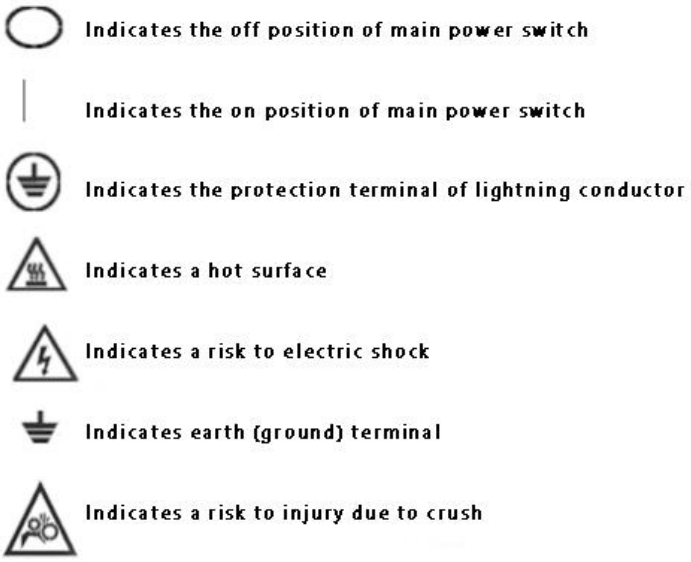
Automatic Headspace Sampler
Operating Manual

INTRODUCTION

About this Manual

This manual explains how to use Headspace Sampler. It includes all information the user needs to install, operate, maintain the product and replace its parts.

Symbols used in this Manual



Safety Information

This product has been designed and tested in accordance with recognized safety standards. It meets Safety Class 1 of International Electrotechnical Commission (IEC). Please carefully read the Manual before use it, especially Caution and Warning, and strictly operate it according to the Manual.

1.Safety matters of carrier gas use

It is prohibited to use flammable or explosive gases, such as hydrogen as the carrier gas. Nitrogen gas, helium gas and other gas can be used as the carrier gas.

2.Electrical equipment safety

Do not operate the instrument if you doubt the electrical safety during use for any reason to prevent violation operation and ask your company’s engineer or other qualified staff to inspect it.

- **Do not operate the instrument if the external or internal protection terminal (ground wire) is not connected.**

●Do not open or move the instrument while it is powered off. Even if it is powered off, it shall not be opened until ensuring that the energy stored in the energy storage element of instrument circuit is discharged.

- **It is forbidden for non-professionals to open the instrument. Only after-sales engineers and other qualified personnel can open the instrument for maintenance.**

- **Do not open the instrument without installing any enclosure. If a sample vial is broken in the instrument, please power off the instrument immediately and do not open the instrument until the sample is completely volatilized.**

3.Safety at high temperature

Pay attention to the high temperature symbol. Do not touch it with hands to avoid burns.

Contents

Chapter I Technical indicator and installation

1.1 Technical indicator

Sample vial

Number:	27
Material:	neutral glass
Volume:	20ml
Septum:	Butylene rubber coated with Teflon
Vial cap	Aluminum/Iron

Equilibration area

Preheating positions:	6
Temperature range:	15°C to 200°C above room temperature With the increment of 1°C
Accuracy:	0.5% of the full range
Stability:	±0.1°C

Sampling system

Temperature range:	15°C to 200°C above room temperature With the increment of 1°C
Accuracy:	0.5% of the full range
Stability:	±0.1°C
Sampling valve:	MV65/106HT, 6-way valve
Sampling loop:	1ml volume, nickel or 3 ml volume, nickel (optional)

transfer line

Temperature range:	15°C to 200°C above room temperature With the increment of 1°C
Accuracy:	2.0% of the full range
Stability:	±0.5°C

needle

Temperature range:	15°C to 200°C above room temperature With the increment of 1°C
Accuracy:	2.0% of the full range
Stability:	±0.5°C

Electric requirement

Power supply:	220V(±10%),50Hz,600VA
	110V(±10%),60Hz,600VA (optional)
Fuses:	5AT, 250V(220V)
	0AT,250V(115V) (optional)

Dimensions and weight

Dimensions: 40W*51H*60D cm

Weight: 30Kg

1.2 INSTRUCTION INSTALLATION

Environmental requirement

A comfortable environment (relatively constant temperature and humidity environment) is recommended for the optimal performance and service life of HSS.

It is necessary to avoid exposure to corrosive materials (gas, liquids, and solids) which can corrode the materials used in HSS as far as possible.

Site

A bench, 75cm long and 50cm wide, which can withstand a weight of 30KG, is enough for HSS. It is required that there should be no cover or other obstacles on the top of the bench, otherwise the top of the instrument cannot be opened and its cooling may be affected.

The transfer line is 80cm long (100cm, optional), so the distance between the injector and GC must be suitable.

Gas supply

The instrument requires high purity gas because it is used as the carrier gas for the column.

The carrier gas for headspace analysis is primarily determined by the type of injection port and the detector.

Connect the carrier gas to the inlet on the rear panel of the instrument. The maximum allowable input pressure is 0.4 MPa.

Caution:

Do not use hydrogen gas as the carrier gas of the HSS due to the risk to explosion.

Power connection

HSS Headspace Sampler shall be operated under any of the following voltage supply conditions:

220V(±10%),50Hz,600VA

110V(±10%),60Hz,600VA

The actual voltage is shown in the label on the back of the instrument.

Caution:

To protect users, the HSS is grounded with a three-pin power connector according to the requirements of the International Electrotechnical Commission.

Insert the three-pin power connector in appropriate slot.

Caution:

HSS must be operated with proper grounding to maintain ground protection function. Make sure that a correct grounding receptacle is used.

1.3 Relevant parameters

1.3.1 Temperautre parameter

Equilibrium temperature

The equilibrium temperature greatly influences the concentration of the analyte in the headspace gas. In general, the amount of analyte in gas chromatogram and the sensitivity of the method increase with the increase of the equilibrium temperature. To obtain the best results, the oven temperature shall be as high as possible providing that safe operation is ensured and the sensitivity of the required analysis is met.

Needle temperature

It is necessary to set the needle temperature slightly above the equilibrium temperature but below the loop temperature

Temperature of quantitative vial

It shall be set slightly above the equilibrium temperature to achieve the maximum accuracy

Temperature of transfer line

It shall be set higher than or equal to the temperature of quantitative vial

1.3.2 Gas regulation

Flow rate of carrier gas

The flow rate of carrier gas shall be set high enough to blow the headspace sample out of the sampling loop. For example, when a packed column is used, the normal peak width time is about 15 s. The carrier gas takes about 10 s to blow the sample out of the sampling loop. For the 1 ml sampling loop, the flow rate of carrier gas shall be 6 ml/min, which is not the minimum flow rate which causes peak expansion. The minimum flow rate for the 3 ml loop was 18 ml/min under the same conditions. For the capillary column, except that the appearance time is 1s, the other conditions are the same as those of the packed column. Therefore, the flow rate shall be at least 60 ml/min avoid peak expansion. Too large flow rate will affect the column capacity and split ratio: Obviously, the higher the split rate is, the higher the split ratio is and the lower the sensitivity is. This is why it is required that the flow rate of the carrier gas shall be enough to blow the sampling loop.

Pressure

Pressure is used to transfer the gas phase of the sample into the sampling loop. The ideal situation is that the pressure is set enough just to fill the loop with the gas phase. Too much pressure will dilute the headspace gas. The pressure of auxiliary gas can be optimized as follows:

1 Equilibrate the sample at the desired oven temperature.

2 Measure the pressure in the sample vial and insert the inlet of the connected pin gauge (connecting the pressure gauge to the needle of syringe needle) into the septum.

3 Read the pressure of headspace sample. You can see the effect of heating on the vial from the pressure gauge.

4 Set the pressure to slightly higher than the reading of pressure gauge (eg, greater than 0.2 bar). This is the lowest set pressure of auxiliary gas that can be used.

5 Run a series of samples, the pressure of auxiliary gas for a sample is higher than that for the previous sample.

6 Obtain the peak area of sample versus the pressure of auxiliary gas curve, using the highest pressure of auxiliary gas against the peak area in step 4.

Note: This optimization procedure is only valid for analyzing a specific sample at a specific oven temperature.

1.3.3 Time parameters

Analysis cycle

The time during which all components of the sample have been completely separated and flowed out of the column.

Equilibrium time

The components in the vial should be well equilibrated at the desired equilibrium temperature. At a certain equilibrium temperature, the sample may take several hours to be equilibrated. The equilibrium time is a function of the viscosity of the sample. Increase the equilibration time, run a series of samples, and plot the peak area versus equilibration time curve to determine the optimal equilibration time. The time when the peak area is stable represents the best equilibrium time.

Pressurization time

Fill the vial with carrier gas. This time shall not be set too long to avoid excessive dilution of the sample.

Filling time

The time for the sample gas to be filled in the loop should not be set too long to avoid excessive dilution of the sample.

Equilibrium time of filling

Equilibrium time of sample gas

Injection time

The time from the injection status to the cleaning status after the sample goes through the six-way valve.

1.3.4 Seal sample vial

Seal septum of sample vial

It is recommended to use a septum with PTFE face to confirm that a smooth chromatogram is obtained when the vial is sealed for blank experiments.

Two types of septum with PTFE face can be used:

-D 20 BUT/PTFE

-D 20 SIL/PTFE

D20 BUT/PTFE septum is made of butylene rubber. It has a temperature limit and the maximum operating temperature is 120°C. D20 SIL/PTFE is recommended for a higher equilibrium temperature of oven.

Seal sample vial

Poor sealed vial caps can cause leaks, which is often an important cause of analytical errors. After the aluminum cap is snapped onto the vial, try to turn the cap to check whether it is tightened. If not, tighten it again until it cannot be rotated at all or a lot of effort needs to do that.

1.3.5 Factors which affect sensitivity, accuracy and precision

The size of sampling loop

A 1ml sampling loop is installed on the injector at the factory. It is enough for most cases and matches with all models of column. However, a 3ml sampling loop can be chosen to enhance the sensitivity, especially when a large volume loop will not cause peak expansion after a packed column is used analysis.

Sample volume

For headspace analysis, the sample volume shall be 25-75% of the capacity of sample vial.

Sample components

How to prepare a calibration standard which reflects the sample components is a major problem in headspace analysis.

With the internal standard or standard addition method, multiple headspace extractions are used to examine the inaccuracies caused by the sample components.

Note: The chemical and physical properties of the internal standard must be similar to those of the sample as possible.

Sample influence

There are some measurable influences related to headspace analysis. In most cases, this influence does not exceed 0.5%. It is advisable to run a batch of samples from low to high concentrations or to run blank samples between the samples with a high and low concentration.

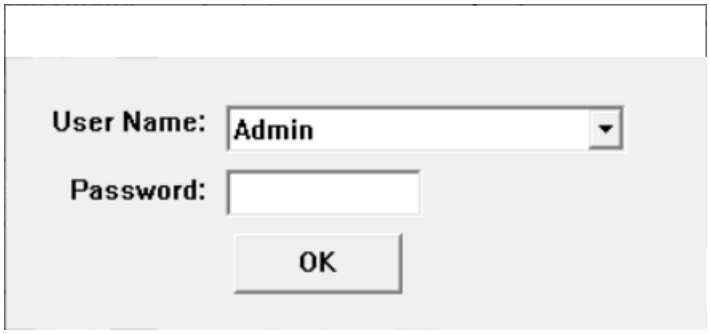
Additives

An important means to improve headspace analysis is to add a substance that can reduce the solubility of the analyte in the sample, so more components in sample can be transferred into the headspace gas. For most solutions, an inorganic salt can be added as an additive to obtain “salt precipitation” effect. This technique is commonly used in various polar analyte solutions and can greatly enhance the sensitivity.

CHAPTER II INTERFACE INTRODUCTION

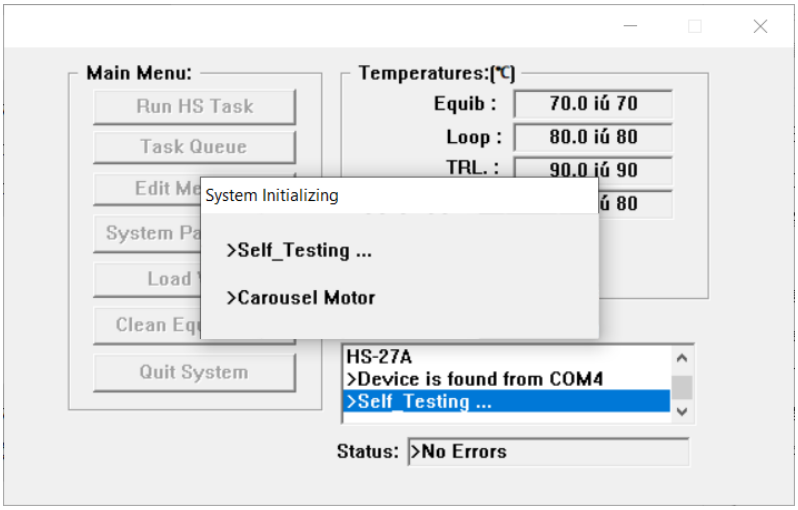
2.1 STARTUP INTERFACE

Turn on the power, enter the user login interface (the default user password is 888888, rights of administrator).

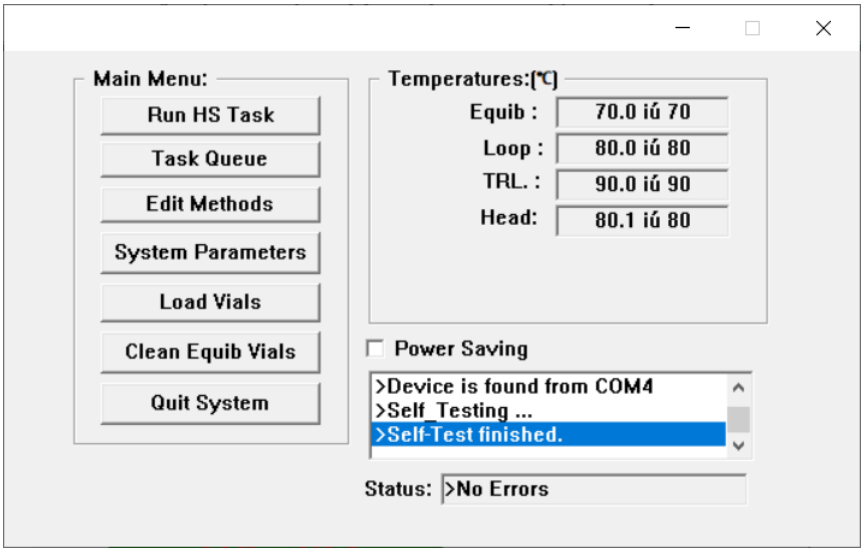


Note: This interface is not available in case of no password login.

Enter the user name and password, click “OK” to enter “Power-on self-test” interface.



The device automatically runs system test and then enters main menu interface:



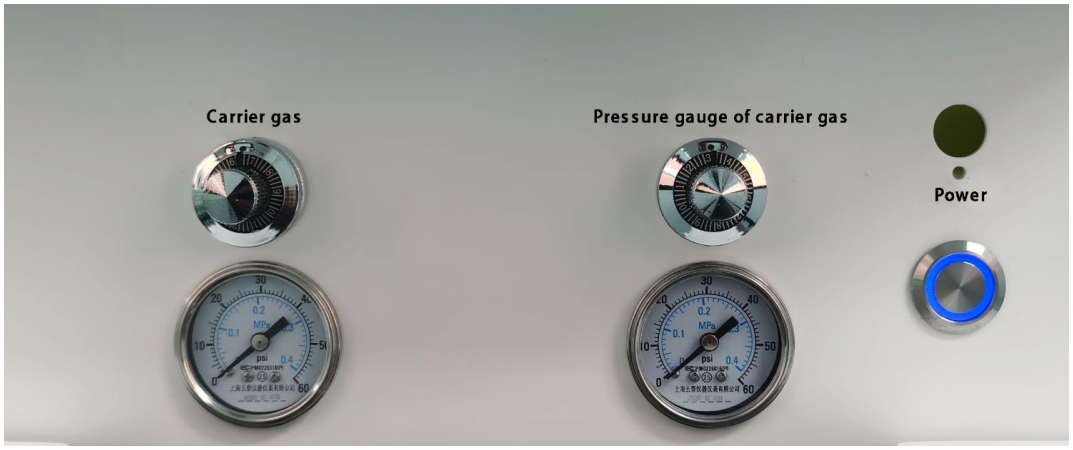
2.2 Interface introduction

The main menu is at the left side of the main interface, and the actual temperature and the set temperature of each heating element are displayed in the upper right corner. System operation, operation messages and error messages are displayed in the bottom right corner.

Energy saving mode: When it is checked, all heating elements will be turned off after the headspace task is completed next time.

Exit: The current user exits the system.

2.3 Introduction to the front panel



Carrier gas valve: used to regulate the flow of carrier gas

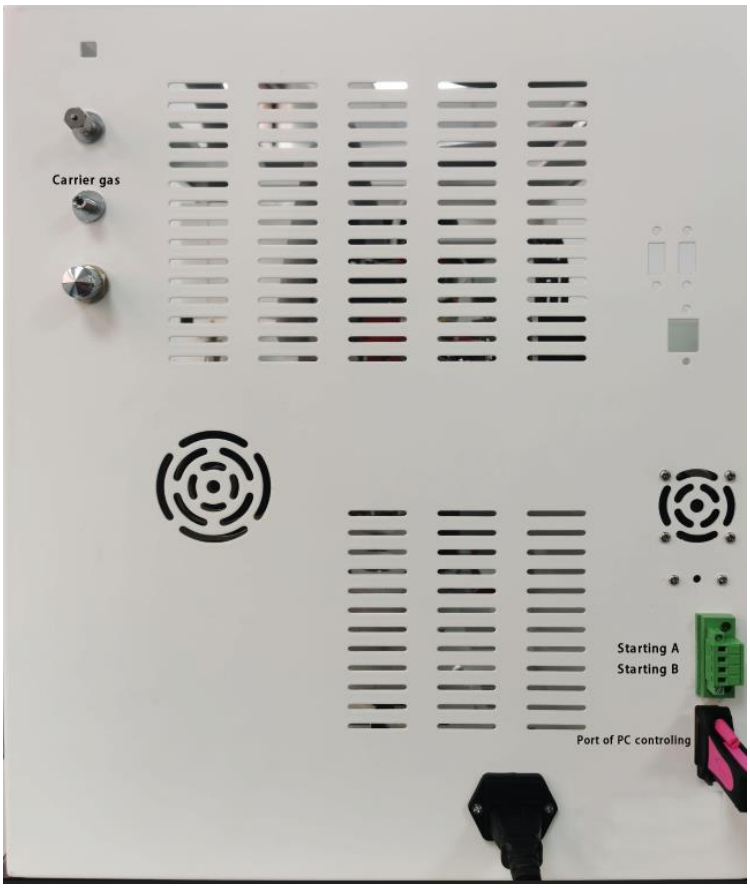
Pressure gauge of carrier gas: used to display the pressure of carrier gas

Pressure gauge: used to regulate the pressurized flow in sample vial

Pressure gauge of pressurized gas: used to display the pressure of pressurized gas

2.4 Layout of the back panel

There are inlet of carrier gas, power port, upgrade port, starting signal port at the back of the instrument.



Inlet of carrier gas: the inlet of carrier gas

Pressure regulator: main pressure regulator of carrier gas

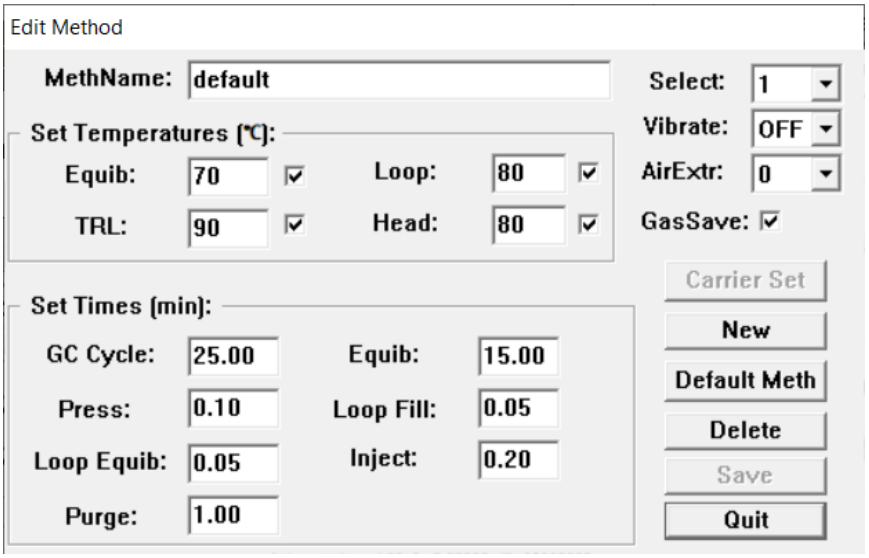
Power port: Power port of the instrument

Port: used to connect a computer (for reverse control software)

CHAPTER III INSTRUMENT OPERATION

3.1 EDIT HEADSPACE METHOD

Click “Method edit” at the main interface menu” to enter method edit interface.



Click “Exit” to exit method edit interface and return to the main interface.

3.1.1 Select and modify a method

1.Click the drop-down menu of method no. to select different method. The parameters of the current method are displayed at the left side.

2.Under “Temperature setting” bar, the box behind each temperature represents the state of the corresponding temperature, that is, it is enabled when it is checked, and otherwise, it is not enabled.

3.The vibration option represents the vibration parameter of the heating position and the vibration speed increases gradually from 1 to 10.

4.Click “Default method” button to recover the current parameters to their default values.

4.To modify a method, select the method, modify the required parameters, and click “Save”.

3.1.2 New method

Click “New” button, the program automatically creates the default method, and then you can modify it to the desired method.

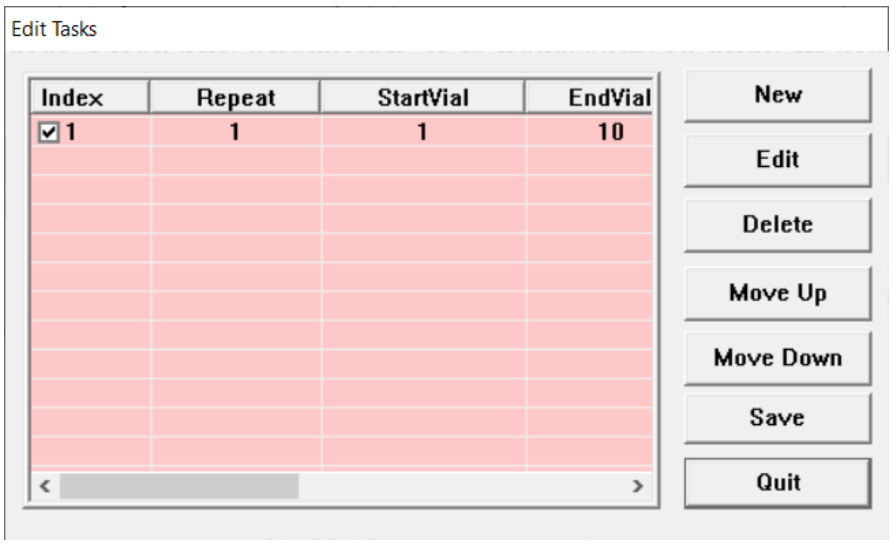
3.1.3 Delete a method

Click “Delete” button to delete the current method.

Note: After any operation, it is necessary to click “Save” before exiting. Otherwise, the operation is invalid.

3.2 TASK LIST

Click “Task Queue” in the main menu to enter the task list interface.



At this interface, you can edit all task parameters. Click “Exit” to exit the method edit interface and return to the main interface.

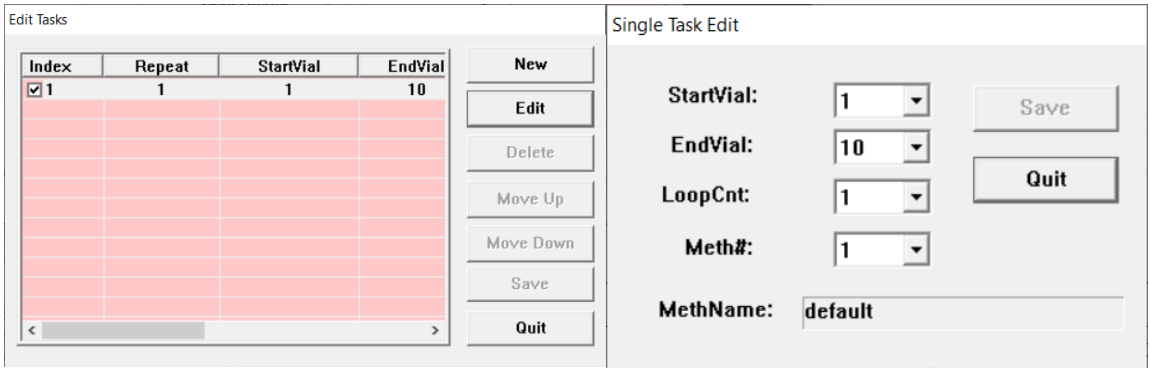
3.2.1 Interface introduction

All tasks are displayed in the task list at the left side of the interface. When the box is checked, the task will be performed. Otherwise, it will not be performed.

2.The operation menu bar is at the right side.

3.2.2 Edit a task

Select a task to be edited, click “edit” button and enter the task edit interface.



1.Starting number of vials indicates which vial at the tray the current task starts from.

2.Ending number of vials indicates which vial at the tray the current task ends at.

3.Loop number refers to the repeat number of current task.

4.Method no. refers to the serial number of method used for current task.

5.After editing, click “Save” to save the task. Click “Exit” to return to the task list interaface.

3.2.3 Delete a task

Slect a task to be deleted, click “delete” to delete it.

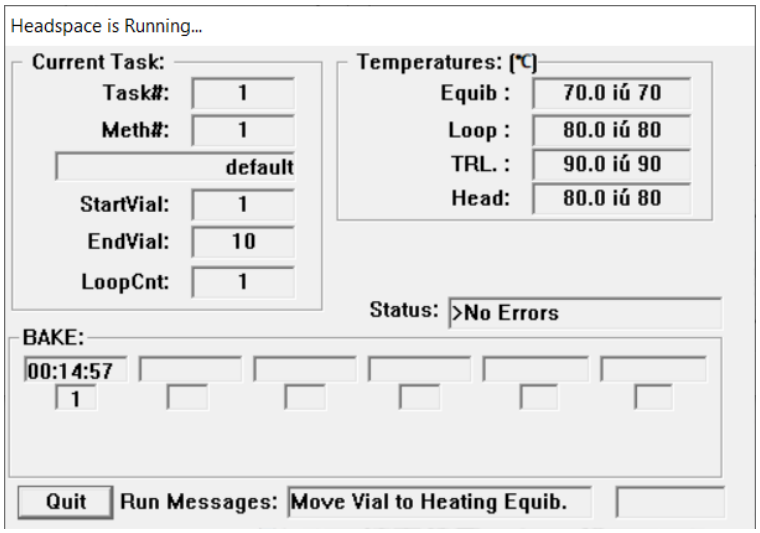
3.2.4 Sort tasks

Select a task, click “shift up” or “shift down” to change its order.

Note: After any operation, it is necessary to click “Save” before exiting. Otherwise, the operation is invalid.

3.3 START A HEADSPACE TASK

Click “Start a headspace task” to enter the task starting interface. The instrument will perform the tasks selected in the task list one by one.



3.3.1 Interface introduction

1.The information on current task is displayed in the upper left corner.

2.The information on temperature and error message is displayed in the upper right corner.

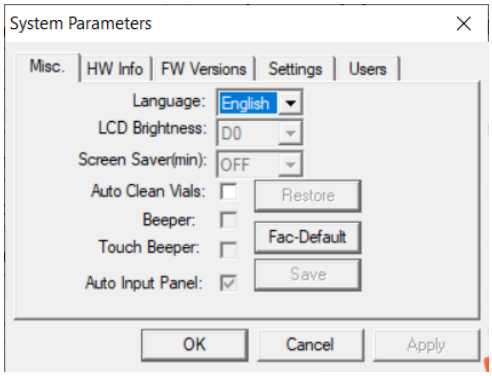
3.There are 6 six equilibrium counters of pre-heating positions and the corresponding vial numbers under the “heating” bar.

4.The “Exit” button and the operating information are at the bottom.

Note: When you click “Exit” to exit the task starting interface, do not power off the instrument, because it will transfer the sample vials at the pre-heating area back before existing.

3.4 SYSTEM PARAMETER

Click “system parameter” in the main menu to enter the system parameter interface.



Click “Ok” to save the parameter modified and exit. Click “Cancel” to exit without saving the parameter.

3.4.1 Important parameter

1.Language: Simplified English (default), Chinese (optional).

2.LCD brightness: used to set the brightness of screen.

3.Screen saver (time): used to set the time of screen saver.

4.Auto cleaning: used to select whether to operate auto cleaning when the instrument is turned on.

5.Sound prompt: used to set system prompt sound.

6.Click sound: used to set whether there is a sound when the screen is clicked.

7.Auto keyboard startup: used to set whether the keyboard is started automatically when entering.

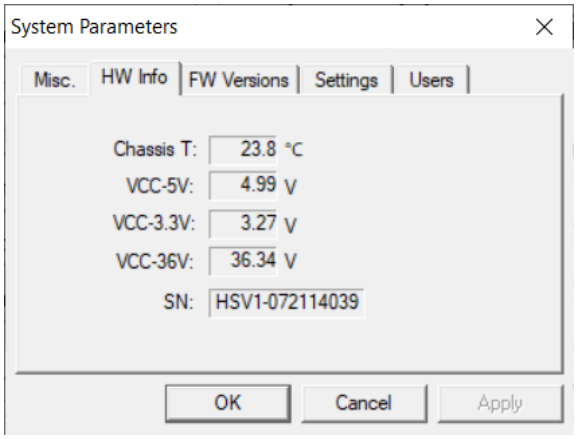
8.。Recover: recover to the original settings.

9.Default: Recover system parameters to default settings, including task list and method edit.

10.Save: Save the settings.

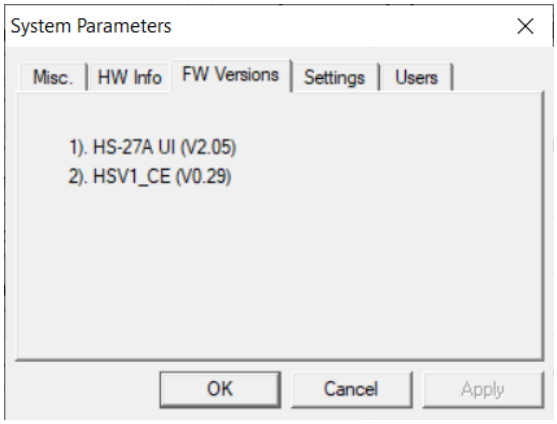
Note: “Default” is used to directly change the parameters without saving, please use it with caution.

3.4.2 Hardware information



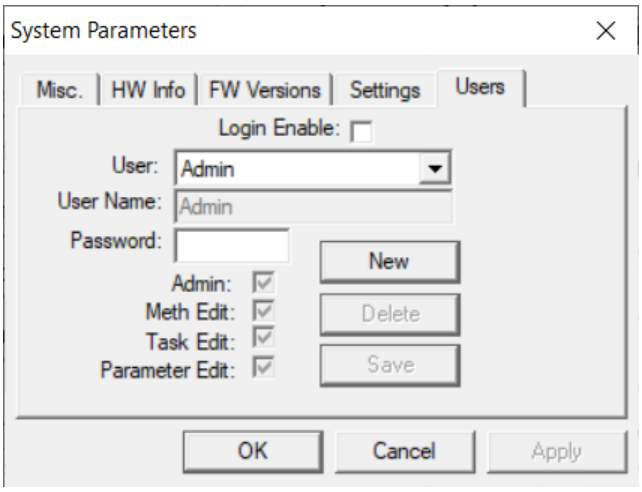
It displays instrument temperature, various important voltage and serial number of the instrument.

3.4.3 Software version



It displays the version of main panel program (HSS_UI) and interface program (HSV1_CE).

3.4.4 User management



Administrator user and rights of user

Note: 1.The original default is the rights of administrator, the password is blank, and you can login without password.

2.Only the administrator has the right to view and change this interface.

3.After modifying, the parameters become effective after restart.

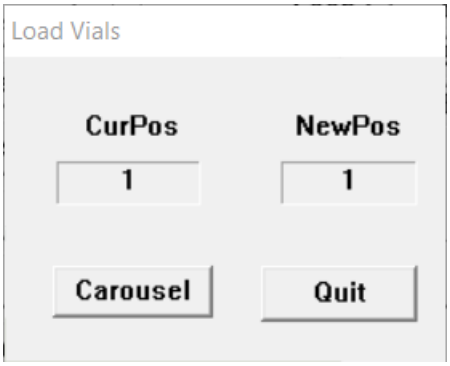
4.Only after “Login with passwords” is checked, the login interface appears when starting.

5.。Different rights are checked for different users, which can perform the corresponding operations.

6.To restore the factory settings (clear all users and passwords), it is necessary to click “Ok” button after the login interface logins short circuit panel J48.

3.5 SAMPLING

Click “Load Vials” in the main interface menu to enter the sampling interface.

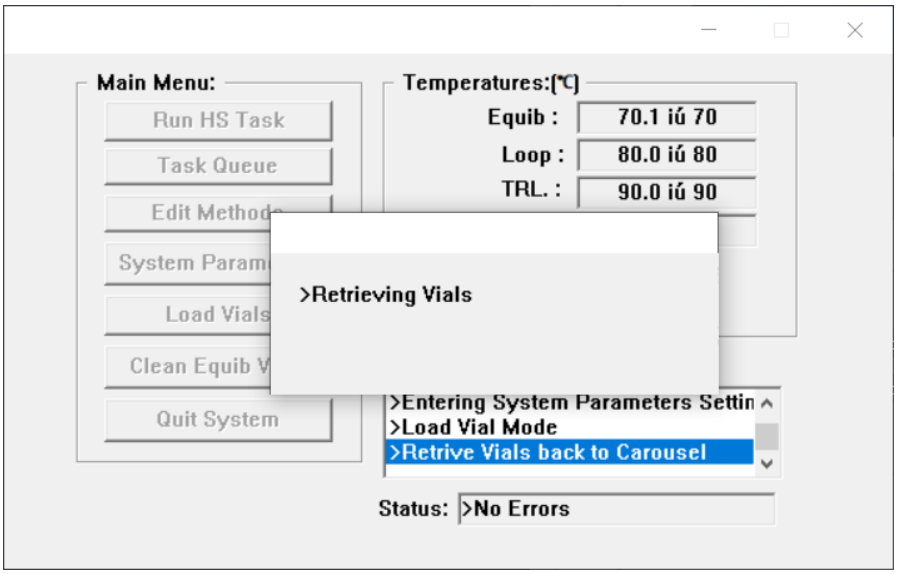


The vial tray rotates a position each time when clicking “Rotate vial tray”. The user can place vial at the tray. After placing the vials, click “Exit” to return the main interface.

Note: The position displayed is the position where the transfer arm takes the vial rather than the position faced by the user. Refer the vial number at the tray for the position of sample vial.

3.6 REMOVE EQUILIBRIUM VIAL

Click “Clean Equib vial” in the main interface menu to enter the remove equilibrium vial interface.



This process needs too long time. At first, the instrument needs to detect the number of vials in the equilibration area, then finds the same number of empty spaces in the tray, and finally returns the sample vials to the tray. This operation is generally used when the sample vials are left in the equilibrium area, such as sudden power failure.

3.7 OPERATING STATE

During normal operation, “normal system” is displayed. In case of any error, the system can automatically detect the fault point and help to repair. Error messages are divided into four types:

1. Power supply error (36V, 5V, 3.3V);

2. Motor error (vibration motor, lifting motor, transfer motor, tray motor, DC motor (six-way valve));

3. Temperature error (too high equilibrium temperature, sampling vial temperature, transfer line temperature, needle temperature, instrument temperature);

4. Serial port error (the port fails to open; indicating communication interruption during operation).

3.8 RESERVE COTNROL SOFTWARE

3.8.1 PREPARATION

1. Connect the instrument to a computer through the serial port.

2.Open the left cover of the instrument and unplug the J42 plug (at the lower left corner of the main panel, default unplugged).

3.Power on, run reserve control software on the computer.

3.8.2 INTERFACE OPERATION

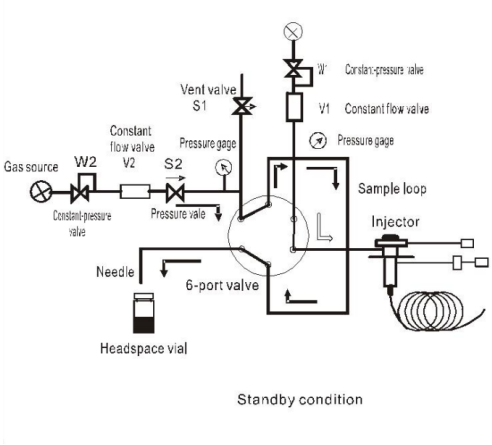
The operation of reserve control software is same as instrument interface. See chapter III for details.

CHAPTER IV OPERATIONAL PROCESS

4.1 OPERATIONAL PROCESS

4.1.1 STANDBY CONDITION

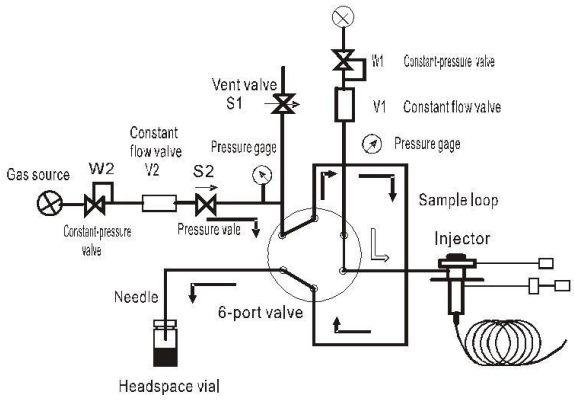
After pressure valve S2 is opened, auxiliary gas passes through valve V2, sample loop and injection syringe. Meanwhile, carrier gas, through valve V1, flows into GC sample inlet. At the beginning, the first sample vial is supplied from the sample carousel to the sample position 1 in the constant temperature oven and balance time starts.



Standby condition

4.1.2 VIAL PRESSURIZATION

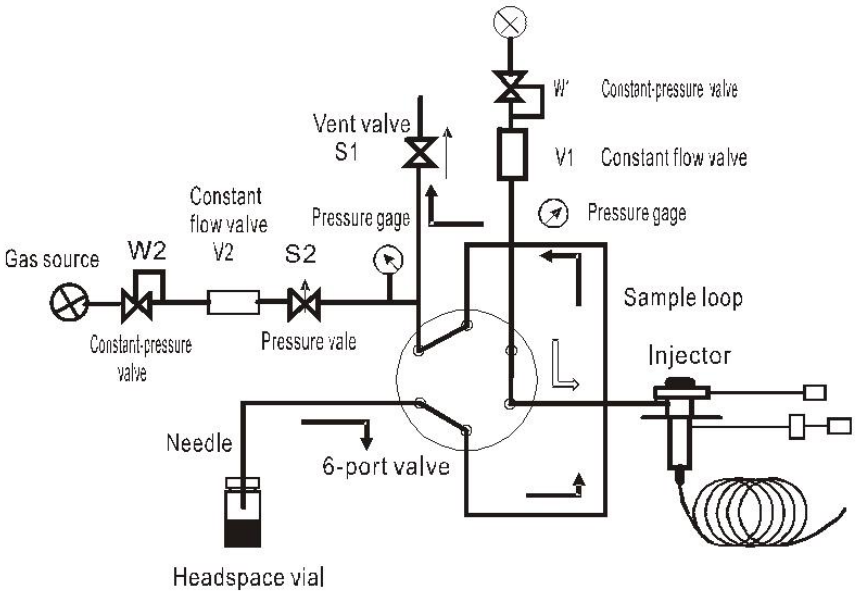
At the end of balance time, the sample vial is moved to the position of syringe needle and lifted up; the needle penetrates 15 mm into the sample vial. During edit operation, S2 pressure valve is activated and sample vial will be pressurized. The pressuring time is generally set to 10s, which is required for obtaining positive pressure of the sample vial。



Pressurization mode

4.1.3 LOOP FILL STATE

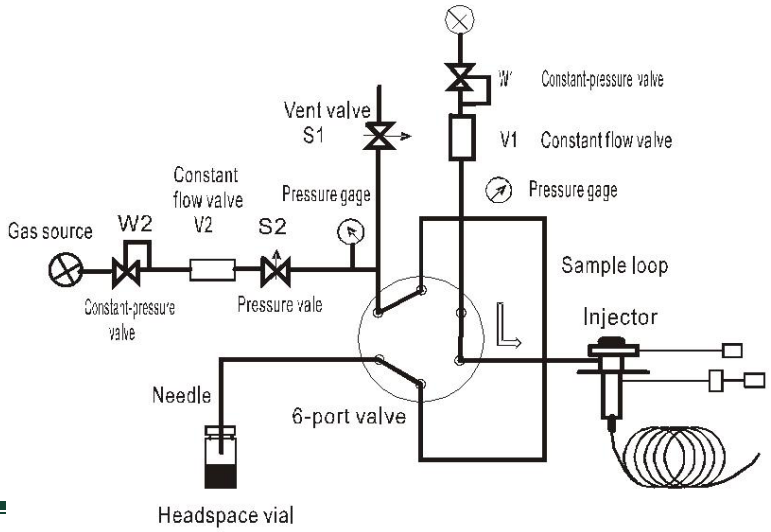
After pressurization, opening discharge valve S1 will make the sample loop filled with headspace gas, some of which will be discharged to the atmosphere through vent outlet. Pressure of sample loop can be balanced
with that of sample vial by activating discharge valve S1 in short time, i.e. 5-10 seconds, or choose to extend opening time of discharge valve S2 to 10-15 seconds to balance pressure in the sample loop and the atmosphere pressure.



Loop fill

4.1.4 LOOP FILL BALANCE

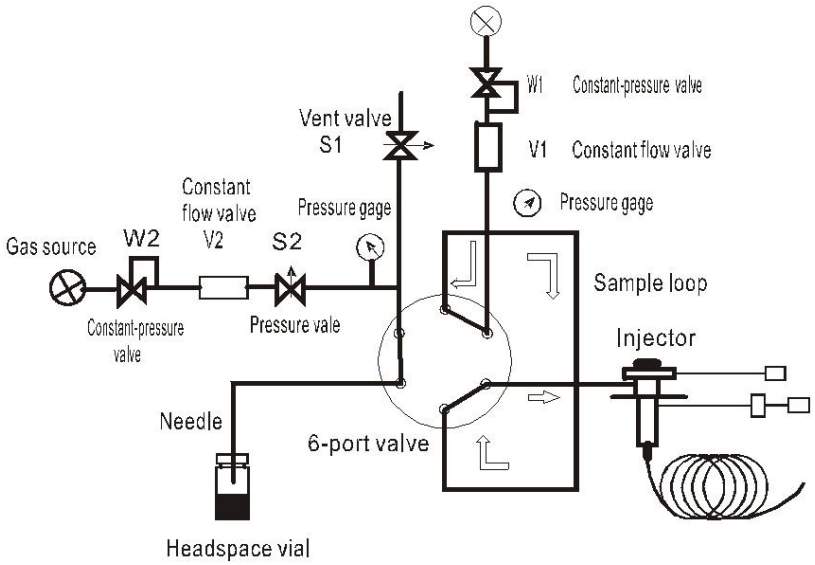
Pressure of sample loop can be balanced with that of sample vial by activating discharge valve S1 in short time, i.e. 5-10 seconds, or choose to extend opening time of discharge valve S2 to 10-15 seconds to balance pressure in the sample loop and the atmosphere pressure.。



Loop fill balance

4.5 INJECTION STATE

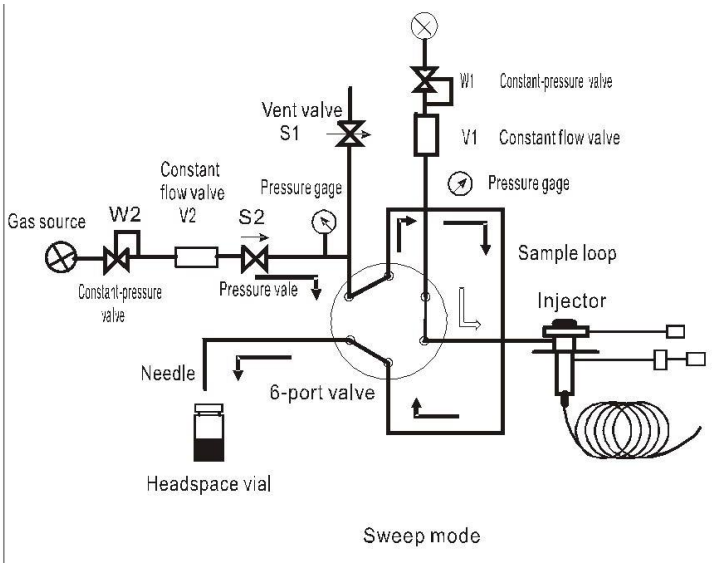
Turn the 6-port valve to allow the sample loop to switch to carrier gas transmission line, which will then enter GC inlet through the sample loop and transmission line. The duration for this section is related to total flow rate entering GC inlet. For example, if the flow rate of installed packing column is 30 ml/min, transmission of 1 ml of gas phase in the sample loop may cost 2 seconds. GC injection time is generally set to 10s.



Injection to GC

4.6 ENDING OF CYCLE

The sample vial is located to the original position in the sample carousel. pressure valve S2 is activated to sweep the whole gas line and the valve circulation is restored to the wait position, i.e. to sweep state.



Note:When a toxic sample is analyzed, a filter should be installed in the headspace gas discharge outlet to absorb toxic gas.

4.2 CONTINUOUS OPERATION OF SAMPLE VIAL

To obtain same balance interval during operation of GC, the sample vial is successively put into constant temperature furnace and resumed to the original position after injection. The editor, based on the time set for the analysis period, minimizes the time required for running all samples. The editor starts the balance of the next sample vial when any one of injection cycle is performed.

If the sample to be detected is toxic, an appropriate filter shield shall be installed at the air outlet to absorb toxic compound.

4.2 CONTINUOUS RUNNING OF SAMPLE VIALS

To obtain the same equilibrium interval during operation, the vials are placed into the preheating area one after another and returned to their original positions after sampling.

According to the time set by the software based on the analysis cycle, next vial is preheated in advance to minimize the time required to run all samples.

CHAPTER V SIMPLE OPERATION

5.1 INSTRUMENT INSTALLATION

- Place the injector on the chromatographic instrument and used the supplied cables to connect the gas line. The carrier gas of headspace is connected to the relief valve of cylinder. Turn off the pressure regulator of carrier gas at the chromatograph and turn on the carrier gas valve of headspace.
- Insert the booster cables of work station and chromatograph in the corresponding holes at the back of the headspace.
- Plug in the power cable.

5.2 PREPARATION

- Prepare the test sample or standard sample and place it in a clean headspace vial.
- Seal the headspace vial with a special sealing pliers to prevent gas leakage.
- Turn on the power switch and the instrument starts a self-test.
- After the self-test, enter the method edit and task list from the main interface and set the parameters.
- The main interface enters the sampling interface. Put the sealed, pre-prepared vials at the appropriate position.
- In the main interface, start the headspace program, and the instrument begins to preheat.
- Adjust the valve stem of pressure regulator at the front panel of the headspace, change the flow rate of pressurization gas and change the pressurization time to adjust the pressure and thus determine the optimal analysis conditions. See the relevant contents of Chapter 1 for details.
- After the conditions are determined, click “Exit” button to exit the headspace program.

5.3 ANALYTICAL OPERATION

After setting parameter, place the vial to be detected, and start the program for analysis.

CHAPTER VI MAINTAINING THE INSTRUMENT

6.1 CHANGE THE SAMPLING LOOP

Loosen the screws on the right sampler cover and lift the cover.

Remove the manifold cover and insulation.

Connect the sample loop to injection valve directly and fix it to the sampling system with a cylinder-shaped aluminum frame.

Use a 7-mm wrench to remove and install two ends and frame of the sampling loop.

Preform a leak test before covering the cover.

6.2 CHANGING THE SAMPLING NEEDLE

Loosen the screws on the right sampler cover and lift the cover.

Remove the screws used to fix the sampling needle, change it, make the joint tight, and perform a leak test before replacing the cover.

6.3 CHANGE THE TRANSFER LINE

Loosen the screws on the right sampler cover and lift the cover.

Remove the manifold cover and insulation.

Use a 7-mm wrench to remove the transfer line, always preform a leak test before inserting the transfer line in GC injection port.

6.4 STEAM CLEANING THE SAMPLING SYSTEM

The inert gas always flows through the transfer line, which means that most of the contaminants are removed during the “normal” injection process.

However, the system may be contaminated with components that cannot be removed. In this case, it is recommended to clean the injector with steam.

In this process, clean water is injected into the sealed vial. The vial is heated to a temperature above the boiling point and produce pressure, and then pressurized steam goes through the transfer line to clean it.

Caution

Due to the risk to explosion and possible contamination of solvents, only water can be used for steam cleaning.

The process is as follows:

Heat to the equilibrium temperature 125°C.

The sampling system and transfer line are heated to 150 °C.

Set the pressure to 1 bar.

Fill 20 ml sample vial with 10 ml clean water. Balance the vial for 15 minutes without shaking.

Caution

When the water temperature reaches 100 °C or higher, the pressure in the vial may increase. Please use a new vial and carefully seal it during this process. Person injuries may be caused due to broken or incorrectly sealed vial, for example, the vial is broken or the lid is too fragile to withstand the pressure.

1. Disconnect the injector and the chromatograph.
2. Enter the start interface.
3. After 5-10 minutes of steam cleaning, blow the steam out.
4. Run an extra cycle with an empty bottle to end the cleaning process. Put an empty vial into the system and repeat the procedure.