

Storage Interface IST16

User manual



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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



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

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.
- 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.

3. Description

3.1 Presentation

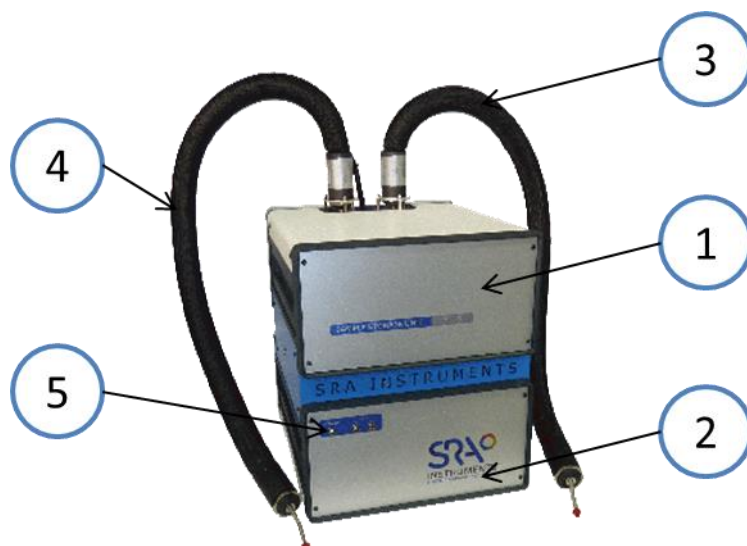
The IST16 is an interface for storing gases, developed by SRA Instruments. It is generally coupled to thermogravimetric balances from different manufacturers (Mettler, Setaram, Netzsch, TA ...). Thus, the IST16 makes it possible to recover and isolate gas fractions resulting from the thermal analysis 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. Power and temperature alarms LEDs on the front panel





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.

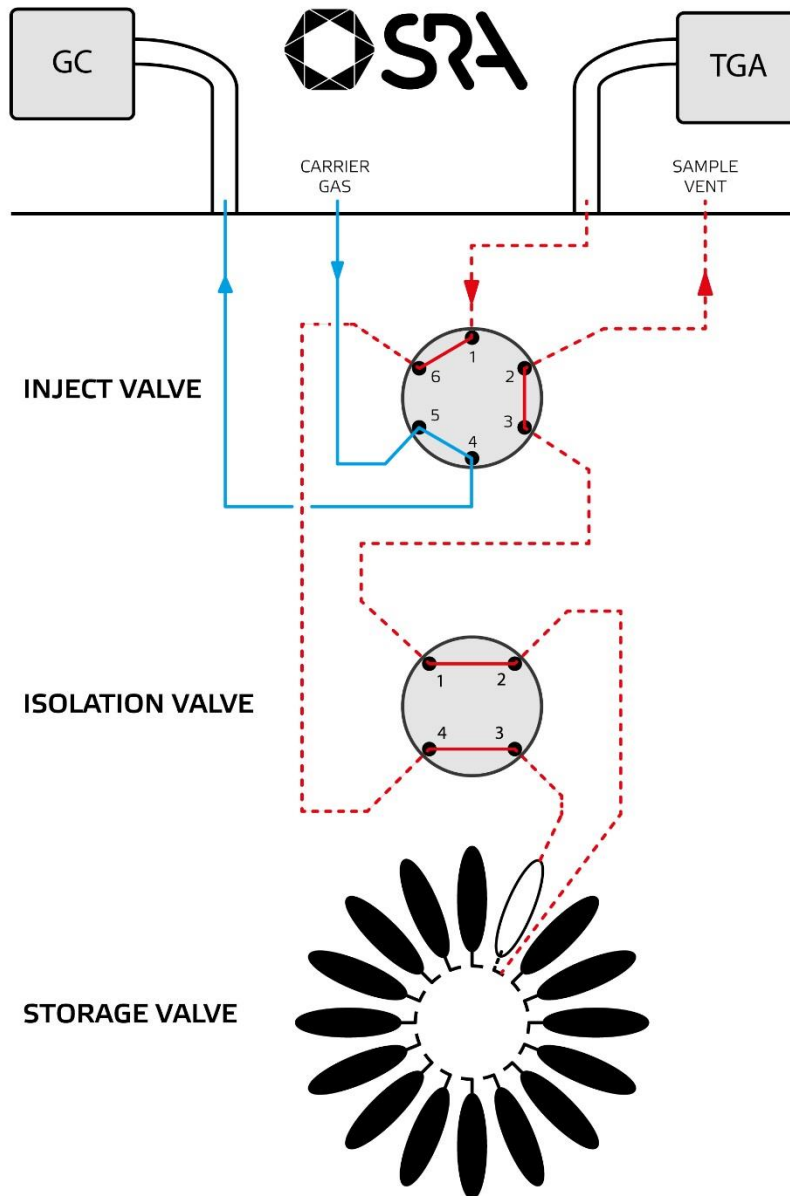
3.3 Fluid flow diagram

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:



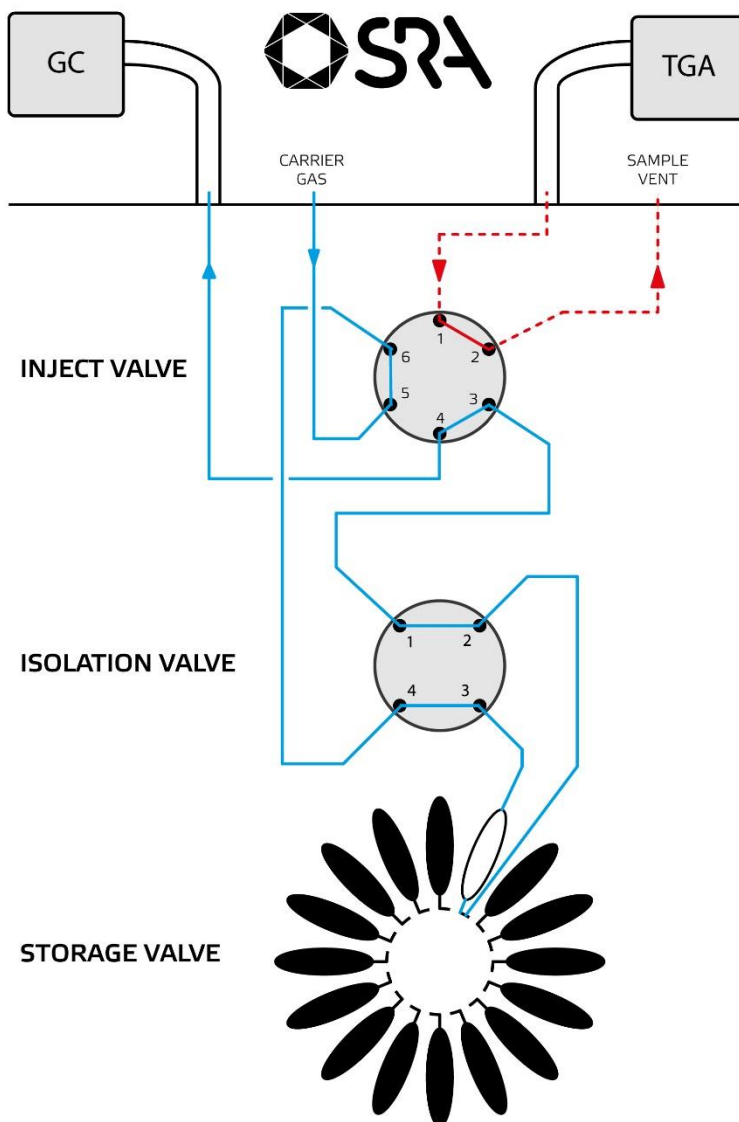
3.3.1 Storage mode



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.

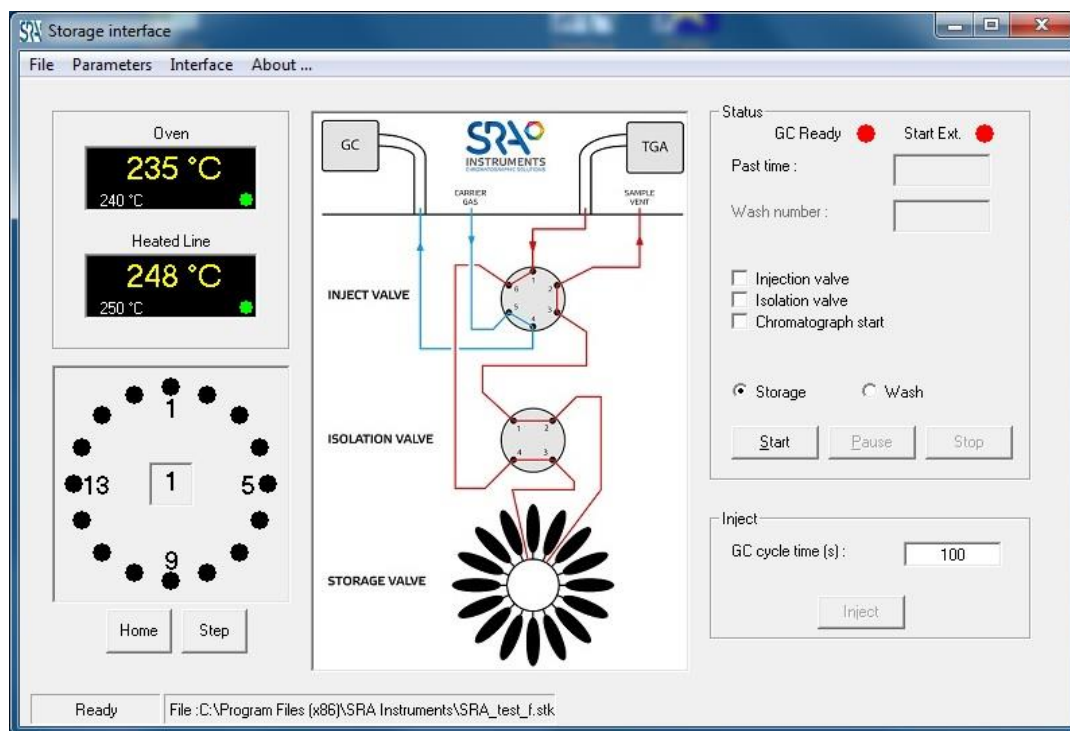
3.4 Automated control software

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.





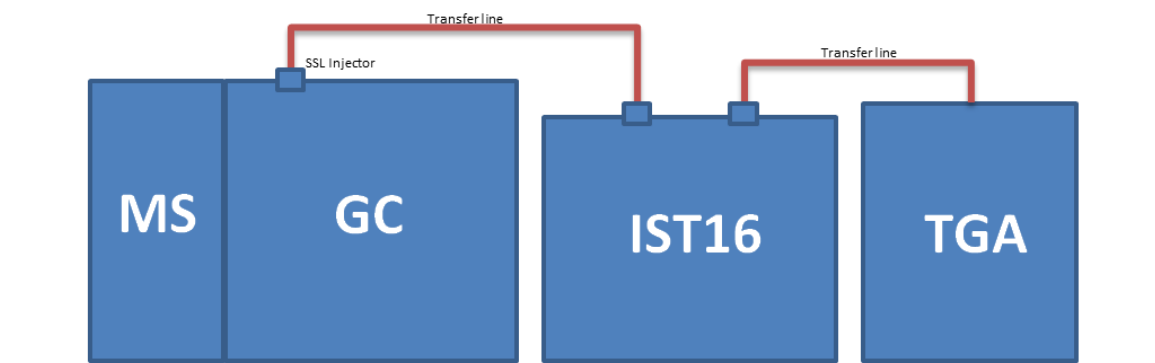
This software is optimized for operation under Microsoft Windows 7 or 10.

4. Installation

4.1 Preparation for the installation

The installation of the IST16 is performed by a 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

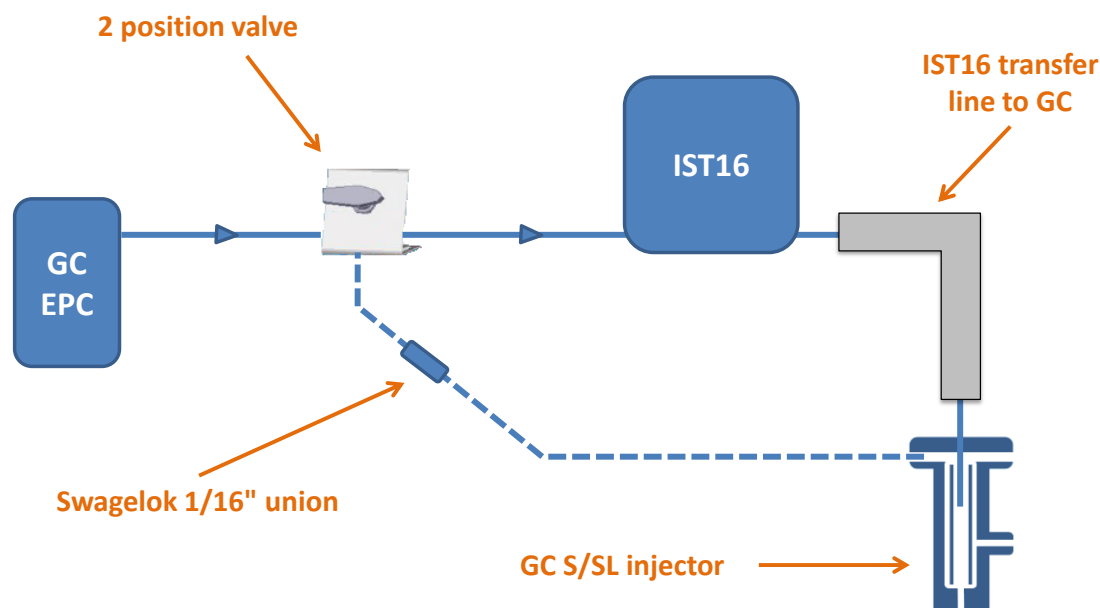


4.2 Modification of the GC carrier gas system

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.





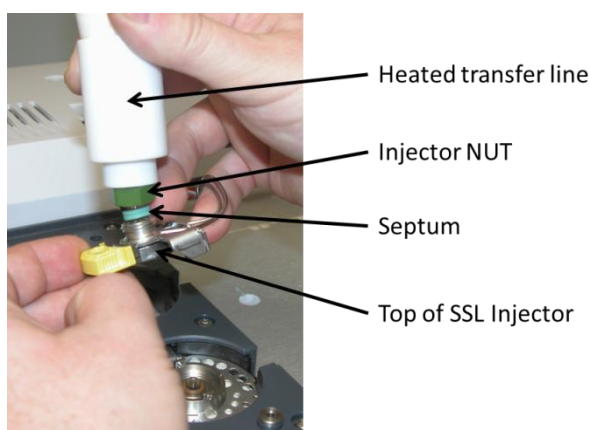
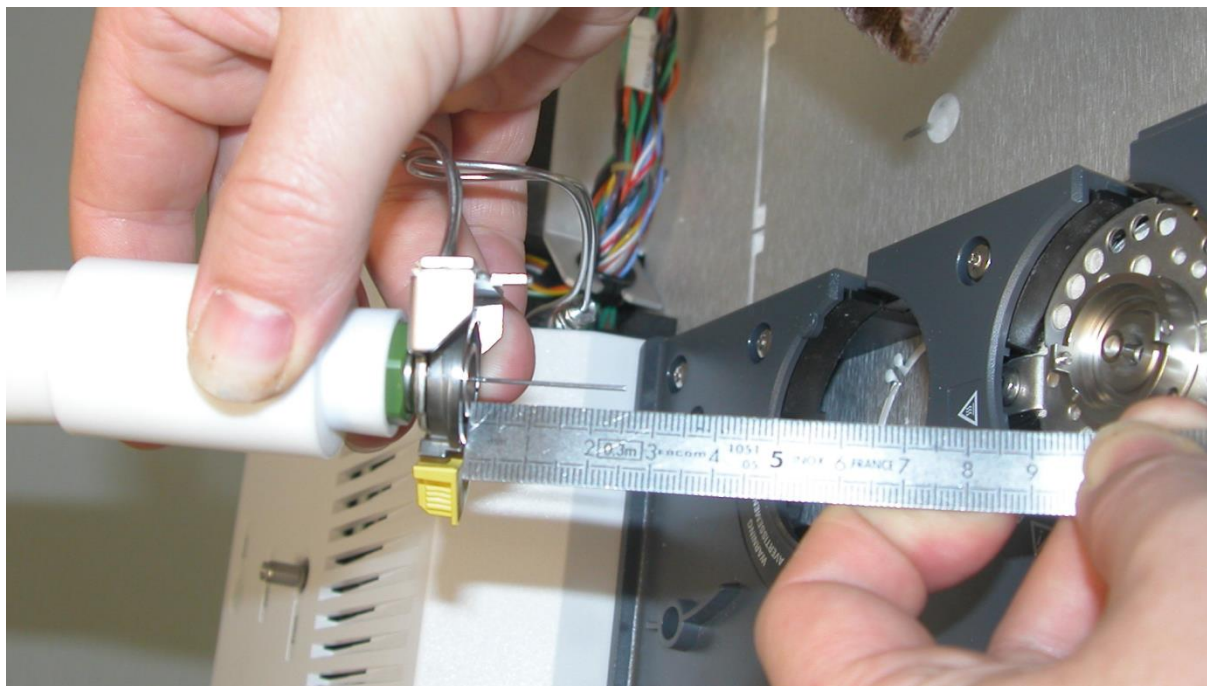
The carrier gas connection to the IST16 is located on the back of the upper panel. The fitting is 1/16" Swagelok type. We recommend that you use new ferrules during installation and follow the coupling manufacturer's recommendations for tightening to ensure the best seal. A leak control with electronic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

**⚠ The use of hydrogen carrier gas for IST16 is not recommended.
It is better to use helium for safety reasons.**

4.3 Installation of the capillary on the GC injector

The Split/Splitless injector line is secured by a bracket on the top of the GC. This transfer line contains an inert stainless-steel capillary of 0.8 mm outer diameter. This capillary must pass into the injector to a depth of about 25mm 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 standard transfer tube of 1/16" diameter to minimize dead volumes. However, in some applications a 1/8" tube may be preferable. It is possible to introduce a 1/8" tube in the transfer line and to perform a reduction inside the valve compartment.

Depending on the type of connection and type of thermo-balance, it may be desirable to mechanically maintain the transfer line in order to do not disturb the TGA or to break the fragile parts of the 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 hardware, see 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 do not disturb the gas flow through the interface.

5. Configuring communication for the Ethernet converter

The valves and heated parts of the storage interface are operated with a serial communication protocol, but to connect the IST16 to the computer it is necessary to use an Ethernet connection. An ADAM 4570 module allows conversion of the transmission from LAN to RS485 and RS232 serial port.

PORT	TYPE
1	RS485 – Module 4022T (temperature control and I/O)
2	RS232 – Valve control

The default IP address for the IST16 is:
10.1.1.103

Configure the IP address and subnet mask of your computer to be compatible with the default address of your IST16 and connect the computer with the storage interface via the LAN cable. Turn on the IST16.

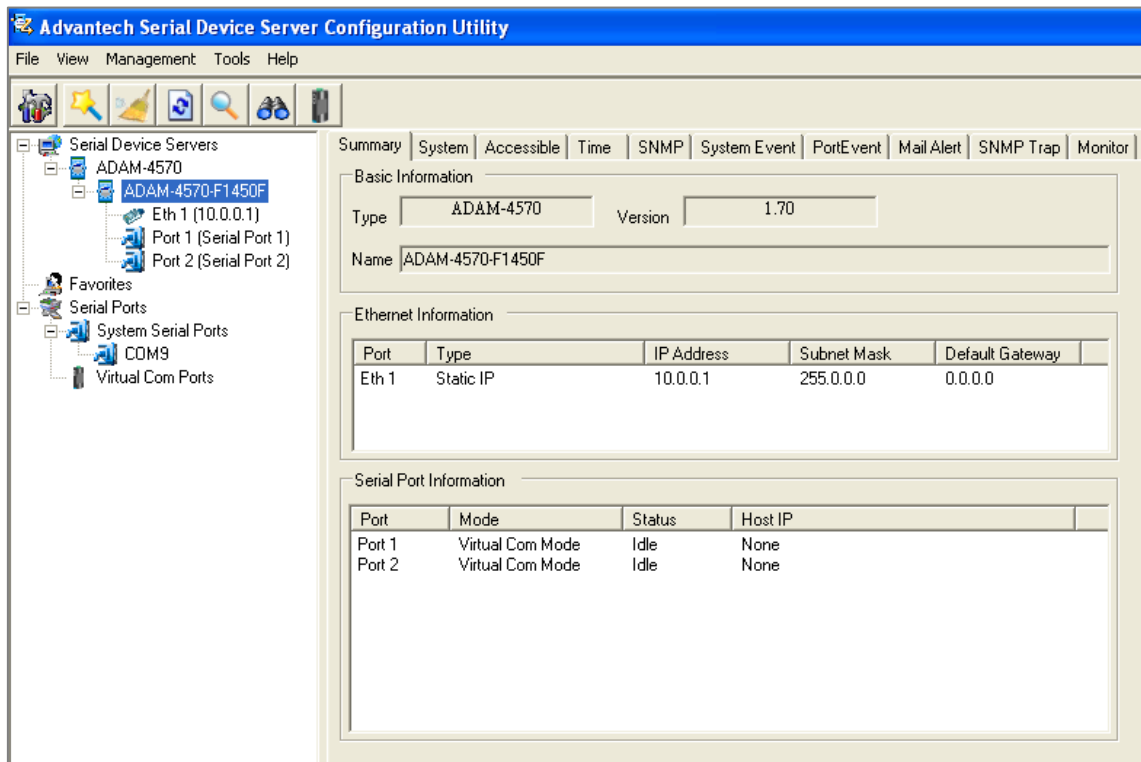
The following steps describe how to configure the ADAM 4570 module. From the provided CD, install **Serial Device Server Configuration Utility** from the Adam 4570 directory and **+\\Utility&Driver\\Serial Device Server Configuration Utility**. If the Windows Firewall requests network permissions for this program, choose the Full Access option (home or workplace).

! To launch **Serial Device Server Configuration Utility** you must right-click on the file and run it as administrator.

After installation, start the program using this shortcut on the desktop:



The main screen is displayed, and the Adam 4570 module is automatically detected if the Ethernet connection is correctly set.



5.1 MAC address

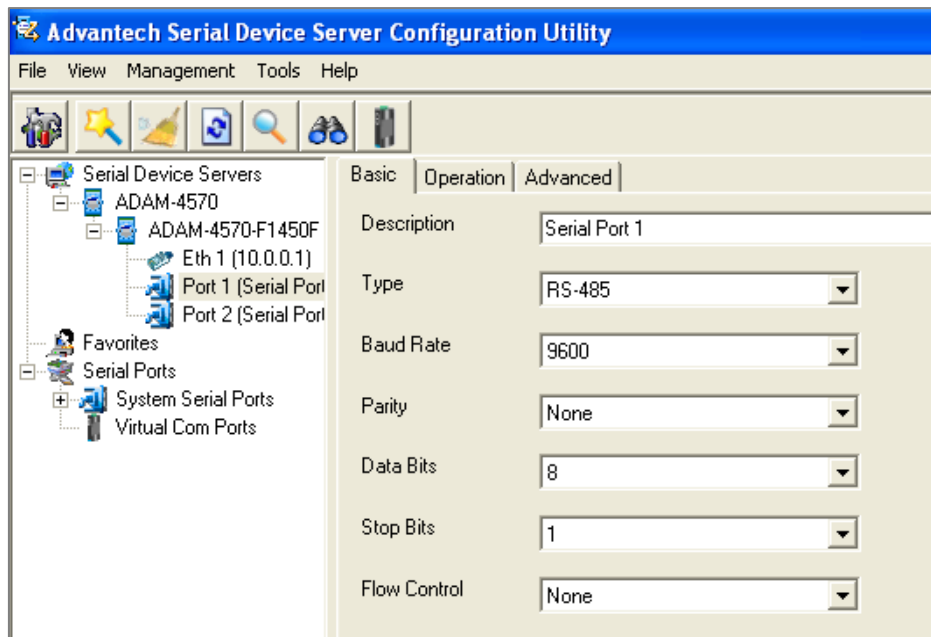
To obtain the MAC address if necessary:

1. Connect to the module as described above.
2. Open a DOS window on the PC by the menu Start\Run and enter the command cmd or command.
3. When the window is opened, enter : Arp -a
4. Get the MAC address in the line corresponding to the module address.

5.2 Ports configuration

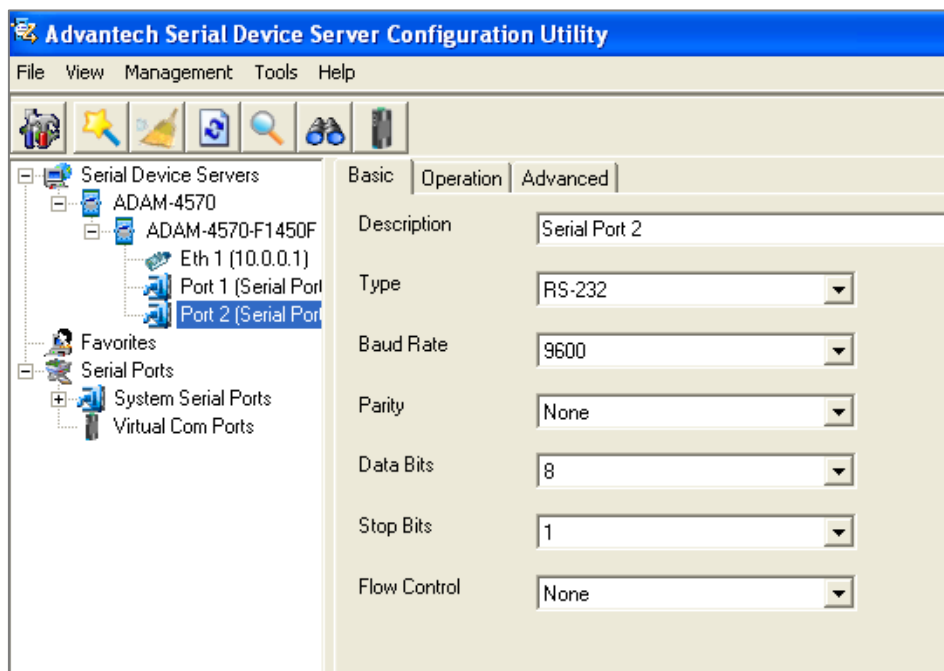
Double-click on **Port 1** and define it as below (RS-485):





Then click on **Apply** or **Apply All Ports** to save and activate the new configuration.

Double-click on the port 2 and define it as below (RS-232):



Then click on **Apply** or **Apply All Ports** to save and activate the new configuration.

During the update, the module resets itself and thus disappears from the screen. It takes a few seconds to see it appear again.

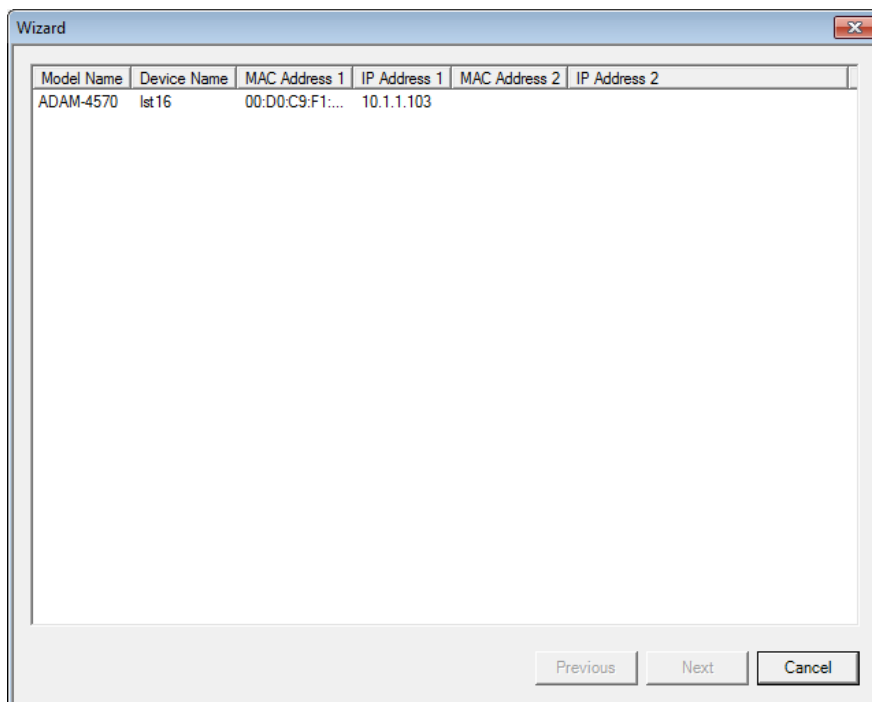


5.3 Serial ports installation

It is now necessary to create the two virtual ports. To do this, select the **Management** menu and click on **Configuration Wizard**.



The following window is displayed:



Select the ADAM-4570 module and click on **Next**.

Select RS-485 as serial port and click on **Next**.



The 'Wizard' window displays configuration options for a device named 'Ist16'. It includes sections for 'Device Setting' (Name: Ist16), 'Serial Port Setting' (Type: RS-485), and 'Ethernet 1' (MAC Address: 00:D0:C9:F1:5B:F2, IP Address: 10.1.1.103, Subnet Mask: 255.255.255.0, Default Gateway: 0.0.0.0). Navigation buttons at the bottom are 'Previous', 'Next', and 'Cancel'.

Select the virtual serial port address to associate it with Port 1/RS-485 and then click on **Finish** (usually COM 4).

The 'Wizard' window shows the 'From System Port' dropdown set to 'COM 4'. It features 'Select All' and 'Clear All' buttons. Below is a table for port selection:

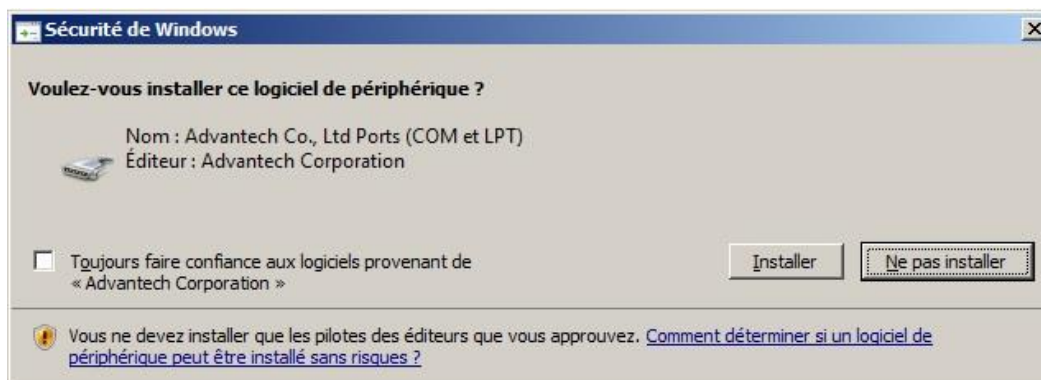
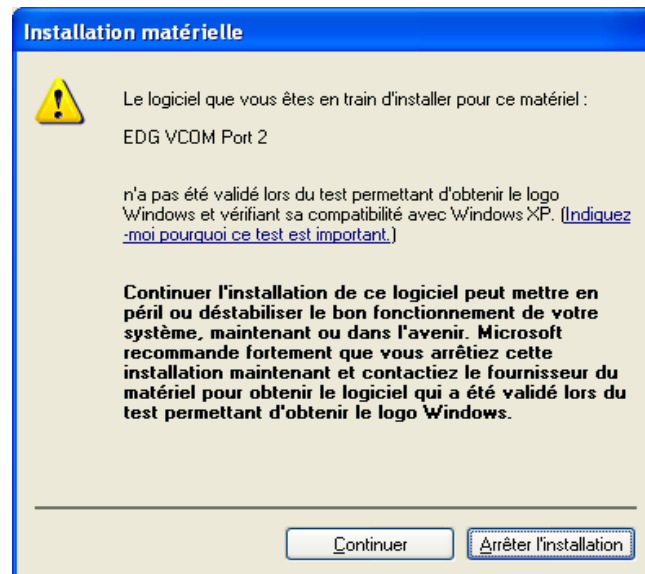
IP 1	IP 2	Device Port	System Port
<input checked="" type="checkbox"/> 10.1.1.180		Port 1	COM 4
<input type="checkbox"/> 10.1.1.180		Port 2	

Navigation buttons at the bottom are 'Previous', 'Finish', and 'Cancel'.

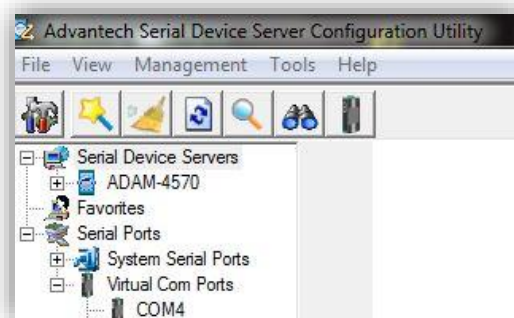
The port installation begins. This may take some time.

Windows may display one of the screens below; click respectively on Next or Install.





When the installation is finished, a message appears: **"Wizard complete!"** The main screen of the software displays the virtual ports in series.



 **This operation must be repeated for the second virtual port (Port 2/RS-232).**

Return to the previous window using the **Configuration Wizard**. For this window, select RS-232 as the serial port type and click on **Next**.



The 'Wizard' window displays configuration options for a device named 'Ist16'. It includes sections for 'Device Setting', 'Serial Port Setting', and 'Ethernet 1'.

Section	Field	Value
Device Setting	Name	Ist16
	Type	RS-232
Ethernet 1	MAC Address	00:D0:C9:F1:5B:F2
	IP Address	10.1.1.103
	Subnet Mask	255.255.255.0
	Default Gateway	0.0.0.0

Navigation buttons at the bottom: Previous, Next, Cancel.

Select the virtual serial port address to associate it with Port 2/RS-232 and then click on **Finish** (usually COM 5).

The 'Wizard' window shows the 'From System Port' dropdown set to 'COM 5'. Below is a table for selecting IP addresses and associating them with device ports.

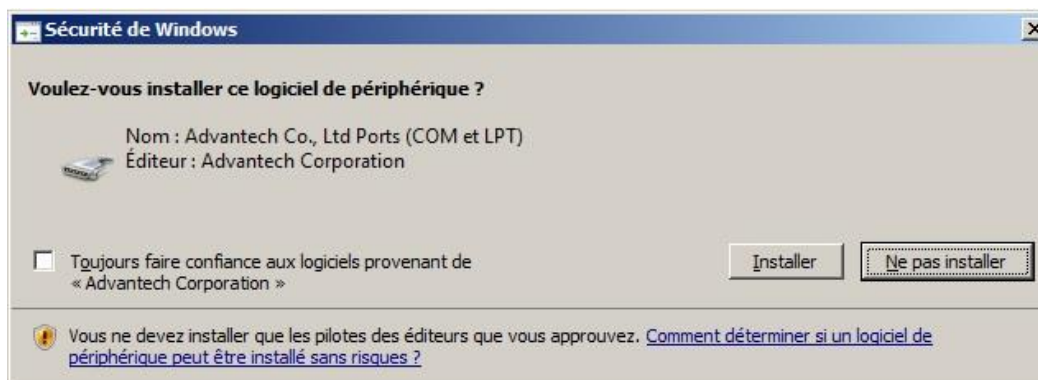
IP 1	IP 2	Device Port	System Port
<input type="checkbox"/> 10.1.1.180		Port 1	
<input checked="" type="checkbox"/> 10.1.1.180		Port 2	COM 5

Buttons at the bottom: Previous, Finish, Cancel.

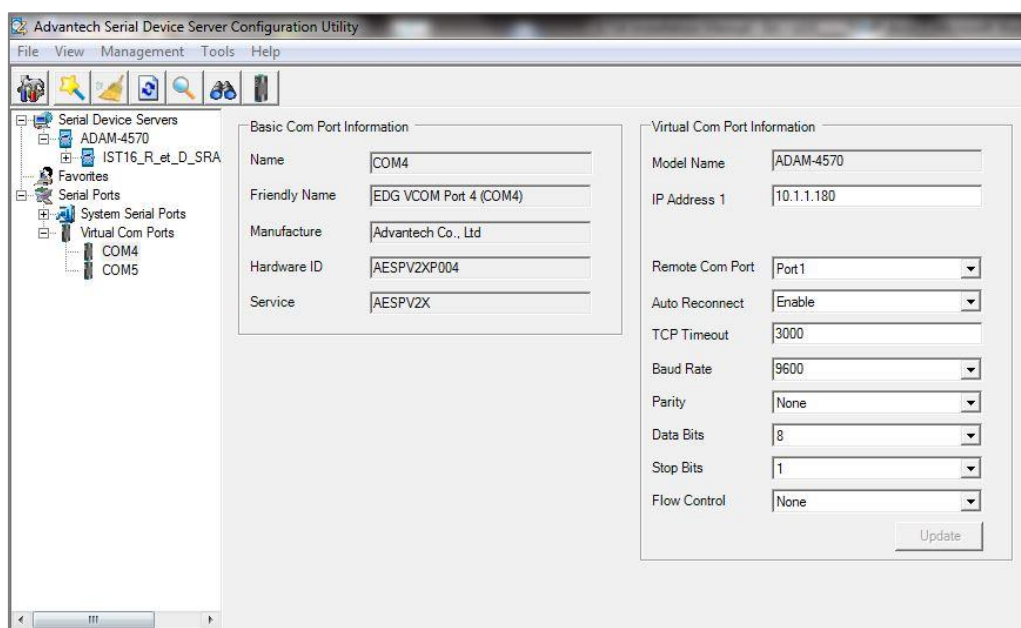
The port installation begins. This may take some time.

Windows may display one of the screens below; click respectively on Next or Install.





When the installation is finished, a message appears: **"Wizard complete!"** The main screen of the software displays the virtual ports in series.



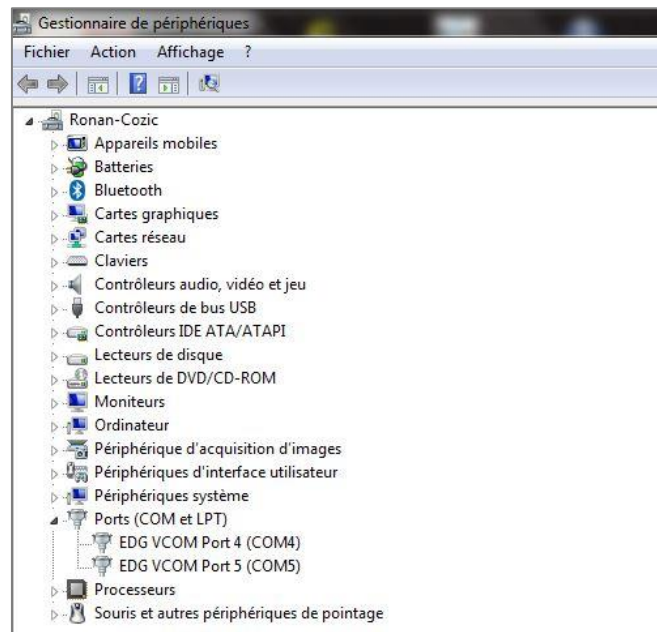


When the configuration of the IST16 is completed, it is necessary to reset it so that the new parameters are taken into account.

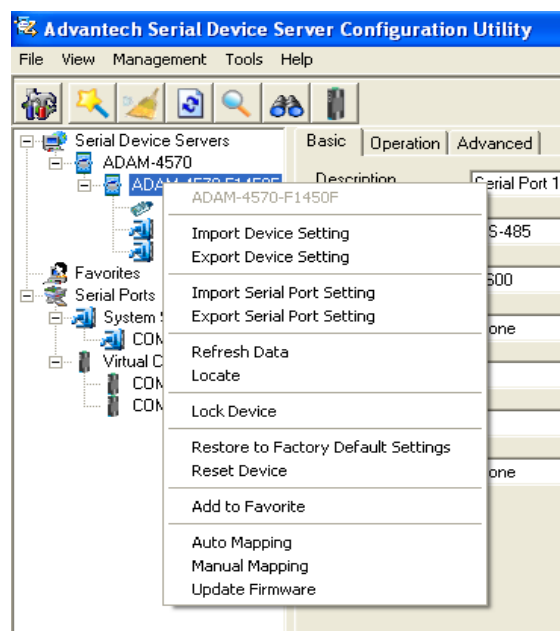
It is not necessary to restart Windows, the ports are automatically recognized.

It is important to set the **Auto Reconnect** parameter to **Enable** (see the window above) so that the port is automatically reconnected each time the computer is started.

When the installation of the virtual ports is completed, you can check that they are correctly detected by using the **Microsoft Windows Device Manager**:



The configuration can be saved.



6. Configuration and installation of the IST16 software

6.1 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.
8. In the new window, check the box "Launch IST16" to launch the application as soon as you exit the installation.
9. Click on the "Finish" button to close the installation of the application.

6.2 Configuration of the valves

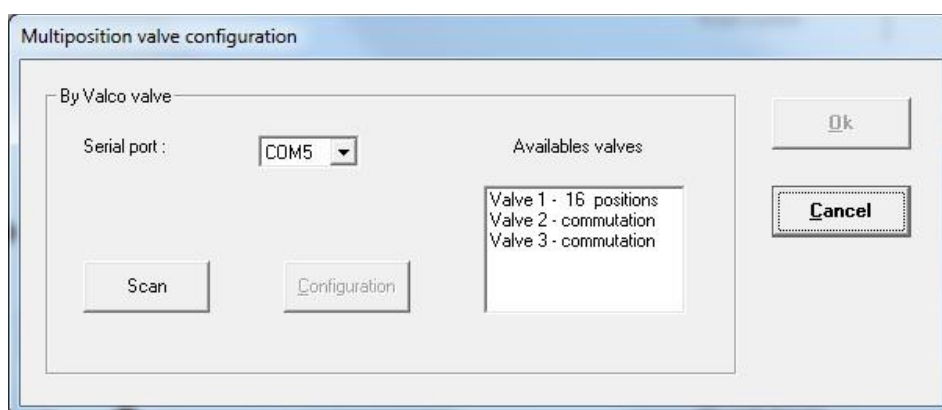
The advanced configuration of the instrument can be accessed via the **Interface\Configuration** menu. You must enter the password and the advanced login.

Login: **System**

Password: **0478442947**

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

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

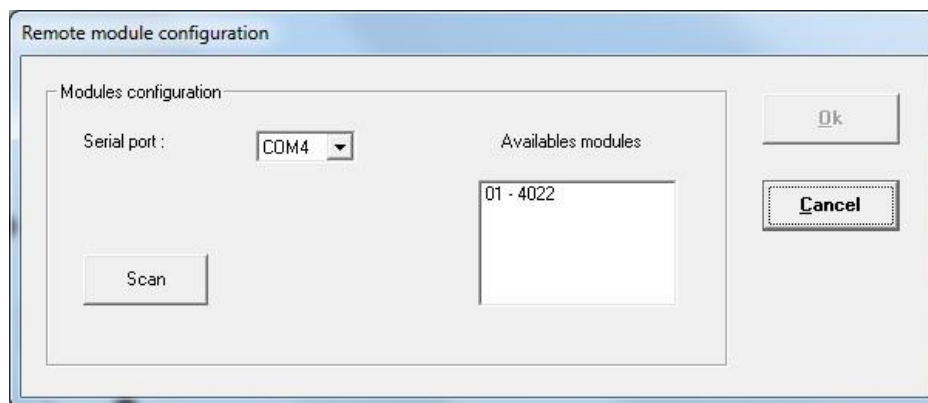


You must select the virtual serial port of the computer associated to Port 2/RS-232 (usually COM 5). You must then click on the **Scan** button to check that the internal rotation of the IST16 valves is effectively detected.

- Valve 1, multi-position valve
- Valve 2, 4 ways switching valve used for the isolation
- Valve 3, 6 ways switching valve used for the injection

6.3 Configuration of the regulation module

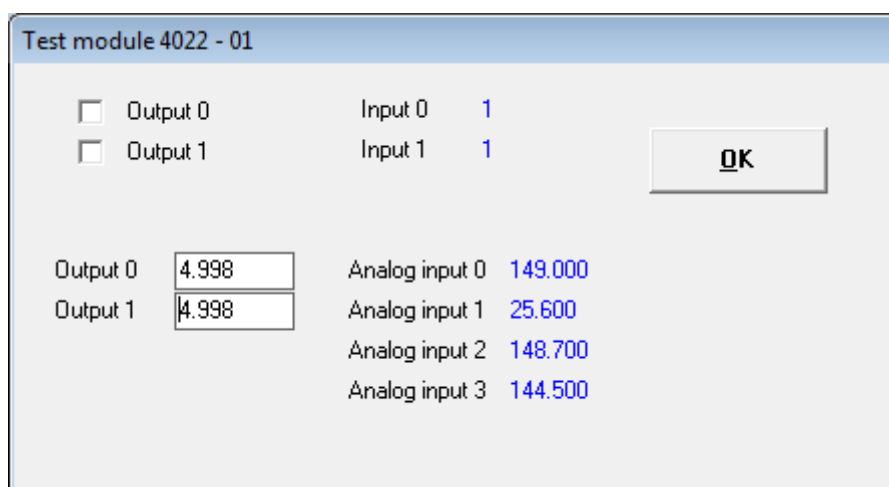
Select the **Interface\System\Remote Module** menu. The following page is displayed:



You must select the serial port of the computer associated to Port 1/RS-485 (usually COM 4). You must then look for the different modules installed in the box with the **Scan** button. In order to validate the configuration, click on the **Ok** button.

6.4 Test of the heating regulation module

When you set the serial connection port of the regulation module, a **Test** button can be displayed according to your access rights. Clicking on this button displays the following window.

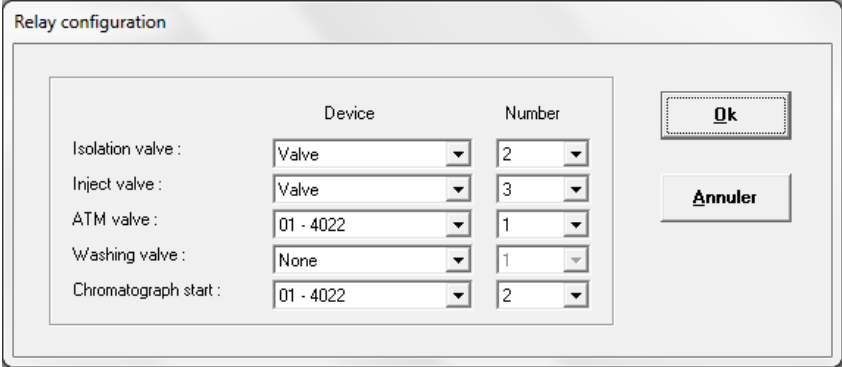


You can then check the operation of the module.



6.5 Configuration of the relays

It is possible to check the configuration of the relays in the following window. To access it, select the **Configuration\Relay** menu. This is the default configuration automatically offered after the software is installed.

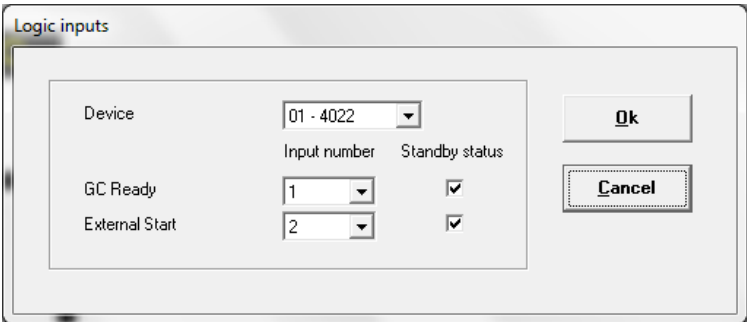


The 'Relay configuration' window displays a table for configuring various valves and the chromatograph start. The table has two columns: 'Device' and 'Number'. The rows are: Isolation valve, Inject valve, ATM valve, Washing valve, and Chromatograph start. The 'Device' column contains dropdown menus with values: Valve, Valve, 01 - 4022, None, and 01 - 4022 respectively. The 'Number' column contains dropdown menus with values: 2, 3, 1, 1, and 2 respectively. To the right of the table are two buttons: 'Ok' and 'Annuler'.

	Device	Number
Isolation valve :	Valve	2
Inject valve :	Valve	3
ATM valve :	01 - 4022	1
Washing valve :	None	1
Chromatograph start :	01 - 4022	2

6.6 Configuration of the logic inputs

It is possible to check the configuration of the logic inputs in the following window. To access it, select the **Configuration\Logic inputs** menu. This is the default configuration automatically offered after the software is installed.



The 'Logic inputs' window displays a table for configuring logic inputs. The table has three columns: 'Device', 'Input number', and 'Standby status'. The rows are: GC Ready and External Start. The 'Device' column contains a dropdown menu with value: 01 - 4022. The 'Input number' column contains dropdown menus with values: 1 and 2 respectively. The 'Standby status' column contains checkboxes with values: checked and checked respectively. To the right of the table are two buttons: 'Ok' and 'Cancel'.

Device	Input number	Standby status
GC Ready	1	<input checked="" type="checkbox"/>
External Start	2	<input checked="" type="checkbox"/>

The **GC Ready** parameter is used by the IST16 to check the Ready status of the gas chromatograph before injecting the samples stored in the column.

The **External Start** parameter is used to start the storage sequence when the TGA begins its temperature ramp.

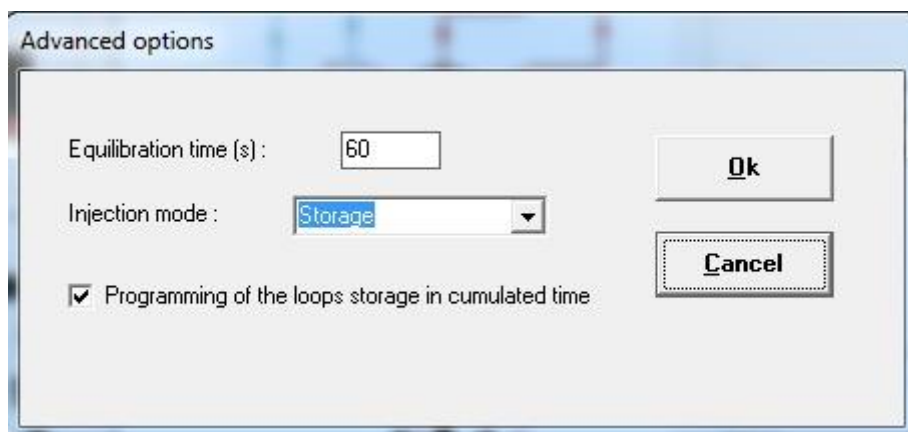
6.7 Advanced options

There are two ways to use the interface and two different modes for entering the different times.

The selection of these modes can be done via the menu **Interface\Configuration\Advanced options**.

The following page is displayed:





The "equilibration time" is the waiting time when the heating of the oven and the lines reach, at $\pm 5^{\circ}\text{C}$, the required temperatures. The "Ready" state of the interface is obtained after this time. If this time is zero, no temperature control is performed.

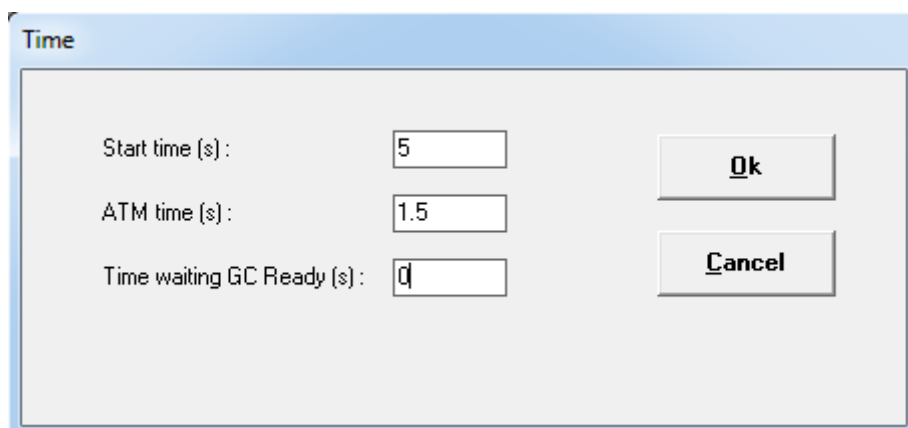
By default, the interface is used in the storage mode. You can also use the injection valve mode which will be described in the chapter 8.

By default, the storage and rinsing times are entered as durations. You can change this mode so that you can enter them as scheduled times from 0.

6.8 Delay of the orders

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\Time** menu.

The following page is displayed:



Delay configuration

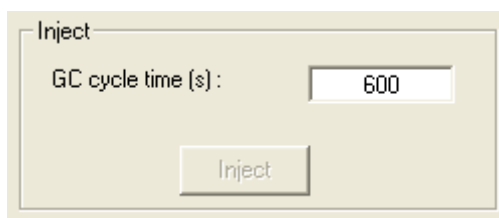


7. Operate with the IST16 software

The IST16 typical sequence is as following:

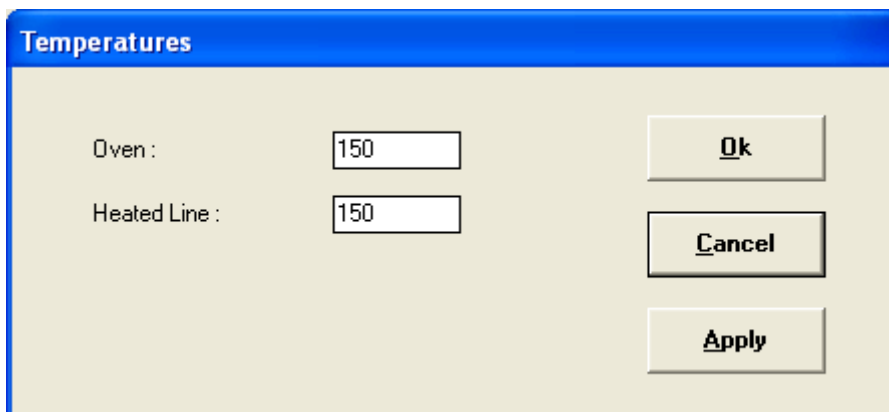
1. First step: storage of the sample in different loops at programmed time.
2. Second step: loops are injected and analyzed one after the other.
3. Third step: loops are cleaned with the flowing gas coming from the TGA at the end of the sequence of GC analyses.

The storage sequence can be initiated only when all temperatures of the storage interface are stable. Before each injection of a loop, it is checked by software that the gas chromatograph is in a ready state (green color). As an alternative for special GC or other devices, a "GC cycle time parameter" can be used to set a delay between two injection loops. If "GC cycle time" is not needed, it is recommended to set it to 600 seconds as below (shorter than the GC analysis time).



7.1 Temperatures

Valves oven and transfer line temperature can be changed via the **Parameters\Temperatures** menu. The following window will appear.



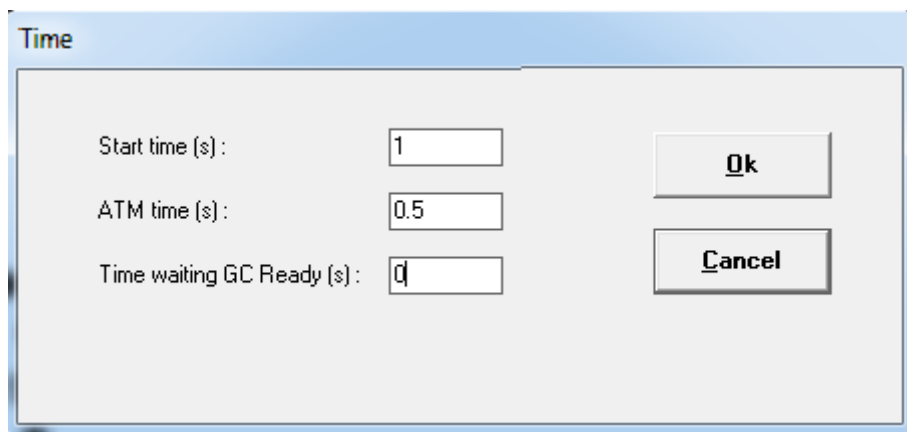
It is possible to set both temperatures in the range 150 - 250°C. Click on "Apply" to send values to the storage interface, then click on "Ok" and save the IST16 method.



Note that when 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 rotary valves.

7.2 Time

Some commands must remain active for a while. The following parameters have been optimized during the tests at the end of the IST16 manufacturing and must not be modified by a non-specialist.



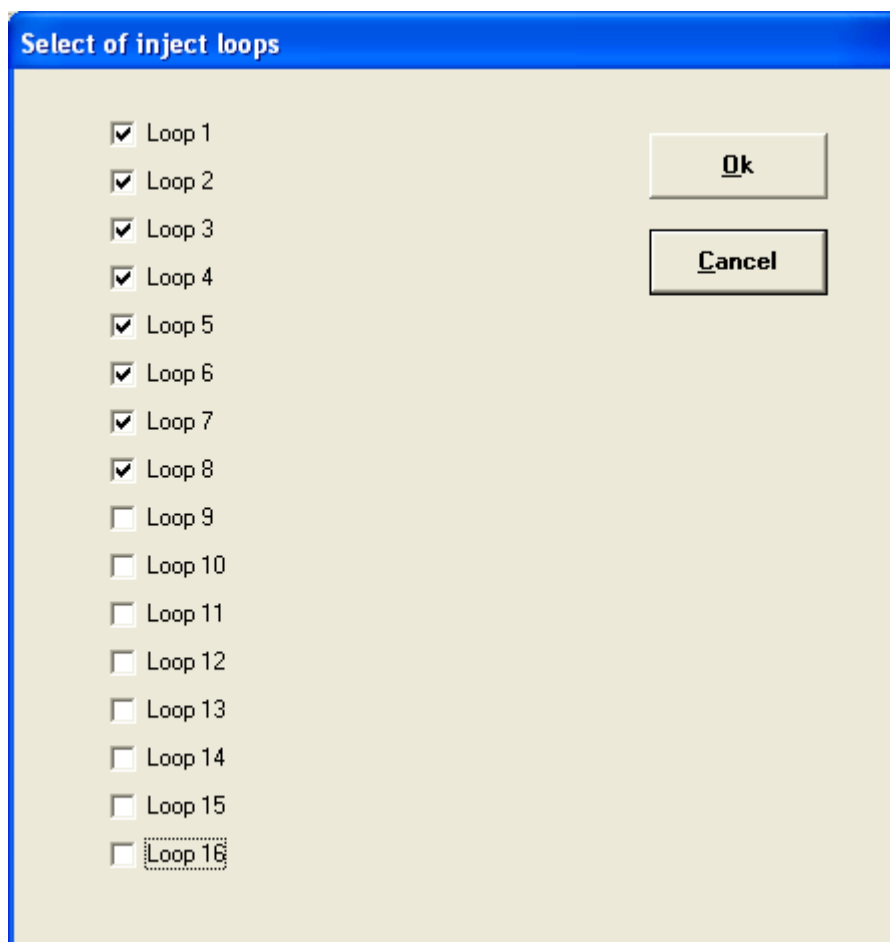
The screenshot shows a software dialog box titled "Time". It contains three labeled input fields: "Start time (s) :" with the value "1", "ATM time (s) :" with the value "0.5", and "Time waiting GC Ready (s) :" with the value "0". To the right of these fields are two buttons: "Ok" and "Cancel".

7.3 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.





7.4 Store time and wash time

During the storage sequence of an experiment, the multi-position valve allows one loop at a time to be flushed with the evolved gas for a certain time. This time is set in the window below accessible from the **Interface\Store time and wash time** menu. A fraction of the evolved gas (typically 250 µL) will be stored in one of the 16 loops at the end of each flushing time.

In addition, you must enter a wash time for each loop (typically from 30 to 300 seconds). This wash time is the duration of the cleaning step of each loop that will be achieved after the end of the injection sequence.



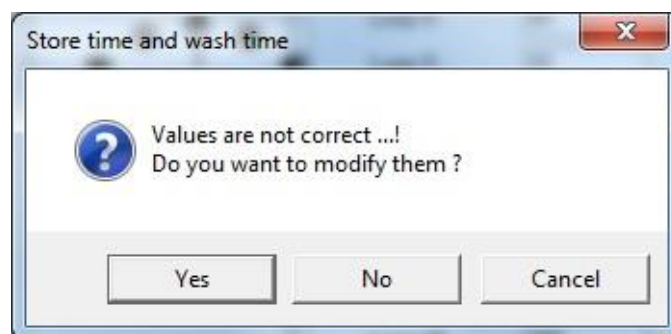
Store time and wash time

Programming of the loops storage in cumulated time

	Storage	Wash
Loop 1 :	<input type="text" value="10"/>	<input type="text" value="10"/>
Loop 2 :	<input type="text" value="20"/>	<input type="text" value="30"/>
Loop 3 :	<input type="text" value="40"/>	<input type="text" value="60"/>
Loop 4 :	<input type="text" value="60"/>	<input type="text" value="90"/>
Loop 5 :	<input type="text" value="80"/>	<input type="text" value="120"/>
Loop 6 :	<input type="text" value="100"/>	<input type="text" value="150"/>
Loop 7 :	<input type="text" value="120"/>	<input type="text" value="180"/>
Loop 8 :	<input type="text" value="140"/>	<input type="text" value="210"/>
Loop 9 :	<input type="text" value="160"/>	<input type="text" value="240"/>
Loop 10 :	<input type="text" value="180"/>	<input type="text" value="270"/>
Loop 11 :	<input type="text" value="200"/>	<input type="text" value="300"/>
Loop 12 :	<input type="text" value="220"/>	<input type="text" value="330"/>
Loop 13 :	<input type="text" value="240"/>	<input type="text" value="360"/>
Loop 14 :	<input type="text" value="260"/>	<input type="text" value="390"/>
Loop 15 :	<input type="text" value="280"/>	<input type="text" value="420"/>
Loop 16 :	<input type="text" value="300"/>	<input type="text" value="450"/>
Total time :	300 secs	

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 “Ok” 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 scanned and stored properly.

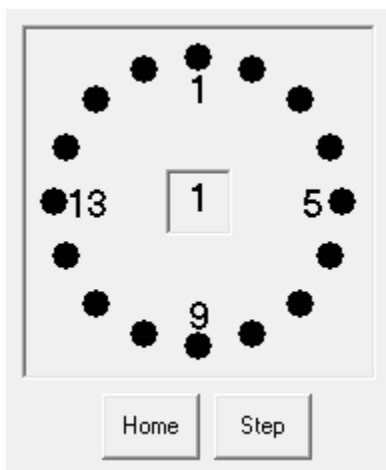
All time values are saved in the method.

7.5 Important remark concerning the first loop

The first loop of a series of storage should not be considered for the study or for quantifying the composition of a mixture. Indeed, it is indeed usual to parameterize its 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.

When all the loops have been injected, the software can, depending on the method, run one or several sequences of washing.

You can also directly launch a flushing sequence when the IST16 is idle by checking the **Rinse** option and by clicking the **Start** button. Always when the interface is at rest, you can switch the multi-position valve via the “Home” and “Step” buttons.



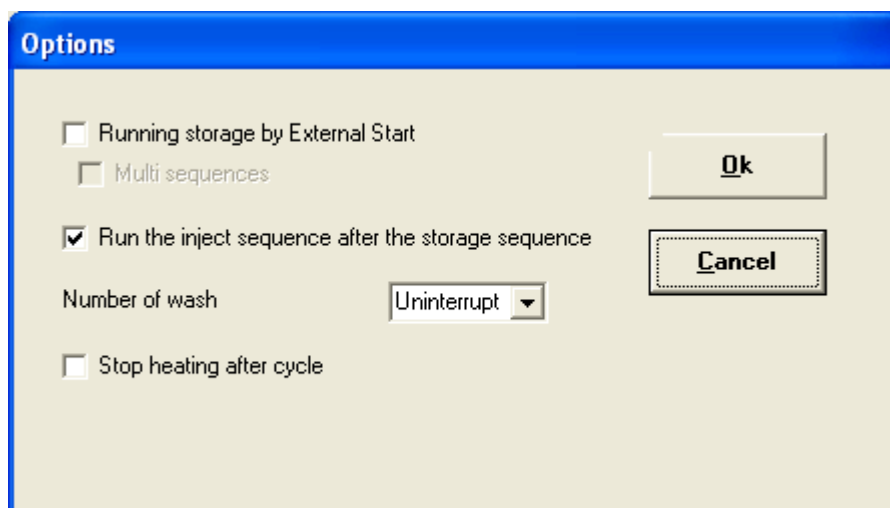
Press the “**Home**” button to position the valve on the first active position.
Press the “**Step**” button to move the valve from an active position.

7.6 Options

By default, the software waits for an operator action to chain each sequence.

You can chain these sequences automatically by the **Parameters\Options** menu.





Check the desired sequence.

Running storage by external start: If checked, after the manual pre-run is done, the IST16 will wait for the external remote given by the external device (i.e. TGA).

Run the inject sequence after the storage sequence: if checked, these 2 steps are automatically chained.

Stop heating after cycle: if checked, the oven and transfer lines will be automatically turned off after the analysis sequence.

When the boxes to check "Running storage by External Start" and "Run the inject sequence after the storage sequence" are checked, the box to check "Multi sequences" is activated.

In this case, the IST16 returns to standby 'external start' at the end of the analysis of the last stored loop. Be careful, it is necessary to change the cycle time of the TGA.

8. Multi-injection and Gas sampling valve 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:

1. Without multi-injection (gas sampling valve): the interface will inject only once into the chromatograph for each analysis. The parameters can be changed via the **Parameters\Injection** menu. The following window appears:



Parameters of injection

Injection time (s) :

GC cycle time (mn) :

Selected loop :

☒ Running by external start

Inject at : ☒ minutes ☐ seconds

Injection num	Inject at
1	0.00
2	21.00
3	41.00
4	61.00
5	

Ok **Cancel**

In the above example, 4 GC analyses from 4 separate samples at the TGA level can be performed.

2. With multi-injection: the interface will inject several times in the chromatograph during the same analysis.

The parameters can be changed via the **Parameters\Injection** menu. The following window appears:

Parameters of injection

Waiting time (mn) :

Injection time (s) :

flush time (s) :

GC Run time (mn) :

Time between injections : 1.00 mn

Injections number :

Selected loop :

☒ Running by external start

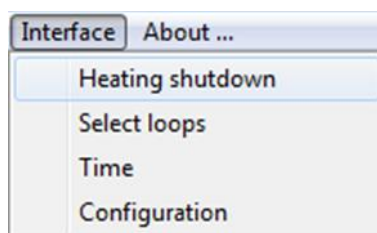
Ok **Cancel**



In the above example, the interface injects every minute during the TGA experiment, with a first injection after 5 minutes of loop sweeping.

9. Shutdown procedure

Before switching off the IST16 electrically, it is mandatory to confirm the "Heating shutdown" menu. It cuts both heaters of the IST16.



Once the IST16 has reached both 100°C set points, it can be electrically switched off. This may take more than an hour.

The IST16 does not heat up automatically when restarting. To return it to service, simply load a method, the IST16 will then launch a dedicated heating procedure.

10. Maintenance operations

10.1 Maintenance schedule

10.1.1 After each experiment

- Clean the TGA furnace with air flow about 200mL/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 1/16" 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 level on MSD signal with carrier gas flowing through IST16, in standby mode. If level is too high, check for leak on your complete configuration.
- Clean the sample path of the IST16 with a solvent (≈ 20mL) 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 position or 4 position valves of IST16.

10.2 Typical cleaning after an experiment

IST16

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

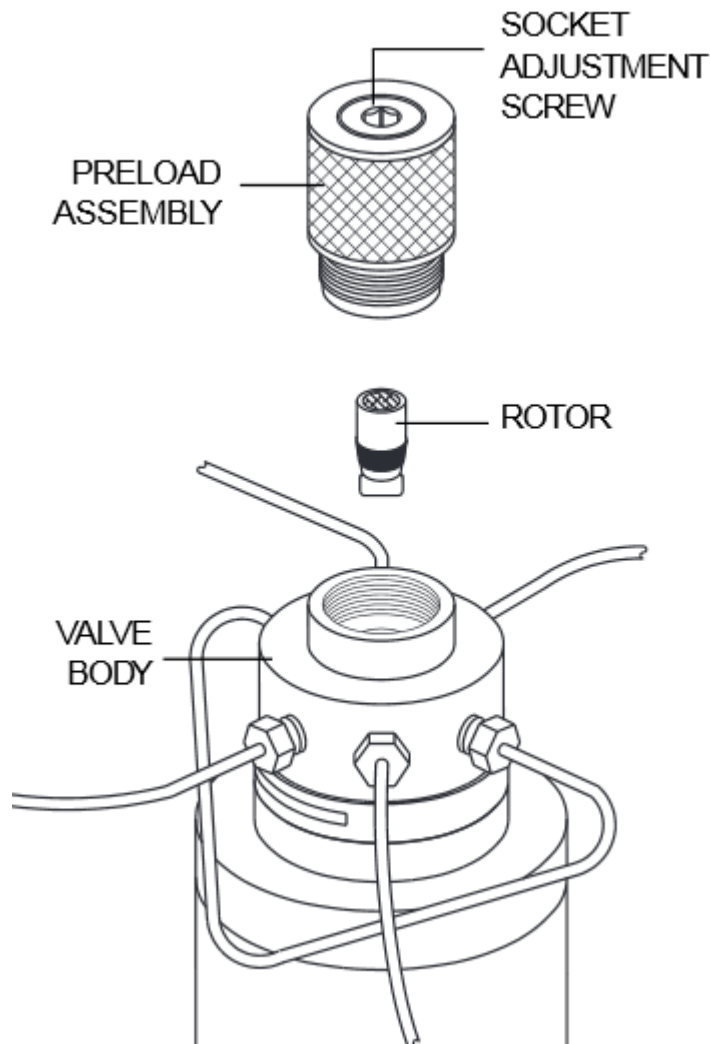
TGA

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

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



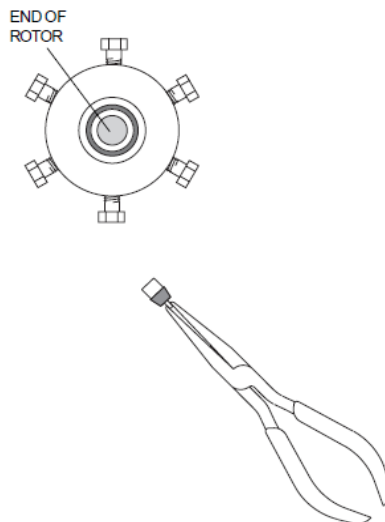
10.3.1 Disassembly the valve

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.



Original rotor

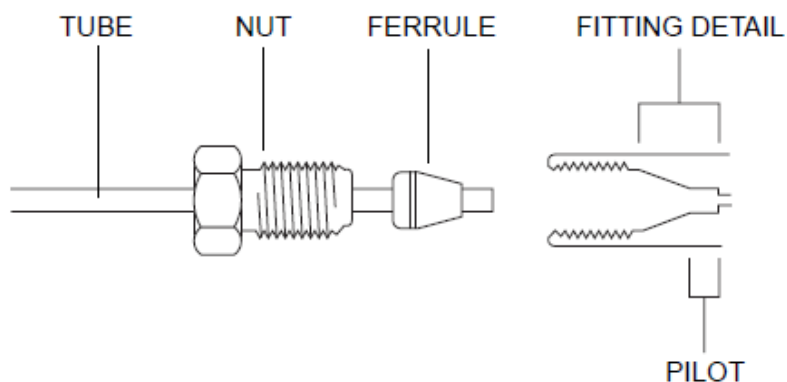
Dirty rotor of the 16 port valve after months of analyses.

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 $\frac{1}{4}$ 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.

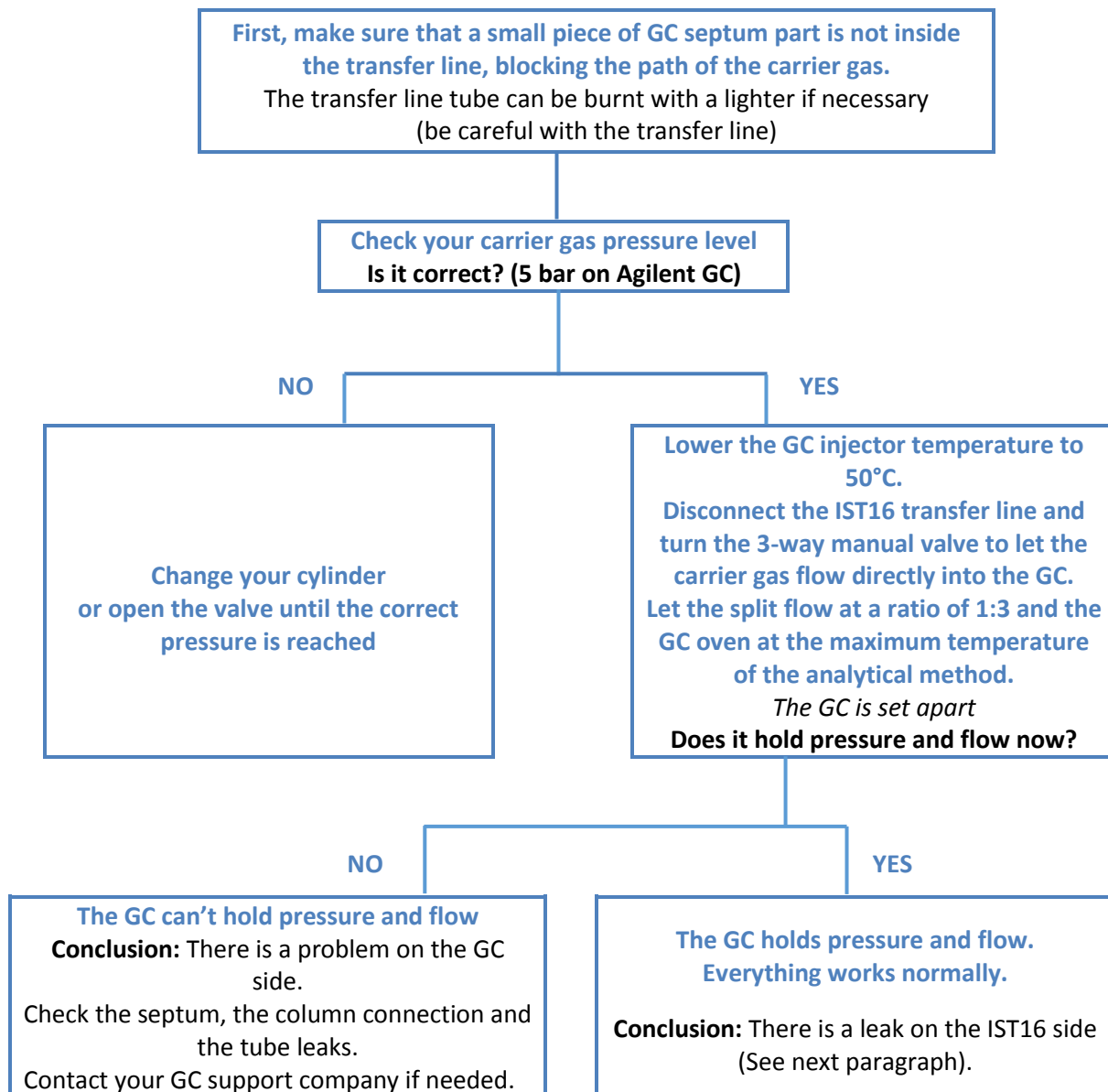


Most common problems	Paragraph
No flow in GC No pressure in GC GC can't hold the pressure	11.1
No peak Small peak Flow restriction TGA oven back pressure	11.2
Memory effect	11.3
High level of air in MS	11.4
Problem with the reading state of the oven, heated line and multi position valve number	11.5



11.1 Flow/pressure problem with GC

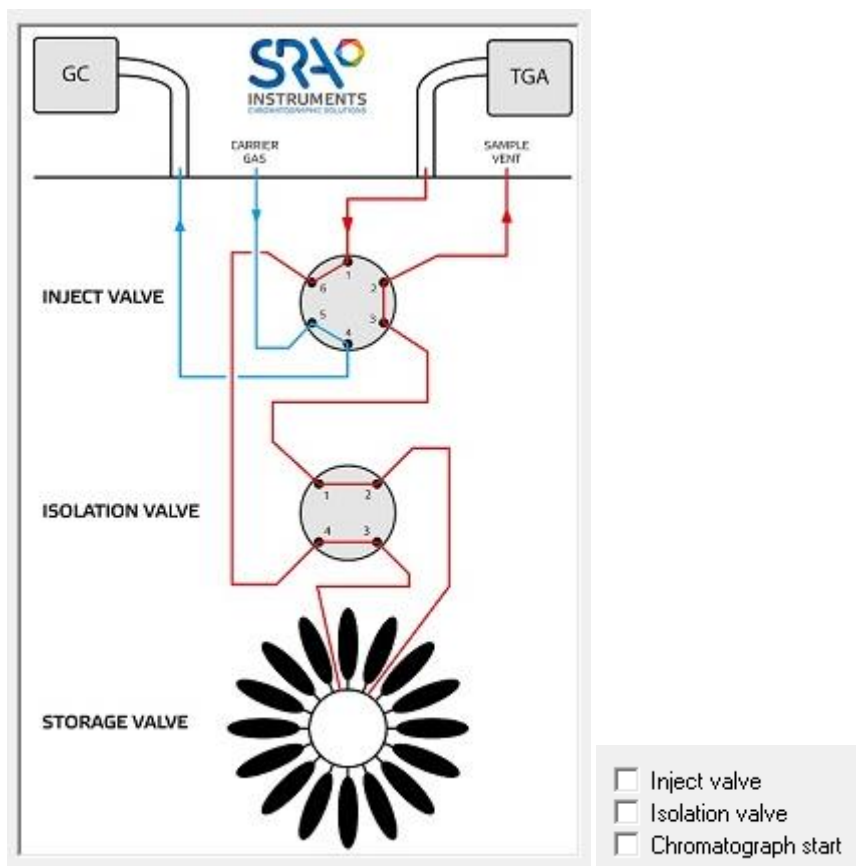
11.1.1 Find the origin of the problem



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 software to help you find the leak.

Step 1:



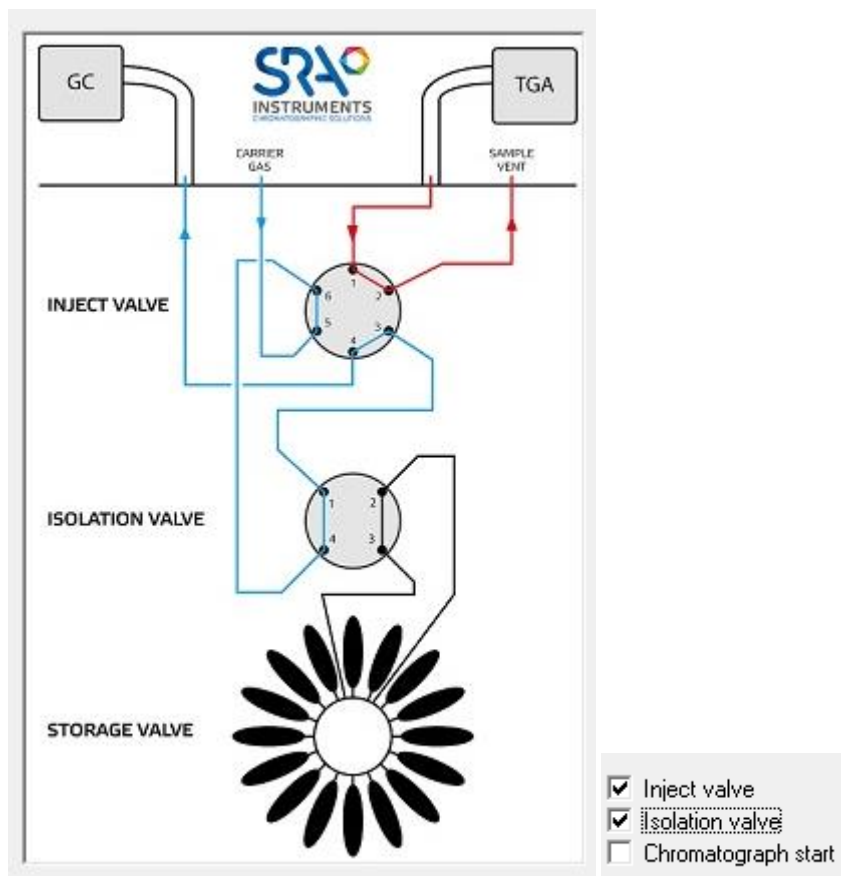
In this configuration of the IST16, the leak may be:

- 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:

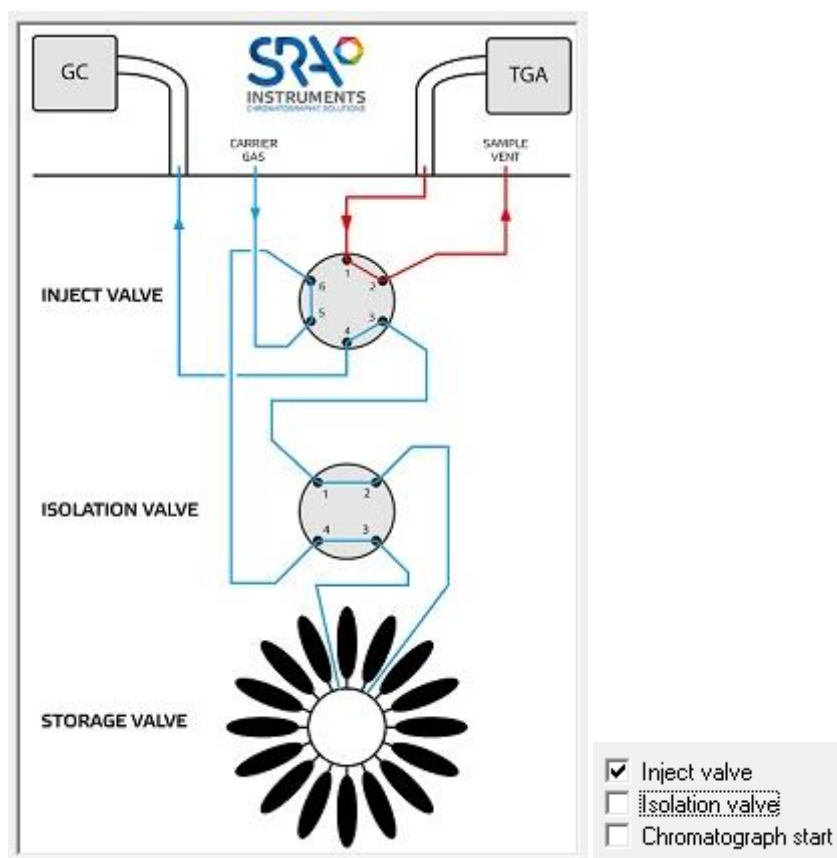


In this configuration of the IST16, the leak may be:

- Between the injection valve connection and the isolation valve connection → **check for leaks and replace tube and ferrules if necessary.**
- On the isolation valve rotor → **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:



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

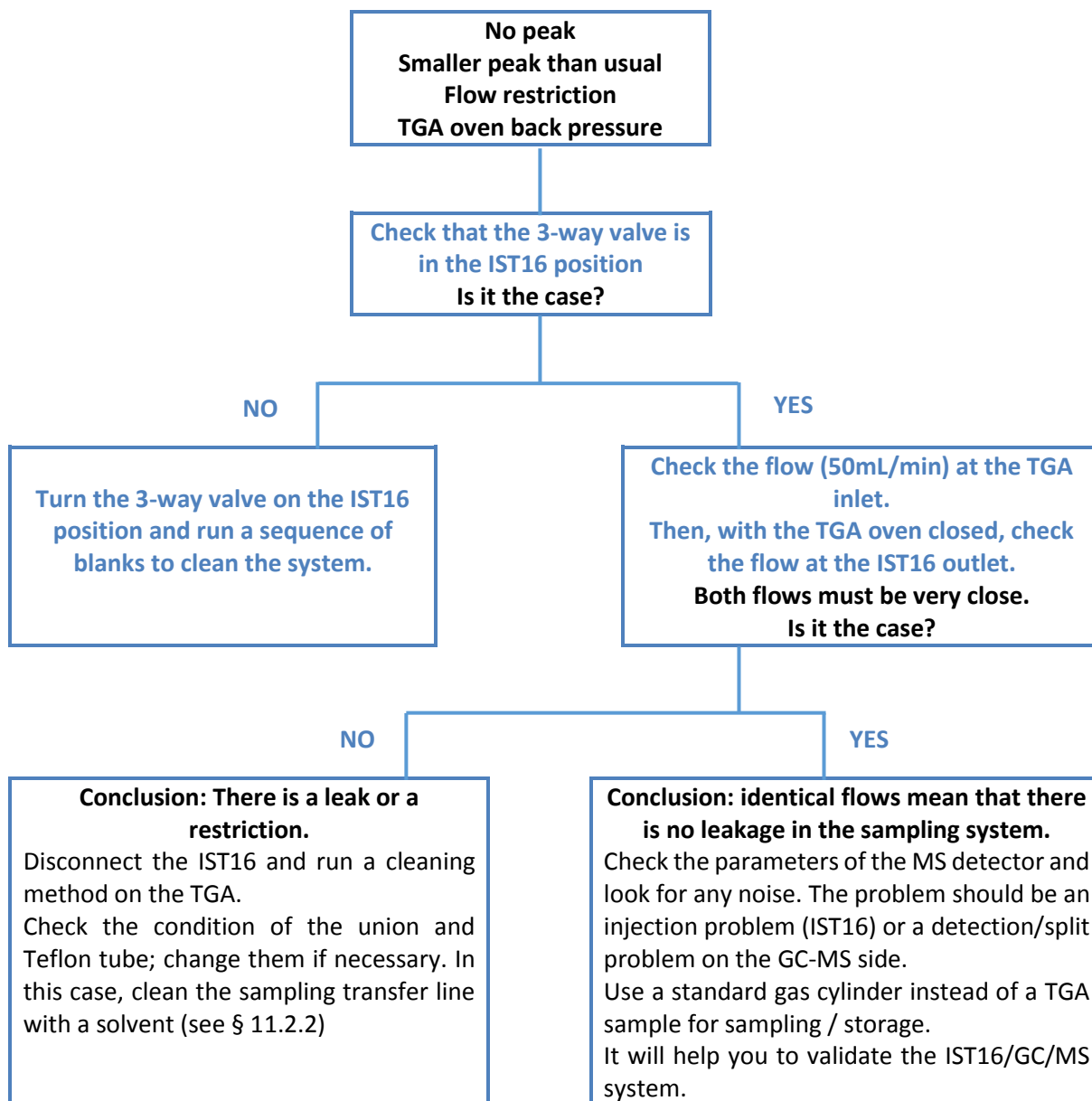
- On the selected loop → **change the loop and check if the leak has disappeared, replace it if necessary.**
- On the rotor of the storage valve → **clean the rotor (paragraph 10.3); replace it if necessary.**
- On the connection between the isolation valve and the storage valve → **check for leaks and replace tube and ferrules if necessary.**
- On the atmospheric pressure relief 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

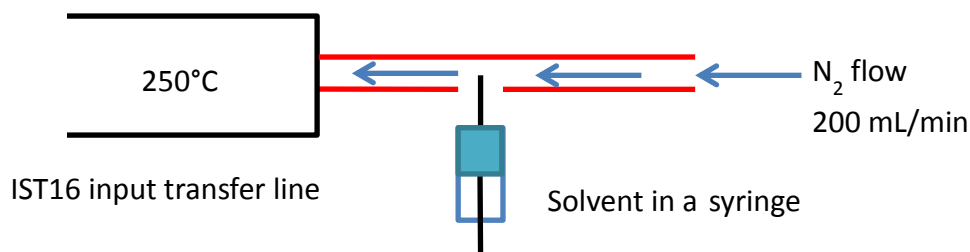
11.2.1 Find the problem



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 200mL/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 cleaning, run a wash sequence from the IST16 software using a nitrogen flow (5 minutes wash per loop) for 2 hours.
4. After 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.



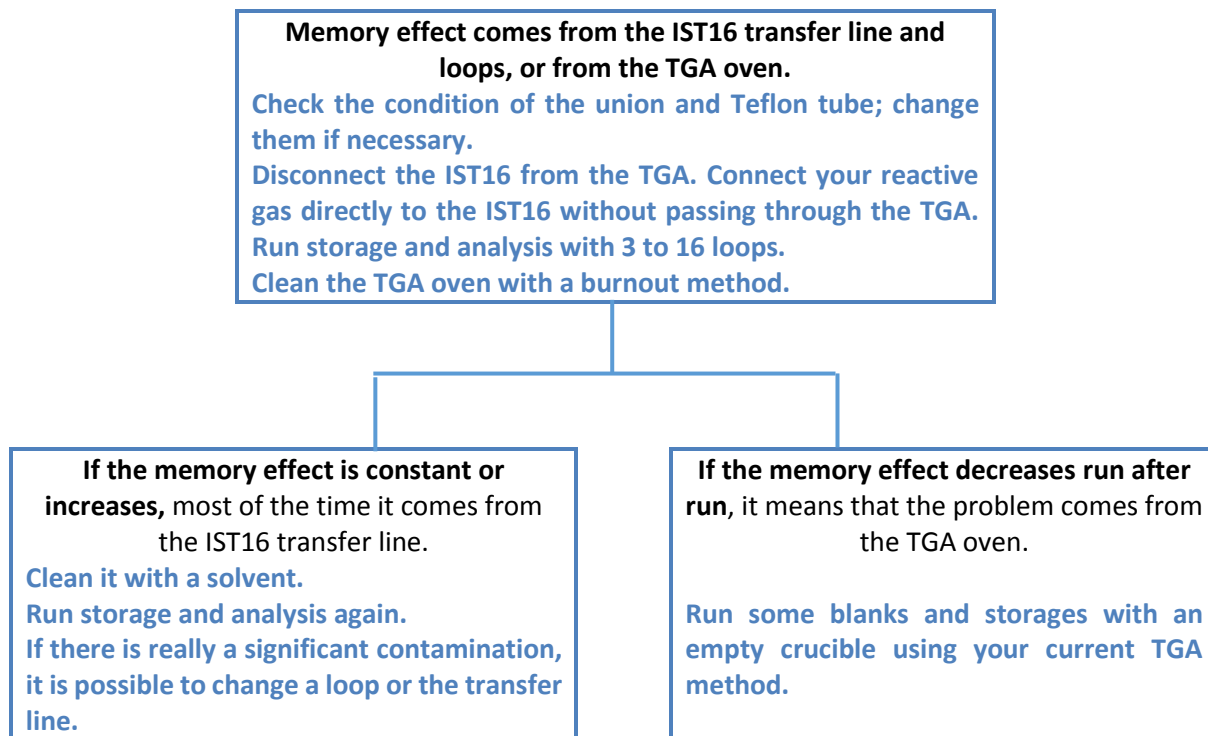
Presence of particles in the PTFE tube after analysis of PE-HD



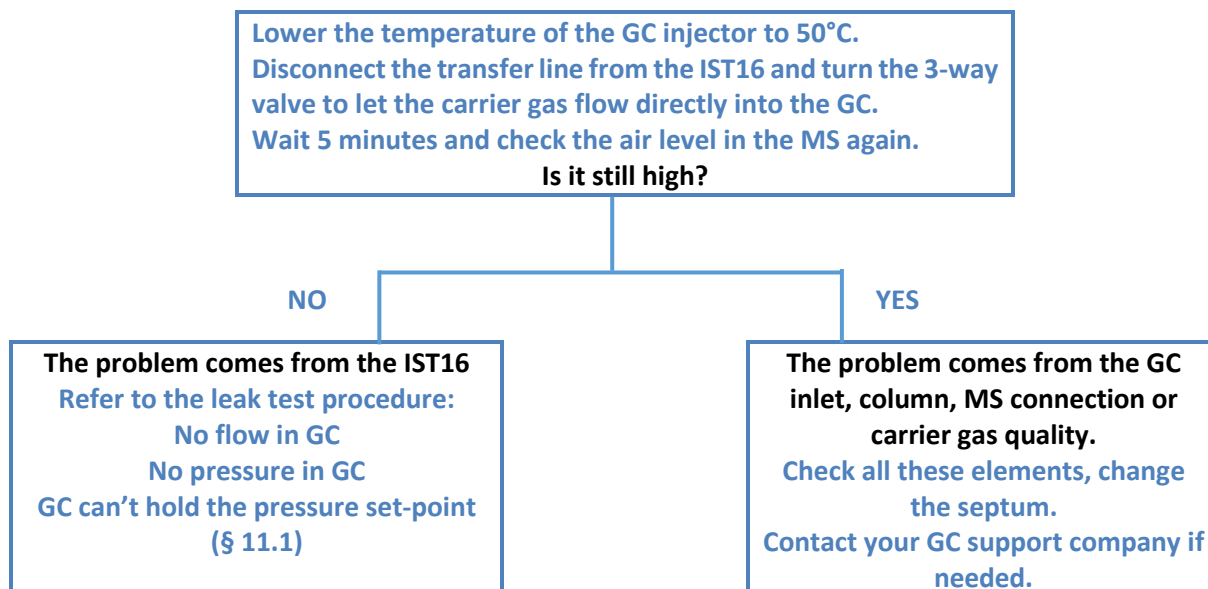
Teflon® tube can also stop halogenated compounds.



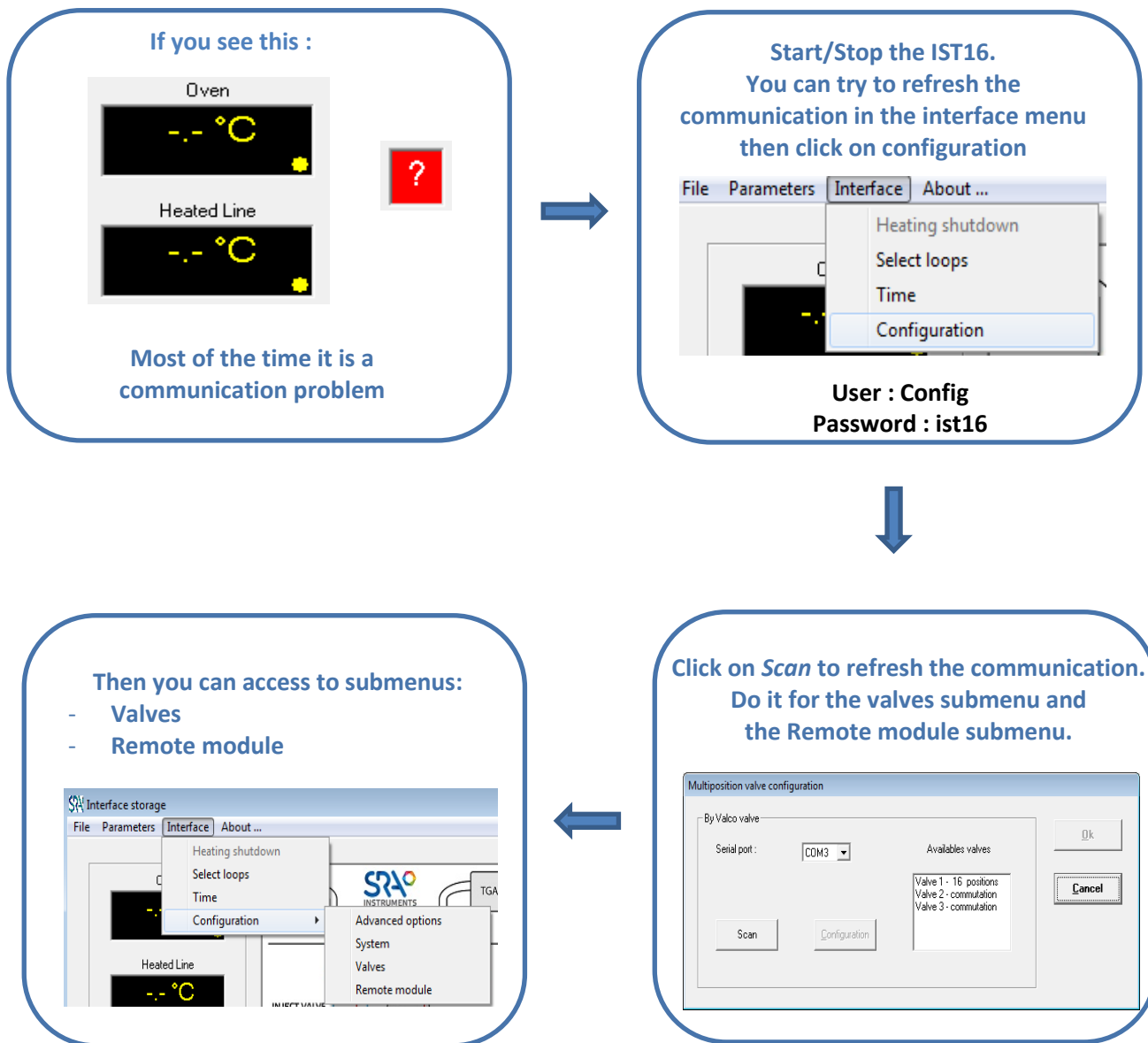
11.3 Problem of memory effect

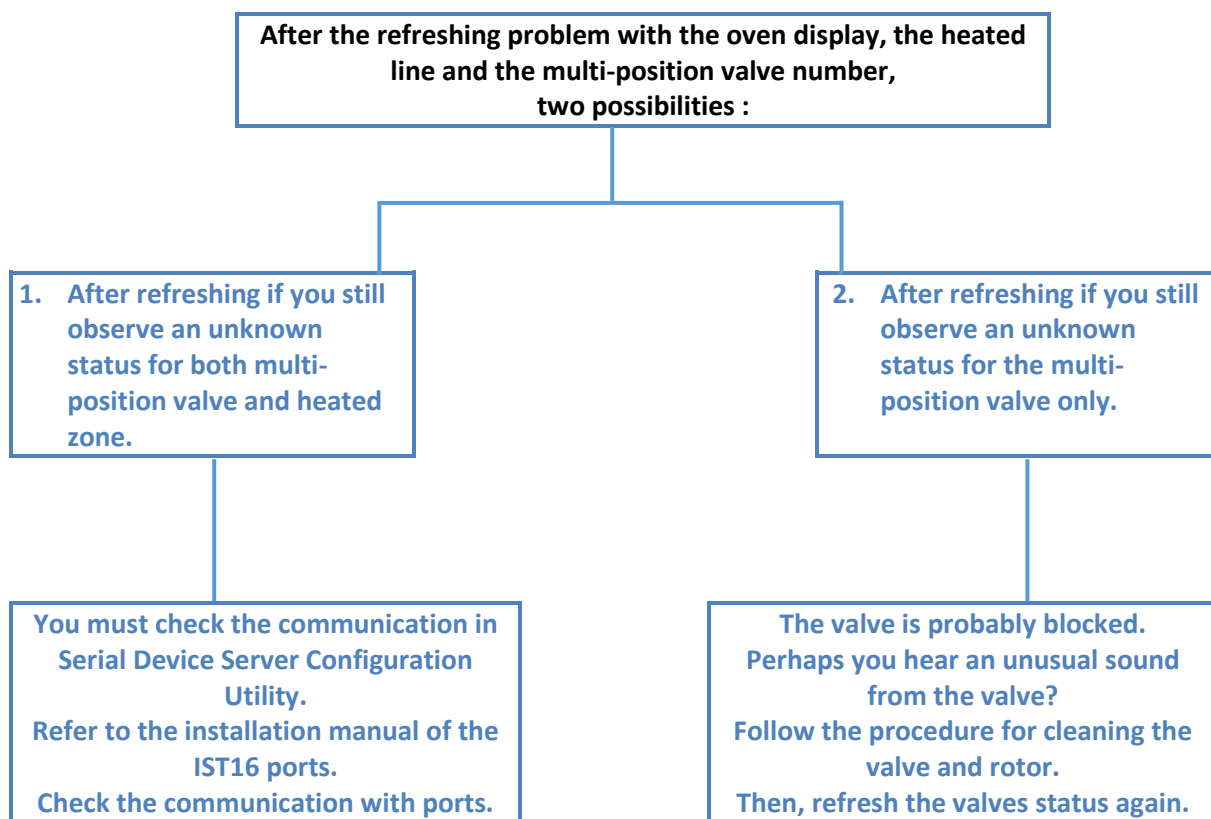


11.4 Problem of high level of air in MS



11.5 Problem with oven display, heated line and multi-position valve number





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 370mm
- 25 Kg

12.3 Instrument

Data System

- SRA IST16 Software.

Number of loop

- 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, and argon, with Swagelok fittings.

12.4.2 Repeatability

Typical RSDs at constant temperature and pressure including SRA MPA system: $\leq 5\%$

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

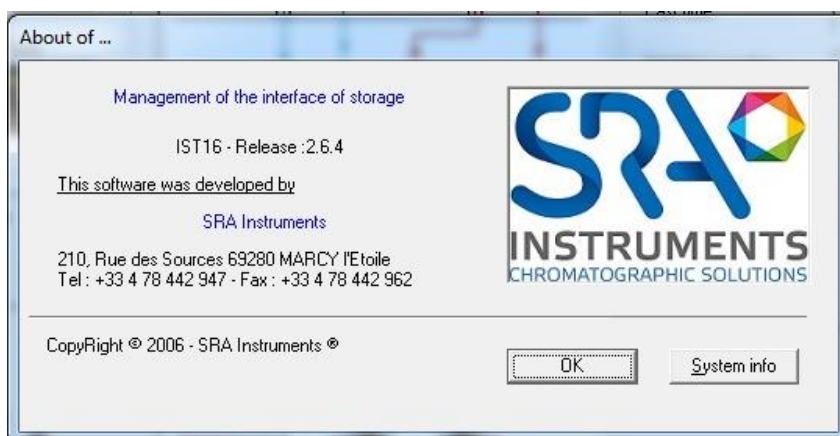
Refer to appendices

12.7 Recycling



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

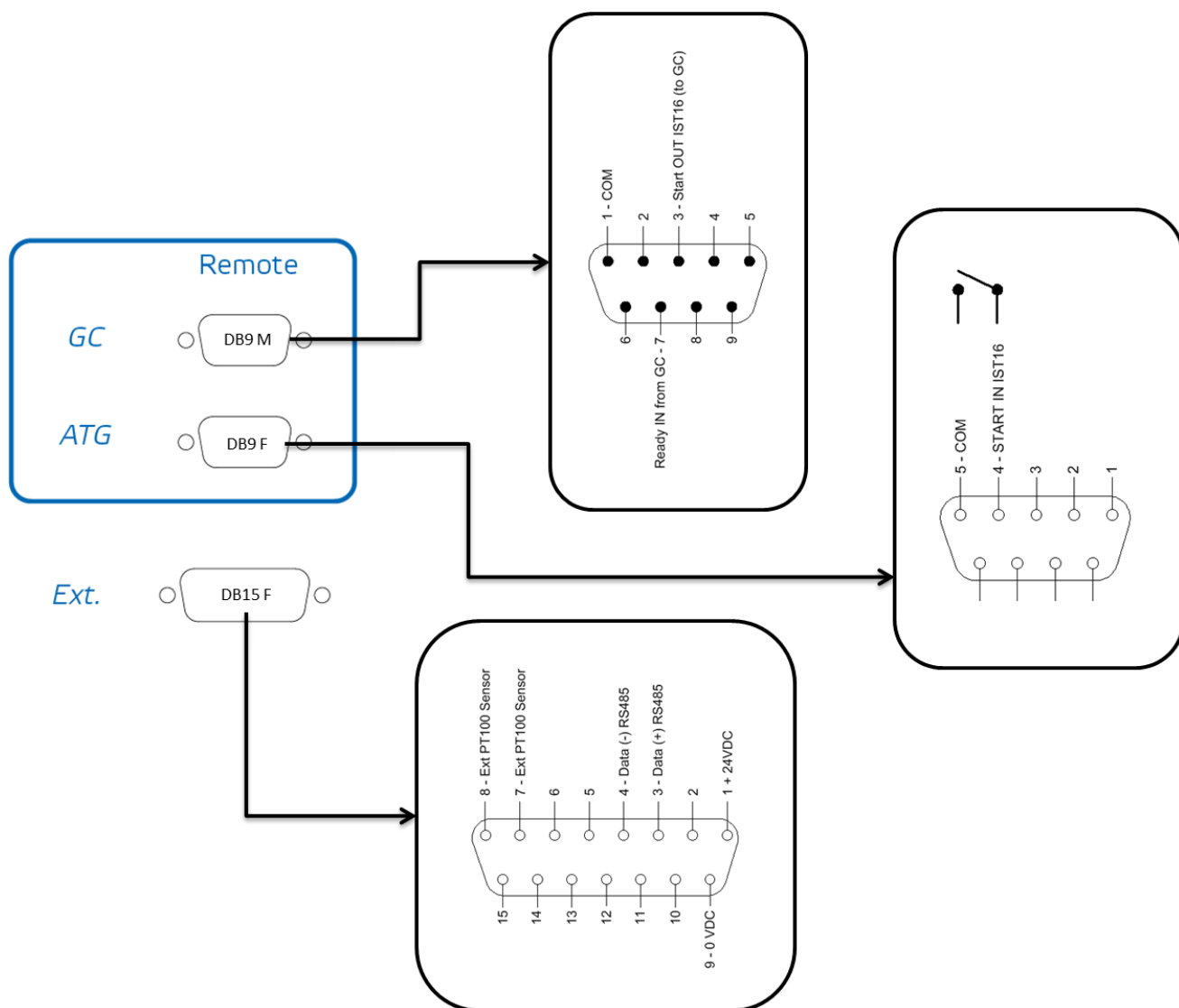
13. About



This menu shows the software version.



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

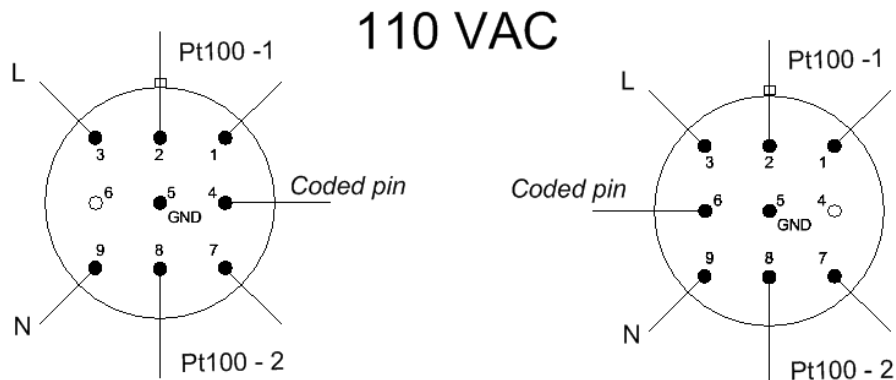


15. Appendix II: Connecting the IST16 transfer lines

The power cable depends on the voltage of your IST16:

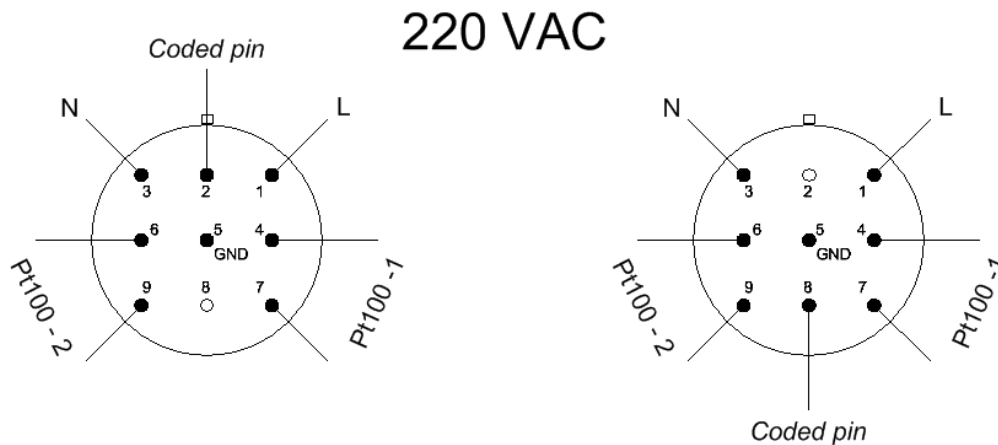
INPUT TRANSFER LINE

OUTPUT TRANSFER LINE



INPUT TRANSFER LINE

OUTPUT TRANSFER LINE



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.
- Pretreatment: 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 temperatures: 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 $^{\circ}\text{C}$ to 1000 $^{\circ}\text{C}$ at 20 $^{\circ}\text{C}/\text{min}$. 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 $^{\circ}\text{C}$ and has its maximum slope around 780 $^{\circ}\text{C}$. 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 ($\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$), the theoretical calculated weight loss is 44%.

The effect observed at about 900 $^{\circ}\text{C}$ 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.

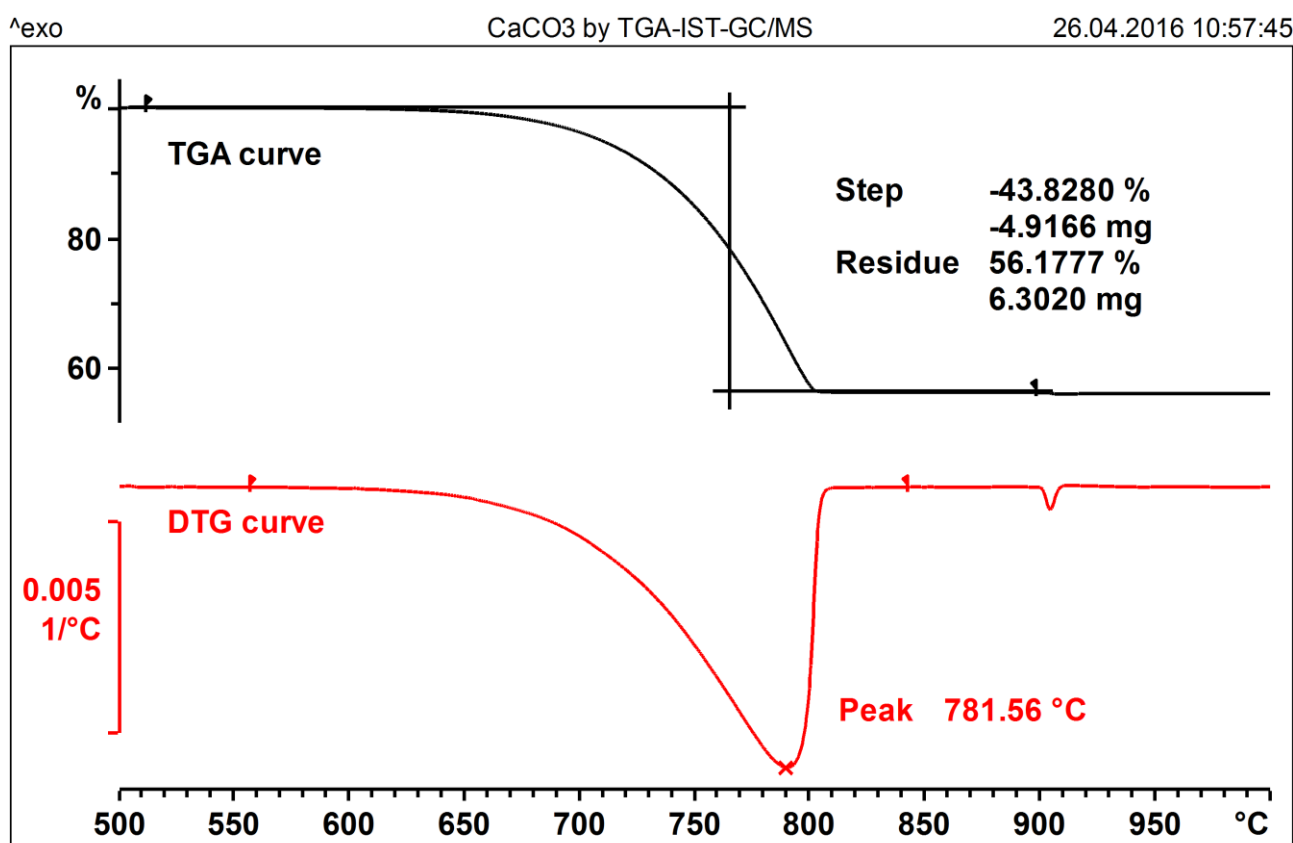


Figure 1: TGA and DTG curves



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 17.3.1 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 CO₂) of loop 06 (750 °C). The peak observed at around 4.6 minutes corresponds to the CO₂ of the calcination of CaCO₃.

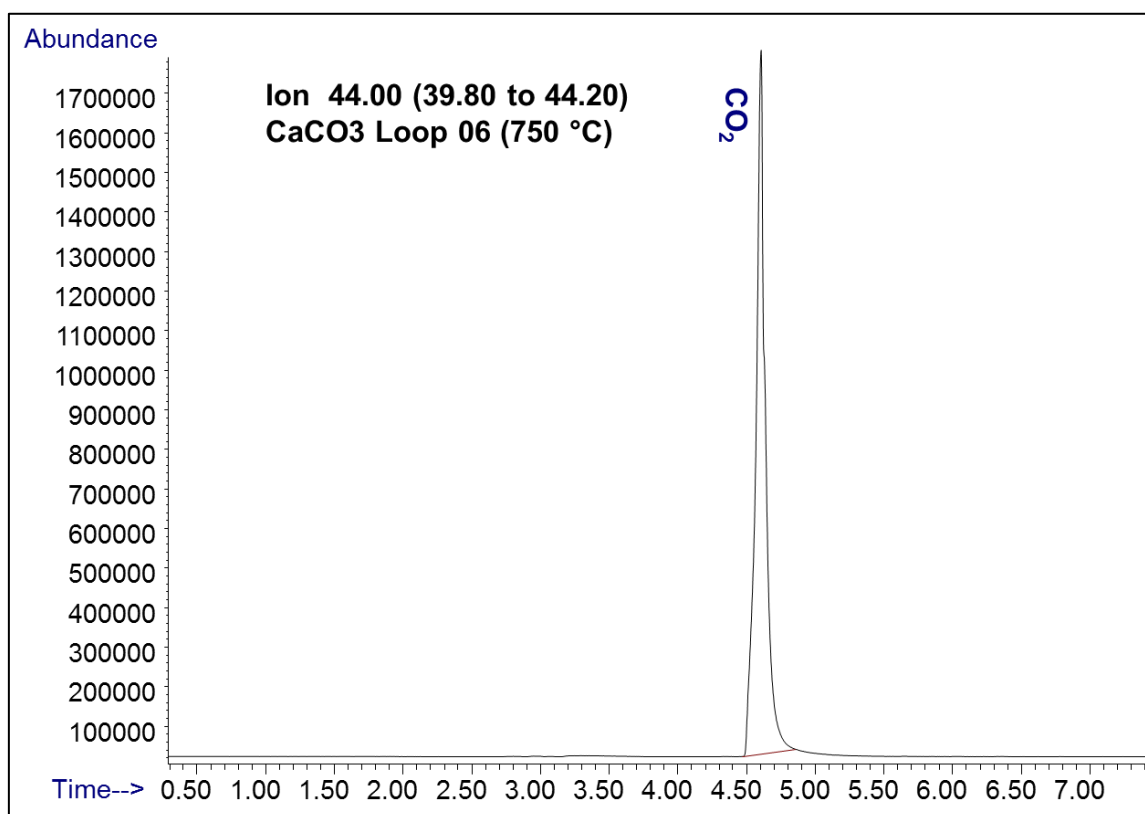


Figure 2: m/z 44 of loop 06 (750 °C) (without background subtracted)

Using all loops, emission profiles of CO₂ along the thermal decomposition is obtained. For this purpose, the area of the fragment peak m/z 44 in the MS spectrum, characteristic for CO₂ 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).

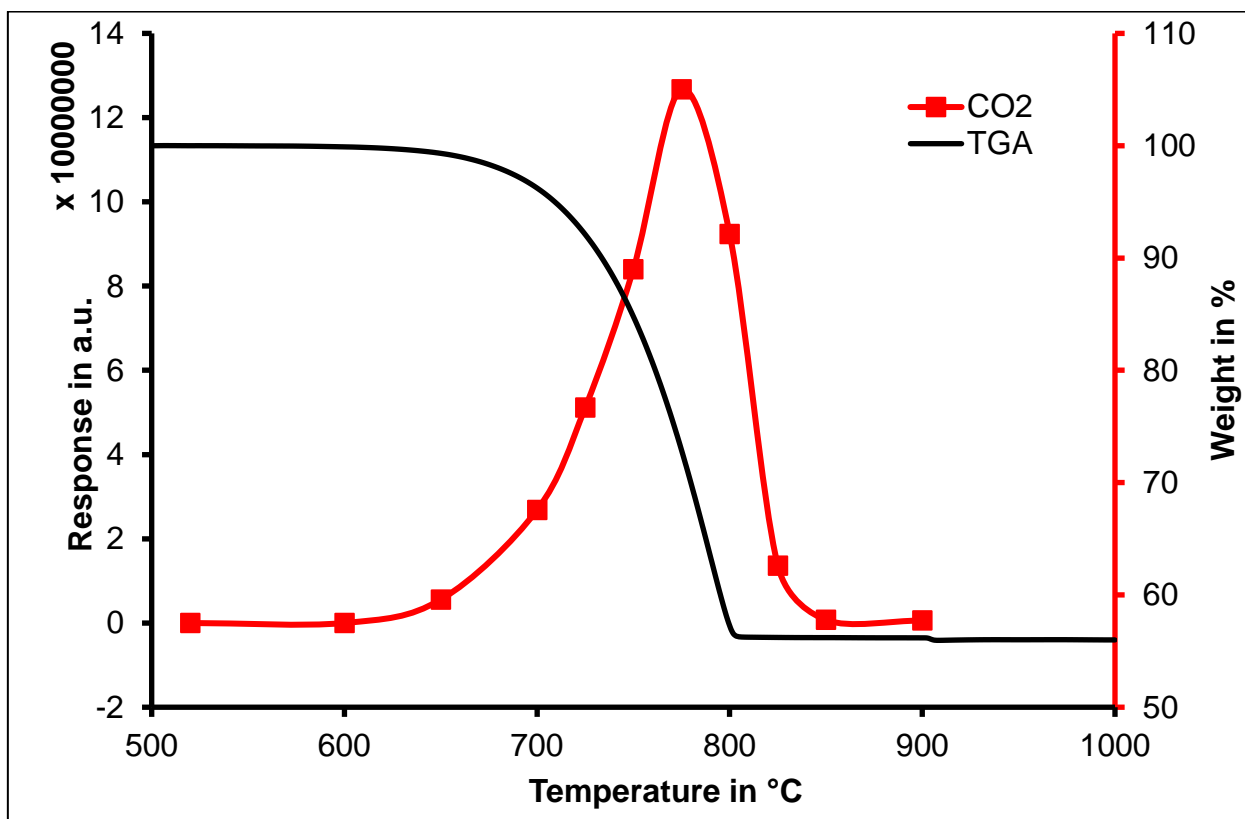


Figure 3: TGA curve and m/z 44 evolution profile

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 ($\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$). 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.

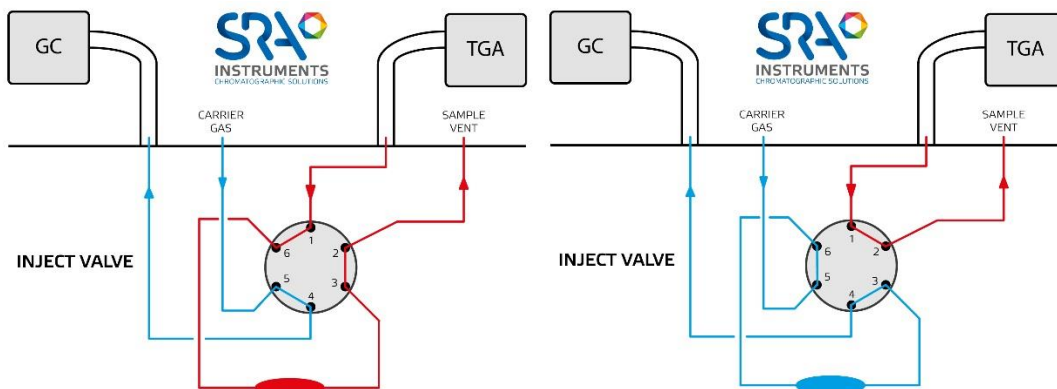
Disclaimer

This report presents one or more examples of analysis. The experiments were conducted with the utmost care using the instruments specified in the description of each application. The results have been evaluated according to the current state of our knowledge. This does not however absolve you from personally testing the suitability of the examples for your own methods, instruments and purposes. Since the transfer and use of an application is beyond our control, we cannot of course accept any responsibility.



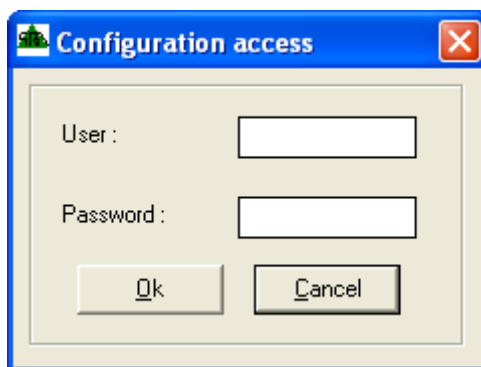
17. Appendix IV: IST1 instrument

The IST1 contains only one 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 is similar to IST16. It is necessary to select the interface type IST1 in the configuration. Only Multi-injection (default) and Gas Sampling Valve modes are available.

All configurations are accessible via the **Interface\Configuration** menu. This menu is protected by a password.

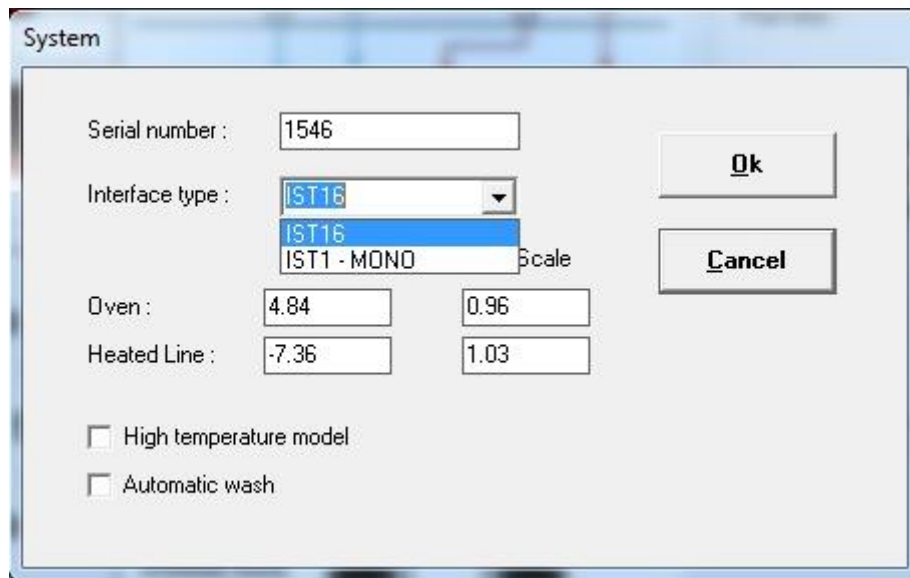
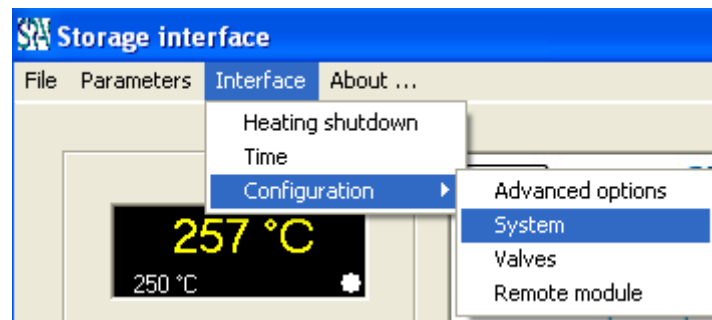


User name: **Config**

and Password: **ist16**



You can choose the type of interface that will be used by the system.



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

Note that it is possible to switch from an IST1 MONO version to an IST16 version by returning the unit to the factory.



18. EU declaration of conformity


We,



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CHROMATOGRAPHIC SOLUTIONS

SRA Instruments
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69280 MARCY L'ETOILE
FRANCE

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

<p>IST16</p> <p>Storage interface for gas</p>	
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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

- EN 61010-1:2010 "Safety requirements for electrical equipment for measurement, control, and laboratory use – Part 1: General requirements"
- EN 61010-2-081:2015 "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

- EN 61326-1:2013 "Electrical equipment for measurement, control and laboratory use – EMC requirements – Part 1: General requirements"
- NF-EN 61000-4-2:2009-06 "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 December 2017

Legal representative,
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