

Gas Chromatograph-Mass Spectrometer

DW-GCMS-II

Operation Guide

(Ver. 1.2)



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General

This Operation Guide describes the procedures for properly operating and maintaining the DW-GCMS-II Gas Chromatograph/Mass Spectrometer. The operator of the instrument should carefully read and 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.

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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 result. Should solvent get into the eyes, immediately flush with large amounts of water and seek medical attention.

Warning:

Do not place solvents near PCs, printers or other instruments, as fire or instrument damage may result.

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. This precaution is required by law for flammable gases.
2. Please note that the temperature of the gas cylinder normally don't exceed 40 °C. 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. The fire extinguisher is 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. 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 our 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.

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1. Introduction

1.1 Some Abbrs.

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

List 1 Abbr.

| <i>Abbr.</i> | <i>Def.</i> |
|--------------|------------------------------------|
| 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 DW-GCMS-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, injection port, 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, injection port 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 are not limited to:

- Injection port
- 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 a fire hazard, keep the mechanical pump and the surrounding environment clean.

Mark

The user must comply with the manual or the *Warning* on the instrument for either operating, or maintenance or repairing of this instrument. Disobeying these precautions violates the related safety standards and correct use of instruments. Drawell Scientific 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 take 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

2.1 Outer Configuration Requirements

2.1.1 Physical Characteristics

The whole package of DW-GCMS-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

Helium cylinder pressure and volume: purity $\geq 99.999\%$, 15MPa;

Regulator--- helium or oxygen valve (output range is 0.5~0.9MPa, no leakage).

2.1.3 Instrument working environment requirements

Table 2-1 Instrument Working Environmental requirements

| Influencing Factor | Unit | Normal operating condition |
|--|------|----------------------------|
| Ambient temperature | °C | 15~30 |
| Relative humidity | % | ≤ 80 |
| 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 $\pm 10\%$ |
| Power frequency | Hz | Rated frequency $\pm 1\%$ |

Warning

The solvent used by GCMS is flammable and toxic, which should be placed in a adequately ventilated room, or will easily cause poisoning or fire.

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 shorten the life of the instrument.

2.2 Installation&Connection

Related materials

| Part name | Part No. | Application |
|-------------------------|------------|---|
| Plug power cord | 3691000075 | Connect MS to AC power |
| Plug power cord | 3691000075 | Connect GC to AC power |
| Computer Cables | 3694000012 | Connect the mechanical pump to MS |
| Bellows | 304742411 | Connect MS outlet to the mechanical pump |
| Corrugated plastic pipe | 304742407 | Exhaust the mechanical pump waste gas outside |
| 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 |

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).

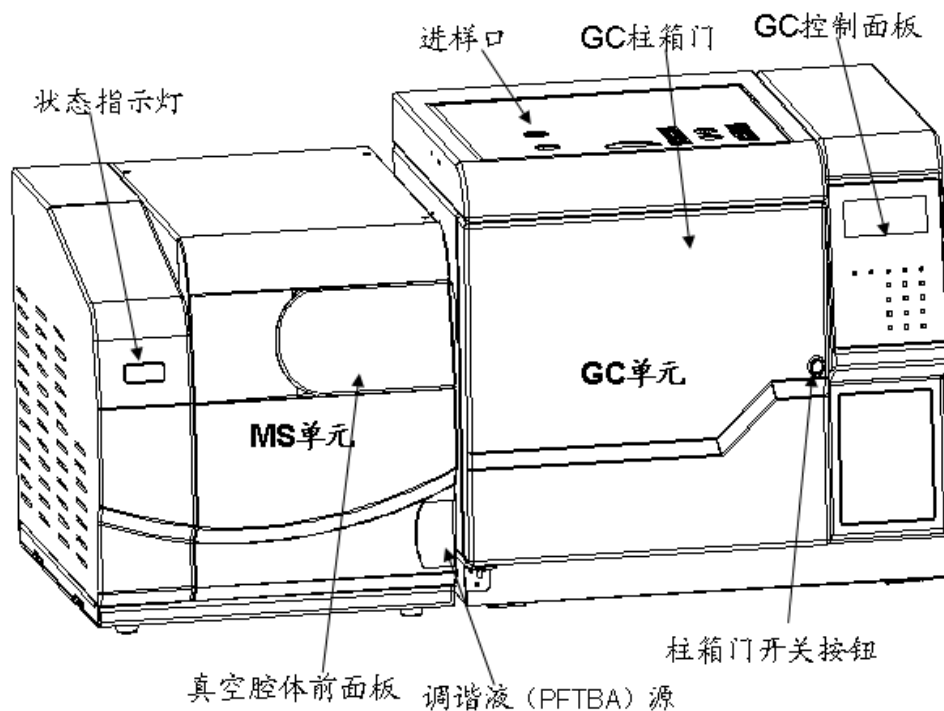


Figure 2-1 DW-GCMS-II Schematic front view

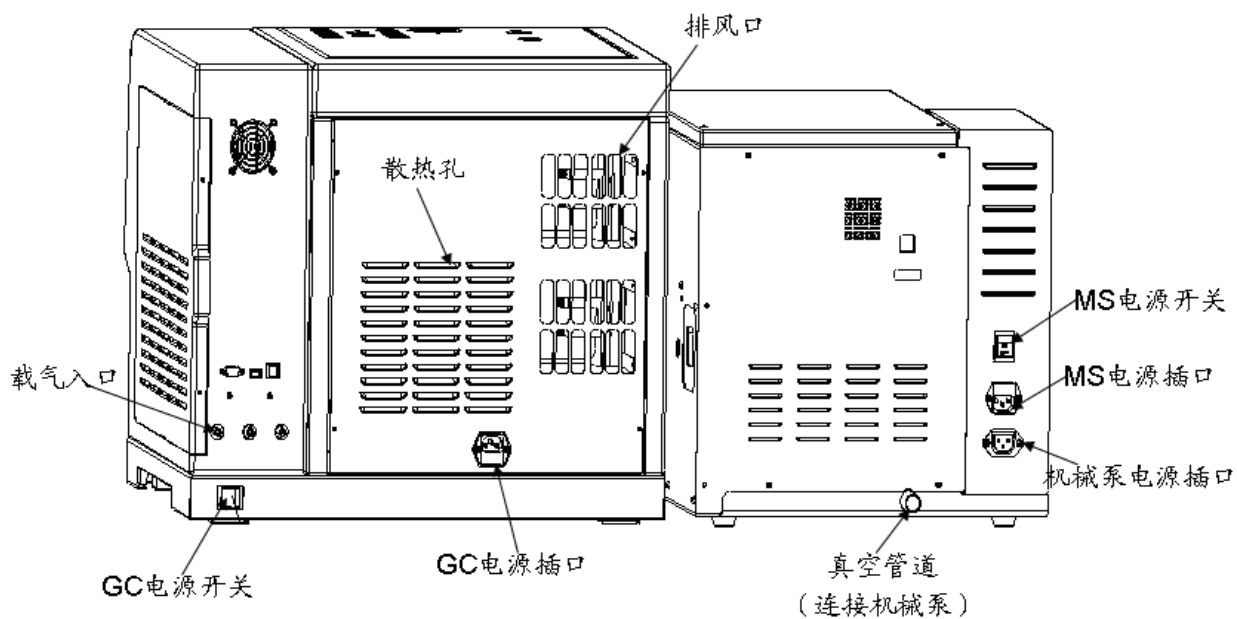


Figure 2-2 DW-GCMS-II Schematic rear view

2.3 External 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 | 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. Fast switch on/off the cylinder valve to clean the valve port, neither face the port directly or open too long, otherwise the reverse pressure of exhaust gas will make the cylinder fall over.
4. The valve injection port is mounted on the cylinder and tighten with a wrench;
5. Release the pressure regulator handle.
6. If open the cylinder pressure valve, the reducer pressure gauge has some indication. If close, the gauge indication should not decline, otherwise it means leakage exists, which should be eliminated before use.

Gas path tube of the stainless steel and valve connector are connected as shown below (O-ring 3×1.8 (optional) is also used for replacement of 1/8" front/back ferrule).

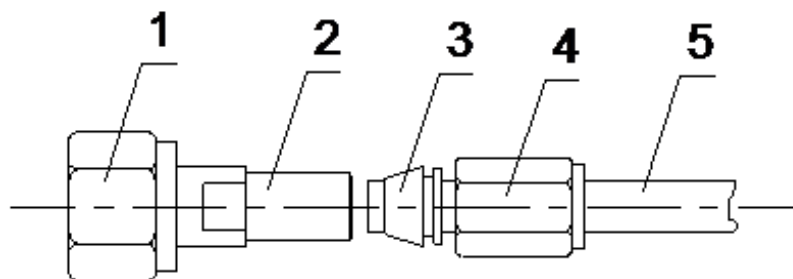


Figure 2-3 Gas path connection schematic diagram

1. Nut (M12) 2. Reducer connector 3. 1/8" front/back ferrule
 4. Nut (M8) 5. Φ1/8"×Φ2 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, contaminate or even damage the instrument.

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

Leakage check must be paid attention 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).

The carrier gas pressure imported into the chromatograph must be within the range of 0.5MPa-0.9MPa.

2.4 External 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, have the low-pressure gauge indicate at 0.5MPa;
2. Smear out the leak fluid at each joint of the external gas path, to observe whether there produce 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!

2.5 Septum, glass insert installation

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

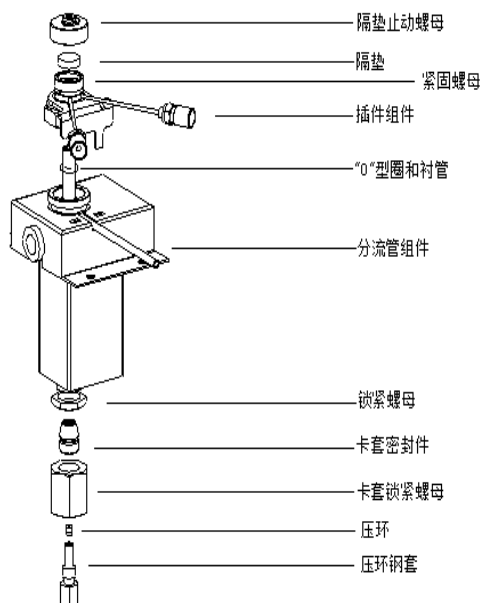


Figure 2-4 Split / splitless injection port exploded view

2.5.1 Glass insert installation

Related consumables and tools

| Part name | Part No. | Application |
|-----------------------|--------------------------------|--|
| Glass insert(split) | 304743254 | Split injection method |
| Quartz wool | 305330043 | Glass insert filler |
| O-ring | 304570315 | Nebulizing chamber temperature:room temperature ~450°C |
| Injection port wrench | 38020004000 | |
| Tweezer | 3802000724 | |
| Glove | 3801001144 (or similar gloves) | |

1. Confirm the position of quartz wool

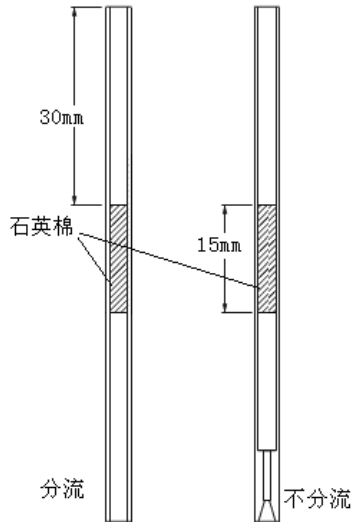


Figure 2-5 Loading position

Of quartz wool in the glass insert

2. Install O-ring: O-ring installed onto the glass insert about 7mm from the top, put the glass insert into the nebulization chamber and push to the bottom, where the ring about 5mm to the top.
3. Fasten glass insert: Carefully tighten the fastening nut by special injection port wrench .

2.5.2 Septum Installation

Related consumables and tools

| Part name | Part No. |
|------------------|--------------------------------|
| Injection septum | 305330042 |
| Tweezer | 3802000724 |
| Glove | 3801001144 (or similar gloves) |

1. Hand-tighten the septum nut (Figure 2-6).
2. Loosen half circle of the nut.

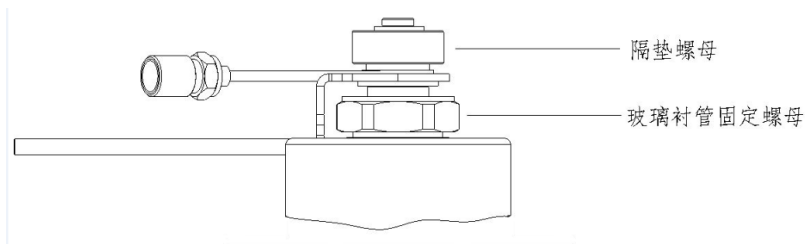


Figure 2-6 Injection port outline

Warning

Do not directly touch the glass insert or septum.

2.6 GC Column Installation

Related consumables and tools:

| Part name | Part No. |
|---|--------------------------------|
| Column | 3709400080 |
| Column cutter | 3802000251 |
| Pressure ring | 304650006 |
| Pressure ring ferrule | 3010205443 |
| Interface column nut | 3041810051 |
| Column mounting jig (for interface end use) | 3010211139 |
| Wrench (1/4 in. x 5/16 in.) | 3802000722 |
| Septa (either used or second-hand is allowed) | 305330042 |
| Gloves | 3801001144 (or similar gloves) |

2.6.1 Injection port side installation

1. The position markers (such as old septum), pressure ring ferrule and the pressure ring pass through the free end of the column (as shown in Figure 2-7) in order. The tapered end of the ring must face the its ferrule.
2. Use the column cutter to cut off the column end for 1~2cm, and to ensure 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 by use of acetone or other solvents prior to be put into the injection port.
5. Note that first hand-tighten the nut to the end, then wrench 1/4 turn, since over-tightening will affect the sealing effect.

Warning

Have the column circle around the metal frame, which hanging on the column rack of the oven. The hanging location depends on the diameter of the frame, 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 injection port and MS interface, do not let any part of the column touch the oven inner wall.

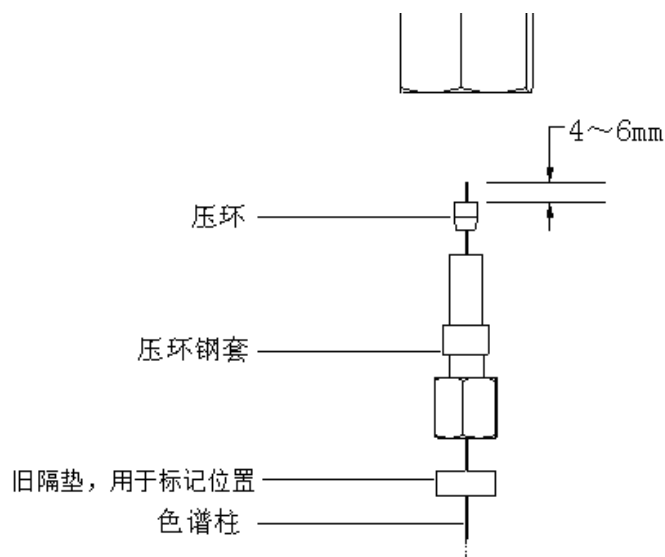


Figure 2-7 Column installation at the injection port end

2.6.2 MS interface end installation

1. The position markers (such as old septa), pressure ring ferrule and the pressure ring pass through the free end of the column (as shown in Figure 2-8) in sequence. The tapered end of the ring must face the interface column nut.
2. Insert the column mounting jig into the front end of the column which protruding the jig about 1cm. Then tighten the nut, cut off the column protruding part and ensure a smooth fracture.
3. Remove the column from the column mounting jig after fasten the position markers, gently wiped with acetone before inserted into the MS interface, then tighten the nut.
4. Note that first hand-tighten the nut to the end, then wrench 1/4 turn, since over-tightening will affect the sealing effect.

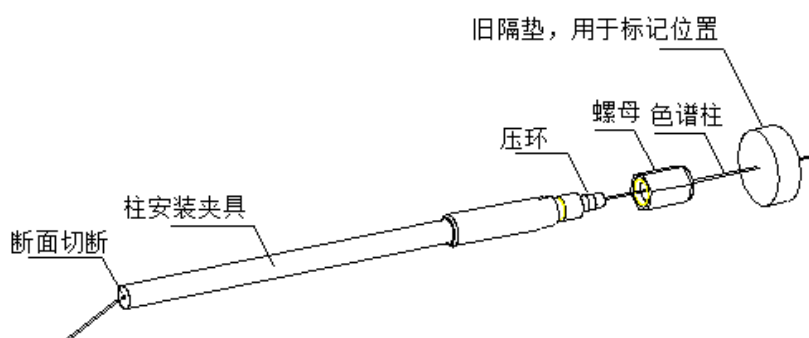


Figure 2-8 Column installation at MS interface end

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, otherwise disassembling 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 |
| Screwdriver | 3802000073 |
| Tweezers | 3802000724 |
| Gloves | 3801001144 (or similar gloves) |

Warning

There's voltage danger inside the MS chamber. Turn off the instrument before the operation and make sure the main power switch is off.

Ion source and interface turn hot during operation, before the maintenance, please turn off the instrument and let it cool for at least 30 minutes.

Warning

The personnel must wear clean, lint-free gloves to avoid contamination during internal operation of MS chamber.

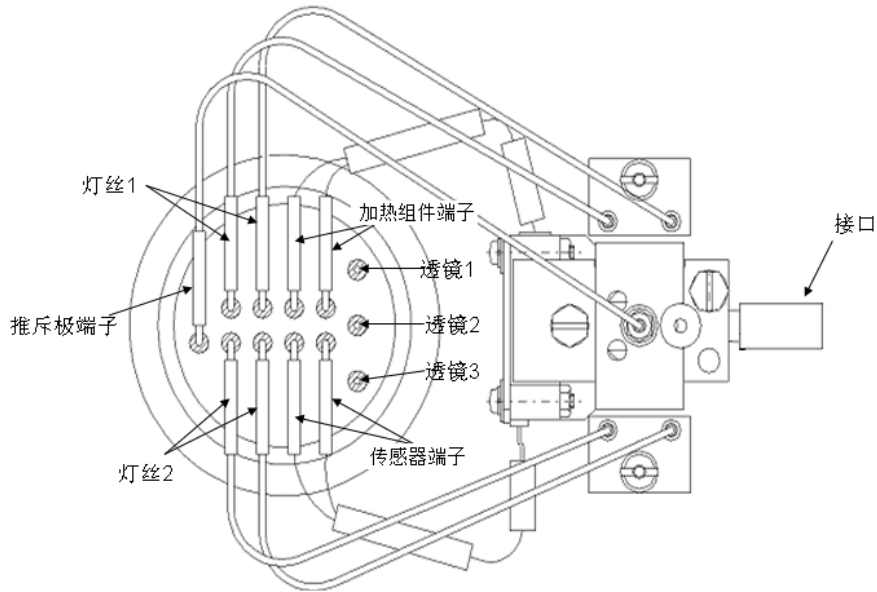


Figure 2-9 Ion source

1. Make sure that the instrument main power switch is off.
2. Loosen the 4 nuts on the front panel by use of a flathead screwdriver, take off the front panel.
3. Use the tweezers wiped by acetone to take off the filament lead terminal (see Figure 2-9).
4. Loosen the filament lock nut and pull out the filament (see Figure 2-10).

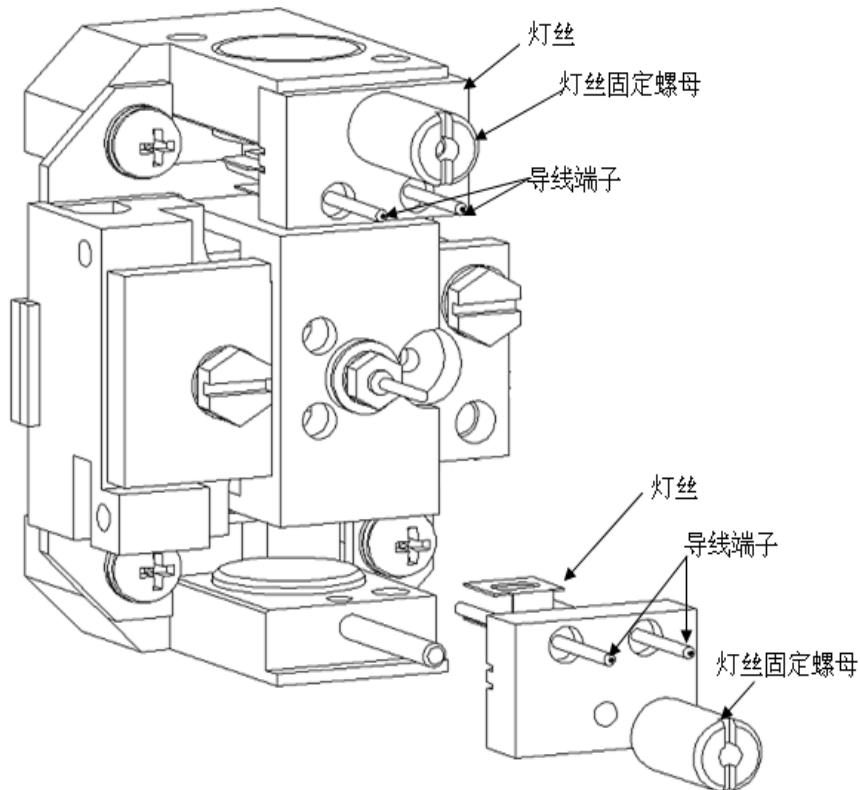


Figure 2-10 Ion source assembly

1. Install the new filament and tighten the nut.

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

Warning

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

2.8 Installing 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 gloves) |

Warning

Notice electric shock . Turn off the instrument and make sure the main power switch is off.
Ion source and interface turn hot during operation. Before maintenance, please turn off the instrument and let it cool for at least 30 minutes.

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.

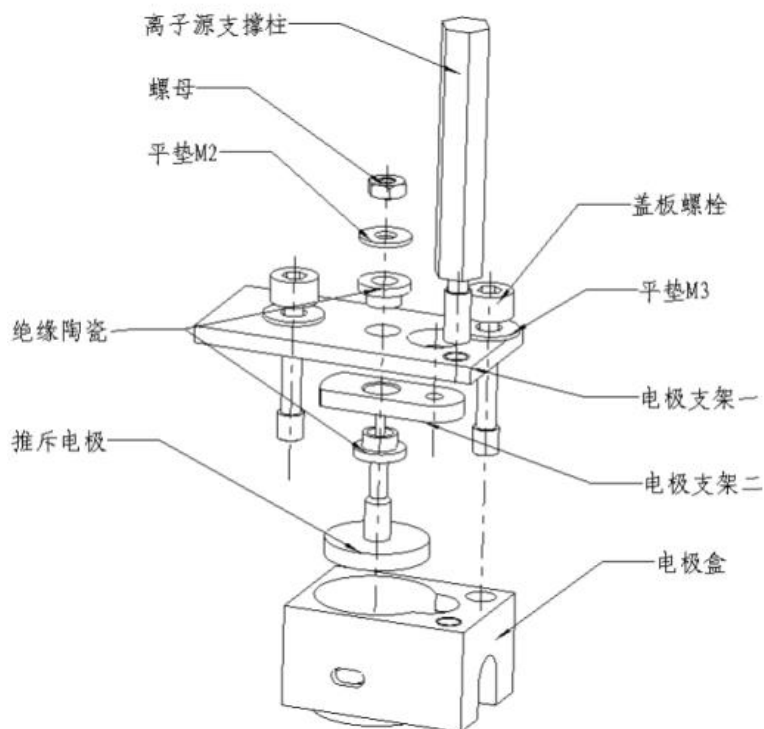


Figure 2-11 Ion source box and mounting jig

1. Put the repulsion electrode into the ion source electrode box and adjust it to situate the repeller electrode in the center of the electrode box.
2. As shown in Figure 2-11, install the parts in an upward order.
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, then the other, 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. Ensure the repulsion electrode and the ion source electrode box are insulated.

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