Gas Chromatograph-Mass Spectrometer
GC-MS-II
User Manual

DRAWELL
Artist of Science

Please read operating manual before installation and operation.

Drawell International Technology Limited
Chongqing Drawell Instrument Co., Ltd.
Shanghai Drawell Scientific Instrument Co.,Ltd.

Chongqing Center : Suite 2705,Building No.12,Shiyou Road No.1, Yuzhong District, Chongqing, China.
Shanghai Office : Suite 1117,Lane561 XiuChuan Rd.,PuDong New Area,Shanghai,China
Homepage : www.drawell.com.cn
Tel : 0086-023-63268643
Email : sales02@drawell.com.cn

General

This Operation Guide describes the procedures for properly operating and maintaining the instrument. The operator of the instrument should carefully read and fully understand this guide herein before running the instrument. This guide should be readily available for reference by operators who will operate and maintain the instrument.

Precautions for Safe Operation

The gas chromatograph mass spectrometer is an analytical instrument used for qualitative and quantitative analyses.

Please note the following points for safe operation of this instrument.

1) Use this instrument only for the specified types of analyses.
2) Follow the procedures as written in this manual.
3) Observe all warnings and precautions.
4) Do not disassemble or modify the instrument without our approval.
5) For service or repair, contact our after-sales service dept..

Warnings and cautions in this manual are specified as follows:

Warning: Indicates a potentially hazardous situation that may result in a serious injury or causes death.

Caution: Indicates a potentially hazardous situation that may result in minor or moderate injury.

Operation Precautions

Warning

Always wear safety glasses or goggles when handling solvents. If solvent gets into the eyes, blindness could occur. Should solvent get into the eyes, immediately flush with large amounts of water and seek medical attention.

Warning:

Do not place solvents near PCs or printers, as fire or instrument damage may occur.

Warning:

Do not use flammable sprays (hair sprays, insecticide sprays, etc.) near this instrument, as they may ignite and cause a fire.

High-Pressure Gas Cylinder Precautions

Warning

A high-pressure gas cylinder will be used to supply the carrier gas. When handling the gas cylinders, observe the following precautions.

1. Keep gas cylinders in a well-ventilated area outside of the instrument installation site. Avoid direct sunlight. Use lines to transport the gas from the cylinders to the instrument. Relevant laws and regulations shall be abided by when flammable gas involved.
2. Please note that the temperature of the gas cylinder normally may not exceed 40℃. No open flame is permitted within 2 m of the cylinder.
3. Use soapy water or other solvent for leakage check before use the high-pressure gas cylinders. Especially in the case of flammable gas (acetylene, hydrogen, etc.) or combustion gas (such as oxygen, nitrogen oxides), no flame is permitted within 5m of the gas cylinders. Valid fire extinguishers are additionally required. (See GB 50140-2005).
4. Secure cylinders with clamps or by some other methods to prevent them from falling over.
5. Be sure to use oil-free valve as pressure reducing valve. In addition, do not use a valve adhesive with oil at the inner gas surface.
6. Close the main cylinder valve immediately after use.
7. Do functional check for the pressure gauges once every month.

Handling Emergencies

The following measures should be taken in case of an emergency. Reuse the instrument with great care and contact Drawell after-sales service dept. if necessary.

In the event of an emergency,

1. Turn off the gas chromatograph and mass spectrometer.
2. Turn off all accessories.
3. Turn off the power supply.
    a. If the power cable is attached to a switch box, turn off the switch box.
    b. If the power cable is plugged into an outlet, unplug the cable.
4. Close the valves of all carrier gas, hydrogen, and air lines.

Table of contents

1. Introduction

1.1 Some Abbrs

For your reading convenience, the abbreviations used in this manual are listed below.

Table 1 Abbr

Abbr.	***Def.***
amu	Atomic mass unit
DC	DC voltage
EFC	Electronic Flow Control
EI	Electron impact ionization source
EPC	Electronic Pressure Control
Full scan	Full scan
GC	Gas Chromatograph
HED	High dynode
m/z	mass-to-charge rate
MC	Mass chromatogram
MS	mass spectrometer
OFN	Octafluoronaphthalene
PCB	Printed circuit board
PFTBA/FC43	Perfluorotributylamine (calibrant)
RF	Radio-frequency
SIM	Selected ion monitoring
TIC	Total ion chromatogram

1.2 Important Safety Warning

Using GC-MS-II, you should always pay attention to the following important safety precautions:

1) If the MS is connected to the power supply even when the power switch is turned off, the following components will still exist potentially dangerous voltages:
   - The wiring connecting MS power cord with AC source;
   - The wiring of AC source itself as well as connecting AC source with the power switch.
2) Turning the power switch on, potentially dangerous voltages also exist in:
   - All the electronic circuit boards in the instrument;
   - Internal cables connected to these boards;
   - All the wiring connecting heater (column oven, inlet, MS interface, etc.).

Warning

All these parts, shielded by covers, can barely touch those dangerous voltages accidentally when those covers are in-situ, unless explicitly stated, don't remove any cover when the detector, inlet or column oven are working.

If the outer insulation of power cord is frayed or worn, the cord must be replaced. Please contact Drawell service department.

3) Parts of high temperature danger

Many parts of the instrument operate with a temperature high enough to cause serious burns; these parts include, but not limited to:

- Inlet
- Column oven and internal units
- GC & MS junction (MS Interface)

These parts of the instrument are allowed to be touched only after being cool to room temperature, if the heating zone temperature is pre-set to room temperature, these parts can be cooled faster. Close the heating zone once reaching the set temperature.

Warning

GC will emit hot gas that may burn the operator during the refrigerating circulation. Be careful when operating behind the instrument.
The combustible material (or flammable / non-flammable filament material) stacking around the mechanical pump would cause fire, keep the mechanical pump and the surrounding environment clean.

Mark

The user must comply with the manual or the ***Warning*** on the instrument for operating, maintaining or repairing this instrument. Disobeying these precautions violates the related safety standards and correct use of instruments. Drawell Instrument bears no responsibility of the damages caused by customers not complying with those standards.

Notice danger.

Indicates hot surface.

Indicates HV danger.

Note

The best way to maintain MS normal operation is to keep “Turn on” and maintain a higher temperature under the condition of carrier air flow. If intend to move or store MS, it is recommended to consult Drawell service department for information. MS must always remain upright and be taken great care when moving. MS vacuum system should not be in a state of long-time communication with the air. While open the instrument case, avoid spilling any liquid on MS.

2. Instrument installation

2.1Outer Configuration Requirements

2.1.1 Physical Characteristics

The whole package of GC-MS-II is rectangular with a length of 105 cm, width 60 cm, height 50 cm, among which the mechanical pump weighs 23 kg, while the whole machine (excluding mechanical pump) weighs 100 kg.

Warning

The instrument bench is required to be stable, able to withstand the instrument, to prevent the instrument from dropping or falling over.

2.1.2 Gas Requirements

Pressure and volume of the helium cylinder: purity ≥ 99.999%, 15Mpa;

Pressure reducing valve: helium or oxygen valve (output press range is 0.5~0.9 Mpa, no leakage).

2.1.3 Instrument working environment requirements

Table 2-1 Instrument Working Environmental requirements

Influencing Factor	Unit	Normal operating condition
Ambient temperature	℃	18～26
Relative humidity	%	40-50
Atmospheric pressure	kPa	70.0～106.0
External electric field, magnetic field, electromagnetic field	—	None
Working position	—	Dust-free Laboratory
Ventilation	—	Good
Mechanical vibration	—	None
Harmful gases	—	None
Supply voltage	V	Rated voltage±5%
Power frequency	Hz	Rated frequency±1%

Warning

The solvent used by GCMS is flammable and toxic, so the instrument should be placed in an adequately ventilated room, otherwise poisoning or fire will be easily caused.
Do not use in an environment with explosive, flammable gas, to avoid fire risk.

Warning

Do not place flammable materials near the column oven vent at the rear of GC to avoid fire risk.

Warning

Avoid placing the instrument in an environment abundant of corrosive gas or dust, otherwise it is impossible to ensure the instrument's performance, or the life of the instrument will be shortened.

2.2 Installation and Connection

Related materials

Part name	Part No.	Application
Plug power cord	3691000040	Connect MS to AC power
Plug power cord	3691000075	Connect GC to AC power
Computer Cables	3694000012	Connect the mechanical pump to MS
Corrugated plastic pipe	304742407	Connect the exhaust port of the mechanical pump to the exhaust duct
Glass fuse	3561000004	Limit input current to MS
Network cable	3699000078	Connect MS network port to that of the computer
Serial Line	3694000077	Connect GC serial port to that of the computer
Corrugated pipe	304742411	Connect the air outlet of MS to the mechanical pump
Sealing ring	304579020	Seal the entire air pipe
Clamp	304741201	Lock the entire air pipe tightly

Place the instrument in sequence as in Figure 2-1; connect the power cords of GC, MS and mechanical pump to the appropriate source (see Figure 2-2).

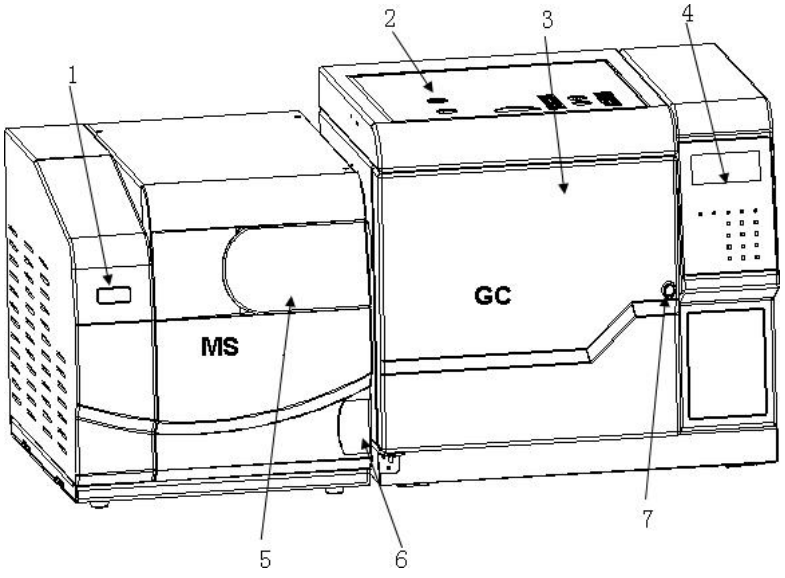


Figure 2-1 GC-MS-II Schematic front view

1-Status Lamp 2-Injection 3- GC Oven door 4-GC Control Panel

5-Vacuum Chamber Front Panel 6-PFTBA 7- Door Lever

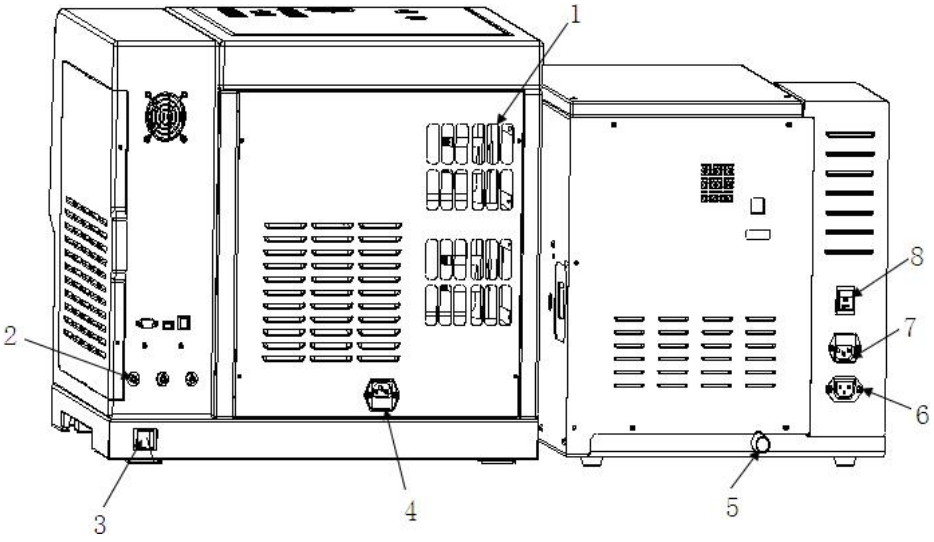


Figure 2-2 GC-MS-II Schematic rear view

1—Exhaust Opening 2—Carrier Gas Inlet 3、8—Power Switch

4—GC Power Socket 5—Vacuum Line 6—Mechanical Pump Power

7—MS Power Socket

2.3External gas path installation

Related consumables and tools

Part name	Part No.
Valve connector	3010605408
Nuts（Φ3.2）	3010603528
1/8”front ferrule	304760233
1/8”back ferrule	304760234
Double wrench（1/4-5/16）	3802000722
Wrench	3802000121
O-ring 3×1.8（optional）	304570322

Installation steps

1. The installation of the external gas path is as shown in Figure 2-3;
2. Keep cylinders upright and be fastened in the anti-falling base;
3. Clean the cylinder valve port with alcohol or acetone;
4. Mount the valve inlet on the cylinder and tighten it with a wrench;
5. Release the pressure regulator handle.
6. There should be some indications on the reducer pressure gauge when opening the cylinder pressure valve; and the gauge indicator should not fall when close the cylinder pressure valve, otherwise it means leakage somewhere, which should be eliminated beforeuse.

   Gas path tube of the stainless steel and valve connector are connected as shown below.

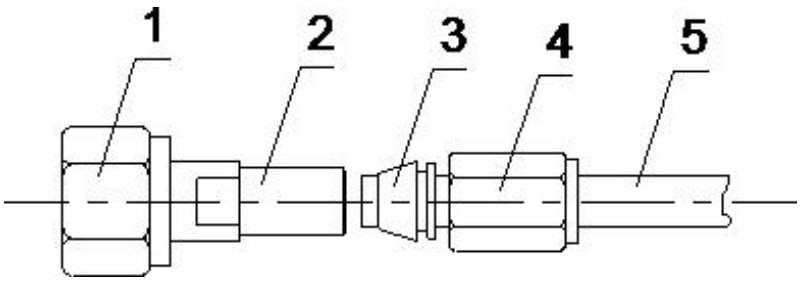


Figure 2-3 Gas path connection schematic diagram

1-Nut (M12) 2-Pressure Reducing Valve Connector

3-1/8”Front/Back Ferrule 4-Nut (M8) 5- Φ1/8”×0.5 Stainless Steel Conduit

Warning

For safety regulations on air source, please refer to the above mentioned *High pressure cylinders Use Precautions*.

The quality of the gas should meet the gas source requirements of GC-MS, to avoid affecting the results or contaminating or even damaging the instrument.

Mechanical pump exhaust outlet uses pipeline to exhaust gas outside, to prevent indoor air pollution when analyzing toxic substances.

Leakage check must be paid attention to during practical operation. Wherever a leakage occurs, the limit is from affecting instrument working to causing an emergency (such as hydrogen leakage may cause an explosion).

2.4External Gas path leakage check

There's a need of leakage check for the installed the external gas path. The check steps are as following:

1. Set the cylinder valve regulator handle at a relaxed state, open the cylinder pressure valve, then slowly adjust the handle until the low-pressure gauge indicates at 0.4MPa;
2. Smear out the leak fluid at each joint of the external gas path, to observe whether there are bubbles;
3. Leak fluid can be soap solution or other mixtures prone to produce bubbles.

Warning

The external gas path needs leakage check after being installed, to avoid helium leakage!

2.5Septum, glass liner installation

Figure 2-4 depicts GC split / splitless inlet exploded view.

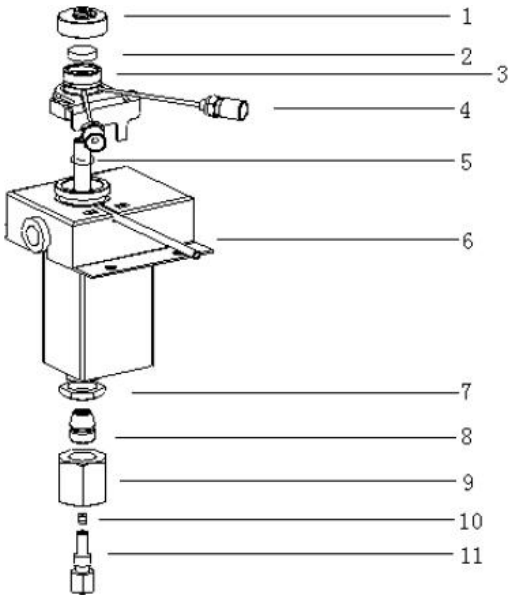


Figure 2-4 Split / splitless inlet exploded view

1— Septum Nut 2—Septum 3—Nut(Injection Port) 4—Injection Port 5—Fluororubber O-Ring 6—Glass Insert Port 7—Nut(Heat Block) 8—SS Ferrule 9—Nut(SS Ferrule) 10—Ferrule(Graphite /Vespel) 11—Nut(Ferrule)

2.5.1 Glass liner installation

Related consumables and tools

Part name	Part No.
Glass liner(for split or splitless)	304743254
Quartz wool	305330043
O-ring	304570315
Inlet wrench	38020004000
Tweezer	3802000724
Gloves	3801001144 (or similar types of gloves)

(1) Confirm the position of quartz wool

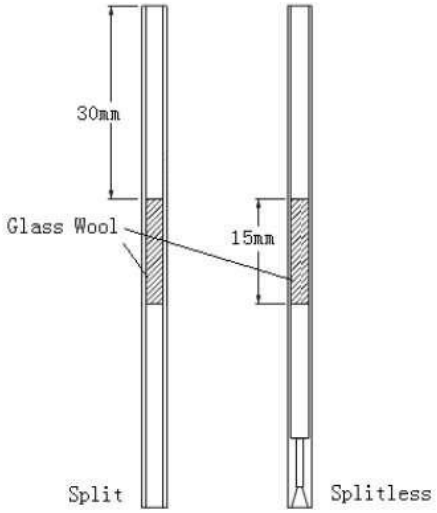


Figure 2-5 Loading position of quartz wool in the glass liner

(2) Install O-ring: O-ring installed onto the glass liner about 7mm from the top; put the glass liner into the nebulization chamber and push to the bottom where the ring is about 5mm from the top.

(3) Fasten glass liner: Carefully tighten the fastening nut by special inlet wrench.

2.5.2 Septum Installation

Related consumables and tools

Part name	Part No.
Injection septum	305330042
Tweezer	3802000724
Gloves	3801001144 (or similar types of gloves)

(1) Hand-tighten the septum nut (Figure 2-6).

(2) Loosen half circle of the nut.

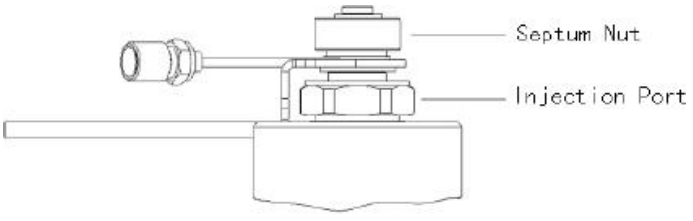


Figure 2-6 Inlet outline

Warning

Do not directly touch the glass liner or septum.

2.6GC Column Installation

Related consumables and tools:

Part name	Part No.
Column	3709400080
Column cutter	3802000251
15% Graphite /85%Vespel pressure ring	304650006
Pressure ring ferrule	3010205443
Interface column nut	3041810051
Column mounting jig (for interface end use)	3010211139
Wrench (1/4 inch×5/16 inch)	3802000722
Septa (used or second-hand is allowed)	305330042
Gloves	3801001144 (or similar types of gloves)
Inlet nut	3010211114

2.6.1 Inlet side installation

1. Pass the position markers (such as old septum), pressure ring ferrule and the pressure ring through the free end of the column (as shown in Figure 2-7) in order. The tapered end of the ring must face the ferrule.
2. Use the column cutter to cut off the column end for 1～2cm, and make sure that the length of the column protruding the pressure ring is 4 ~ 6mm.
3. Use the magnifier to inspect the column fracture, if not clean or smooth, please repeat step 2.
4. Wipe the front end of the column with acetone or other solvents put it into the inlet.
5. Please be noted that the nut shall be first tightened manually as possible as one can, and then with a wrench for 1/4 turn; be careful that over-tightening will affect the sealing effect.

Warning

Have the column circle around the metal frame hanging on the column rack of the oven. The hanging position depends on the diameter of the frame, and it's better to have the column located in the center of the oven. Both ends of the column protrude from the frame bottom, smoothly bending towards the inlet and MS interface; do not let any part of the column touch the inner wall of the oven.

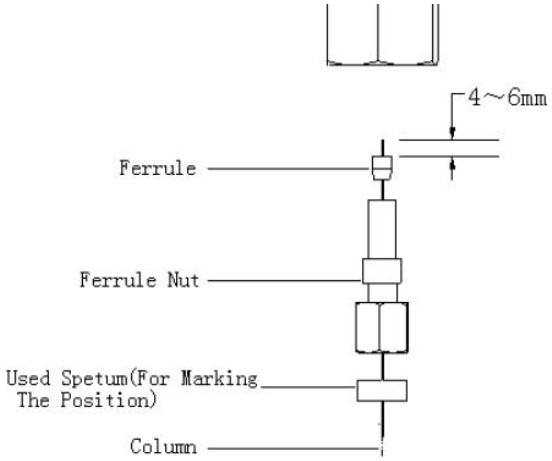


Figure 2-7 Column installation at the inlet end

2.6.2 MS interface end installation

(1) Pass the position markers (such as old septa), pressure ring ferrule and the pressure ring through the free end of the column (as shown in Figure 2-8) in sequence. The tapered end of the ring must face the inlet bolt.

(2) Insert the front end of the chromatographic column into the column measure with 1cm exposed outside of the measure. Then tighten the nut, cut off the exposed part of column and ensure a smooth fracture.

(3) Remove the column from the measure after fasten the position markers, gently wipe the chromatographic column with alcohol or acetone before insert it into the MS interface, and then tighten the nut.

(4) Please be noted that the nut shall be first tightened manually as possible as one can, and then with a wrench for 1/4turn; be careful that over-tightening will affect the sealing effect.

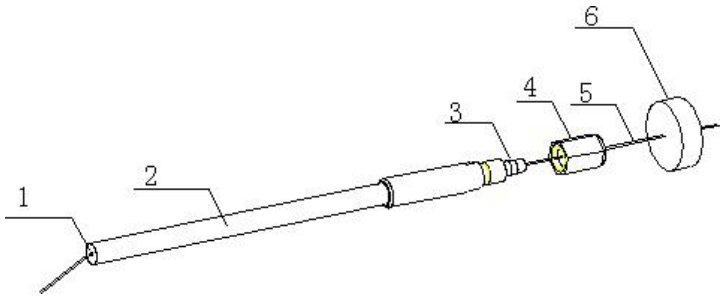


Figure 2-8 Column installation at MS interface end

1—Cut Column Flush With End of Jig 2—Column Mounting Jig

3—Ferrule 4—Nut 5—Capillary Column 6—Used Septum

Warning

When the oven and interface are heated, pressure ring will shrink slightly; it needs leakage check after one or two heating cycles.
Please make sure that the MS interface has been cooled to normal temperature; disassemble the nuts at high temperature will damage the thread of MS interface.

2.7 Filament installation

Related consumables and tools

Part name	Part No.
Ion source filament	2072012
Straight screwdriver	3802000073
Sleeve	3802000077
Tweezer	3802000724
Gloves	3801001144 (or similar type of gloves)

Warning

There's voltage danger inside the MS chamber. Turn off the instrument and make sure the main power switch is off before operation.
Ion source and interface will turn hot during operation; please turn off the instrument and let it cool for at least 30 minutes before maintenance.

Warning

The operator must wear clean and dust-free gloves to avoid contamination when operating inside the MS chamber.

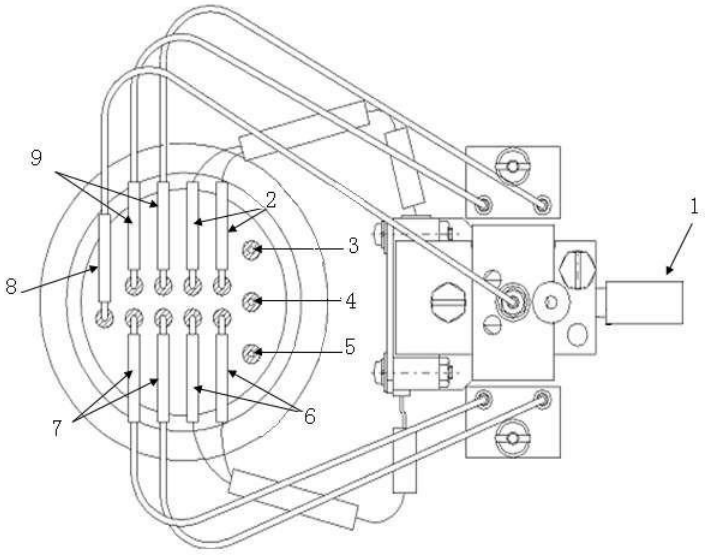


Figure 2-9 Ion source

1—GC-MS Interface 2—Heating Assembly Terminals 3—Lens1

4—Lens2 5—Lens3 6—Sensor Terminals 7、9—Filament

8—Repeller Electrode Terminal

(1) Make sure that the main power of the instrument is switched off.

(2) Loosen the 4 nuts on the front panel with a sleeve; remove the front panel.

(3) Use a tweezer wiped by alcohol or acetone to remove the filament lead terminal (see Figure 2-9).

(4) Loosen fixing nuts of filament with a straight screwdriver and pull out the filament (see Figure 2-10).

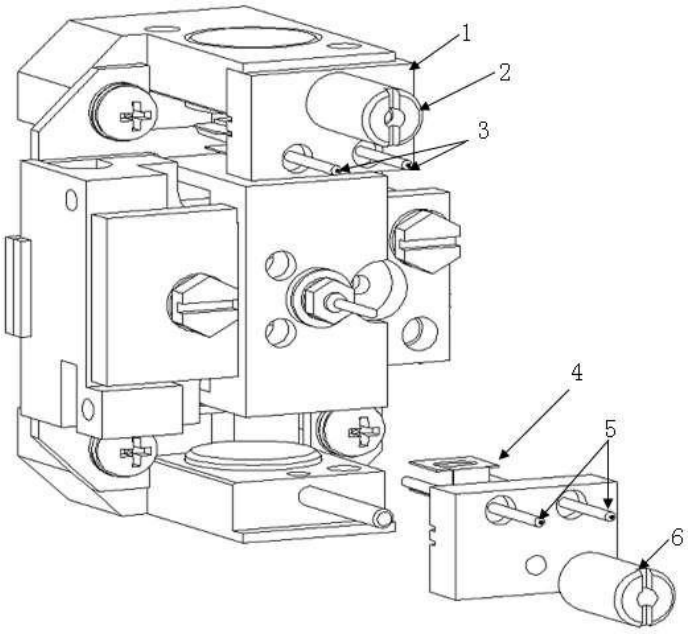


Figure 2-10 Ion source assembly

1、4—Filament 2、6—Locking Nut 3、5—Terminal

(1) Install the new filament and tighten the nuts.

(2) Push the lead terminal into the lead pin, reconnect the filament.

Warning

The filament should be fully inserted and well contacted when installing, and avoid the filament wire contacting other parts.

2.8Install ion source electrode box and repulsion electrode

Related consumables and tools

Part name	Part number
Electrode holder 1	3010211123
Electrode holder 2	3010211125
Insulating ceramic 1	305301217
Repulsion electrode	3010211131
Ion source holder	3010211100
Nut M2	304050001
Flat washer M2	304070001
Flat washer M3	304070002
Cover bolts	3042110010
Allen wrench 2.5mm	3802000001
Tweezer	3802000724
Gloves	3801001144 (or similar type of gloves)

Warning

Be careful of electric shock. Turn off the instrument and make sure the main power switch is off. Ion source and interface turn hot during operation. Please turn off the instrument and let it cool for at least 30 minutes before maintenance.

Warning

Be aware of burns since the ion source electrode box and repulsion electrode are still very hot after drying. Ensure the parts being completely cooled before maintenance.

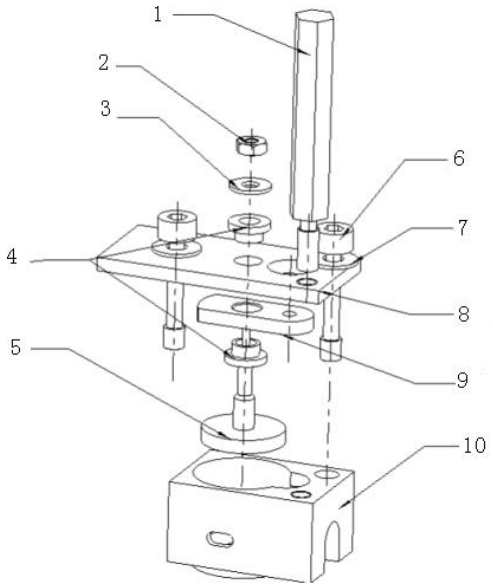


Figure 2-11 Ion source box and repulsion electrode

1—Ion Source Holder 2—Nut 3—Gasket(M2)

4- Ceram Gasket 5—Repeller Electrode 6—Screw

7-Gasket(M3) 8—Electrode Trestle 1 9—Electrode Trestle 2

10—Electrode Box

(1) Install the parts in an upward order, as shown in Figure 2-11.
(2) Put the repulsion electrode into the ion source electrode box and adjust it to situate the repulsion electrode in the center of the electrode box.
(3) Fasten the ion source holder to the electrode box prior to assembly the electrode box to the target position by holding the ion source holder.
(4) Tighten the two screws of electrode box, first the left one, remove the ion source holder, and then the other one to ensure equilibrium force.
(5) Connect to the repulsion electrode lead terminal.

Warning

Do not over-tighten the screws; otherwise it will deform the lens or damage the insulating parts. Make sure the repulsion electrode and the ion source electrode box are insulated.

3. Instrument Startup and Shutdown

3.1 Turn on the instrument

3.1.1 Power on

(1) Start computer and printer. Start Windows.
(2) Open the valve of the helium cylinder after ensuring that the pressure shown on the high pressure gauge is not lower than 3 MPa.
(3) Make sure the power supply of the instrument is switched on; then turn on the power of gas chromatograph (GC)
(4) Turn on the power of mass spectrometer (MS). The power indicator at the upper left corner of the MS front panel will light up.

3.1.2 Start software

(1) Double-click the ChemAnalyst (GCMS) icon on the desktop of computer, and the startup interface of ChemAnalyst software will display.
(2) The software will be connected with the instrument automatically after the software has been completely started. [Connected] will be displayed on the lower left corner of the software interface after the system being connected successfully.
(3) [Security protected] means that although the software has been connected with the instrument successfully, the instrument vacuum does not meet the requirement of working conditions and the system is under security protected.
(4) [Running] means that the instrument has been started successfully and is ready for tuning and measuring.
(5) Real-time monitoring value of main parts of the instrument will be displayed below the surface of the software.

3.1.3 Start vacuum system

Notice

Mechanical pump oil would become very thick if the instrument had been shut down for a long time and the ambient temperature is relatively low. The motor of mechanical pump may bear too much load when the oil is too thick and the mechanical pump is running. When the instrument is shut down at a cold environment, it is required to raise the ambient temperature, and the instrument should not be started before the temperature of the mechanical pump reaches at least the lowest temperature requirement (15℃).

Check if the screws on the door of MS front panel are tightened before starting the vacuum system.

Do not tighten the nuts on the MS front panel after starting the vacuum system. Otherwise, it will be very difficult to move the front panel after turning off the instrument.

(1) Click the function icon [Monitor] on the left of the software interface.

Figure 3-1 the Monitor icon

(2) A dialog box [System monitoring] will be displayed on the screen, as shown in Figure 3-2.

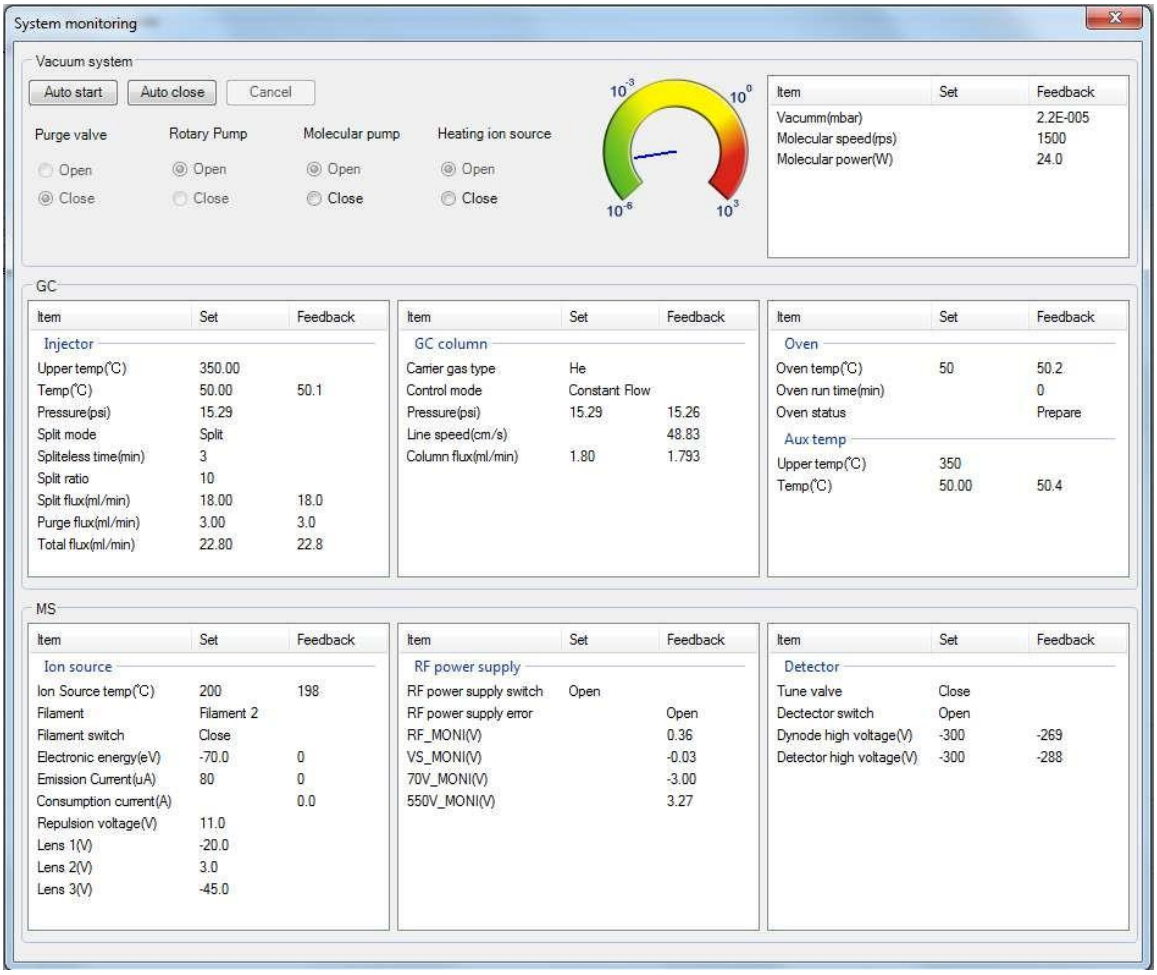


Figure 3-2 System Monitoring

(3) Click [Auto Start] or [Auto Shutdown], the system will start or shutdown the vacuum automatically.

(4) Operations of opening or closing the [Vent Valve], [Mechanical Pump], [Molecular Pump] or [Ion Source Heating] are also allowed.

(5) During the process of [Auto Start] or [Auto Shutdown], click [Cancel] would stop the current operation, but the system would not restore to the status before the operations of [Auto Start] or [Auto Shutdown] was taken.

After clicking [Auto Start], the system will start the mechanical pump first, and then start the turbo molecular pump when the vacuum degree is below 22 mbar.

If an alert of [Mechanical Pump Startup Timeout] or [Molecular Pump Startup Timeout] pops up, the instrument may have a gas leak. Please check if the O-ring is well-placed or if the screws are tightened at the places such as the front panel of the ion source, GC-MS interface, and so on.

Click [Auto Start] again, the green indicator on the MS panel will light up and flick. The green indicator keeps on after the system starts.

After clicking [Auto Shutdown], the ion source heating will be shut down first; the molecular pump will be shut down after the ion source temperature drops to 120℃ and below; and after the revolving speed of the molecular pump is lower than 200rps, the mechanical pump will be shut down and the vent valve will be open, and the vacuum is shutdown.

Note

After starting vacuum, it is required for at least 3 hours of waiting for stability of the instrument. Leak check may be performed during this period of time, and it is not recommended to start sample analysis test during the time.

3.1.4 Power off

(1) Power off the computer, monitor and printer.
(2) Switch off GC.
(3) Switch off MS.
(4) Close the main valve of the gas cylinder.

3.2 System standby settings

(1) Click [Instrument] [Standby], the instrument will switch to Standby status.
(2) At the Standby status, MS turn off the detector high voltage and RF, and lower the speed of the molecular pump to less than 1000 rps while the temperature of ion source keeps at 200℃.
(3) The temperature at the inlet, oven and GC-MS interface of GC will drop to 50℃ and low, and the inlet will be set to splitless status.
(4) If the instrument will not be used for a long period of time, set to Standby status to stable the vacuum status of the system, prevent from being contaminated and shorten its stabilizing time.
(5) It is not recommended to set the instrument to Standby status every day after work, the temperature of ion source dropping to 200℃ is only required.

Note

Do not frequently start up and shut down vacuum if not necessary; it will shorten lifetime of the turbo molecular pump.

Pay attention to the value of [Power consumption of Molecular Pump] on the status feedback bar of the instrument. Normally, the value of [Power consumption of Molecular Pump] should be < 30W after the molecular pump starts. If the value of [Power consumption of Molecular Pump] is higher than 30W for a long period of time, please check the flow settings of GC column and the sealing status of the system.

3.3 Check vacuum status of the system

(1) Further operations can be performed only after the vacuum has being started for 3 hours and the vacuum degree is better than 5.0E-005mbar.
(2) Open the tuning files recently used on the [Tuning] interface.
(3) Select [H_2O, Air] mode.
(4) Click [Set Parameters].
(5) Click [Start Acquisition].
(6) It's better the parameter of [Detector HV] is not higher than -700V before being aquired, otherwise it cannot be sure if there is a leakage; the value of [Detector HV] may be increased appropriately if the peak intensity is too low after acquisition started.

Note

If the vacuum degree does not reach 5.0E-005mbar, do not light up filament or collect data, otherwise it may shorten lifetime of filament or even cause damage to the other parts.

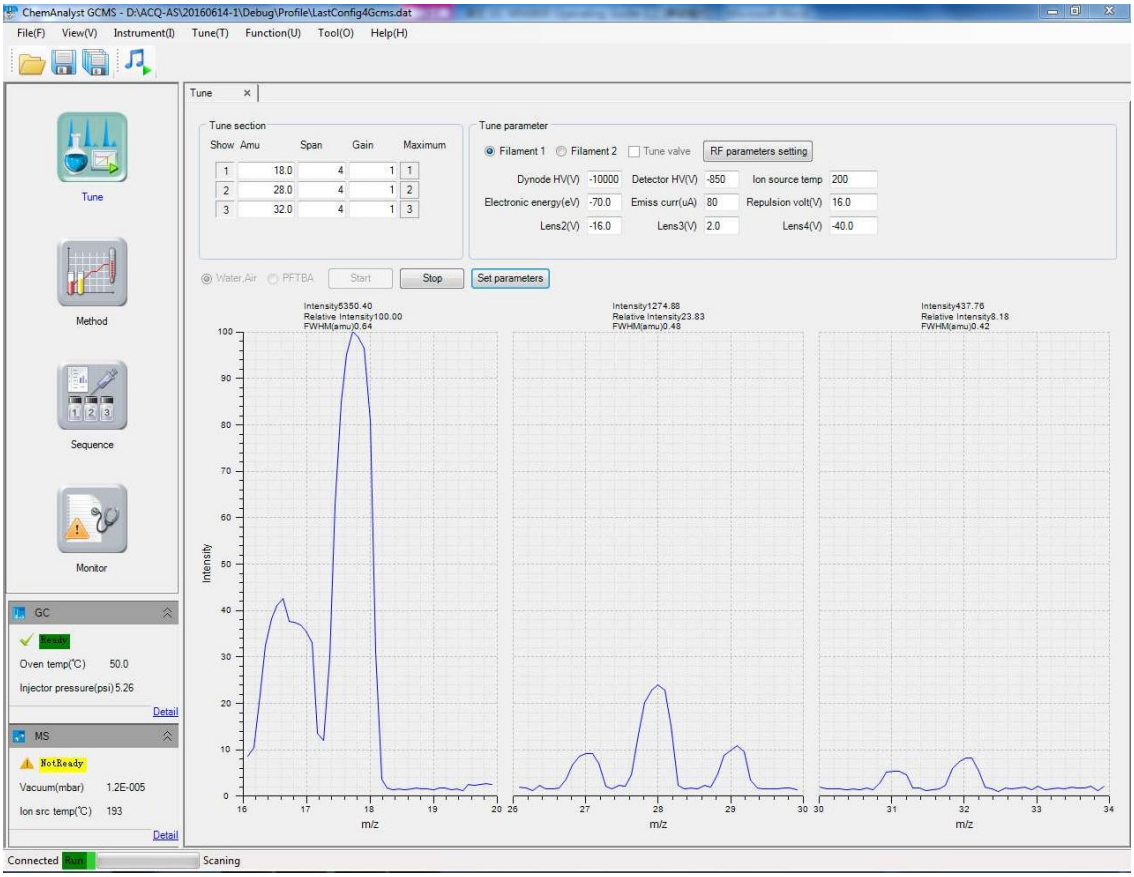


Figure 3-3 Tuning Panel

(7) If the peak intensity of the spectrum is m/z 18(H2O) ＞m/z 28(nitrogen) ＞ m/z 32(oxygen) (As shown in Figure3-3), and the nitrogen- oxygen ratio is lower than 3.7, the instrument has no gas leak.

(8) If the above mentioned conditions are not satisfied, the instrument may have a gas leak somewhere; stop scanning immediately, check for leakage points and resolve the problem; otherwise the lifetime of filaments will be severely shortened.

Possible reasons of gas leak:

(1) After removing and cleaning the ion source, whether the screws on the front panel of the ion source are appropriately tightened; whether the O-rings need to be replaced.

(2) After replacement of GC columns, whether the ferrules on either GC sample inlet or GC-MS interface are well sealed;

(3) Whether the GC inlet is tightened after replacement of GC glass liner and septum.

4. Tuning and calibrating

Tuning the instrument is to correct the instrument, adjusting various parameters of ion source, mass analyzer and detector to acquire desired resolution, sensitivity, and accurate mass axis and the correct ion abundance ratio. One of the purposes of tuning is to know about the state of the instrument and check if the instrument runs properly and the performance can meet the specifications. The second purpose is to meet the requirement of different analytical methods and to obtain the best qualitative and quantitative analytical conditions.

Tuning can be divided into automatic tuning and manual tuning. Automatic tuning means that the system will automatically adjust various parameters of the instrument based on collected peak of PFTBA's mass spectrum to enable various indicators of the instrument reach the target value. Manual tuning means that various parameters of the instrument will be adjusted manually, hence the operator is required to have a technical basis

4.1 Automatic tuning

(1)After ensuring that the instrument has no leak, click [Tune], [Automatic tuning];
(2)Open the tuning files recently used; also it can be set to always open the tuning files created during last time of automatic tuning.
(3)When [Calibrate mass number] is selected, the mass axis will be automatically calibrated during automatic tuning process.
(4)When [Create tuning reports] is selected, tuning reports will be automatically created after automatic tuning.
(5)Click [Advanced settings] to set the target mass and resolution for sensitivity tuning.

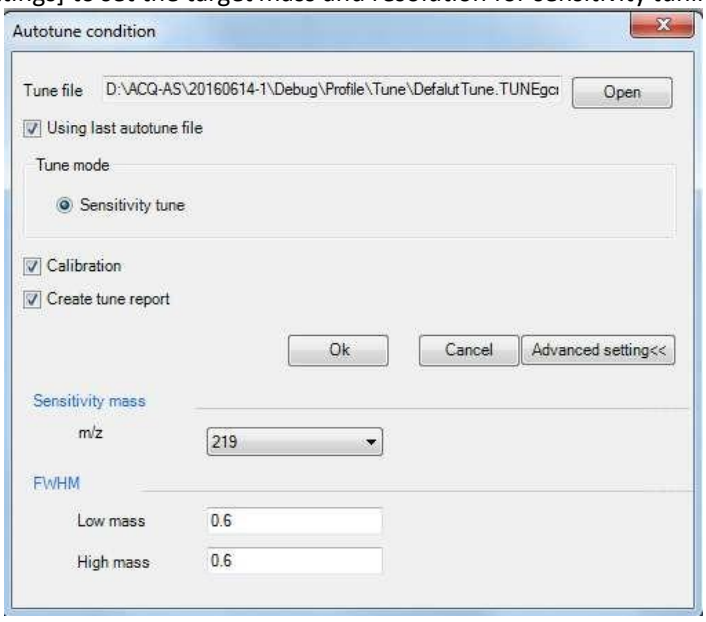


Figure 4-1 Tuning Panel

(6)When automatic tuning is finished, tuning files and tuning reports will be automatically

created.

(7)Click [File], [Automatic tuning report] and choose tuning files to create corresponding reports if needed.

4.2 Manual tuning

4.2.1 Start scanning

(1) Open a tuning file, select perfluorotributylamine mode, select filament, open the tuning valve, click [Set parameters], [Start scanning].

(2) Adjust [Delay electrode voltage], [Introduce electrode voltage], [Focus electrode voltage] in turn to achieve optimal peak intensity and ratio.

(3) Re-tuning is required when switching the filament.

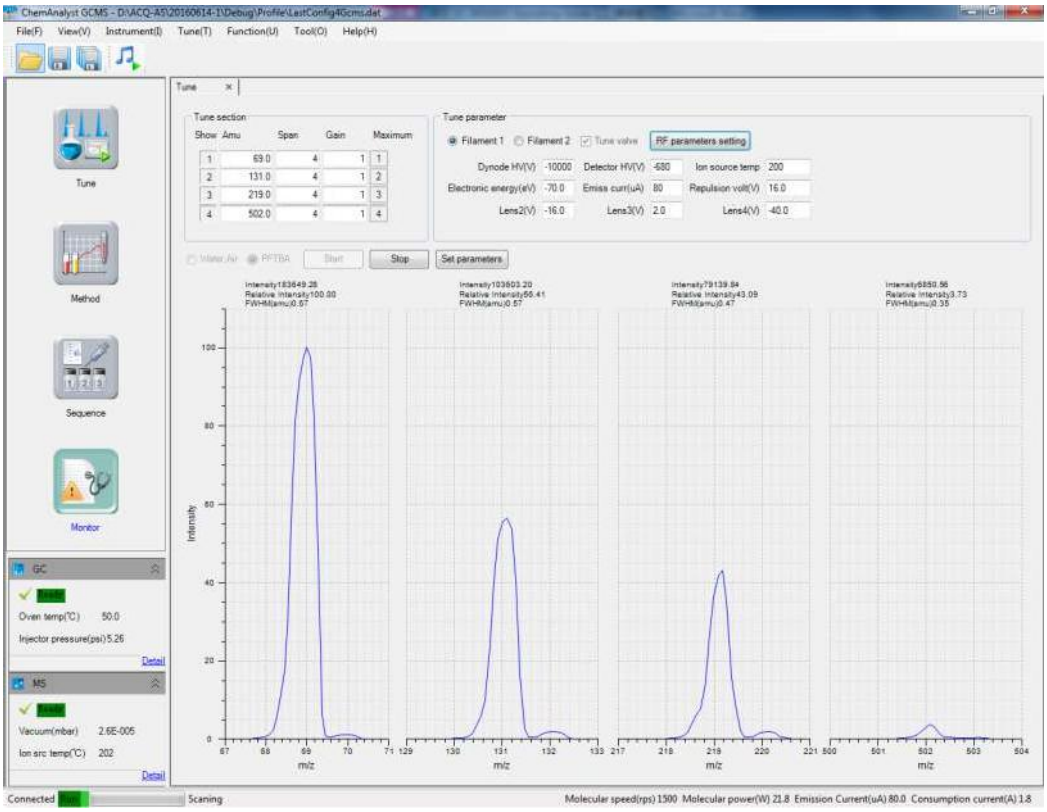


Figure 4-2 Tuning Panel

4.2.2 Adjust resolution

(1) Click [Initialize RF parameters], as shown in Figure 4-3.

(2) Click [Parameter M] to set the offset value. The larger the value is, the larger the full width at half maximum (FWHM) of MS peaks is and the worse the resolution is. The smaller the value is, the better the resolution is.

(3) Click [Parameter R] to set the offset value. This value has a stronger influence on the FWHM of high mass peaks than low mass peaks.

(4) After setting completed, click [OK] to observe changes in peak shape.

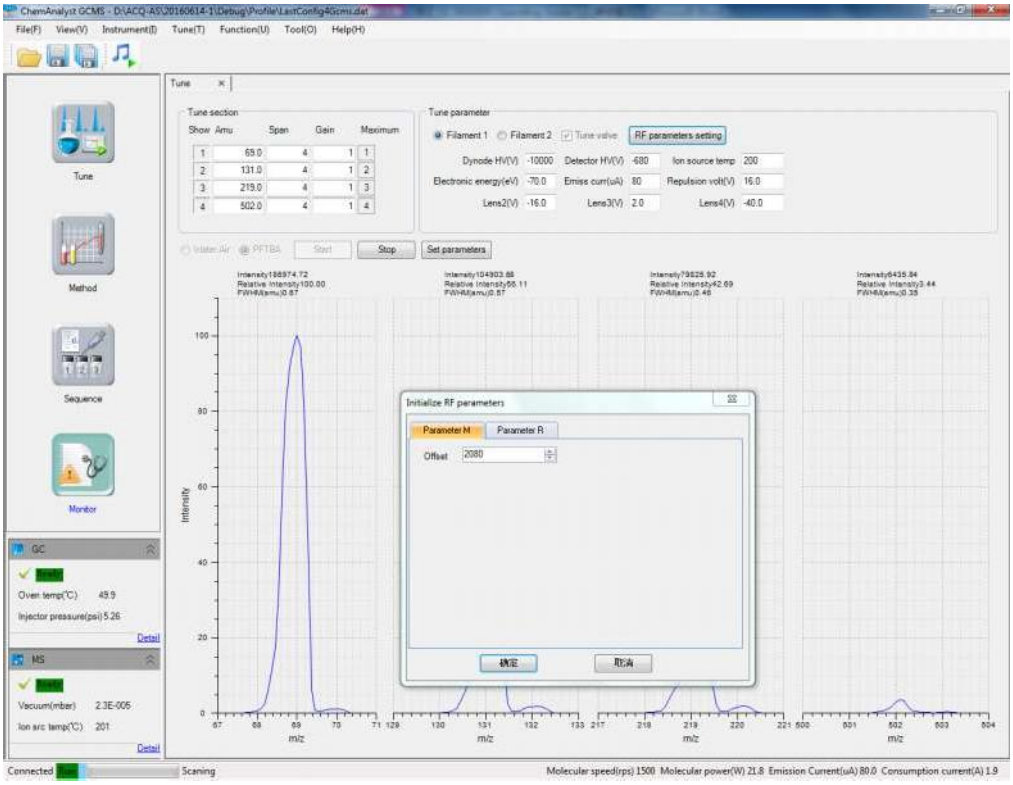


Figure 4-3 RF Parameter Setting Panel

4.3 Calibrate mass number

(1) Click [Tune], [Automatic tuning] to open the mass number calibrating page.
(2) Click [Add] to add the corresponding numbers of peaks to be calibrated.
(3) Enter the number 69, 131, 219 and 502 in turn in the [Calibrate peak] column.
(4) Start [Tune valve].
(5) After starting acquisition, click the left calibrating peak and the corresponding mass spectrum peak will be displayed in the right mass axis calibrating box.
(6) Click [Center], the mass spectrum peak will be centered automatically by the system, and the location data of the peak will be recorded.
(7) After centering each calibrating peak, click [Calibrate] to calibrate the mass axis.
(8) Save the tuning file, and manually tuning results can be saved at the same time.

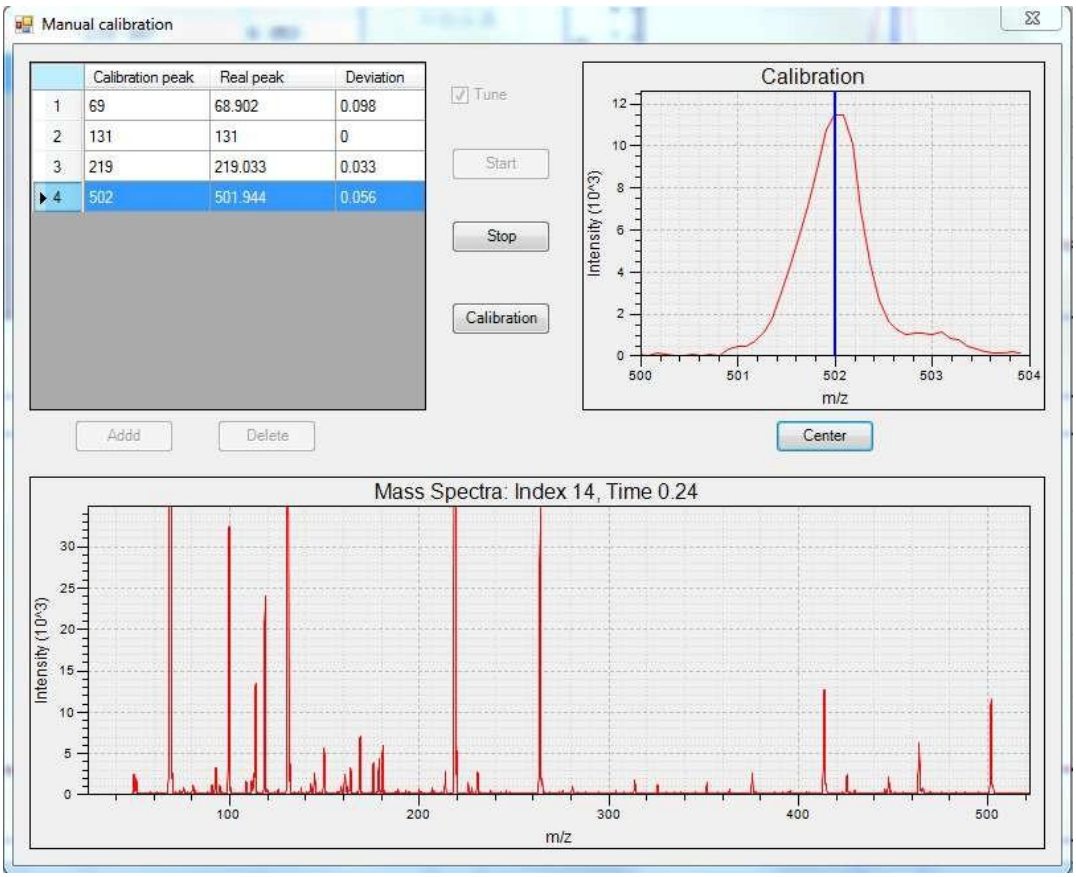


Figure 4-4 Mass Axis Calibration Panel

5. Method Setting

5.1 Set GC method

5.1.1 Set method on GC control panel

Figure 5-1 GC Control Panel

(1) Set the inlet temperature: [Inlet] – [Settings], input the temperature value you want to set, [Enter].

(2) Set the chromatographic column parameters: [Chromatographic Column] – [Settings], input the chromatographic column parameters, [Enter].

(3) Set the warming program of the oven: [Oven] – [Settings], input the temperature value you want to set, [Enter], use the  or  to set the warming rate and holding time.

(4) Set the temperature of the GC-MS interface: [Auxiliary temperature] – [Settings], input the temperature value you want to set, [Set].

5.1.2 Set GC Method from the software “ChemAnalyst”

(1) Click [Method] to go to the GC method setting page, as shown in Figure 5-2.

(2) Click [Retrieve from GC] to get the set value and real-time values from GC.

(3) Set related parameters.

(4) Click [Download to GC], the set value on the current page can be downloaded to GC.

(5) Click [Retrieve all], all set value on GC panel can be received by the software.

(6) Click [Download all], all set value on GC panel can be r downloaded to GC.

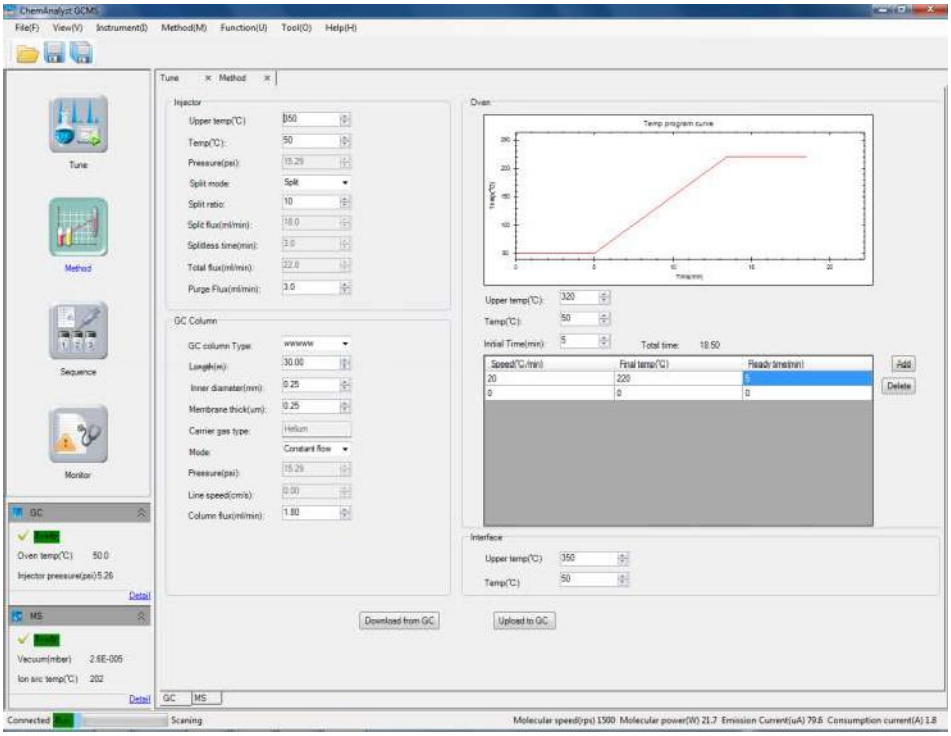


Figure 5-2 GC Method Setting Panel

5.2 Set MS method

(1) Click [MS Settings] to go to MS settings page, as shown in Figure 5-3.

(2) [Customize tuning file] to load the tuning file used by MS method.

(3) Set the ion source temperature, emission current and detector voltage.

(4) Only after a certain period time will the ion source temperature be stable, so set the ion source temperature before clicking [Download to MS] in order to let the ion source temperature reach the set value in advance.

(5) The detector voltage could be the tuning voltage plus 300V, for example: if the detector voltage is -700V during tuning process, the detector voltage can be set to -1000V; and the specific voltage value should be determined based on the concentration and response value of the sample.

(6) Set the Solvent Cut Time; the Solvent Cut Time refers to the time that the solvent of sample takes to flow to MS from GC. During the Solvent Cut Time MS would turn off the filament, RF power supply and detector HV to avoid the possible damage to MS parts caused by the change of vacuum degree resulted from the solvent into MS.

(7) Set Scanning Duration.

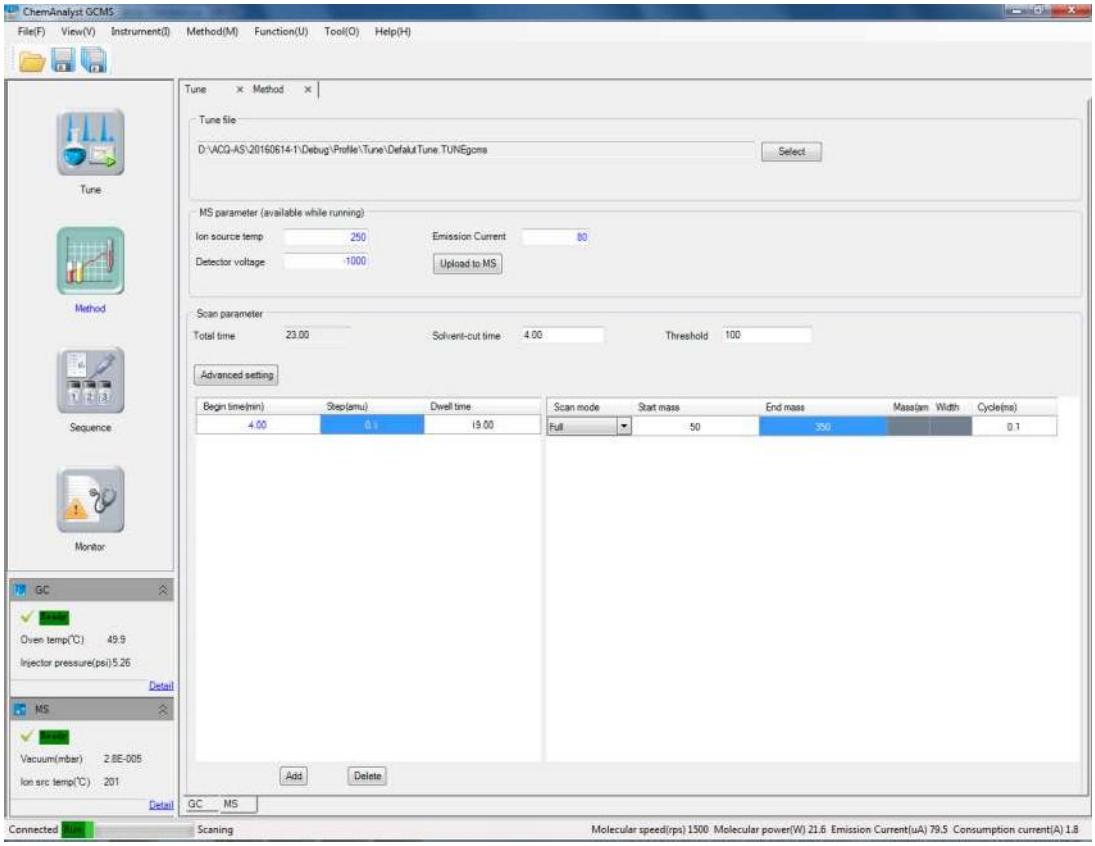


Figure 5-3 MS Settings Panel

5.2.1 Full Scan Mode

(1) [Duration] plus [Solvent Cut Time] equals the total scanning time.

(2) Select [Full] from [Scan Mode], set the mass range for scanning, dwell time for each point is as the default value.

Figure 4-4 Full Scan

5.2.2 Selected Ion Monitoring (SIM) Scan Mode

(1) Add or delete SIM segment with [Add] or [Delete].

(2) Modify the duration of each SIM.

(3) Set the scan mode as SIM, set the mass number. Default value of scan width is

1. Default value of dwell time for each point is 4. As shown in Figure 5-5.

Begin time(min)	Step(amu)	Dwell time
4.00	0.1	3.00
7.00	0.1	4.00
11.00	0.1	5.00

Scan mode	Start mass	End mass	Mass(am	Width	Cycle(ms)
SIM			77	1	4
SIM			135	1	4
SIM			163	1	4
SIM			194	1	4
SIM			0	0	0
SIM			0	0	0
SIM			0	0	0
SIM			0	0	0
SIM			0	0	0
SIM			0	0	0
SIM			0	0	0
SIM			0	0	0
SIM			0	0	0
SIM			0	0	0
SIM			0	0	0

Figure 5-5 SIM Scan Mode Settings

(4) The time used for one time SIM scanning T(ms)= Width10 *4* the number of mass number; under the conditions set in Figure 5-5, T=1*10*4*3=120ms, i.e. it can scan 8.3 times in 1 second.

5.2.3 Advanced settings

(1) Click [Advanced Settings], click the advanced function during acquisition, as shown in Figure 5-6.

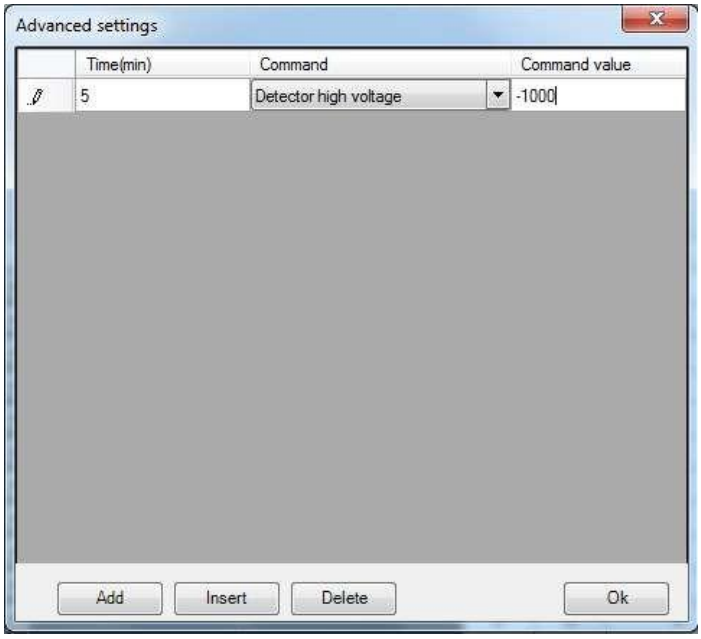


Figure 5-6

(2) Set open/close and target value of the filament, tuning valve, detector HV, dynode HV in the dialog box of advanced settings.

(3) Figure 5-6 implies that the detector HV will switch to -1000v from the fifth minute of sequence acquisition.

5.2.4 Save Method

(1) Click [File], [Save Method File].

(2) Enter the Method name and save.

6. Sequence

6.1 Sequence Settings

(1) Click [Sequence] to switch to sequence acquisition window, as shown in Figure 6-1.
(2) Add sequences.
(3) Set the data file information and sample information.
(4) Click the Start Acquisition button on the toolbar to be ready for acquisition.
(5) If GC and MS are [Not Ready], the sequence will be waiting and not enter the waiting-for-injection status until GC and MS are Ready.
(6) After entering the waiting-for-injection status and injection completed, press the [Start] button on GC panel to start sequence acquisition.
(7) If the instrument is running with air or performing some tests of less repeatability requirement for injection, the operator may press the [Start] button on GC panel to force to start sequence acquisition.

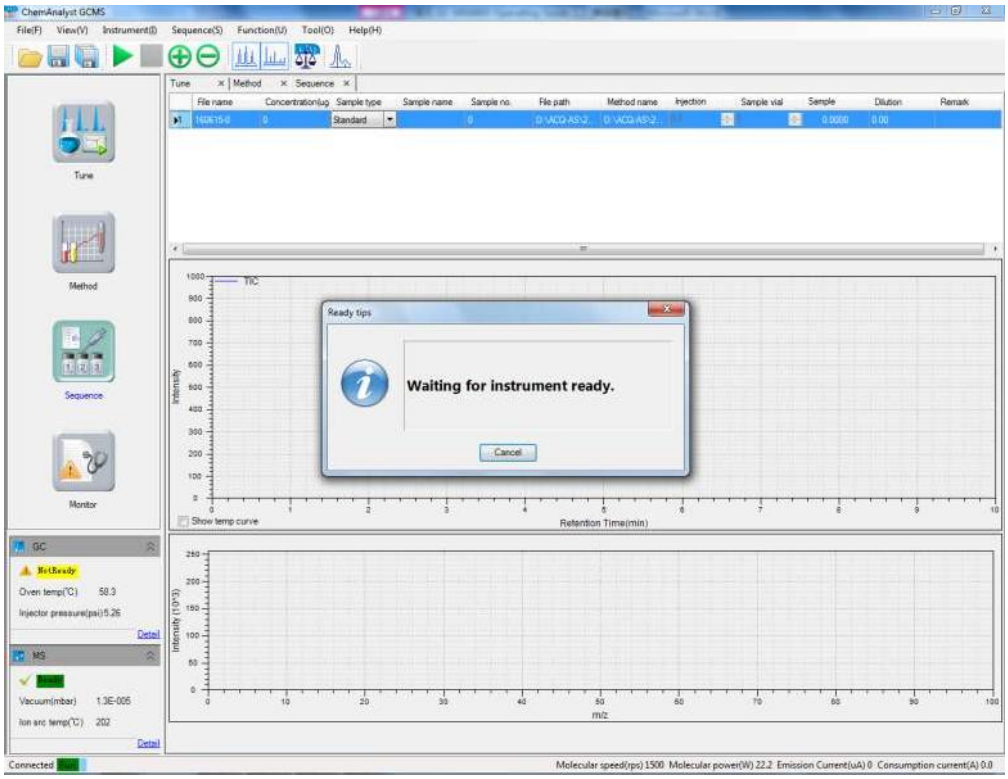


Figure 6-1 Sequence Feedback Panel

6.2 Sample injection and data acquisition

(1) As shown in Figure 6-2, use a syringe to extract 1μL of sample, quickly insert the syringe into the inlet to the bottom, quickly inject the sample into GC and pull out the syringe quickly.

Figure 6-2 Sample Injection

(2) As shown in Figure 6-3, after quickly pulling out the syringe, immediately press the [Start] button on GC control panel to start data acquisition.

Figure 6-3 Start data acquisition

7. Data Processing

Double-click the icon of the data analysis software 

on the desktop of the computer with the left mouse button, start running the data processing software.

7.1 Function introduction of the menu icon on the toolbar

Figure 7-1 Toolbar

(1)  The button for opening files, used to open data files.

(2)  The button for saving component data, used to save component data.

(3)  Printing button, used to open the print-report window.

(4)  Hiding button, used to hide all open windows. But the directory note will not be deleted.

(5)  Closing current files, click it to close current windows, and the corresponding directory note will be deleted.

(6) See Section 7.4 for functions of other icons.

7.2 Function introduction of [Option] on the menu bar

(1) Mass range, refers to the width on the left and right of the corresponding mass number when extracting MC diagram. For example, set the mass range as 0.5 when extracting MC diagram of 100 mass number, the corresponding mass number on the generated MC diagram will be 99.5-100.5.

(2) Smoothing points: refer to the points used by the software to smooth the chromatographic peak; 5, 7, 9 and 11 points may be selected, and if 0 point is selected, no smoothing will be carried out.

(3) Decimal places displayed for mass number: the digits after the decimal point of the mass number displayed by the chromatographic peak; 1 represents one digits after the decimal point will be displayed.

(4) Chromatogram Annotation: Chromatogram Note can be set, including the color, font size, forms of separators for Annotation, and the prefix of Annotation.

7.3 Qualitative Analysis

7.3.1 Compare the Retention Time

(1) Click the  icon on the menu toolbar to open the standard and sample chromatograms and compare the retention times. If they are the same or similar, the sample peak may be the target peak, and then compare its mass spectrogram with the standard mass spectrogram, as shown in Figure 7-2.

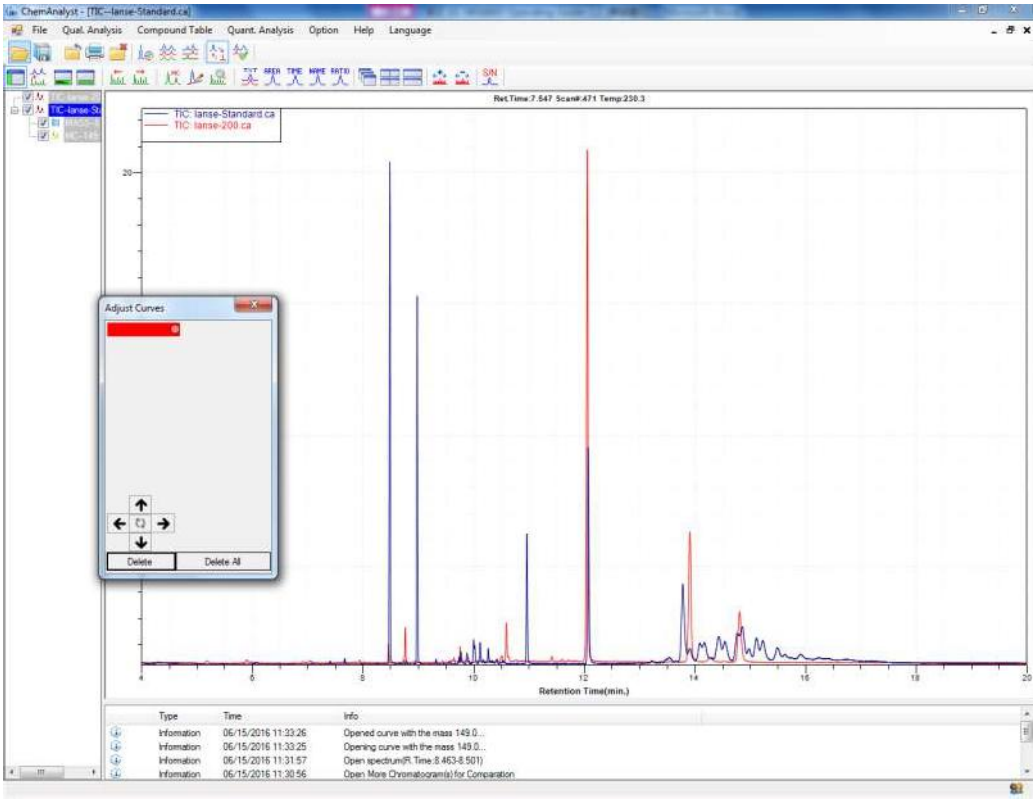
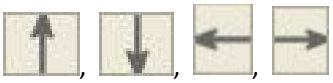


Figure 7-2 Retention Time Comparison

(2) Use arrows in the dialog box of [Adjust and Contrast chromatography] to adjust the positions of the chromatogram:

 , Means to move the selected curve upward.  Means to restore the size and position of the selected curve.

 After selecting a curve, click this button to delete the currently selected curve.

 Click this button to delete all the curves.

7.3.2 Check Mass Chromatogram

After selecting  , click the  icon and input the characteristic ion of the

target compound in the below interface popped up, click OK to check the Mass Chromatogram (MC) of the ion, as shown in Figure 7-3, 7-4.

Figure 7-3 Search the Characteristic Ion

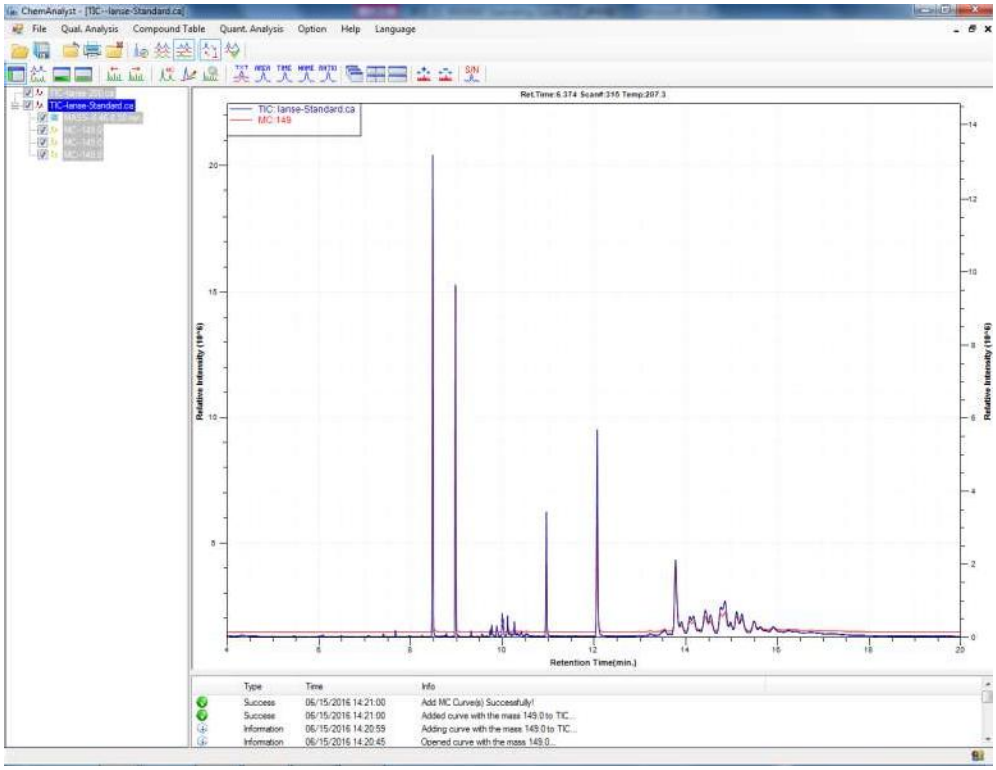


Figure 7-4 MC Displayed

If the shape of peak on MC diagram is similar with the chromatographic peak, and the retention times are close, that peak might be the target peak; determine its nature through its mass spectrogram.

7.3.3 Check Mass Spectrogram

Press the Shift key and hold down, click with the left mouse button on any point of the mass spectrogram, and the mass chromatogram of that point will be displayed, as shown in Figure 7-5.

Press the Shift key and hold down, click with the left mouse button and drag a segment, and the mean mass chromatogram of that segment will be displayed.

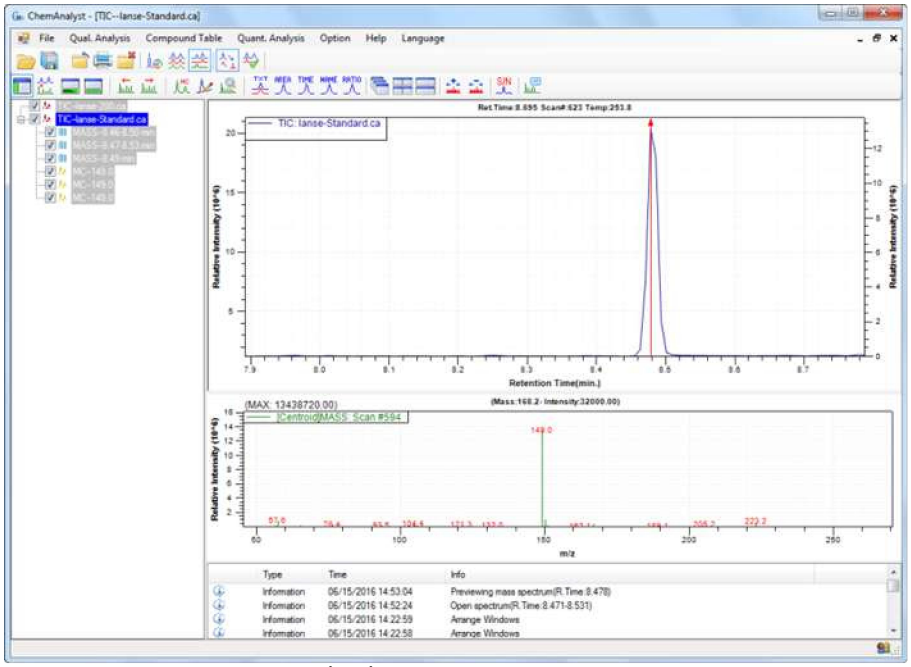


Figure 7-5 Check Mass Spectrogram

7.3.4 Remove Background

(3) Click the  icon on the toolbar, press and hold the left mouse button to select a segment of straight baseline on the left or right side of the target chromatographic peak as the mean noise level, as shown in Figure 7-6.

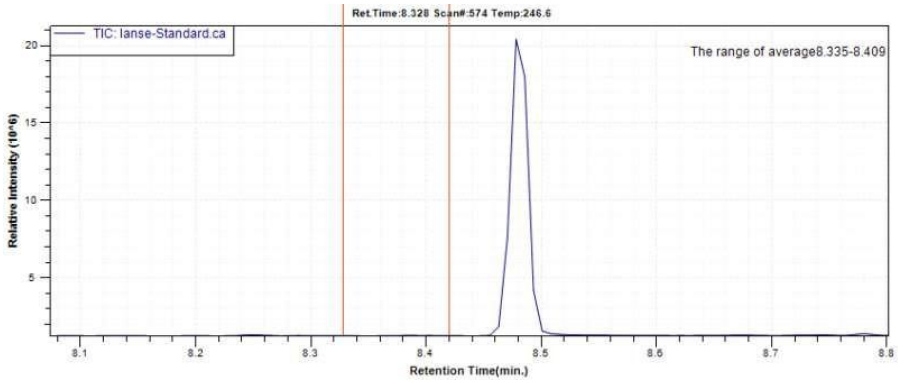


Figure 7-6 Select Noise Range

(4) Click the  icon on the toolbar, press and hold the left mouse button to select the range of the chromatographic peak for removing background, as shown in Figure 7-7.

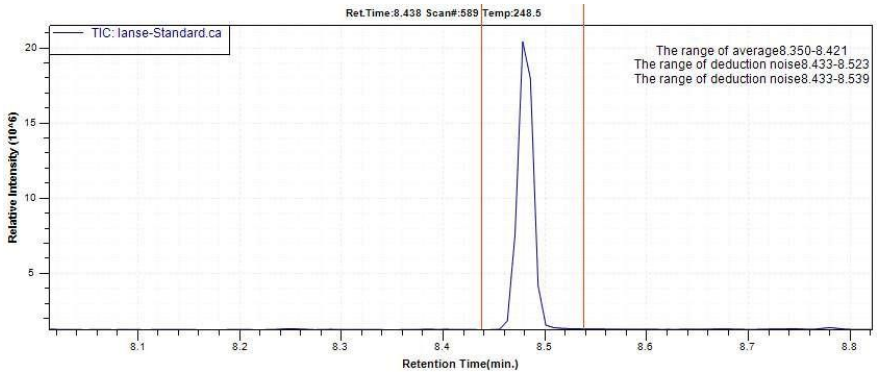


Figure 7-7 Range for Removing Background

(5) Press the Shift key and hold down, click the mass spectrogram or hold the left mouse button and drag for a short distance, the mass chromatogram without background will be seen, as shown in Figure 7-8.

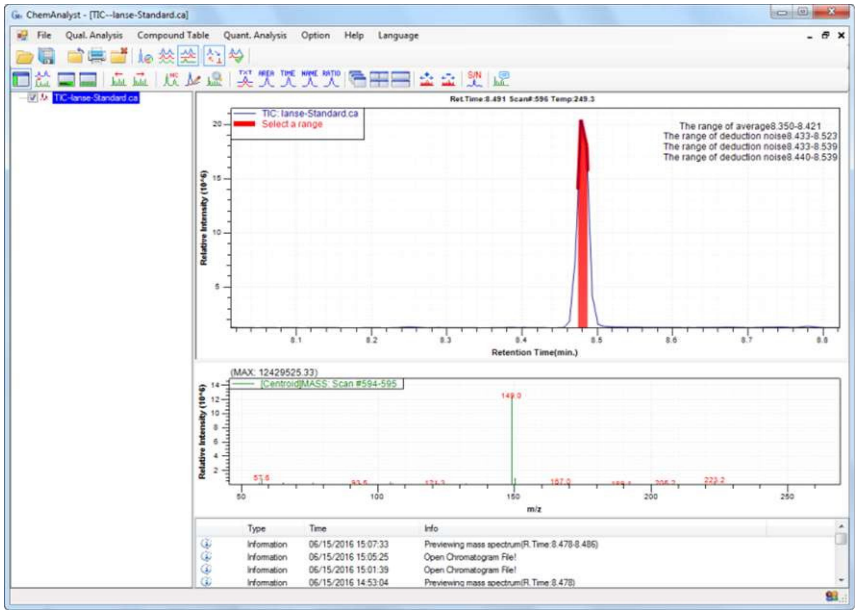


Figure 7-8 Check Average Spectrum

7.3.5 Spectral Library Search

(6) Click the right mouse button on the centroidal mass spectrogram, select [Search from NIST and Preview…] and preview the search results, as shown in Figure 7-9.

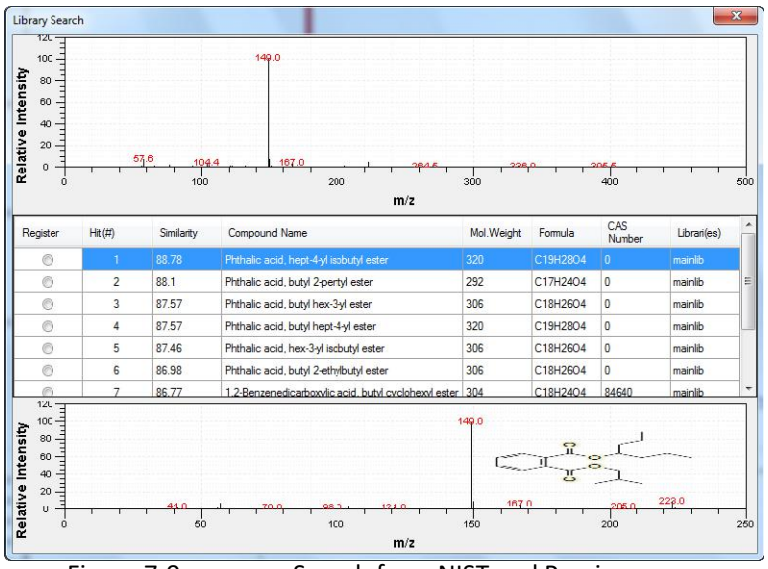


Figure 7-9 Search from NIST and Preview

(7) Click the right mouse button on the mass spectrogram or integral component and select [Register to Qualitative Table]. Click the  icon to open the Qualitative Table where batch searching from NIST library can be performed. Double-click to check the search results. As shown in the below figure.

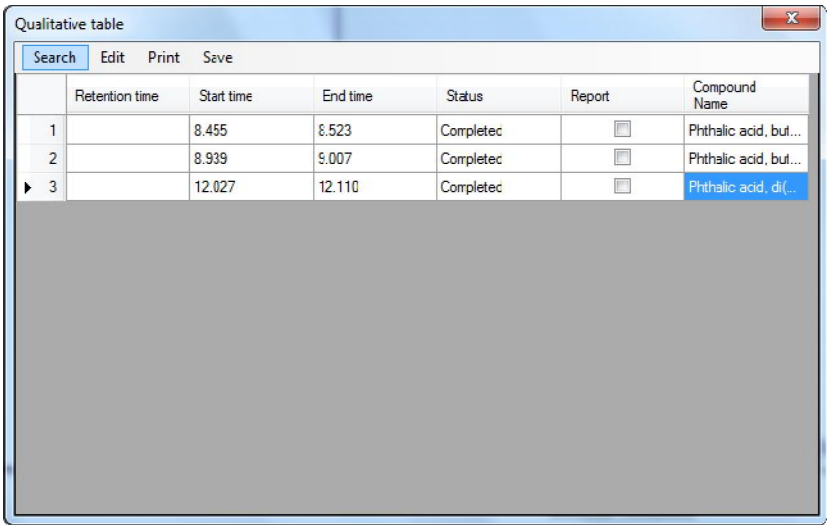


Figure 7-10 Search Table Preview

7.3.6 Index Search

(8) Click [Qualitative Analysis], [Index Search] to open the dialog box for selecting index database, as shown in Figure 7-11.

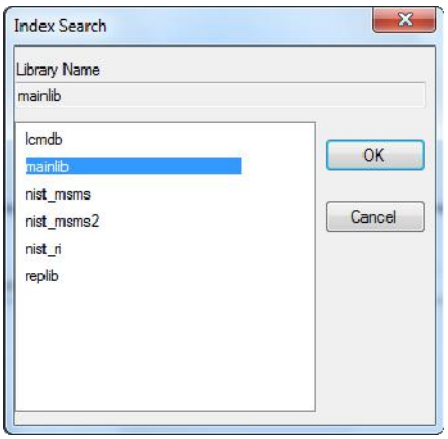


Figure 7-11 Index Search

(9) Select an index database.

(10) Conduct search according to [Name and Synonym], [Molecular Weight], [Chemical Formula], [CAS Number] or [NIST Library Number] in the dialog box of [Edit Database]; as shown in the below Figure, search the matter with CAS Number 64-17-5.

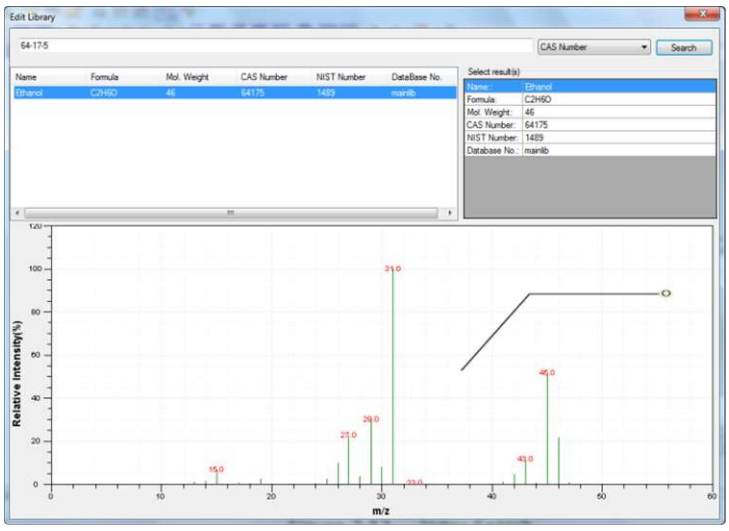


Figure 7-12 Index Search

7.4 Quantitative Analysis

: Integration toolbar, as shown in Figure 7-13.

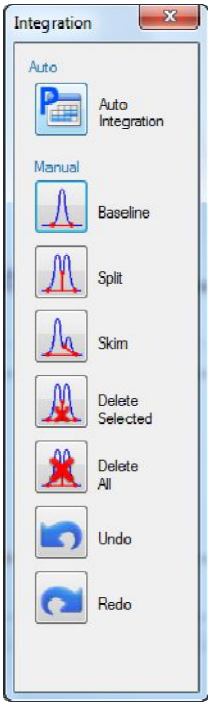


Figure 7-13 Integration Toolbar

7.4.1 Automatic Integration

(11) Click the  icon, and users can set Automatic Integration parameters in the dialog box as required, as shown in Figure7-14.

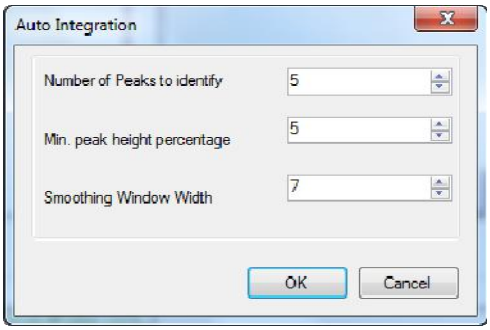


Figure 7-14 Parameter Settings for Automatic Integration

7.4.2 Manual Integration

(12) Click the baseline icon. 

(13) Click the start point and end point of chromatogram peak respectively, as shown in Figure 7-15.

(14) The corresponding component information will be displayed below the chromatogram.

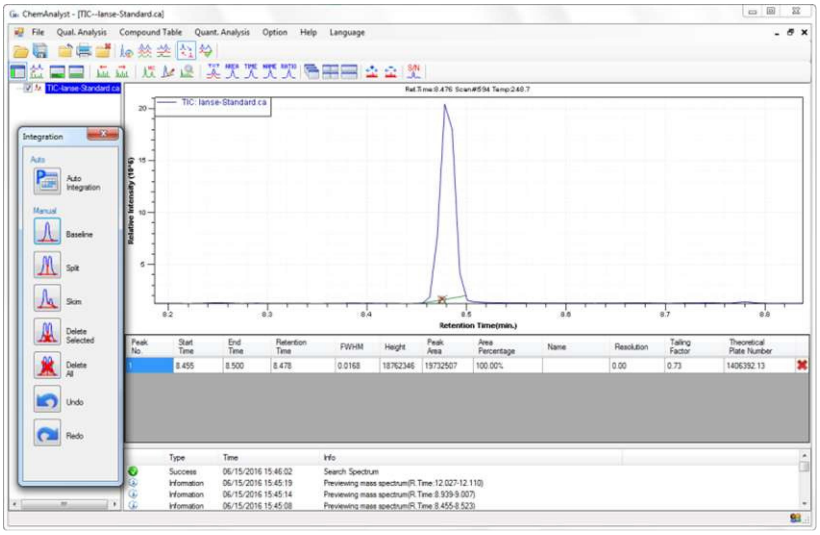


Figure 7-15 Manual Integration

The following is the introduction of other icons on the ***Integration*** toolbar:


Split the chromatogram which cannot be completely separated.


Slash shoulder peak and perform integration.


Delete integration of selected chromatographic peak: click the icon and the selected integration will be deleted.


Delete all integration data and components data.


Cancel the previous operation.


Redo the cancelled operation.

(1) Save integration data: [Component Table], [Save Component Table]
(2) Import integration results to Excel: [Component Table], [Import Component Data to Excel].
(3) Export integration results: [Component Table], [Export Component Data].
(4) Import integration results to current program: [Component Table], [Import Component Table].

7.4.3 Quantitative Analysis -External Standard Method

(1) Click [Quantitative Analysis], [Quantitative Analysis Guide], a window will pop up as shown in Figure7-16.

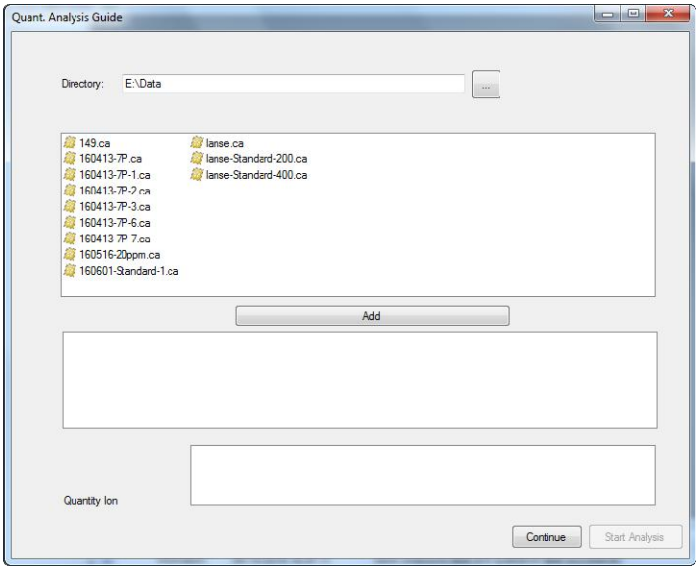


Figure 7-16 Quantitative Analysis Guide

(2)Select a data file and click [Add] to add it to the below textbox.

(3)Select a data file in the below textbox, press the Delete key on the keyboard to delete the selected file.

(4)Under the Quantitative mode, TIC or corresponding quantitative ions can be selected in the [Quantitative Ion] box.

(5)Click [Continue] and a window will pops up as shown in Figure 7-17.

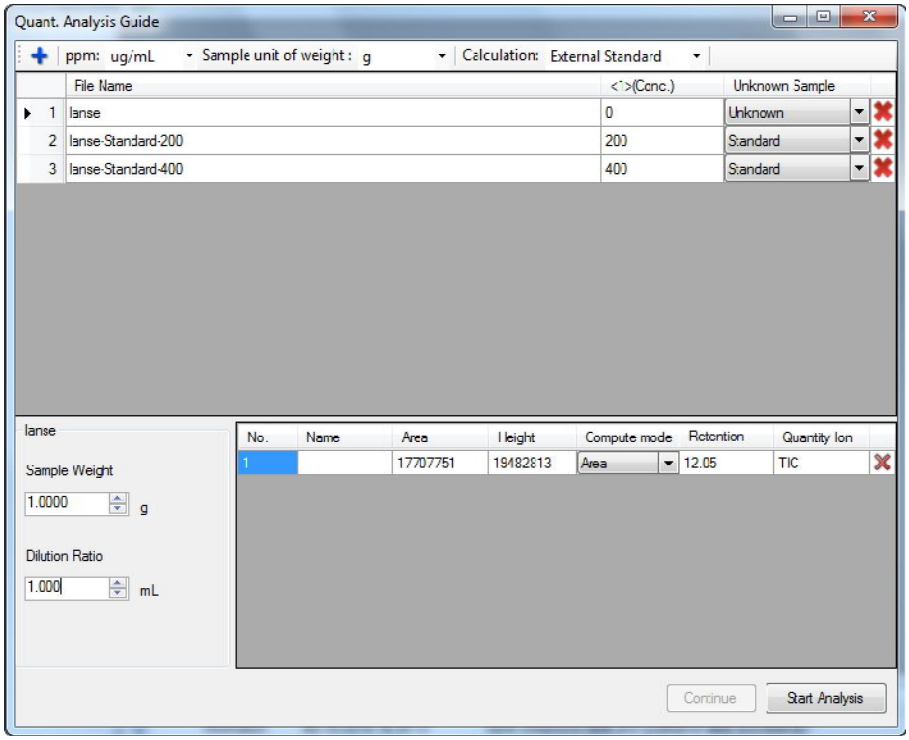


Figure 7-17 Quantitative Analysis Guide

(6)Set the concentration and unit of weight for the standard sample.

(7)[Calculating Method], select [External Standard].

(8)[Sample Type], select [Standard] or [Unknown].

(9)Enter [Sample Weight], [Dilution Ratio].

(10)Click [Start Analysis], and the standard curve window will pop up, as shown in Figure 7-18.

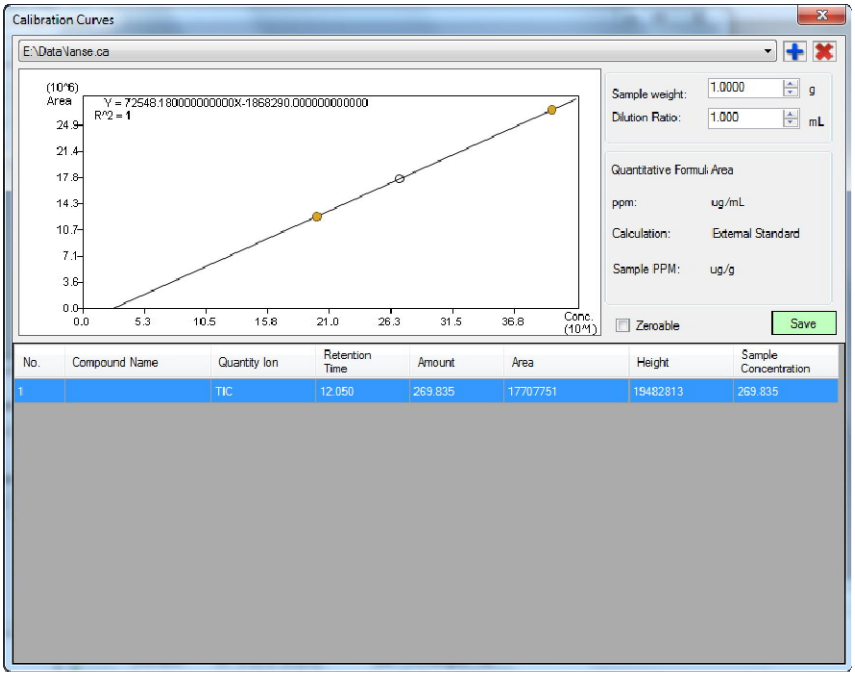


Figure 7-18 the Curve- External Standard Method

(11)Click  icon on the upper right corner of the window, the unknown sample can be added to the standard curve for analysis.

(12)Click the yellow filled dot  on the standard curve, and when the dot becomes to  , that dot will not be involved in quantitative calculation.

(13)Click [Save] to save the results.

(14)Click [Quantitative Analysis]→[Quantitative Analysis Result] to check the quantitative analysis result.

(15)Click [OK], and the saved results can be open.

7.4.4 Quantitative Analysis -Internal Standard Method

(1)When applying Internal Standard Method, the steps to add the data files are the same to that of External Standard Method.

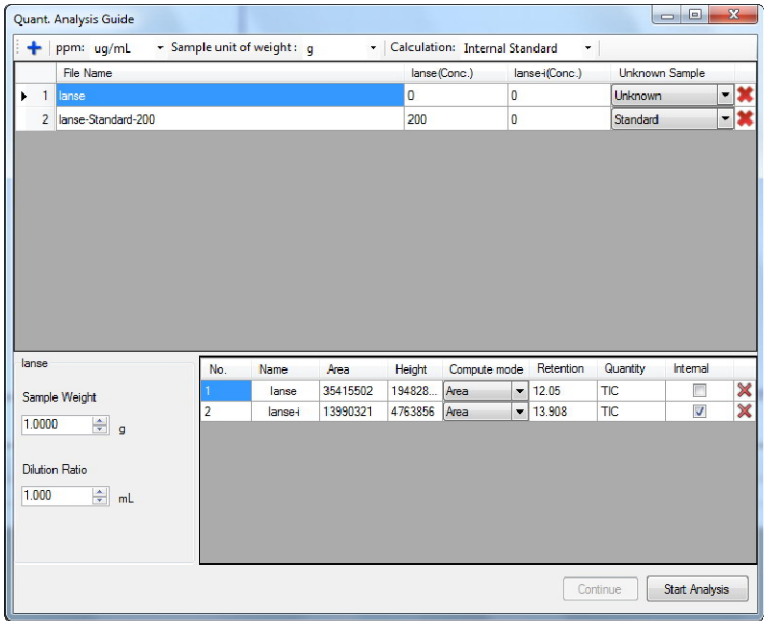


Figure 7-17 Internal Standard Method

(2)[Calculating Method], select [Internal Standard].

(3)Select [Internal Standard Matter], and input the concentration of non-internal-standard matter in the standard sample.

(4)Click [Start Calculating], and the result is as shown in the below figure.

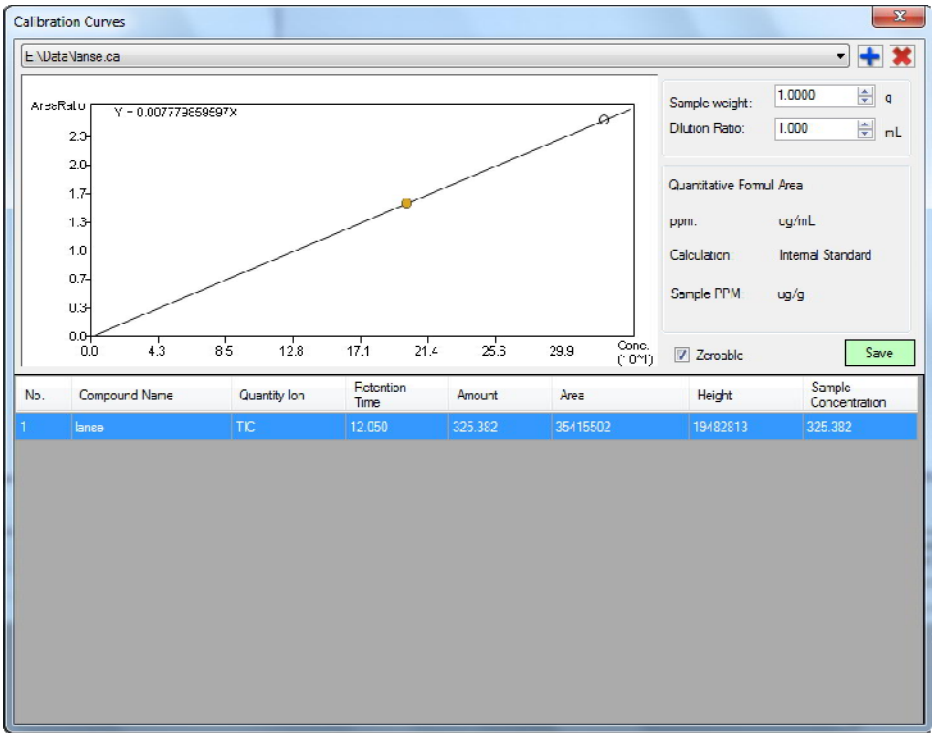


Figure 7-20 the Curve- Internal Standard Method

7.5 Functions of other icons

(1)  Comparison of mass chromatograms: if the button is selected, the newly opened mass chromatogram will be added to current TIC diagram directly. If the button is not selected, the newly opened mass chromatogram will be shown separately in a new window.

(2)  Spectrum list: show or hide the content of spectra list.

(3)  Centroidal: if selected, all the mass spectrogram will be automatically centered.

(4)  MC Summary: if there are several MC curves open on the TIC diagrams, with this button, all peaks including characteristic ion on TIC will be found.

(5)  Hide (or show) preview: Click the button to hide or show MS preview window. If the window is displayed, the mass spectrum will be displayed on the preview area after clicking TIC or MC; otherwise, the mass spectrum will be displayed in a new window, as shown in Figure 7-21.

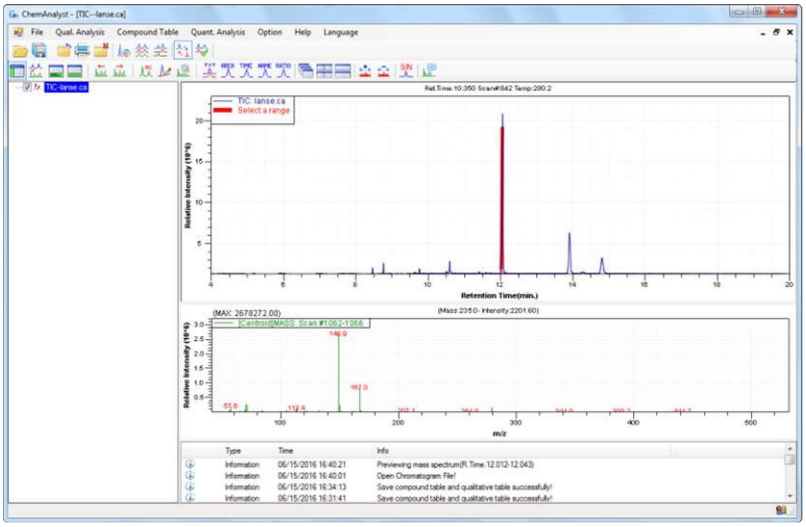


Figure7-21 Preview of Mass Spectrum

(6)  Display log: click to hide or display the log area.

(7)  Display component results: click to display or hide component table.

(8)  Display the previous mass spectrum: click to display the previous one mass spectrum according to the current retention time. The mass spectrum will be displayed in the preview window if the preview window is already open; otherwise a new window will be open with the mass spectrum in it.

(9)  Display the next mass spectrum: click to display the next one mass spectrum according to the current retention time. The way of display is similar to the above.

(10)  Annotation switch, click to switch the peak point to display the peak area.

(11) 

Annotation of area, time, name and ratio.

(12)  Arrangement of data windows: layered, vertical, horizontal.

(13) 

Signal to Noise Ratio: click the icon, select a segment of baseline as the noise range, move the mouse pointer to the peak point, and then the Signal to Noise Ratio of the peak will be displayed, as shown in Figure 7-22.

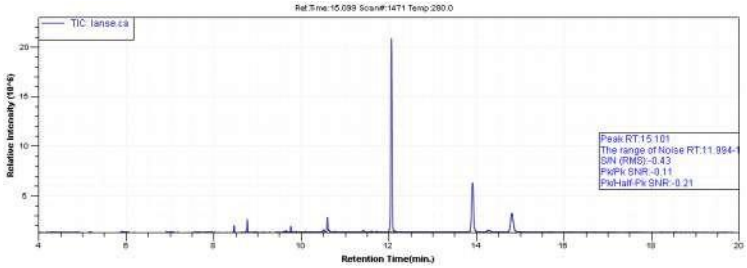


Figure 7-22 Signal to Noise Ratio Calculating

(14)Click [Help]—[Shortcuts] to check the shortcuts used in the program.

8. Analysis Report

8.1 Print Report

（1） Click  in the menu bar and enter the print interface, as shown in Figure 8-1:

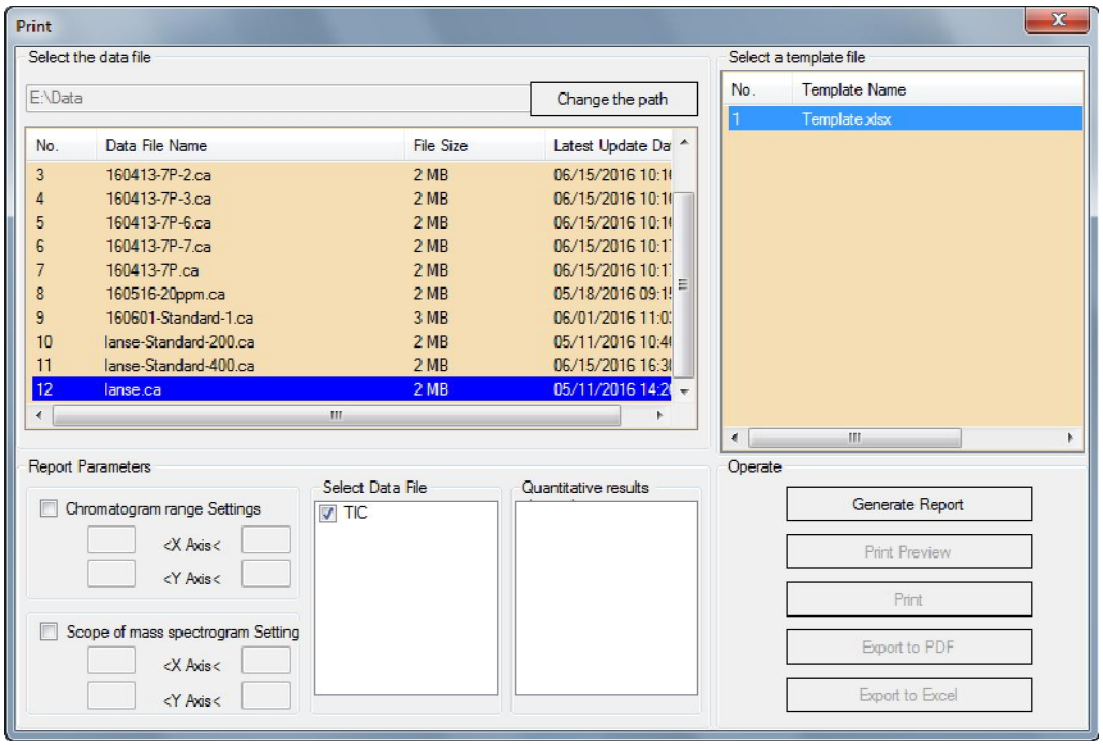
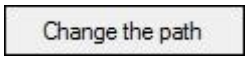


Figure 8-1 Print Report

（2） Select the data file: here you can select the data file you want to print. Click 

to change the file directory.

（3） Select a template file: here you can select a template you want to use.

（4） Report Parameters: here you can set the ranges of X- and Y-axes of the chromatogram and the mass spectrogram, the chromatographic curve and the quantitative ions.

（5） Operate: here you can generate a report; use the print preview; print; export to PDF or EXCEL.

8.2 Template Design

（1） Create a template: Create an EXCEL file under the template directory.

（2） Edit the template: click  in the menu bar and enter the print interface. Select the template you want to edit in the list of templates and then double-click

to open it; drag the template tab into EXCEL, as shown in Figure 8-2:

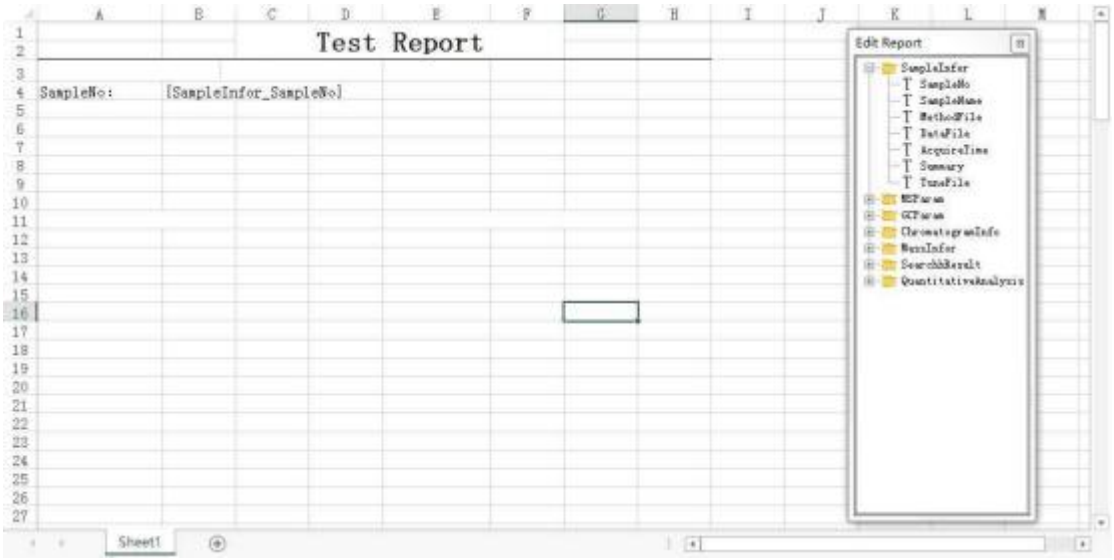


Figure 8-2 Report Template

a) SampleInfor: basic information of the sample.
b) GC Param/MS Param: basic parameters of GC and MS.
c) Chromatogram Info: the related information of chromatogram.
d) MassInfor: the related information of mass spectrogram. It is required to right-click on the mass spectrogram or integral component and choose [Register to the qualitative list] to add it.
e) Search Result: the search result from NIST database.
f) Quantitative Analysis: the result of quantitative analysis.

（3） Save the template: you can save the EXCEL file after the editing is complete, as shown in Figure 8-3:

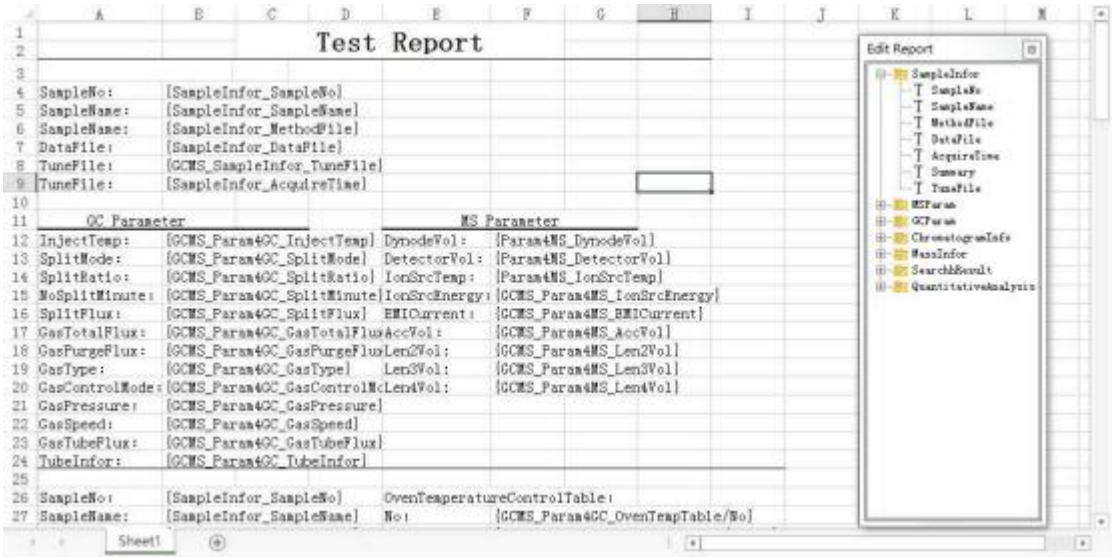


Figure 8-3 Report Template

9. Trouble Shooting

<table>
<tr><th>Trouble</th><th>Possible Reasons</th><th>Solutions</th></tr>
<tr><td rowspan="2">After GC is powered on, the main panel has no display and the oven fan has no response.</td><td>1. GC power is not well connected.</td><td>1. Check the power supply of GC.</td></tr>
<tr><td>2. Fuses of GC main power are blown.</td><td>2. Replace the fuses. Contact Drawell after-sales service department for details.</td></tr>
<tr><td rowspan="2">After MS is powered on, the red indicator of MS is off.</td><td>1. MS power is not well connected.</td><td>1.Check the power supply of MS.</td></tr>
<tr><td>2. Fuses of MS main power are blown.</td><td>2. Replace the fuses. Contact Drawell after-sales service department for details.</td></tr>
<tr><td rowspan="3">Software can not be connected with MS.</td><td>1. The network cable is not connected.</td><td>1. Check the connection of MS network cables and GC serial port cables.</td></tr>
<tr><td rowspan="2">2. The MS-side Ethernet port crashes.</td><td>2. If the vacuum is not started, power off and restart GC and MS.</td></tr>
<tr><td>3. If the vacuum is started, execute the cmd command to restart. Steps as follow:
A． Input telnet 10.80.120.182 in [Run] toolbar (as shown in Figure1) and click OK.
 

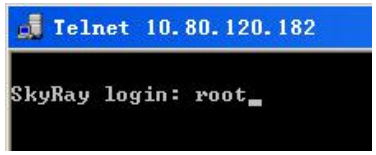
Figure 1
B． Input root in the pop-up dialog box (as shown in Figure2), then input password Drawell and press Enter.
 

Figure 2
C. Input reboot (as shown in Figure3), and press Enter. Wait for 30s.</td></tr>
</table>

		 Figure 3 D. Reconnect the instrument.
		4. Contact Drawell after-sales service department if problem is still not solved with the above solutions.
The ion source can not be heated.	1. The heating assembly of the ion source is not well installed.	1. Re-install the heating assembly of the ion source.
	2. The heating rod is damaged.	2. Contact Drawell after-sales service department.
The temperature of the ion source is displayed as a negative value.	The temperature sensor of the heating assembly is short-circuited.	Adjust the wires of the heating assembly.
The intensity of peak fluctuates too much while tuning.	1. Filament aging leads to fluctuations of emission of electron flow.	1. Check whether the fluctuation of the emission current is over 2μA on the instrument status feedback bar in the software.
		2. If it is the first time acquisition after maintenance of the ion source, the deviation of the mounting position of the ion source electrode box may cause the fluctuation. In this case, re-install the electrode box to solve the problem.
		3. Replace the filament.
	2.Other reasons	Contact Drawell after-sales service department.
There is no PFTBA peak while tuning.	1. Short of tuned liquid.	1. Open the panel of tuned liquid on MS to check its amount.
		2. If the tuned liquid runs out, please contact Drawell after-sales service department.
	2.Other reasons	Please contact Drawell after-sales service department.

Appendix 1: PFTBA Mass Fragment Information Table

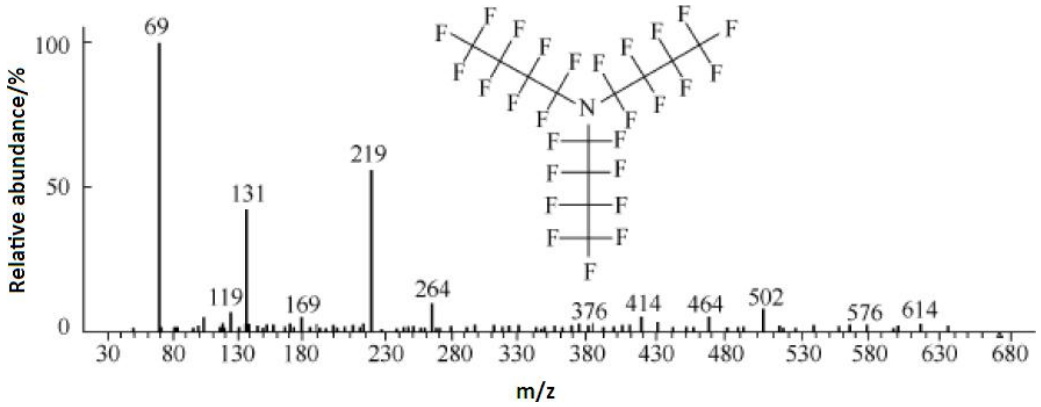


Figure 10-1 Standard Mass Spectrum of

PFTBA/FC43 Table 10-1 MS Data of PFTBA/FC43

Amu	Relative Intensity	Amu	Relative Intensity
49.99379	0.73	218.9856	38.07
68.99518	100	225.9903	0.56
92.99518	0.56	230.9856	0.47
99.99358	6.31	263.98705	8.38
113.99669	2.34	313.98386	0.32
118.99199	7.8	325.98386	0.16
130.99199	36.31	375.98067	0.38
149.99039	1.29	413.97748	1.64
163.9935	0.56	425.97748	0.85
168.98877	3.24	463.97429	1.02
175.9935	0.91	501.9711	1.95
180.98877	1.31	575.96796	0.32
213.9903	0.76	613.96471	0.64

Drawell International Technology Limited
Chongqing Drawell Instrument Co., Ltd.
Shanghai Drawell Scientific Instrument Co.,Ltd.

Chongqing Center : Suite 2705,Building No.12,Shiyou Road No.1, Yuzhong District, Chongqing, China.
Shanghai Office : Suite 1117,Lane561 XiuChuan Rd.,PuDong New Area,Shanghai,China
Homepage : www.drawell.com.cn
Tel : 0086-023-63268643
Email : sales02@drawell.com.cn