic Data Sheet Channel Control checksum incorrect.	
39	0	EDS C.C. Logbook checksum incorrect	Electronic Data Sheet Channel Control Logbook checksum incorrect	
40	0	EDS C.C. Protected checksum incorrect	Electronic Data Sheet Channel Control Protected checksum incorrect	
41	0	EDS A.M. Config. checksum incorrect	Electronic Data Sheet Analytical Module Configuration checksum incorrect	
42	0	EDS A.M. Logbook checksum incorrect	Electronic Data Sheet Analytical Module Logbook checksum incorrect	
43	0	EDS A.M. Protected checksum incorrect	Electronic Data Sheet Analytical Module Protected checksum incorrect	
44	0	EDS Config SVER incorrect	Electronic Data Sheet Configuration Structure Version incorrect	
45	0	EDS Protected SVER incorrect	Electronic Data Sheet Protected Structure Version incorrect	
46	0	EDS C.C. Config SVER incorrect	Electronic Data Sheet Channel Control Structure Version incorrect	
47	0	EDS C.C. Protected SVER incorrect	Electronic Data Sheet Channel Control Protected Structure Version incorrect	
48	0	EDS A.M. Config SVER incorrect	Electronic Data Sheet Analytical Module Configuration incorrect	
49	0	EDS A.M. Protected SVER incorrect	Electronic Data Sheet Analytical Module Protected Structure Version incorrect	
50	0	Pressure Offset calibration complete	Notification Pressure Offset calibration is completed	
51	0	Pressure Offset calibration Failed	Calibration offset out of range	
52	0	Unable to store pressure offset	Pressure off set is out of valid range	
53	2	Temperature sensor disconnected	Temperature sensor not connect to instrument	Call Service
54	1	Not ready to start run	Issued by Safety Control Object in Hardware domain. Bridge Call to GC domain (Reporting Not Ready To Start Run Error)	Check method
54	1	Stream selection failed	Stream selector (VICI) failed switching	Check valve
55	1	Ambient pressure or temperature alarm	Issued by Safety Control Object in Hardware domain whenever ambient temperature has passed a certain value.	
56	1	Column cleaning	Instrument in column cleaning state	NA

Error number	Error class	Event/error code	Description	Action needed
57	1	Equilibrating temperature zones	Instrument stabilizing after column cleaning	Wait until Ready
76	3	IOC Communication error	MPU is not able to communicate with IOC	Call service
77	3	Read main board EDS error	Not able to read Main board EDS	Call service
78	3	Read channel controller EDS error	Unable to read EDS controller	Call service
79	3	Read channel analytical module EDS error	Not able to read analytical module EDS	Call service
990	3	Watchdog Error: Store Application report on flash error	Internal Software Error, can't store application report on flash memory.	Auto reboot
991	3	Watchdog Error: Store ErrorLog report on flash error	Internal Software Error, can't store ErrorLog report on flash memory.	Auto reboot
992	3	Watchdog Error: Instrument frozen (hazardous error)	Internal Software Error, software hanging	Auto reboot
993	3	Watchdog Error: OOA Timer error	Internal Software Error, OOA Timer could not be created.	Auto reboot
994	3	Watchdog Error: ACE reactor stopped	Internal Software Error, ACE reactor stopped.	Auto reboot
995	3	Watchdog Error: Event pump stopped for 20 s	Internal Software Error, Event pump stopped.	Auto reboot
996	3	Watchdog Error: IOC Fatal error 0	Internal Software Error, IOC fatal error 0	Auto reboot
997	3	Watchdog Error: IOC Fatal error 1	Internal Software Error, IOC fatal error 1	Auto reboot
998	3	Watchdog Error: IOC Fatal error 2	Internal Software Error, IOC fatal error 2	Auto reboot
999	3	Watchdog Error: IOC Fatal error 3	Internal Software Error, IOC fatal error 3	Auto reboot

12. Technical data

12.1 Power supply

220-240 VAC, 50-60 Hz

12.2 Dimensions and weight

SOLIA (MicroGC only)

Width : 191 mm

Height : 480 mm

Depth : 525 mm

SOLIA (MicroGC + Coupling Interface)

Width : 383 mm

Height : 480 mm

Depth : 525 mm

Weight : \approx 15 kg

SOLIA coupled to MS

Width : 700 mm

Height : 480 mm

Depth : 542 mm

Weight : \approx 85 kg

12.3 Working conditions

- Relative humidity : 0 to 95 %
- No condensation
- Room temperature :
 - Temperature : 0 °C to 40 °C
 - The MicroGC automatically shuts down if the ambient temperature exceeds 65 °C.
- Ambient pressure : The MicroGC automatically shuts down if the ambient pressure is greater than 120 kPa.
- Maximum operation altitude : certified up to 2000 meters above sea level.
- Indoors use

12.4 Chromatographic modules

- Up to 4 modules
- 1 or 2 carrier gases

12.4.1 Carrier gases

- Compatible with helium, hydrogen, nitrogen, and argon with 1/8" Swagelok fittings.
- Inlet pressure: minimum = 550 ± 20 Kpa (80 ± 3 psi) 5.5 bars
- Minimum purity: 99.9996 % (for trace analysis 99.9999% is recommended)

12.4.2 Sample and injection

- Gas or vapor samples only
- Sample pressure: from atmosphere to 14.5 psi max (1 bar)

12.4.3 Injector

- Micro-machined injector without moving parts
- Injection volume from 1 to 10 µL
- Heated injector up to 110 °C, including a heated sample transfer line.
- Optional back-flush

12.4.4 Column

Temperature range, up to 180 °C, isothermal

12.4.5 Detector

- Micro-machined thermal conductivity detector (TCD)
- Dual channel (sample flow and reference flow)
- Internal volume per 200 nL channel
- Filaments, oven

12.4.6 TCD operating range

- Concentration, from 1 ppm to 100 %.
- Dynamic linear range, 10^6

12.4.7 TCD detection limits

Detection limits are typical for selected components, as long as the column length and conditions used are appropriate.

- 0.5 ppm for WCOT capillary columns between 4 and 10 m long.
- 2 ppm for PLOT columns

12.4.8 Repeatability

< 0.5 % RSD for propane at 1 % molar level for WCOT columns at constant temperature and pressure.

12.5 Driving software

Soprane II by default

13. EU declaration of conformity

We,



SRA Instruments
210 Rue des Sources
69280 MARCY L'ETOILE
FRANCE

As a manufacturer, declare under our sole responsibility that the instrument type

SOLIA 990 MicroGC / MS analyzer	
---	--

to which this declaration relates, meets the Essential Health and Safety Requirements applicable to it and which are defined by the following Directives and subsequent additions and / or changes:

- 1/ Directive 2014/35/EU, Annex I
- 2/ Directive 2014/30/EU, Annex I

Compliance with the above requirements has been ensured by applying the following standards:

1/ Directive 2014/35/EU – Low voltage

- NF EN 61010-1:2010+A1:2019 "Safety requirements for electrical equipment for measurement, control, and laboratory use – Part 1: General requirements"
- NF EN IEC 61010-2-081:2020 "Safety requirements for electrical equipment for measurement, control and laboratory use – Part 2-081: Particular requirements for automatic and semi-automatic laboratory equipment for analysis and other"

2/ Directive 2014/30/EU – Electromagnetic compatibility

- NF EN 61326-1:2013 "Electrical equipment for measurement, control and laboratory use – EMC requirements – Part 1: General requirements"
- NF EN 61000-4-2:2009 "Electromagnetic compatibility (EMC) – Part 4-2: Testing and measurement techniques – Electrostatic discharge immunity test"

In accordance with the above-mentioned directives (Module A), the above-mentioned equipment is subject, regarding design and production aspects, to *internal production control*: **E FAB 24**

Marcy l'Etoile, 23 November 2020

Legal representative,
Armando MILIAZZA

210 rue des Sources
69280 MARCY L'ETOILE
Tél. 04.78.44.29.47
Fax 04.78.44.29.62
www.sra-instruments.com
SIRET: 642 068 731 00054

14. Appendix I : Driving a SOLIA from Soprane II

Soprane II, when combined with MassHunter GC-MS Acquisition and MSD Chemstation Data Analysis, enables the MicroGC/MS SOLIA coupling to be controlled.

14.1 Installation

Communication between Soprane II, MassHunter and MSD Chemstation Data Analysis is carried out via computer macros. These macros are automatically deployed in the MassHunter and MSD Chemstation Data Analysis tree structure when Soprane II is installed. It is therefore necessary to install Agilent softwares before installing Soprane II.

Install in this order: MassHunter GC-MS Acquisition, MSD Chemstation Data Analysis (MassHunter version) and then Soprane II as administrator and following the recommended settings.

14.2 Instrument configuration

14.2.1 Creating the SOLIA instrument in Soprane II

The screenshot displays the configuration window for an instrument. It is divided into two main sections: 'Instruments' and 'Connection'.
 - The 'Instruments' section has a blue header and shows 'CP 990 LAN' in a dropdown menu.
 - The 'Connection' section has a blue header and contains several fields:
 - 'Analyzer' is set to '10 . 1 . 1 . 101'.
 - 'Adam modules' is a dropdown menu.
 - 'Valve' is a dropdown menu.
 - 'Solia' is set to '990 - 2'.
 - 'Keller' is a dropdown menu.
 At the bottom of the window is a green 'Confirm' button with a checkmark icon.

From Soprane Configurator, create a new analyzer by clicking on the "+" icon. Then enter the IP address of the instrument. Select "990 - 2" for the SOLIA category (as shown in the image above).

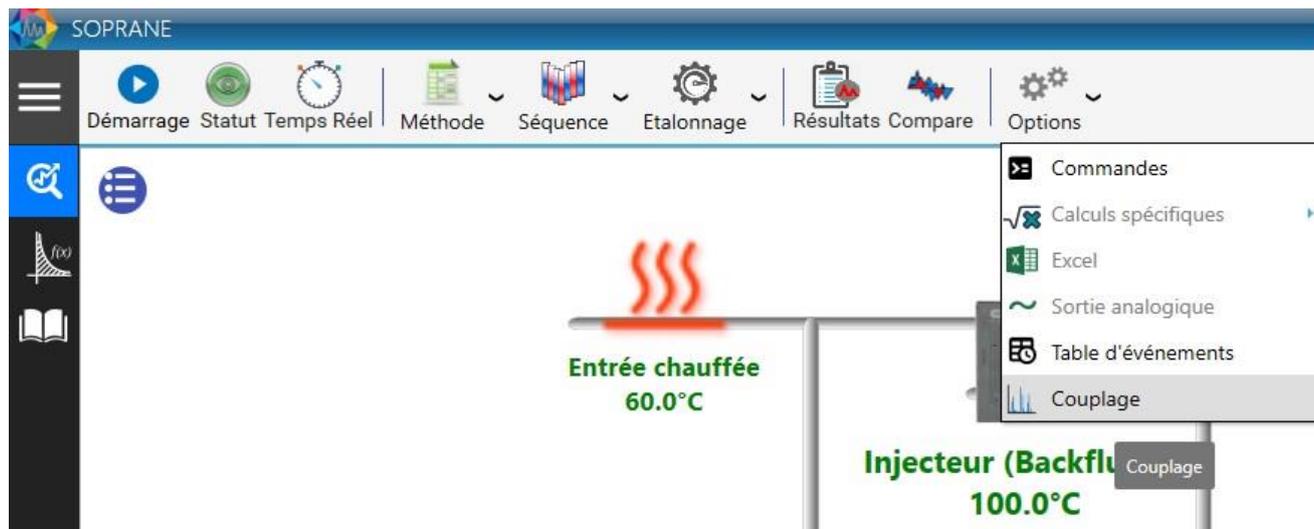
14.2.2 Creating the MSD instrument in Agilent GCMS Configuration

- Start Agilent GCMS Configuration as an administrator
- Select the instrument number to be configured (default is "1")
- Fill in the name of the instrument (for example "MSD") and the identity (ID) of the laboratory (optional)
- Select the MSD model
- Enter the IP address of the MSD. The IP address of the instrument is displayed on the screen inside the top cover of the MSD.

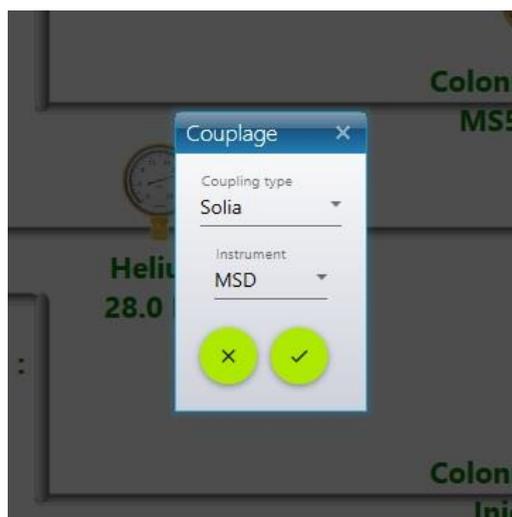
- Specify the polarity of the quadrupole in "DC Polarity". This information is located inside the top cover ("Pos" or "Neg")
- Select "None" for the GC model
- Select "Workflow mode: Enhanced".

14.3 Configuration of the coupling

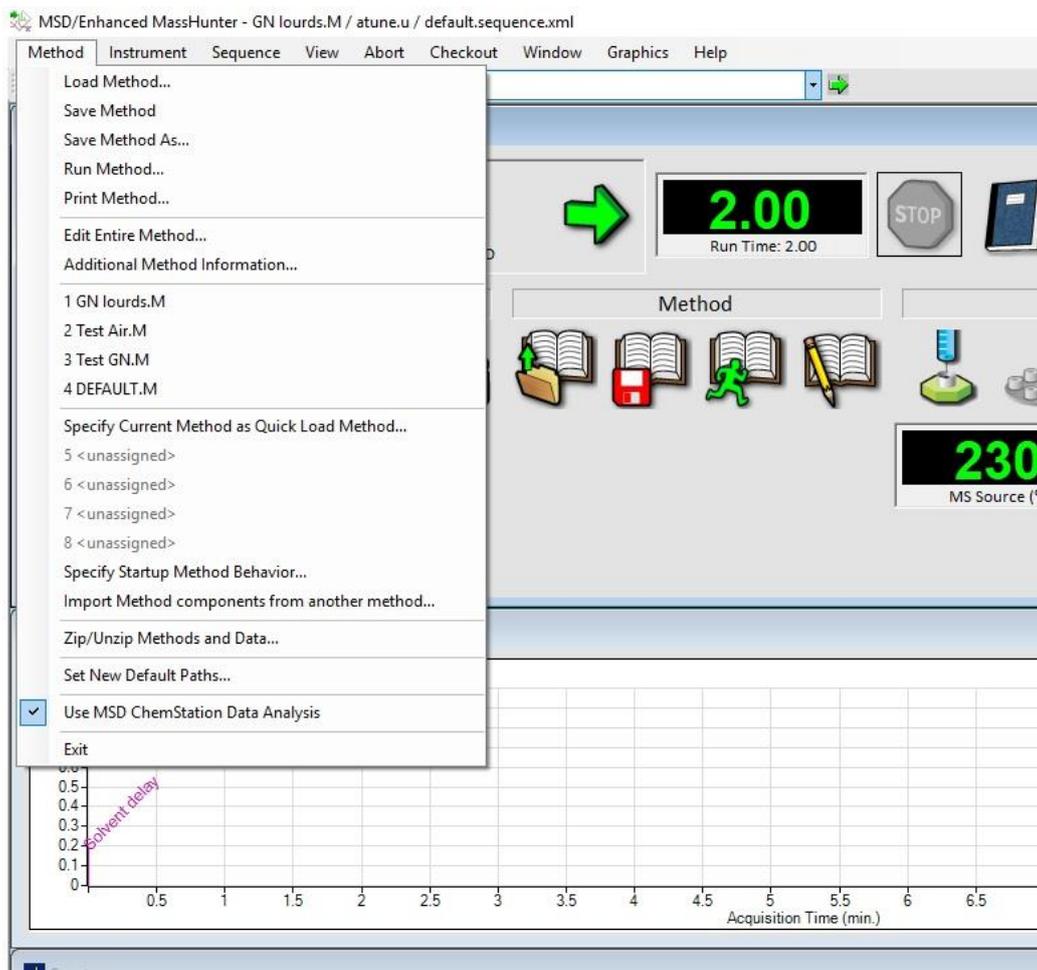
In order to drive the coupling, it is necessary to configure the coupling in each of the software packages. In Soprane II, the activation of the coupling is done from the "option" tab and then "coupling".



A window will then open. Select "SOLIA" and then the name of the MS instrument created in the Agilent GCMS configurator, as below:



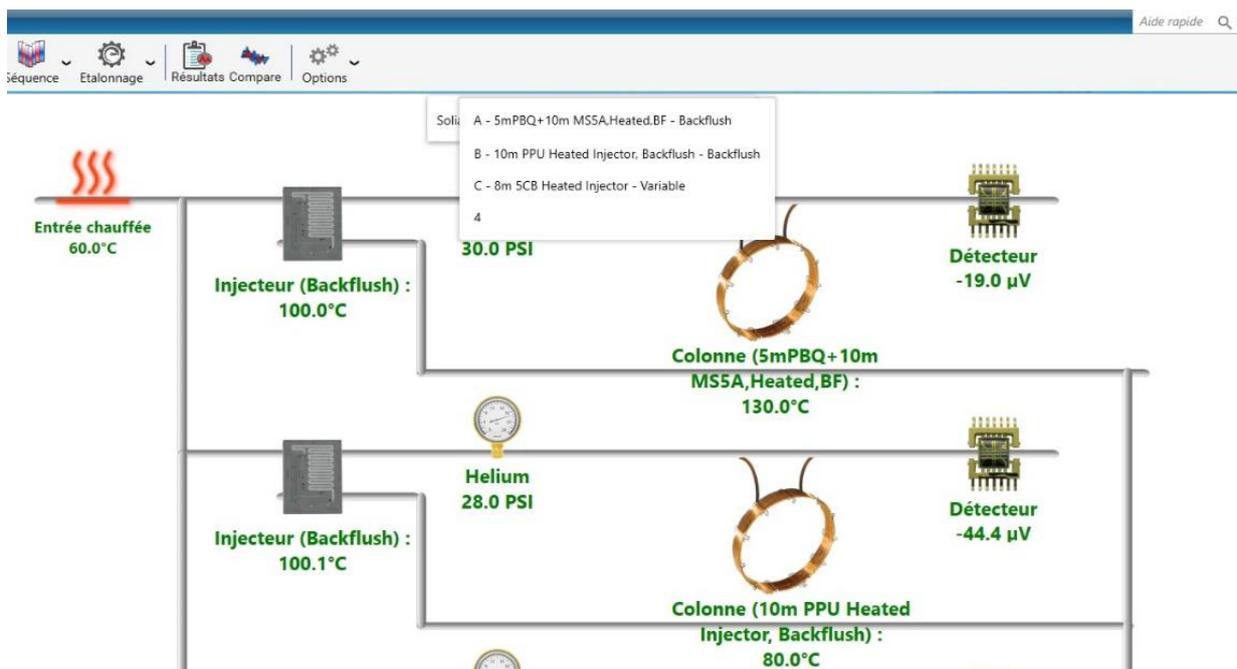
In MassHunter, check the line "Use MSD ChemStation Data Analysis" in the "Method" menu.



14.4 Controlling the SOLIA

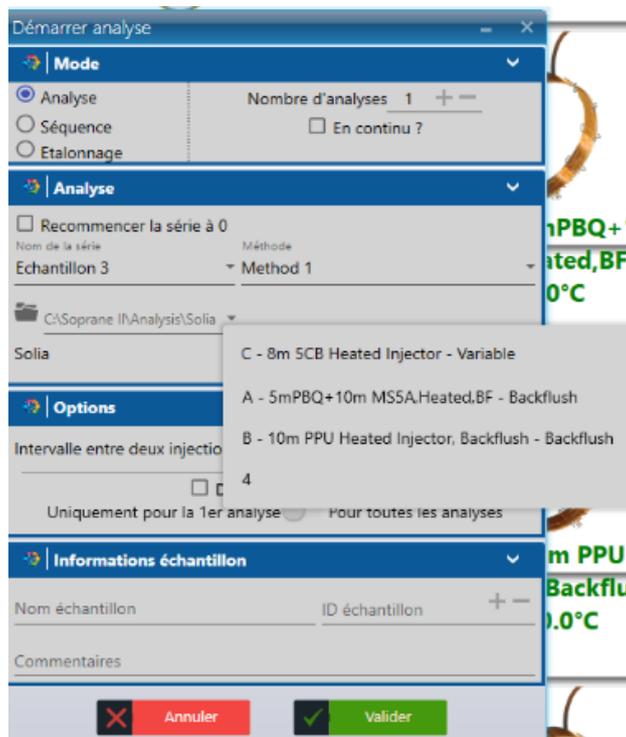
Once these configuration requirements have been completed, it is possible to control the channel selection valve between the μ GC modules and the MS detector. There are several ways to change the position of this valve.

From the "Status" tab, by clicking on the bar above the diagram:

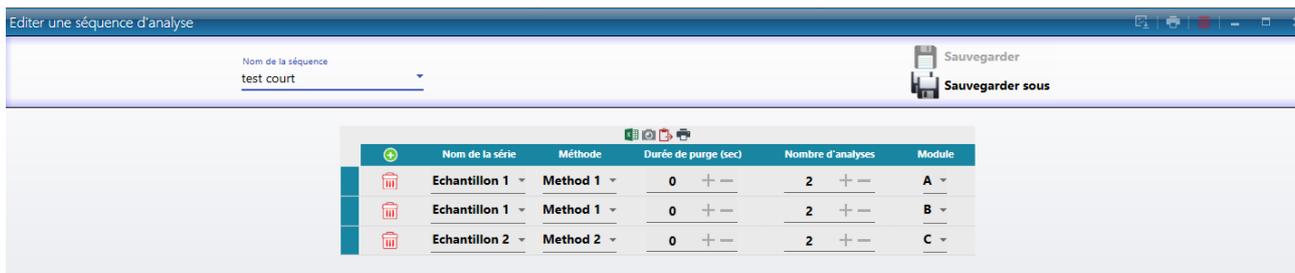


The names of the different modules are displayed on the screen. Click on the module to be coupled to the MS detector.

When starting analyses, in the "Start" tab:



When writing an analysis sequence: the valve position will change between the analyses and during the sequence, in order to couple the desired μ GC module to the MS detector for each analysis:



14.4.1 Creating an analysis method

A SOLIA analysis method includes a Soprane analysis method and a MassHunter analysis method.

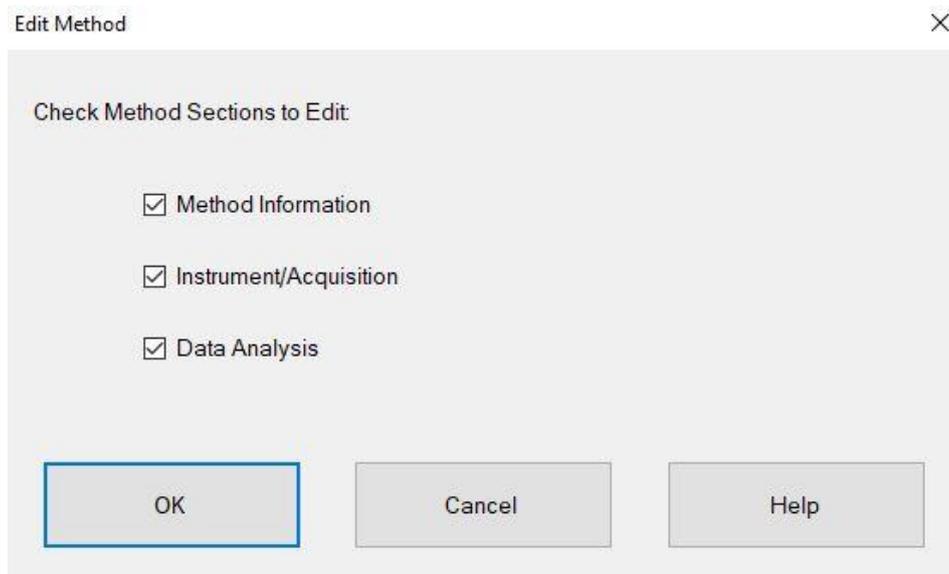
14.4.2 Creating a Soprane II analysis method

Refer to Chapter 4.3.1.

14.4.3 Creating a MassHunter analysis method

From the "File" tab, save the method under the desired name. Then edit the method.

Check all three boxes:



Check "Data Acquisition" and "Data Analysis":

Method Information

Method Comments:

Save Copy of Method With Data

Method Sections to Run

Pre-Run Macros/Commands

Instrument Control: Browse...

Data Analysis: Browse...

Data Acquisition

Data Analysis

Post-Run Macros/Commands

Instrument Control: Browse...

Data Analysis: Browse...

OK Cancel Help

Select "Sample Inlet: Other/None" and "Injection Source: External Device".

Inlet and Injection Parameters

Sample Inlet: Other/None

Injection Source: External Device

Use MS

Inlet Location: Front Rear

MS Connected to: Front Rear

OK Cancel Help

Define MS parameters :

Tune File

Tune File: atune.u

Tune Type: EI

Tune EMV: 786

MS Source: 230 (Actual) / 230 (Setpoint)

MS Quad: 150 (Actual) / 150 (Setpoint)

Acquisition Type: Scan

Detector Setting

Run Time: 2.00 min

Solvent Delay: 0.00 min

Trace Ion Detection

EM Setting: Gain Factor

Gain Factor: 1.00

Applied EM Voltage (V): 903

EM Saver

Limit: Sum Limit 1e8 (Default)

Scan Time Segments

Time	Start Mass	End Mass	Threshold	Scan Speed (u/s)	Frequency (scans/sec)	Cycle Time (ms)	Step Size (m/z)
0.00	5.00	150.00	150	3,125 [N=1]	14.8	67.76	0.1

SIM Time Segments

Time	Group Name	Number of Ions	Total Dwell Time (ms)	Cycle Time (Hz)	Resolution	Gain Factor	Calculated EMV
0.00							

Method Last Saved: 10/19/2018 4:01:52 PM

SRA recommends the above default settings, which should be modified according to the application. The Run Time must be equal to or greater than the Soprane II analysis time.

Check "Quant Report" (essential for the transmission of results to Soprane II) and save the method.

Select Reports

- Percent Report
- LibSearch Report
- Quant Report
- Custom Report
- Update Custom Database

OK Cancel Help

14.5 Results processing

Soprane II offers the possibility to combine all μ GC (TCD detection) and MS results in a same table, as shown below.

Analyse	Date d'injection	Série	Methode...	C1 (A)
Gaz_002	19/10/2018 11:53	Gaz	Test BF	10,012
Gaz_003	19/10/2018 11:56	Gaz	Test BF	10,021
Gaz_004	19/10/2018 11:58	Gaz	Test BF	10,014
Gaz_005	19/10/2018 12:01	Gaz	Test BF	10,035
Gaz_006	19/10/2018 12:04	Gaz	Test BF	10,041
Gaz_007	19/10/2018 12:07	Gaz	Test BF	10,044
Gaz_008	19/10/2018 12:09	Gaz	Test BF	10,021
Gaz_009	19/10/2018 12:12	Gaz	Test BF	10,022
Gaz_010	19/10/2018 12:17	Gaz	Test BF	10,024
Min				10,012
Avg				10,026
Max				10,044
Rsd (%)				0,114

The table above groups the TCD results of the μ GC modules (indicated by the letters (A), (B) and (C)), as well as the MSD results.

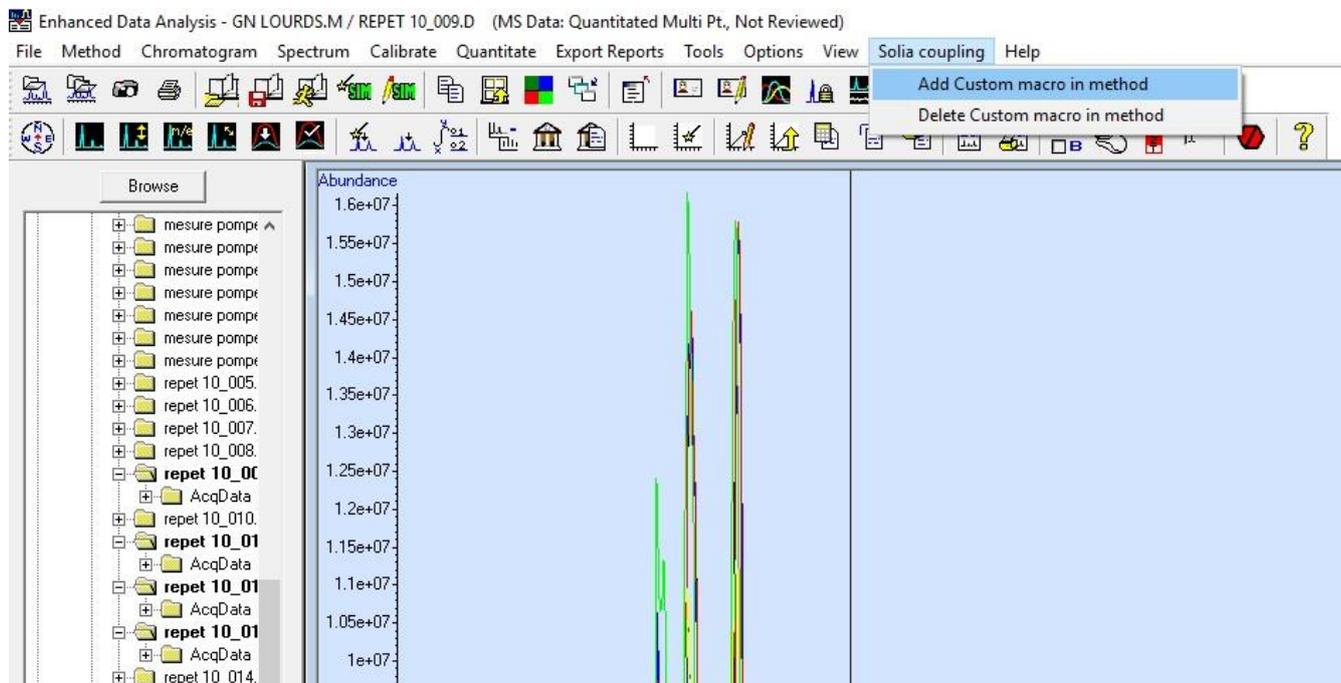
14.5.1 Creating a Soprane II processing method

Refer to Chapter 4.5.

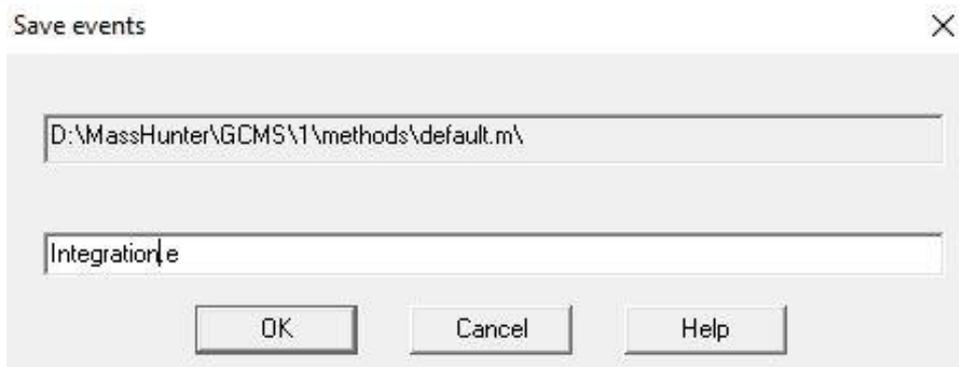
14.5.2 Creating an MSD Chemstation Data Analysis processing method

Open MSD Chemstation Data Analysis and load the acquisition method used. The processing method must be the same as the acquisition method for the results to be transmitted to Soprane II.

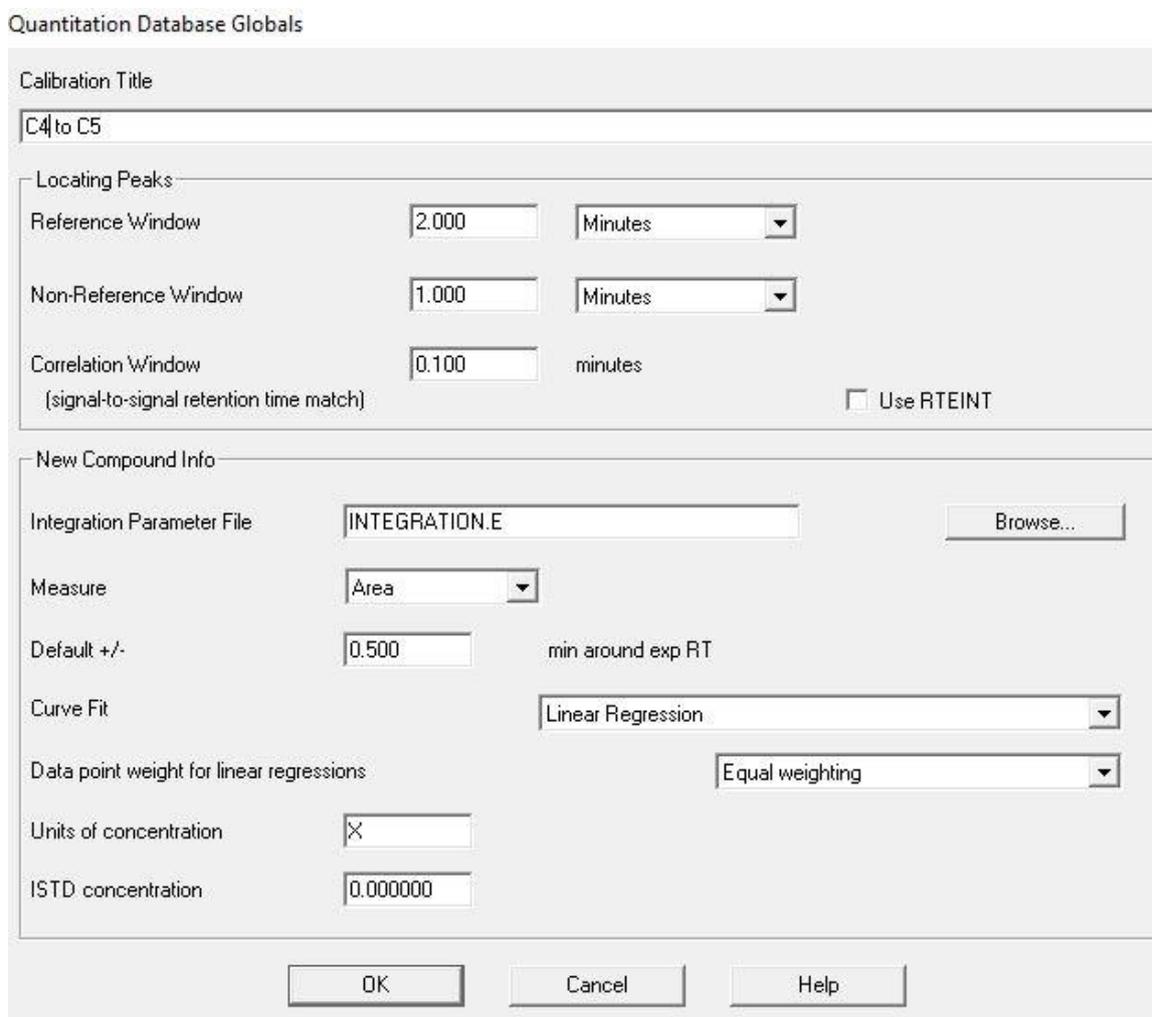
In the "SOLIA Coupling" tab of MSD Chemstation Data Analysis, click on "Add Custom macro in method".



From the "Chromatogram" tab, click on "AutoIntegrate" and then modify the integration parameters in "MS Signal Integration parameters" if necessary. Then save the integration parameters under the desired name:

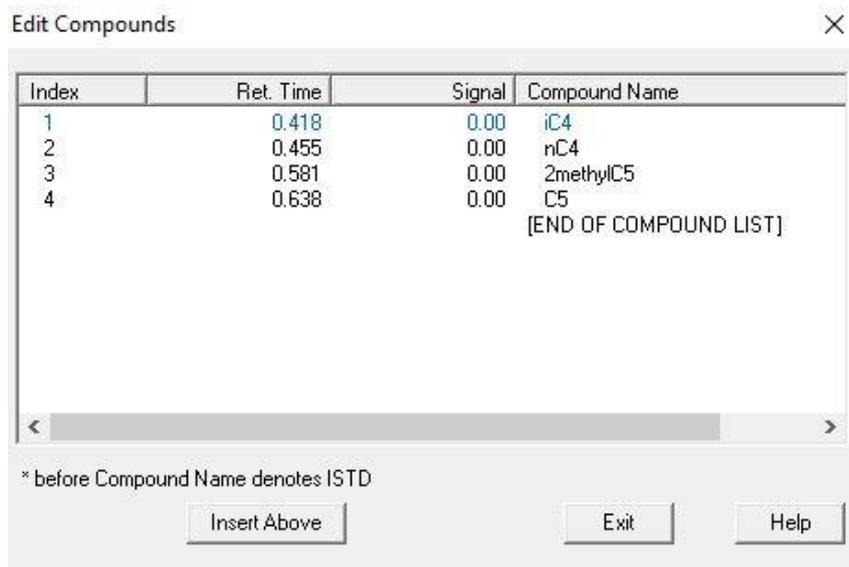


From the Calibration tab, click on "Set Up Quantitation".

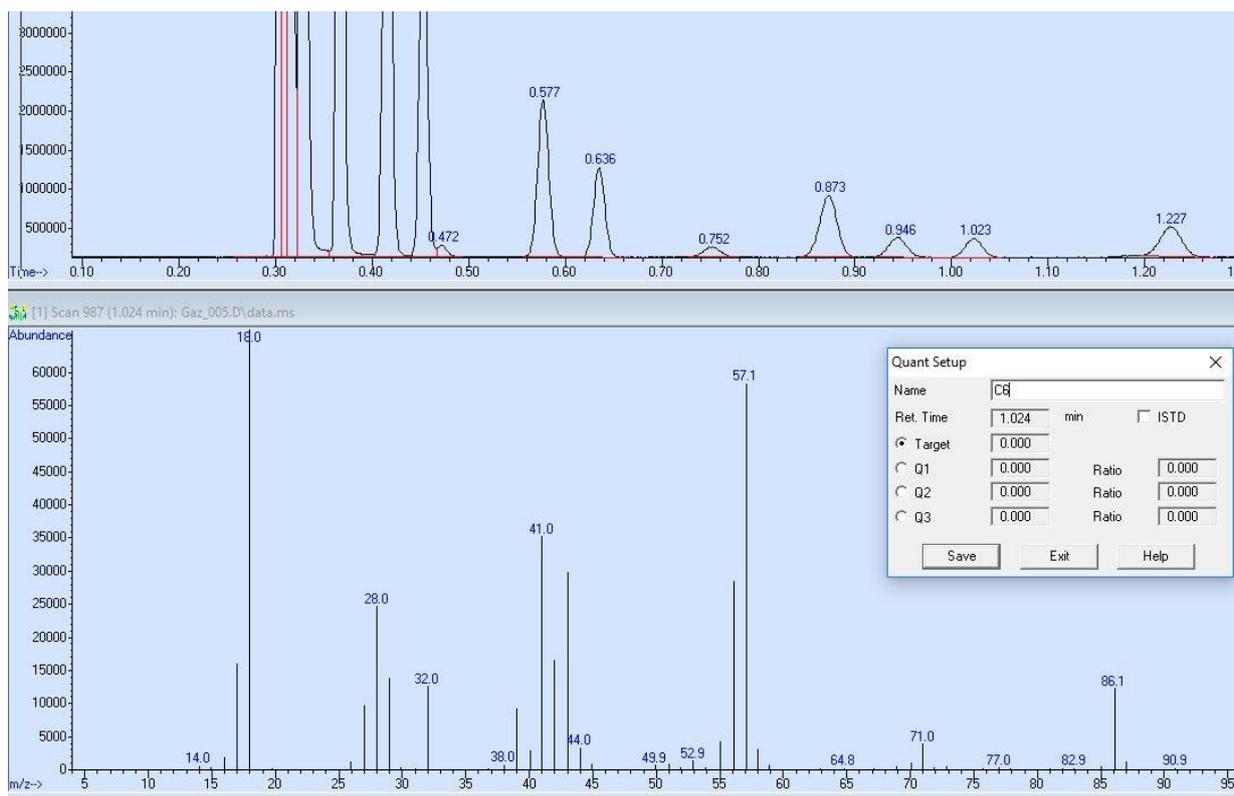


Fill in the fields "Calibration Title", "Integration Parameter File" (click on browse and load the previously saved integration file) and "Units of concentration". Uncheck "Use RTEINT" and click on OK.

The following window allows you to add the desired compounds to the calibration table. Click on "Insert Above".



Right double-click on the chromatographic peak's apex to be added to define its retention time in the calibration. Define the name of the compound.



At this stage, it is also possible to select target ions used for quantification. To do this, click simultaneously on the left click and right click on the m/z peak of the mass spectrum to be considered.

Click on "Save" to move to the next compound, and on "Exit" when all the compounds have been added to the table.

15. Appendix II : Columns

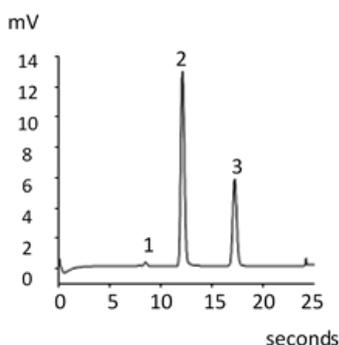
15.1 Molsieve 5Å columns

The Molsieve 5Å column is designed to separate: hydrogen, carbon monoxide, methane, nitrogen, oxygen, and some noble gases. Higher molecular weight components have much higher retention times on this column.

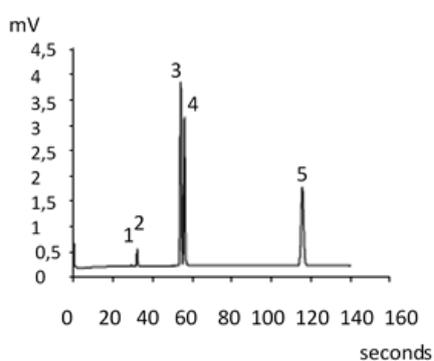
Parameter	4 m Heated	10 m Unheated	20 m Unheated
Column temperature	110 °C	40 °C	40 °C
Injector temperature	110 °C	NA	NA
Column pressure	100 kPa (15 psi)	150 kPa (21 psi)	200 kPa (28 psi)
Sample time	30 s	30 s	30 s
Injection time	40 ms	40 ms	40 ms
Run time	25 s	140 s	210 s
Detector sensitivity	Auto	Auto	Auto
Peak 1	Hydrogen 1.0 %	Neon 18 ppm	Neon 18 ppm
Peak 2	Argon/Oxygen 0.4 %	Hydrogen 1.0 %	Hydrogen 1.0 %
Peak 3	Nitrogen 0.2 %	Argon 0.2 %	Argon 0.2 %
Peak 4	_____	Oxygen 0.2 %	Oxygen 0.2 %
Peak 5	_____	Nitrogen 0.2 %	Nitrogen 0.2 %

- Table 1 -

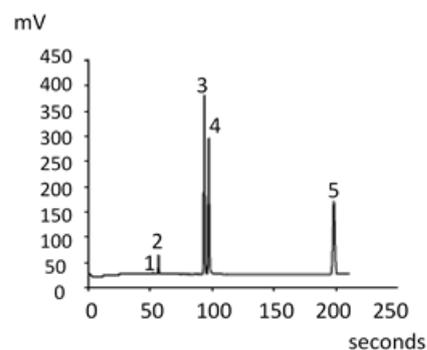
Molsieve 5Å 4 m heated



Molsieve 5Å 10 m unheated



Molsieve 5Å 20 m unheated



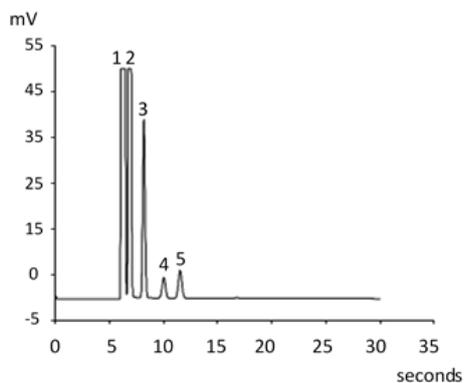
15.2 CP-Sil 5 CB columns

The natural gas components, mostly hydrocarbons, separate in the same order on the non-polar and medium-polar CP-Sil CB columns. Nitrogen, methane, carbon dioxide, and ethane are not separated on these columns. They produce a composite peak. For separation of these components, consider a HayeSep A column.

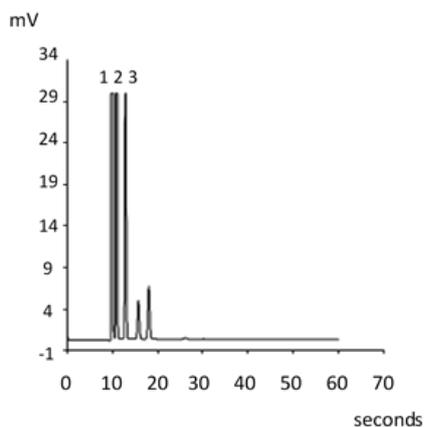
Parameter	4 m Heated	6 m Unheated
Column temperature	50 °C	50 °C
Injector temperature	110 °C	NA
Column pressure	150 kPa (21 psi)	150 kPa (21 psi)
Sample time	30 s	30 s
Injection time	40 ms	40 ms
Run time	30 s	30 s
Detector sensitivity	Auto	Auto
Peak 1	Composite balance	Composite balance
Peak 2	Ethane 8.1 %	Ethane 8.1 %
Peak 3	Propane 1.0 %	Propane 1.0 %
Peak 4	i-Butane 0.14 %	i-Butane 0.14 %
Peak 5	n-Butane 0.2 %	n-Butane 0.2 %

- Table 2 -

CP Sil 5 CB 4 m Heated



CP Sil 5 CB 6 m Unheated

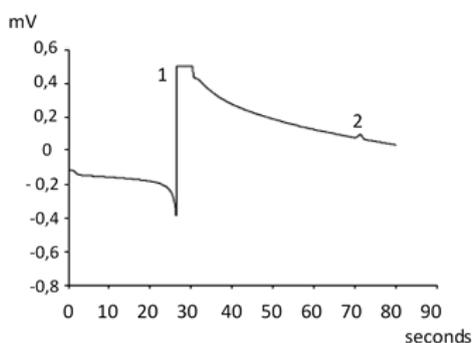


15.3 CP Sil 13 and 19 CB columns

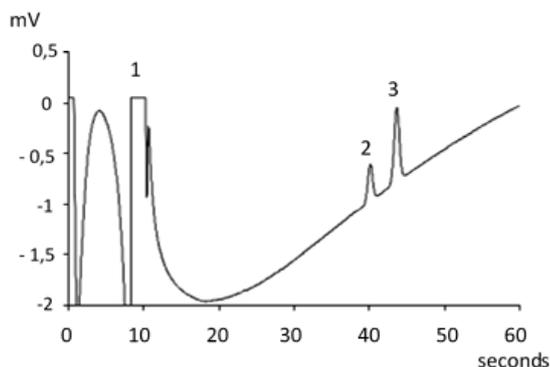
Parameter	CP-Sil 13 CB 12 m Heated (TBM)	CP-Sil 19 CB 6 m Heated (THT)
Column temperature	40 °C	85 °C
Injector temperature	50 °C	85 °C
Column pressure	250 kPa (38 psi)	200 kPa (25 psi)
Sample time	30 s	30 s
Injection time	255 ms	255 ms
Run time	80 s	35 s
Detector sensitivity	Auto	Auto
Peak 1	Methane balance	Helium balance
Peak 2	TBM 6.5 ppm	THT 4.6 ppm
Peak 3	_____	n-Decane 4.5 ppm

- Table 3 -

CP Sil 13 CB 12 m Heated (TBM)



CP Sil 19 CB 6 m Unheated (THT)

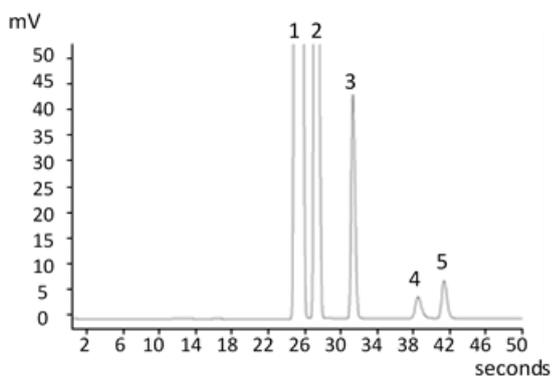


15.4 PoraPlot 10 m column

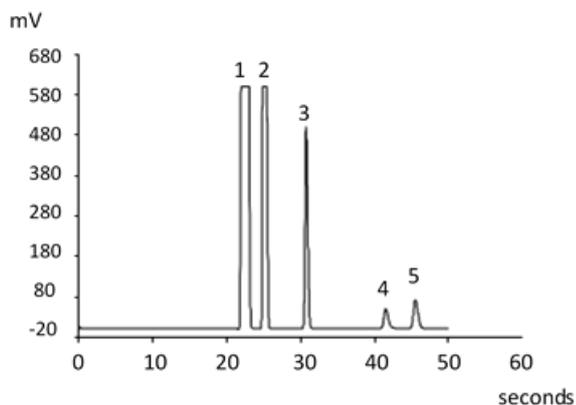
Parameter	PoraPlot U 10 m Heated	PoraPlot Q 10 m Heated
Column temperature	150 °C	150 °C
Injector temperature	110 °C	110 °C
Column pressure	150 kPa (21 psi)	150 kPa (21 psi)
Sample time	30 s	30 s
Injection time	40 ms	40 ms
Run time	100 s	50 s
Detector sensitivity	Auto	Auto
Peak 1	1	Composite balance
Peak 2	2	Ethane 8.1 %
Peak 3	3	Propane 1.0 %
Peak 4	4	i-Butane 0.14 %
Peak 5	5	n-Butane 0.2 %

- Table 4 -

PoraPlot U 10 m Heated



PoraPlot Q 10 m Heated



15.5 Hayesep A 40 cm heated column

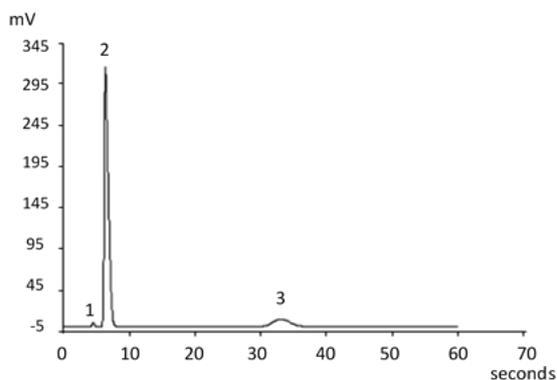
The HayeSep A column separates oxygen, methane, carbon dioxide, ethane, acetylene, ethylene, and selected sulfur gases. Nitrogen coelutes with oxygen. Components with a higher molecular weight than propane have long retention times on this column.

! Maximum allowable column temperature is 160 °C.

Parameter	Hayesep A 40 cm Heated
Column temperature	50 °C
Injector temperature	110 °C
Column pressure	150 kPa (21 psi)
Sample time	30 s
Injection time	40 ms
Run time	60 s
Detector sensitivity	Auto
Peak 1	Nitrogen 0.77 %
Peak 2	Methane balance
Peak 3	Ethane 8.1 %

- Table 5 -

Hayesep A 40 cm Heated

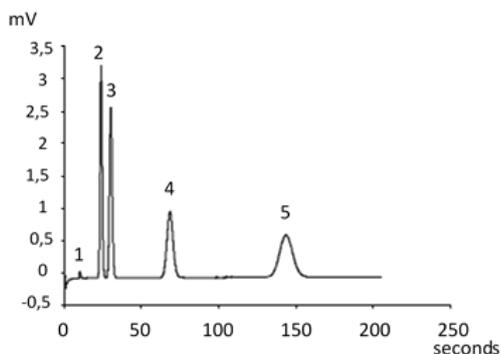


15.6 CO_x and Al₂O₃/KCl columns

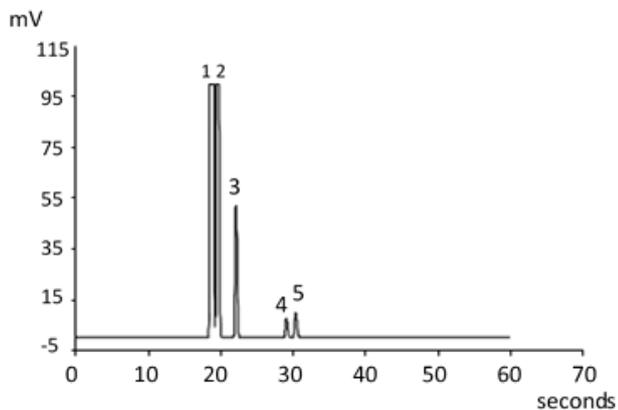
Parameter	CO _x 1 m Unheated	Al ₂ O ₃ /KCl 10 m Heated
Column temperature	80 °C	100 °C
Injector temperature	NA	110 °C
Column pressure	200 kPa (28 psi)	150 kPa (21 psi)
Sample time	30 s	30 s
Injection time	40 ms	40 ms
Run time	204 s	60 s
Detector sensitivity	Auto	Auto
Peak 1	Hydrogen 1.0 %	Composite balance
Peak 2	Nitrogen 1.0 %	Ethane 8.1 %
Peak 3	CO 1.0 %	Propane 1.0 %
Peak 4	Methane 1.0 %	i-Butane 0.14 %
Peak 5	CO ₂ 1.0 %	n-Butane 0.2 %
Helium balance		

- Table 6 -

CO_x 1 m Unheated



Al₂O₃/KCl 10 m Heated

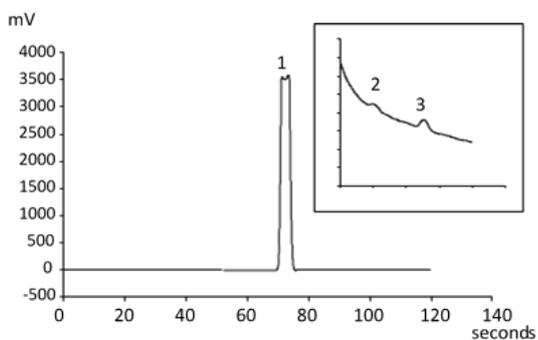


15.7 MES (NGA) and CP-WAX 52 CB columns

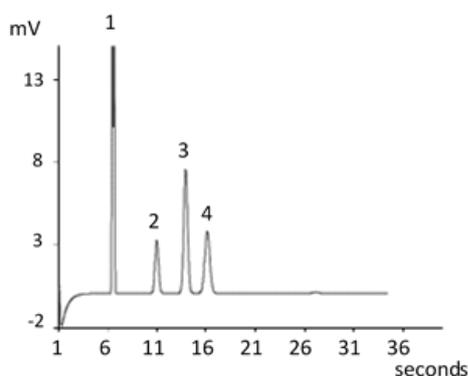
Parameter	MES 10 m Heated (NGA)	CP-WAX 52 CB 4 m Heated
Column temperature	90 °C	60 °C
Injector temperature	110 °C	110 °C
Column pressure	70 kPa (10 psi)	150 kPa (21 psi)
Sample time	30 s	30 s
Injection time	500 ms	40 ms
Run time	120 s	35 s
Detector sensitivity	Auto	Auto
Peak 1	Nitrogen balance	Nitrogen 0.75 %
Peak 2	n-Decane 11.2 ppm	Acetone 750 ppm
Peak 3	MES 14.2 ppm	Methanol 0.15 %
Peak 4		Ethanol 0.30 %
		Helium balance

- Table 7 -

MES 10 m Heated (NGA)



CP-WAX 52 CB 4 m Heated



16. Appendix III : Frequently Asked Questions

16.1 My detector is in default status, what should I do ?

If the detector displays default in Soprane Status:

1. Check that you have correctly purged columns by downloading a purge method before starting the detector, that the carrier gas tubing is tight and of quality, connected with a stainless steel tube at 5.6 bars pressure.
2. Check that a carrier gas flow is present at the columns output (on the back of the MicroGC). If it's not the case on one of the two outputs, contact SRA Instruments after-sales service.
3. Check that the carrier gas used is correctly configured in Soprane Setup software.

If all these verifications are carried out and correct, please, download again the purge method and look at the status.

If the detector is again in default, contact SRA Instruments after-sales service.

16.2 My pressure sensor is in default status, what should I do ?

1. Check that the carrier gas tubing is correctly fed, tight and with a pressure at 5.6 bars.
2. If there are two carrier gas inputs on the MicroGC, check that the two inputs are correctly connected.
3. If necessary, check that the carrier gas is effectively coming at the carrier gas tubing output, at the MicroGC inlet.
4. Check that the columns outputs are at atmospheric pressure and not blocked.

If all these verifications are carried out and correct, download again the method and look at the status.

If the pressure sensor is again in default, please contact SRA Instruments after-sales service.

16.3 I change the carrier gas, what should I do ?

Before changing of carrier gas type, follow the procedure to switch off your MicroGC.

Then, configure the carrier gas type as it is described in paragraph 7.1.1.

It is then highly advised to carry out a bake out during minimum one night to purge all the columns and to rebalance the column with the use of a new gas.

Don't forget that a bad carrier gas configuration can irremediably damage the detector.