FULI INSTRUMENTS

GC9790 II GAS CHROMATOGRAPH

Installing and Operating Manual

ZHEJIANG FULI ANALYTICAL INSTRUMENTS Co., Ltd.

CONTENTS

Introduction to basic operation of instrument

1. Power supply
   1) 220V AC±10%
   2) Frequency : 50±0.5Hz
   3) Power consumption : ≥2000W
   4) Grounding resistance ＜0.1Ω
2. Requirements gas source

   N_2 : 99.995%

   H_2 : 99.995%

   Air : dry, oil-free
3. Requirements Fid operation

   Carrier gas : flow rate 30ml/min.

   H_2 : at ignition, 0.15 ~ 0.2Mpa

   after ignition, 0.1Mpa, i.e., 30ml/min.

   Air : at ignition 0.03Mpa

   after ignition 0.1Mpa, i.e., 300ml/min.

4. Requirements of TCD operation
   1) Initiate & preheat, before current is supplied, aeration should first be implemented.
   2) Flow rate calibration : Identical two way output flow rate, i.e., 25 – 30ml/min.
   3) N_2 taken as carrier gas, current can not be set too large (Refer to the operating manual.).

CHAPTER 1 OUTLINES

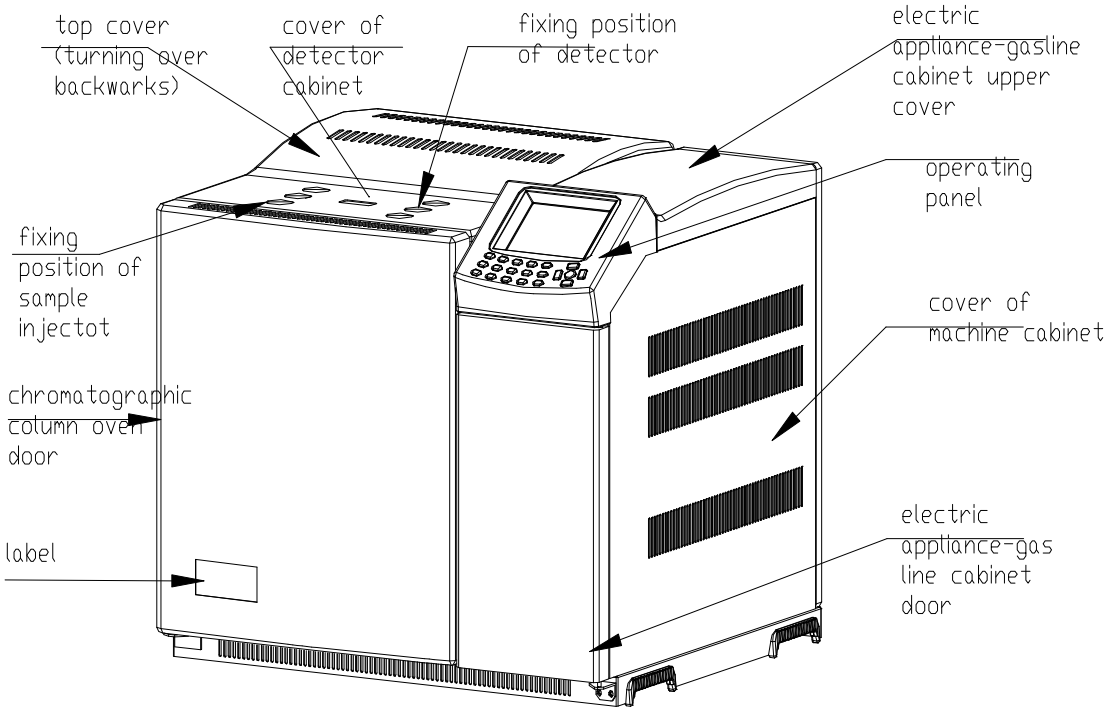


Fig. 1-1 Basic structure of instrument

Type GC – 9790 Ⅱ Gas Chromatograph is a serialized single detector device which is a kind of popular, multifunctional and high performance instrument (Its structure is shown in Fig.1-1.). Analytical system of double gas lines is adopted in this instrument witch basic configuration and hydrogen flame ionization detector is matched with the instrument. The operating modes of both constant temperature and programmable temperature can be implemented by the instrument and packed column or capillary chromatographic column can be installed in the instrument. Either column head sampling or rapid vaporous sampling analysis' objects and application aspects.

Microcomputer control, keyboard operation and LCD screen are adopted in the instrument. It has the advantages of high integration of electronic circuits, high reliability, easy operation and good adaptability to long term operation etc..

Auto cooling system of back door opening is matched with the column oven and every zone of heating (injector oven, capillary injector oven, detector oven, auxiliary oven and column oven) is respectively insulated from each other with thermal protective material in order to minimize heat transmission so that the temperature of every heating zone can be controlled independently.

Two packed column injectors or rapid vaporous injectors can be contained at the same time in the injector oven. A capillary injector can be fixed in the capillary injector cabinet. In order to meet the requirements of special users, two detector cabinets, one standard detector cabinet and one thermal conductivity detector cabinet can be installed, at the same time on the instrument. Between them, the standard detector cabinet is of the double base mode , which can install two detectors at the same time.

Both the gas flow rate & pressure control systems of the instrument with basic configuration are

installed in the gas line cabinet of the instrument and the sample injection system installed at the top of the instrument demands a vertical injection mode. In addition, an option of auto sample injection system can be adopted for the instrument. The output signals of the instrument can be connected to recorder, integrator and computer etc. peripheral drawing equipment...

CHAPTER 2 SPECIFICATIONS

2.1 General parameters

Outline dimensions : 565×510×490mm.（W×H×D）
Column cabinet dimensions : 260×250×150mm.（W×H×D）
Chromatographic column installation gap size : 152.4mm.(6″ standard joint)
Chromatographic column : Outside diameter of packed column Φ 3~ Φ 5mm. (metal or glass column)
Wight : 50Kg.

2.2 Temperature control

Temperature control of column cabinet:
Room temperature plus 6℃~350℃ (1℃ increment), (guaranteed specification), (Upper limit of temperature setting can be up to 399℃ and it is allowed for user to use, but the specification is not guaranteed.).
Temperature floatation : less then ±0.1℃(environmental temperature variation 10℃ or power voltage variation 10%)
Temperature gradient :±1% (temperature range 100℃~350℃)
Programmable temperature rise
program steps : 7
rate of temperature rising : 0.1 ~40℃/min.(0.1℃ increment)
rate of temperature falling : Temperature of column cabinet falls down from 200℃ to 100℃ less than 3 min..
Time setting : 6000min..
Temperature control of the thermal conductivity detector
Room temperature plus 20℃~350℃(guaranteed specification) (Upper limit of setting parameter can be up to 399℃ and it is allowed for user to use, but the specification is not guaranteed.).
Accuracy of temperature control : less than ±0.1℃
Temperature of the other heating zones :
room temperature plus 20℃~350℃(guaranteed specification), (Upper limit of temperature setting can be up to 399℃ and it is allowed for user to use, but the specification is not guaranteed.).
Accuracy of temperature control : less than ±0.2℃

2.3 FID detector

Detection threshold : less than 3×10^{-12} g/s[n-c16]
Noise : less than 3×10^{-14} A
Drift : less than 5×10^{-13}A/h.
Linear range: more than 10^6

2.4 Application environment

Environment temperature : 5℃~35℃
Relative humidity : less than 85%
Power voltage : AC 220±22V
Power frequency : 50±0.5Hz
Max. power consumption : 2500W
The operating room for the instrument should be protected from corrosive gases, violent mechanical vibration, strong electromagnetic field interference about it, violent indoor temperature variation and strong air convection.

CHAPTER 3 INSTRUMENT PACKAGE

3.1 Type GC–9790 GAS CHROMATOGRAPH (offered according to order) 1 set

3.2 Type GC–9790 GAS CHROMATOGRAPH installing & operating manual 1 piece

3.3 Operating manual of the detector corresponding to Type GC–9790 GAS CHROMATOGRAPH (not including FID) 1 piece

3.4 Qualified certificate of manufacturer for Type GC–9790 GAS CHROMATOGRAPH 1 piece

3.5 Packing certificate for Type GC – 9790 GAS CHROMATOGRAPH 1 piece

3.6 Spare parts for Type GC – 9790 GAS CHROMATOGRAPH 1 kit

3.7 Spare part list for Type GC – 9790 GAS CHROMATOGRAPH 1 piece

3.8 Certificate of product maintenance 1 piece

3.9 GPI – 2 GAS Purifier (offered according to order) 1 set

3.10 Integrator or recorder (offered according to order) 1 set

CHAPTER 4 OPERATING PRINCIPLE OF INSTRUMENT

This instrument manufactured on the basis of gas chromatography is an effective device to carry out gas chromatographic analysis. Gas is adopted as mobile phase for the instrument. After a mixed multi-component sample to be analyzed is injected into an injector and vaporized for an instant, the sample (gas) is carried by carrier gas, i.e. mobile phase and goes through chromatographic column filled with stationary phase. The absorption, de–absorption and dissolution etc. processes will happen between the molecules of the components and stationary phase in chromatographic column, so that those components with close performance and structure will separate greatly because the individual molecules' distributive phenomenon will repeatedly happens many times between the two phases. In addition, the absorptive and de–absorptive acting force of each component of sample is quite different and its separated time needed is different, too. As a result, all components of the mixed sample have been separated completely. Next, the every separated component will sequentially goes into the detection system, the separation information will be converted into electrical signals by the detector and they will be applied to a recorder or integrator to draw in chromatogram. Its flowchart is shown in Fig.4.1. Similar to other analytical instruments, GC (Gas Chromatograph) is applied to testing chemical components and physical properties of substance. The chemical components denote that a kind of compound or admixture consists of what molecules, atoms or aggregates, how much content they have individually. The physical properties of substance denote its partition coefficient (at stationary phase), activity coefficient, molecular weight, vapor density, specific surface and pore size distribution etc. physical constants. GC can be applied being widespread to petroleum, chemical, organic synthesis, papermaking, electric power, metallurgical, medical-pharmaceutical etc. industries, and pesticide residual, soil & environment supervisory, labor protection, commodity inspection, food hygiene, and public security investigation, as well as blank analysis of super pure substance research department etc.. Today, GC instruments have become one kind of necessary analytical equipment for various chemical analysis labs.

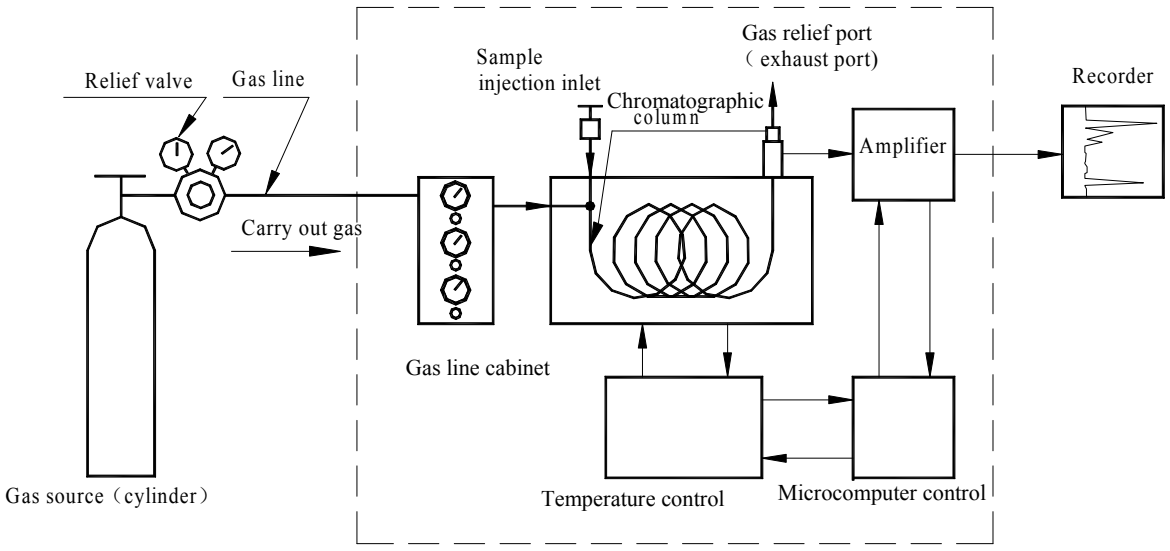


Fig.4.1 Flow chart of gas chromatograph

CHAPTER 5 INSTALLATION

5.1 Preparations for lab

The instrument should be installed in a special analytical lab for GC in order to manipulate the instrument and gas source respectively. It should be put on a sturdy cement or wooden bench without vibration, on which there is sufficient space for placing recorder, integrator etc. peripheral equipment and on whose back a certain distance should be left between the bench and wall for its maintenance. Sufficient power supply should be guaranteed. The instrument should be kept away from tinder and protected from corrosive gases, exceedingly venting, and violent temperature fluctuation. The arrangement of the bench may be referred to Fig.5.1.

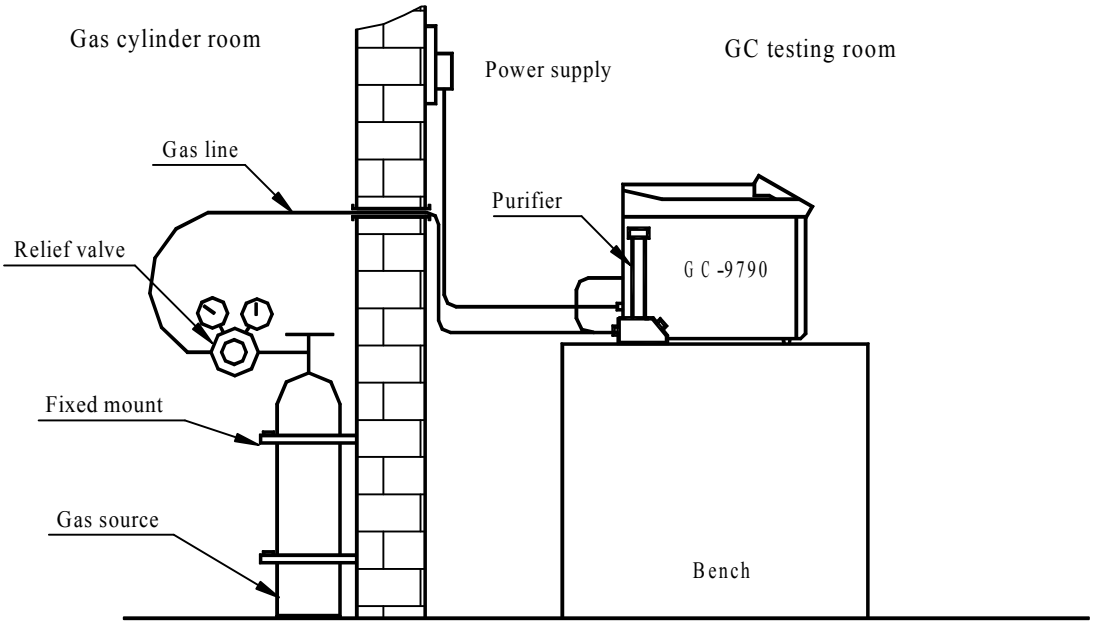


Fig.5.1 Arrangement of lab

The carrier gas and auxiliary gas needed by the instrument, in general, can be supplied by a high pressure cylinder, gas generator or oil-free air compressor. High pressure gas must pass through the relief valve to supply the instrument. In general, gas source is not matched with the instrument, so users will have gas source prepared for themselves. Gas source is the prerequisite condition for initiating the instrument, so it is necessary to take it into first consideration in advance. If user puts forward the concrete requirements of application when the instrument is ordered, the manufacturer will offer help to purchase the necessary equipment instead. According to the detector configuration, various gases can be chosen and the sorts of gas, their basic parameters and selective principle can be referred to table 5.1. The gas generator based on the principles of chemistry & electrolytics, oil-free air compressor etc. devices can be applied to offering working gas sources for the instrument. Because, in general, the water content of the gases generated by gas generator or air pump is rather high, particularly, special attention should be paid to those older gas generators. Thus, while those devices are applied, the protective measure must be taken and gas purified device should be matched with the instrument. In addition, it is noticed that regular maintenance for purifier is necessary and the filling material in the purifier should regularly be activated and replaced, otherwise its purification function

will be lost. If the gas with high water content applied for a long time both the service life of chromatographic column and stability of the instrument will be reduced or the gas line system of the instrument will be polluted, so that it will cause the instrument to work abnormally (Its activating method and period can be referred to the relevant contents about the gas line in chapter 7.).

Detector	Gas source	Inlet pressure	Purity
TCD	H_2 or He	0.3Mpa	99.999%
FID	H_2	0.3Mpa	99.995%
	N_2 or He	0.4~0.5Mpa	99.998%
	Air	0.3Mpa	No dust, oil mist, water content etc.
ECD	N_2 or He	0.4~0.5Mpa	99.998%
	or Ar/CH4		H_2O≤ 0.002 Pma
			O_2 ≤1ppm
EPD	H_2	0.3Mpa	99.995%
	N_2 or He	0.4~0.5Mpa	99.998%
	Air	0.3Mpa	99.998%

Table 5.1

5.2 Unpacking

＜CAUTION＞

whenever open up the covers or flank aprons on which electric symbol is denoted please pay attention to hazardous voltages inside the instrument. During maintenance, if it is necessary to open them must pull out power plug firstly to ensure personnel safety.

After the instrument arrives, inspect the perfection of its outer package in time and count & check the number of parts according to the delivery order. If find the outer package damaged or matched with parts lost, stop unpacking or return the instrument to its manufacturer, or contact with the carrier, and advise the relevant department of the manufacturer of the event in order to deal with it properly or to ask for compensation in time so that the loss of both the user and manufacturer will be minimized.

After the instrument is unpacked, investigate its outward appearance & damage caused during transportation, and count whether its number of accessories & spare parts are consistent with its packing list. Check up whether the instrument configuration is consistent with its order, its functions are perfect, and its every movable part is flexible & reliable. If find that any article is left out, assembling mistake exists or the working performance is not satisfied, come into contact with the manufacturer in time in order to avoid unnecessary economical loss or delaying your work.

After check up and make sure there are no problems, open up the cabinet's door of the chromatographic column and use a slender screwdriver to push the motor fan slightly to check up whether its fan rotates smoothly, looses, is blocked. If the fan looses it should be repaired in time. Take out the power cord from its spare part box, complete connection according to Fig.5.2, and it is ready to turn on power supply. Before turning on, it is needed to be pre-checked up whether there is short-circuit between the phase & neutral line of its input socket, the fuse holder looses, the voltage, power & phase position at its power plug satisfy the instrument, and it is grounded well. The position

of its power switch is shown in Fig.5.3, the installation of the instrument on the bench can be referred to Fig.5.4.

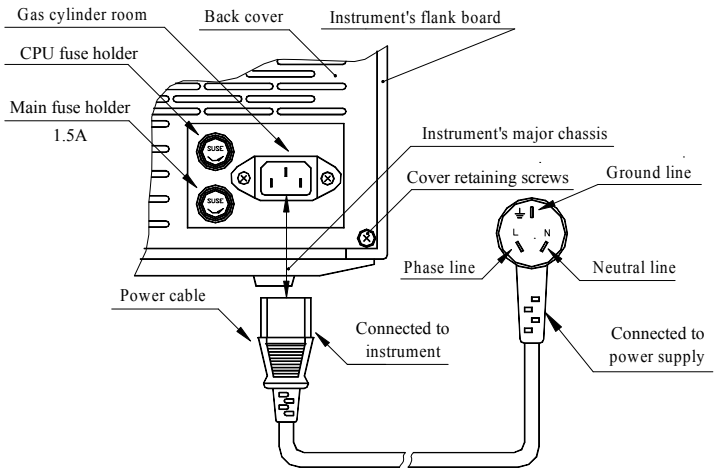


Fig.5.2 Installation position scheme of power cord

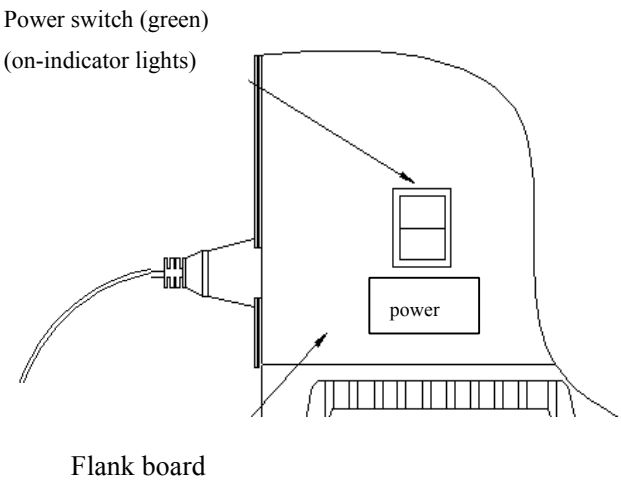


Fig.5.3 Power switch position scheme

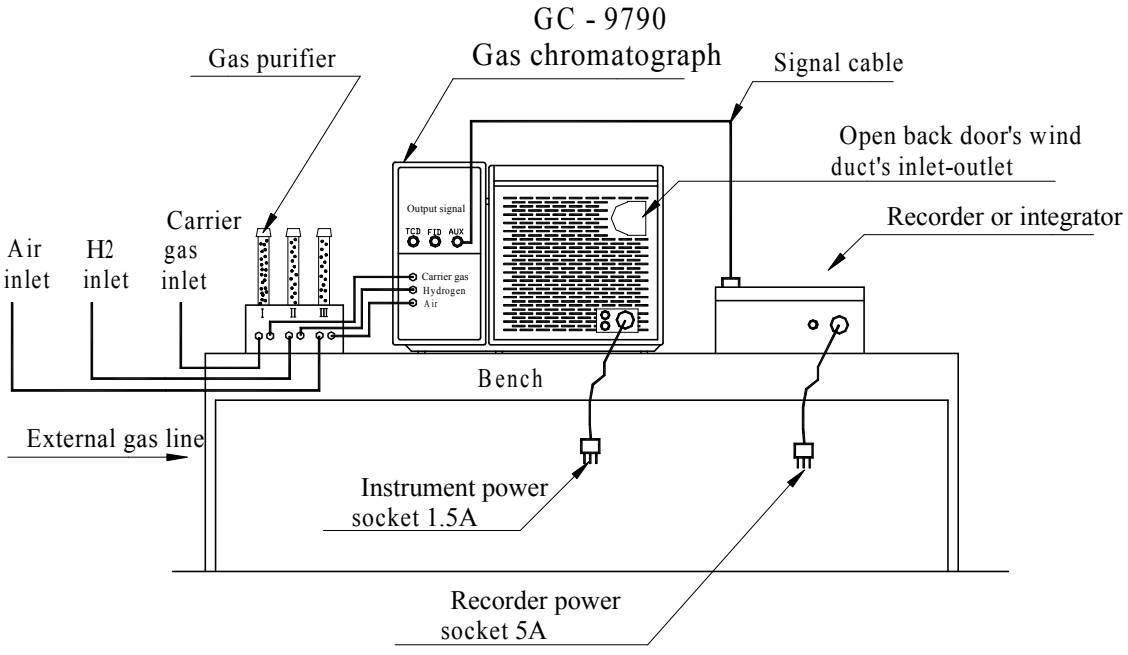


Fig.5.4 External circuit & gas connecting scheme of bench

There are two signal output channels, 《TCD》 & 《FID》 and the output signals can be connected to recorder, integrator and chromatographic workstation etc. recording equipment. Its connection mode is shown in Fig.5.5. According to application situation, user can properly select suitable recording

equipment. The number of detectors, in general, is decided according to the instrument configuration, but their installation position, in general, is kept unchanged. The instrument with basic configuration only has one《FID》channel. The assembly of its detector cabinet and circuit, gas line arrangement are shown in Fig.5.6.

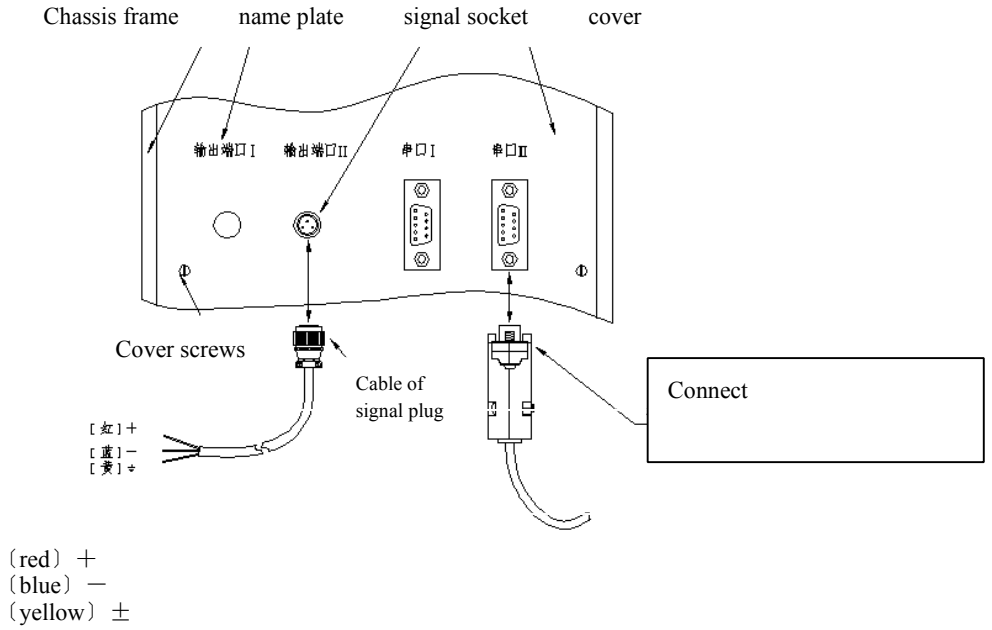


Fig.5.5 Signal cable connecting scheme of detector

5.3 Gas line connection

CAUTION
The pipeline not having been purged will directly pollute the gas line and detection system of the instrument to make its stability reduced and it will not be able to work normally for a long time.

All gas sources of the instrument are connected through Φ3mm pipelines to the gas inlets of its gas line cabinet, at which the signs to denote the applied gas names are put and their joints are connected with M8 × 1 nuts. To operate TCD, ECD, only carrier gas pipeline is needed to be prepared, and besides that, H_2 and air pipe lines are needed to be prepared for the other detectors. The connecting method can be referred to Fig.5.6.

To connect the instrument and gas sources, it is most suitable to adopt red copper or stainless steel pipes. In order to prevent the pollution of the instrument's gas line system from the mist and other chemical residues in the pipelines. The applied pipelines must strictly be purged according to the following procedure, then they can be connected with the instrument.

Purging method :

1. Purge inner wall of the pipelines with acetone detergent solvent to clear residual oil and the consumed amount of the solvent is about 150ml per meter (pipe) and after oil is taken off, de-absorb the pipe thoroughly with absolute alcohol.
2. Wind the purged pipelines, put it in a dry oven with temperature of 300℃ and meantime ventilate nitrogen gas at 30ml/min into the oven continuously for an hour, then after the temperature of the pipe drops, seal its ends and put it into a special bag in order to avoid being polluted again.

The other pipes, such as nylon pipe can also be applied, but this kind of pipe is not easy to be cleared and easy to generate evaporable material further to affect the stability of the instrument. In addition, the nylon pipe line is easily to be aged and leakage will easily happen. While hydrogen gas is applied, the leakage occurrence is very dangerous, so it is necessary to check, maintain this kind of pipe regularly to avoid the leakage happing.

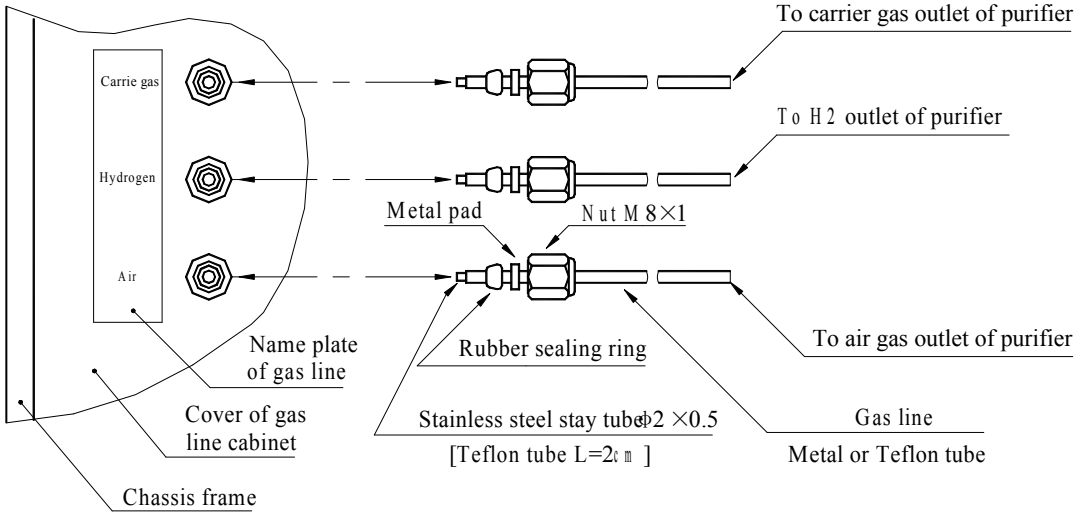


Fig.5.6 Connecting scheme of external gas lines

5.4 Check up system leakage

《CAUTION》

Before leaving the factory, all the gas lines are strictly tested for tightness performance, so leakage phenomenon, in general, will not happen in interior of the instrument. The gas leakage, in general, happens at the joint of chromatographic column or sample injection pad (Because of injecting sample many times, the injection pore size is enlarged, so that the tightness performance is worse and the sample injection pad should be replaced in time.)etc. places.

Both the hydrogen gas and air after reaching FID will meet little resistance and although there may be a little bit of gas leakage, it will not affect the instrument severely. So, if no

particular events happen, don't disassemble the gas lines and detector. After the instrument is started, if there is no gas leakage caused from the external gas lines, next testing can be implemented.

When trouble or abnormal operation happens to the instrument and it is necessary to disassemble the parts of the detector and gas lines, please ask for skilled professionals to execute maintenance. First of all, a clean maintenance measure should be taken and don't directly touch, particularly, certain key parts, such as the ceramic parts, nozzles etc. with hand. Instead, should put on yarn protective groves to avoid polluting the parts and to keep the instrument's stability.

Leakage investigation for the instrument system can be divided into two steps. After gases are supplied first check up the section of the gas lines between the gas sources' outlets and the inlets of the purifier (including the relief valve and its joints, and the relevant gas pipe lines), then check up the section of the gas lines between the instrument's gas line system and the outlets of the purifier and the tightness performance of the gas line system of the instrument.

The procedure of leakage investigation between the steel cylinder and inlets of the purifier :

After the gas sources are connected, the pressure of 0.5 MPa is given by the relief valve, close the corresponding shutoff valves on the panel of the purifier; Turn off relief valve, observe low pressure indication at it and take down the pressure variation values for 10 min.. If the pressure drops obviously, it denotes that the gas leakage exits in the system and testing of leakage detection must be done at this moment. Leak detecting liquid can be applied to leakage detection of the gas line system (If have no special leak detecting liquid at hand, can have a mixture solution of detergent/water instead. Its make up method is that put detergent of certain dose into water and stir it up until bubbles appear.). Under the conditions of the system sustaining a constant pressure, smear a bit of the liquid on the joints or nodes at which leakage may happen and observe whether there is a bubbling phenomenon. According to this method, check up and eliminate gas leakage at one point after another. During checkup, use the liquid as little as possible and after checkup, clean up the liquid in time to prevent it from contaminating the gas line system as the pressure drops.

The investigating procedure of the system's gas tightness :

Open up the cover of the detector cabinet, loosen the corresponding gas line's fastening nut and seal the joint end of the gas line with a blinding plug to insure the tightness of the gas outlet. Refer to Fig.5.7.

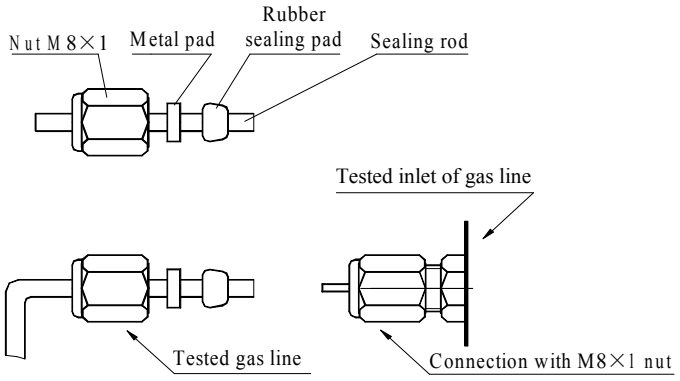


Fig.5.7 Structure of sealing end

Open up the flank board of the gas line cabinet :
Open up the shutoff valve of the corresponding purifier and fill the system with nitrogen gas up to 0.35Mpa, then shut up the shutoff valve; After the gas has reached a balanced status for 2 minutes, observe the pressure variation of the corresponding pressure gage in the cabinet of the gas line. If the pressure is obviously changed after 10 minutes it denotes that the system is leaking and hence, a test of leakage detection is needed. Its method is idem (During leakage detection, leak detecting liquid must be applied as little as possible and after checking up, clean up the liquid in time to prevent from contaminating the gas system.).
(Note : Before leaving the factory, the gas tightness of the system is strictly tested, so before initiating the instrument, this test is not necessary to be done. Only if there is a trouble in the system or replace parts of the gas line this test can be implemented.)

5.5 Check up before turn power on

CAUTION
Before the chromatographic column is not built into the instrument, initiate it to execute every operation on its panel as an exercise. But any gas, especially, hydrogen gas is forbidden to be applied to the instrument in order to avoid danger happening.

Check up whether power supply is correctly connected;
Check up whether the gas line system is completely connected and the gas sorts are consistent with the requirement;
Check up whether the steel cylinder is reliably fixed and the pressure range the relief valve is consistent with the requirement;
Check up and familiarize the whole structure of the instrument, setting method of the keyboard, every control switch, gas line system. In addition, the relevant chapters & sections of the operating manual can be referred to familiarizing the function of every gas flow rate adjustment valve and corresponding adjustment technique.

CHAPTER 6 OPERATIONS

6.1 Gas sources are the gas supplier that offers carrier gas and auxiliary gas for gas chromatograph. Hydrogen, nitrogen, helium and argon gas etc. are commonly used as carrier gas and oxygen gas and air etc. are used as auxiliary gas for the instrument.

Selecting carrier gas, firstly, should meet the requirements of detector and consider analytical method which will affect analytical period, column efficiency and sensitivity. Taking column efficiency, for example, should require carrier gas with small diffusion coefficient, and to obtain better peak pattern, nitrogen is commonly adopted as carrier gas. If desire to shorten analytical period taking helium gas is better than nitrogen gas. In terms of TCD, often take hydrogen (helium) gas with high thermal conductivity coefficient as carrier gas to improve its sensitivity and nitrogen & argon gas are not applied. As viewed from safety and analytical period, helium gas is better then hydrogen gas, but China is lacking in helium resource and its price is rather high, thus hydrogen is commonly taken as carrier gas. In regard to nitrogen gas applied to FID as carrier gas, it not only has good safety but also high sensitivity. In regard to hydrogen gas applied as carrier gas, in order to improve the sensitivity, it is necessary to have a make-up gas treatment before it is applied to detector. As the summary mentioned above, hydrogen and helium gas are better to be applied to TCD, and when nitrogen, argon gas and air are applied, its sensitivity is rather low and Type N and W will appear easily. Nitrogen gas is often taken as carrier gas for FID and FPD, but on special occasion, hydrogen gas can be applied too. Nitrogen gas is commonly taken as carrier gas for ECD. In general, the principles of choosing carrier gas are mentioned in the following :

Inert gas (It will not react chemically with sample or stationary phase during analyzing.) has no corrosiveness and will not be dissolved in the range of 200~400℃；

Gas with low diffusion coefficient is applied for improving column efficiency；

Its price should be reasonable and it can be satisfied with the requirement of detector’s application;

It should be based on the sample analyzed and analytical accuracy standard to choose the sort of gas source and its purity reasonably. The messages can also be looked up from chemical analytical manuals.

《Warning》

When debugging TCD,if there are two kinds of Carrier Gas H_2 and Air, the chromatogram nut must be tightened,and then shut the Total Pressure of the Capillary Column gas line to avoid explodig happening.

Φ3mm pipelines which are used for the all inlets of gas sources and connected to the joints on the rear panel of the gas line cabinet are adopted and their spiral burr of M8 × 1 is adopted. The sealing structure can be referred to Fig.6.1. Only one carrier gas channel is needed for operation of TCD and ECD, while for operation of FID, FPD and NPD extra hydrogen gas and air channels are needed.

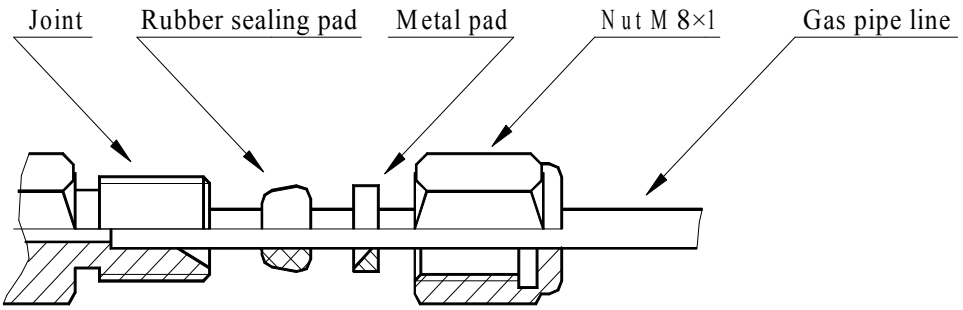


Fig.6.1 Sealing structure scheme

When steel cylinders are applied to supplying gas, each of them must be fixed a gas relief valve to lower the high pressure of the gas to the available lower pressure value. Relief valve can only be used for one kind of gas and mixed use is not allowed. The notice of option and application about relief valves can be referred to sec.7.2 of the manual.

In general, the highest pressure grades of gas contained in steel cylinder are divided into 15, 20 and 30Mpa three kinds. Among them, gas steel cylinder of 15Mpa is most commonly used and its gas volume is 40 l approx.. Safe attention must be paid to the steel cylinder application and the rules of its safe application must strictly be observed because of very high pressure in the steel cylinder. The advantages of applying the gas stored in steel cylinder are a complete variety of kinds, stable pressure, high purity of gas, easy installation, and easy replacement. Its disadvantages are the supply with difficulty in some cities, trouble in transportation and higher price.

《CAUTION》

Don't move the steel cylinder fixed with relief valve. Take down the relief valve and fit a safety head during transportation to protect the output nozzle of the steel cylinder from impact & collision.

After a relief valve is fitted for applying the cylinder, leakage testing must strictly be implemented. The standing cylinder should be fixed with a rack. The cylinder should be kept away from tinder & heat source and should not be placed at those sites where it will be violently exposed to the sun & rain and encounter excessive temperature difference.

6.2 Adjustment of gas flow rate

The control system of three gas lines of carrier gas, hydrogen gas and air is only offered for the instrument with the basic configuration. Its gas line flowchart can be referred in detail to the gas line part of Chapter 7. Before leaving the factory, the control valves of the total carrier gas pressure, and the gas lines of hydrogen & air are set to shutoff status.

Adjustment of carrier gas-line : Adjust 《Total pressure》 to let the pressure indication of carrier gas be at 0.3Mpa; According to the application requirement of the chromatographic column, properly choose the sort and flow rate of the carrier gases. Refer to the curve of gas flow rate to adjust《Carrier Gas I》 and《Carrier Gas II》 to define the flow rate of carrier gases (After the flow rate of carrier gases is defined, the pre-column pressure gauge will indicate the respective pre-column pressure according to the resistance of the chromatographic column. If the temperature of the instrument begins rising after

the adjustment of the flow rate is completed, at the moment, the indication of the pre-column pressure gauge will be regulated a bit along with the temperature rise and it is determined by the operating property of the stabilizing flow valve and it means the valve is working well.).

Adjustment of hydrogen gas line : Refer to the curve of the gas pressure-flow rate to adjust《Hydrogen Gas I》&《Hydrogen Gas II》to let the respective indication of the pressure gauge within the respective flow rate range. In general, when the gas flow rate is 30ml/min. the respective pressure indication will be 0.1Mpa (At the moment, the gas flow rate is 30 ± 2ml/min. This is the conventional operating condition for FID.).

Adjustment of air gas line : Adjust《Air》to let the indication of the respective pressure gauge be at 0.03Mpa (At the moment, the gas flow rate is 300 ± 20ml/min. This is the conventional operating condition for FID.).

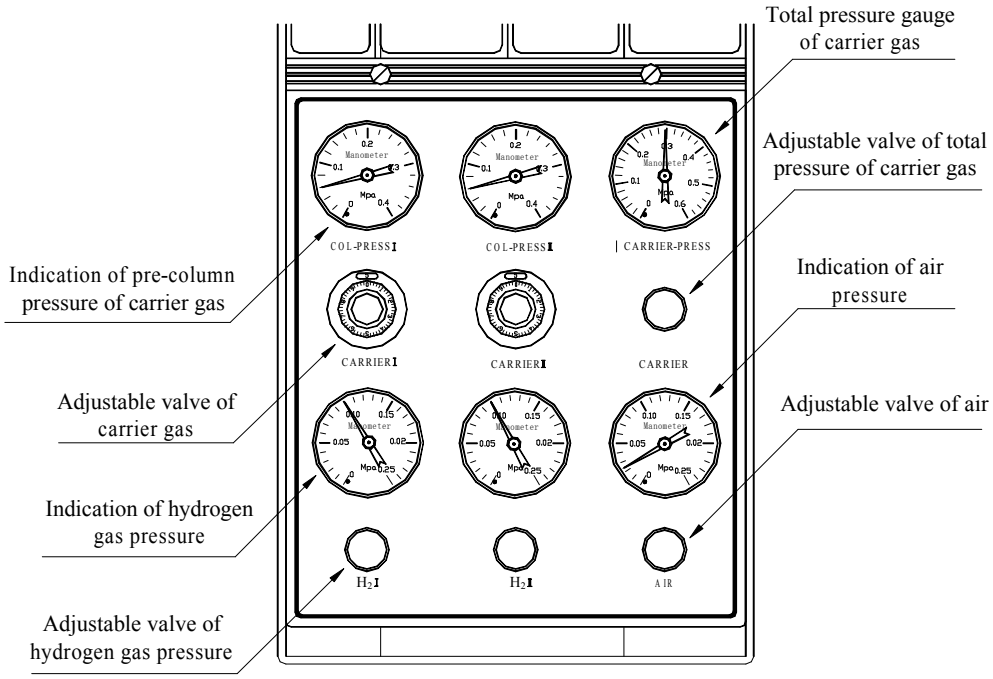


Fig. 6.2 Gas line control panel

6.3 Measurement of flow rate

If a special demand is put forward to the instrument's gas line control system, a soap film gas meter can be applied to recalibrating the gas velocity of flow. Its gas line's connecting method is shown in Fig. 6.3. Bubbling agent (Common detergent can be adopted to make up the bubbling agent and its making up method is identical with that of leak detection liquid.) is put into the soap film meter which is connected to the tested detector. In order to reduce the measurement error, a larger soap film gas meter should be adopted when the gas flow rate of the blank gas line is measured. After the gas is

ventilated into the gas line, the stopwatch's function of the instrument can be utilized for measuring the elapsed time the soap film passes from point 0 to point 10 to calculate the gas flow rate expressed with ml/min.. In order to avoid polluting the gas line, the applied soap liquid height of the meter must be noticed to keep the soap film liquid from flowing into the gas line from the meter.

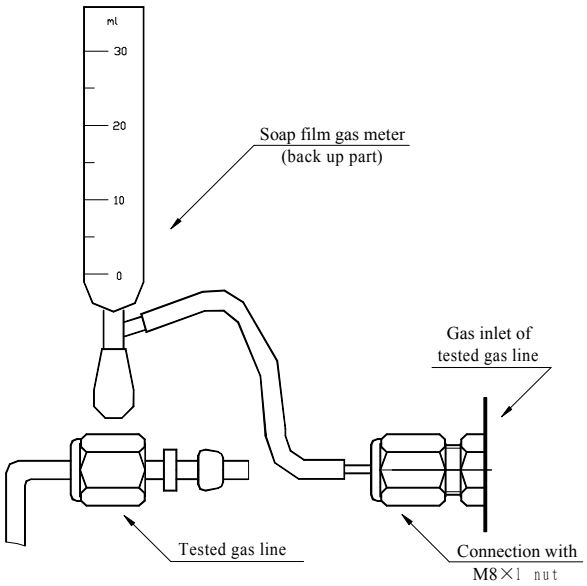


Fig. 6.3 Scheme of flow rate measurement

6.4 Column installation

《CAUTION》

After heating, even if the column oven and chromatographic column have been cooling, the temperature of the injector, detector's joint are still considerably high. So must wear heat shielding gloves or take certain protective measures to operate for keeping from scalds.

During column installation,《Heating》switch must be turned off to keep the crumbles blown about by the blower of the column cabinet from flying into eyes;

After column installation is completed, the all unnecessary articles in the oven must be cleaned up and the pollutants on the surface of the cabinet should be wiped up;

Once hydrogen gas is introduced, ignition should begin immediately to avoid the gas accumulation causing danger.

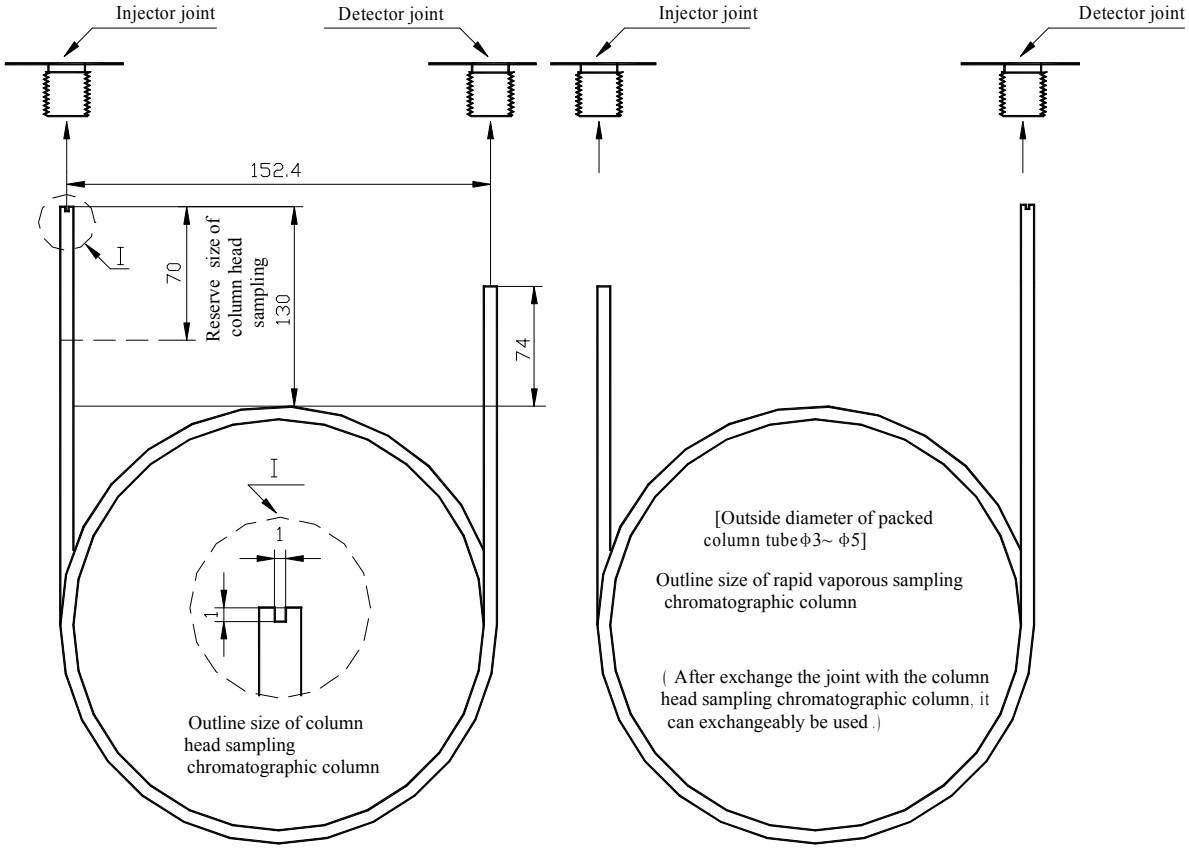
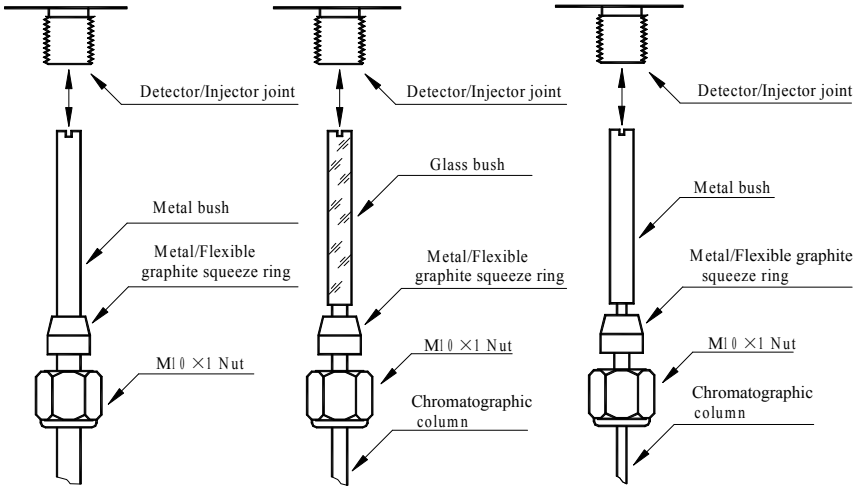


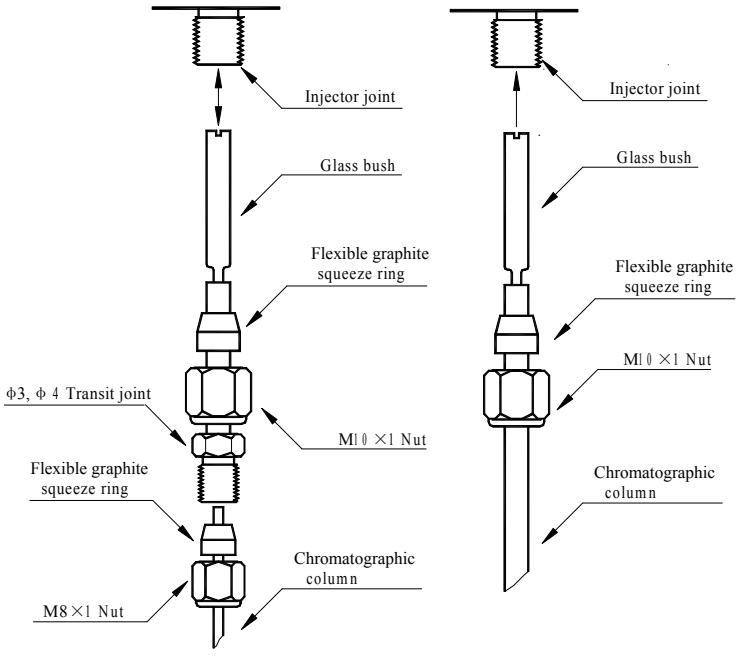
Fig. 6.4 Outline sizes of chromatographic packed column

In GC, the separated process of sample happens in chromatographic column. Therefore, in terms of the instrument, the quality of packing material and column efficiency of the chromatographic column is very essential. The separation efficiency of chromatographic column is mainly determined by the option of stationary phase in the column and the packing technique. In addition, the category of chromatographic column, material, shape, size, installation, sealing, and activation processing of column tube will highly affect the separation & detection of the sample as well.

The injector & detector joints (the inlet & outlet of the chromatographic column) matched with the instrument are located at the top of the column oven. If a system with double gas lines or TCD is applied to the instrument, at least two chromatographic columns are assembled in the instrument. The outline and open size of the chromatographic column are shown in Fig.6.4. The sealing structure and installation method of the chromatographic column are shown in Fig.6.5. Metric M10 × 1 nuts are used as joints to connect the detectors, chromatographic columns and injectors



Installation method of column head injector



Installation method of rapid vaporous injector

Fig. 6.5 Plug-in packed chromatographic column installation

The sequence of chromatographic column installation is as follows :

Turn off《Heating》switch at the bottom of the instrument's flank board, at this moment, the detector & injector can be sustained at the operating temperature;

Turn off the shut off valves of the hydrogen gas & air at the gas purifier and keep the carrier gas continuously flowing in order to keep the air from reversely diffusing into the gas lines and filter;

Open up the column cabinet's door (If it has been heated, should let the inside of the cabinet naturally cool to the ambient temperature to avoid scalds.). With regard to a new instrument, first take down the sealing nuts of the injector & detector;

Rotate and take down the cooling head of the injector and take out the injection shock insulator, take out a metal blockage shaped like a shock insulator from the part box to install it at the position of the injection shock insulator, then rotate the cooling head of the injector tightly. Its function is to guarantee the gap between the chromatographic column and shock insulator to reduce the dead volume (The operation can be referred to the installation diagram of injector in Chapter 8.);

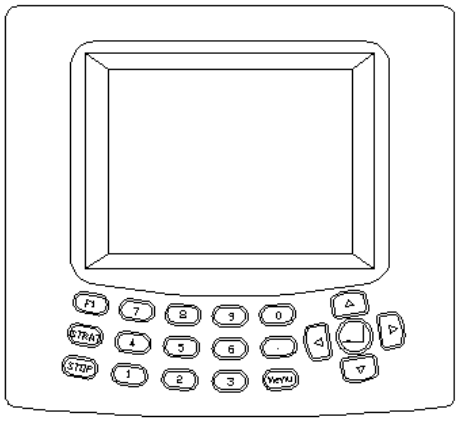
After fix the nut & sealing pad on the activated chromatographic column or testing column, insert the column along the inner hole of the injector and detector to the end, no gap is left at its top and then fasten the sealing nut;

Re-take down the cooling head of the injector and replace the injection shock insulator;

Testing of leak detection at the joint of the chromatographic column : In general, metal column is matched with stainless steel nut, brass or graphite squeeze ring. Metal squeeze ring has better tightness and longer operating life. Graphite squeeze ring is easy to use and convenient to seal, but during installation, the housing nut can not be rotated too tight in order to prevent the squeeze ring from loosing sealed function.

Glass column is commonly applied at lower column temperature, so brass nut is suitably matched to use. Silicone rubber or graphite material is commonly used for the sealing squeeze ring. Slightly install column and if the joint part of the column is deformed too much, the column should be replaced. If the deformation is not so serious, slightly loosen the fixed screws of the detector or injector oven, and after the chromatographic column installation is completed, re-fix these screws. When glass column is installed fastening the sealing nut should be very carefully, never wring the nut too tightly with spanner to prevent the breakdown of the glass column caused by temperature rise or strain change of the column tube, and it will be satisfied that the tightness is only guaranteed.

6.5、Keyboard operation



〔Fig6－6〕Instrument control panel

The operating method of the keyboard, function keys' setting, parameters' detection etc. operating procedures of Series GC-9790 Ⅱ Gas Chromatographs will be introduced in this section and its purpose is to help users familiarize and master the operating method of the instrument to bring the instrument into a full play as soon as possible.Its keyboard is located at the top of the gas line cabinet to let users be able to operate it and observe its display status conveniently. The arrangement plan of the keyboard is shown in Fig.6.6. There is a LCD screen on the panel whose function is to display various messages of the instrument. There are 28 operating keys. Brief description of the function keys ,as follow:

《0～9》-----Numeric keys;;

《 •》-----first time Decimal point key;second time clear key;

《START》-----Program run start key;

《STOP》-----Program run stop key;

《Menu》-----Menu key;

《F1》-----To clear the alarm;

《△》-----To move the cursor up;

《▽》-----To move the cursor adown;

《◁》-----To move the cursor left;

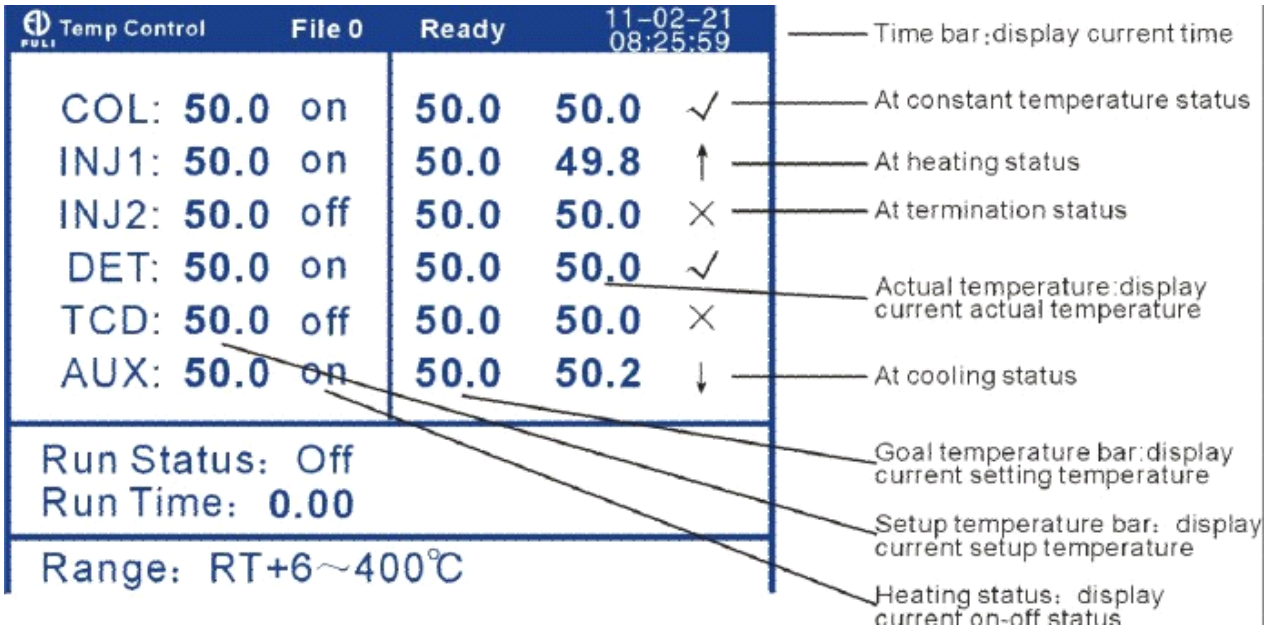
《 ▷》-----To move the cursor right;

《↵》-----Enter。

Turn on the instrument left-hand power switch,the LCD display holds to order bright.The computer begins firstly to enter self-checking program.After several second,the screen display is as shown in 〔fig 6.7〕 ,and after several second, as shown in〔 Fig 6.8 〕.

（When the cursor is at the following any interface of bottommost the condition, may return to this interface press key"0" . ）

〔Fig6-7〕



〔Fig6-8〕

This interface for temperature control , then cursor located at temperature setup bar. If let the detector heating up to 320℃，Just move the cursor to detector's temperature setting place，Press down in turn：《 3 》+《 2 》+《 0 》+《↵》key,then detector's setting temperature is320℃，Detector's goal temperature becomes 320℃ .If the input error can press key“ • ” twice to elimination ， reset temperature.Can use the same method setting oven、injector、detector temperature.Running time bar is from starting timing to shutdown,next time retiming.Heating bar，“0”mean off，“1”mean on。Let the cursor moves to the heating status bar, presses in turn《1 》+《 ↵ 》key will display“ON”, Press in turn 《0》+《↵》key，will display "OFF"。

When setup temperature must pay attention cannot surpass the protection temperature, instrument initial protection temperature is 400℃. When the instrument some temperature heating zone temperature parameter establishes improper or because each kind of reason has the instrument temperature to lose control. When reach to protective temperature,instrument heats up completely automatic shut-off the power source ， Buzzer warning ， and visual display alarm code number and error message ，This status has maintained until closes the instrument total power source (or artificial processing and eliminates warning status). The warning later please do not close the instrument total power source immediately, must examine the warning code number, the search reason and the error message, and according to error message solution breakdown.

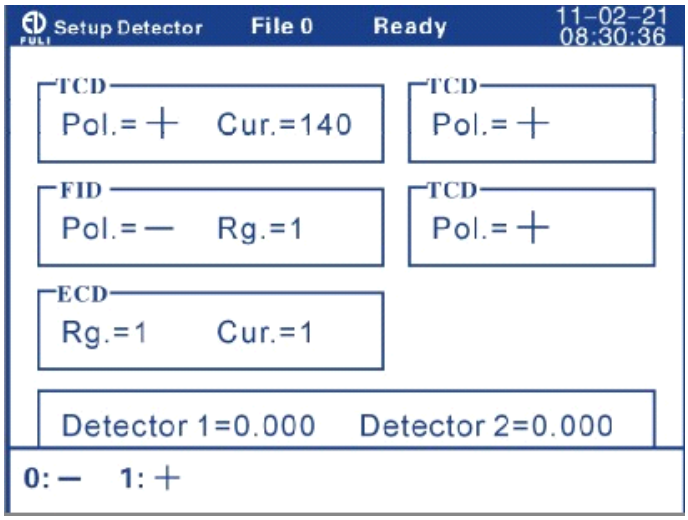
Can use heating protection reasonably in the instrumentation process, once temperature out of control, can to the instrument and the auxiliary equipment protects effectively, in order to avoid unnecessary loss.

《Attention》

The overheat protective temperature setup temperature must be set at 10℃ or 10℃ higher than its working temperature and less than 400℃. If it is less than the operating temperature or setup error, system will present the warning,protective temperature setup data also original data,the instrument will lie in protective status and abnormal operation.

Press《Menu》key，the status bar display 

, and press《▷》key，let cursor move to《Detctor》，And then press《↵》key，LCD be display〔Fig6-9〕。



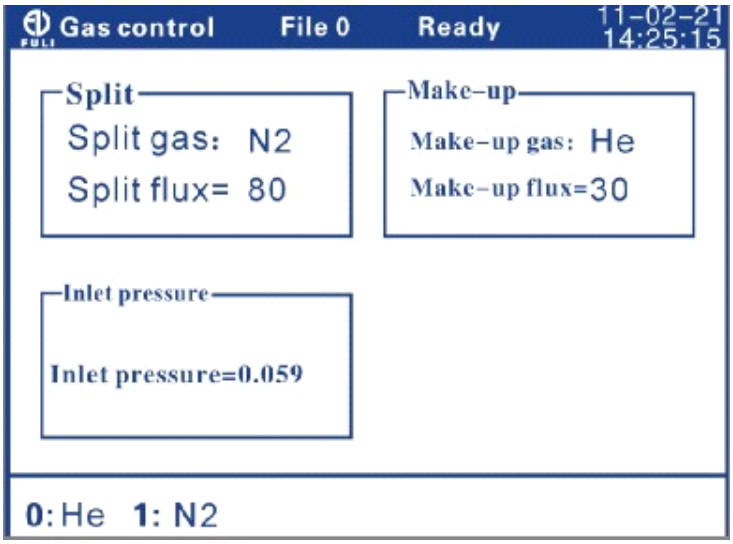
〔Fig6-9〕

When cursor located at TCD polarity，underneath status bar display:"0:-"，"1:+"，Press《0》+《↵》, input "-" Press《1》+《↵》, input "+" When cursor located at TCD current，underneath status bar display：TCDheater current 0～250mA,TCD heater current range（0～250）mA。FID polarity display is the same as TCD polarity"0:-"、"1:+"， FID sensitivity is 0 ～4， 0 means highest sensitivity 、4 lowest。ECD sensitivity is 0 and 1，0 means hight sensitivity、1 lowest，ECD current: 0=0.5nA、1=1nA 、2=2nA。FPD sensitivity is 0～3，0 means hightest sensitivity、3 lowest。NPD sensitivity is 0～3，0 means highest sensitivity、3 lowest。、When operation different detector，must chose corresponding polarity、current、range.Specific input method:the same as preceding text ，move cursor to you would like setup polarity、current or range location press in turn key 《figure》+《↵》.

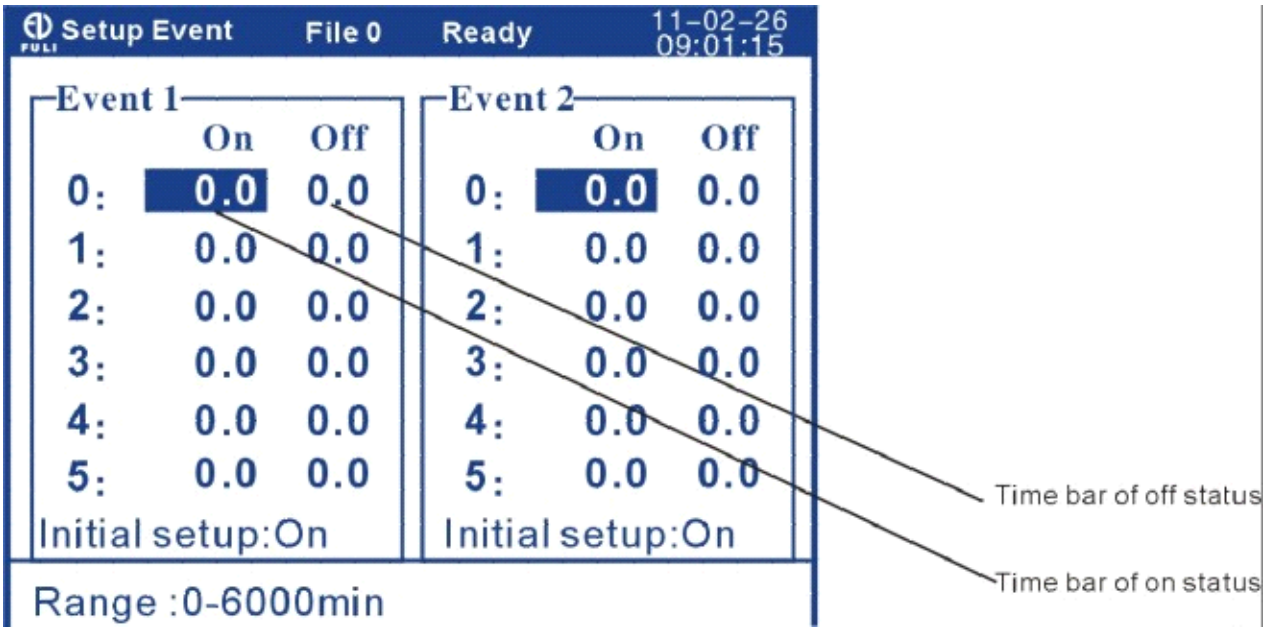
Press《Menu》key，status bar display as 

and press《▷》key，move cursor to 《Gas》, 

press《↵》key，enter into next interface：



This interface is the gas control interface，cursor located at split gas "N_2"，Press《0》+《↵》combination key input H_2，Press《1》+《↵》combination key input N_2。Split flux switch split needle valve；switch make-up needle vale get make-up gas flux.Pre-column pressure can switch carrier gas pressure maintaining valve.（flux unit :ml/min，pressure unit:Mpa）

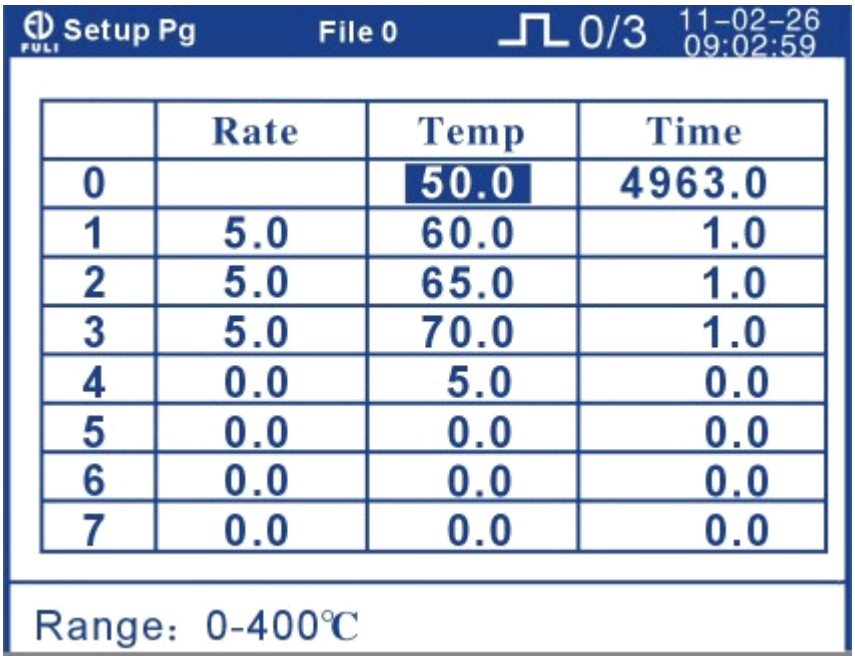


〔Fig6-10〕

Herein status bar 

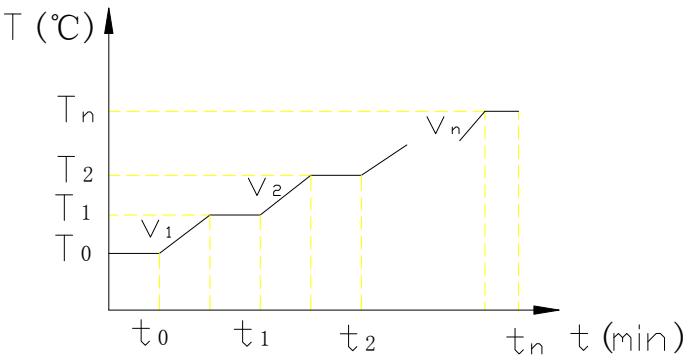
，press key《▷》，move cursor to《Event》，after that press《▷》key，LCD turn to [Fig 6-10]interface。This is event control setup interface，have event1 and event 2，event 1、event 2 all have 5 on and 5 off.Can proceed all setup，For example solenoid valve switch control etc.Move cursor to initial setup，Press《0》+《↵》combination key ，input on，initial setup is on，split always open ,split status.When initial setup off,split always close,splitless status.

After press key《Menu》，move cursor right to《Time Pg》，and press《↵》，LCD display〔Fig 6-11 〕 .As Figure 0 the status is the constant temperature (Initial temperature constant status).Temperature programming 7 ramps，constant temperature range（0～400）℃，time range（0～6000）Min，heating rate（0～40）℃/min.Program 1 display 5℃/min rate from 50℃ to 60℃，60℃ holding 1 Min.In the temperature program active status, interface display



〔Fig6-11〕

figure/figure、figure/figure 或 figure/figure mark，means located at constant temperature status，means located at heating up status，means located at cooling status，figure /figure：First figure means ramps，the other figure means temperature program total ramps.The column oven temperature program curve see〔Fig 6-12〕.



T1、T2…Tn means ramp 1、ramp 2… The n ramp is the final temperature，T0 means initial temperature.

t1、t2…tn means ramp 1、ramp 2…The n ramp final temperature holding time，t0 means initial temperature holding time.

V1、V2…Vn means ramp 1、ramp 2…The n ramp heating rate.

〔Fig 6-12〕Temperature programming time program schematic

Setup heating rate、constant temperature、constant temperature time，press《START》 key to start procedure or press twice key《Menu》，status bar display Start | Stop | Alarm，Next press key《↵》start procedure。

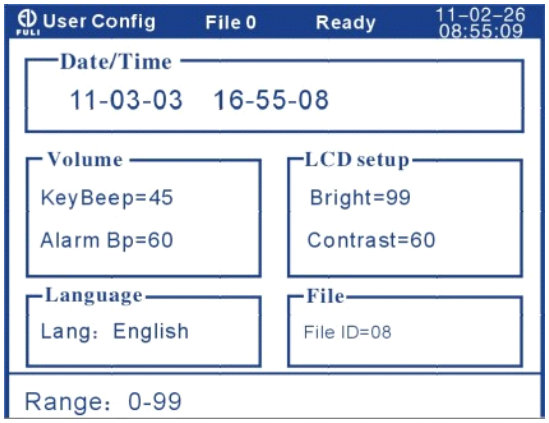
Press key《Menu》，status bar display TempCtrl | Detector | Gas | Event | Time Pg | AUX ↵，move cursor to 《 AUX 》， and press key 《 ↵ 》， The status bar display User | System | MaxTemp | Stopwatch，then cursor located at 《User》，after that press《↵》，that interface display〔Fig 6-13〕.

Keyboard beep setup：0～99，0 volume lowest、99 volume highest.Setup method：move cursor to key beep，press in turn《4》+《5》+《↵》，then key beep volume is 45.

Alarm beep setup：0～99，0 volume lowest、99 volume highest .Setup method is the same like key beep setup.

LCD bright setup：bright range0～99，0 darkest 、99 brightest.Setup method is the same like key beep setup.Also can press《Menu》+《▷》or《◁》increase or decrease brightness.

Contrast setup：contrast range:0～99，0 minimum、99 maximum.Also can press《Menu》+《△》or《▽》increase or decrease contrast.

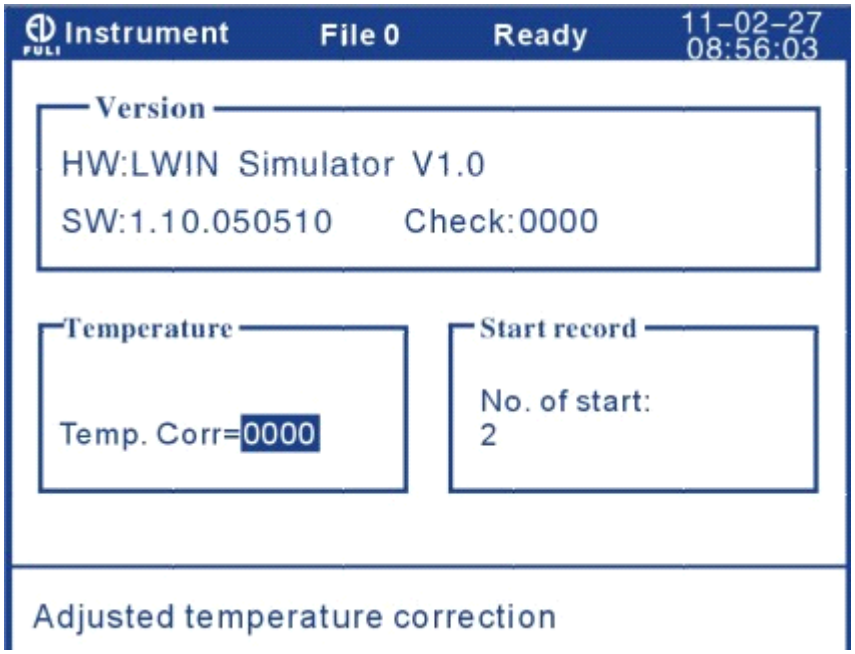


〔Fig 6-13〕

Language choice have English and Chinese，0=Chinese，1=English，Move cursor to language，press in turn 《0》+《↵》，then language choice is English.

File number：0～9，90 is clean up now、99 is clean up all.Input file number just move cursor to file ID and press in turn《8》+《↵》.And then file number is 8th，Then in the instrument all setup

parameter is the 8th file，If inputs other file number ，then the 8th file saved.If retrieves the 8th file, only need input file number 8 and press key enter.



〔Fig 6-14〕

Press key《Menu》，Then status bar display 

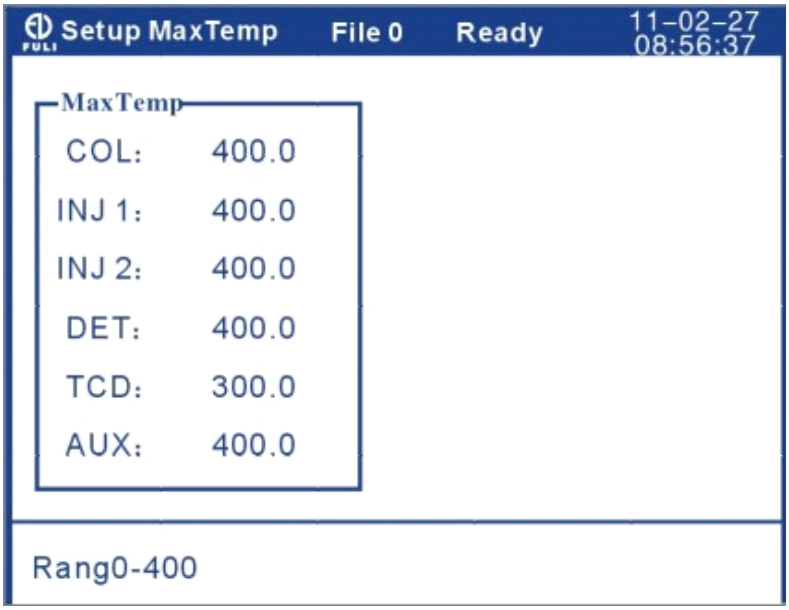
and move the cursor to 《System》，after that press key《↵》，LCD display〔Fig 6-14〕interface. This interface is instrument specification.User do not modify.“Start record”Has recorded the number of times which the instrument starts.

Press key《Menu》，status bar display 

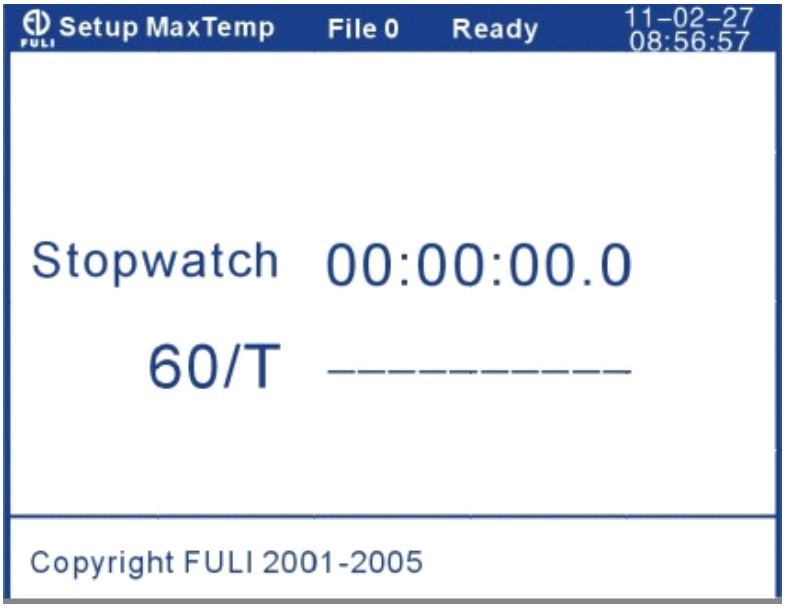
，and move the cursor to《Max Temp》and press《↵》LCD diplay〔Fig6-15〕interface.This interface is Maximum temperature.According to physical truth to setup column oven、injector、detector、TCD、AUX maximum temperature.Instrument system setup maximum temperature is 400℃. The instrument practical work's setup temperature requests to be lower than the protection temperature 10℃ or 10℃ below, otherwise the system auto-alarm.

Press key《Menu》，status bar display 

，move the cursor to 《Stopwatch》，and press 《↵》，LCD display〔Fig 6-16〕interface。



〔Fig 6-15〕



〔Fig 6-16〕

This interface is stopwatch setup ，First press《↵》，start timing，twice《↵》 stop timing and stop time simultaneously display 60 second dividing immediate time value ， Convenient user calculate minute flux. Third time press《↵》，clean up timing.or press key《△》start timing，press《▽》stop timing，press key《 ▷ 》clean up timing .When the cursor is at the following any interface bottommost status bar, may press key "·" return to this interface.

Press twice《Menu》，status display TempCtrl | Detector | Gas | Event | Time Pg | AUX ，then cursor located at《AUX》， move cursor to choice menu，press enter into any interface.

CHAPTER 7 GAS LINES

The stabilizing pressure valve, stabilizing flow rate valve, needle valve and the all other pipeline parts are installed in the gas line cabinet of the instrument and the regulating valves are installed on the gas line panel. Open up the small door of the gas line cabinet and the various gas flow adjustments can conveniently be adjusted. The configuration of the gas line parts is determined by the detector system. When TCD, ECD are applied the carrier gas flow rate control is only offered. While the other detectors are applied, besides that of the carrier gas, the control of hydrogen gas and air flow rate are offered as well. If the low temperature operating system, capillary injector, gas injection valve, column switching valve, supplementary gas valve and other auxiliary devices are added to the column cabinet, the gas line parts should relevantly be amended.

7.1 Gas line technological process of the instrument with basic configuration

Its flowchart is shown in Fig.7.1.

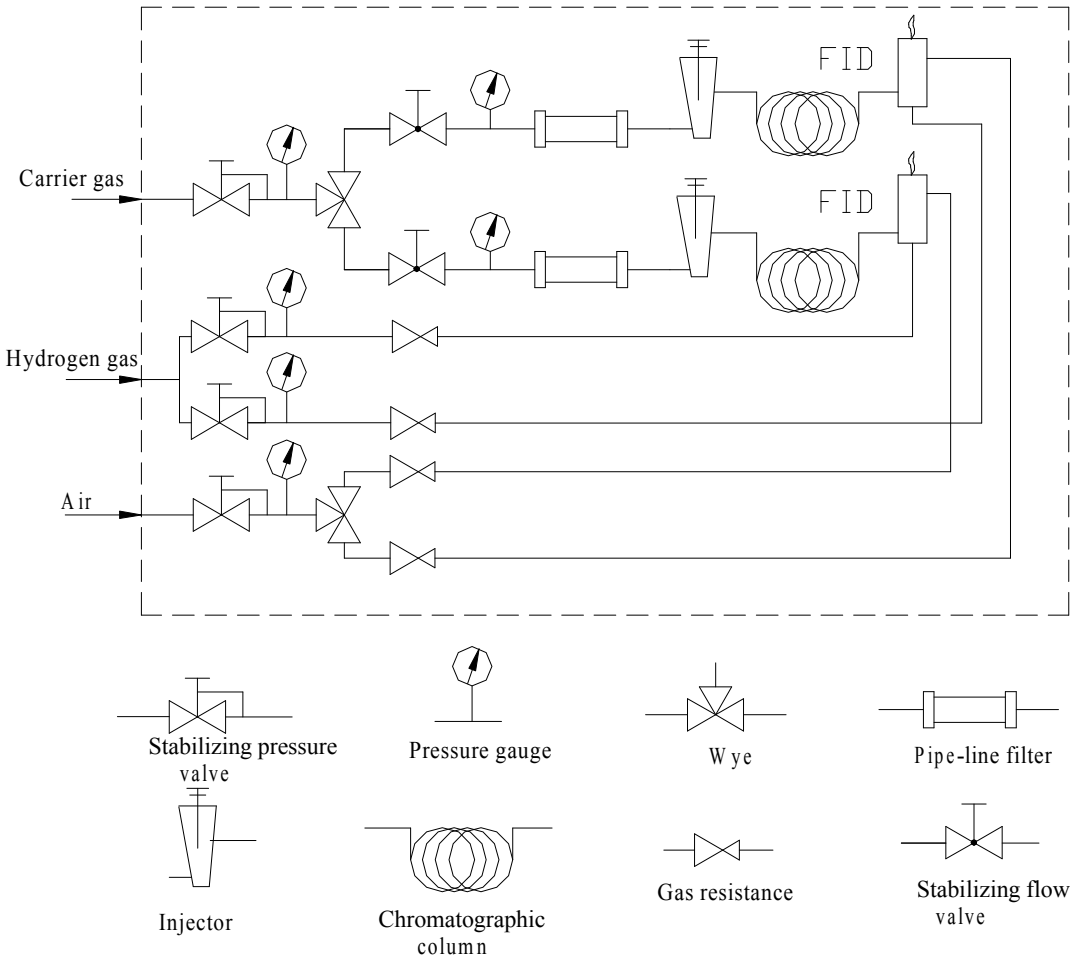


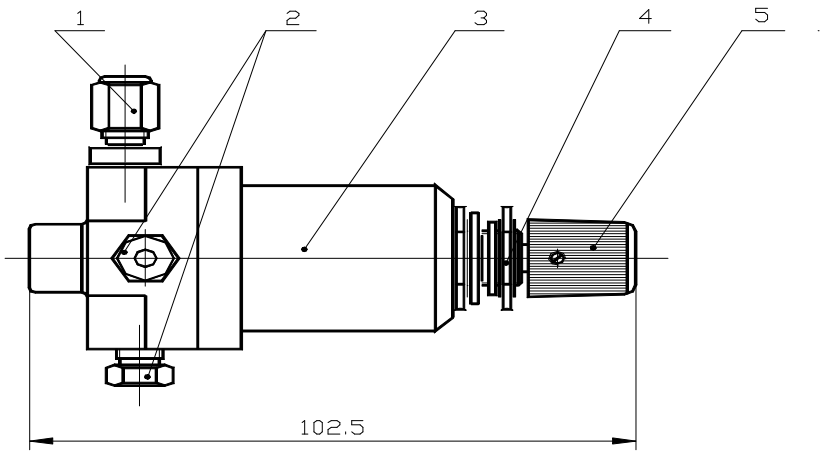
Fig.7.1 Instrument's gas line flowchart with basic configuration

7.2 Introduction to parameters of major parts

7.2.1 Stabilizing pressure valve

a. Technical parameters
   Max. input pressure : 0.6Mpa
   Max. output pressure : 0.45Mpa
   Max. output flow rate : 750ml/min.
b. Application and maintenance

There is a powder sintering filter fixed at the inlet of the stabilizing pressure valve, but the gas supplied to the valve is still required to be clean and without dust, mechanical impurities and mist etc.. When the valve is closed (The valve stem is turned anti-clockwise.) there should be no gas flowing out from its outlet. If a little bit of flow rate exists it denotes that the interior of the valve parts is dirty and the clean up is needed. We hope that user had better not disassemble the valve himself. The best way is to send back the valve to the manufacturer for repair or replacement. The outline structure of the stabilizing pressure valve is shown in Fig.7.2.



Item :	Descriptions :
1. Inlet	1. Panel installation hole □12 $^{+0.20}_{+0.10}$
2. Outlet	2. Inlet-outlet joint spiral burr M8×1
3. Valve body	3. Suitable for gas line diameter □2～3mm
4. Retaining nut	4. Gas line pipe diameter >ø3mm or adopting non-standard joint (Please put forward your demand for order)
5. Knob	

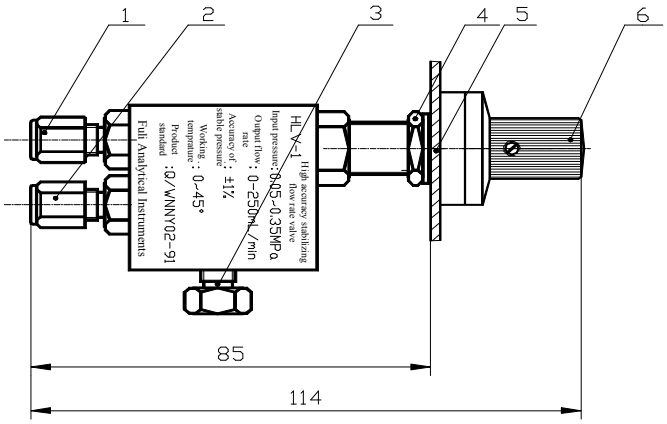
Fig.7.2 Outline structure of stabilizing pressure valve

7.2.2 Stabilizing flow rate valve

The carrier gas line system supplies the carrier gas through a stabilizing flow rate valve to the instrument with the basic configuration. The inlet of the stabilizing flow rate valve is matched with a stabilizing pressure valve in order to offer a constant gas source to ensure that the stabilizing flow rate valve is able to operate normally. The operating characteristic of the stabilizing flow rate valve is that when a constant inlet pressure is given the valve will offer a constant output flow rate not affected by

the restriction of the outlet resistance and the flow rate is only determined by the opening of the valve needle. This advantage can be utilized when the operation of programmable lifting temperature or replacing the chromatographic column is implemented. Although the resistance variation behind the valve may happen the flow rate value originally set will keep constant. Thus the adjusting procedure in the instrument operation can be simplified.

It can also be observed that the resistance of the chromatographic column will correspondingly vary and the pressure in front of the column will increase or decrease too due to the variation of the column temperature. Before leaving the factory, a curve of flow rate-adjusted number of turns for the stabilizing flow valve has been drawn (hydrogen, nitrogen gas). If the adjustment of the gas flow rate is needed, the correspondent scale value can be looked up on the curve to turn the handle of the valve stem to obtain the correspondent gas flow rate. The outline structure of the stabilizing flow rate valve is shown in Fig.7.3.



Items: Descriptions:

1. Inlet
2. Outlet
3. Outlet [connected to pressure gauge]
4. Retaining nut
5. Panel
6. Scale knob

1. Panel installation hole □12 $^{+0.20}_{+0.10}$
2. Inlet-outlet joint spiral burr M 8×1
3. Suitable for gas line diameter □2～3mm
4. Gas line pipe diameter >ø3mm or adopting non-standard joint (Please put forward your demand for order)

Fig.7.3 Outline structure of stabilizing flow rate valve

Specifications :

Maximum input pressure : 0.6Mpa

Maximum pressure drop : 0.5Mpa

Maximum output flow rate : 300ml/min.

7.2.3 Needle valve

a. Specifications

Maximum input pressure : 0.6Mpa

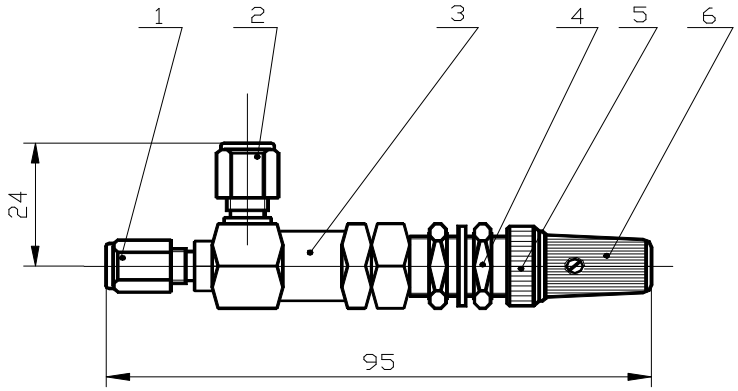
Maximum output flow rate : 350ml/min.

b. Application and maintenance

The gas supplied to the needle valve is required to be clean and without dust, mechanical impurities and mist etc.. When the needle valve is adjusted it is best not to adjust it to shut off status to prevent

the valve needle from damage. The outline structure of the needle valve is shown in Fig.7.4.

Note : The instrument with the basic configuration does not use needle valve. When the configuration is upgraded the needle valve is needed.



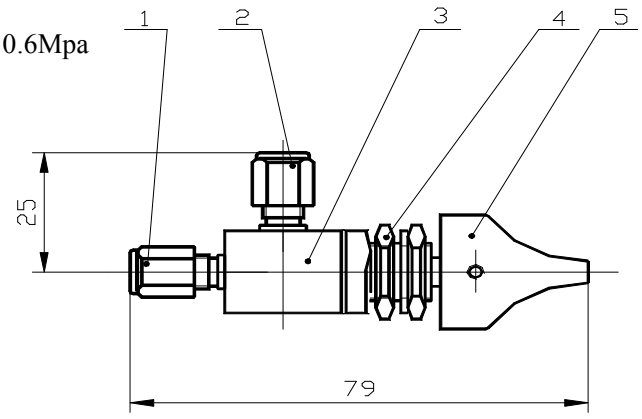
Items :	Descriptions :
1. Inlet	1. Panel installation hole □2 $^{+0.20}_{+0.10}$
2. Outlet	2. Inlet-outlet joint spiral burr M8×1
3. Valve body	3. Suitable for gas line diameter □2～3mm
4. Retaining nut	4. Gas line pipe diameter >ø3mm or adopting non-standard joint (Please put forward your demand for order)
5. Retaining ring	
6. Knob	

Fig.7.4 Outline structure of needle valve

7.2.4 Switch valve

Specifications

Maximum input pressure : 0.6Mpa



Item :	Descriptions :
1. Inlet	1. Panel installation hole □2 $^{+0.20}_{+0.10}$
2. Outlet	2. Inlet-outlet joint spiral burr M8×1
3. Valve body	3. Suitable for gas line diameter □2～3mm
4. Retaining nut	4. Gas line pipe diameter >ø3mm or adopting non-standard joint (Please put forward your demand for order)
5. Knob	

Fig.7.5 Outline structure of switch valve

7.2.5 Gas resistance

Adopting the fixed gas resistance in the gas line of the instrument has many advantages. The gas resistance, which is fitted at the outlet of the stabilizing pressure valve, can increase the range of the output pressure and adjusting accuracy of the flow rate. In addition, the stabilizing pressure valve can be controlled in the working region with higher accuracy of the stabilized pressure. In conventional operation, operator can execute the operation according to the gas flow rate given by the instrument to simplify the operating procedure. The fixed gas resistance is applied to the hydrogen gas and air lines for the instrument. Its assembly structure is shown in Fig.7.6. The gas flow rate under the rated pressure is :

Hydrogen gas (H_2) : Pressure 0.1Mpa, flow rate 30±2ml/min. ;

Air : Pressure 0.03Mpa, flow rate 300±5ml/min. ;

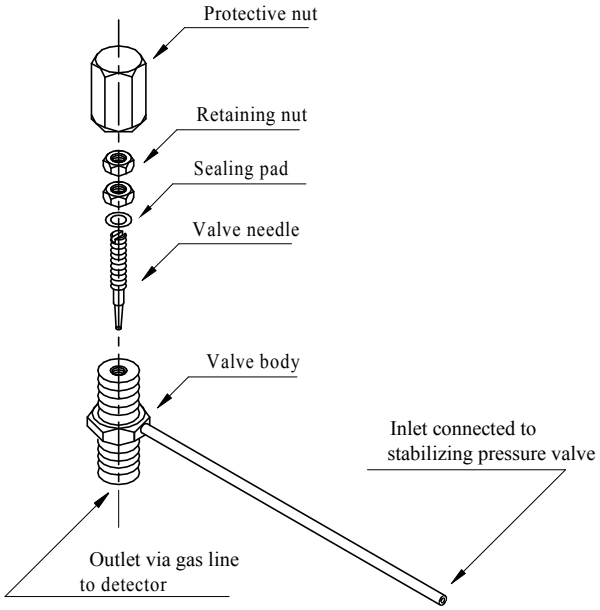


Fig.7.6 Gas resistance assembly structure

7.2.6 Relief pressure valve

The relief pressure valve is a device which can reduce the high pressure gas contained in the steel cylinder down to 0.1~0.6Mpa low pressure gas. The output low pressure gas can be adjusted according to the requirement and meanwhile the low pressure of the output gas can almost be kept unchanged under the variation of the high pressure of the gas and the flow rate fluctuation.

Therefore the function of the relief pressure valve is reducing pressure and playing a certain role of stabilizing pressure.

In general, the relief pressure valve is not matched with the instrument, user can order according to the following technical requirements.

Technical requirements :

a. Maximum inlet gas pressure is 15Mpa and maximum working pressure should be two times more than the output low pressure;
b. Range of the output working pressure : 0.1~0.6Mpa;

c. Maximum output gas flow rate > $40m^3$ /hr.;
d. Accuracy of input & output pressure gauge : grade 2.5;
e. When the fluctuation of the input pressure and output flow rate is tolerated in the range, the fluctuation of the output pressure should not be more than 1.5% of the maximum output pressure;
f. Inlet joint spiral burr size : G5/8 in.

Notice of the option of relief pressure valve :

a. The relief valve structures between those of common gas and inflammable gas (H_2) are all the same, but only their traverse directions of the connecting spiral burr are in sharp contrast;
b. In general, its output pressure of 0~0.6Mpa is suitable and the higher the pressure, the lower the accuracy of the stabilizing pressure;
c. In general, the maximum output flow rate (air) for GC is 60 l/hr. which is one-thousandth of the output flow rate of the relief pressure valve sold in the market, so should purchase that valve with as small output flow rate as possible.

7.2.7 Gas purification

The purpose of gas purification is to take off H_2O, O_2 , harmful organic substances etc. and mechanical impurities.

The gas purifier is a special device matched with the instrument for purifying the working gases. The device has three gas line paths and each of them is respectively independent. Its structure and applied method can be referred to the operating manual of Type GPI – 2 gas purifier.

Application and activation of purification materials in common use :

a. Activated carbon :

Before applying the purchased product, its particles should be screened out and its should be immersed with benzene several times to take off sulfur, tar etc. substances, then ventilate superheated steam of 380℃ to flow the activated carbon until its milky white color disappears. Keep it in a holding bottle and bake it at 160℃ two hours before application.

b. Silica gel

Before applying the purchased silica gel, its particles should be screened out and it should be immersed with 6N hydrochloric acid 1-2 hours, then immersed with distilled water until it has no chlorine ions (testing with $AgNO_3$). Put it into oven for baking 6~8 hours and then keep it ready for application. Before application, ventilate gas at 20℃ to activate 2 hours.

c. Molecular sieve

It is used to screen out the particles of the purification material and the screened material is baked at 350~580℃ 3~4 hours.(The maximum activation temperature will not be beyond 600℃.)

d. 105 catalyst

105 catalyst is a kind of palladium in oxygen out catalyst. The activation method is that put the catalyst into a deoxidizing tube, let the catalyst dewater two hours at 360℃, cool it to ambient temperature, ventilate inactivated hydrogen into the catalyst to deacidize one hour, and let the hydrogen of 1% oxygen pass through the catalyst once more and finally the oxygen content of the catalyst can be reduced to 0.2ppm.

e. Activated copper catalyst

This catalyst presents brown strips. Before application, ventilate hydrogen to activate in 300℃~400℃ and under this temperature the oxygen in nitrogen can effectively be taken off to let the oxygen

content of the catalyst be reduced to less than 10ppm. When the color of the catalyst becomes black it denotes the reactivation is needed.

f. Type silver X molecular sieve

Both Type 201 and 202 silver X molecular sieves are multifunctional catalysts whose taking off oxygen performance is especially outstanding. Type 201 catalyst has the ability to take off not only the paucity oxygen in hydrogen but also the paucity oxygen in nitrogen and noble gas under atmospheric temperature. Before application, it is needed to activate at 100~160℃, use hydrogen to blow and purge slowly and to make type silver X molecular sieve be deacidized in metal status for use. If the catalyst is inactivated hydrogen can be ventilated to deacidize. After deacidize over ten times it is necessary to activate at higher temperature to take off the moisture.

CHAPTER 8 INJECTOR

The injector which is matched with the instructor with the basic configuration can be applied to column head sampling or rapid vaporous sampling (The applied chromatographic column is of 3~5mm diameter.). Both metal column or glass tube can be applied with the injector. The other kinds of injectors can be referred to their operating manuals respectively.

8.1 Technical parameters

Injector body : Outer diameter of chromatographic column 3~5mm, length 122mm;
Material : Full stainless steel injector body, aluminum-alloy heating block;
Injection head : Aluminum cooling head assembled with stainless steel coned guiding vent of syringe needle;
Chromatographic column bush of column head sampling : Stainless inside diameter 3~4mm;
Chromatographic column bush of rapid vaporous sampling : Glass outer diameter 5mm;
Injector pad : Silicon rubber, outer diameter 5mm;
Installation manner : Clamped in injector cabinet;
Temperature control : Keyboard setting, microcomputer control;
Heating power : 150w;
Sensor : Ceramics platinum resistor R_0=100Ω;
Temperature range : Higher than ambient temperature plus 20℃~350℃, increment 1℃;
Temperature stability : Temperature fluctuation less than ±0.5℃ within 24 hours.

8.2 Injector installation

Two injectors can be installed in the packed column sampling system of the instrument with the basic configuration and design of the injector ensures for the instrument an operating mode of vertical sampling. During applying the injector, except that the sampling pad, which is consumable, often needs to be replaced (After having been applied many times, the needle pore of the injector has enlarged, so that if the leak phenomenon happens the pad should be replaced in time.), the injector, in general, needn't be disassembled and maintained.
The basic structure and installation method of the injector can be seen in Fig.8.1, in terms of the application & installation method of the injector chromatographic column and bush can be referred to Fig.6.5 of chapter 6.

8.3 The temperature option of the injector

The temperature option of the injector will highly affect the peak pattern and component separation of the sample. If the temperature is too low, the delayed peak will happen and if the temperature is too high, the leading edge of the peak waveform will be erect or the chromatographic peak caused by the sample decomposition will happen. The applied temperature of the injector can generally be determined on the basis of the sample composition, column temperature and applied amount of the sample. In order to ensure that all the components of the sample would be vaporized for an instant under not decomposing, properly increasing the injector temperature, especially during applying

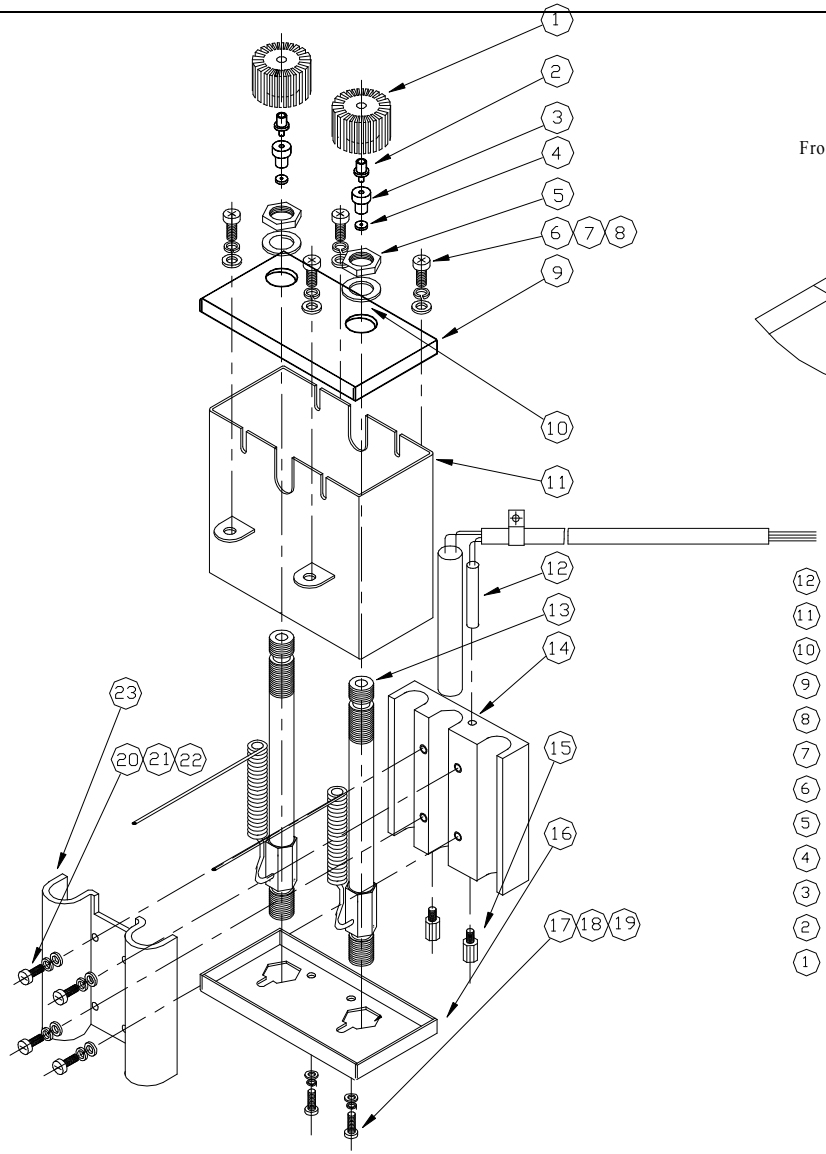
relatively large amount of sample, is comparatively advantageous. The stationary phase which is in front of the chromatographic column, will be decomposed and skinned to cause an unstable baseline and presenting a “ghost peak” (especially, when the operation of a column head injector is applied.). These phenomena should be prevented as far as possible.
In general, the injector temperature should be controlled at 30℃~50℃ higher than the boiling point of the component.

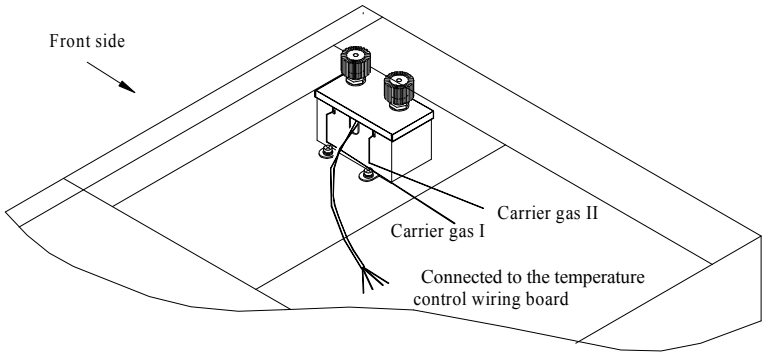
8.4 Application of injection shock insulator

The unstable baseline and ghost peak will appear in high sensitivity analysis because of the volatile of the interior organic substance of the shock insulator and sample pollution. Especially during the operation of capillary and programmable lifting temperature, these phenomena are sticking out. In order to minimize the analytical interference caused by the shock insulator, during higher application requirement, the shock insulator should be preprocessed before its application. The processing method is that put the shock insulator into the column oven, have it baked at 200℃~250℃ for 8 hours, and then it can be applied after cooling. Its service life of the baked shock insulator may be reduced and the leakage should be prevented in operation.
In operation of high temperature, before lifting temperature, the injector head should not be screwed too tight in order to prevent the injection needle from not being able to stick into or bending after temperature rising. In severe case, the affinity of the injector head may happen, so that the head can not be turned.

8.5 Injector purge

A common high temperature injector is only suitable for the sample with good stability and not reacting chemically with metal surface. If the sample contains high boiling point substances and the components with thermal instability or corrosiveness, the residuals in the sample will precipitate or be carbonized in the injector. Along with time elapse, the precipitation-hardening will occur and then become carbonic substance. Once this substance makes contact with other sample the corresponding chemical reaction will happen. It is possible that the waveform of the peak may be changed or the redundant strange peak may appear, so that the quantitative & qualitative repeatability of the instrument will become worse. Based on the application of the instruction and the variation of analyzed samples, purging in time is needed.
The commonly adopting purging solutions are acetone, ether, n-hexane and absolute alcohol etc.. After purging with the solution, purge with distilled water, dry up with a blower, put it on the instrument to be ventilated for 30 minutes and heat it at 120℃ for 4~8 hours and then the injector can be used again.





Scheme of the instrument installation position (for the injector)

12	Heating wire bunch	23	Heating bar hold down
11	Cabinet body	22	Flat washer φ3
10	Retaining washer φ10	21	Spring washer φ3
9	Cabinet cover	20	Hold down retaining screw M3
8	Flat washer φ4	19	Flat washer φ3
7	Spring washer φ4	18	Spring washer φ3
6	Ⅱ 4 Retaining screw	17	Heating block retaining screw M3
5	M10 × 1 Retaining nut	16	Cabinet body bottom plate
4	Bulkhead	15	Carrying screw
3	Injection pad	14	Heating block
2	Guiding sleeve	13	Injector body
1	Injector head		

Fig.8.1 Scheme of injector installation method

CHAPTER 9 FLAME IONIZATION DETECTOR (FID)

9.1 Outline

The operation, installation and application notice etc. contents of the Flame Ionization Detector (FID) are described in this chapter. The FID is installed in the detector oven of the instrument and its double gas line configuration can be matched with and equipped for two detectors. The control part of FID is installed in the electrical appliance box on the upper side of the gas line cabinet. The basic structure and electric control part can be referred to Fig.9.1 and Fig.9.2 respectively.

The gas flow rate adjustment and control setting method can be referred to the gas line adjustment part in Sec.5.6.

1. Technical specifications of FID:

Under the normal working conditions, when 5% OV – 101 packed column is adopted, the carrier gas is high purity nitrogen (N_2) and take hexadecane as a tested sample, the specifications of the detector should be satisfied with the following requirements :

Linearity range : more than 10^6;

Measuring threshold : less than 2×10^{-11}g/s;

Noise : less than 2×10^{-13}A[0.02mv];

Drift : less than 4×10^{-12}A/h[0.4mv/hr.];

2. Technical parameters of FID amplifier :

Range setting : 0 ---1×10^{-12}A/mv;

1--- 1×10^{-11}A/mv;

2 --- 1×10^{-10}A/mv;

3 --- 1×10^{-9}A/mv; (The linearity for all ranges is ±5%)

Polarization voltage : 180±10V

9.2 Operational principles

The operational principles of FID are described in this section as follows. The analyzed sample is put and burned in hydrogen flame to generate ion flow. Its mechanism of ionization is the chemical ionization. The ion flow caused by ionization is detected under the external electric field role. Its electric signal level denotes the content of the analyzed sample. The operational principle scheme of FID is shown in Fig.9.3.

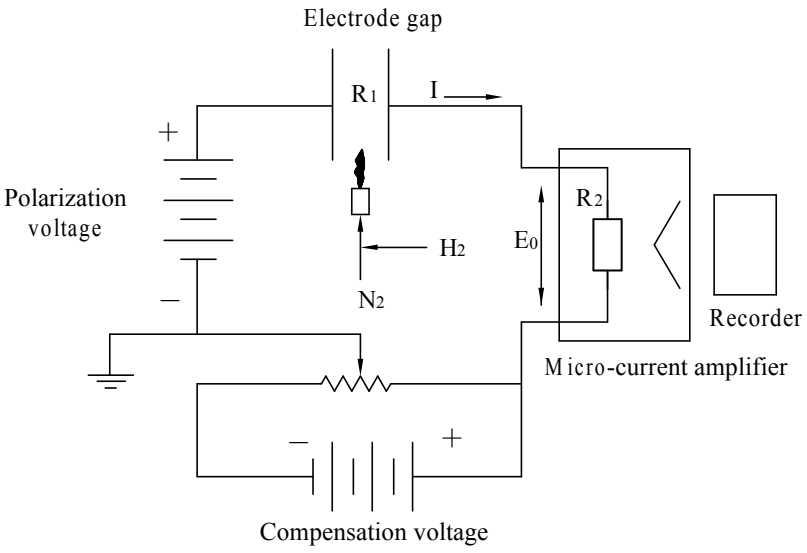


Fig.9.3 Scheme of FID operational principles

The carrier gas carrying the sample components flows out from the chromatographic column and passes through the electrode gap. Some molecules of the gas is ionized by the hydrogen gas flame to generate a number of charged particles and under the generate a number of changed particles and under the role of the electric field, a current I forms. Then the current flows through the gap and a measuring resistor R_2 to generate a voltage E_0 across R_2 . The voltage is supplied to a micro-current amplifier and the amplified signal is successively supplied to a recorder. The electrode gap can be considered as a variable resistor whose resistance is decided by the number of the charged particles in the gap. Only if the pure carrier gas (In practice, the carrier gas always contains organic substances and stationary liquid washed away from chromatographic column etc. substances.) passes through the electrode gap, a constant convection current I which is called the base current or background current will form. When FID is applied, the base current should be small as far as possible. Only in the small base current case, can the slight current variation be detected. When the carrier gas is only supplied, in order to offset the base current influence and to make the input/output of the amplifier be zero, a compensation voltage, which is equal to $I \times R_2$ and whose polarity is in contrast with voltage E_0 , is supplied at the terminal of the amplifier and at the moment, the positive voltage E_0 is offset by the negative compensation voltage, so that a straight line is drawn on the recorder. When the carrier gas with the detected sample passes through the electrode gap, the molecules of the components are ionized and the charged particles rapidly increases to make the variable R_1 caused by gas conduct have an increment R_2, so that a signal chromatogram is drawn on the recorder.

The normal operation of FID needs three kinds of gases, hydrogen gas, air and carrier gas. The performances of FID depend on the proper option of these gases flow rate. If attempt to obtain good stability and sensitivity, their purity and pressure range option should be consistent with the requirements of tables 5-1.

9.3 Basic characteristics

The interior structure and installation method of FID can be referred to Fig.9.1. A fully-enclosed structure is adopted for the detector to reduce the influence of the detector operation caused by the external gas flow variation. The non-metal nozzle structure has good chemical inertia. The nozzle diameter is 0.5mm and the nozzle-end at the top of the nozzle is sealed with special material and non-mental. The polarization voltage source is clamped at the nozzle-end and such a design not only makes the ion flow conduct well, but prevents the analyzed sample from generating thermal decomposition phenomenon.

The design of the cylinder body volume of the detector guarantees the high efficiency of the gas combustion. The carrier gas and hydrogen gas are mixed in the inside of the nozzle, while the burning acceleration gas goes into the combustion chamber around the nozzle, thus benefiting sufficiently to mix the gases so that it will create the sufficient conditions for high efficient combustion and not being extinguished easily. After detection, the gases pass through the drainage port to be emptied, meanwhile the gases also play a role of purging the cylinder body of the detector, so that it strengthens the detector's ability to resist pollution.

The design of the detector ensures the chromatographic column installation is vertical. There is only the naked stainless steel join surface with diameter 1.5mm and length 2mm between the nozzle and chromatographic column and when the nozzle and the glass packed column are combined in the analytical system, it's effective to reduce the absorptive function of the metal surface against the sample.

9.4 Application scope

The FID is in response to the nearly almost organic compounds, but it is in little or no response to H_2, He, Ar, Kr, Ne, Xe, O_2, N_2, CS_2, COS, H_2S, SO_2, NO, N_2O, NO_2, NH_3, CO, CO_2, H_2O, $Sicl_3$, SiF_4, HCHO, HCOOH etc. Because the detector is in no response to water and air it is specially suitable for detecting the water phase sample containing biological substances and air pollutants. In addition, because the sensitivity response to CS_2 is low, CS_2 becomes a very good kind of solvent for FID to detect the analyzed sample.

In the quantitative analysis, the detector sensitivity variations to various hydrocarbon materials are very close, therefore during the analysis for the petroleum components etc. hydrocarbon materials, the normalizing computation directly based on the peak area can be adopted instead of the quantitative correction factor.

FID belongs to the type of mass flow rate sensitive detector and it not only has the advantages of the high sensitivity & wide linear range, but has the relative insensitivity to the change of the operational conditions and good stability. So it is specially suitable for the conventional micro or macro analysis. Because of the rapid response, FID can be matched with the capillary analytical technique to complete the fast trace analysis. Therefore, FID is one of the most comprehensively applied detectors in GC.

9.5 Selection of gas flow rate conditions

The gas proportioning of the optimum point not only has high response value, but has the least response influence caused by the gas flow rate variation. Therefore, the optimum gas proportioning can make the quantitative analytical error reduced, promote the instrument stability and be advantageous to micro component analysis.

9.5.1 The proportion between carrier and hydrogen gas

The carrier gas optimum flow rate depends on the optimum separation condition of the chromatographic column and the hydrogen optimum flow rate should be based on the carrier gas to choose reasonably. In general, the proportioning between the two gases is 1.1 ~ 1.2. The relationship between the hydrogen flow rate and sensitivity can be seen in Fig.9.4. The optimum hydrogen flow rate can be decided through an experimental method, that is, after a little bit of adjusting the hydrogen flow rate, the injector syringes a certain quantity sample and repeatedly adjust to compare the S/N ratio of the detector to find out the optimum flow rate. Alternatively, it can be chosen through the relationship between the hydrogen flow rate and base current. During the instrument operation, the certain amount of the organic substances in the carrier gas and running off of the stationary phase paucity always exist, of course, under the optimum hydrogen flow rate, they cause the maximum base current to be displayed. Thus the optimum hydrogen flow rate can be chosen.

The operating method is that under the proper sensitivity, choose rather small (or large) hydrogen flow rate, adjust the recorder in an available range and at the moment, slowly increase (or decrease) the hydrogen gas flow rate to make the recorder pen move in a single direction. Once an inflection point appears during moving, this flow rate is the optimum one.

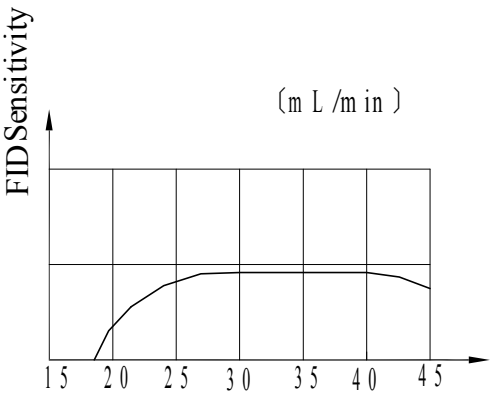


Fig.9.4 Relationship between FID sensitivity and H_2

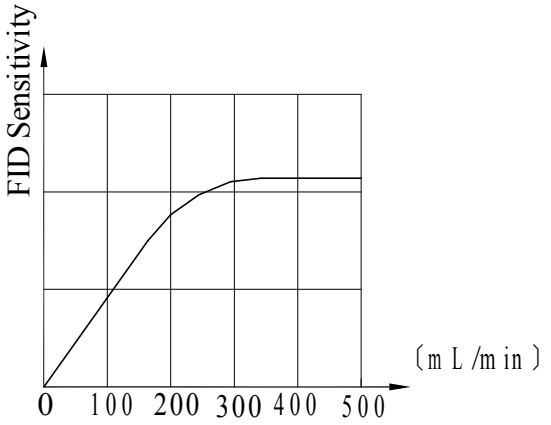


Fig.9.5 Relationship between FID sensitivity and air

9.5.2 The optimum air flow rate

The function of air in the detector not only offers the burning acceleration gas for the flame, but offers the purge function to wash away the gas combustion material. When the air flow rate is comparative small, the sensitivity increases along with the increase of the air flow rate and after it arrives at a certain point, the sensitivity will almost be unchanged if the air flow rate increases continuously. In order to let the air play a role of purging sufficiently, the selective principle of the optimum air flow rate is that under the unchanged sensitivity along with the flow rate variation, the flow rate for the moment plus 50ml/min. approx. is the right air flow rate. If the air flow rate is too large, the air will blow the flame to cause the rather high baseline noise, so that it is easy to produce the irregular response or flameout phenomenon. The relationship between the FID sensitivity and air flow rate can be seen in Fig.9.5. In general, if the carrier gas flow rate is 30ml/min., the air flow rate is 300~400ml/min. and the hydrogen flow rate obtained through an experiment is 30ml/min., the detector's maximum response value to propane should appear. Therefore, when the hydrogen flow rate, in general, is 30ml/min. approx., while the air flow rate is 10 times of the hydrogen flow rate, the flame is more stable and the ionization efficiency is the highest.

9.6 Operation

The assembly and disassembly sequence of FID on the instrument can be referred to Fig. 9.1. In general, before leaving the factory, the instrument has been adjusted and has strictly been tested. After the instrument has arrived at user, been normally installed, and the gases are ventilated to implement leak detection and then the instrument can normally be applied. Only when the interior of the detector system is severely polluted by the sample, in general, user should not disassemble the detector to prevent its parts from polluting the instrument, otherwise it will cause the instrument not to work normally. Disassembling the detector must be done by the skillful and qualified operators or ask the manufacturer for help. The basic operating procedures of initiating the instrument are described as follows :

1. Leak detection of the carrier gas line in front of the injector outlet :

First, use a blinding to seal the injector outlet, ventilate the carrier gas, and adjust the stabilizing flow rate value of the gas line to let the indication of the pressure gauge be fixed at 0.4Mpa, then close the

shutoff valve of the carrier gas line of the purifier to observe the gauge for half an hour and the gas pressure should be dropped less than 0.005Mpa.

2. Installation of chromatographic column :

The one end of the column connected to the injector should first be installed and ventilate the carrier gas of 0.4Mpa to measure the gas flow rate being consistent with the requirement at the outlet of the chromatographic column and purge the column at least half an hour in the open status (If the chromatographic column is inactivated, the column should be processed according to the activation method.), then connect the other end of the column to the detector.

3. Instrument initiation :

Turn on the power switch, heating switch and after the instrument self-checking is executed, make sure every thing is all right. Then it is ready for user to set the parameters. At the moment, if user would not like to set the parameters, the instrument will operate on the basis of the last parameter setting and the instrument is in the status of the temperature lifting (or initialization).

4. Detector ignition :

After the temperature of the instrument is stable, open up the recorder to adjust the recording stylus to a proper position. In accordance with the gas pressure-flow rate curve given by the instrument, adjust the respective gas flow rate control valve, set the hydrogen gas flow rate at 30ml/min. & air flow rate at 150ml/min, and then ignite at the gas relief port of the detector with an electronic igniter (referred to Fig.9.6). For the moment, observe the recorder. After the gas is ignited, the signal should rapidly increase. If the displayed line returns to the original base line position, it denotes the flame is not ignited. Then properly increase the hydrogen gas flow rate and ignite once again. If it still does not work, check the following events :

a. The hydrogen and air gas lines are connected whether they are correct and unblocked;

b. Check whether the connection of the igniter cell is correct;

c. Put a small mirror near the gas relief port of the detector to observe whether there is a condensation phenomenon. When the condensate water appears it denotes that the gas has been ignited. After the flame is ignited, adjust the air flow rate to 300ml/min. According to the method mentioned above, select the optimum gas proportioning condition.

After the adjustment is completed, ensure that the instrument has a certain stable period of time and how long the period of the time to be selected reasonably depends on the sensitivity of the instrument and its applied scope.

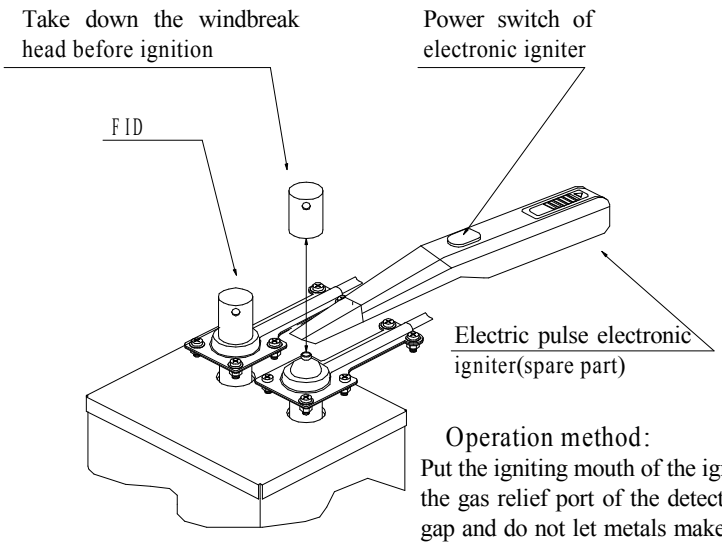


Fig.9.6 Scheme of detector ignition

<CAUTION>

1. In order to prevent the hydrogen accumulation from causing an accident, only before ignition can the hydrogen gas source be opened. After the instrument is shut off the hydrogen gas source must be closed in time.
2. When igniting, do not take down the collector to look in the cylinder body of the detector.
3. The FID operating temperature is at least 50℃ higher than the column oven temperature and can not be lower than 150℃ in order to prevent the generation of the condensation phenomenon.
4. During assembling and disassembling the parts, do not directly contact the parts with hand. At high temperature, do not wear plastic gloves to operate.
5. When the detector operates in high temperature status, although the instrument is turned off for a short time; the detector surface is still hot , do not touch the surface with hand to prevent yourself from scald.

9.7 The method of checking before acceptance

9.7.1 Noise and drift investigation

Operating conditions :
a. Carrier gas : N_2, 30ml.min.;
b. Hydrogen gas : H_2, 30ml/min.;
c. Air : Air, 300ml/min.;
d. Column oven temperature : 140℃;
e. Injector temperature : 200℃;
f. Detector temperature : 220℃;
g. Range : One position [1×10^{-11} A/mv];
h. Full scale of recorder : 1mv, paper speed 60cm/min.;
I. Measuring chromatographic column : 5% 0V – 101 stainless steel, outer diameter of 3mm, L(length) of 500mm.
After the instrument is in stable status, continuously records for an hour and the base line should be consistent with the requirement. If the base line is not qualified, the column oven temperature and the bake time can be increased.

9.7.2The operating conditions of sensitivity:

After the stability specifications are qualified, continue to operate according to the every item of the stability.
a. Range amendment : one position (1×10^{-11} mv/A);
b. Measuring sample : nC16 of ISO – octane (0.3 μ g/ μ l), injection quantity 1 μ l. Continuously inject the sample three times, take the average area and substitute it into formula 9.1 to calculate the measuring threshold of the detector :

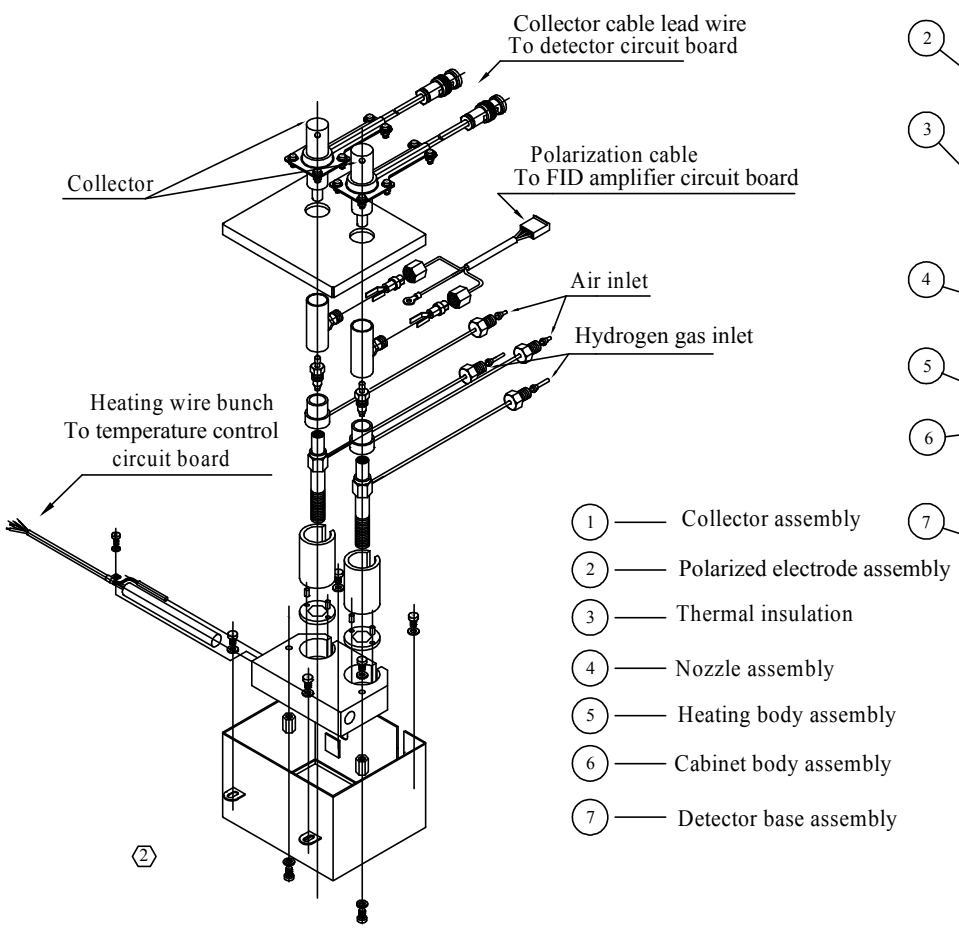
$$Mt = \frac{2R_n \cdot m}{A} [g/s] \qquad (9.1)$$

Where : Mt--- Measuring threshold of FID [g/s]

R_n --- Baseline noise [μV]

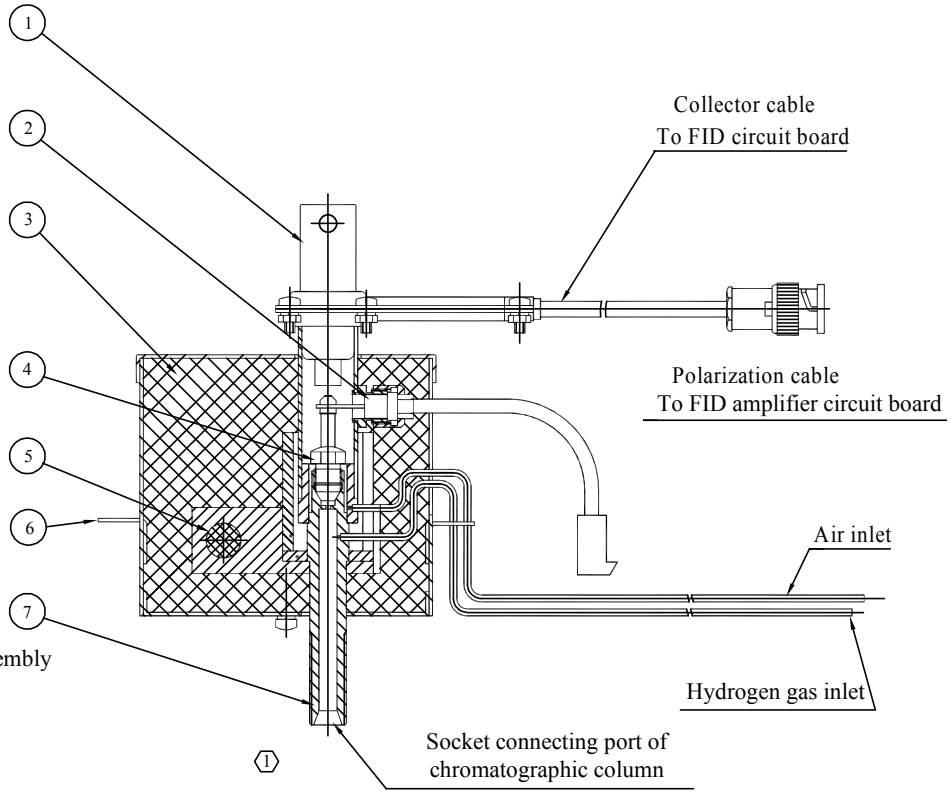
m --- Mass of injection sample [g]

A --- Average peak area [μV・S]



Assembly structure scheme

1 —— Collector assembly
2 —— Polarized electrode assembly
3 —— Thermal insulation
4 —— Nozzle assembly
5 —— Heating body assembly
6 —— Cabinet body assembly
7 —— Detector base assembly



Operation mode assembly scheme : When the double detectors are adopted the one near the column oven door is detector 1.

Fig.9.1 FID assembly structure scheme

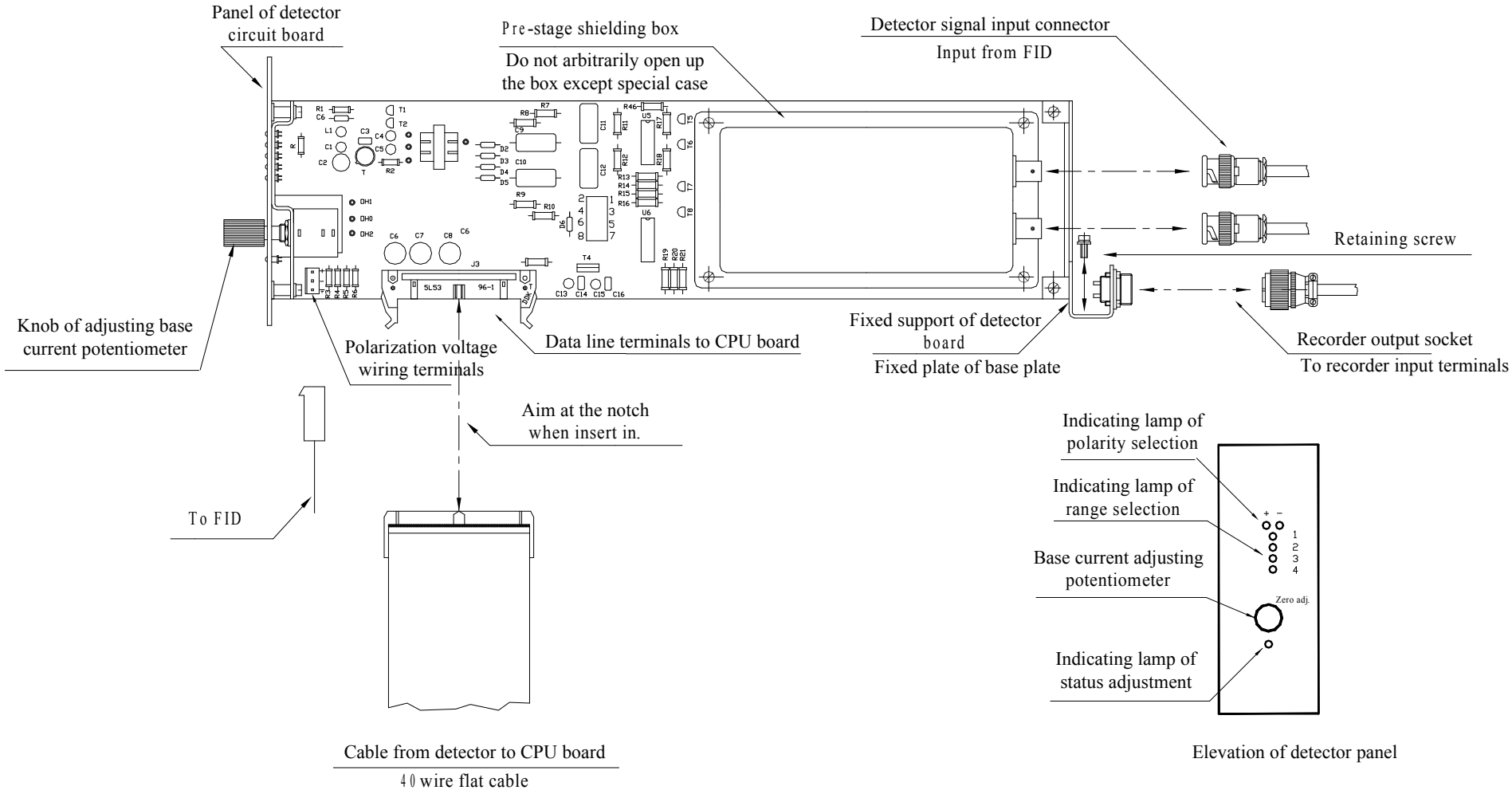


Fig.9.2 Structure scheme of FID amplifier board

CHAPTER 10 INSTRUMENT MAINTENANCE AND TROUBLESHOOTING

Application and maintenance

a. Before every time start up instrument it is needed to check the tightness of the gas line system to prevent the accident of gas leak from the leak happening;
b. Before ignition of the instrument, should make the amplifier stabilize half an hour and adjust the zero point of the recorder;
c. When the instrument is ignited the sensitivity of the amplifier should be adjusted slightly low, that is, position "3" is more suitable;
d. While the instrument is operating the cover of the detector should be put on to avoid the temperature fluctuation and reducing the instrument's stability;
e. The signal output terminal of the detector is forbidden to be grounded, otherwise the components of the detector will be burned out;
f. The power supply of the instrument should be grounded well and connecting the ground wire to running water pipe is forbidden;
g. While the instrument is idle, the stabilizing pressure valve should be adjusted in a relaxation status (anti-clockwise turned) to prevent the elastic devices from losing effectiveness;
h. While the instrument is idle for a long time, it should regularly be electrified;
i. If unnecessary, do not open the flank or rear cover etc. of instrument to prevent user from getting an electric shock..

Troubleshooting:

No.	Symptom	Presumable cause	Troubleshooting
1.	Instrument can not be started up.	a. Power supply is not connected; b. Instrument fuse is burned out	a. Check the cause of the failure; b. Replace a new fuse.
2.	Instrument temperature can not be raised and alarm is given.	a. Heating switch is not turned on; b. Heating fuse is burned out.	a. Turn on heating switch; b. Replace a new fuse.
3.	Individual heated zone temperature can not be raised and alarm is given.	a. Heating bar circuit is open; b. Measuring Pt resistor circuit is open; c. Failure of temperature control.	a. Check up, replacement; b. Check up, replacement; c. Examine and repair or replace temperature control circuit board.
4.	Detector's high temperature sensitive operation noise is high..	a. Low purity of applied gas (gases); b. Detector parts are polluted.	a. Replace gas (gases) with high purity; b. Washing detector.
5.	Detector baseline is unstable.	a. Column is washed away; b. Gas leak of column	a. Reactivation or chromatographic column

		connection; c. Condensate pollution exists in detector system.	replacement; b. Recheck gas leak; c. Properly raise temperature of detector and injector; raise carrier gas flow rate to purge instrument 2 hours.
6.	Detector in low or no response.	a. Detector flame out; b. Improper gas proportioning; c. Chromatographic column resistance is too large and carrier gas is blocked; d. Flame nozzle is blocked by foreign body.	a. Re-ignite; b. Re-adjust gas proportioning; c. Replace chromatographic column; d. Dredge gas line.
7.	Detector can not be ignited.	a. Air flow rate is too small; b. Hydrogen gas flow rate is too small; c. Power supply of igniter is not sufficient and there is no discharging phenomenon; d. Gas line is blocked.	a. Properly reduce air flow rate; b. Properly raise hydrogen gas flow rate; c. Replace cell of igniter; d. Dredge gas line.
8.	Peak form becomes wider.	a. Carrier gas flow rate is small; b. Low column temperature; c. Low injector, detector temperature; d. Large dead volume of system.	a. Properly raise carrier gas flow rate; b. Properly raise column temperature; c. Properly raise temperature; d. Check chromatographic column installation;
9.	Abnormal peak form appears.	a. Polluted shock insulator or gas leak; b. Sample decomposition; c. Pollutants exists in detector; d. Column pollution.	a. Replace or activate shock insulator; b. Properly change analytical conditions; c. Washing detector; d. Replace or activate chromatographic column.

《APPENDIX I》

ALARM CODES

1. Code : 00
   Display : ×SYSTEM ALARM!
   CODE=00
   Oven ALL Alarm None
   Cause : No
   Processing : Heated zone system in normal operation
2. Code : 11
   Display : SYSTEM ALARM!
   CODE=11 1/1
   Oven COL alarm
   Rpt short
   Cause : Column oven Pt resistor is short circuited.
   Processing : Check column oven Pt resistor lead wire.
3. Code : 21
   Display : SYSTEM ALARM!
   CODE=21 1/1
   Oven TCD alarm
   Rpt short
   Cause : TCD Pt resistor is short-circuited.
   Processing : Check TCD Pt resistor lead wire.
4. Code : 31
   Display : SYSTEM ALARM!
   CODE=31 1/1
   Oven DET alarm
   Rpt short
   Cause : Detector Pt resistor is short-circuited.
   Processing : Check detector Pt resistor lead wire.
5. Code : 41
   Display : SYSTEM ALARM!
   CODE=41 1/1
   Oven INJ alarm
   Rpt short
   Cause : Injector Pt resistor is short circuited.
   Processing : Check injector Pt resistor lead wire.
6. Code : 51
   Display : SYSTEM ALARM!
   CODE=51 1/1
   Oven AUX1 alarm
   Rpt short
   Cause : Auxiliary (AUX.) 1 Pt resistor is short circuited.
   Processing : Check AUX. 1 Pt resistor lead wire.
7. Code : 61
   Display : SYSTEM ALARM!
   CODE=61 1/1
   Oven AUX2 alarm
   Rpt short
   Cause : AUX. 2 Pt resistor is short circuited.
   Processing : Check AUX. 2 Pt resistor lead wire.
8. Code : 12
   Display : SYSTEM ALARM!
   CODE=11 1/1
   Oven COL alarm
   Rpt open
   Cause : Column oven Pt resistor circuit is open.
   Processing : Check column oven Pt resistor.
9. Code : 22
   Display : SYSTEM ALARM!
   CODE=22 1/1
   Oven TCD alarm
   Rpt open
   Cause : TCD Pt resistor circuit is open.
   Processing : Check TCD Pt resistor.
10. Code : 32
   Display : SYSTEM ALARM!
   CODE=32 1/1
   Oven DET alarm
   Rpt open
   Cause : Detector Pt resistor circuit is open.
   Processing : Check detector Pt resistor.
11. Code : 42
   Display : SYSTEM ALARM!
   CODE=42 1/1
   Oven INJ alarm

Rpt open

Cause : Injector Pt resistor circuit is open.

Processing : Check injector Pt resistor.

12. Code : 52

Display : SYSTEM ALARM!
CODE=52 1/1
Oven AUX1 alarm
Rpt open

Cause : AUX. 1 Pt resistor circuit is open.

Processing : Check AUX. 1 Pt resistor.

13. Code : 62

Display : SYSTEM ALARM!
CODE=62 1/1
Oven AUX2 alarm
Rpt open

Cause : AUX. 2 Pt resistor circuit is open.

Processing : Check AUX.2 Pt resistor.

14. Code : 13

Display : SYSTEM ALARM!
CODE=13 1/1
Oven COL alarm
Over Max Temp

Cause : Column oven temperature is beyond setting max. temperature.

Processing : Check whether column temperature setting is correct and heated zone is out of control.

15. Code : 23

Display : SYSTEM ALARM!
CODE=23 1/1
Oven TCD alarm
Over Max Temp

Cause : TCD temperature is beyond setting max. temperature.

Processing : Check whether TCD temperature setting is correct and heated zone is out of control.

16. Code : 33

Display : SYSTEM ALARM!
CODE=33 1/1
Oven DET alarm
Over Max Temp

Cause : Detector temperature is beyond setting max. temperature.

Processing : Check whether detector temperature setting is correct and heated zone is out of control.

17. Code : 43

Display : SYSTEM ALARM!
CODE=43 1/1
Oven INJ alarm
Over Max Temp

Cause : Injector temperature is beyond setting max. temperature.

Processing : Check whether injector temperature setting is correct and heated zone is out of control.

18. Code : 53

Display : SYSTEM ALARM!
CODE=53 1/1
Oven AUX1 alarm
Over Max Temp

Cause : AUX. 1 temperature is beyond setting max. temperature.

Processing : Check whether AUX.1 temperature setting is correct and heated zone is out of control.

19. Code : 63

Display : SYSTEM ALARM!
CODE=63 1/1
Oven AUX2 alarm
Over Max Temp

Cause : AUX. 2 temperature is beyond setting max. temperature.

Processing : Check whether AUX. 2 temperature setting is correct and heated zone is out of control.

20. Code : 14

Display : SYSTEM ALARM!
CODE=14 1/1
Oven COL alarm
Fail Heat up

Cause : Column oven heated zone can not be

heated.

Processing : Check column oven heating cord and Pt resistor lead wire.

21. Code : 24

Display : SYSTEM ALARM!
CODE=24 1/1
Oven TCD alarm
Fail Heat up

Cause : TCD oven can not be heated.

Processing : Check oven heater and Pt resistor lead wire.

22. Code : 34

Display : SYSTEM ALARM!
CODE=34 1/1
Oven DET alarm
Fail Heat up

Cause : DET oven can not be heated.

Processing : Check oven heater and Pt resistor lead wire.

23. Code : 44

Display : SYSTEM ALARM!
CODE=44 1/1
Oven INJ alarm
Fail Heat up

Cause : INJ oven can not be heated.

Processing : Check oven heater and Pt resistor lead wire.

24. Code : 54

Display : SYSTEM ALARM!
CODE=54 1/1
Oven AUX1 alarm
Fail Heat up

Cause : AUX.1 oven 1 can not be heated.

Processing : Check oven heater and Pt resistor lead wire.

25. Code : 64

Display : SYSTEM ALARM!
CODE=64 1/1
Oven AUX2 alarm
Fail Heat up

Cause : AUX. 2 oven can not be heated.

Processing : Check oven heater and Pt resistor lead wire.

26. Code : 15

Display : SYSTEM ALARM!
CODE=15 1/1
Oven COL alarm
Temp abnorm

Cause : Temperature of column oven heated zone is abnormal.

Processing : Check COL oven heating cord and Pt resistor lead wire.

27. Code : 25

Display : SYSTEM ALARM!
CODE=25 1/1
Oven TCD alarm
Temp abnorm

Cause : TCD heated zone temperature is abnormal.

Processing : Check oven heater and Pt resistor lead wire.

28. Code : 35

Display : SYSTEM ALARM!
CODE=35 1/1
Oven DET alarm
Temp abnorm

Cause : Detector heated zone temperature is abnormal.

Processing : Check oven heater and Pt resistor lead wire.

29. Code : 45

Display : SYSTEM ALARM!
CODE=45 1/1
Oven INJ alarm
Temp abnorm

Cause : Injector heated zone temperature is abnormal.

Processing : Check oven heater and Pt resistor lead wire.

30. Code : 55

Display : SYSTEM ALARM!
CODE=55 1/1
Oven AUX1 alarm
Temp abnorm

Cause : AUX. 1 heated zone temperature is abnormal.

Processing : Check oven heater and Pt resistor lead wire.

31. Code : 65
Display : SYSTEM ALARM!
CODE=65 1/1
Oven AUX2 alarm
Temp abnorm
Cause : AUX. 2 heated zone temperature is abnormal.
Processing : Check oven heater and Pt resistor lead wire.

32. Code : 16
Display : SYSTEM ALARM!
CODE=16 1/1
Oven COL alarm
Door is open
Cause : Column oven door is opened.
Processing : Close column oven door.

33. Code : 17
Display : SYSTEM ALARM!
CODE=17 1/1
Oven COL alarm
Back door error
Cause : Fail to open rear door for positioning.
Processing : Check connecting line of rear opening door mechanism.

Description : After the failure has been removed, use key《Clear》to shut up alarm. If not to do, then the alarm will given 3 times every second minute. Several alarms (Ex. Display 2/2 denotes there are two alarms) can use《↑》,《↓》keys to examine.

APPENDIX A SCHEME OF INSTRUMENT POWER SUPPLY CONFIGURATION

Ordinal number of cables and connection

① CPU board－Display board (16 wire flat cable)

② CPU board－Temperature control board (20 wire flat cable)

③ CPU board－Detector board(50 wire flat cable)

④ CPU board－Heated zone

⑤ CPU board－Rear opening door mechanism

⑥ Power transformer－CPU board

⑦ Temperature control board－Red power switch

⑧ CPU board－Column oven door switch

⑨ Temperature control board－Column oven stirring motor

⑩ Temperature control board－Heated zone (power supply)

⑪ Power transformer－TCD board

⑫ FID －FID amplifier board (Polarization collector cable)

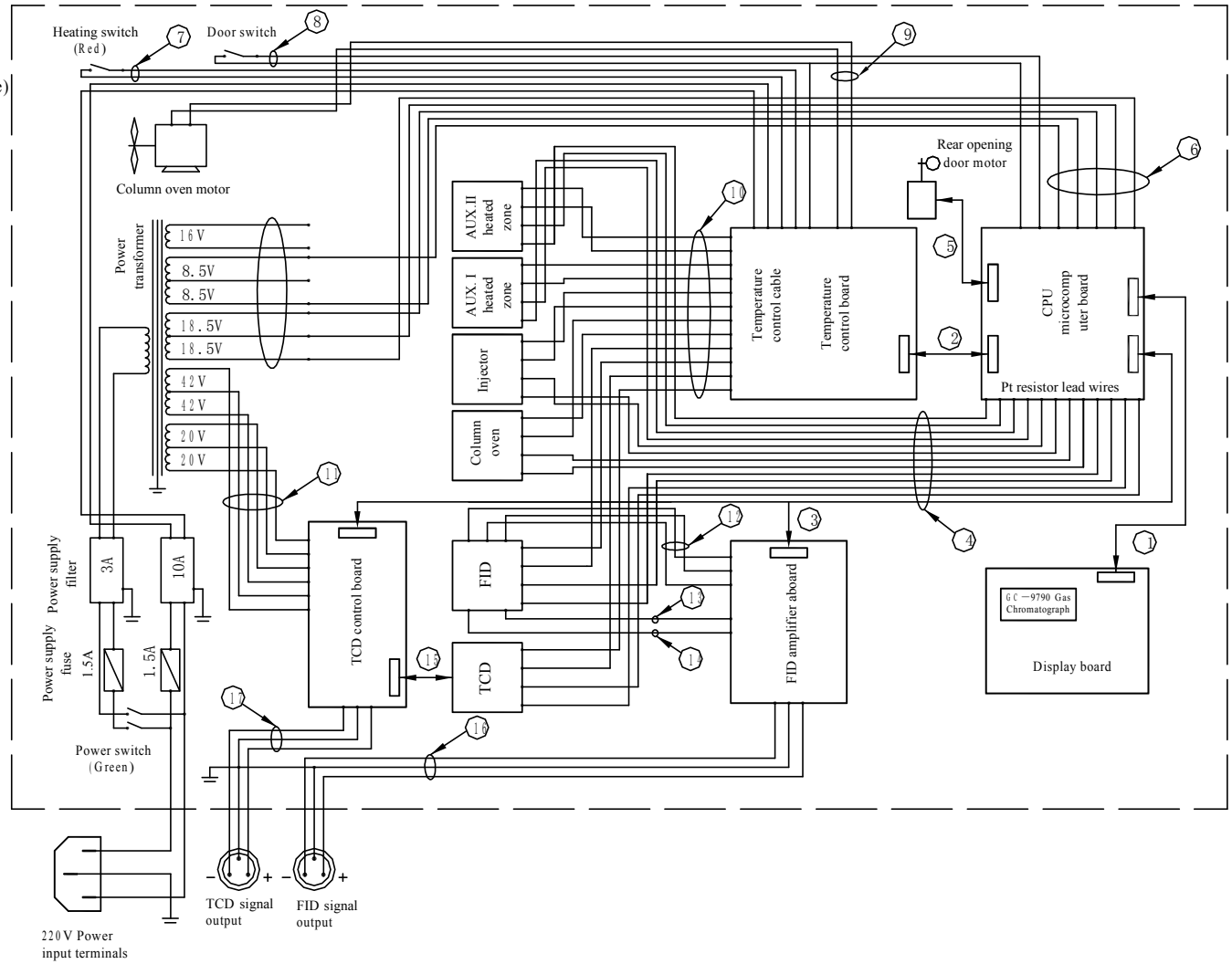
⑬ FID －FID amplifier board (FID collector cable)

⑭ FID －FID amplifier board (FID collector cable)

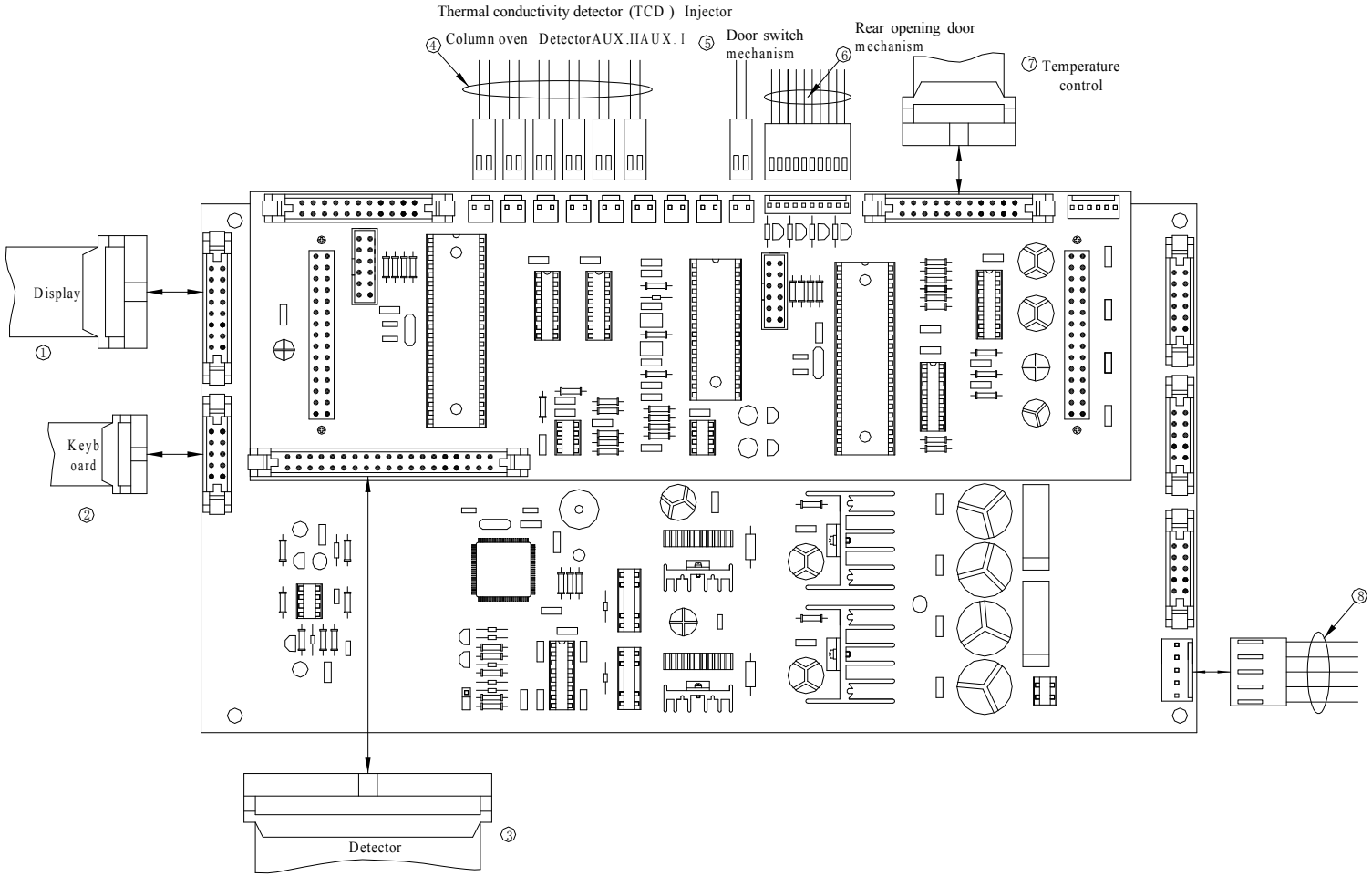
⑮ TCD －TCD amplifier board (TCD bridge current cable)

⑯ FID signal output wire

⑰ TCD signal output wire



APPENDIX B INSTALLATION SCHEME OF CPU CIRCUIT BOARD



① CPU —Display board [Data bus 16 wire cable]

② CPU —Keyboard [Connecting wire]

③ CPU —Detector [Control line,50 wire cable]

④ CPU —Pt resistors [Signal line, bunched cables]

⑤ CPU —Door switch mechanism [Connecting line]

⑥ Rear opening door mechanism [Control line, bunched cables]

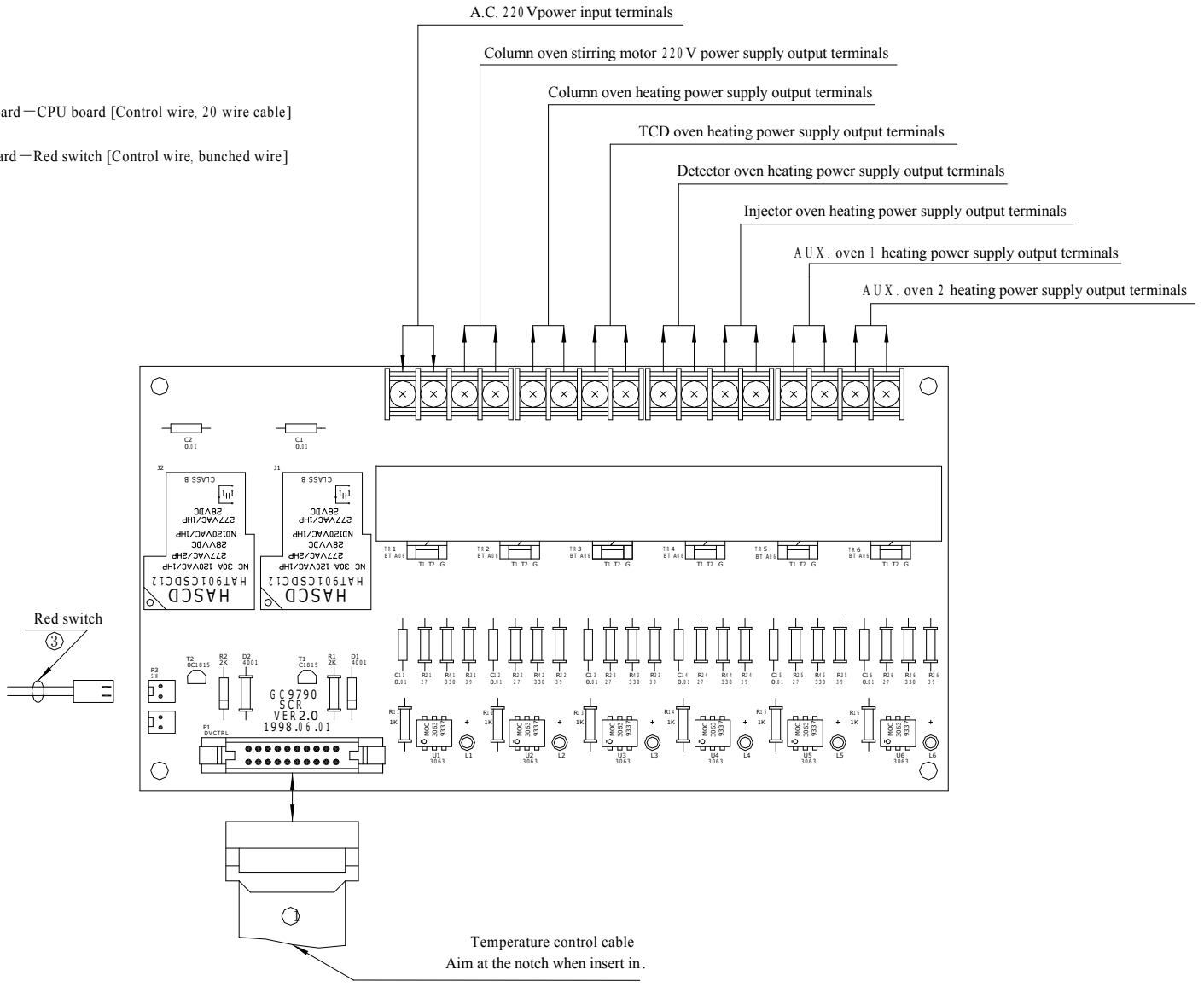
⑦ CPU —Temperature control board [Control wire,20 wire cable]

⑧ CPU —Transformer power wire [Power cord]

APPENDIX C INSTALLATION SCHEME OF TEMPERATURE CONTROL CIRCUI

① Temperature control board—CPU board [Control wire, 20 wire cable]

② Temperature control board—Red switch [Control wire, bunched wire]



Operating Manual's Application Range and Notice of Instrument Operation

1. Description

This manual is the installation and operating manual of the instrument with the basic configuration for series GC – 9790 Gas chromatograph. Both the instrument and FID are mainly introduced (The corresponding installation and operating manuals will be attached according to the configuration situation of the instruments for the other types of detectors or injectors.). The lab preparations, external gas line connection, basic structure, initiation, check before acceptance and key parts etc. contents about the gas chromatograph are also mainly introduced. In terms of the specific situation which may endanger personal safety or incorrect operation, the manual will give the form of "CAUTION" to prompt user and the attention prompting the safety measures will briefly be described in the relevant chapters and sections. We hope that this operating manual can offer you a help to start the instrument smoothly and successfully.

2. Notice

To prevent operator from getting an electric shock :

1) Some electric devices will be exposed by taking down some covers and, general, there are danger signs on these covers. Before taking down these covers attention must be paid to pull out the power plug.

2) In order to meet the requirement of connecting the power supply at the installation site and to replace the power plug, it should be consistent with the specifications mentioned in the manual and the polarity of the power supply should be guaranteed.

3) In accordance with the requirement to replace the fuse, its specifications are denoted on the panel or can be found out in the manual. If the insulation of the power cord is broken it should be replaced in time to get rid of the accident of short circuit.

4) When the position of connecting the power cord is replaced, the voltage, polarity, power of the applied electric network must be examined. Only if these parameters mentioned above are consistent with the requirement the instrument can be linked to the network.

To prevent operator from getting an scald :

In instrument operation, the heated zones have comparative high temperature, so after the instrument is turned off the heated zones continue to have certain temperature for a period of time. In order to get rid of scald, operator should avoid making contact with them. If it is needed to replace parts you must wait for the instrument cooling or use thermal protective gloves or take the other thermal protective measures to contact with the heated zones.

Gas cylinders :

1) The regulations related to gas cylinder transportation, storage, management and safety application should be observed.

2) The store place of gas cylinder should be kept away from heating sources and tinder and should be ventilated well. When the cylinder vertically stands it is needed to have a sturdily fixed support. Do not move it in operation status.

3) The applied gas cylinders should clearly be marked in order to prevent the instrument from mistaken installation to make the instrument not work normally or danger happening.

4) The connecting gas lines between the cylinders and instrument should be kept clean : The pressure tolerance strength should higher than the maximum output pressure of the relief valve and gas leak examination must strictly be executed by user before operation.

Safety measures :

1) The tightness of the gas line system should regularly be checked.

2) Reasonably arrange the gas line system and the distance between the cylinders and instrument should not be too far away. If the gas lines are too long or winding the resistance of them will be increased to cause the leak phenomena with ease.

3) The instrument uses and consumes a small amount of sample, so, in general, it can not generate the air pollution and, in particular, when the type of mass flow rate sensitive detectors is applied the sample will be burned by the flame and then drained off. Therefore, in general, it is unnecessary to adopt the special ventilating equipment. But when the type of the concentration detectors is applied to analyzing harmful materials, the instrument only separates the sample and the components of sample in not broken down. In this case, it is necessary to use a pipeline to drain off the exhausted gases from the instrument to outdoors.

4) The organic solvents in common use should be placed far away from the instrument and stored in the fireproof ventilating cabinet.

A clear sign should be put on every poisonous and inflammable article.

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