OLIS
Online Liquid Injection System
for gas chromatograph

User manual

SRA INSTRUMENTS
ANALYTICAL SOLUTIONS

OLIS valve installed on Intuvo 9000 GC

Version 2.1

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:

Hotline: +33 (0)4 78 44 22 09
E-mail : service@sra-instruments.com

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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 is designed for use in very specific conditions. 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 OLIS valve 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 OLIS 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.

In particular, the flash heating stem must not be used while the OLIS controller is turned ON.

Warning: Hot surfaces

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

These parts include, among others:

- Needle interface

You must be extremely careful to avoid touching these heated surfaces. Do not use the instrument if the OLIS module is disassembled.

Warning: Moving parts

Many parts of the OLIS move during operation. Do not touch the OLIS valve while the power is on; make sure that you have unplugged the power cord.

These parts include, among others:

- Stem and motor

You must be extremely careful to avoid injury to your fingers.

Warning: Electrostatic discharge is a threat to electronics

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

Warning: Use of gases

A mixture of gases can lead to an explosion. Carefully use hydrogen as carrier gas. You must perform a leak test and have a hydrogen detector.

2.2 Before starting

- 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.
- Have your instrument serviced by SRA Instruments.
- Use only gases and solvents specified in the operating procedures.
- Do not open the valve if the temperature is above 50°C.
- Do not open the electronic box.
- Eliminate from the environment of the instrument: vibrations, magnetic effects and explosive gases.
- The OLIS must be used indoors only; it is designed for use at room temperature and under conditions where no condensation can occur. Install the OLIS and the GC on a rigid and stable surface.

3. Description

3.1 Presentation

The OLIS valve is an online liquid injection system for gas chromatography, developed by SRA Instruments. It can accept samples up to 60 bars and can be installed on all GC models, online or in laboratory, equipped with split/splitless injectors.

Designed for the introduction of liquid samples under pressure which may contain very polar, high boiling point or high viscosity analytes, OLIS is mainly used for online analyses in pilot plants, solvent plants or for liquids under pressure in a canister coupled to a gas chromatograph.

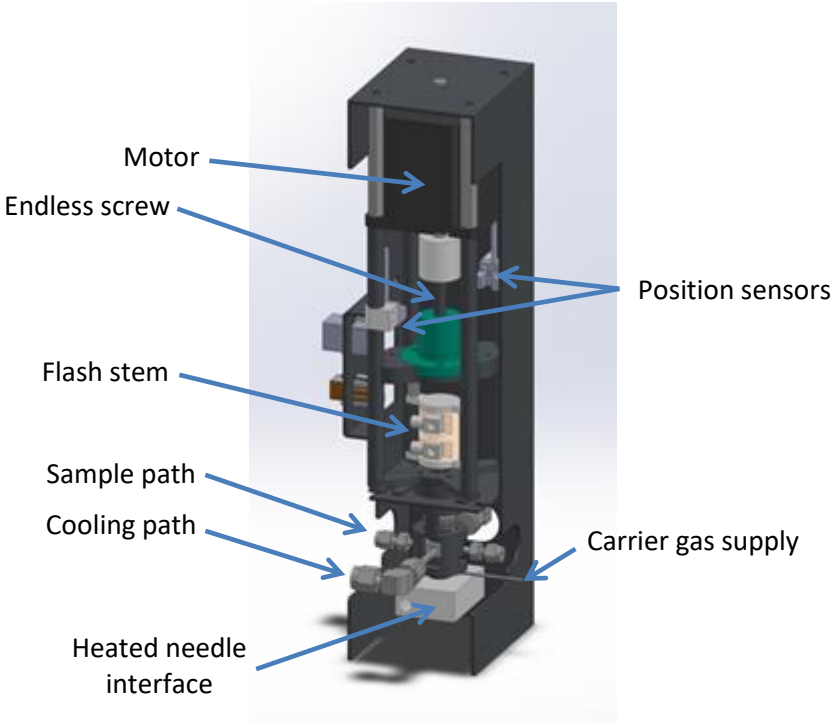
OLIS is designed to obtain directly online results similar to those obtained with laboratory chromatographs. In comparison, the other advantage is that it minimizes the user's contact with the liquid sample since there is no need to collect the sample and bring it to the laboratory.

3.2 Conception

The OLIS valve consists of:

- A motor
- An endless screw
- A flash stem
- A sample path chamber
- A cooling path chamber (optional)
- A carrier gas supply
- A needle connection through the septum of the GC (Split/Splitless injector)

The stem has its lower end machined to allow sample circulation.



The valve is controlled by the **OLIS controller**. It is an electronic interface including embedded software.

Power and alarm lights are present on the front panel.

3.3 Principle of operation

The valve uses an actuator which is driven by an electric motor from its upper position (filling stage) to its lower position (injection stage).

The sample flows continuously through the valve even when the stem is in the injection position. In the sampling position, the (fixed) volume of the stem fills with the sample. When the GC starts, the stem goes down, vaporizing the sample in the carrier gas at high temperature (350 °C in 1 to 2 seconds), in order to optimize the vaporization and injection processes. The sample, via a heated needle, then goes into the split/splitless injector to which the separation column is connected.

OLIS ensures complete heating of the sample after injection. The vaporization chamber has independent heating to avoid cold spots. A specific Teflon seal pair for high temperatures is necessary to ensure a constant tightness even after a high number of injections.

The independent heating zone is doubled by a safety device that cuts off the power if the threshold is exceeded. The alarms on the front panel are then active and it is recommended to contact the after-sales support in this case.

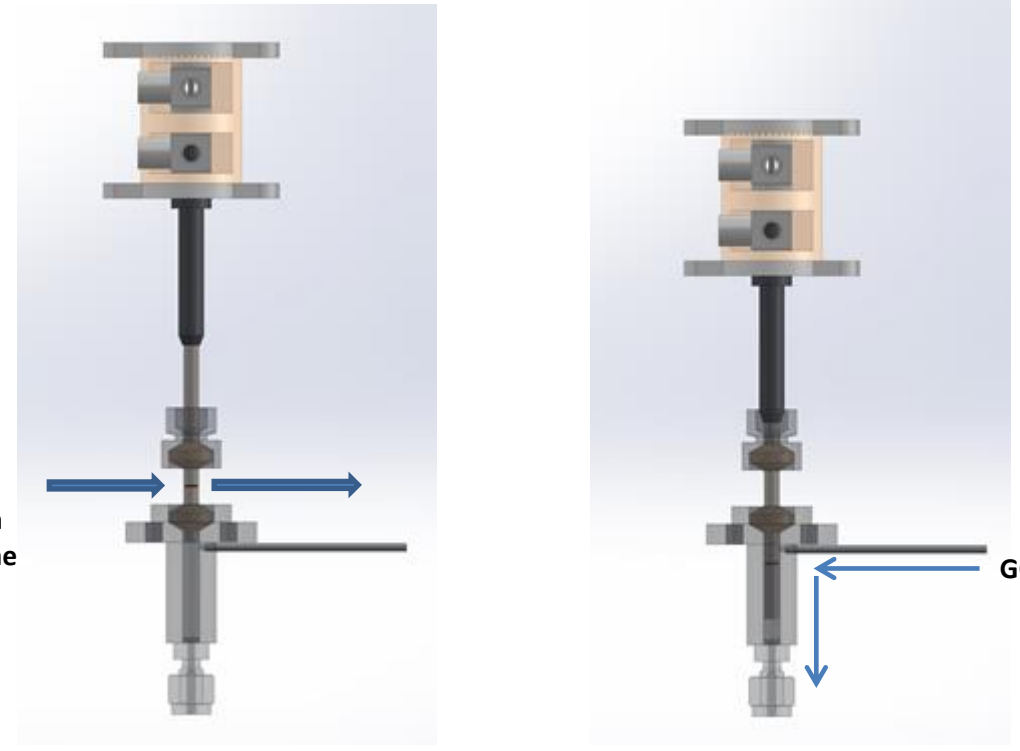
Notes:

- For calibrations for example, you can easily remove the OLIS valve from the GC injector and use your standard GC injection port without disassembling parts and fittings.

- Optionally, it is possible to cool the sample flow path to avoid internal vaporization generated by the temperature of the GC injector.

Stem in sampling position

Stem in injection position



3.4 Embedded software

The software was developed to communicate with the OLIS controller, which controls the injection system, via an embedded internet server. The control parameters (STEM temperature, STEM position at injection, injection counter) and diagnostics are therefore available on web pages.

Access to the interface requires only a LAN connection and a compatible web browser (Internet explorer, Firefox, Chrome).

The OLIS controller can also be used offline, with predefined parameters during manufacture or after LAN configuration. Closing the web browser does not stop the valve method and does not cancel either the settings or the heating, so you can perform an injection.

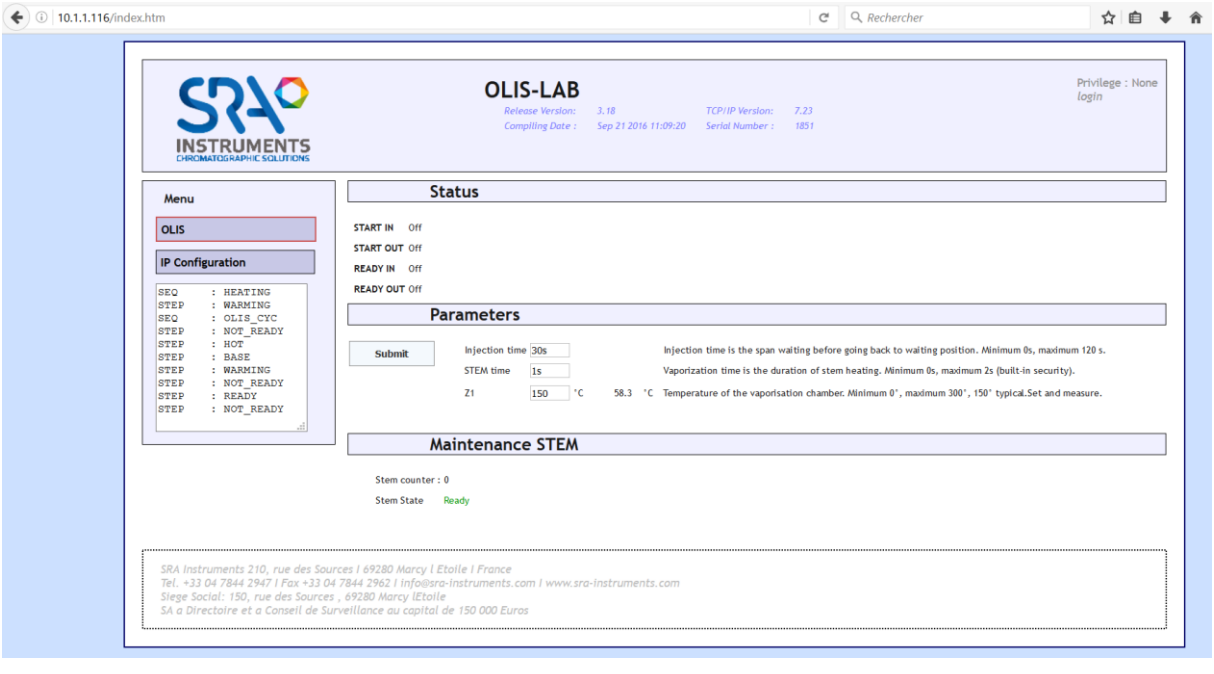
The software controls the safety of the instantaneous heating and allows to adapt the heating temperature for specific applications.

IMPORTANT: Each time the controller is turned on, the software automatically starts heating the vaporization chamber.
When the temperature is reached, the valve automatically moves to the sampling position in case the stem is not in the correct position after maintenance.

Default IP configuration:

Host Name :	OLIS-XXXX (XXXX= Serial Number)
IP Address :	10.1.1.116
Gateway :	10.1.1.1
Subnet Mask :	255.255.255.0
Primary DNS :	10.1.1.1
Enable DHCP :	enables the auto-attribution of an IP address from a DHCP server
Enable Auto-IP :	enables the board to change IP itself in case of conflict.

Visualization of the interface :



3 parameters are available:

- Injection time: the valve remains in injection position typically for 30 seconds after injection.
- The STEM time: typically, this is 1 second to reach at least 300 °C.
- Z1: the temperature of the vaporization chamber for transferring the sample to the split/splitless injector, which is typically 150 °C.

4. Installation

4.1 Preparation for the installation of equipment

OLIS installation is carried out by a SRA Instruments technician or a recognized partner.

To be able to set up the equipment in your laboratory, it is necessary to provide:

- A 240 VAC or 110 VAC power supply depending on the model - 6 A
- Approximately 30 cm wide on the left side of the GC
- Approximately 60 cm wide between the controller and GC input (for valve cables)
- Usually, the OLIS is installed on a split/splitless injector.

Tools required:

- 2 wrenches 5/16"
- 2 wrenches 7/16" (for cooling sample path option)
- 1 electronic leak detector
- 1 electronic flowmeter

Accessories delivered with OLIS:

- 3 hex screwdrivers 1.5 mm, 2 mm and 3 mm respectively
- 1 needle key 8 mm
- 1 wrench 16 mm
- 1 Torx T10 angled screwdriver and 1 Torx T20 screwdriver

Accessories supplied with the controller:

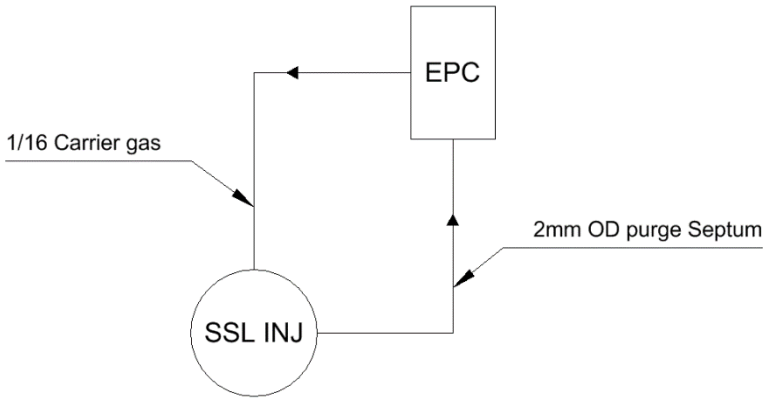
- Ethernet cable
- Power supply cable

4.2 Modification of the GC carrier gas circuit

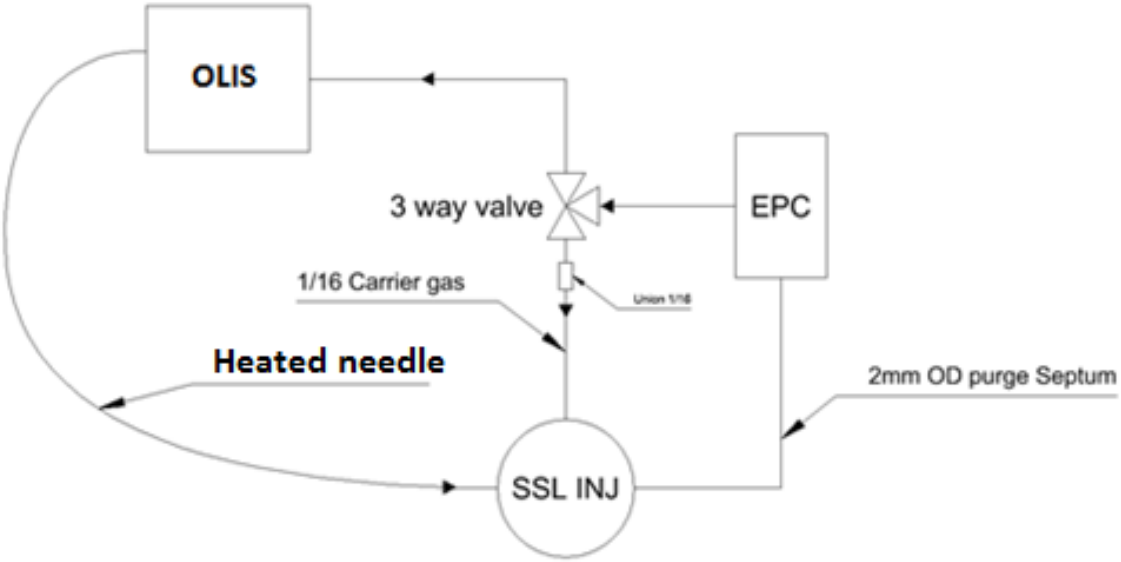
In order to supply the OLIS with carrier gas, a 3-way valve will be mounted on the GC. This valve allows switching the carrier gas:

- Via the transfer line, allowing injection with the OLIS.
- Directly to the injector for standard use of the GC. It is also possible to isolate the OLIS for maintenance.

GC standard circuit



Modification of the circuit for OLIS



The carrier gas connection to the OLIS valve is located on the rear panel. The connection is Swagelok 1/16" type. We recommend that you use new ferrules during installation and follow the recommendations of the fitting manufacturer for tightening in order to ensure the best sealing. A leakage control with electronic detector is recommended periodically to check the absence of leakage that can degrade the analysis and create carrier gas overconsumption.

The use of hydrogen as carrier gas for OLIS is possible but not recommended.
It is preferable to use helium for safety reasons.
If hydrogen is to be used as carrier gas, you must perform a leak test and use a hydrogen detector for safety reasons.

4.3 OLIS needle

To avoid breaking the needle during transport, it is not connected to the valve base before shipping. You must connect it when received before installing the valve. The needle is included in the GC installation kit, ready to use and including the ferrule.

4.4 Installing the valve on the GC using the installation kit

The OLIS valve is a product whose standard design allows adaptation to any GC. To install the OLIS valve on a GC you need to order the correct installation kit to ensure compatibility.

The kit always contains:

- parts for modifying the connection of the carrier gas: union, ferrules, tubes and the 3-way valve.
- a support base adapted to the type of GC
- a needle guide nut adapted to the GC
- a remote cable for OLIS adapted to GC

Refer to the specific kit installation manual or contact SRA Instruments for more information.

4.5 Installing the valve on an Agilent 7890 GC

BEFORE MODIFICATION

Be sure to correctly identify the carrier gas tube before cutting.
Do not cut the tube of septum purge (connected to the small chimney).

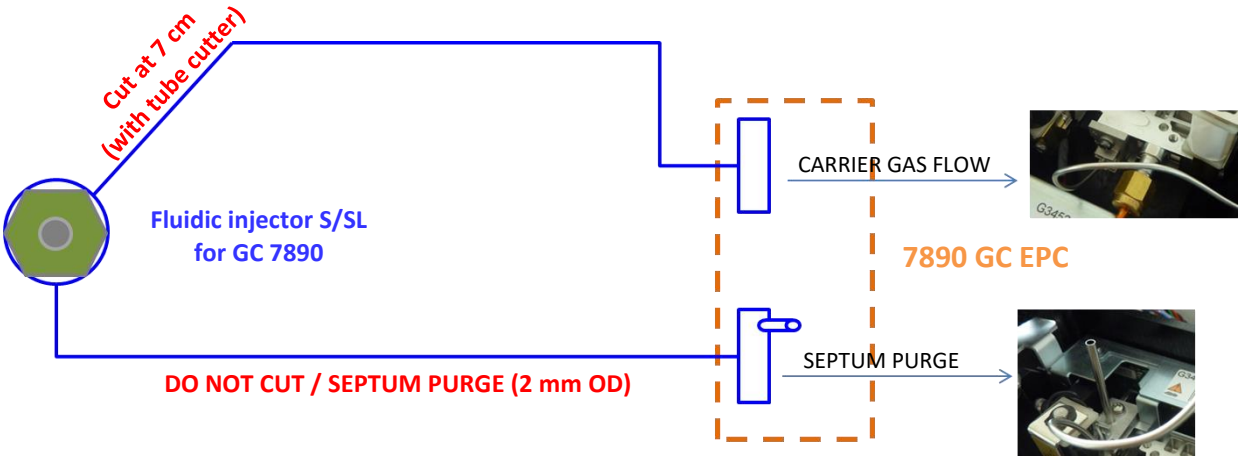


Fig. 1 - Diagram of Agilent GC standard S/SL EPC before modification for mounting the OLIS valve. According to § 4.2 of the manual, the path of the carrier gas must be modified to pass through the OLIS valve.

AFTER MODIFICATION

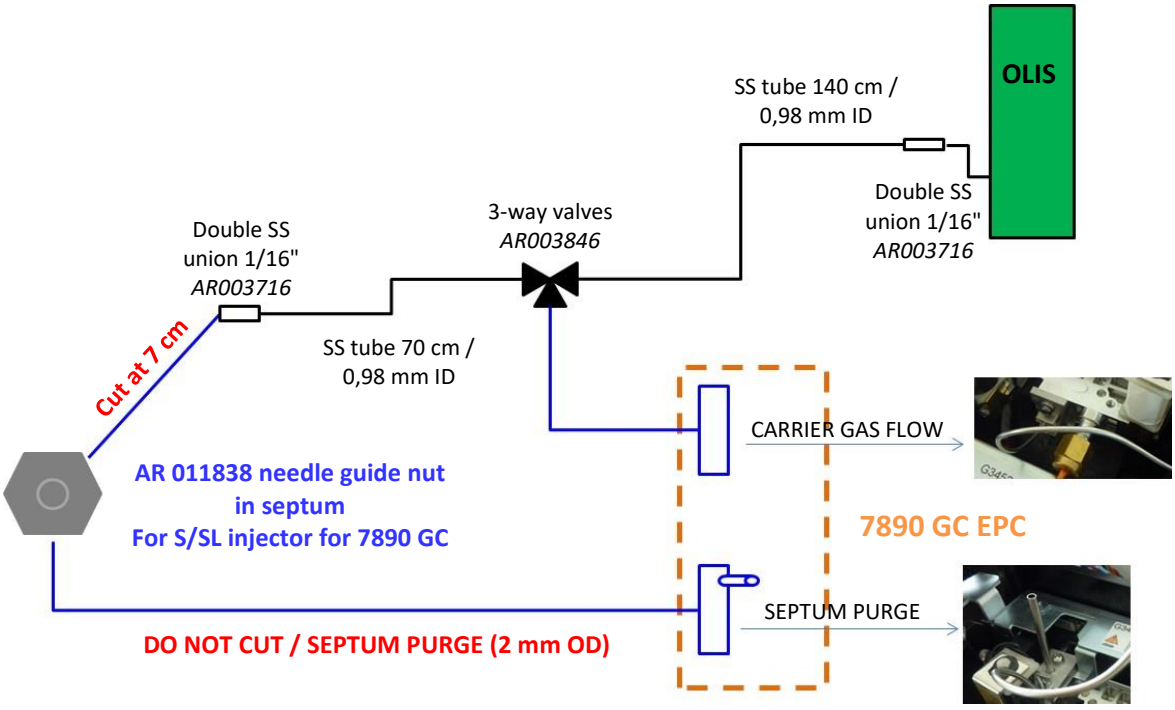


Fig. 2 - Diagram of Agilent GC standard S/SL EPC after modification for mounting the OLIS valve. The 3-way valve allows to quickly change the path of the carrier gas in order to use either the OLIS valve with the GC or the GC alone in manual or automatic injection.
If you wish to use the GC alone, be sure to remove the OLIS valve from the S/SL injector before turning the 3-way valve.

5. OLIS controller

5.1 Description

The function of the controller is to accompany the integration of the OLIS injector on the GC for safe use.

The controller powers and checks the OLIS status, and triggers an alarm in case of excessive internal temperature. In this case, safety measures are taken:

- Heating of the OLIS base is switched off (heated and temperature controlled by the controller).
- STEM heating capacity is switched off.
- The electric motor control is stopped.
- A visual alarm (LED) is triggered and the output contact is closed when the status is not ready.

In case of an alarm, the controller must be reinitialized by an operator using the integrated software. The initialization will only be activated if the threshold temperature is below the limit value. In this way, any anomaly, even brief, will be listed by the operator.

For this, it is necessary to initialize the system. The integrated software will automatically apply the method and attempt to move the STEM to the sampling position within minutes.

Some errors are critical (stem, heating system) and it is necessary to stop the controller in order to clear the defects.

The controller consists of:

- ✓ An indicator light that lights up on the front panel, corresponding to the injection control resulting from the start of the GC.
- ✓ On the rear panel, a REMOTE connection for starting the analysis from the GC (start in GC) and one for the ready/not ready state of the OLIS valve transmitted to the GC (ready out to GC).
- ✓ External remote I/O on rear panel for specific applications and needs.

The LED on the front panel indicates the OLIS status:

off	OLIS is OFF
	GREEN: OLIS is ready and idle OLIS waits for Start and/or GC ready
	GREEN blinking: OLIS is running
	RED/GREEN blinking: OLIS is not ready or in stabilization. The external START will not be taken into account.
	Fast blinking: Cycle error (a physical parameter such as temperature didn't behave as expected). Check the web page for more information.
	RED: OLIS is frozen due to a software error. Physical reset is mandatory.

This controller includes electronic control of the OLIS and control of the electric motor used to move the STEM.

To function, it requires:

- A 240 VAC or 110 VAC power supply depending on the model - 6 A
- Connections provided to connect the controller to the OLIS valve
- A REMOTE cable between the GC and the OLIS box (included in the installation kit)

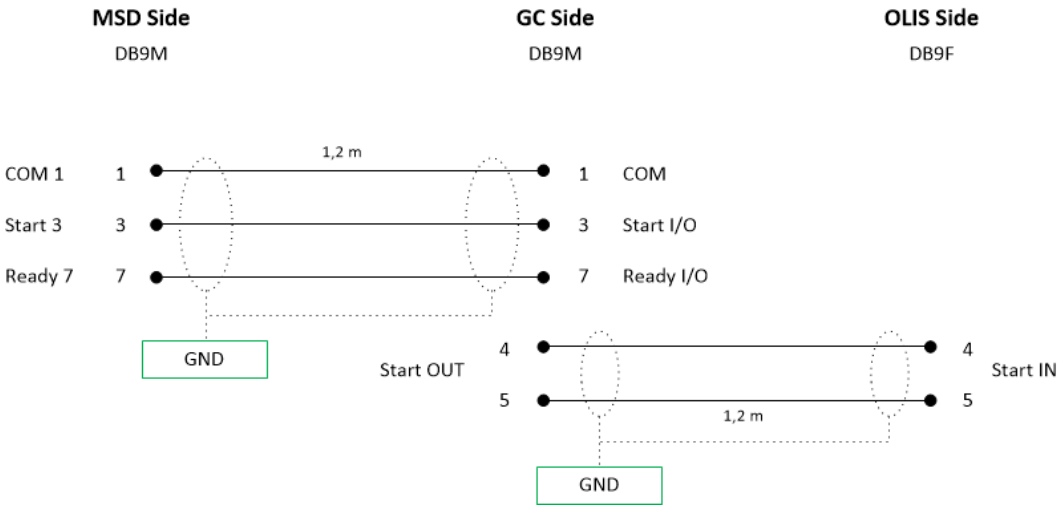
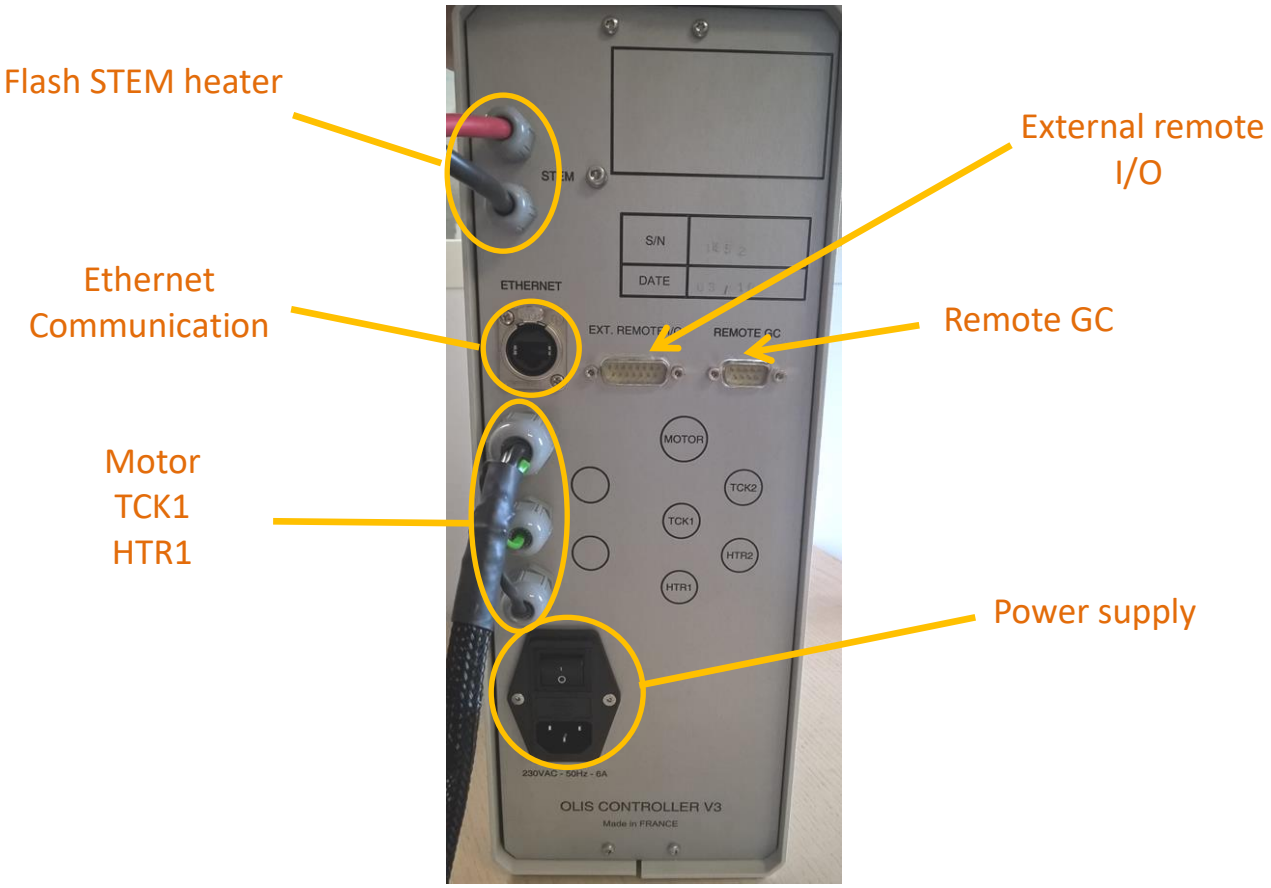


Fig.3 – Example of a wiring diagram between OLIS, Agilent 7890 GC and Agilent 5977 MSD.

5.2 Installation

1. Install the controller to the left of the GC. If you use a mass spectrometer, you must place it to the right of the GC as it must be close to the valve, with the STEM flash heating cables measuring about 1 m.
2. Connect the motor and controller heating cables to the OLIS valve.
3. Connect the power supply of the STEM heating system to the OLIS valve. (Red high position and black low position).
4. Connect the GC remote control cable.
5. Connect the external remote I/O if necessary.



5.3 Sample flow path and vent

The sample flows to the vent through a 1/16-inch tube located on the bottom of the valve. In the case of a toxic sample, it is important before use to extend this outlet to a common vent or hood to protect anyone in the environment of the instrument.
For a better functioning of the interface, it is recommended to connect this outlet at constant pressure and if possible, not to break the flow through the valve.

6. Maintenance operations

6.1 Maintenance schedule

6.1.1 Every month

- Check the glass liner state in the GC injector. Change the septum if necessary.

6.1.2 Every 6 months

- Check the needle state. Change the OLIS Teflon seals.

6.1.3 In case of GC carrier gas shutdown

- The needle interface and Teflon seals are the main elements in contact with the GC carrier gas. In case of leakage or pressure drop of the carrier gas, new parts must be installed.

Replacement as soon as necessary:
OLIS performance is strongly linked to GC condition. Perform GC maintenance as recommended by the manufacturer.

6.2 Changing the needle, stem, and Teflon seals

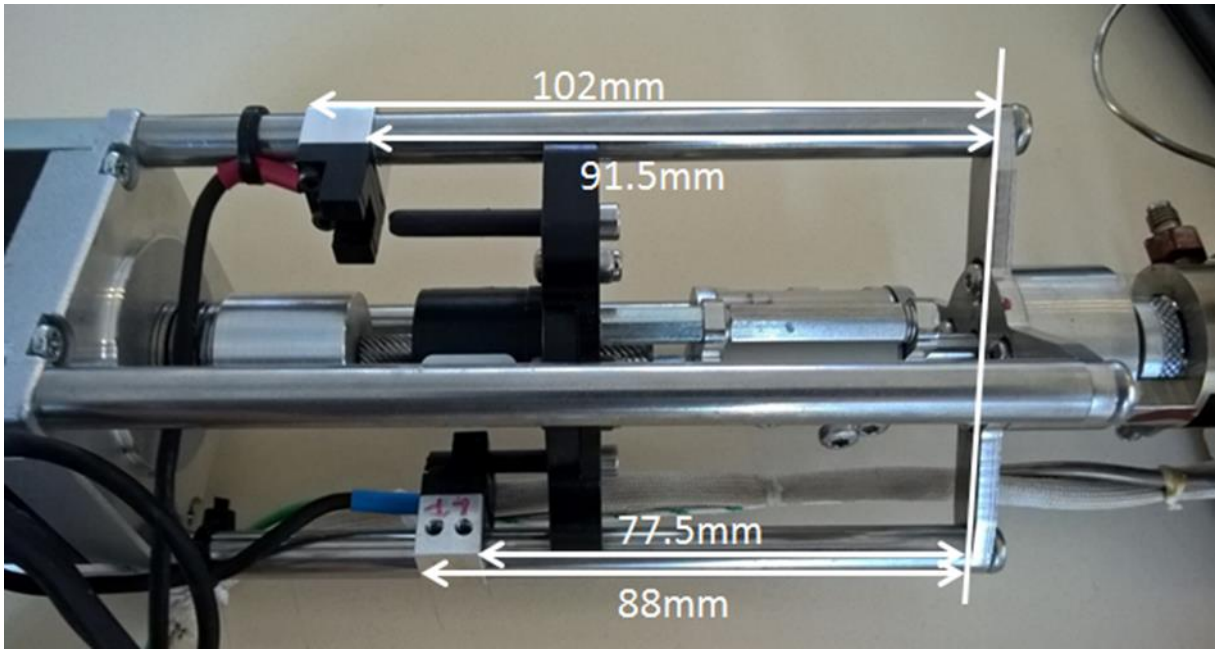
To change the needle, stem, and Teflon seals, refer to the maintenance manual that covers the lower part of the OLIS valve. These operations can be carried out directly on site by any trained technician.

Code	Designation		Minimum qty
AR007424	High T°C seal (200 – 450 °C) (1 unit)		2
AR000351	Direct liner 1,5mm ID (Agilent GC only) (1 unit)		1
AR010722	Interface needle N (Agilent GC only) (3 units)		1 set

6.3 Changing the motor and sensors

To change the motor and sensors, refer to the maintenance manual that covers the upper part of the OLIS valve. These operations are carried out by a trained reseller partner or directly by SRA Instruments by returning the device.

6.4 Position of the sensors/high and low positions of the valve



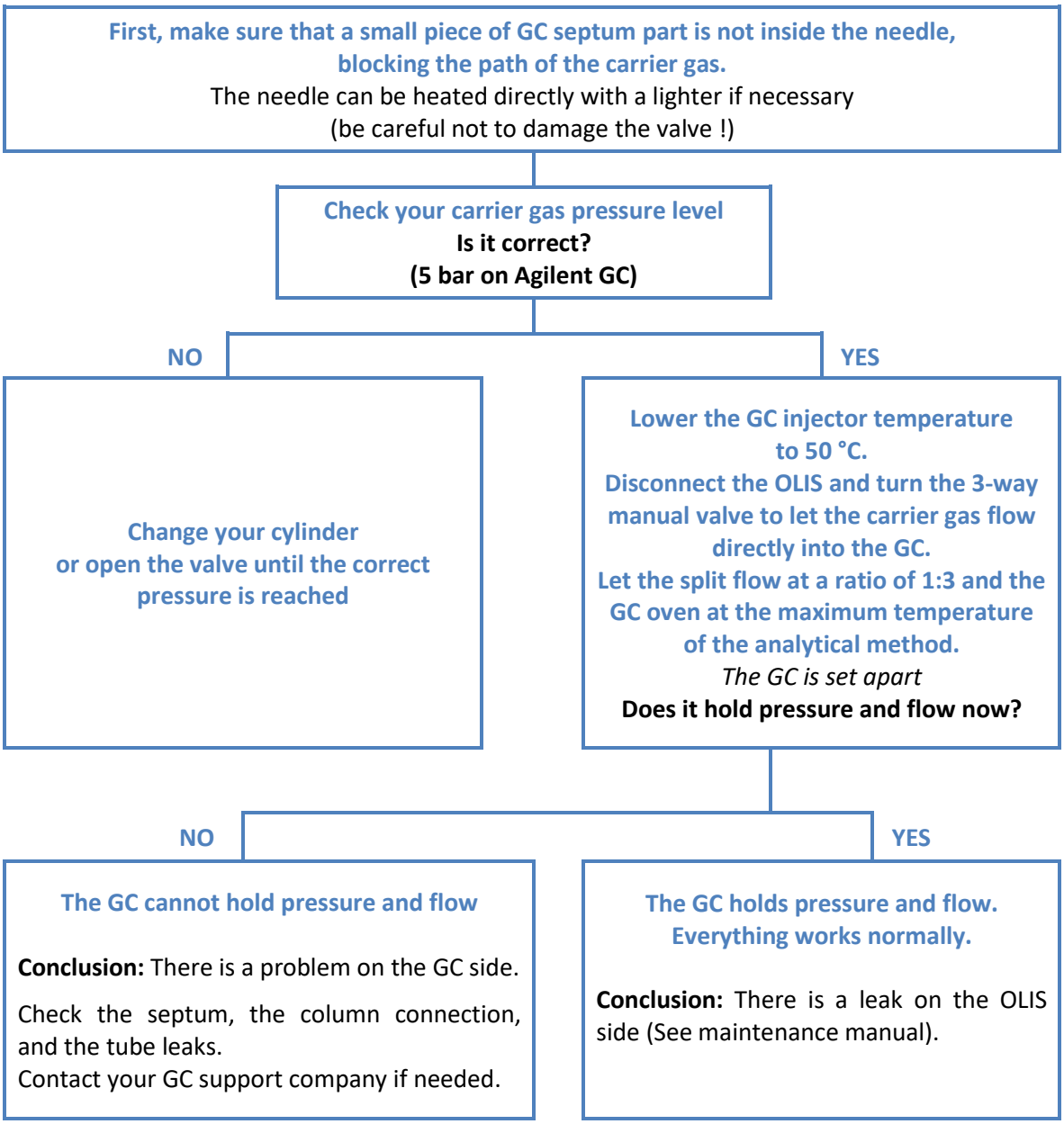
The position of the sensors is important for loading the sample and injecting it into the GC carrier gas.
The position of the sensors can be easily changed using a 1.5 mm hex screwdriver.
Make sure not to over-tighten the screw to avoid any problems and check the position of the sensor in relation to the high and low rods.

6.5 Flow/pressure problem with GC

Find the origin of the problem, for this:

- Lower the injector temperature and oven temperature below 50 °C.
- Let the split flow at a ratio of 1:3.
- Lower the OLIS temperature to 50 °C.
- Make sure that the 3-way manual valve is in the OLIS position.

Contact SRA Instruments or your reseller for more details on the next steps.



7. Technical data

7.1 Power supply

Power supply input: + 240 VAC
Power supply output (motor, stem heating...): + 24 VDC
Power consumption, max. 2.5 A / 240 VAC

7.2 Dimensions and weights

Valve:

- H 290 ; D 120 (150 mm including in/out sample tube) ; W 120 mm (150 mm including in/out cooling tube)
- 2 kg

Controller:

- H 370 ; D 300 ; W 140 mm
- 10 kg

7.3 Utilities

Power: 220-240 VAC; 1000 W max.

The GC requires a split/splitless inlet, remote start-in, remote ready-out.

The PC requires Windows 7 or higher, Ethernet connection.

7.3.1 Carrier gases

Compatible with helium, nitrogen, and argon, with Swagelok fittings.

7.3.2 Repeatability

Typical RSDs at constant temperature and pressure: ≤ 1%.
The sample must be liquid in the OLIS sampling chamber.

7.4 Fuses

Presence of 2 fuses on the back of the instrument at the bottom right.

220 VAC Instrument

- Fuses T4AH, (250 VAC)

110 VAC Instrument

- Fuses T6.2AH, (110 VAC)

To change them, turn off the power and unplug the power cable.

7.5 Inputs/Outputs

DB9 connector on the side of the OLIS control unit.

1-3 pin start out
1-7 pin ready out
4-5 pin start in
1-9 pin ready in

In most cases, "OLIS valve is the slave and GC the master". In this case, start in and ready out are used.

OLIS receives GC startup using the 4-5 pin start in. It gives the GC the status ready/not ready by using the 1-7 pin ready out. The GC detects whether the OLIS valve is ready or not; then it can start the valve and GC analysis begins.

7.6 Recycling

Do not throw away this equipment. Contact a competent recycling organism.

8. 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

OLIS Online Liquid Injection System	

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 purposes"

2/ Directive 2014/30/EU – Electromagnetic compatibility

- 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 21**

Marcy l'Etoile, 23 November 2020

Legal representative,
Armando MILIAZZA