Storage Interface IST16

User manual

Version 8.3

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 the responsibility of the customer to inform SRA Instruments after-sales service if the IST16 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 IST16 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 IST16 work at temperatures high enough to cause severe burns.
These parts include, among others:

- the transfer lines
- the valve oven

You must be extremely careful to avoid touching these heated surfaces. Do not use the instrument if the IST16 module is disassembled.

Warning: Electrostatic discharge is a threat to electronics

Electrostatic discharge (ESD) can damage the printed circuit boards of the IST16. 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 For experiments

- 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 or one of its distributors.
- Use only gases and solvents specified in the operating procedures.
- Do not open the oven if the temperature is above 100 °C.
- Do not open the electronic box.
- Eliminate from the environment of the instrument: vibrations, magnetic effects and explosive gases.
- The IST16 must be used indoors only; it is designed for use at room temperature and under conditions where no condensation can occur. Install the IST16 on a rigid and stable surface.

2.3 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 IST16 complies with the legislation for all aspects of electrical safety.

3. Description

3.1 Presentation

The IST16 is an interface for storing gases, developed by SRA Instruments. It is generally coupled to thermogravimetric balances from different manufacturers (Mettler, Setaram, Netzsch, TA ...). Thus, the IST16 makes it possible to recover and isolate gas fractions resulting from the thermal analysis with a sequence defined by the user. These fractions can then be injected separately into GC-MSD for a detailed analysis. Thanks to IST16, the duration of the analysis is no longer a limitation for the study of TGA profiles.

3.2 Principle of operation

The interface consists of:

1. An upper box containing the oven integrating the 3 valves constitutive of the system
2. A lower box containing the entire electronic control unit for gas circulation
3. A 1.2m transfer line for interfacing to the TGA
4. A 1.2m transfer line for interfacing to the Split injector of the GC system
5. A touch interface

The IST16 provides full heating of the sample from the thermo-balance to the GC. It has 2 independent heating zones to maintain the temperature up to 250 °C.

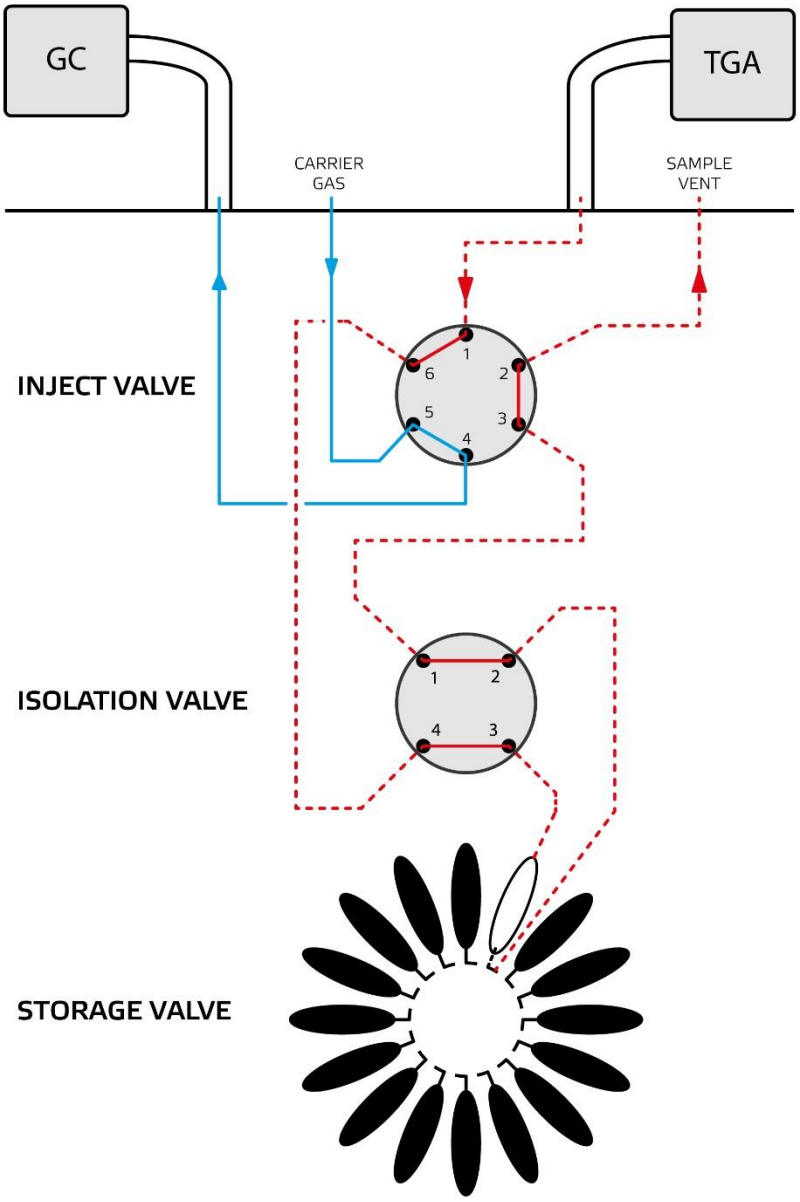
Each of these 2 heating zones is coupled with an independent safety device which switches off the power if the threshold is exceeded. The alarms on the front panel are then active and it is recommended to contact the after-sales support in this case.

3.3 Fluid flow diagram

The IST16 is equipped with 3 high temperature switching valves. The 16 loops of the storage valve have a default volume of **250 µL** and have an external tube diameter of 1/16 inch.
The INOX used has a Sulfinert®-type treatment.

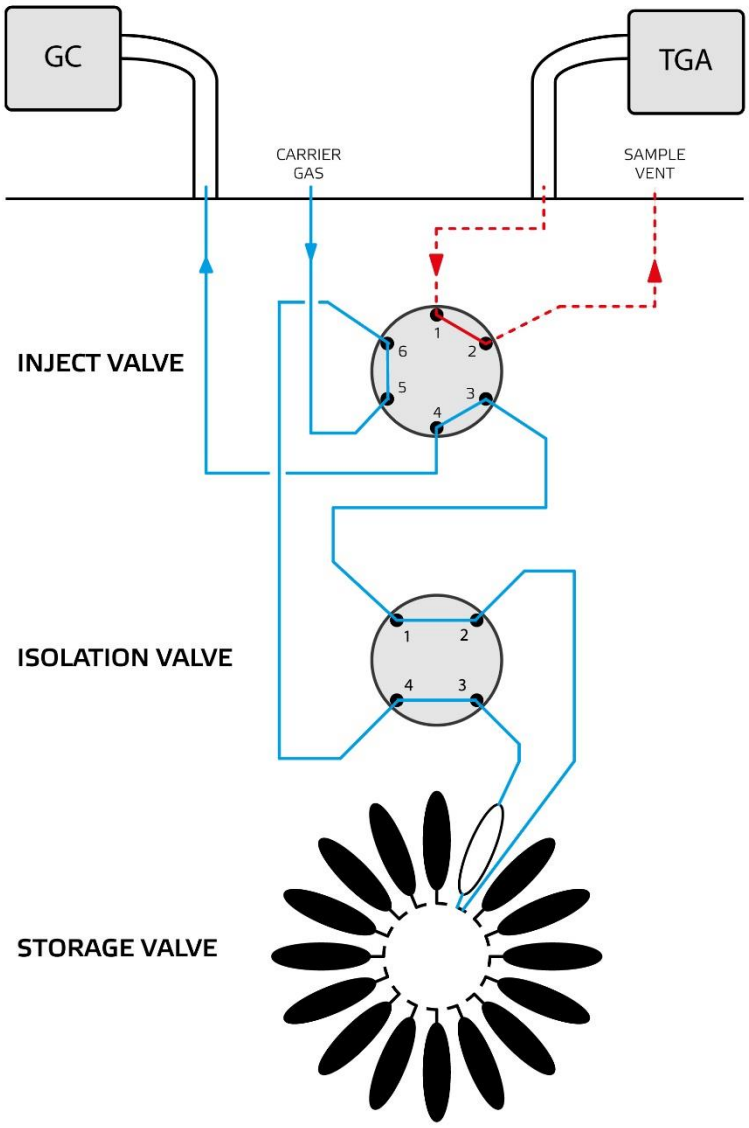
The fluid flow diagram is as follows:

3.3.1 Storage mode



This storage mode is not available on IST1. For this latter, refer to appendix IV chapter 17.

3.3.2 Injection mode



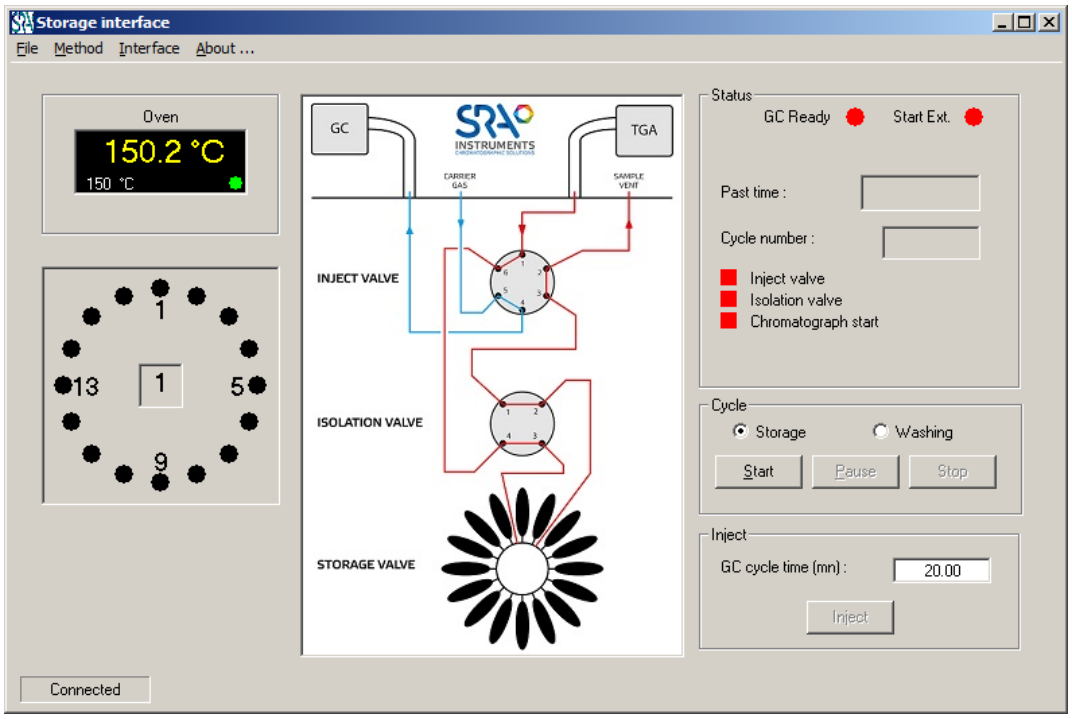
This injection mode is not available on IST1. For this latter, refer to appendix IV chapter 17.

3.4 Automated control software

The IST16 software has been developed to control the IST16 storage interface.

The storage sequence can trap up to 16 samples.

The injection sequence then allows injecting these samples into a chromatograph (optionally coupled to a mass spectrometer) in an automated way.



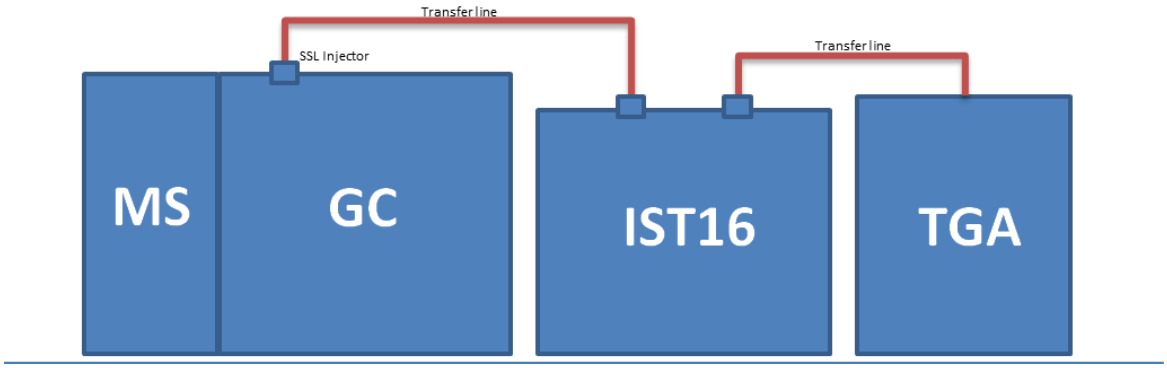
This software is optimized for operation under Microsoft Windows 7 or 10.

4. Installation

4.1 Preparation for the installation

The installation of the IST16 is performed by an SRA Instruments technician or a recognized partner. To be able to set up the equipment in your laboratory, it is necessary to provide:

- 1 power supply 237 VAC – 16 A
- Approximately 40 cm wide on the right side of the GC
- Usually, the interface is placed between the analytical system and the Thermo-balance:

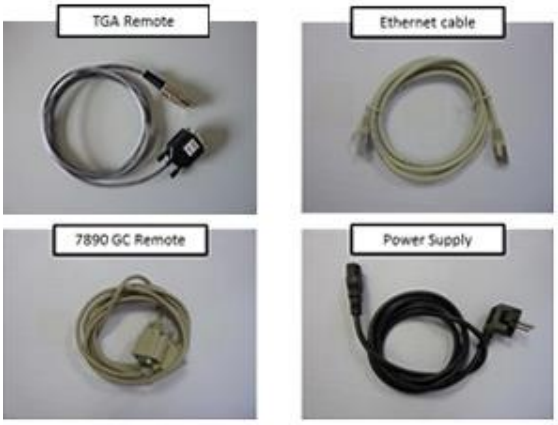


Tools:

- 1 wrench 7/16'' and 2 wrenches 5/16''
- 1 wrench 5 mm
- 1 Torx T20 screwdriver and 1 Torx T10 screwdriver
- 1 electronic leakage detector
- Allen key 9/64'' and Allen key 7/64''
- 1 electronic flowmeter

The following cables are supplied with the IST16:

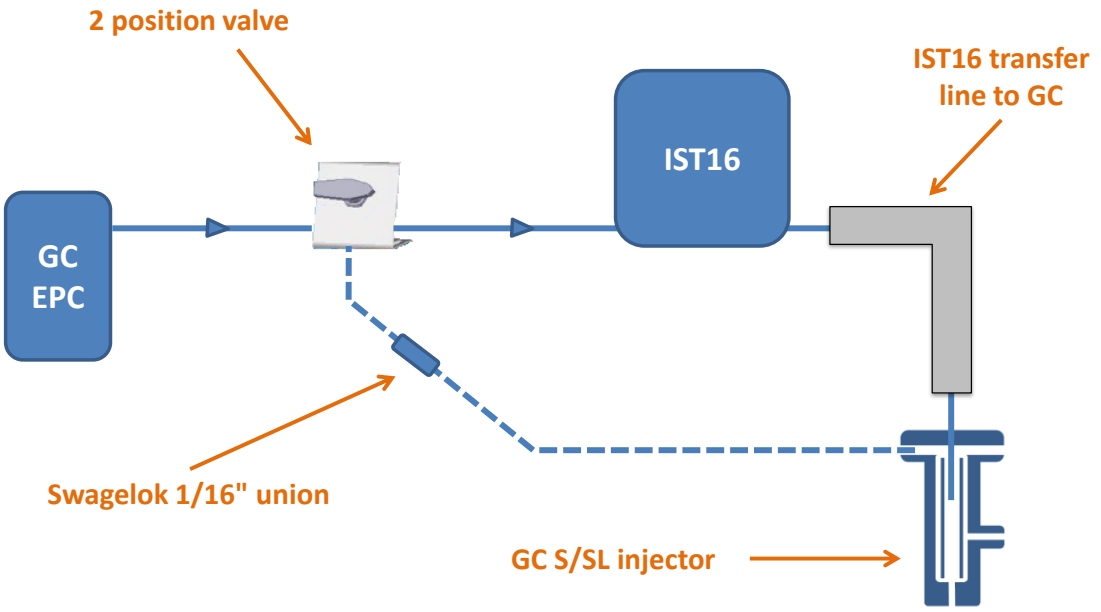
- TGA remote
- Ethernet
- GC remote
- Power Supply



4.2 Modification of the GC carrier gas system

In order to supply the IST16 with carrier gas, a 3-way valve will be mounted on the GC. This valve allows switching the carrier gas:

- Through the storage interface for the TGA/IST16/GC-MS mode, allowing injection with the IST16.
- Directly to the S/SL injector for standard use of the GC. It is also possible to isolate the storage interface for maintenance.

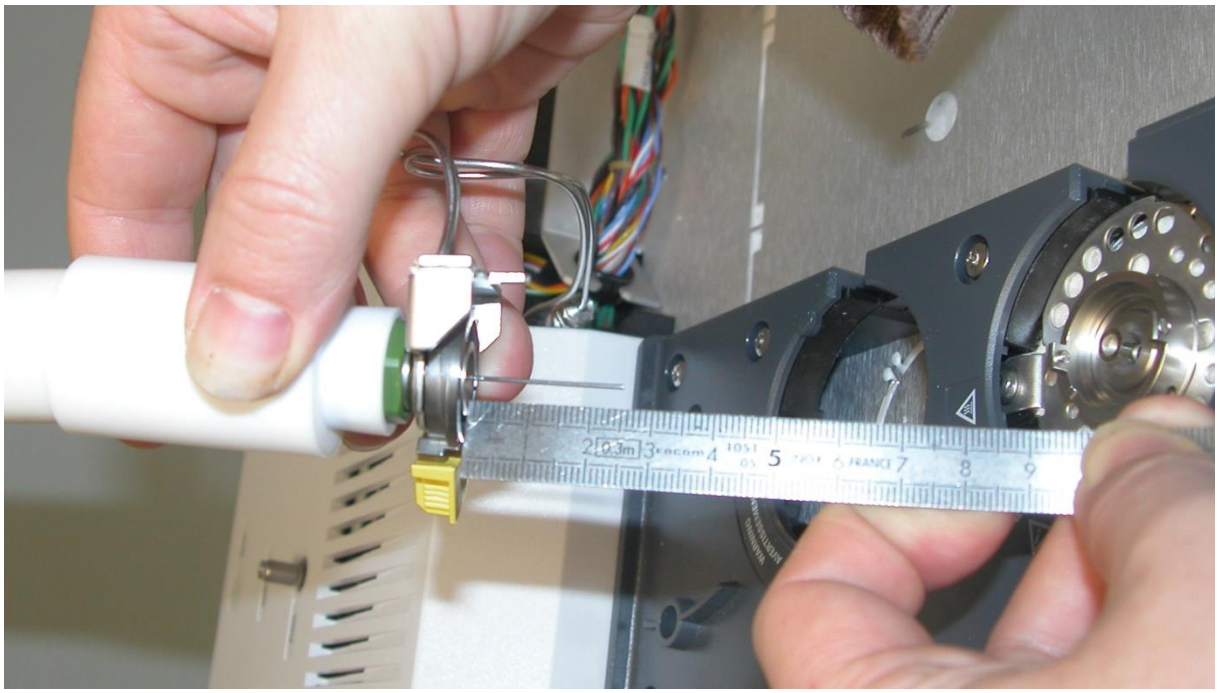


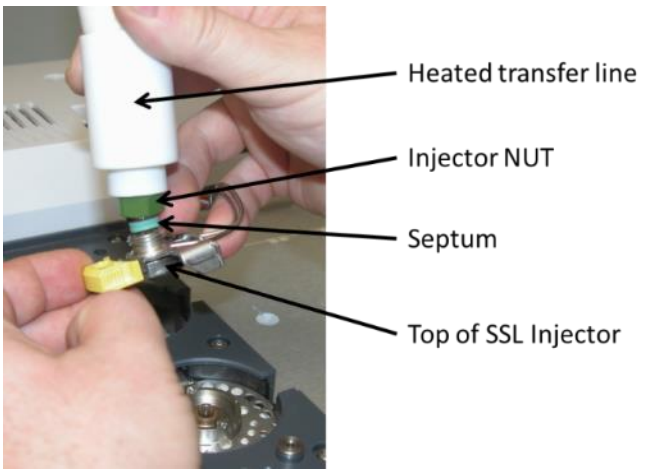
The carrier gas connection to the IST16 is located on the back of the upper panel. The fitting is 1/16" Swagelok type. We recommend that you use new ferrules during installation and follow the coupling manufacturer's recommendations for tightening to ensure the best seal. A leak control with electronic detector is recommended periodically to check for leakage that can degrade the analysis and create overconsumption of carrier gas.

The use of hydrogen as a carrier gas for IST16 is possible.
If the choice is available, it is preferable to use helium for safety reasons.
If you use hydrogen as a carrier gas, you must first perform a leak test and use a hydrogen detector for safety reasons.

4.3 Installation of the capillary on the GC injector

The transfer line on the Split/Splitless injector side is fixed by a bracket on top of the GC. This transfer line contains an inert stainless-steel capillary with an outer diameter of 0.8 mm. This capillary must pass into the injector to a depth of about 25 mm with respect to the septum nut.





We recommend to possibly put a mark to adjust the depth once the line is in place.

4.4 Installation of the transfer line on the TGA

The transfer line is normally supplied with the connection corresponding to your TGA model. This can be a DN16 flange (Mettler case) or a simple double ring fitting (TA, Setaram cases...).

We recommend the use of a transfer tube of 1/8" diameter.

Depending on the type of connection and type of thermo-balance, it may be desirable to mechanically maintain the transfer line in order to not disturb the TGA or risk breaking any fragile parts composing its oven. We can assist you in finding a support adapted to your conditions of use. Do not hesitate to contact us for any special requests.

To find out how to install the IST16 transfer line on your equipment, please refer to the corresponding appendix in the dedicated installation kit.

4.5 Vents

The sample vent is located on the upper back side of the instrument.

⚠ **It is recommended to connect this vent to a conventional vent or a hood to prevent the emission of toxic or odorous compounds in the immediate environment of the instrument.**
In all cases, the IST16 vent must remain at atmospheric pressure in order to not disturb the gas flow through the interface.

5. Installation and configuration of the IST16 software

5.1 Software installation

1. **First, make sure that the computer is configured with Administrator rights.**
2. Insert either the USB key or the CD-ROM provided and use the Windows Explorer to display the files contained. Select the file **Setup.exe**, right-click on this file and select the menu **Run as Administrator**.
3. A welcome window is displayed; click on Next.
4. Then accept the terms of the license agreement so that you can go on with the installation.
5. In the window “Destination location”, select the folder where the application files will be stored by entering its name or by clicking on Browse.
6. In the window “Select start menu folder”, select the folder where the software shortcut for launching the application will be stored by entering its name or clicking on Browse.
7. In the window displayed, check the box if you want an icon to be created on your computer desktop.
8. Click on the Install button to complete the installation.
   Before launching the entire installation of the application, you can check the data entered by clicking on the Back button and correct any mistake.
9. In the new window, check the box “Launch IST16” to launch the application as soon as you exit the installation.
10. Click on the “Finish” button to close the installation of the application.

5.2 Configuration of the IST16

The dialogue with the IST16 uses an Ethernet connection and it is necessary to indicate the IP address to the software. This configuration is accessible through the **Interface\Configuration\IP Address** menu.

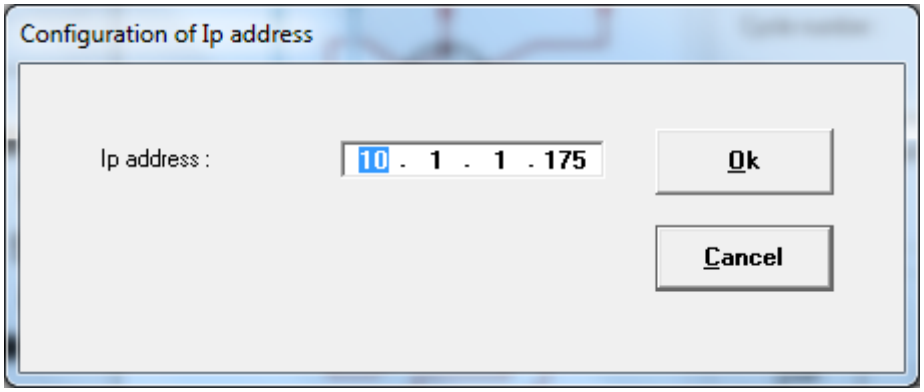
If a configuration has already been carried out, this menu can be accessed with a password. In this case, the following login and password must be entered:

Login: **Config** Password: **ist16**

⚠ **The advanced configuration of the IST16 must not be used by people not trained.**

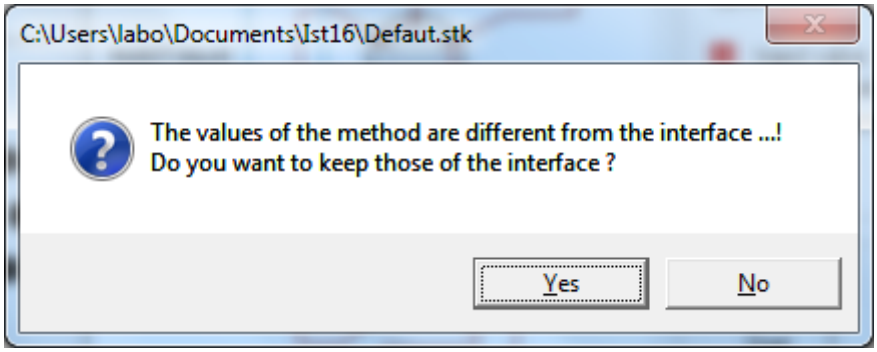
When you have entered the password, select the **Interface\Configuration\Ip address** menu.

The following window is displayed:



Enter the IP address and validate with Ok; the software automatically tries to connect to the interface.

When connecting to the IST16 and if a method has already been loaded, the following message may be displayed:



This message means that the parameter values of the IST16 method are different from those of the method loaded in the application.
If you click Yes, the application method parameters will be cleared and replaced by the IST16 method parameters.
If you click No, the application method will not be changed.

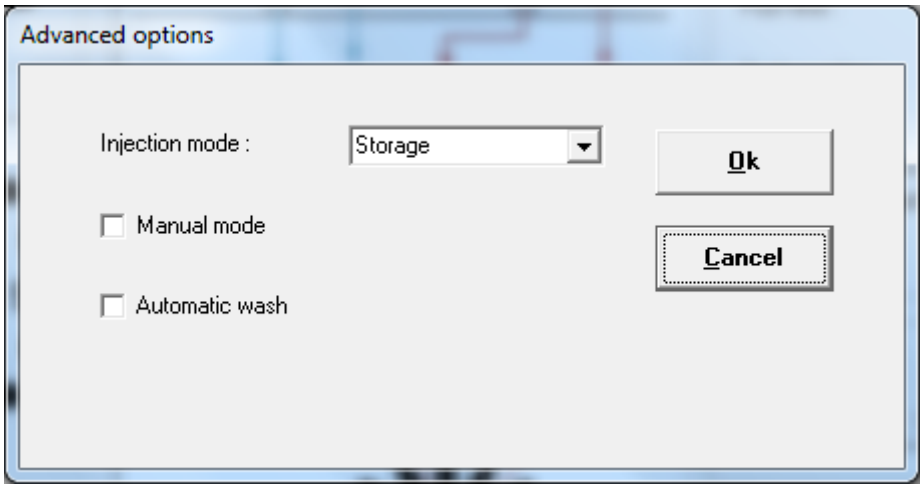
5.3 Advanced options

There are three ways to use the interface.

These modes are selected through the **Interface\Configuration\Advanced Options** menu.

! This menu is accessible only if the software is connected to the interface.

The following page is displayed:



By default, the interface is used in **Storage** mode. You can also use the Gas sampling valve or Multi-injection modes which will be described in chapter 7.

When the 'Manual mode' checkbox is checked, the user can work manually with the IST16 and has access to the method parameters. For more information on the manual mode, please refer to chapter 9.3.

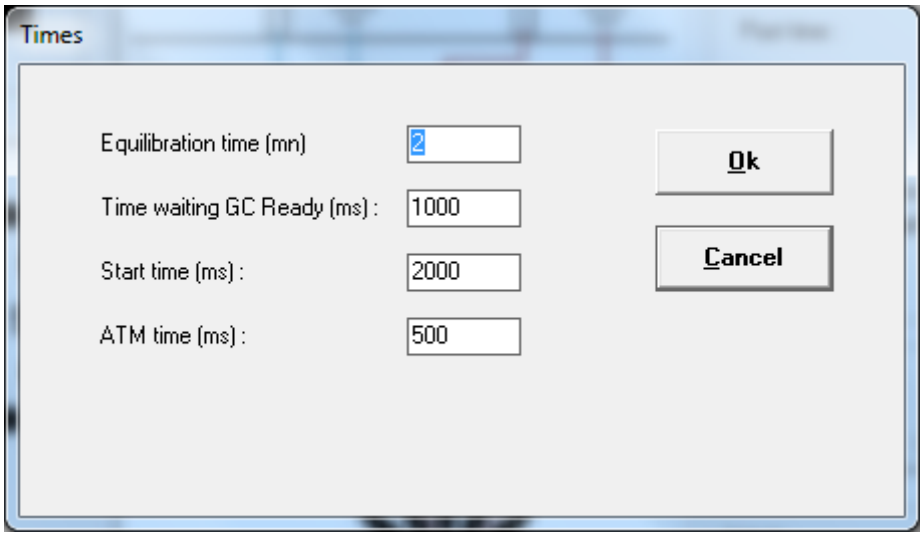
The 'Automatic wash' checkbox allows the user to activate the request for washing after analysis.

5.4 Commands time delay

Several commands must remain active for a given time. The default values are suitable for most configurations, but you can modify these times if necessary by using the **Interface\Configuration\Times** menu.

! This menu is accessible only if the software is connected to the interface.

The following page is displayed:



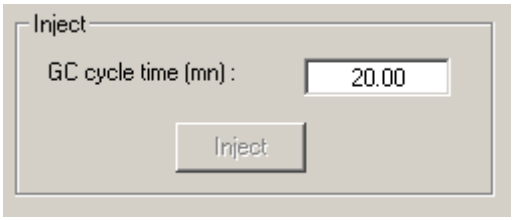
Delay configuration

6. Operate with the IST16 software

The IST16 typical sequence is as following:

1. First step: storage of the sample in different loops at programmed time.
2. Second step: loops are injected and analyzed one after the other.
3. Third step: loops are cleaned with the flowing gas coming from the TGA at the end of the GC analysis sequence.

The storage sequence can only be started when the interface oven temperature is steady. Before each injection of a loop, the software checks that the gas chromatograph is in a ready state (green color). The GC cycle time must correspond to the GC analysis time. It is used to adjust the delay between two injection loops.



6.1 Washing

The washing sequence can be started when the IST16 is idle by checking the Washing option and clicking on the **Start** button.

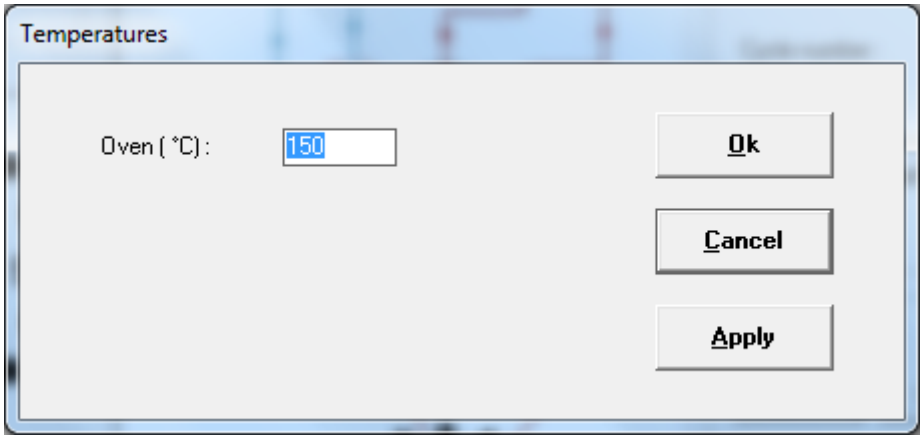
If the **'Automatic wash'** option has been checked, it can be started as soon as the injection cycle has been completed on all loops. Depending on the method, there may be several washing sequences.

6.2 Temperatures

The temperature of the transfer lines is self-regulated at 250 °C and therefore cannot be changed.

The valves oven temperature can be changed in the **Method\Temperatures** menu.

The following window appears:



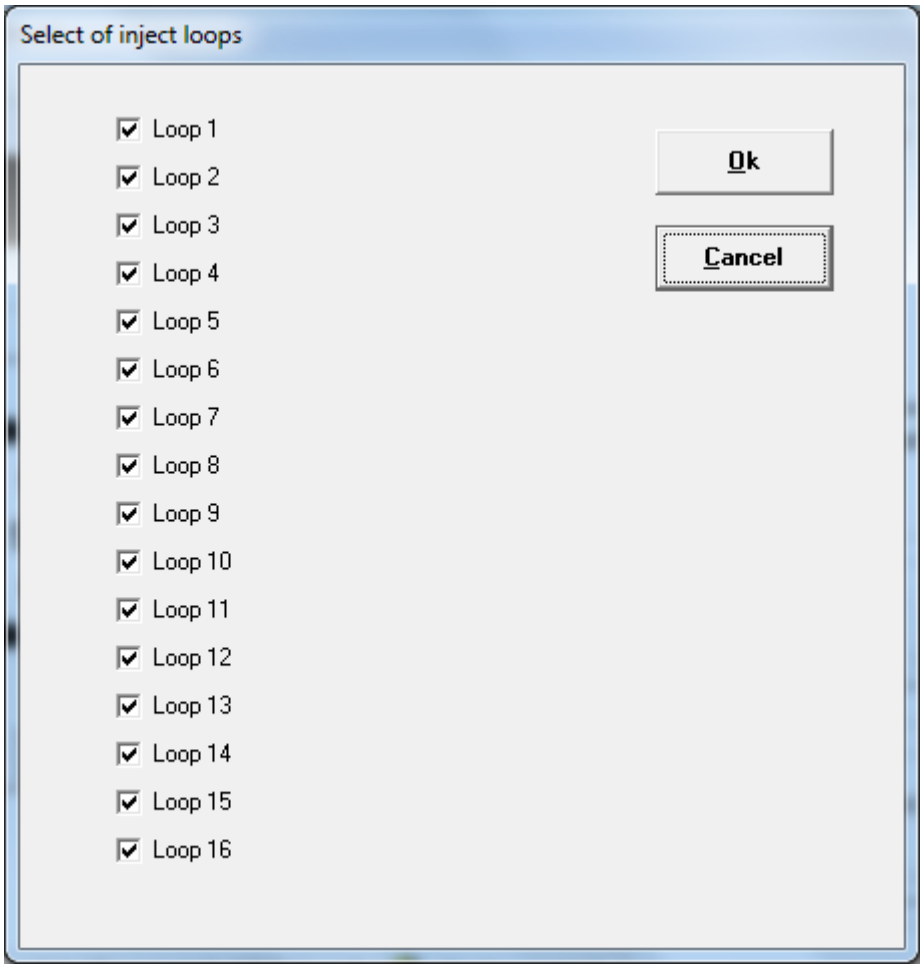
The temperature can be set between 150 and 250 °C. Click on "Apply" to send values to the storage interface, then click on "Ok" and save in the IST16 method.

Note that when the temperature from 150 to 250 °C is applied, the storage interface will start its own heating procedure and will automatically trigger the switch valves during the temperature raise. This operation is done to preserve the system from mechanical problems with the rotary valves.

6.3 Loops selection

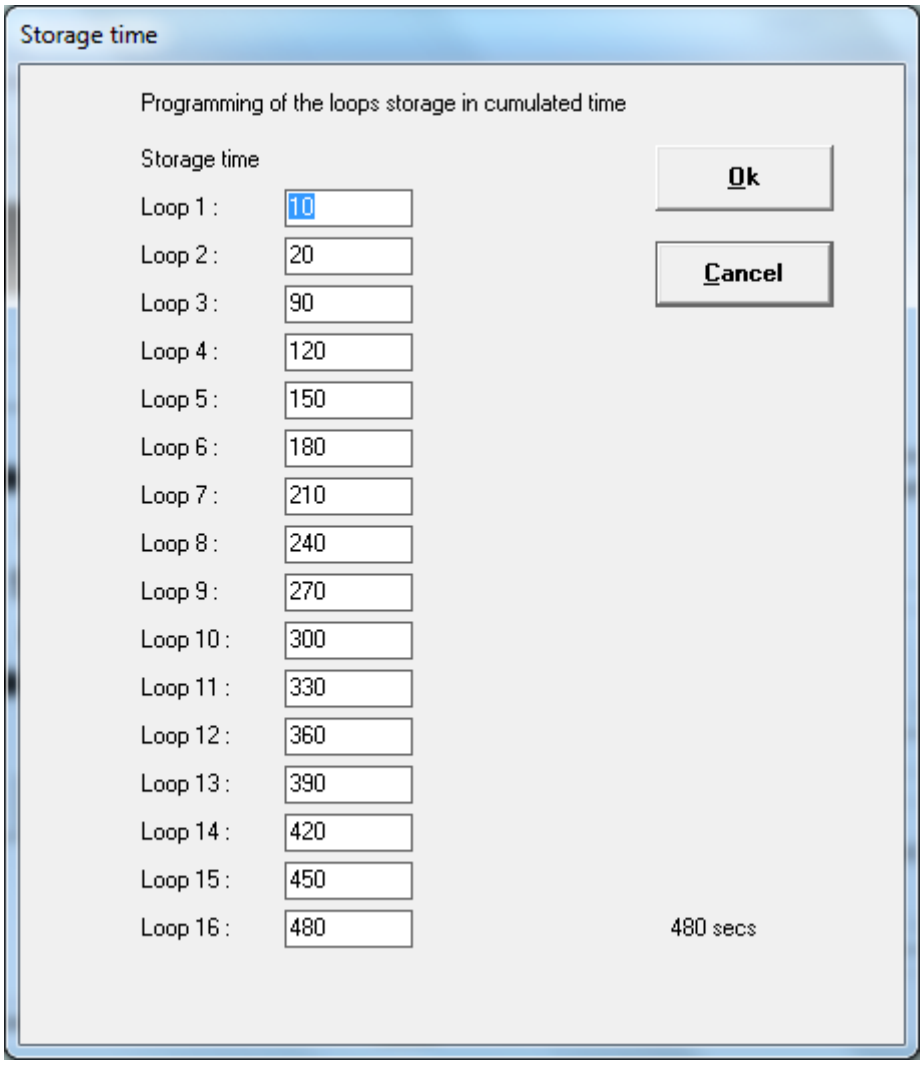
For some experiments, it is not necessary to use all the 16 storage loops. In this case, you can disable the loops which will not be used from the **Interface\Select loops** menu.

It is strongly recommended to work only with consecutive loops to keep the performance in terms of reproducibility of results.



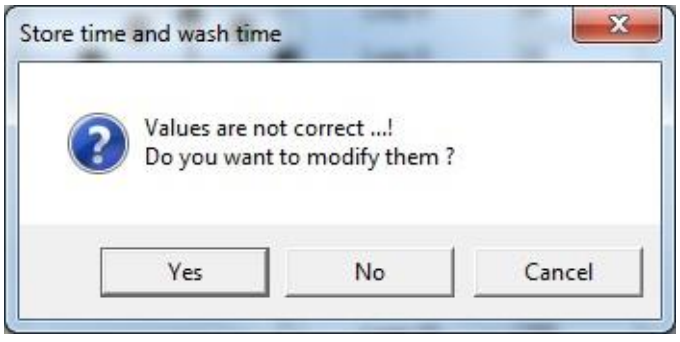
6.4 Storage time

During the storage sequence of an experiment, the multi-position valve allows each loop to be flushed with the emitted gas for a period of time. This time is defined in the window below accessible from the **Method\Storage Time** menu. A fraction of the emitted gas (typically 250 µL) will be stored in one of the 16 loops at the end.



Note that if a set time is not correct, the input value is displayed in red.

When all changes are made, click on the "OK" button to confirm. The software checks the values and if there is an error, it reports it with the message:



- If you click on the "Yes" button, the software corrects the values and displays again the window.
- If you click on the "Cancel" button, it displays again the window without making any changes.
- If you click on the "No" button, it makes no change. In this case, it may be that some loops are not flushed and stored properly.

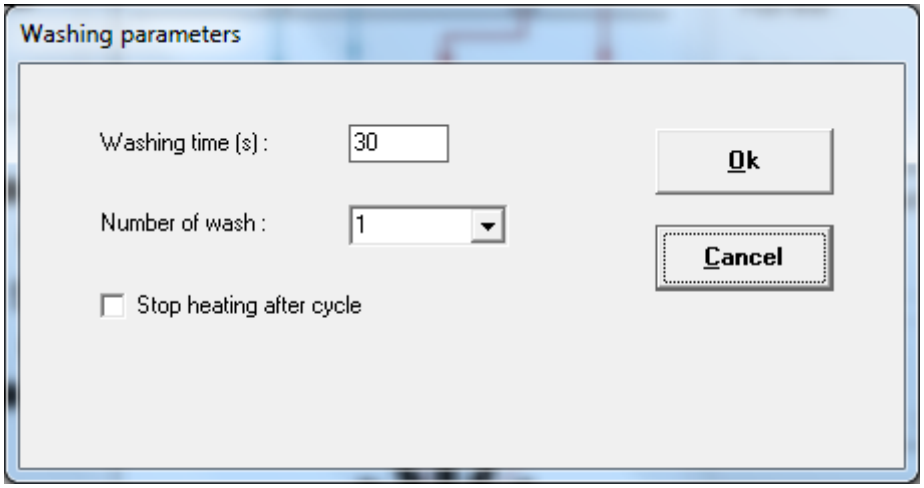
All time values are saved in the method.

Important remark concerning the first loop:

The first loop of a storage series shall not be considered for quantification or for the study of the composition of a mixture. It is indeed customary to set up the storage sequence with a very short time on the first loop of the series in order to start storing the first compounds of interest on the second loop of the series.

6.5 Washing parameters

After the analysis sequence, you must flush the injection loops. A washing time, common to all the loops used, is accessible through the **Method\Washing** menu.



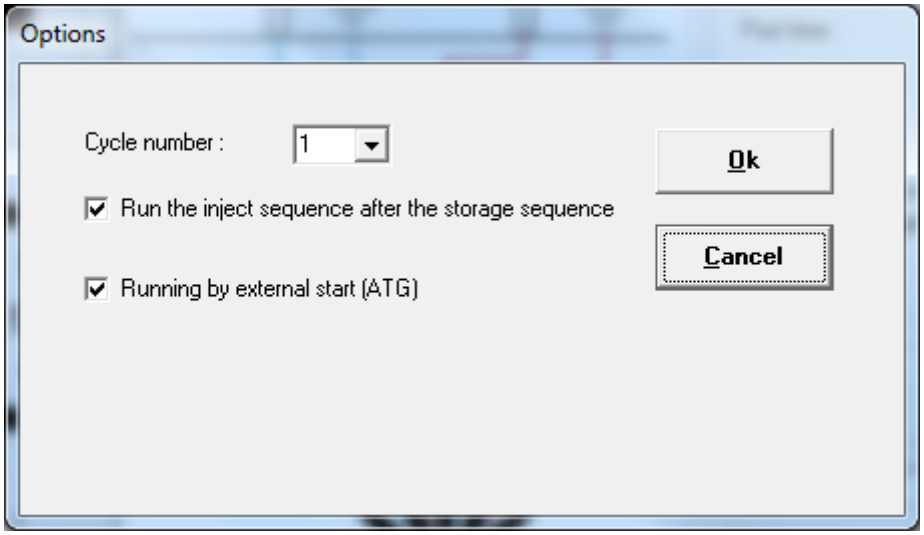
You can enter a time between 5 and 900 seconds.

You can set several washing cycles or even carry out a continuous washing.

The checkbox 'Stop heating after cycle' allows you to stop heating the oven and the lines when the washing cycle is finished. This is useful when the operation is finished and you want to put the IST16 in standby mode.

6.6 Options

The **Method\Option** menu allows you to automate several injection cycles. Note that between the injection cycle (analysis) and the next storage cycle, there is no washing cycle.



The 'Cycle number' parameter allows you to define the number of storage and analysis cycles you wish to perform.

Usually in the case of multi-cycle, not all loops are used. It is important to set up the TGA analysis cycle correctly so that the loops used by the storage sequence are injected and analyzed. It is also important to check the box 'Running by external start' so that the IST16 waits for the signal from the TGA at the end of the analysis of the last loop to restart the next storage cycle.

The checkbox 'Run the inject sequence after the storage sequence' allows to start the injection cycle as soon as the storage cycle is finished.

If the 'Running by external start' checkbox is enabled and you start the storage cycle by pressing the 'Start' button, the IST16 waits for the TGA to send a Start request to start the storage cycle.

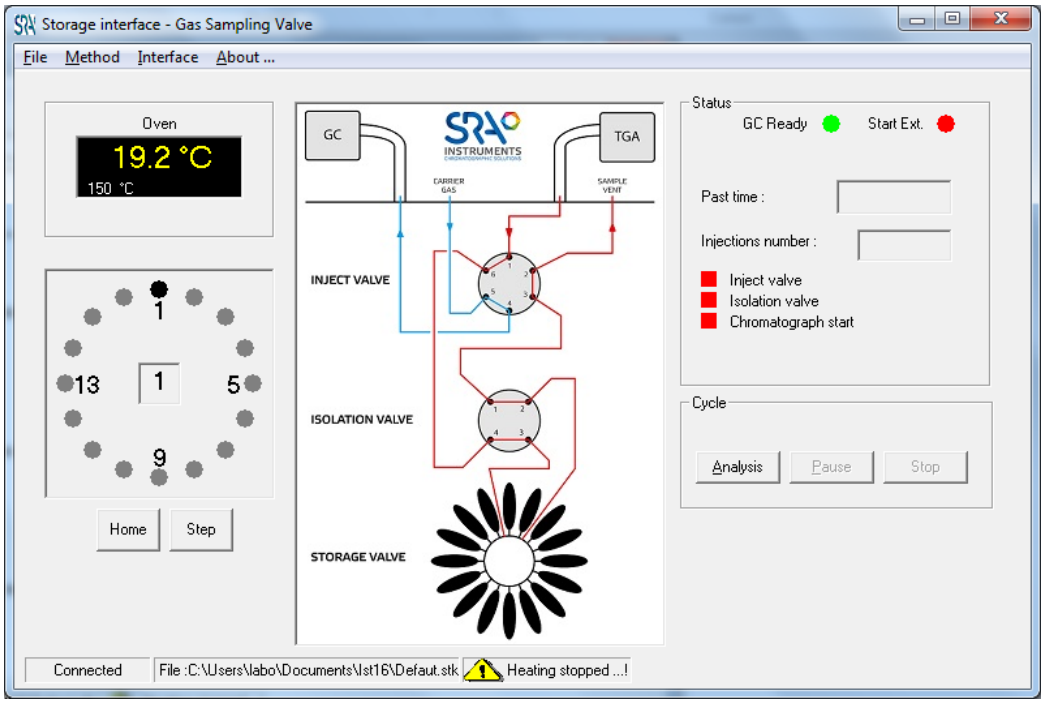
7. Gas sampling valve and Multi-injection modes

Both modes can be accessed via the **Interface\Configuration\Advanced options** menu.

With these two modes, the interface allows you to store and then inject a single loop several times in a row for a set period of time. As soon as the loop is injected, it returns to the storage position.

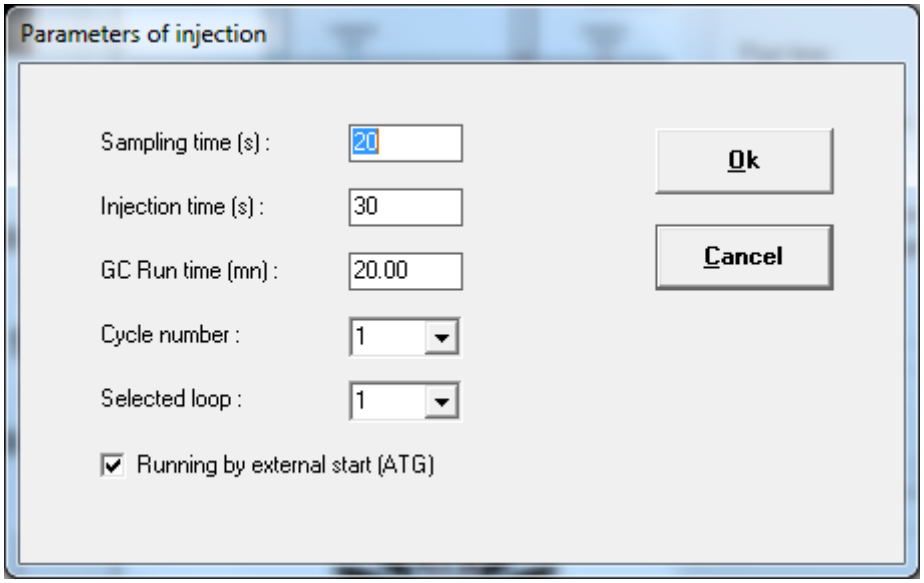
There are two ways of working, which are described in the following paragraphs.

7.1 Gas sampling valve (without multi-injection)



The interface will inject only once into the chromatograph for each analysis. The parameters can be changed via the **Method\Injection** menu.

The following window appears:

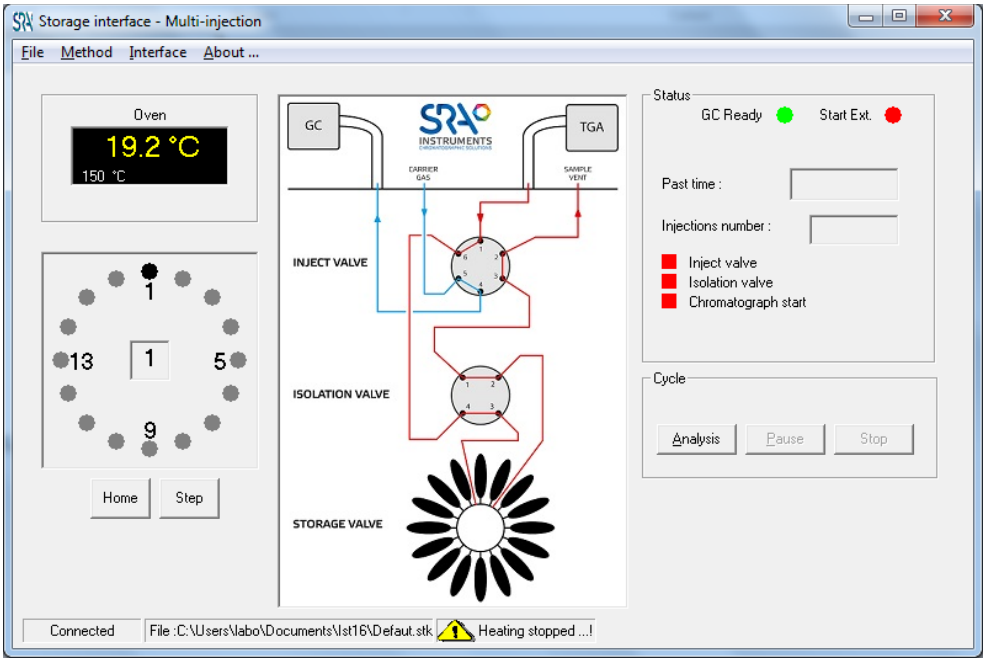


The injection will be carried out after the indicated sampling time and the analysis cycle will be started after the injection.

You can repeat the cycle several times.

If the 'Running by external start' option is checked, the IST16 will wait for the TGA to start before performing the sampling.

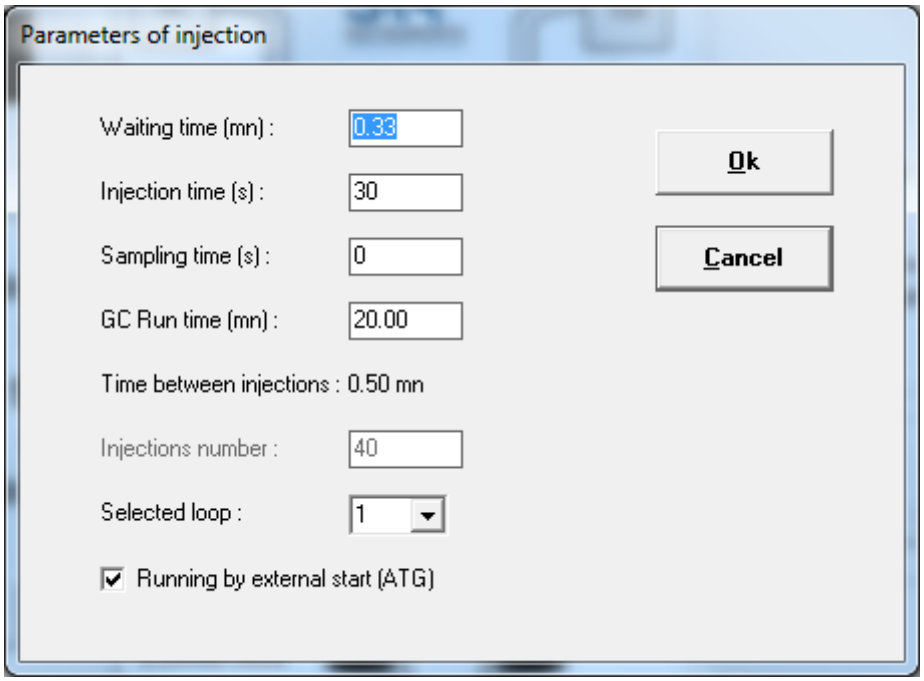
7.2 Multi-injection



The interface will inject several times in the chromatograph during the same analysis.

The parameters can be changed via the **Method\Injection** menu.

The following window appears:

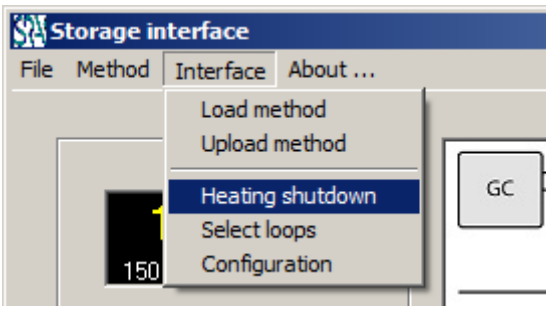


In the example above, the interface injects every 30 seconds during the TGA experiment, with the first injection after 20 seconds of loop flushing.
If the option is checked, the cycle will only start after receiving the external start from the TGA.

8. Dialog with the interface

Two menus allow either to load the method from the application to the interface (‘Load method’) or to retrieve the method of the interface (‘Upload method’).

These two actions are available under the **Interface** menu :

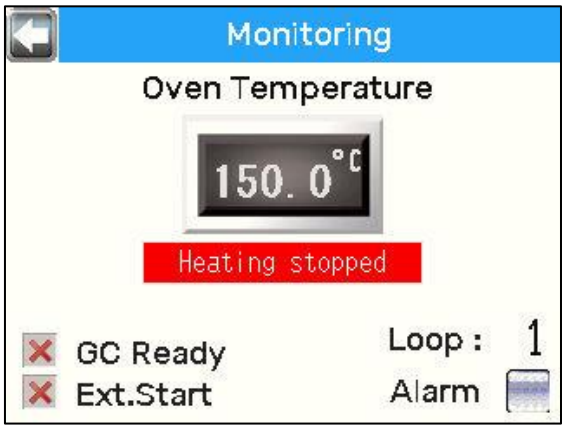


The 'Heating shutdown' sub-menu allows you to stop the heating of the oven from the application.

9. Storage interface

The storage interface is equipped with a touch screen from which various operations can be performed.

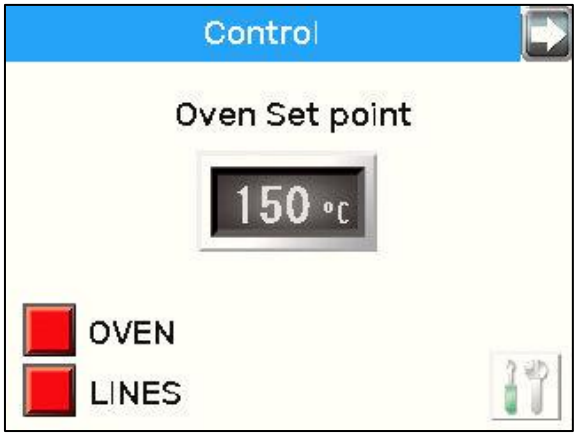
From the 'Monitoring' page, it is possible to view the status of the interface.



The display shows the various faults present or alarm, the status of the logic inputs and the oven temperature.

X : GC Not Ready
O : GC Ready

By clicking on the arrow at the top left of the screen, the 'Control' page is displayed.



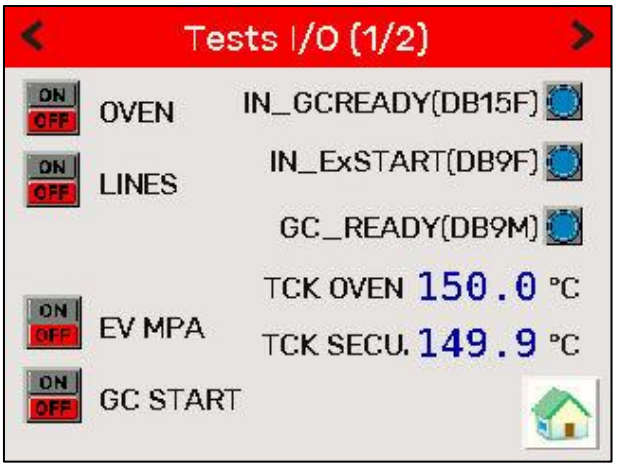
From this page it is possible to switch on the heating of the oven and the heated lines.
The 'Oven Set Point' field is used to set the oven temperature set point.

If the button is blue, the heater is off.
If the button is red, the heating is on.

9.1 Control and maintenance

The Tools button  in the bottom right corner of the 'Control' screen allows you to access the screens to perform different control or maintenance. This mode is protected by a password.

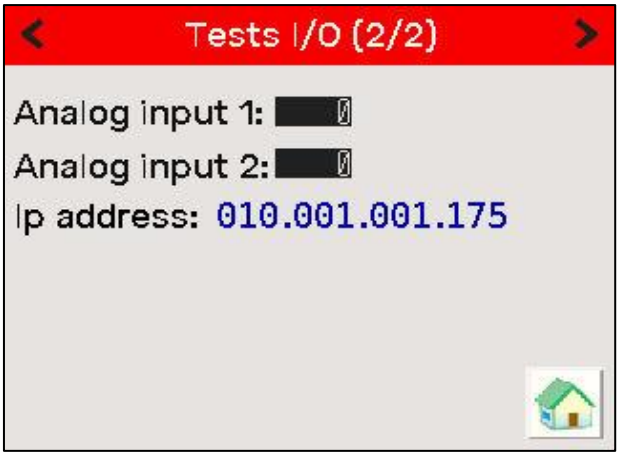
The password is: 2947



From this page you can switch on either the oven only or the heated lines.
You can control the atmospheric pressure release valve and monitor the temperature of the oven and of the safety sensor as well as the status of the logic inputs.

The following screen allows you to view the Analog input1 value that is used to retrieve the 'Ready/Not Ready' status of an Agilent chromatograph, Analog value. It can take two values:

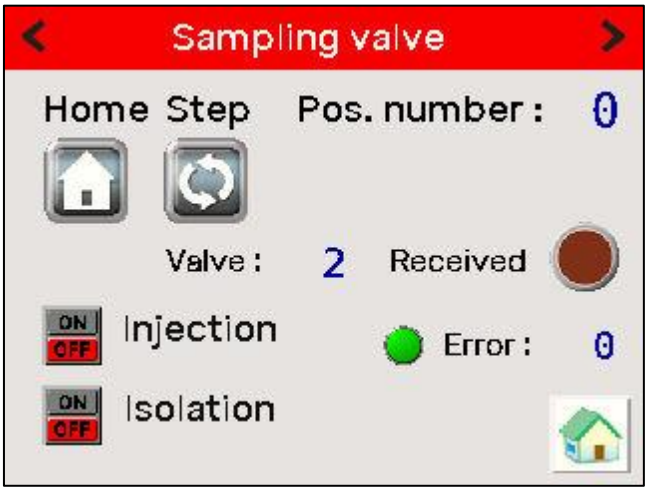
- 0 or 1, the chromatograph is 'Not Ready'.
- 30, the chromatograph is 'Ready'.



The Analog input 2 value is only used if the IST16 **pressure reading** option is enabled. Indeed, it could be considered to equip the IST16 with a pressure sensor and this input would allow to read it.

This page also displays the IP address of the interface. To change this address, refer to chapter 9.2.

The last page of this mode allows to control the position of the different valves.



9.2 Changing the IP address of the interface

When power is turned on, a first screen is displayed during interface initialization and a second screen is displayed during parameter initialization.

In the upper left corner, the  button gives access to the low level resources of the PLC.

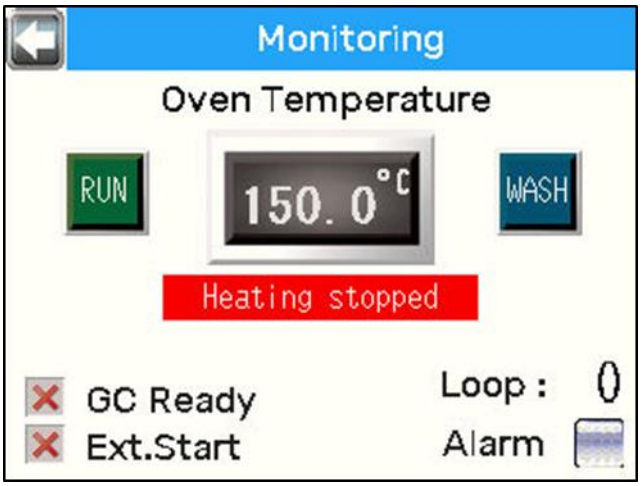
- Press this button. After a beep, the PLC goes into low level configuration mode, the display of the **'Home'** tab shows the firmware version of the Proface.
- Click on the **'MainUnit'** tab, a new screen gives access to different buttons.
- Press the **'Ethernet'** button.

- From the new screen, it is possible to change the IP address and the 'Subnet Mask'. Keep the port value at 8000. The bottom right arrow displays the Mac address of the interface and the fields for changing the 'GateWay' address.
- When you have modified the IP address, click on the **'Exit'** button to reset the interface, then on the **'Save changes and exit'** button the values will be automatically saved. At the question **'Offline mode will be terminated. Is that all right'**, press the **'Yes'** button.

9.3 Manual mode

If the manual mode is activated on the interface, it is possible to change the parameters directly from the PLC.

There are two buttons on the 'Monitoring' screen.



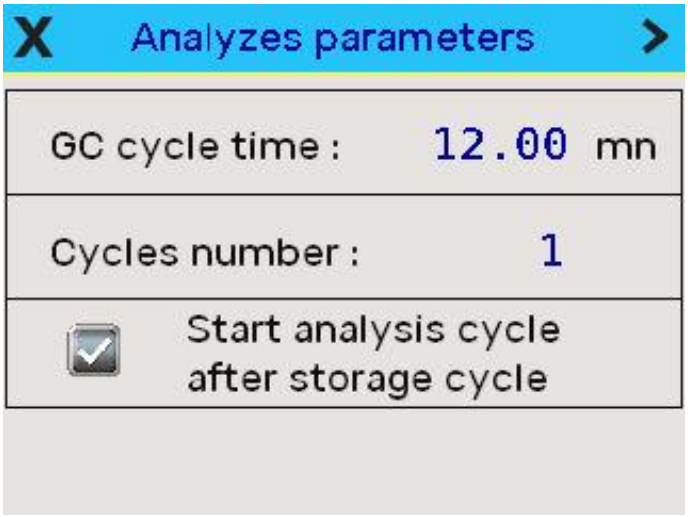
The Run button 

is used to start the storage cycle or the injection cycle when the storage mode has been completed.
Press the Wash button 

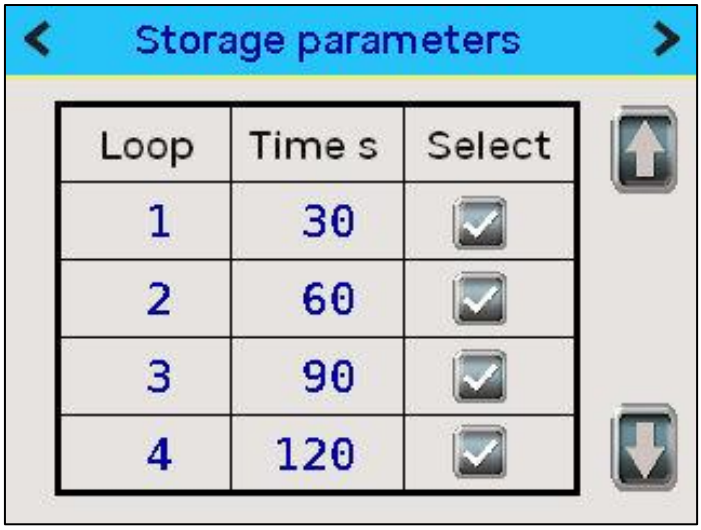
to start the washing cycle.

An additional button is displayed on the 'Control' screen. This button gives access to the parameters of the method.

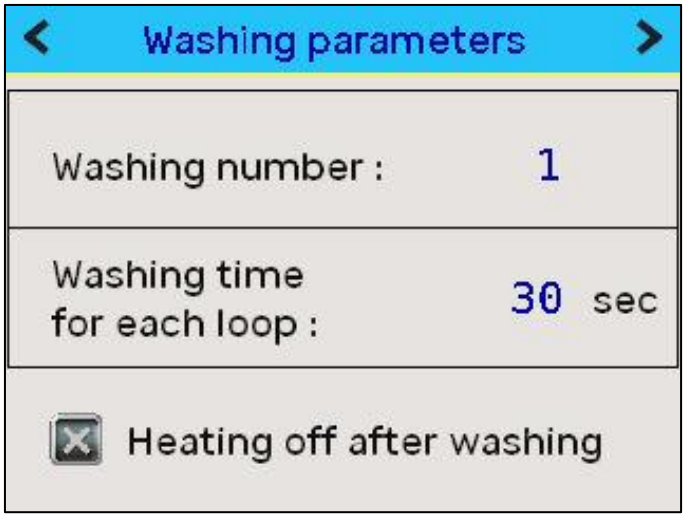
9.3.1 Analysis parameters



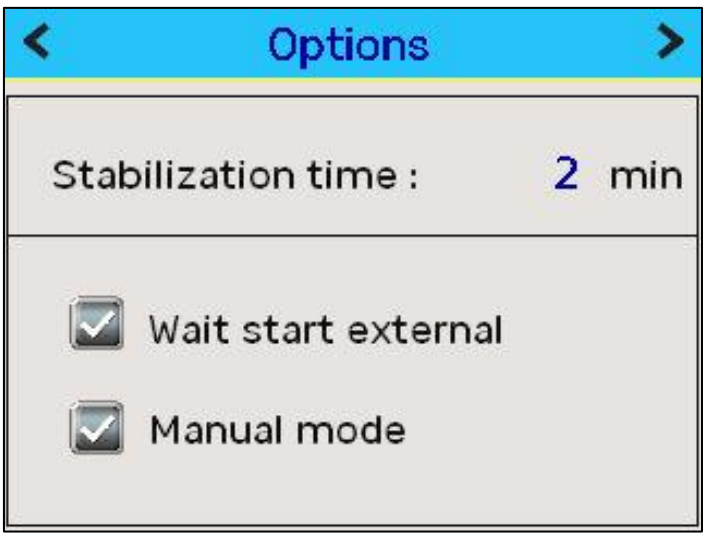
9.3.2 Storage parameters



9.3.3 Washing parameters

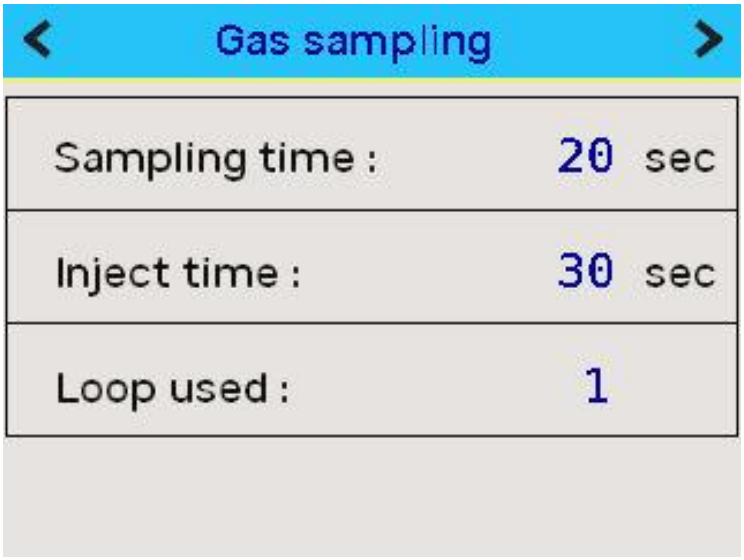


9.3.4 Options



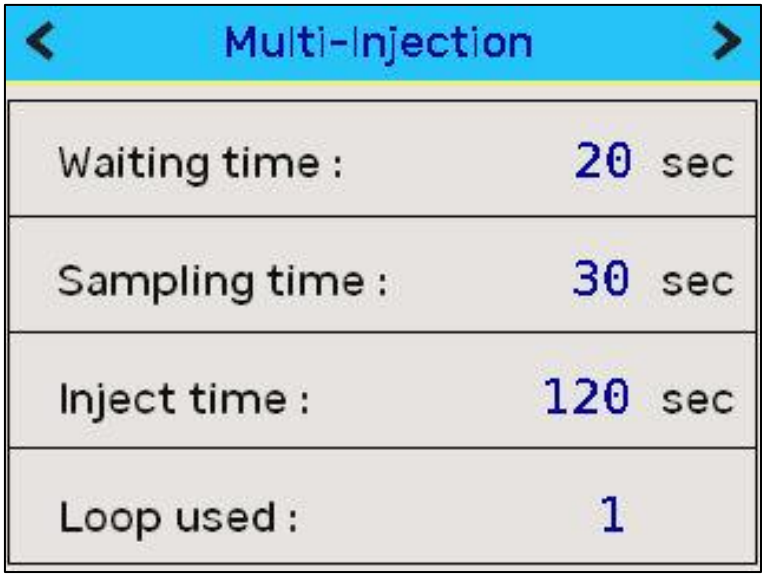
9.3.5 Gas sampling parameters

When the IST16 is configured in Gas Sampling mode, the 'Storage parameters' screen is not available and is replaced by the 'Gas Sampling' screen.



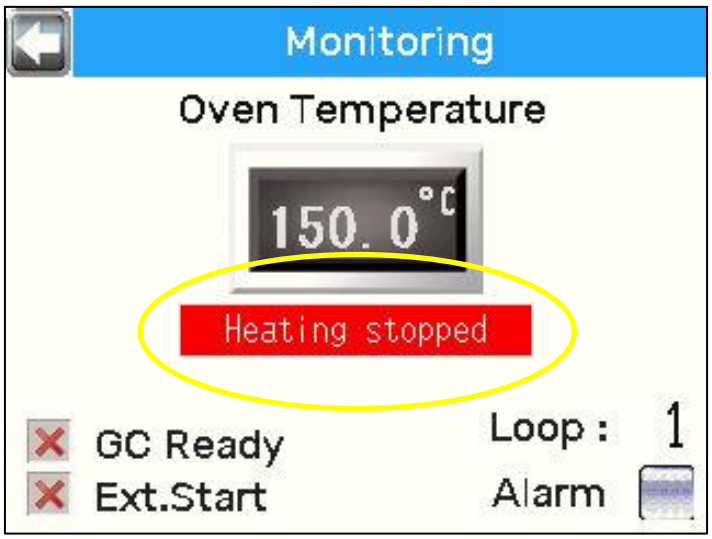
9.3.6 Multi-injection parameters

When the IST16 is configured in Multi-injection mode, the 'Storage parameters' screen is not available and is replaced by the 'Multi-injection' screen.



9.4 Error and fault messages

The various error or fault messages are displayed on the **'Monitoring'** screen.



Different information can be displayed:

- Oven Ready message: This means that the oven temperature has been reached and the storage sequence can be performed.
- Oven Not Ready Message: This means that the oven temperature is changing.
- Heating stopped message: This means that the heating is stopped and that no storage cycle can be performed.
- Heating alarm message: This message appears if there is a heating fault in the oven.
    - Either the oven does not heat while the heating is on. After 3 minutes, the temperature has not exceeded the safety threshold.
    - Or the temperature does not drop while the heating is switched off.
    - Or the oven temperature does not reach the required setpoint.
- Heating security message
    - Either the temperature read from the safety sensor is higher than the maximum permissible setpoint temperature and the deviation exceeds the safety threshold.
    - Or the difference between the temperature read from the oven sensor and the temperature read from the safety sensor is above or below the safety threshold.
- Position fault message: This message appears if the multi-position valve is not recognized (loss of ID, power off or faulty) or if it fails to move to the requested position.
- Isol.valve default message: This message appears if the isolation valve is not recognized (loss of ID, power off or faulty).
- Inject valve default message: This message appears if the injection valve is not recognized (loss of ID, power off or faulty).

This information is also displayed in the software.

At the end of the cycle, the IST16 resets these parameters and checks the presence of the valves.

10. Maintenance operations

10.1 Maintenance schedule

10.1.1 After each experiment

- Clean the TGA furnace with air flow about 200 mL/min.: TGA heating rate at 50 °C/min. to 1000 °C for 30 min.

  ⚠ **TGA must not be coupled to IST16 at this time.**

- Clean the 1/8" stainless steel union between PTFE tube and IST16 input transfer line with solvent.
- Replace the Teflon® protective tube if necessary.

10.1.2 Every week

- Check air and water levels using the MSD signal with carrier gas flowing through IST16, in standby mode. If the level is too high, check for leaks throughout the entire configuration.
- Clean the sample path of the IST16 with a solvent (≈ 20 mL) if you don't use a Teflon® protective tube.

10.1.3 Every month

- Check the glass liner state in the GC injector.

10.1.4 Every 6 months

- Check the storage valve rotor state, clean it with solvent or replace it if necessary.

Replace if required:

- MSD filament and electron multiplier
- 6 or 4 position valve rotor of IST16.

10.2 Typical cleaning after an experiment

IST16

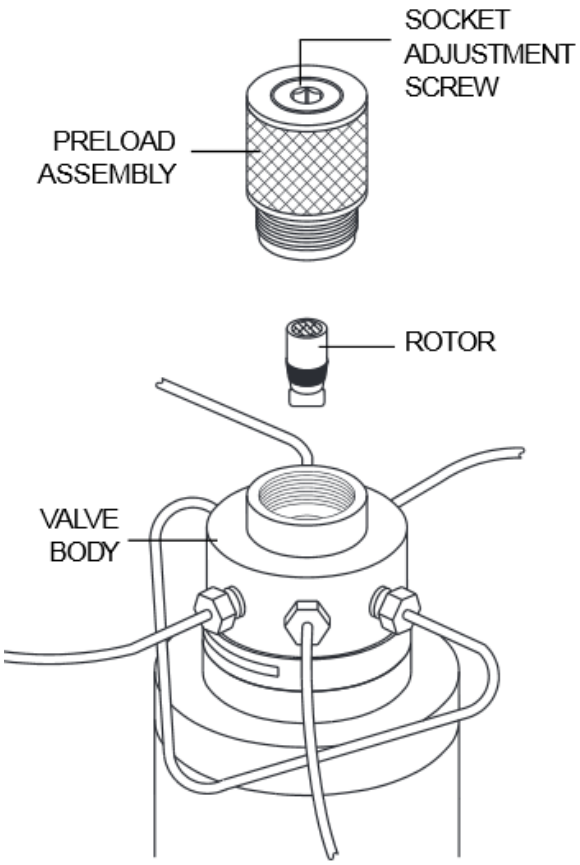
a. Check all the removable fittings between the input transfer line and the TGA and if necessary clean them with ethanol or isopropanol.
b. You can also replace the Teflon® protective tube if necessary.
c. The programmable IST16 cleaning sequence is used to clean the loops after a sequence (a total cleaning time of at least 5 minutes for each loop is recommended).
d. As a reference blank, you can run a sequence with the TGA purge gas to verify that all loops are clean.

TGA

e. Disconnect IST16 from TGA.
f. Run a step at 1000 °C for 30 min, using a high air flow rate.

Note: steps a, b, e and f can be done after the end of the TGA run and during GC-MS analyses.

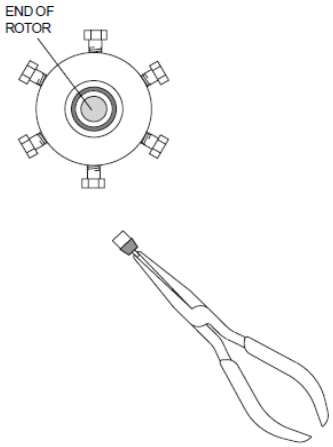
10.3 Clean the valve and rotor



10.3.1 Disassembly the valve

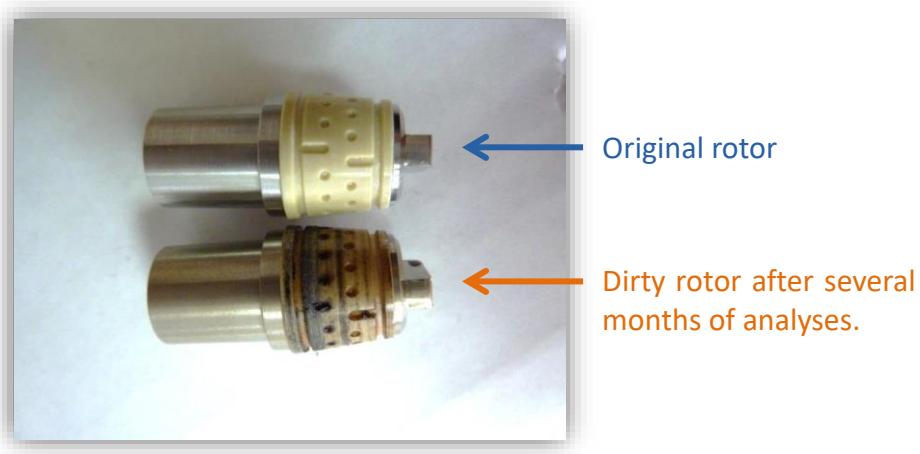
1. Shutdown IST16 heating.
2. Wait until the oven temperature is around 50 °C, otherwise the preload assembly thread may become permanently blocked.
3. Unscrew totally the preload assembly.
4. Operate the storage valve from the software to disengage the rotor from the valve body.
5. **Note the direction in which the rotor is positioned** and carefully remove it with a magnet or flat clamp.

⚠ **The direction of the rotor is critical**



10.3.2 Clean the inside of the valve body

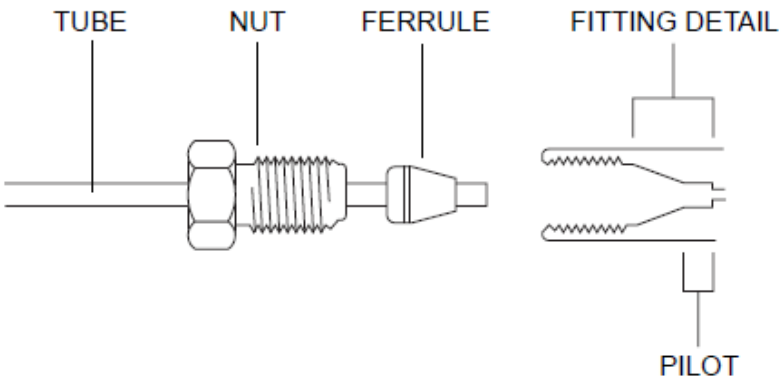
1. After taking out the rotor, wet a cotton stab with methanol or isopropyl alcohol.
2. Clean gently by dabbing the inside of the valve body to remove any residue.
3. Also, be careful to remove any fiber left during the cleaning.
4. Visually check the inside of the valve body. The conical surface must be smooth. The presence of a scratch between the holes can lead to leakage, in this case the valve body must be replaced.



10.3.3 Clean the rotor

1. Carefully grasp the rotor tip, taking care not to damage the metal or polymer.
2. Immerse it briefly in the solvent (methanol or isopropyl alcohol).
3. Carefully wipe the polymer with a clean tissue and make sure that no fiber is left by the tissue.
4. Inspect the rotor. If it has scratches or shrinkage, it must be replaced.

10.3.4 Change tubing or fitting



Parts of a Valco fitting

1. Pay attention to the condition of the 1/16" tubing; it must be clean inside and scratch-free outside.
2. The temperature of the IST16 oven must be below 50 °C.
3. Insert the new fitting assembly into the valve port, screwing the nut 2-3 turns by hand.
4. Push the tubing so that it gets in place; this is essential for proper connection with zero dead volume.
5. Turn the nut manually until you can tighten it by hand.
6. Use the appropriate wrench and screw the nut ¼ of a turn (90°) after the point where the ferrule begins to touch the tubing.

11. Locate the origin of the problem

TGA/IST16/GC-MS coupling is a complex and powerful solution having 3 independent devices: a TGA, an IST16 and a GC-MS.
Each device can have a problem and therefore affect the results given by the other 2 devices.

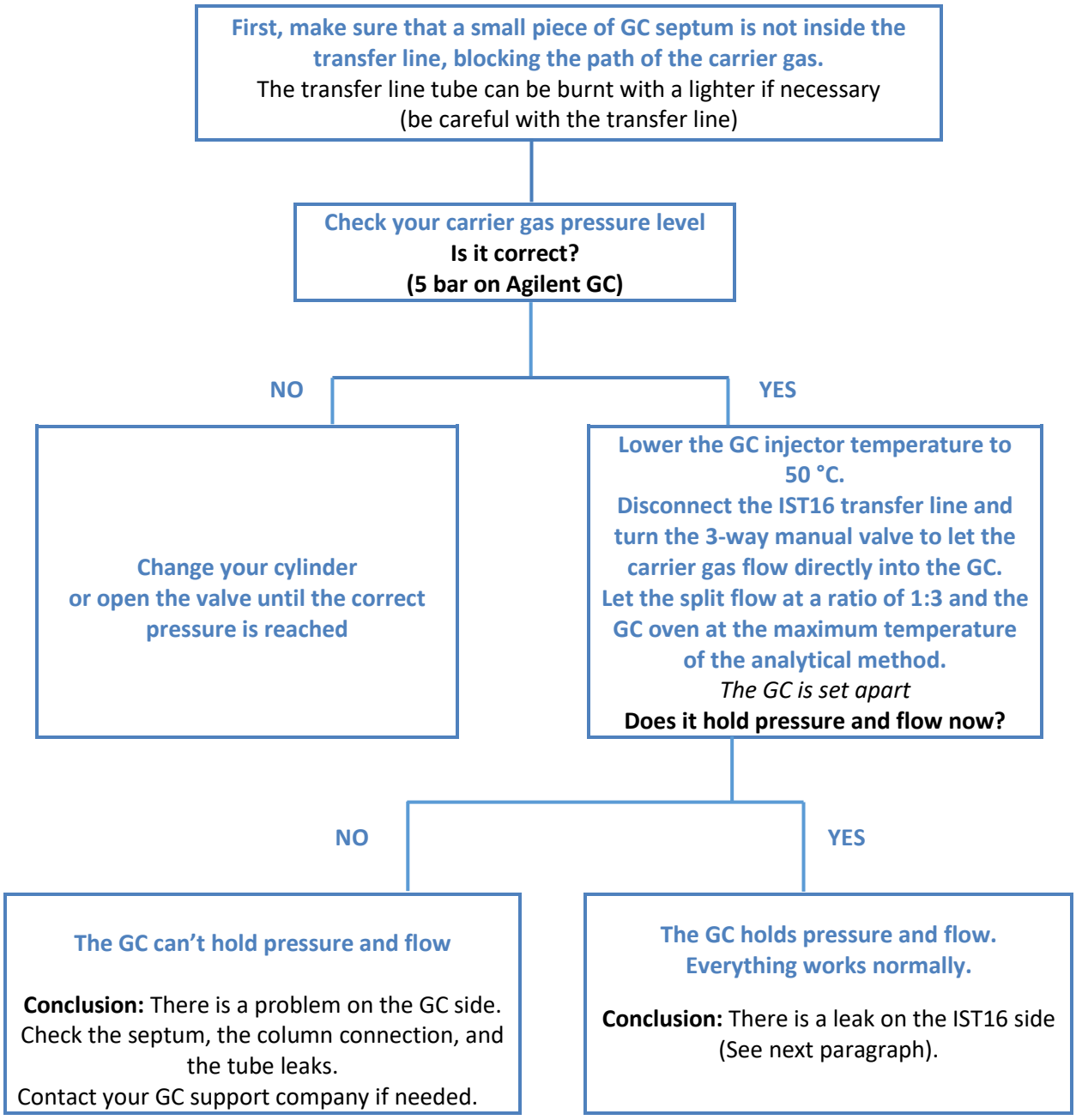
This chapter tries to help you locate the most common problems.

A leak detector and a flow meter are needed to detect and solve most of the problems encountered with the IST16 and GC-MS.

Most common problems	**Paragraph**
No flow in GC No pressure in GC GC can't hold the pressure	11.1
No peak Small peak Flow restriction TGA oven back pressure	11.2
Memory effect	11.3
High level of air in MS	11.4
Problem with oven display and multi position valve number	0

11.1 Flow/pressure problem with GC

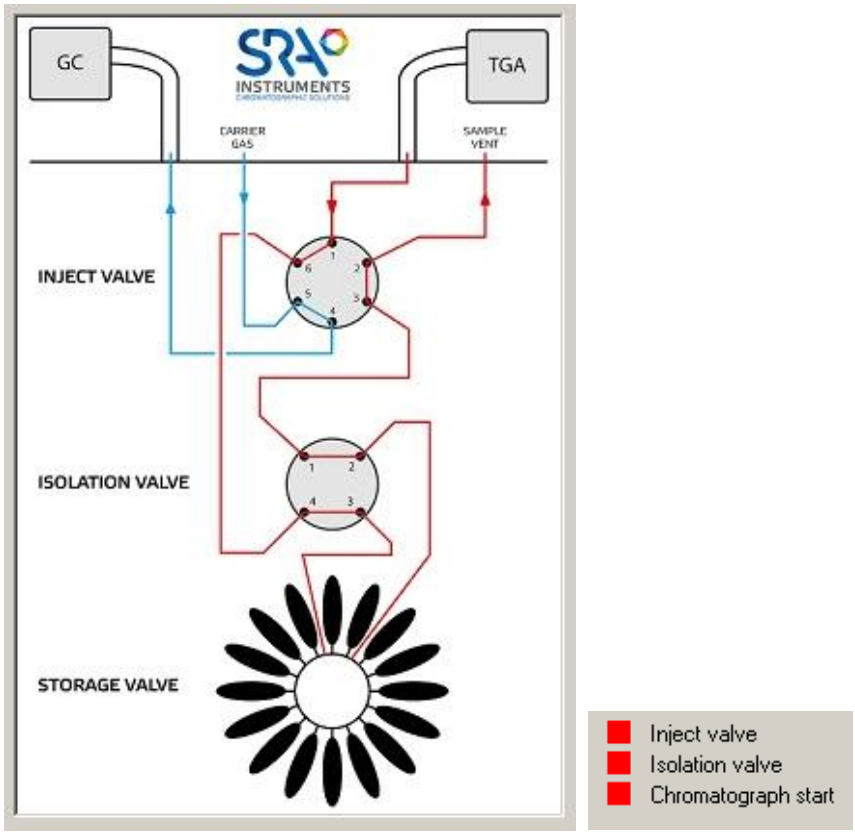
11.1.1 Find the origin of the problem



11.1.2 There is a leak on the IST16 side

1. Lower the temperature of the injector and oven to 50 °C.
2. Reconnect the IST16 transfer line to the injector.
3. Let the split flow at a ratio of 1:3 and the GC oven at the maximum temperature of the analytical method.
4. Lower the temperature of the IST16 to 50 °C. Make sure that the 3-way manual valve is in the IST16 position.
5. Use the IST16 interface to help you find the leak, Maintenance menu, 'Sampling valve' screen.

Step 1:



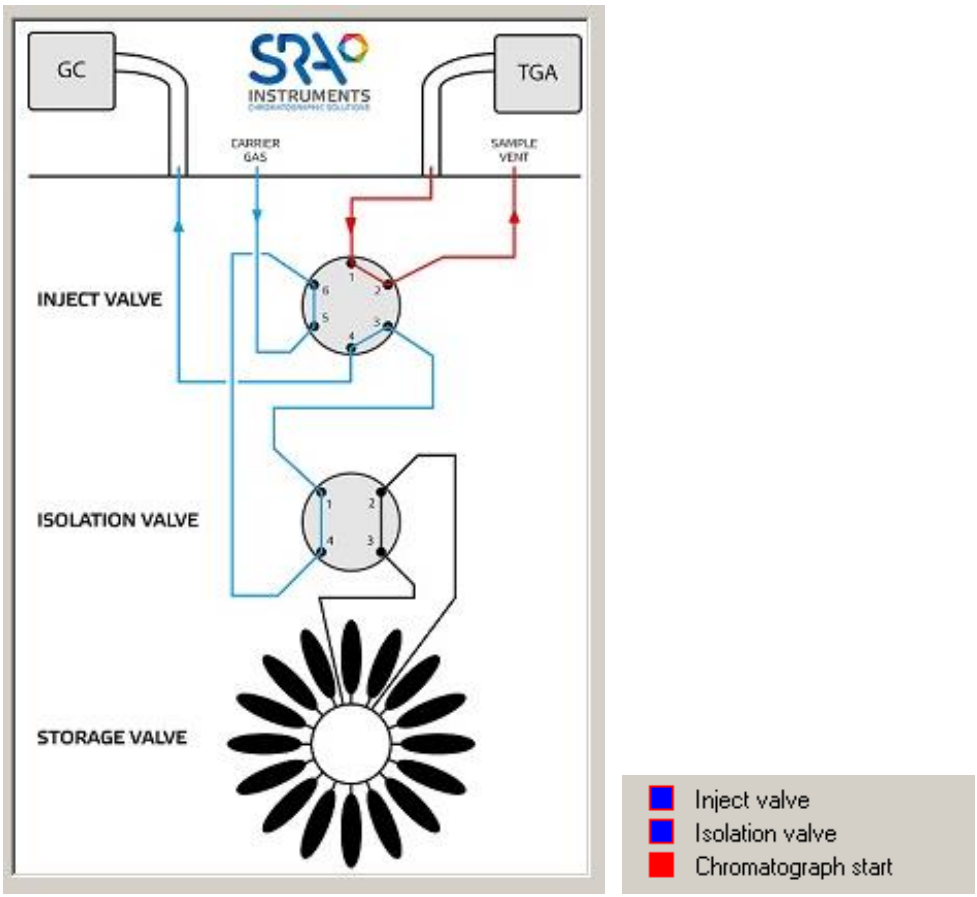
Legend : Inactive Active

In this configuration of the IST16, the leak may be:

- On the GC septum side → change the septum if necessary.
- Check the transfer line if the path is blocked by the septum → **cut a small part of the transfer line on the GC side or change it if necessary.**
- Between the 3-way manual valve and the carrier gas connection panel on the back of the IST16 → **check for leaks and replace tube and ferrules if necessary.**
- In the IST16 oven, located between the rear panel of the IST16 and the injection valve → **check for leaks and replace tube and ferrules if necessary.**
- On the rotor of the injection valve → **clean the rotor (see paragraph 10.3); replace it if necessary.**

- If the GC holds the pressure in this position, go to step 2.

Step 2:

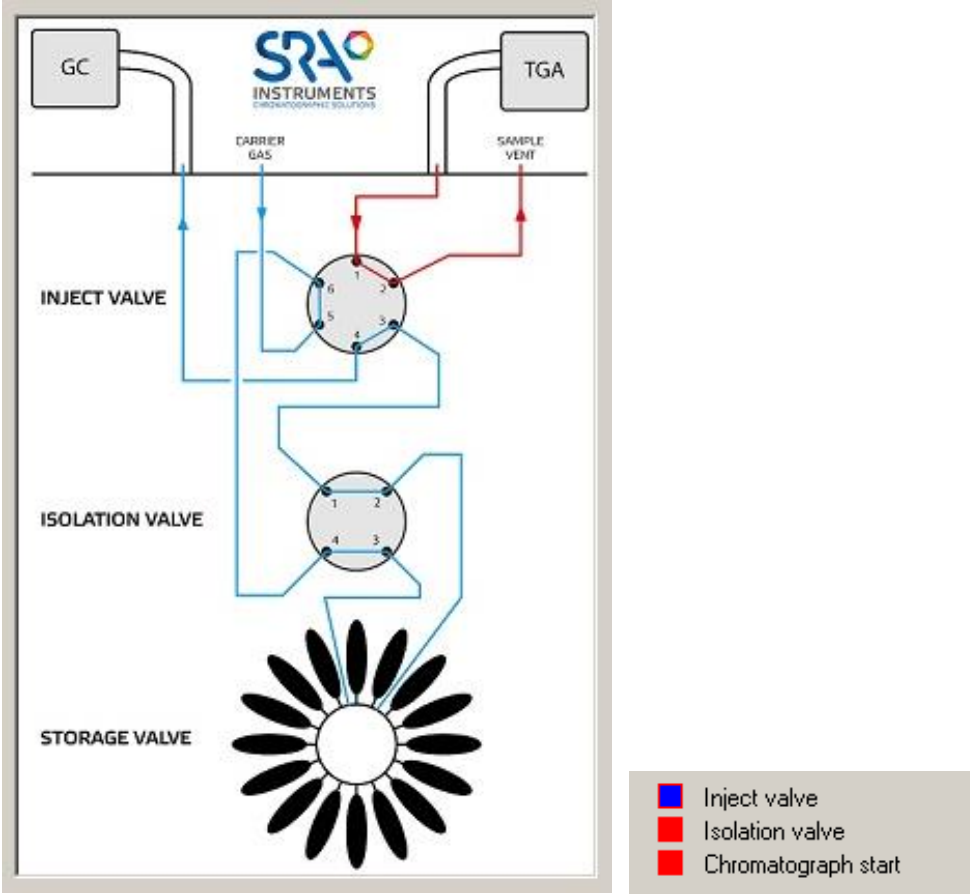


Legend : Inactive Active

In this configuration of the IST16, the leak may be:

- Between the injection valve connection and the isolation valve connection → **check for leaks and replace tube and ferrules if necessary.**
- On the rotor of the isolation valve → **clean the rotor (paragraph 10.3); replace it if necessary.**
- If the GC holds the pressure in this position, go the step 3.

Step 3:



Legend : Inactive Active

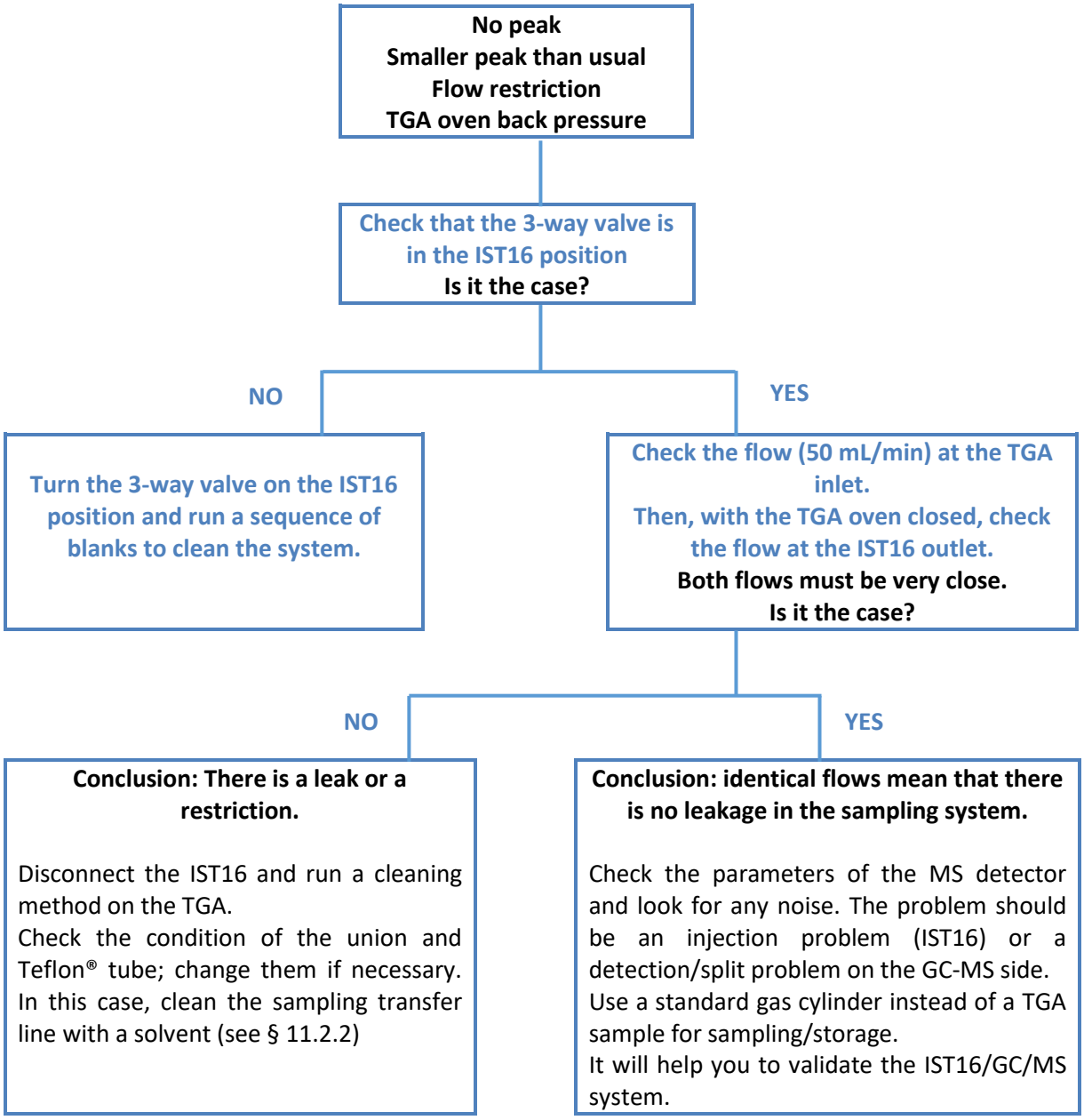
In this configuration of the IST16, the leak may be:

- On the selected loop → **change the loop and check if the leak has disappeared, replace it if necessary.**
- On the rotor of the storage valve → **clean the rotor (paragraph 10.3); replace it if necessary.**
- On the connection between the isolation valve and the storage valve → **check for leaks and replace tube and ferrules if necessary.**
- On the atmospheric pressure release valve, inspect the plastic vent located on the rear panel → **if you detect helium at the tube outlet in this configuration, it means that the atmospheric pressure valve is leaking. Change the atmospheric pressure valve.**

Contact SRA Instruments or your TGA support company for the next steps.

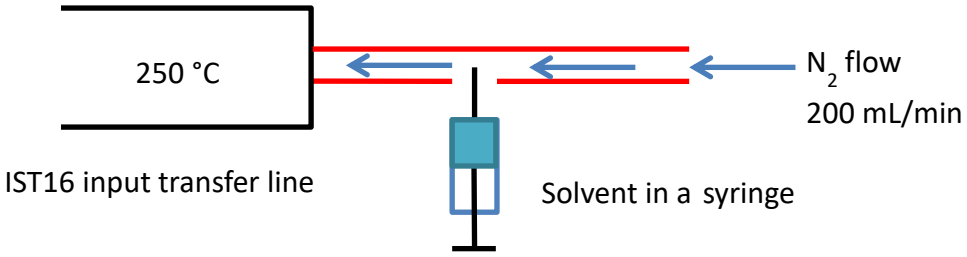
11.2 Problem of flow restriction

11.2.1 Find the problem



11.2.2 Clean the inside of the IST16

After significant contamination or persistent restriction it is possible to clean the inside of the IST16 using a solvent.
Use a nitrogen flow of about 200 mL/min (not through TGA if possible) to push a small volume of solvent (isopropyl alcohol or methanol) into the system, through the input transfer line.



1. Program the instrument to 250 °C (line and oven) and use a nitrogen flow to push slowly a small volume of solvent (isopropyl alcohol) inside the system.

2. A contaminated loop or a transfer line can also be replaced. The rotor must be cleaned after this operation. Place an evacuation at the outlet of the IST16 in a recovery vial (products may come out in liquid form).

Exothermic reactions may occur at this stage.

3. At the end of the cleaning, run a wash sequence from the IST16 software using a nitrogen flow (5 minutes wash per loop) for 2 hours.

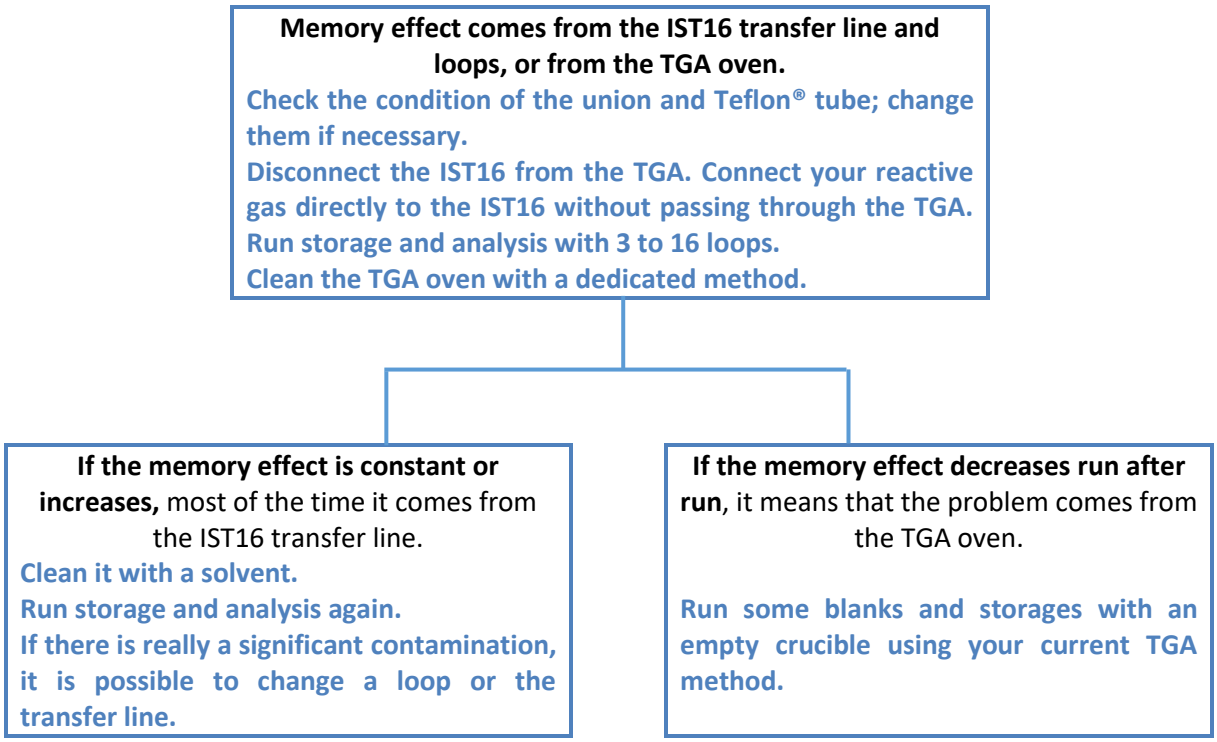
4. After the cleaning: start a GC-MS analysis using storage with nitrogen to check the memory effect.

5. Use a 20 cm Teflon® tube to protect the IST16 from the heaviest compounds.

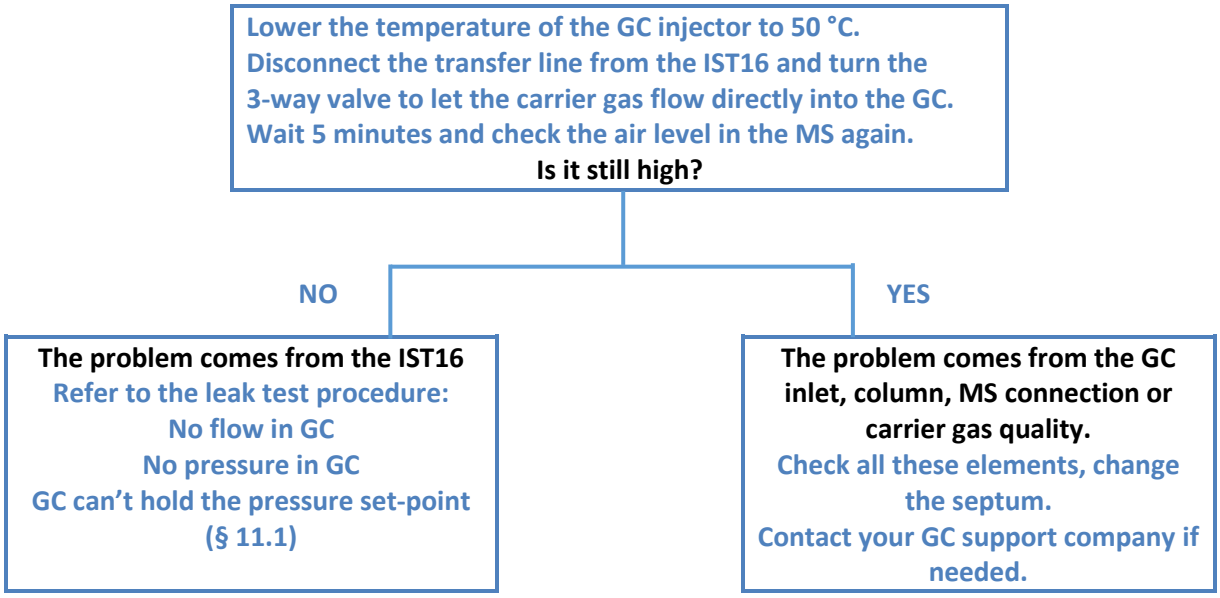
Presence of particles in the PTFE tube after analysis of PE-HD

Teflon® tube can also stop halogenated compounds.

11.3 Problem of memory effect

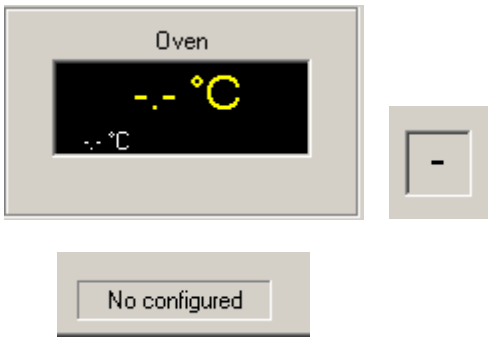


11.4 Problem of high level of air in MS



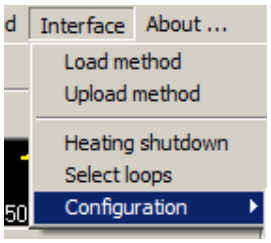
11.5 Problem with oven display and multi-position valve number

If you see this :



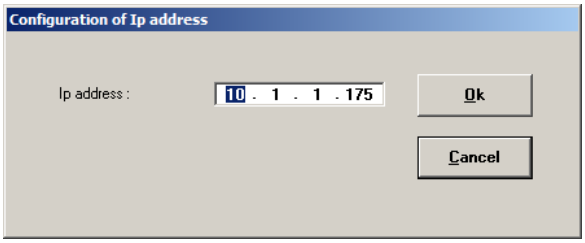
The indication "No configured" means that the connection with the IST16 has been lost.

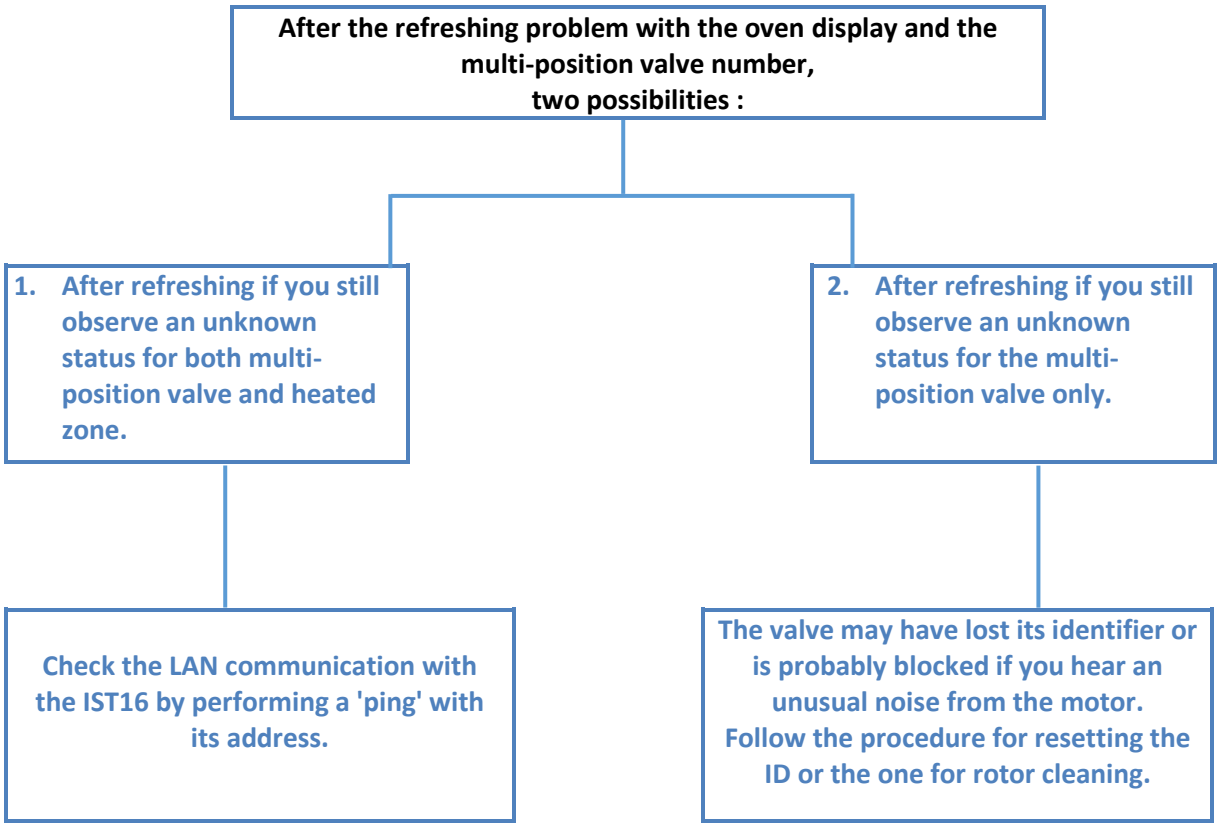
Close the application and switch off the IST16.
Then switch it on and restart the application.
If the connection is still not established, enable configuration with the **Interface\Configuration** menu using the **Config** user and the **ist16** password.



Display the **'Configuration of Ip Address'** window with the **Interface\Configuration\Ip Address** menu.

Change the IP address if necessary and confirm with the OK button.




After the refreshing problem with the oven display and the multi-position valve number, two possibilities :
1. After refreshing if you still observe an unknown status for both multi-position valve and heated zone.
2. After refreshing if you still observe an unknown status for the multi-position valve only.
Check the LAN communication with the IST16 by performing a 'ping' with its address.
The valve may have lost its identifier or is probably blocked if you hear an unusual noise from the motor. Follow the procedure for resetting the ID or the one for rotor cleaning.

12. Technical Data

12.1 Power supply

Power supply input: + 220 VAC
Power supply output: + 24 VDC
Power consumption, max. 2.5 A /220 VAC

12.2 Dimensions and weights

- H 450 ; D 400 ; W 370 mm
- 25 Kg

12.3 Instrument

Data System
- SRA IST16 Software

Number of loops
- 16 in Sulfinert™ stainless steel

Number of valves
- 3 (injection, storage, isolation) with automatic management

Heated zones
- 2 electronically regulated

Loop volume
- 250 µL in standard, customized volumes on demand

Heated transfer lines
- Small internal diameter x 1.15 meter in Sulfinert™ stainless steel ; Tmax = 300 °C

Valve box temperature
- 250 °C as standard working temperature (300 °C can be reached for specific applications)

Ethernet port
- Ethernet port IEEE 802.3u 10/100 BASE-T fast Ethernet Compatible

12.4 Utilities

The SRA IST16 interface must be located between TGA and GC. This requires a space of at least 40 cm wide.

Power: 220-240 VAC; 1000 W max

GC specification
- Requires a split/splitless inlet, remote start-in, remote ready-out

PC requirements
- Windows 7, Ethernet connection

TGA requirements
- remote start-out (contact closure)

12.4.1 Carrier gases

The IST16 is compatible with helium, nitrogen, argon, and hydrogen with Swagelok fittings.

12.4.2 Repeatability

Typical RSDs at constant temperature and pressure including SRA MPA system: ≤ 5 %

12.5 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.

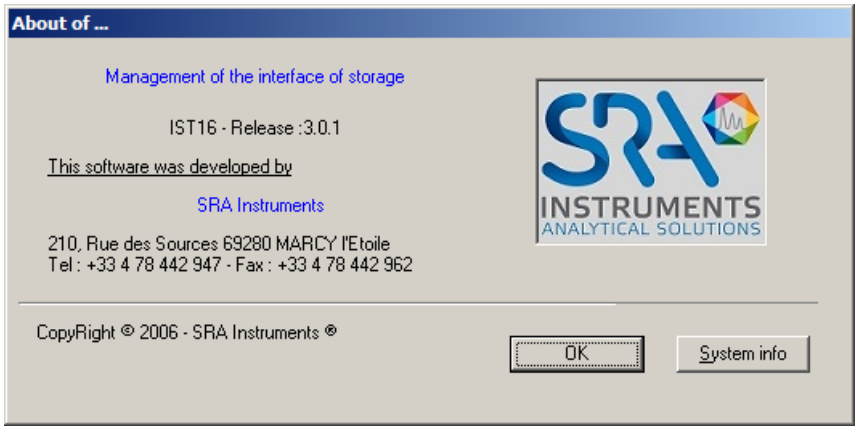
12.6 Inputs/Outputs

Refer to appendices

12.7 Recycling

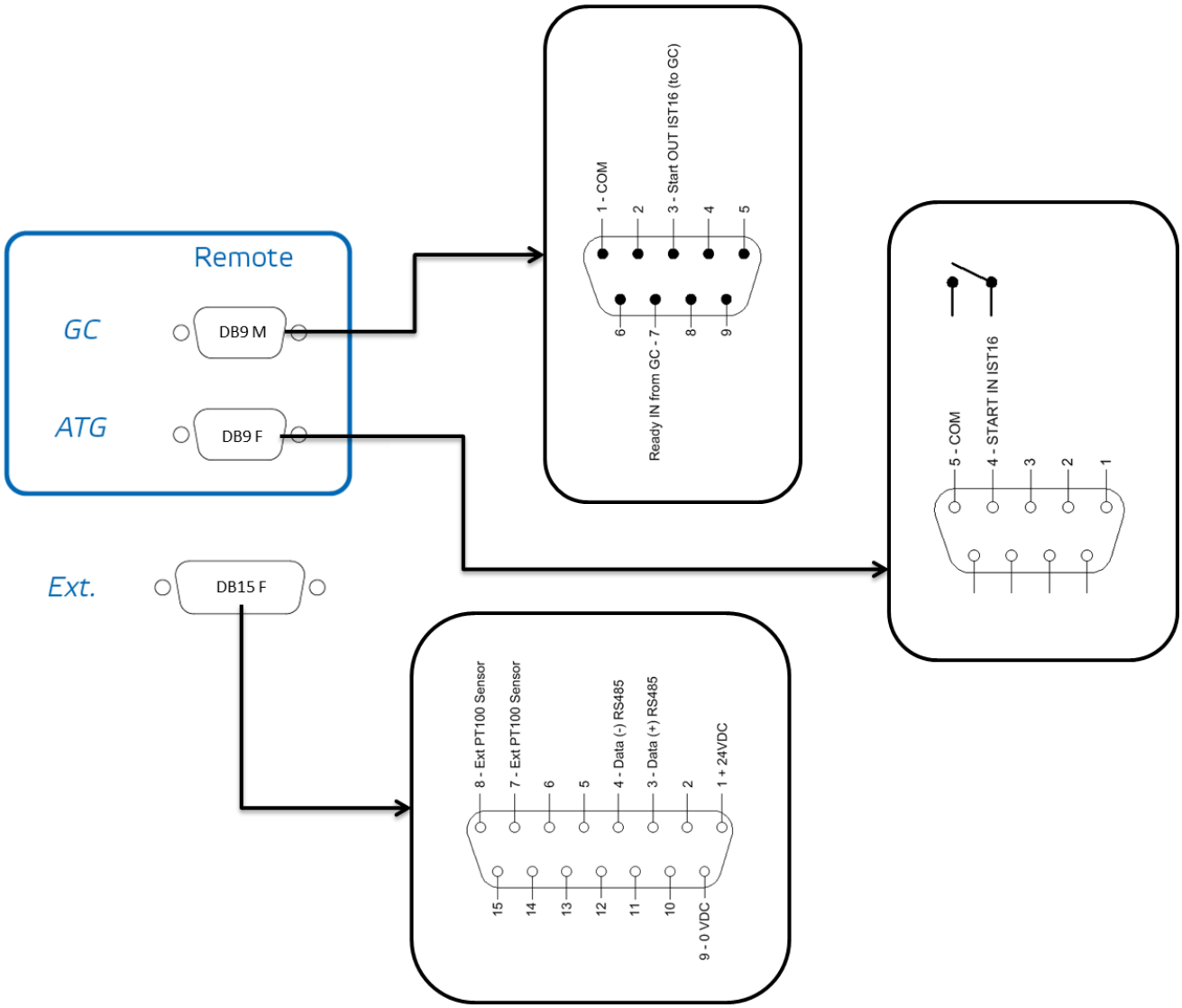
Do not throw away this equipment. Contact a competent recycling organism.

13. About



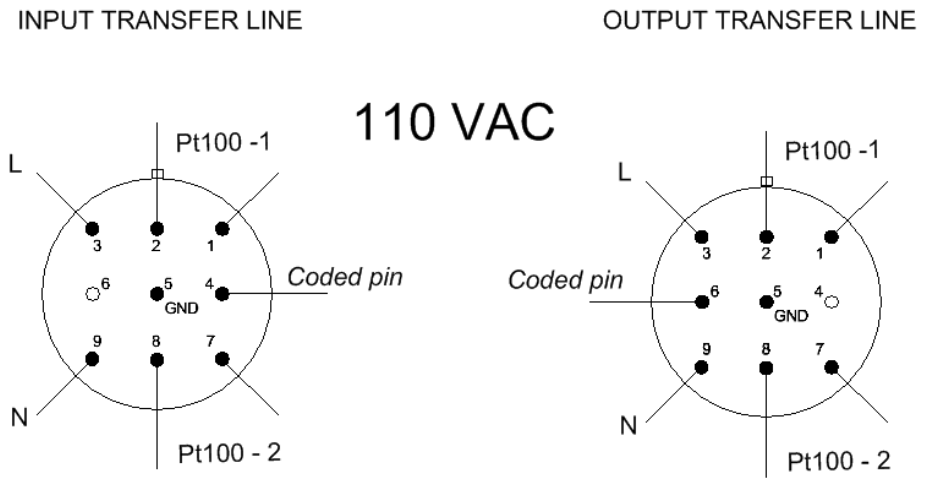
This menu shows the software version.

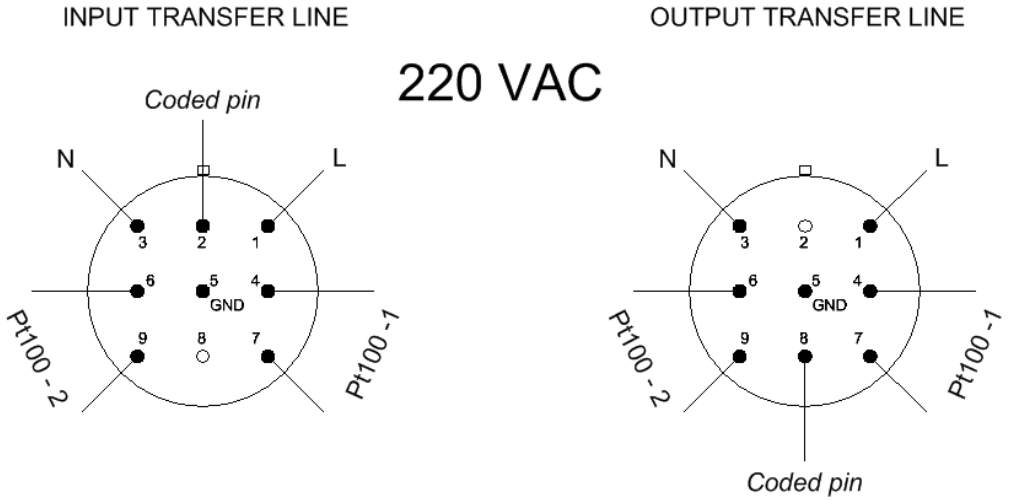
14. Appendix I: IST16 Remote outputs & I/O



15. Appendix II: Connecting the IST16 transfer lines

The power cable depends on the voltage of your IST16:





16. Appendix III: Installation checkout sample

16.1 Sample and conditions

- Sample: Calcium carbonate ($CaCO_3$)
- Measuring cell: TGA/DSC 3+ with SDTA sensor,
  IST16 storage interface,
  Agilent 7890A GC / 5975C MSD.
- Pre-treatment: None
- Sample preparation: See respective figures

16.2 Instrument parameters

16.2.1 TGA

- Reactive/Purge gas: 30 mL/min, N_2 grade 5.0
- Protective gas: 20 mL/min, N_2 grade 5.0
- Method: 500 °C to 1000 °C at 20 °C/min.
- Pan: Alumina 70 µL
- Sample mass: around 10 mg

16.2.2 IST16

Transfer lines and valves temperature: 250 °C

16.2.3 GC

- Inlet port temperature: 280 °C
- Split: 3:1
- Septum purge flow: 1 mL/min
- Column specifications: HP-5 60 m x 0.32 mm x 0.25 µm
- Column flow: 0.8 mL/min.
- Oven temperature program: 50 °C for 4 min, 20 °C/min. to 100 °C, isothermal at 100 °C for 1 min.

16.2.4 MSD

- Scan mode: m/z from 35 to 55
- EMV gain: 1

16.3 Results

16.3.1 TGA/DSC

Figure 1 displays the TGA results using the 70 µL alumina crucible of around 10 mg of calcium carbonate. The method consisted of a heating from 500 °C to 1000 °C at 20 °C/min. Nitrogen was used as protective and reactive gas at 20 mL/min and 30 mL/min flow rate, respectively.

The TGA curve normalized to the initial sample mass is shown in black and the DTG curve is shown in red. The DTG curve corresponds to the first derivative of a TGA curve. In a DTG curve, a TGA step is displayed as a peak. This makes it easier to determine the limits for the TGA step evaluation.

The sample exhibits one decomposition step of about 44 % in the studied temperature range under nitrogen atmosphere. It starts at about 600 °C and has its maximum slope around 780 °C. The measured mass loss of 44 % for the calcination of calcium carbonate agrees well with the expected mass changes. From the stoichiometry of the reaction ($CaCO_3 \rightarrow CaO + CO_2$), the theoretical calculated weight loss is 44 %.

The effect observed at about 900 °C corresponds to IST16 position change from storage mode to injection mode (after collection of the last loop) and is not related to a thermal effect.

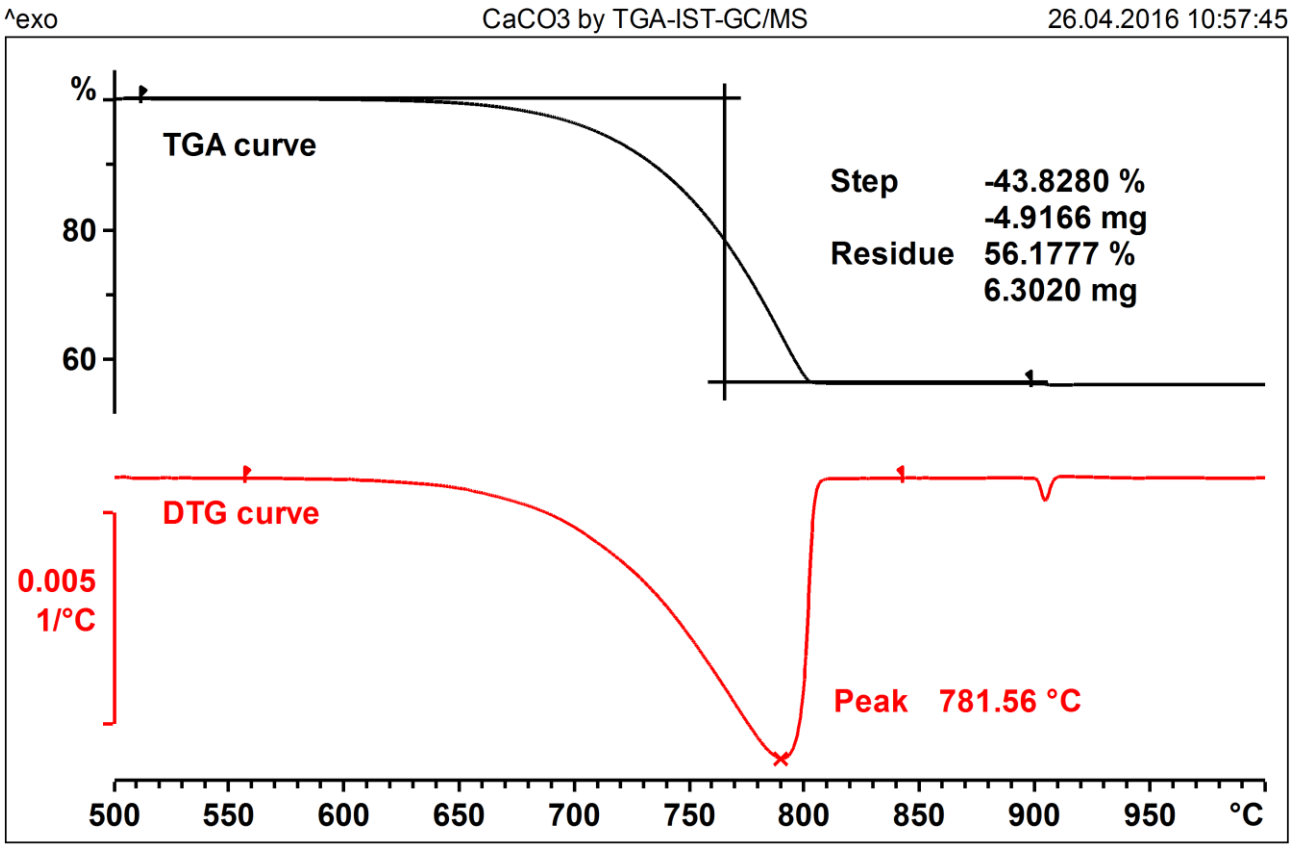


Figure 1: TGA and DTG curves

16.3.2 TGA/IST16/GC-MS

The TGA was coupled to an Agilent 7890B GC / 5977A MS by means of the IST16 storage interface. The same method as described in section 16.3.1 was used for the TGA. The GC inlet temperature was 280 °C using a split 3:1. The oven program was: 50 °C for 4 min followed by a heating at 20 °C/min to 100 °C and an isotherm for 1 min at 100 °C. The column was an HP-5ms of 60 m x 0.32 mm x 0.25 µm. The column flow was 0.8 mL/min. Scan mode from m/z 35 to m/z 55 and an EMV gain of 1 were used for the MS detector. The IST16 interface transfer lines and oven were set to 250 °C. A PTFE filter tube is placed between the TGA outlet and the IST16 transfer line to protect the interface.

Based on figure 1, gas storage temperatures were set according to table 2 below.

IST16 Loop number	1	2	3	4	5	6	7	8	9	10	11
TGA Temp. in °C	520	600	650	700	725	750	775	800	825	850	900

Table 2: Storage loop numbers at selected temperatures

After collection of the loop 11, the samples stored in the 11 loops had been automatically injected one by one into the GC. For example, figure 2 displays *m/z* 44 (for CO_2) of loop 06 (750 °C). The peak observed at around 4.6 minutes corresponds to the CO_2 of the calcination of $CaCO_3$.

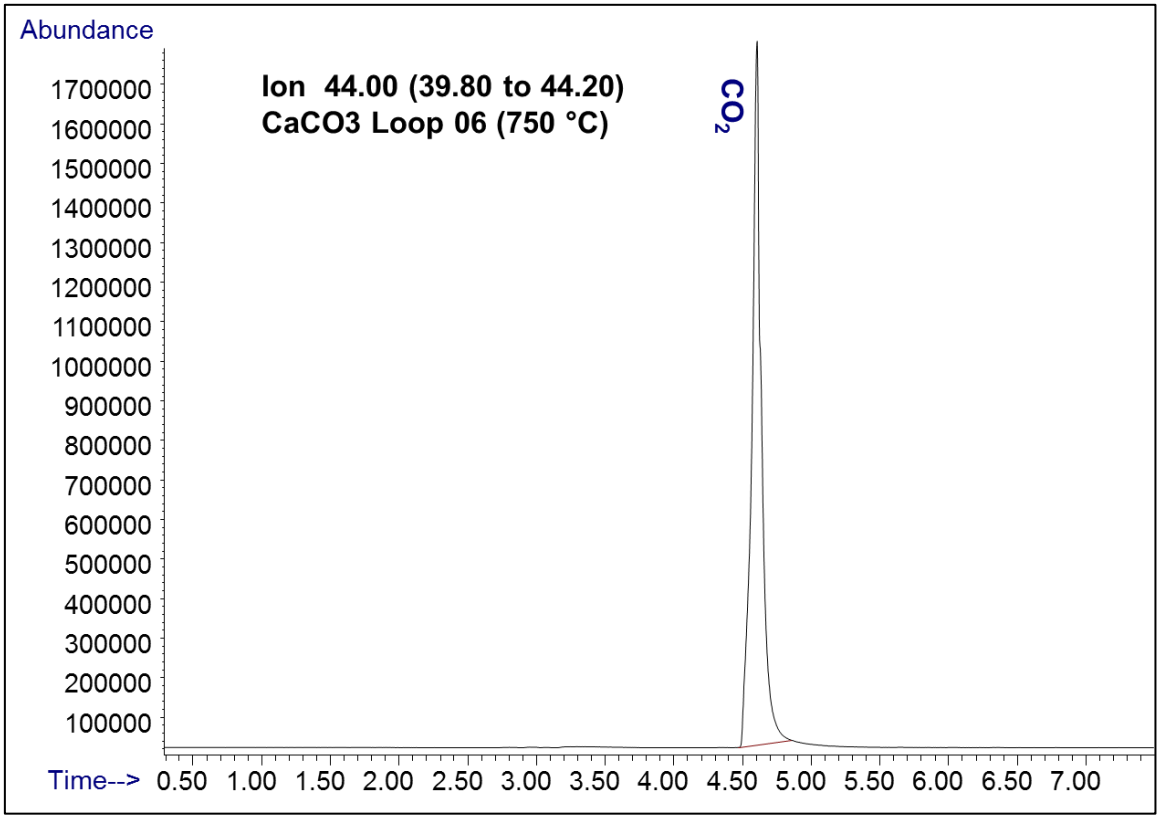


Figure 2: *m/z* 44 of loop 06 (750 °C) (without background subtracted)

Using all loops, emission profiles of CO_2 along the thermal decomposition is obtained. For this purpose, the area of the fragment peak *m/z* 44 in the MS spectrum, characteristic for CO_2 is calculated for each loop. In such a way its temperature dependent evolution profile is established. This is illustrated in figure 3. The TGA curve is shown in black (right axis) and *m/z* 44 is shown in red (left axis).

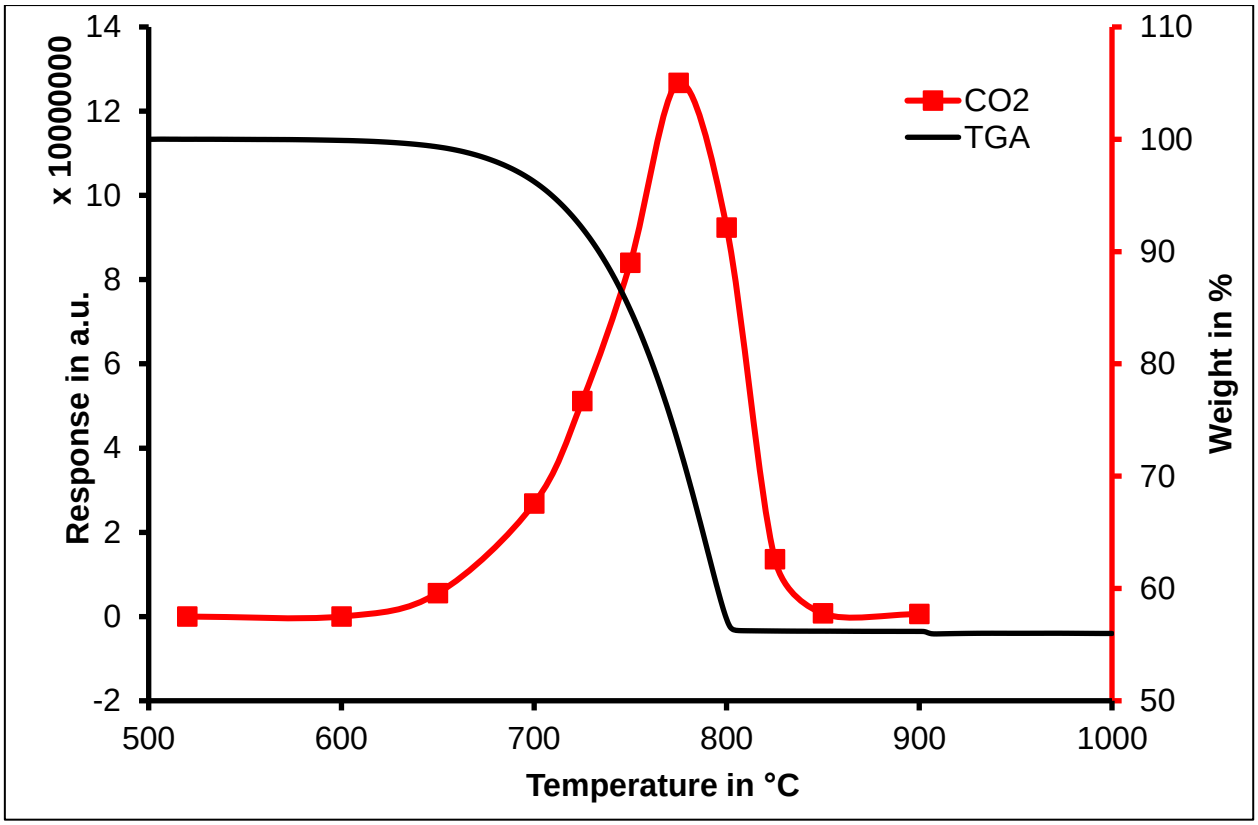
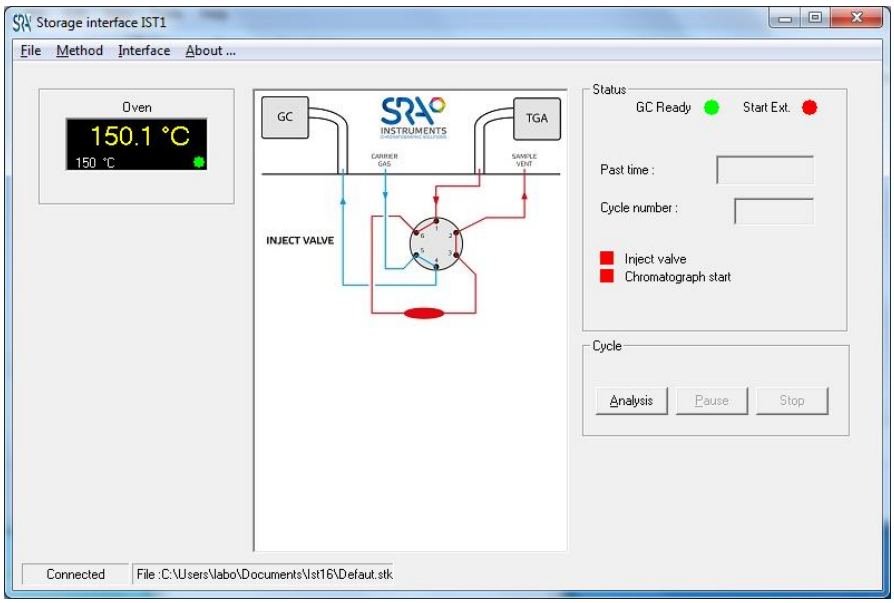


Figure 3: TGA curve and *m/z* 44 evolution profile

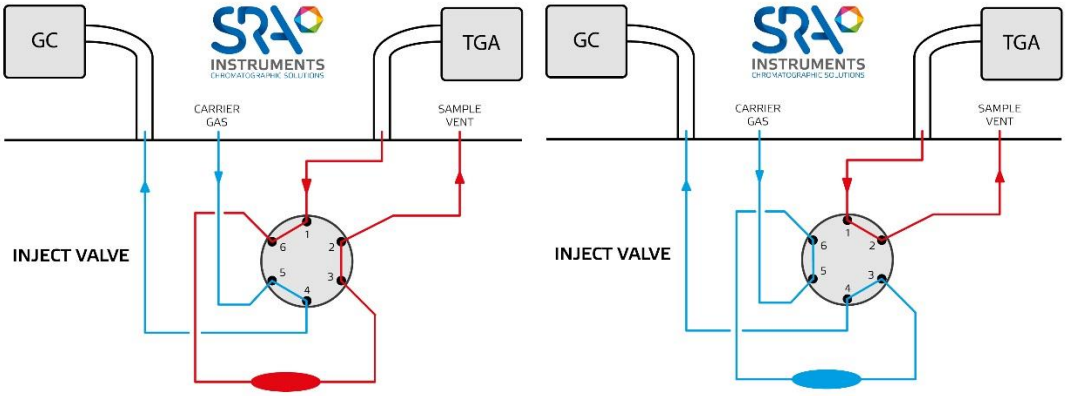
16.4 Conclusion

Calcium carbonate ($CaCO_3$) can be used as a standard for the TGA-IST16-GC/MS system proof of concept for on-site verification. $CaCO_3$ decomposes to CaO and CO_2. The mass loss measured by the TGA should be 44 % according to the stoichiometry of the calcium carbonate calcination ($CaCO_3 \rightarrow CaO + CO_2$). As only CO_2 is evolved, there are no risks of contamination and the GC acquisition method is only 7.5 minutes. As it can be observed in figure 3, CO_2 is still detected even the decomposition process is finished. This can be resolved by either using a lower sample mass such as e.g. 5 mg, a bigger reactive gas flow such as e.g. 50 or 70 mL/min or a bigger split ratio such as e.g. 10:1 or 20:1. The same test should be repeated using other columns delivered with the GC/MS system during installation.

17. Appendix IV: IST1



The IST1 is a special configuration because it is equipped only with a high temperature transfer valve (see diagram) with two positions, the loading position (left) and the injection position (right).



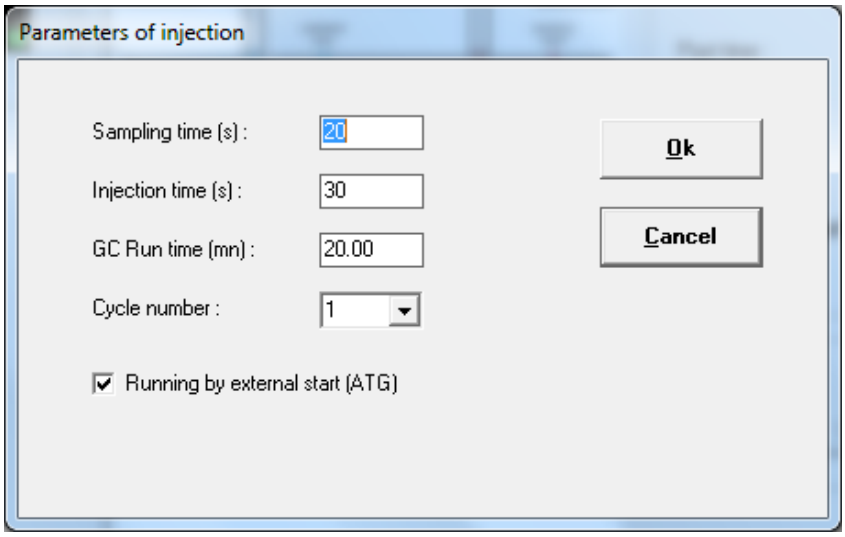
The installation and configuration of the software are similar to the IST16. Only the Gas sampling valve (default) and Multi-injection modes are available.

For maintenance operations, the recommendations are the same as for the IST16.

Note that it is possible to upgrade from an IST1 MONO version to an IST16 version by returning the unit to the manufacturing plant.

17.1 Injection parameters – Gas sampling mode

The injection parameters are available under the **Method\Injection** menu. The following window is displayed:

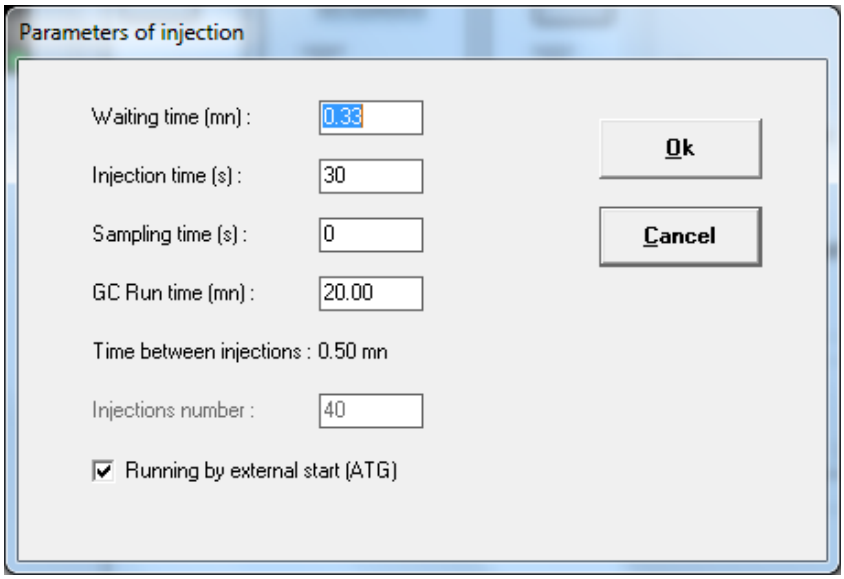


The parameter 'GC Run time' must correspond to the analysis time of the GC method.

It is possible to launch several cycles; each cycle can start on receipt of an external start from the TGA. The recommendations are the same as for use with an IST16.

17.2 Injection parameters – Multi-injection mode

The injection parameters are available under the **Method\Injection** menu. The following window is displayed:



The use is the same as with an IST16.

18. 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

IST16 Storage interface for gas	

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

- NF EN IEC 61326-1:2021 "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 13**

Marcy l'Etoile, 20 January 2023

Legal representative,
Armando MILIAZZA

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