OPERATION MANUAL

GAS CHROMATOGRAPHY

GC1120

Content

1 Overview

GC1120 gas chromatography is a computerized，high performance，low cost and general purpose gas chromatograph. It have many advantages as high reliability，compact structure and easy to operate. It can be used in very wide fields for QA/QC like trace analysis for environmental protection, atmosphere and water pollution, analysis and research for poison, clinical application, research of pathology and virus, food fermentation, petrochemical industry, petroleum processing, oil analysis, research of geology and prospecting, organic chemistry, synthesis research, health quarantine, etc. The main features of GC1120 gas chromatography are as following:

1) The microcomputer temperature control system with advanced control technology has remarkable performance, which achieve high precision of temperature control (better than ± 0.1℃), high reliability and strong capability of resisting disturbance. There are 6 separate temperature control zones in the system with the maximal temperature up to 400℃. The limit temperature settings and over-temperature protection function guarantee the safety of this instrument.
2) Big screen for display give more information and more easy operation. The curve of program temperature control, baseline can be displayed. And setting and real temperatures of oven, inlet, and detectors can be displayed in one screen.
3) Packed-column injector and capillary split/splitless injector modules can be installed and achieved 4 kinds of injector combinations. Up to 3 injectors can be installed at the same time.
4) Up to three sample injectors can be installed in the instrument at the same time. 6 kinds of detectors can fit with the instrument, including FID, TCD, ECD, FPD, NPD and PID. Each unit can be assembled very easy when requirement.
5) The internal DAQ device for acquiring instrument statues and data is used to carry out real-time control and data handling with one connect cable between instrument and computer. The real control include temperature control for every parts, detector selection and setting, program temperature, temperature's curve display and flow rate display (optional).
6) Data handling include dual channels (can be extended) high speed data acquirement, integral with manual or auto parameters setting, 5 kinds of quantity methods, baseline correction and making report functions.
7) On packed-column injector can be achieved to fit many kind of column. With connecting pieces used, the instrument could complete analysis by wide bore capillary (ID: 0.53mm). Six-way valve is optional.
8) Gas channel have dual stable control system to achieve high precision, high repeatable flow.
9) Large volume column oven is easy to install one capillary and two packed columns at same time. The oven has the quick heating/cooling function with auto-open back door mechanism. It can achieve accurate control at ambient temperature and 7 stages temperature program.

Notes

Except FID, this operating manual does not include contents of other detectors, wide bore capillary direct injector, capillary cold column injector, split /splitless capillary injector, six-way valve, converter, pyrolysis apparatus, deoxidation device, FJ-2003 chromatograph work station, CDP chromatographic data processor, etc. The operating manuals for these accessories will be provided with the item when the optional accessories are selected.

Notes

Criterion which GC1120 Gas Chromatography implemented are: Q/SXAG21-2010《GC1120 Gas Chromatography》. And this criterion accords with the newest version of followed criterion:

GB191-2000	《Packs Chu Yuntu to show a sign》
GB/T2829-2002	《sampling procedures and tables for lot-by-lot inspection by attribute》 (Apply to inspection of successive lots or batches)
GB9969.1-1998	《Instructions for use of industrial products General Princepals》
JB/T6244-1992	《Gas chromatography for laboratory use》
JB/T9329-1999	《Basic environmental conditions and testing methods for instruments transportation and storage in the transportation》
JJG700-1990	《Gas chromatography》

Every GC1120 Gas Chromatography has been tested strictly before it leaves the factory. The testing results and original spectra accompany with the instrument. The testing results accord with the technical specification of the instrument and Q/SXAG-2008 《GC112A Gas Chromatography》.

1.1 Specifications and operational requirements

1. 1. 1 Column oven temperature

Temperature range: 7℃above room temperature (R.T.) ~ 400℃ (1℃ increment)
Temperature control accuracy: better than ±0.1℃ @200℃
Temperature programming: 7-stage temperature programming
Temperature programmed rate setting: 0.1℃~ 40℃/min (0.1℃increment, measured at 200℃)
Thermostatic timing per stage: 0 ~ 655 min (1 min increment)

1. 1. 2 Injectors, detectors and aux temperature

Temperature range: 7℃ above R.T. ~400℃ (1℃ increment)

Temperature control accuracy: better than ±0. 1℃ @200℃

Notes

In software, there are [detector A] and [detector B] for temperature setting. Normally, ion detectors such as FID, ECD and PID etc, is set by [detector A]. TCD and FPD etc is set by [detector B].

For injector setting, there are A and B, too. Packed Inlets is set by B. Split /splitless inlet is set by A.

And the aux is controled accessories such as converter, pyrolysis oven, etc.

1.1.3 Hydrogen flame ionization detector (FID)

Limit of detection: Dt≤8×10^{-12} g/s (n-$C_{16}H_{34}$ in n-hexadecane)
Drift: ≤6×10^{-13} A/30min
Linear range: ≥ 10^6
Maximum temperature: 400℃

1.1.4 Operation requirements

Power supply: 220V±22V, 50Hz ±0.5Hz
Instrument power: ≤2000W
Ambient temperature: +5℃ ～+35℃
Relative humidity: ≤85%
The environment where the instrument is to be placed should be free of corrosive gas、 electric field or magnetic field that may affect the normal operation of the instrument. The workbench which the instrument is to be placed should be stable and no vibration is allowed.

Warning

The instrument has a strict requirement for grounding. The room where the instrument is placed should have good power grounding.

1. 2 Package and optional accessories

1. 2. 1 Package

GC1120 is series products including the following 6 normal configurations.

GC1120-1	Capillary inlet (including blowing-up function of diaphragm and shunting regulation with back pressure valve) , make-up gas flow control, SE30 ø0.32mmx15m Capillary Column, one FID
GC1120-2	Inlet with double packed columns, one TCD
GC1120-3	Inlet with double packed columns, double FIDs
GC1120-4	Inlet with single packed column, capillary inlet accessories (including blowing-up function of diaphragm and shunting regulation with back pressure valve), double FIDs
GC1120-5	Inlet with double packed columns, capillary inlet accessories (including blowing-up function of diaphragm and shunting regulation with back pressure valve), make-up gas flow control, double FIDs
GC1120-6	Inlet with double capillary inlets (including blowing-up function of diaphragm and shunting regulation with back pressure valve) , double make-up gas flow control, double FIDs, packed columns inlet accessory

GC1120 provides a complete set of accessories for original installation, such as purifying device, exterior gas circuit pipes, spanner, injector and various kinds of fitting, users just need to prepare the gas supply (Please ref the attached list of accessories).

1. 2. 2 Optional accessories

Users can order the following optional accessories of GC1120 gas chromatography when purchase.

1) All kind of detectors
2) Inlet six-way valve
3) Converter (including methane nickel catalyst)
4) Thermal desorption device
5) Split/splitless inlet
6) Simulation and educational presentation software of chromatography
7) Chromatograph work station
8) CDP chromatographic data processor
9) Deoxidation tube
10) Glass thin-layer chromatographic column (ID 2mm, OD 5.7mm, length ≥lm. Central line distances for the two tubes are 186.8mm and 59mm respectively, suitable for this instrument)

1. 3 Instrument working principle

Gas chromatograph uses gas as the mobile phase (carrier gas). Samples of a mixture are inserted into a stream of carrier gas passing through a packed column or capillary chromatograph column. Each solute in the original mixture will distribute itself between tile stationary medium and the carrier gas in a unique way. The result is that some solutes will travel across tile absorbent medium faster than others, leaving tile individual solutes separated and spread. The end of tile tube leads straight into detection device to detect individual solutes according to their physical and chemical properties. Please refer to figure 1-1 for working scheme of GC1120.

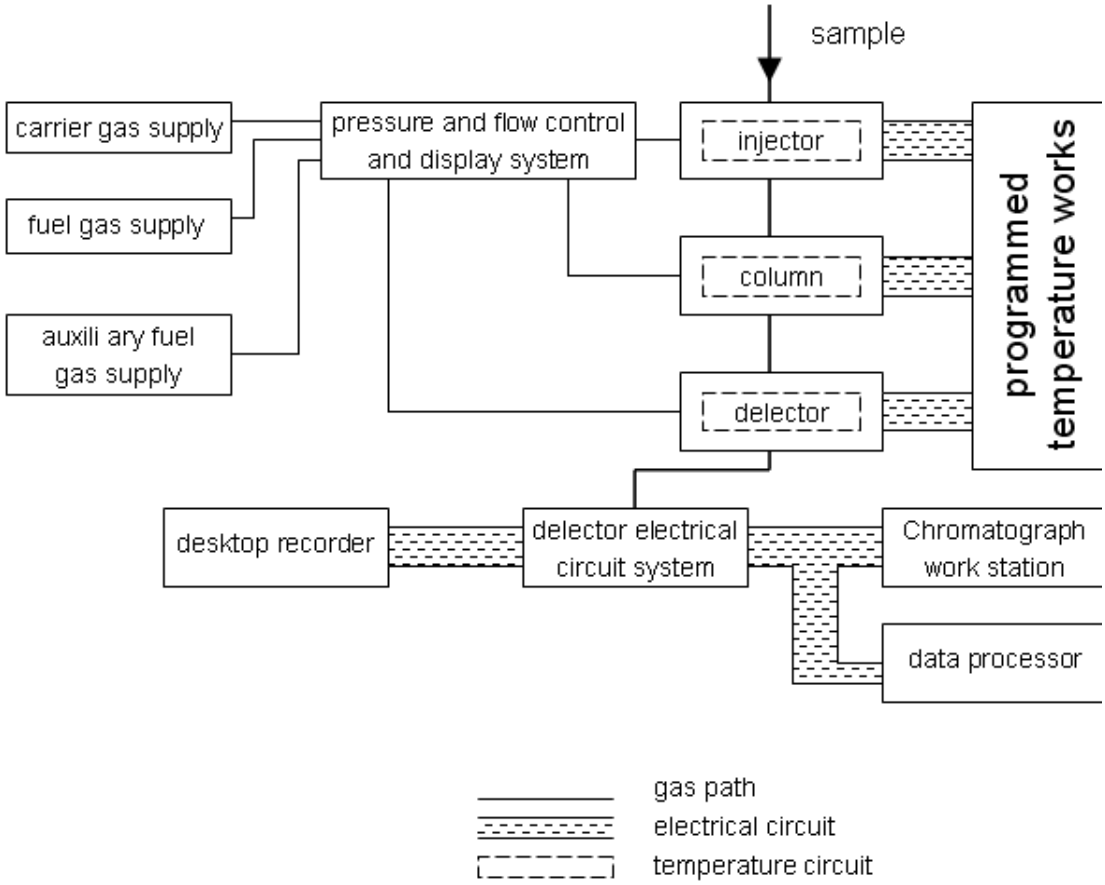


Figure 1-1 GC1120 Gas Chromatograph Working Scheme

1.4 Mainframe structure

GC1120 consists of detector, inlet, chromatography column compartment, flow control accessories, temperature control and detector circuit, etc. Please refer to figure 1-2.

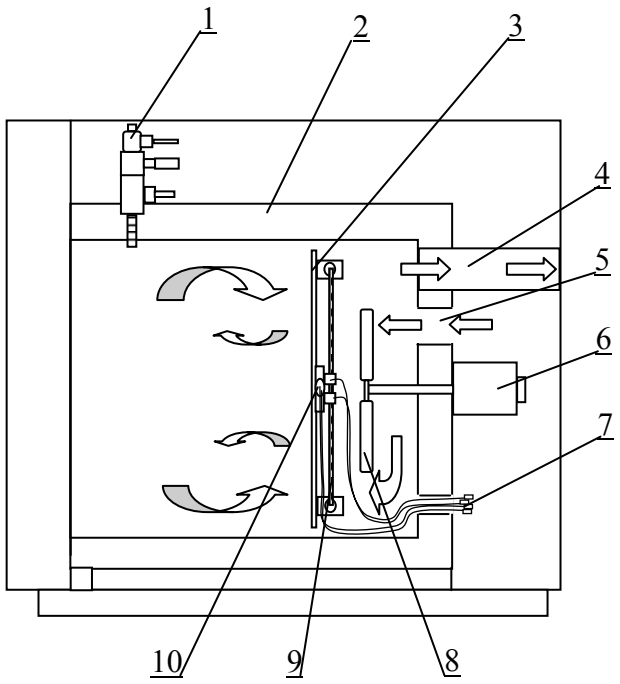
For basic configuration of GC1120 chromatograph，the middle part is chromatograph column compartment，upper right is programmed temperature controller，down right is detector control, left is flow control accessories and gas path panel, upper left to the column compartment is the installation space for ion-detector (normally two FID will be installed) and TCD．Upper right to the column compartment is the double packed inlet or capillary inlet．

Figure 1-2 GC1120 Gas Chromatography

1.5 Oven

With its large oven space，GC1120 is capable of convenient installation for capillary column or double packed column, and quick temperature raising or cooling. Heating filament of the column compartment is hidden behind the grid. Therefore, heating filament radiation, which may cause peak split, can be reduced. It also has low motor noise, smooth running and little vibration. When the oven is to be cooled, cooling air intake and hot air vent behind the oven will be opened automatically to cool the oven immediately.

The total heating power is about 1500W. Please refer to figure 1-3 for schematic diagram of the oven structure



1. Detector
2. Insulation
3. Grid
4. Hot air exhaust
5. Cool air inlet
6. Air blower motor
7. Connection of heating and Pt resistance
8. Fan
9. Heating
10. Pt resistance

Figure 1-3 Sketch of Oven（side-glance）

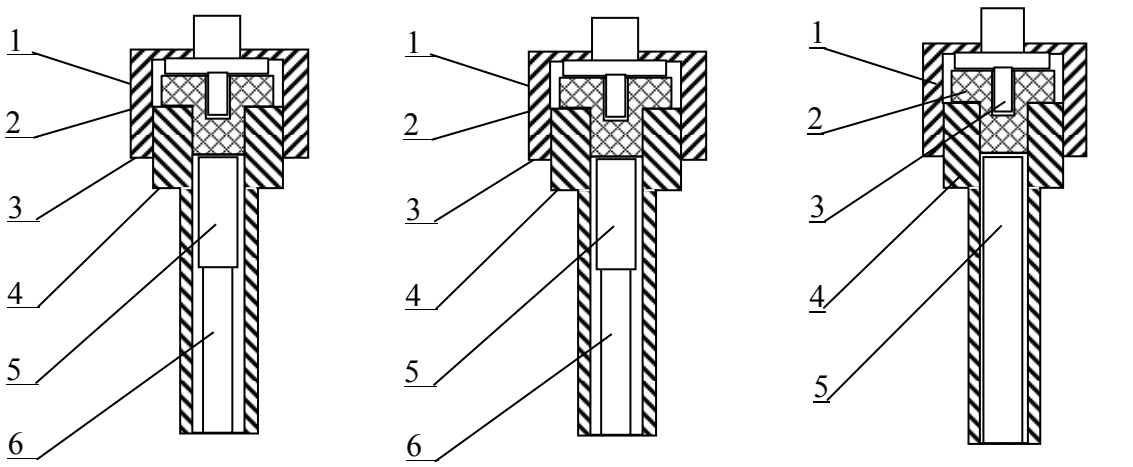
1.6 Inlet

There are three kinds of inlets could be selected: single/double packed inlet, capillary split/splitless inlet. Select according to customer needs.

1.6.1 Packed column inlet

The instrument can be filled with single-or dual-column injector packed column injector (optional).

Packed column injector structure shown in Figure 1-4. Single packed column / double packed column injector installed on the host at the top of the left side of the body heat, heat the body to install a heating element (100W) and ceramic platinum resistance temperature controller by a microcomputer to control its temperature.



φ3mm、φ4mm packed column

1. Exhaust
2. Seal ring（GC0035）
3. Guide frame
4. Inlet
5. Tube φ5（GC0054）
6. Connector (sampling) φ3（GC0050）
   φ4（GC0051）

φ5mm packed column

1. Exhaust
2. Seal ring（GC0035）
3. Guide frame
4. Inlet
5. Tube φ5（GC0054）
6. Column（φ5）

φ6mm packed column

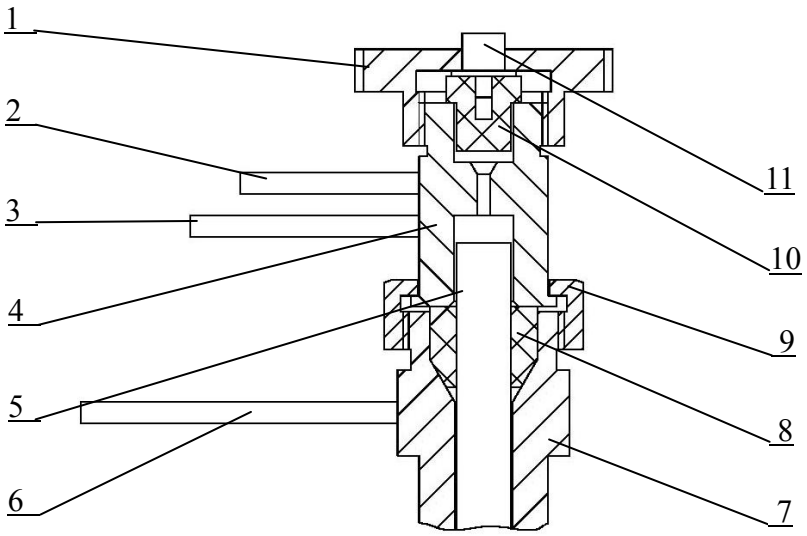
1. Exhaust
2. Seal ring（GC0035）
3. Guide frame
4. Inlet
5. Column（φ6）

Figure 1-4 Packed column inlet（part）

Installation step refer to Chapter 4.4 of this manual.

1.6.2 Capillary inlet

Inlet equipped on GC1120 is special capillary inlet, which can be used for split/splitless injection. This configuration includes independent capillary inlet system (including carrier gas flow control, septum purge, split with back pressure valve and makeup flow control). Figure 1-5 is the illustration of special capillary inlet structure.



1. Heat abstraction cap
2. Septum purge vent
3 Carrier gas inlet
4. Position sleeve
5. Split quartz liner tube(GC0056)
6. Split vent
7. Capillary inlet base
8. Silicon/graphite seal（GC0041）
9. Nut
10 Silicon seal（GC0035）
11. Needle guide

Figure 1-5 Illustration of capillary inlet structure（part）

1. 7 Gas flow control system

The carrier gas flow of GC1120 adopts double stabilizing design by pressure maintaining valve and flow maintaining valve. The air and hydrogen flow adopt pressure maintaining valve and needle valve with mechanical dial as adjusting modes. Capillary gas flow use backpressure valve for split adjust and needle valve for septum purge.

Dial and panel for air and hydrogen flow control is on the left frontal of the instrument, referring to figure 1-6. Upper left is the dial and panel for capillary flow (Open the cover plate when on use), see figure 1-7.

1. 7. 1 Carrier gas flow path

Carrier gas flow rate is regulated by a mechanical dial flow control valve. The inlet pressure for the flow control valve is regulated by a pressure control valve (adjusted to 3kg/cm^2 for ex-work). Flow through the flow control valve can be obtained from the corresponding flow chart (notice: flow rate relates with individual gas). That means each scale on the valve represents a standard curve relation with the flow rate. Since the scale-flow rate curve has a precision of 0.5%, rotary flow meters can be omitted. If more accurate flow measurement is required, soapsuds flow meter can be used.

1. 7. 2 Hydrogen and air flow path

The auxiliary gas of GC1120 chromatograph includes air and hydrogen. Dial needle valves are used for air & hydrogen flow control. Gas pressures are regulated by upper stream pressure control valves to ensure constant pressures for hydrogen & air needle valves. Hydrogen & airflow rate coming out of tile needle valves can be obtained from the corresponding dial-flow rate chart. That means you can set or change the hydrogen & airflow rate by setting the dial of the needle valve.

Notes

1). Scale-flow rate curve sheet for carrier gas, hydrogen and air are provided attached Table-1. Please refer to these curve sheet for the corresponding flow rate. Curve sheet or Table-1are valid for each configuration of GC1120 chromatograph.

2). In Figure 1-7，carrier gas pressure gauge indicates pressure for carrier gas before coming into the column，hydrogen pressure gauge indicates pressure for hydrogen coming out of the hydrogen pressure control valve, air pressure gauge indicates pressure for air coming out of the air pressure control valve.

Warning

1). Please do not change pressure output of the internal pressure valves of the gas flow. Any difference may affect the validity of the scale-flow rate curve and the output precision.

2). Most valves used for GC1120 gas path are scale valves. They have been subjected to strict inspection and regulation before leaving factory. Users should not dismantle the dials on the valve, or the displayed scale would not match the scale-flow chart. If the dials get loose, soapsuds flow meter should be used to correct the scale under the pressure condition specified on the chart so that the scale-flow rate relation consists with that on the scale-flow rate chart. The dial should not be turned to "0" to protect needle valve and flow control valve. To shut off gas, turn off the on-off valve on the purifier or turn the valve dial to a position which represents "0" flow rate on curve cards.

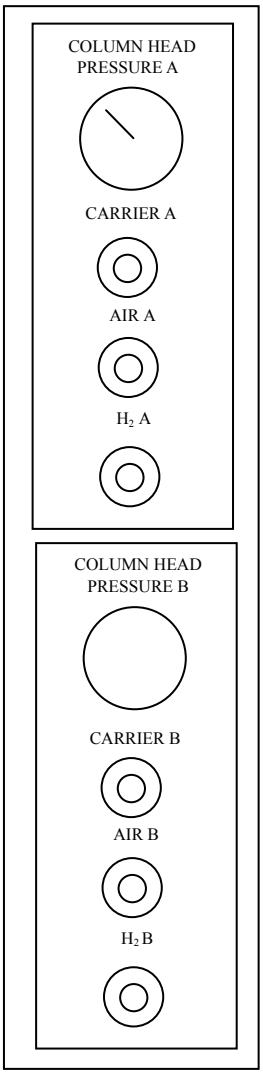


图 1-5 Carrier Gas Regulation Panel (front view)

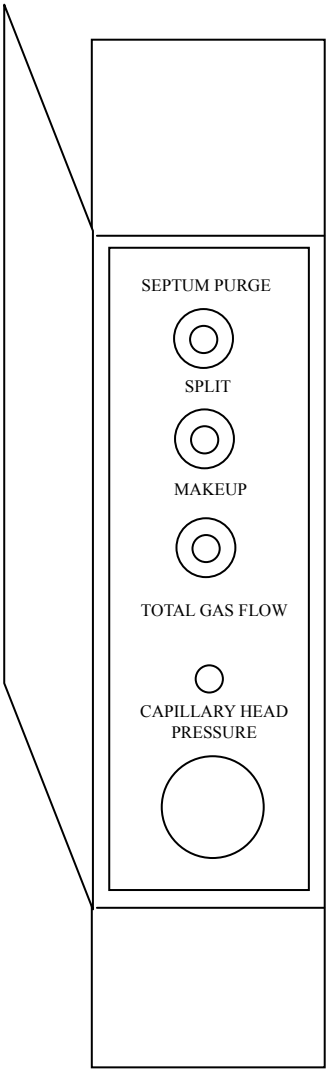


图 1-6 Capillary Regulation Panel (platform)

2 Programmed temperature works

Programmed temperature works of GC1120 chromatography have wide range and precise temperature control for 6 loops. For column compartment a 7-stage programmed temperature is available. This control system has high performance, reliability, disturbance resistance and minimum temperature fluctuation due to its advanced software and hardware technology and structure.

The system also has such functions as temperature limit setting, time recorder and analysis time counter, temperature retention, dynamic scanning of actual temperature, automatic opening of column compartment back door while cooling, data protection from power failure, automatic opening or closing heating, etc. In addition, RS-232 connection can be selected to chromatograph workstation to carry out 2-way data transmission and control for temperature setting and system status.

Programmed temperature works of GC1120 chromatography has an integrated circuit that includes voltage regulator, platinum resistance sampling & A/D switch, CPU, SCM, controllable silicon, etc. The circuit board has a minimized area and components therefore ensures reliability , easy installation and maintenance. This circuit board is called main board. In addition a keyboard display board and a RS-232 serial communication board (optional) together with the main board consist of the temperature control system.

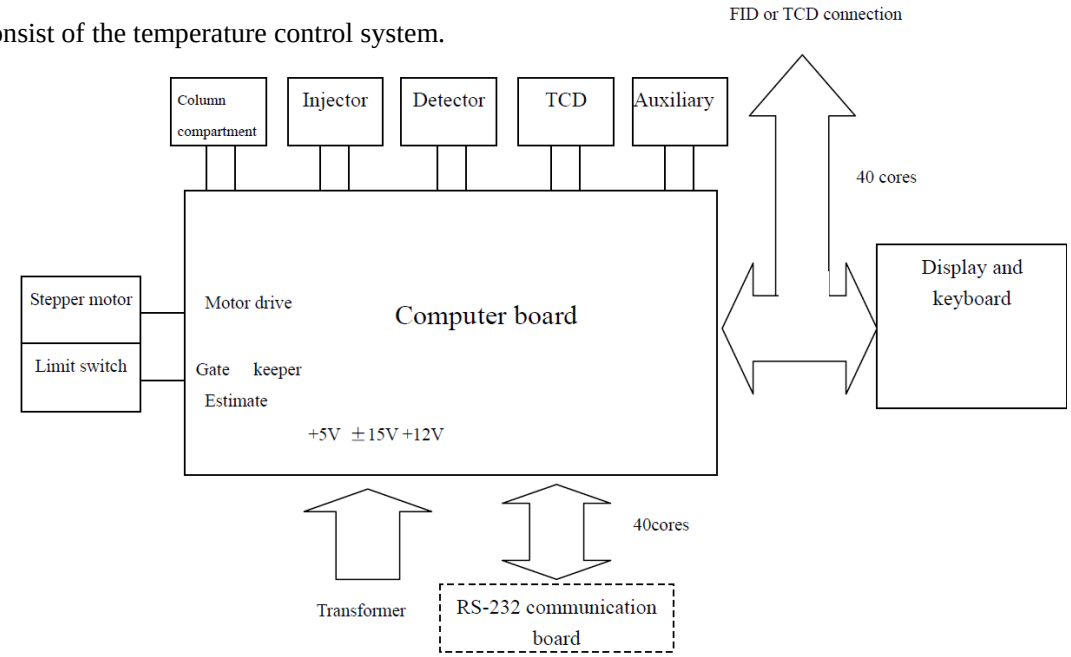


Figure 2-1 Illustration of the microcomputer system

2. 1 Operating panel and keyboard

Temperature control panel is as shown in Figure 2-2.

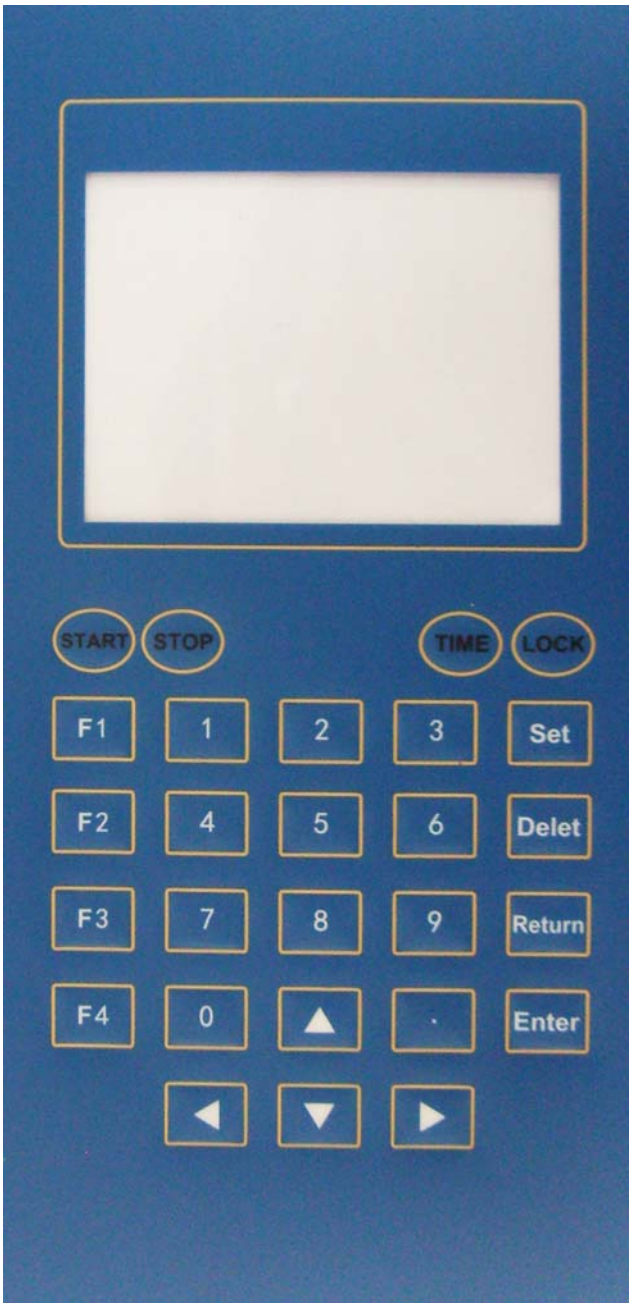
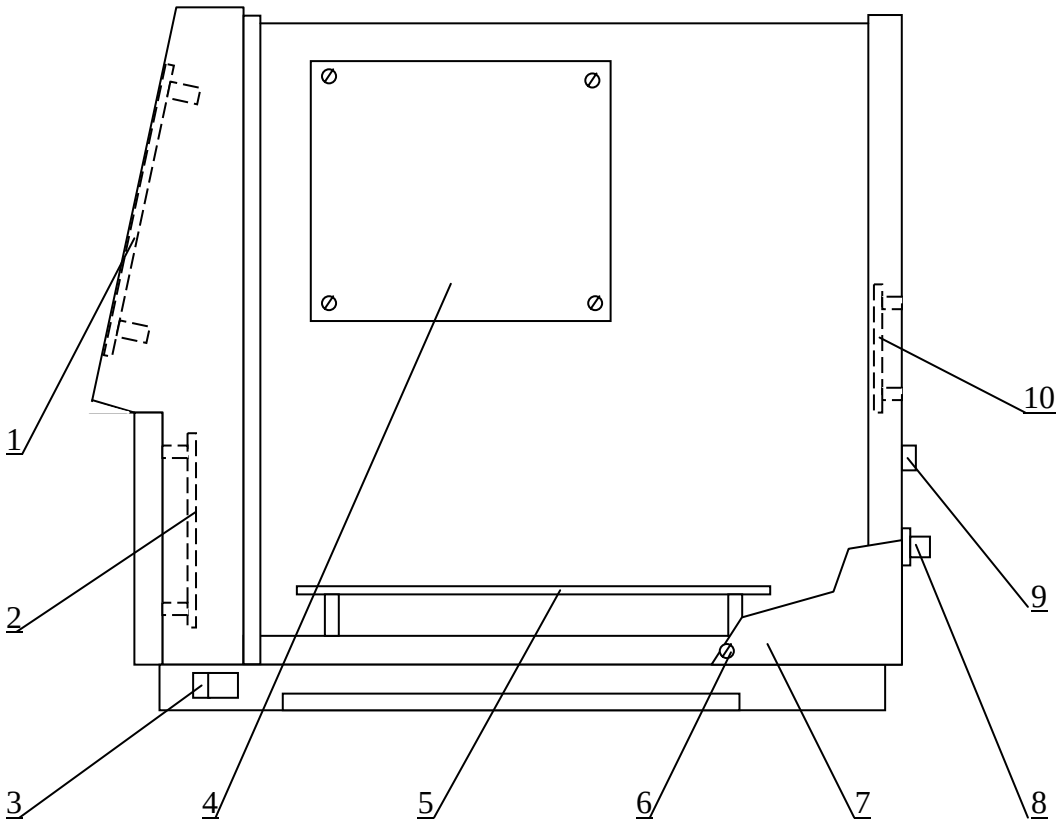


Figure 2-2 Temperature Control Panel

Circuit board installation position for GC1120 chromatography (basic configuration) is shown in figure 2-3.



1. Display and keyboard
2. Detector zero setting board
3. Power switch
4. Main board
5. Detector circuit board
6. Electrical side board fix screw
7. Electrical side board
8. Detector signal port
9. RS-232 parallel communication port
10. Built-in chromatogram work station interface board (optional)

Figure 2-3 Circuit Compartment (side view)

2.1.1 Keypad

Programmed temperature panel has 27 function keys and number keys. They can be classified as follows:

Number keys:

[0] [1] [2] [3] [4]

[5] [6] [7] [8] [9]

[.] — Decimal

Most often used keys:

[STAR] — Temperature program begin running after press.

[STOP] --- Stop the temperature program.

[EDIT] ----Set the status to edit mode for modify parameters after press.

[ENT] — For data input purpose. Press the number keys and press **[ENT]** to input the data.

[CE] — If entered a wrong number, you can press **[CE]** key and then input the correct number and press **[ENT]. [CE]** can also be used to clear error message.

[RETURN]---Return to upper menu item.

[LOCK] — This key can be used to prevent incorrect data input. Pad locking information will be displayed on the screen after press this key. And all other keys can not work except **[LOCK]** key. To resume functions of other keys, press **[LOCK]** again.

[Timing]---Go into timing screen

Function keys

[F1]— Open all temperature controllers.

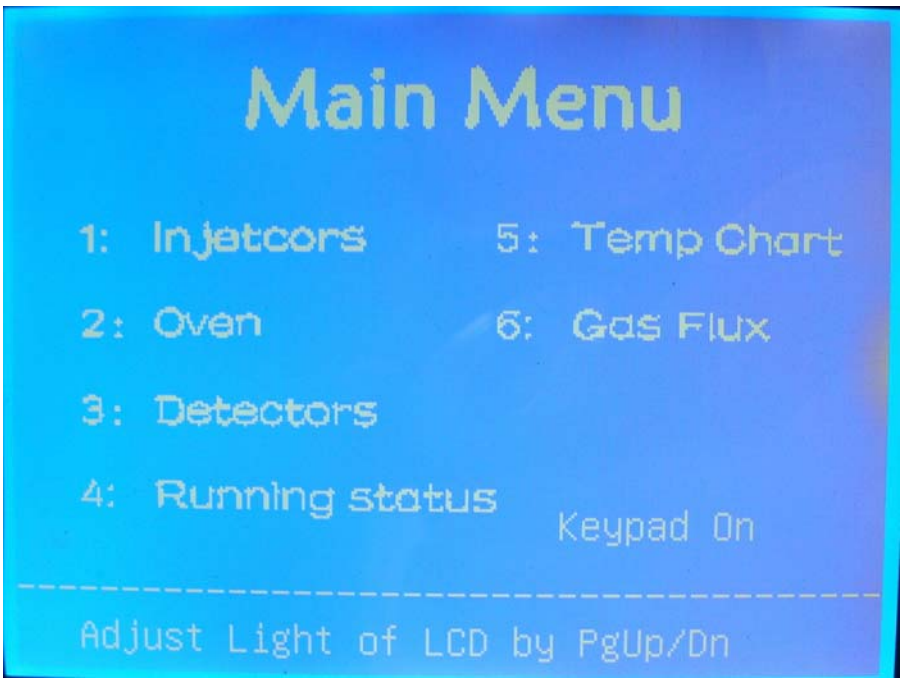
[F2]---Shut down all temperature controllers.

[F3]---Setting the max temperature for all controllers.

[F4]---Back to main menu in any item. If the screen has wrong display, this key will reset the screen to main menu.

2.1.2 Main menu

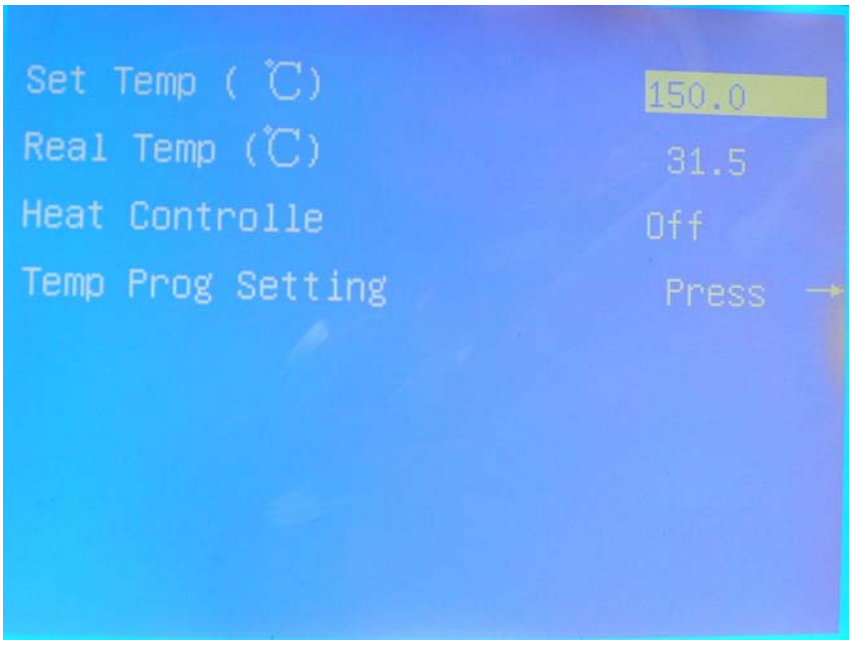
As shown in figure below.



In main menu, number keys 1-7 used to select items. PgUp and PgDn keys set the light of the screen. When [LOCK] key pressed, “Keypad On” at the right corner of the screen will change to “Keypad Off”

2.1.3 Injectors menu item

After select 1, the screen is shown as figure below.

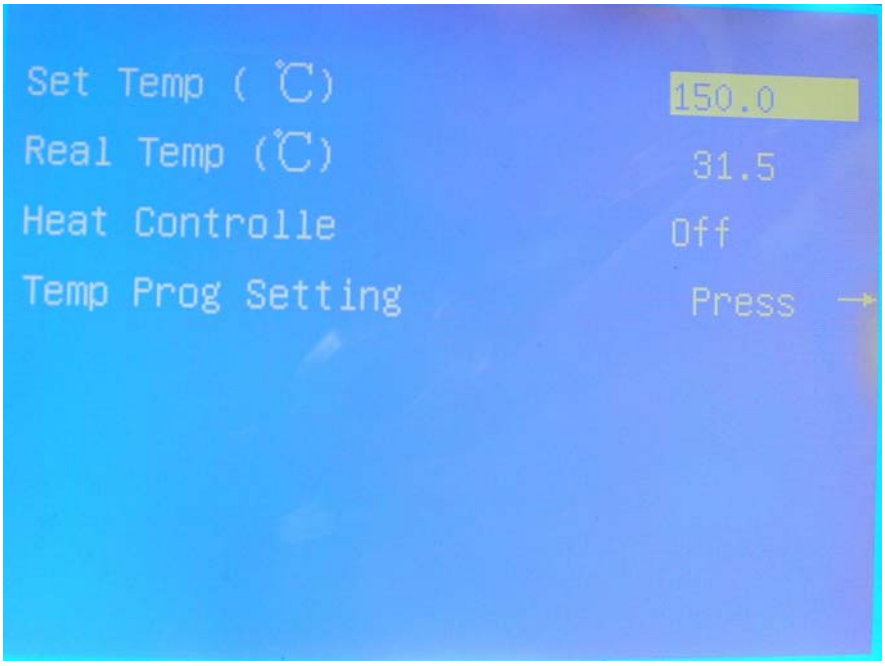


Use number key 1 or 2 to select to set or display parameters of front or back injector.

On setting screen, use PgUp or PgDn to select the item. Then press [EDIT] to change to edit mode. Use number key to input data and [ENT] to finish settting.

2.1.4 Oven menu item

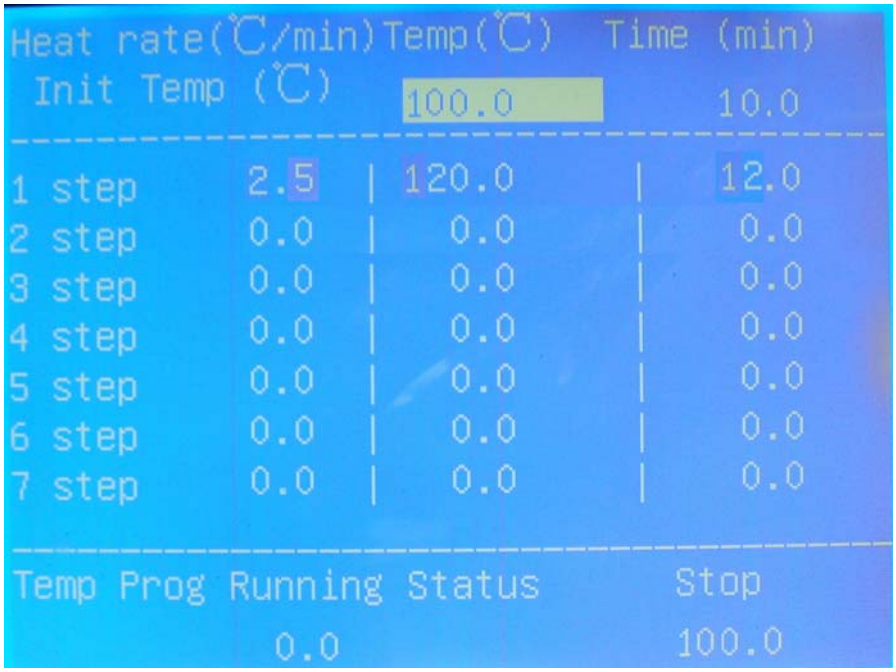
After select 2, the screen is shown as figure below.



Oven screen display information about oven temperature.

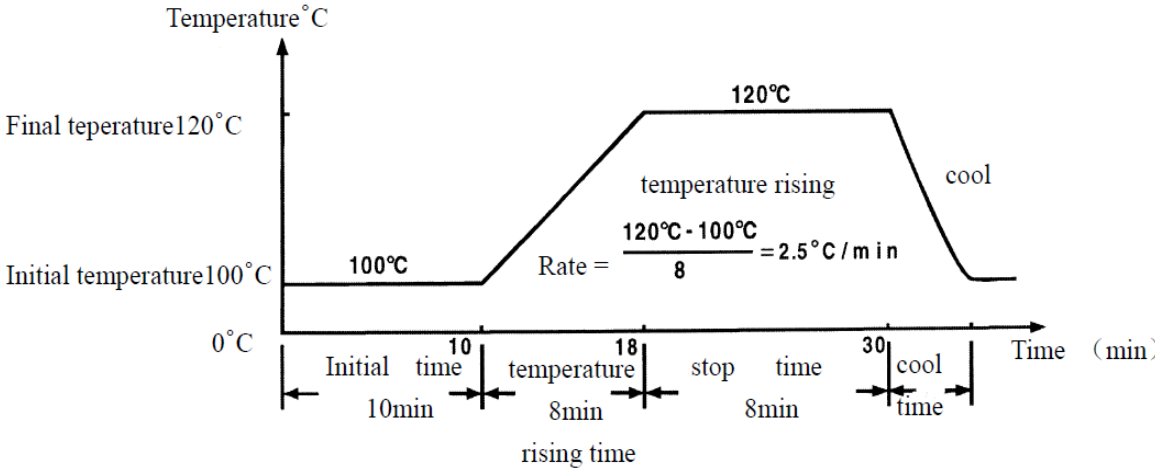
Use PgUp or PgDn to select the item. Then press [EDIT] to change to edit mode. Use number key to input data and [ENT] to finish settting.

Use right arrow key into temperature program screen as figure below.



Use PgUp or PgDn to select the item. Then press [EDIT] to change to edit mode. Use number key to input data and [ENT] to finish settting.

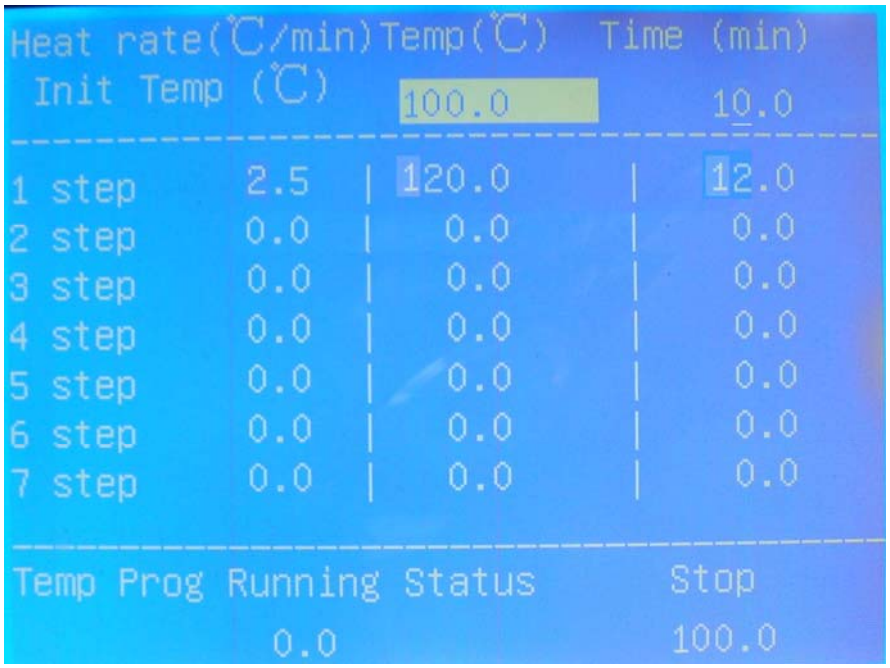
Example: a one stage programmed temperature is compiled according to the curve shown in the following picture.



Operating procedures are as follows:

Operation	**Key sequence**
Set initial temperature 100°C	**Select Init Temp item, then [EDIT] [1] [0] [0] [ENT]**
Set initial time 10min	**Select Init Time item, then [EDIT] [1] [0] [ENT]**
Set program rate 2.5°C/min	**Select 1 step first col, then [EDIT] [2] [.] [5] [ENT]**
Set final temperature 120°C	**Select 1 step second col, then [EDIT] [1] [2] [0] [ENT]**
Set final time 12min	**Select 1 step third col, then [EDIT] [1] [2] [ENT]**

The screen as figure below:

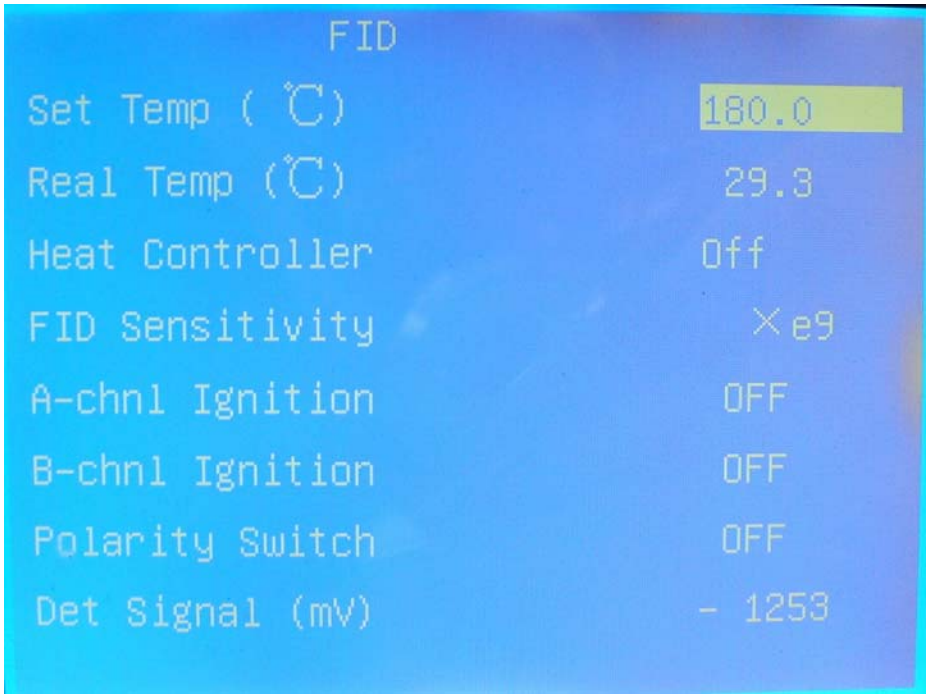


2.1.5 Injector menu item

After select 3, the screen is shown as figure below.

All detectors GC1120 can be installed will display on this screen. And a ✓ mark display on the item if the detector was installed. Detectors installed can be automatic checked when power on.

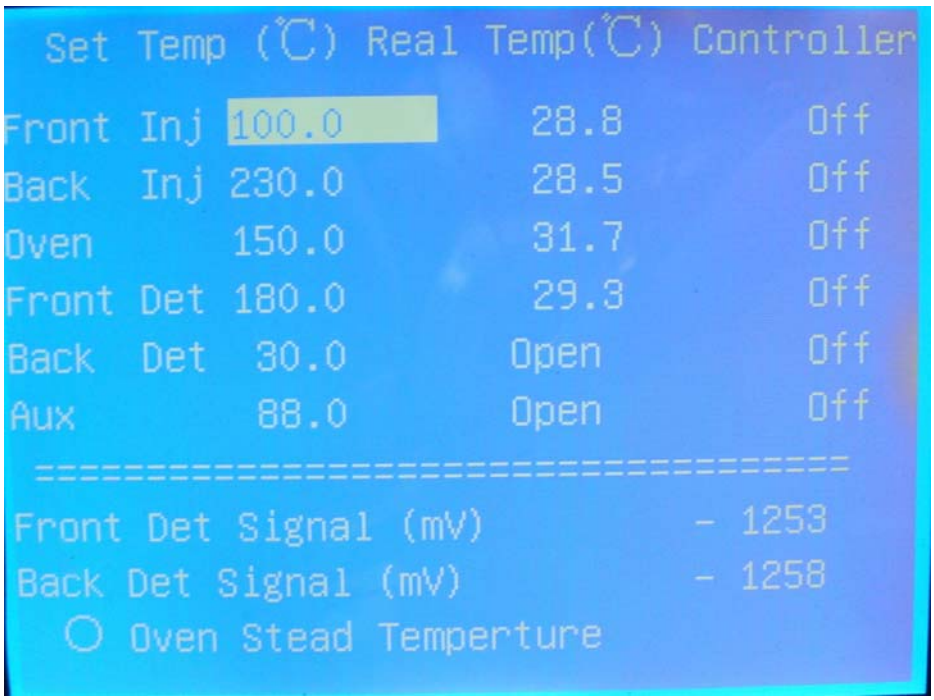
Use number key to select the detector item and go into setting screen. Use FID1 as an example. Press [1], then FID setting screen as figure below.



Use PgUp or PgDn to select the item. Then press [EDIT] to change to edit mode. Use number key to input data and [ENT] to finish settting.

2.1.6 Running status menu item

After select 4, the screen is shown as figure below.



On this screen, Injectors, oven, detectors and aux's status and information will display. And all parameters can be edited on the screen. Use PgUp or PgDn to select the item. Then press [EDIT] to change to edit mode. Use number key to input data and [ENT] to finish setting.

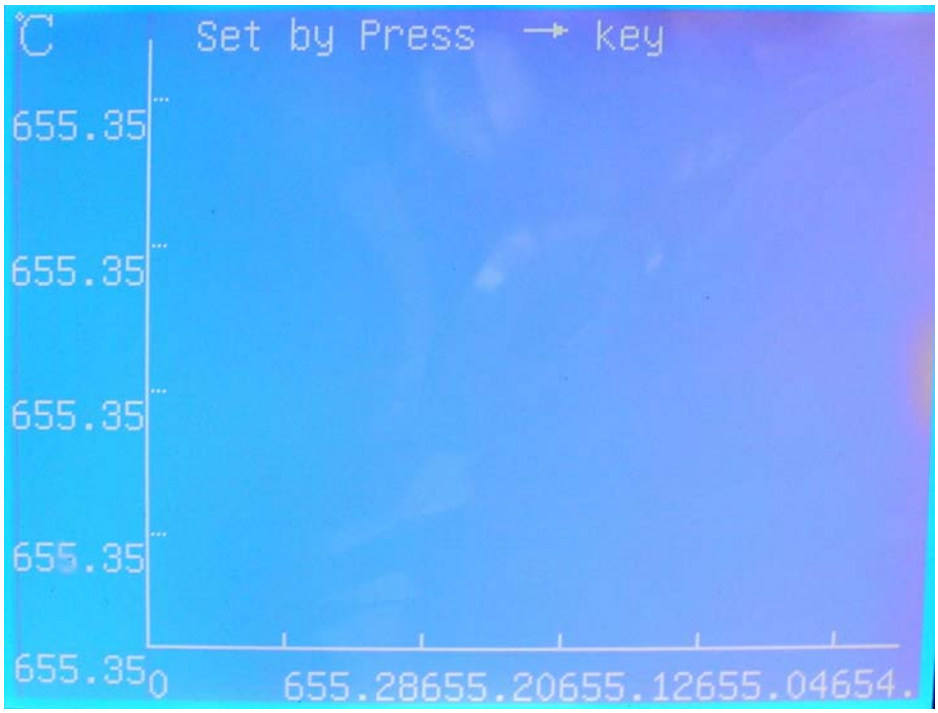
[F1] and [F2] can open or close all heating controller.

Normally, Front Inj is for capillary's injector, Back Inj is for packed column's injector or the second capillary's injector. Front Det is for FID, ECD, PID or NPD detector. Back Det is for TCD or FPD detector.

At the bottom line, oven status display. When heating, LED will blink. When constant temperature, LED will keep light. And step will be displayed when temperature program is running.

2.1.7 Temp Chart menu item

After select 5, the screen is shown as figure below.



On this screen, temperature curve will display. This is useful for program temperature. Use right arrow key into curve display parameters setting screen. On setting screen, max and min temperature displayed and sampling frequency can be set. Use PgUp or PgDn to select the item. Then press [EDIT] to change to edit mode. Use number key to input data and [ENT] to finish settting.

2.1.8 Gas Flux menu item

After select 6, the screen is shown as figure below.

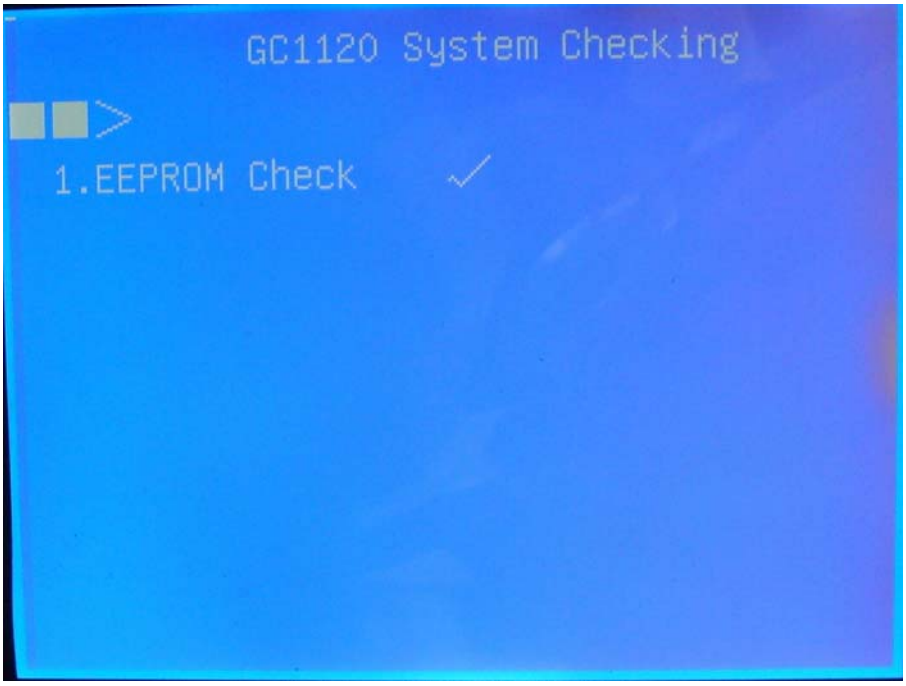
On the screen, gas flux in each channel will display. The accessories for this function is optional and do not include in basic configure.

2. 2 Operation of the programmed temperature works

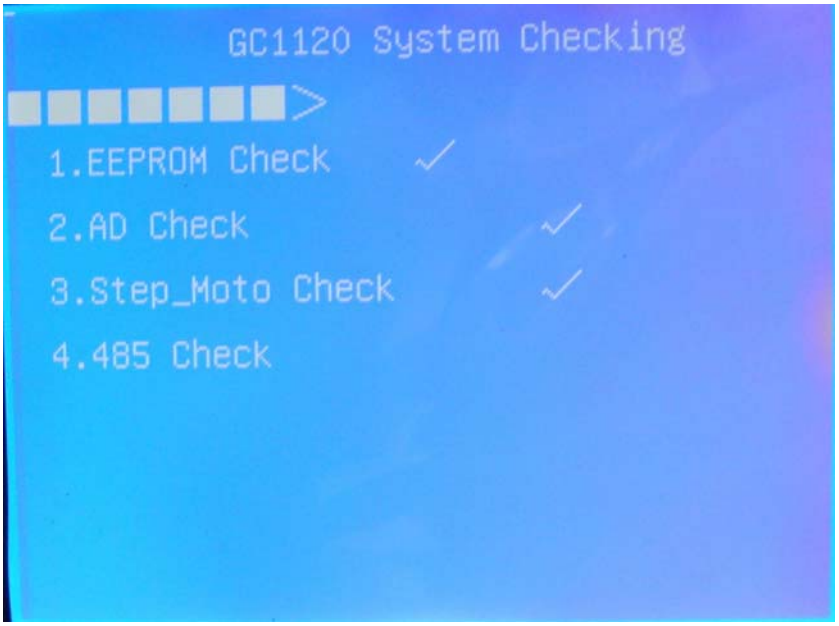
Please refer to this operating manual the first time you installed this instrument and carry out the following operations:

2. 2. 1 Startup

Turn on mainframe machine power switch (the power switch is positioned at the lower right of the mainframe machine, please refer to Figure 2-3). The following screen will be displayed for self-testing and initialization.



4 items will be checked as shown in figure below. And the 3rd is for back door of oven. Error for this item is not a problem, it only means the back door open when power on. And it will be closed automate.



After self-check finished and no error, two beeps can hear and screen will go into main meun.

Notes

When power cord connected to the host, the power switch will light. It indicate that the host has already connect to ~ 220V voltage power supply, not means power on.

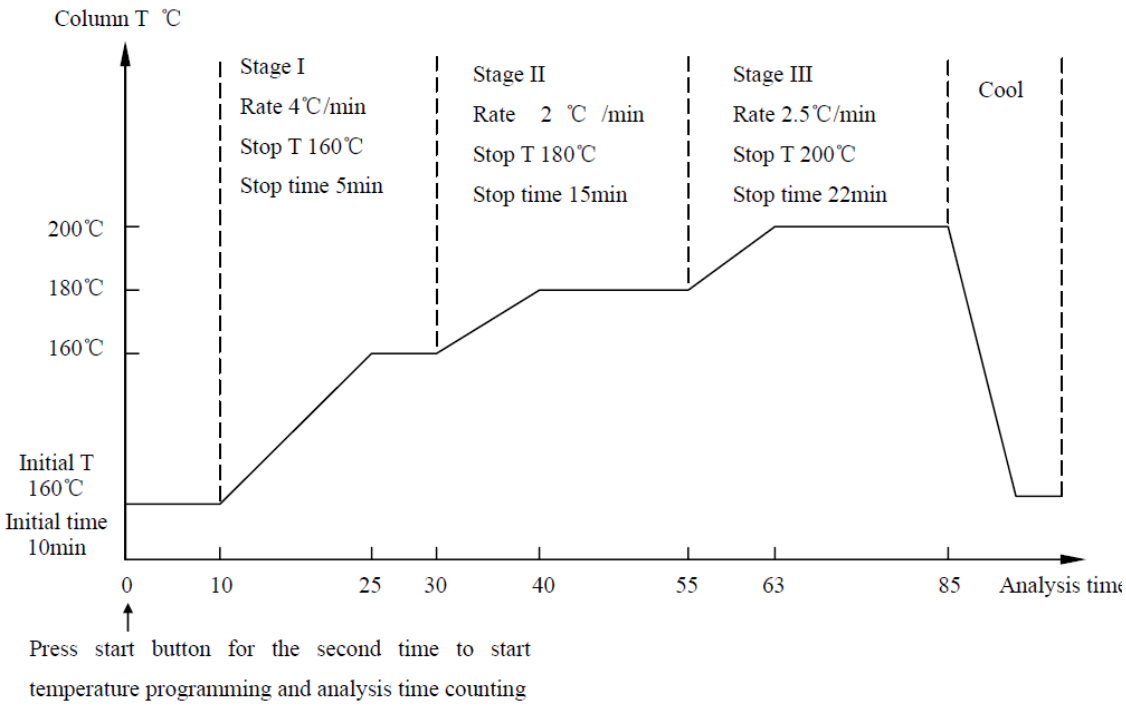
2. 2. 2 Temperature and temperature programming parameters setting

The initial temperature is set to 100°C for column compartment, 200°C for injector, detector and auxiliary, 30°C for thermo conductivity cell. The maximum column compartment temperature is 400°C. The set temperature control parameters can be saved and will be restored to the previously set value for each startup. The column compartment temperature is determined by the maximum temperature allowed for the column. Other temperature parameters are determined by analysis conditions and analysis object. Therefore, maximum temperature and analysis temperature should be reset according to specific requirement for column compartment, injector and detector.

Normally, temperature's setting and display can use item 4 in main menu. Refer chapter 2.1.6.

For temperature programming of oven, use item 2 on main menu screen. Refer chapter 2.1.4.

Here a 3-stage temperature programming will be used as an example for parameter setting. And how to input the data please refer chapter 2.1.4



So the setting sequence will be:

1. Set initial temperature to 100, time to 10.

2. Set 1 step: rate 4, temp 160, time 5
3. Set 2 step: rate 2, temp 180, time 15
4. Set 3 step: rate 2.5, temp 220, time 22.

2. 2. 3 Sample analysis process

You can start to analyze sample when you have understanding in injector, detector column compartment and operations of other parts of the chromatography.

{A} Thermostatic analysis

Programmed temperature works will set thermostatic temperature for injector and detector. Column compartment temperature will be initialized to the value required by thermostatic analysis (please notice that injector set temperature should be 30°C higher than the column compartment initial temperature). Then press [F1] and temperature will rise. After temperature and detector signal are stable, sample can be injected for analysis. Press [START] on the chromatograph data processor at the same time when the sample is injected. Chromatograph data processor or chromatograph workstation starts records peaks, integration and quantitative calculation for the detector signal.

{B} Temperature programming analysis

Programmed temperature works set temperature programming parameters for column compartment, injector and detector (please notice that injector set temperature should be 30C higher than the column compartment initial temperature). Then press [F1], injector temperature and detector temperature will rise to the set value, column compartment temperature will rise to the initial temperature. After temperature and detector signal are stable, sample can be injected for analysis. Press [START] of the programmed temperature works and the [START] on the chromatograph data processor at the same time while the sample is injected. Then column compartment starts temperature programming and chromatograph data processor or chromatograph workstation starts record peaks and integration calculation.

Status can be checked on status range at bottom of screen or Temp chart screen. When stage final time is completed or [STOP] key is pressed, the back door of oven will open for cooling the oven.

It requires several minutes for the column compartment to cool from the maximum temperature to the set initial temperature. During this cooling process, the column compartment back door will remain opened until the set initial temperature has been reached for several minutes. Then the door will be closed slowly. The reason is that it takes some time the column compartment to reach its internal temperature equilibrium. Otherwise temperature fluctuation may occur.

Therefore, you have to wait about ten minutes between one analysis is completed to the start of the next. After that, you can press [START] again to start the next temperature programming process.

2. 2. 4 Detector parameters

GC1120 presently has three connections and can fit 6 types of detectors, i.e. FID, TCD, ECD, PID, NPD and FPD. Detector selection, measuring range, polarity and current setting can be set by keypad on the control panel.

Use FID as example. Select 3 on main menu screen and select 1 on detectors screen to set parameters. Refer chapter 2.1.5.

For sensitivity switch, the initial set value is 10^{10}. You can set 10^{10}, 10^{9}, 10^{8} and 10^{7} respectively according to requirement. LED on FID amplifier operating board indicates sensitivity steps.

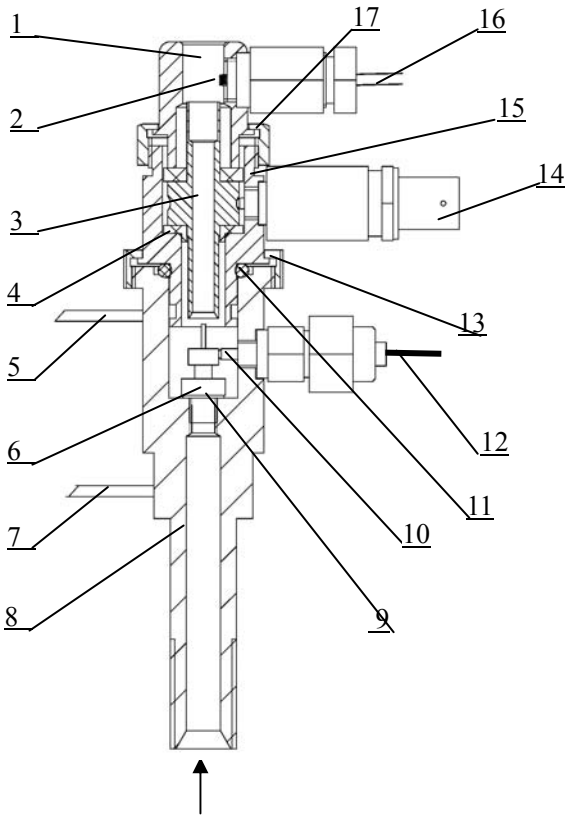
3 Detector system

GC1120 provide single or double flame ionization detector (FID) installed configure. And thermo conductivity detector (TCD), electron capture detector (ECD), photoionization detector (PID), flame Photometric Detector (FPD), and nitrogen phosphorus detector (NPD) are provided for GC1120 chromatography in optional. This chapter will introduce FID's installation and usage.

3.1 Flame ionization detector (FID)

FID of GC1120 gas chromatography is a tube-type structure. Figure 3-1 shows the structure of FID detector. Cylindrical detector base has a structure that ensures small post column dead space between column and nozzle. Flame nozzle has good insulation against ground and resistance to high temperature. Please refer to figure 3-2 for its structure. Independent of the thimble-type emitter structure can be easily fixed in the nozzle in order to ensure that FID detector with good performance consistency. Around the system by the platinum wire from the ignition filament mounted on top of detector. The stainless steel cylindrical receiver has good insulation against ground and high receiving efficiency.

Collector sign seat can be easily sign line connector, and the sign line with the FID of the other side of the input amplifier connected to shielding box.



1. Cap
2. Ignition filament（GC0070）
3. Collector
4. Insulated gasket
5. Air input
6. Nozzle（GC0059）
7. H_2 input
8. FID seat
9. Sealing gasket(septa)(GC0060)
10. Thimble-type emitter
11. Sealing gasket（GC0034）
12. Emitter cable
13. Big nut
14. Collector sign seat
15. Upside set
16. Ignition cable
17. Small nut

Figure 3-1 FID Structure

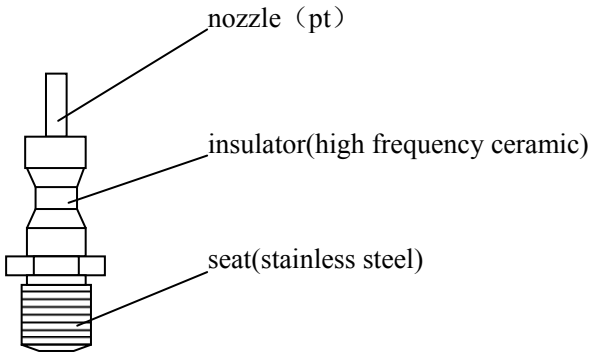


Figure 3-2 Nozzle Structure

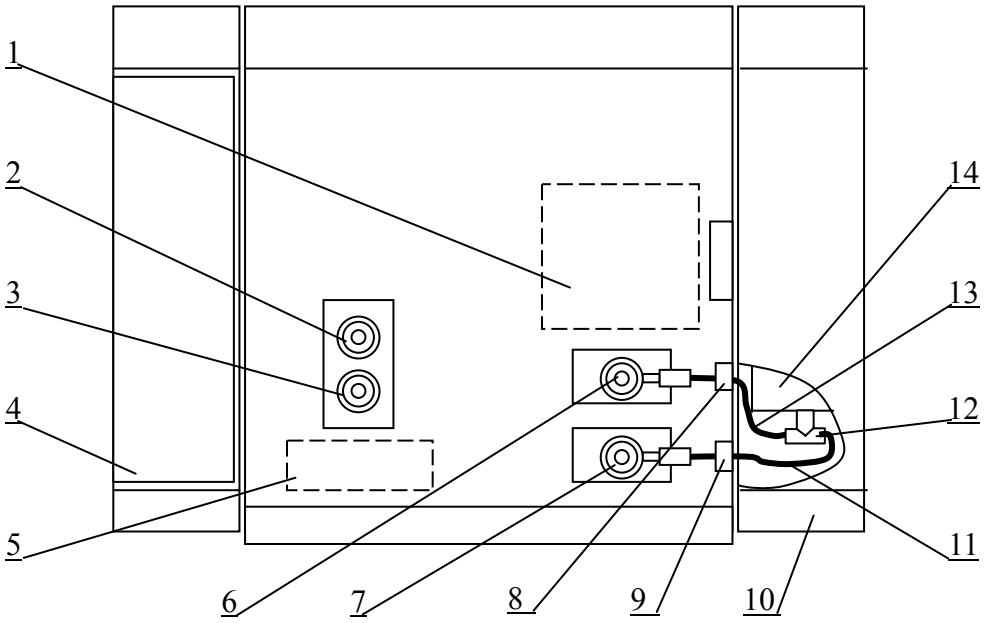
Notes

FID mounted on the nozzle diameter of the vent about φ0.4mm, analysis of adaptation in both packed column capillary column analysis. If the user needs analysis dedicated FID capillary column vents (the diameter of the nozzle about φ0.3mm), please contact us.

GC1120 provided 8 graphite sealing gasket (GC0060) in the accessories. The nozzle can be replaced if blocking or contamination occurs during using. When a new graphite sealing gasket, sleeve (GC0093) vents will be tightened up so that with the vents sealed strictly between FID base.

3.2 Connection of FID to mainframe

Single/Double FID is located at front top of the mainframe. FID base is installed in a heat conductive aluminum case. This heat conductive case also embodies an electrical heating element and a ceramic platinum resistance which are connected to the main circuit board of the programmed temperature works. The signal wire is connected to FID micro electric current amplifier by high frequency cable. The outlet of emitting through the rear of a host of line-line adapter to plug high-pressure input module (+250 V,-250V). Ignition filament wire connected to the electrical box inside the upper part of the ignition switch. The outlet of chromatographic column is packed into the inlet of the FID on the top of the column compartment. Connected and sealed with nuts and graphite gaskets. Hydrogen and air are drawn from the gas path control system by a stainless pipe. Please refer to figure 3-3 for connection sketch of FID and mainframe.



1．TCD setting　2．injector B　3．injector A

4．gas path system　5．capillary injector setting　6．FID（B）

7．FID（A）　8．FID（B）ignition switch　9．FID（A）ignition switch

10．electrical box　11．FID（A）high frequency cable

12．Q9-50KJK three adapters　13．FID（B）high frequency cable

14．FID Shielding Box Amplifier

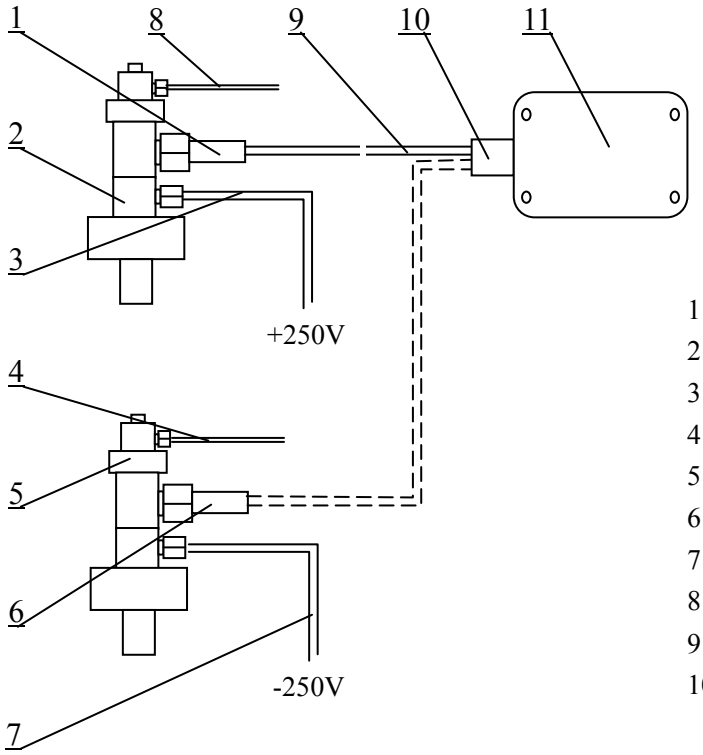
Figure 3-3 Connection sketch of FID and mainframe（Top）

3. 3 FID working scheme

FID of GC1120 has two working schemes. One is used as single detector, the other is used as double detectors with mutual compensation.

3. 3. 1 Single detector working scheme

Since only one micro current amplifier is equipped, only one end of the long high frequency part is connected to the single inlet of shield box in FID micro current amplifier; the other end is connected to the signal outlet of the detector to be used. Single detector working scheme applies to thermostatic packed column and capillary column analysis. Please refer to figure 3-4 for the signal connection diagram of single detector.



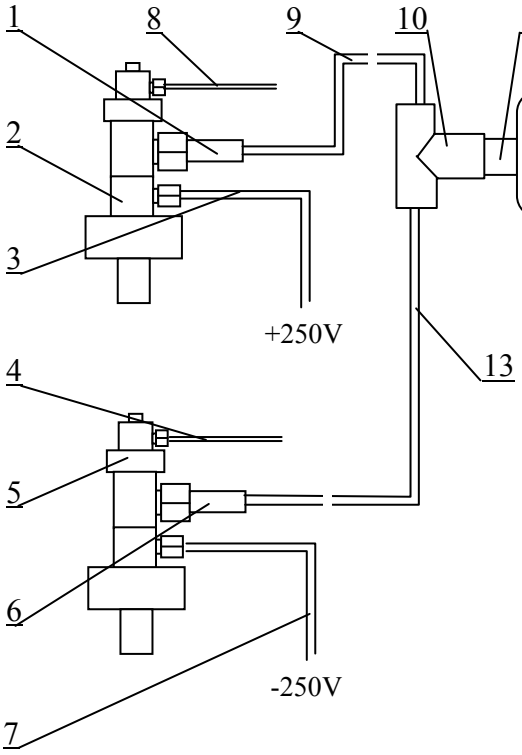
1. FID（A）collector outlet
2. FID（A）
3. FID（A）emitter cable
4. FID（B）Ignition cable
5. FID（B）
6. FID（B）collector outlet
7. FID（B）emitter cable
8. FID（A）Ignition cable
9. high frequency cable
10. Shielding Box Amplifier Input
11. Shielding Box Amplifier

Figure 3-4 Signal Connection Diagram Of Single Detector

3. 3. 2 Double detectors working scheme

Please refer to figure 3-5 for signal connection diagram of double FID detector.

Two emission probes are connected to positive high voltage and negative high voltage respectively. Outlet signals from the two probes can be superimposed by a short high frequency cable and a Q9-50KJK three way connection. Then the superimposed signal is connected to the inlet of the micro current amplifier by a long high frequency cable (see figure 3-6). Compensation connection applies to double packed column temperature programming process and to thermostatic process. Compensation connection can offset the base current signal of the two detectors and therefore is beneficial to base line drift reduction.



1. FID（A）collector outlet
2. FID（A）
3. FID（A）emitter cable
4. FID（B）Ignition cable
5. FID（B）
6. FID（B）collector outlet
7. FID（B）emitter cable
8. FID（A）Ignition cable
9. FID（A）high frequency cable
10. Q9-50KJK three adapters
11. Shielding Box Amplifier Input
12. Shielding Box Amplifier
13. FID（B）high frequency cable

Figure 3-5 Double Connection Diagram Of Double Detector

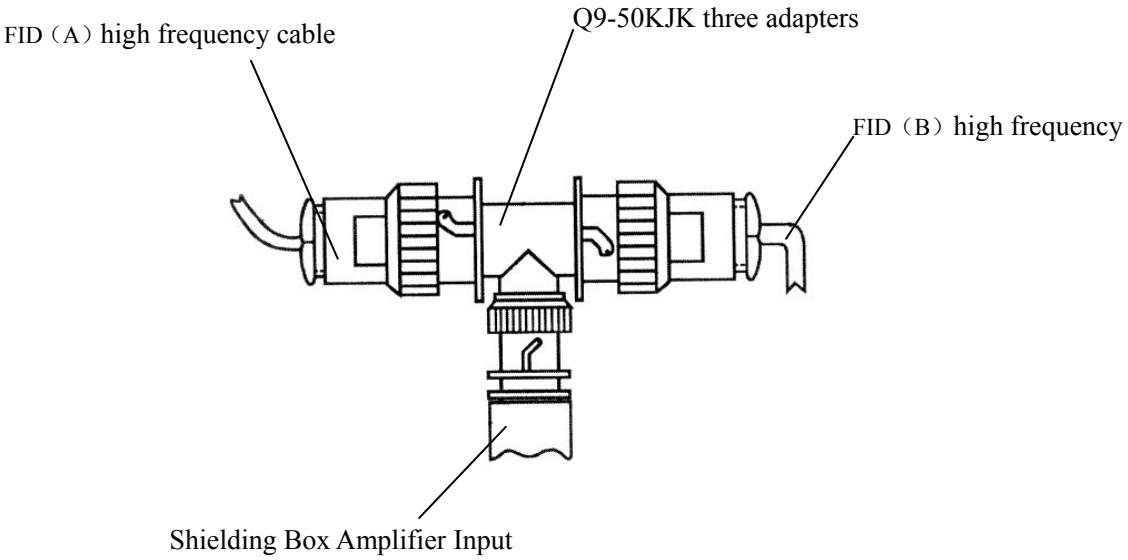


Figure 3-6 Connection Diagram Of Double FID Compensation High Frequency Cable

Notes

1. For Ex-works: FID (A) emission probe is connected to +250V high voltage; FID (B) emission probe is connected to –250V high voltage. When the two detectors are used separately, the peak direction is reversed.
2. The emission and ignition probes of FID (A) and FID (B) share one platinum-gold filament filament respectively, controlled by ignition switch of FID (A) and FID (B). The probe is charged with high voltage when the switch is off. The ionization chamber ignites when the switch is on. The contact time should be controlled to 4~5 seconds when you turn on the switch. Do not press the switch for a long time. Otherwise the filament may burn.

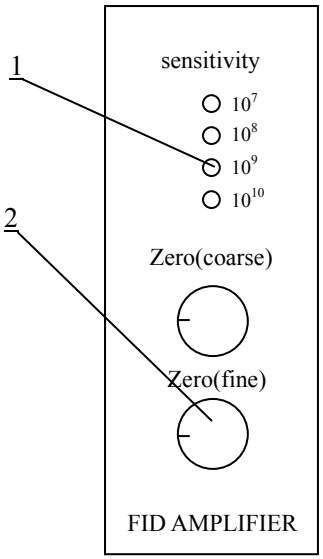
Notes

The double column can not have samples injected simultaneously since only one micro amplifier is equipped.

3.4 FID micro-current amplifier and operating board setting

GC1120 FID micro current amplifier employs the working principle of current/ voltage shift. The ion current collected by FID collector is transformed and amplified by high frequency cable and transferred to recorder or data processor.

GC1120 FID amplifier measuring range and polarity setting are accomplished by computer system (please refer to 2.2.4). Zero adjustment (coarse and fine adjustment) function is accomplished by two knobs on the FID amplifier operation panel. Zero adjustment (coarse and fine adjustment) knob (base current compensation knob) can adjust the position of the recording pen of recorder, data processor and chromatograph workstation. Please refer to figure 3-7 for FID micro current control amplifier panel diagram



1. sensitivity (range) indication light
2. zero(current compensation) adjusting knob

Figure 3-7 FID micro current control amplifier panel diagram

Notes

GC1120 FID amplifier panel has no attenuation switch. Signal attenuation will be accomplished by data processor or chromatograph workstation.

Notes

FID amplifier circuit board and control panel are installed for GC1120 except GC1120-2 configuration.

4 Instrument installation and running

4.1 Power source requirement

220V power source should be supplied to GC1120 chromatography (about 2 KW). No other high load equipment should share the power supply circuit with the chromatograph. If electrical network voltage exceeds 220V±10%, or severe interference occurs, a 3 KW electrical AC voltage regulator should be installed. The instrument should be perfectly earthed.

Warning

A dedicated earth line should be installed. Do not use the neutral line as earth line.

Warning

When power cord connected to the host, the host power switch will light that indicate the host band has ~ 220V voltage. The power cord must be unplug from the host, before working for disassembly.

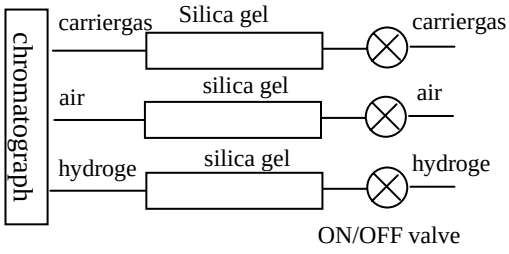
4.2 Gas supply preparation and treatment

4.2.1 Gas supply

Three types of gas are needed for GC1120 FID, they are carrier gas (normally nitrogen), hydrogen and air. Nitrogen purity should be no less than 99.99%, hydrogen no less than 99.9% and air should be free water, free oil and free pollutant.

4.2.2 Gas supply treatment

These gases should undergo strict purification treatment before entering instrument. A general purpose purifier (accessory GC0010) is provided for the basic configuration chromatograph. Please refer to Figure 4-1 for details. The purifier consists of purification tube and switch valve that is installed between chromatograph and gas source. Purification tube is equipped with activated silicon gel. Turn the switch on to supply gas to chromatograph.



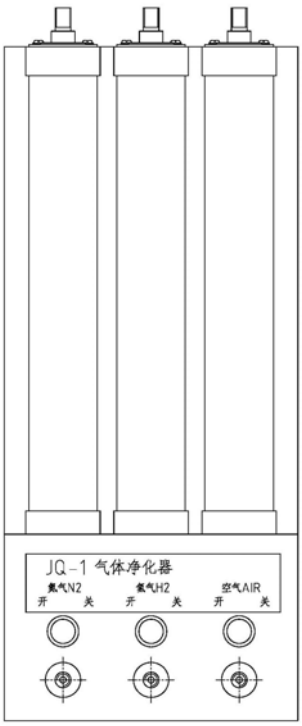
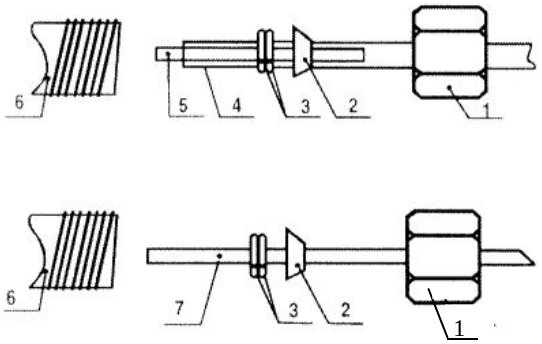


Figure 4-1 Purifier

4.3 External gas path connection

4.3.1 Connect gas supply pipe to gas path

GC1120 gas chromatograph gas supply pipe is made of Φ3×0.5 PE pipe (accessory GC0011) or Φ3×0.5 SS pipe. Nuts size is M8×1, Φ3.2 (accessory GC0020). Please refer to Figure 4-2 for connection diagram of these two kinds of pipe to joint. In the figure, Φ3×0.5 PE pipe uses sealed liner to enhance sealing point strength to ensure gas supply and sealing. If Φ3×0.5 SS pipe is used, Φ2×0.5×20 sealed liner is not required. The sealing ring can be replaced with a 5mm length Φ5×1 PTFR pipe. Two sealing rings should be used for the purpose of sealing. Maximum sealing pressure is 0.5 Mpa~0.8Mpa (5kgf/cm^2~8kgf/cm^2). Check gas path leakage. Soapsuds with high alkalinity should not be used to avoid corrosion. Diluted Lauryl sodium sulfate solution is recommended.



1. Screw cap（M8×1，φ3.2）（GC0020）
2. Sealing gasket(phosphorous copper)（GC0017）
3. Sealing ring（6×1.9）2 只（GC0019）
4. φ3×0.5 PE pipe（GC0011）
5. Sealing gasket(φ2×0.5×20 Ss pipe)(GC0016)
6. connection
7. φ3×0.5 Ss pipe（GC0013）

Figure 4-2 Connection Diagram Of Outer Gas Path Joint

Notes

If Φ3×0.5 SS pipe (GC0013) as a gas tube, it should order separately.

4.3.2 Outer gas path connection

Cut 6 Φ3×5 PE pipe (GC0011) into the length required, then connect the cylinders, the purifier , and the mainframe with these pipes as shown in figure 4-3.

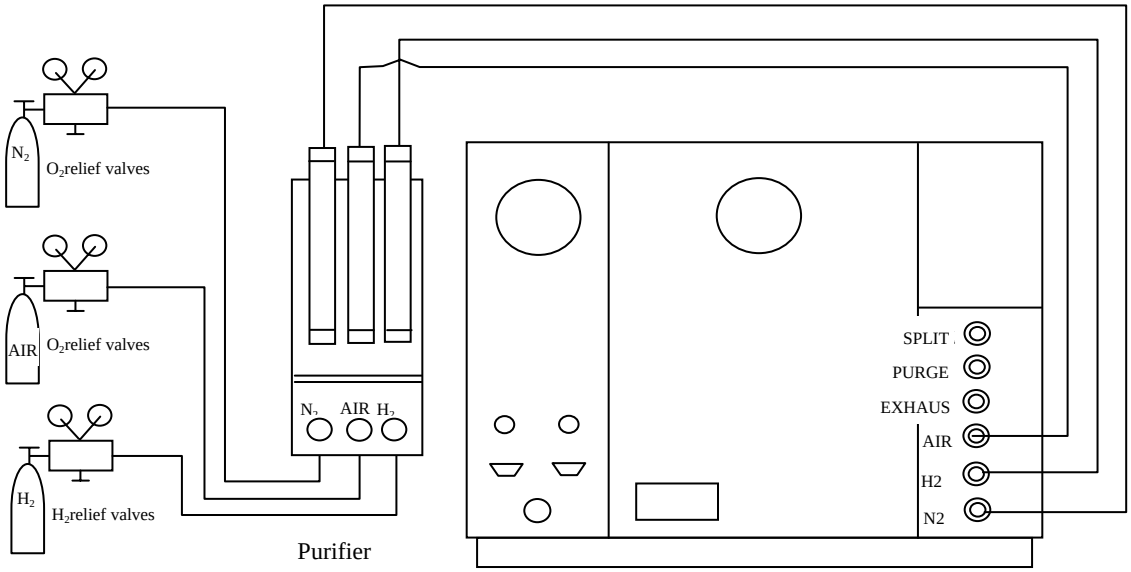


Figure 4-3 Connection Diagram Of Outer Gas Path

4. 3. 3 Outer gas path leakage test

Leakage test should be done after the outer gas path is connected. Test procedures are:

With gas cylinder:

1) Turn off the carrier gas flow control valve, hydrogen, air needle valve on the packed column (scale indicator is about “1”).

2) Turn on high pressure valve on cylinder. Turn the low pressure adjusting rod slowly until the pressure gauge reaches an indication of 3kg/cm^2.

3) Turn off high pressure valves on all cylinders

4) Turn off all high pressure valves on all cylinders. The low pressure gauge indication should remain. Otherwise leaking is indicated and should be excluded.

With gas generator:

1) Turn off the carrier gas flow control valve, hydrogen, air needle valve on the packed column (scale indicator is about “1”).

2) Turn on power of generator, the flow indicator on generator should be zero. Otherwise leaking is indicated and should be excluded. This method only suit for generator with flow rate display.

4.4 Packed column installation

When installation, enough length of empty column (at least 50mm) should be left for Φ5-6mm column injection so that the injection needle can be inserted into the vaporizer fully. For Φ3-4mm column, do not need leave empty column because a connector will be used.

Due to the rigidity of the column, the Φ5.7mm packed glass column should be installed both ends of injection and detector inlet at the same time.

4.4.1 Installation of Φ3mm and Φ4mm metal column to packed column inlet

Please refer to Figure 4-4 and Table 4-1 for installation procedures:
1) Fix nuts (No.7), graphite sealing gasket (No.5), packed column Transition tie-in (Inj) (No.2) to the packed column in turn.

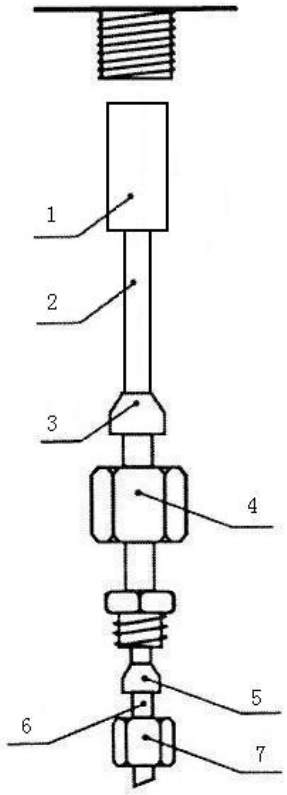
2) To enable the column head in the transition tie-in (Inj) hole (see Figure below), who live in this location first hand tighten the nut, and then use the appropriate two wrenches, one caught in the nut, and a second clip in the transition joints on the reverse screw tight seal.

3) Fix nuts (M12×1, Φ5.2) and graphite sealing gasket (Φ5) to transition tie-in (Inj) in turn.

4) Bushing (No.1) set during the transition tie-in (Inj), and then transition tie-in (Inj) conjunction with the bushing pushed into the inlet outlet connector, the insert deeply as possible.

5) Hold this position, tighten the nuts (M12×1, Φ5.2) with injector outlet joint manually, then tighten up.

packed column inlet



No	Name	Size	
1	bushing	ϕ5mm（GC0054）（already installed）	ϕ5m（GC0054）
2	Transition tie-in (Inj)	ϕ3mm（GC0052）（already installed）	ϕ4mm（GC0053）
3	sealing gasket	ϕ5mm（GC0041）	ϕ5mm（GC0041）
4	nuts	M12×1，ϕ5.2mm（GC0045）（already installed）	M12×1，ϕ5.2mm（GC0045）
5	sealing gasket	ϕ3mm（GC0039）	ϕ4mm（GC0040）
6	packed column	ϕ3mm（o.d.）	ϕ4mm（o.d.）
7	nuts	M8×1，ϕ3.2mm（GC0043）（already installed）	M8×1，ϕ4.2mm（GC0044）

Table 4-1

Figure 4-4 installation of Φ3mm and Φ4mm metal column to packed column inlet

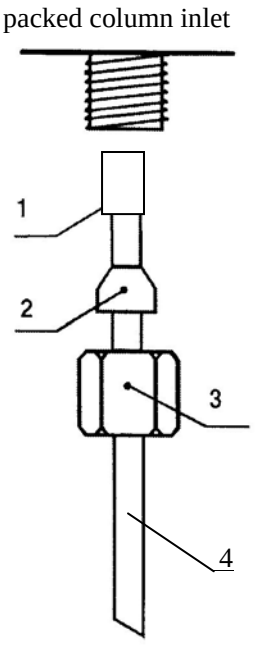
4.4.2 Installation of Φ5mm metal column to packed column inlet

Please refer to Figure 4-5 and Table 4-2 for installation procedures:

1) Fix nuts (No.3), graphite sealing gasket (No.2) and pushing (No.1) to packed column in turn.

2) Insert column into injector outlet joint as deep as possible.

3) Hold this position, tighten nuts and injector outlet joint manually, then tighten up by spanner.



No	Name	Size
1	bushing	ϕ5 (GC0054)
2	sealing gasket	ϕ5 (GC0041)
3	nuts	M12×1，ϕ5.2 (GC0045)
4	packed column	ϕ5 metal column

Table 4-2

Figure 4-5 installation of Φ5mm metal column to packed column inlet

4.4.3 Installation of Φ6mm metal column and Φ5.7mm glass column to packed column inlet

Please refer to Figure 4-6 and Table 4-3 for installation procedures:

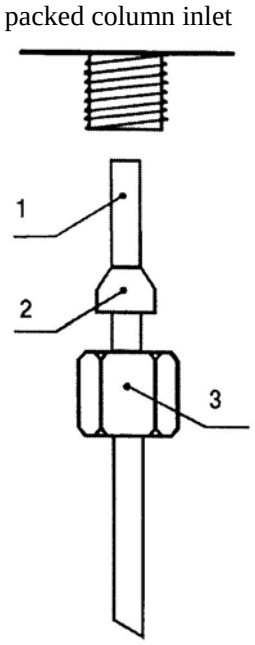
1) Fix nuts (No.3), graphite sealing gasket (No.2) and pushing (No.1) to packed column in turn.

2) Insert column into injector outlet joint as deep as possible.

3) Hold this position, tighten nuts and injector outlet joint manually, then tighten up by spanner.

Warming

Installation of the glass, the nut screw too tight may cause column broken.



No	Name	Size	
1	packed column	Φ6 metal column	Φ5.7 glass column
2	sealing gasket	Φ6 （GC0042）	Φ6 （GC0042）
3	nuts	M12×1， Φ6.2 （GC0046）	M12×1， Φ6.2 （GC0046）

Table 4-3

Figure 4-6 installation of Φ6mm metal column Φ5.7mm glass column to packed column inlet

4.4.4 Installation of Φ3mm and Φ4mm metal column to FID inlet

Please refer to Figure 4-7 and Table 4-4 for installation instruction.

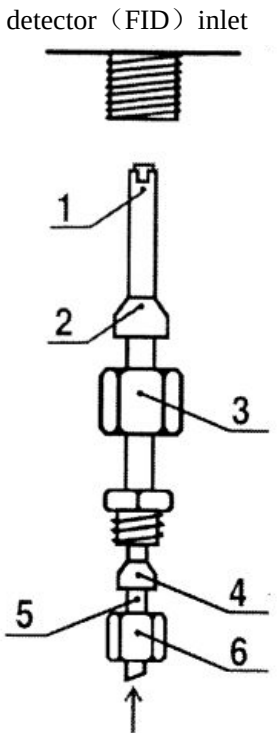
1) Fix nuts (No.6), graphite sealing gasket (No.4) and transition tie-in (Det) (No.1) to packed column in turn.

2) So that the transition tie-in (Det) around 1 ~ 2mm (see Figure below), who live in this location first hand tighten the nut, and then use the appropriate two wrenches, one caught in the nut, and a second clip in the transition joints on the reverse screw tight and sealed.

3) Fix nuts (M12×1, Φ6.2) and graphite sealing gasket (Φ6) to transition tie-in (Det) in turn.

4) Push column head into joint.

5) Hold this position. Tighten nuts and joint manually. Then tighten up with a spanner.



No	Name	Size	
1	Transition tie-in (Det)	ф3mm （GC0050）	ф4mm （GC0051）
2	sealing gasket	ф6mm （GC0042）	ф6mm （GC0042）
3	nuts	M12×1， ф6.2 mm （GC0046）	M12×1，ф6.2mm （GC0046）
4	sealing gasket	ф3mm （GC0039）	ф4mm （GC0040）
5	packed column	ф3mm （o.d.）	ф4mm （o.d.）
6	nuts	M8×1， ф3.2mm （GC0043）	M8×1， ф4.2mm （GC0044）

Table 4-4

Figure 4-7 installation of Φ3mm and Φ4mm metal column to FID inlet

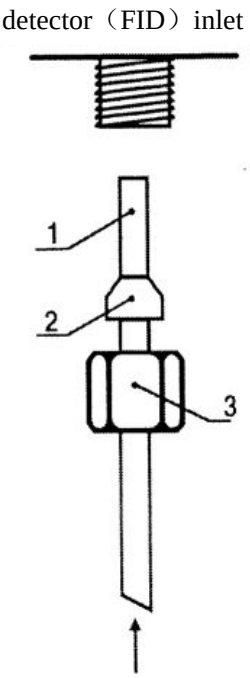
4.4.5 Installation of Φ5mm, Φ6mm metal column and Φ5.7mm glass column to FID inlet

Please refer to Figure 4-8 and Table 4-5 for installation instruction.

1) Fix nuts (No.3) and graphite gasket (No.2) to packed column in turn with no transition joint.
2) Insert column head into FID inlet to the bottom.
3) Hold this position. Tighten nuts (M12×1 Φ6.2) and FID inlet joint manually. Then tighten up with a spanner.

Warning

After the column is installed, all joints and nuts should be subjected to leak test under room temperature and operating temperature of column compartment, injector and detector.



No	Name	Size		
1	packed column	ϕ5 metal column	ϕ6 metal column	ϕ5.7 glass column
2	sealing gasket	ϕ5 （GC0041）	ϕ6 （GC0042）	ϕ6 （GC0042）
3	nuts	M12×1，ϕ5.2 （GC0045）	M12×1，ϕ6.2 （GC0046）	M12×1，ϕ6.2 （GC0046）

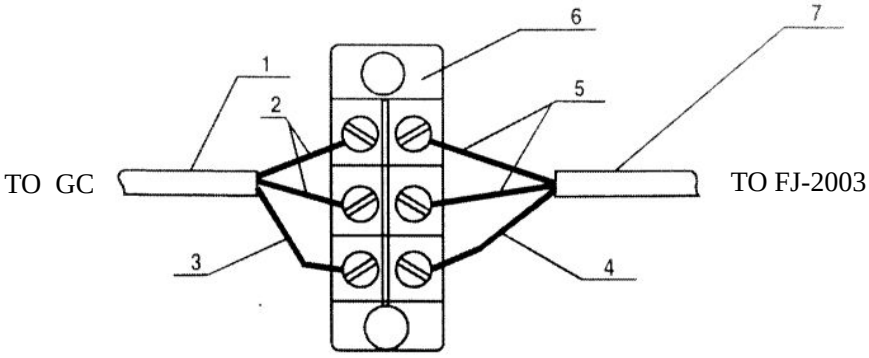
Table 4-5

Figure 4-8 installation of Φ3mm and Φ4mm metal column to FID inlet

4.5 How to connect recorder or data processing equipment

GC1120 FID amplifier output signal is connected to “detector signal” socket located at the lower right part of the mainframe (please refer to Figure 2-3). The signal cable can be connected to recorder, data processor or FJ-2003 chromatograph workstation. This signal is controlled by the zero knob on the microprocessor panel. FID amplifier sensitivity (range) and change of polarity can be set on the panel for recorder, data processor or chromatograph workstation. Signal attenuation function can be controlled by recorder, data processor or chromatograph workstation. The procedures are:

1) Insert one end of the data processor signal cable (GC0068) into the socket printed “detector signal” located at the lower right part of the mainframe (note: port No.3 is for earth, port No.1 & No.2 are for chromatographic signal).
2) The other end of the cables are connected to signal input of the chromatograph workstation. Please refer to Figure 4-9.



1 data processor signal cable（GC0068）
2、5 plastic cable for chromatographic signal transfer
3、4 metal shielded cable earthing
6 connection outlet, FJ-2003 chromatograph workstation accessory
7 chromatograph signal cable, FJ2003 accessory

Figure 4-9 Signal connection of GC1120 with data equipment

4.6 FID thermostatic work

When installation completes, the chromatograph can start running. Operation procedures of FID under thermostatic condition are:

1) Connect external gas path of carrier gas, air and hydrogen and then carry out leak test.
2) Install aged chromatograph column (from injector to FID)
3) Open carrier gas supply valve, adjust low pressure regulator rod until the low pressure gauge indication reaches 3.5 kg/cm^2~6 kg/cm^2. Adjust the two carrier gas flow control valves on the gas path panel until the flow rates of path A and path B reach an appropriate value (refer to the dial-flow rate chart under specific separation condition for knob adjustment).
4) Turn on mainframe power, set temperature for column compartment, detector and injector as specified in chapter 2. For example: column compartment 150°C, injector 180°C and detector 180°C.
5) Set FID amplifier parameters on the microprocessor panel. For example: sensitivity (range) 10^8, polarity "1" (set output to "+")
6) Recorder zero setting: turn on recorder power and recording pen switch, short circuit three recorder input ends, set recorder measuring range to 1mV, adjust the adjustable resistor of the recording pen until the recording pen is positioned to baseline.
7) Connect recorder signal cable (GC0068), please refer to the previous chapter for details.
8) After temperature of injector, FID and column compartment reach equilibrium, open air and hydrogen gas source, adjust low pressure regulator rod until the low pressure gauge of air reaches 3kg/cm^2~6kg/cm^2, hydrogen pressure gauge reaches 2kg/cm^2~3.5kg/cm^2. Adjust the two air needle valves and two hydrogen needle valves on gas path control panel (please refer to Figure 1-6). Adjust the A & B air flow valves and A & B hydrogen valves until an appropriate flow rate is reached (refer to the dial-flow rate chart under specific separation condition for knob adjustment).
9) Ignition: press ignition buttons on FID amplifier control panel. Baseline will deviate the original position after ignition. To check if the flame is ignited or not, the two normal practices are :
    a) Change the flow rates of two hydrogen paths alternatively. If the recording pen responses, flame is ignited.
    b) Put a smooth metal surface or glass sheet at ion chamber "vent" (please refer to Figure 3-1). If steam condensed on the surface of metal or glass, flame is ignited.
10) Adjust FID amplifier "coarse" and "fine" base current compensation knob (zero adjustment) until recording pen is positioned to an appropriate baseline.

Note

1) If peak direction is reversed, press [POLAR] key on the microprocessor panel to reverse polarity to change peak direction.
2) If chromatograph data processor or FJ-2003 chromatograph workstation is used, press [POLAR] to shift polarity of the signal, or substitute the positive and the negative cable connection.
3) FID sensitivity is determined by the velocity ratio of hydrogen to carrier gas (or to capillary column carrier gas + make-up gas). There is an optimum ratio. Normally if a target solute has high concentration, increase air velocity is necessary. If the target solute has low concentration, decrease air velocity. Figure 4-5 shows the optimum ratio of hydrogen to carrier gas velocity.

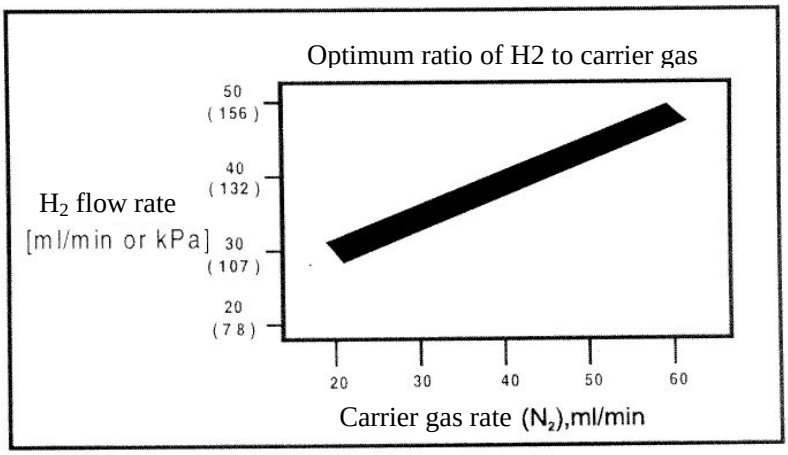


Figure 4-9 Optimum ratio of hydrogen to carrier gas velocity

4. 7 FID temperature programmed work

FID operation procedures at temperature programming are:

1) Same as 1)~10) in the Chapter 4.5.
2) Set all temperature programming parameters according to the procedures specified in Chapter 2.
3) Adjust base current, improve baseline drift as specified below:
4) Reset FID operating mode to double FID. Set FID amplifier range (sensitivity) to desired status. Adjust “Zero” knob to position the recording pen properly.
5) After the baseline is stabilized, check actual temperature of controlled device. Injection may start if the actual temperature reaches the set value. Press [START] key to start temperature programming. One stage of programmed temperature analysis is completed when the “COOL” light turns on.

4.8 FID operational notice

1) This detector is a high sensitive detector and high purity carrier gas (99.99% N_2) is required. Carrier gas, hydrogen and air should be treated by purifier.
2) When column is being aged, do not connect column to detector to avoid detector contamination. The maximum operating temperature of chromatographic column is 230°C. Do not open hydrogen source when column is being aged.
3) Turn off hydrogen and air source before temperature equilibrium is reached to avoid water accumulation in detector.
4) Do not press the FIRE button too long to avoid decaying the ignition filament.
5) The chromatographic column should be fully aged when the maximum sensitivity or temperature programming is being employed.
6) Turn on carrier gas first after the chromatograph is switched on. Start ignition when FID reaches a temperature higher than 100°C.
7) For ignition convenience, it is recommended that the hydrogen flow rate be increased before ignition. Decrease the hydrogen flow rate slowly to the flow rate desired after ignition.
8) Turn off hydrogen (extinguish) first, then start cooling, and cut off carrier gas to switch the chromatograph off.

Warning

When hydrogen is used as a fuel for FID, if hydrogen valve is opened while the column is not connected to the detector entrance joint, hydrogen will flow into the heating chamber leading to explosion. Therefore, column should be installed between the detector entrance and the injector, or M12×1 nuts (GC0049) and Sealing gasket(septa) Φ10×5 (GC0033) should be inserted into FID inlet and tightened up with a spanner.

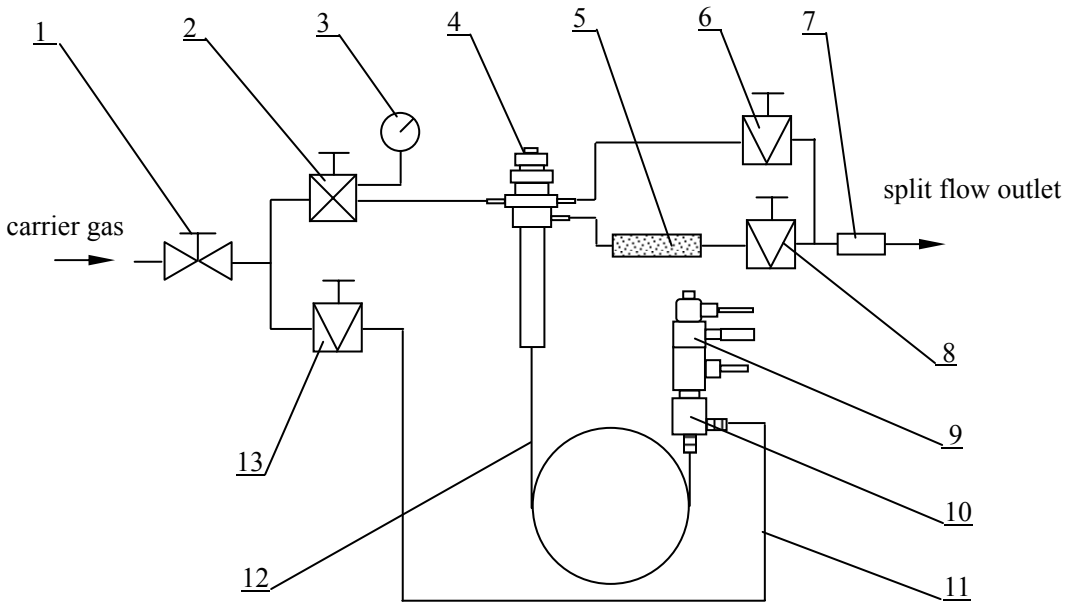
5 Capillary column analysis system

This instrument can analyze with capillary column. Capillary column injection may be in split/splitless flow or direct injection. This chapter mainly deals with installation and operating manual for capillary split injectors. Operating manuals are available for users of other types of capillary column injectors.

5.1 Introduction to capillary flow

GC1120-1, GC1120-4, GC1120-5 and GC1120-6 types are not only equipped with a capillary column injector, but also with an independent carrier gas flow control system. Gas system has the "Split" back-pressure valve knob, "Septum Purge " needle valve knob, "Makeup" needle valve and capillary pre-column pressure gauge (see Figure 1-6).

Figure 5-1 shows capillary injector gas path.



1．pressure control valve　2．flow control valve　3．pre-column pressure indicator
4．Capillary injector　5．filter　6．purge needle valve
7．split flow outlet　8．split back-pressure valve　9．FID DET
10．makeup gas connection　11．makeup gas pipe　12．capillary column
13．makeup gas needle valve

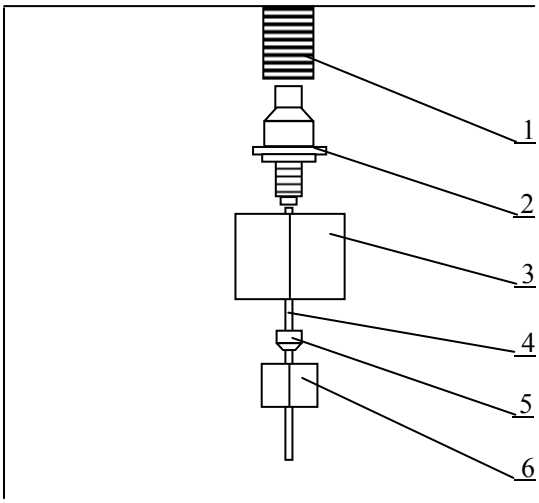
Figure 5-1 Capillary injector path diagram

5. 2 Capillary Split Injector Installation

Capillary injection device (including an independent carrier gas flow control) GC1120 models have factory installed capillary injector, including carrier gas, septum purge, split and makeup gas. Carrier gas The adjustment knob and the display of GC1120-5 on top panel, and GC1120-1 and GC1120-6 are carrier gas knob A in the instrument panel on the front. (See Figure 1-7)

Capillary injector installation is for the requirement of injector cleaning by user self.

1. Joint sets will be converted into the Screw cap (M12 × 1, φ8) in, pushed into the capillary injector exit, first hand the Screw cap (M12 × 1, φ8) and tightened sampling outlet, and then use Spanner to seal tightly. To tight metal seals require a larger force. (See Figure 5-2).
2. To divert split quartz liner tube (GC0056) one set of silicone rubber into the φ5 (or graphite) sealing gasket, and then inserted from the top of capillary column injector of imports, and as far as possible the segregation of quartz into the lining at the bottom of tube. Silicone rubber gasket can be used in capillary injector temperature 200 ℃ below, 200 ℃ above graphite gasket. (See Figure 1-5)
3. Positioning presentation will be streaming into the split quartz liner tube at the top of liner.
4. Positioning of the pressure put on the cap tightened up in the capillary injector on imports.
5. First Sealing gasket (silicon) (GC0035) put into the position of the hole, and then lead sealed in sealing gasket(silicon), sets down into the cooling cap and tightened up.



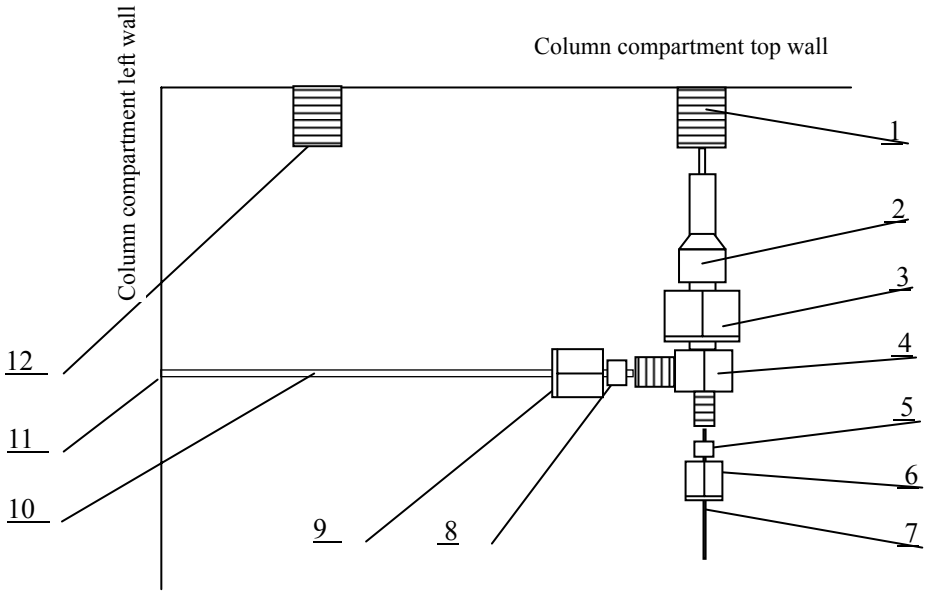
1．Capillary injector　2．Joint sets　3．M12×1，φ8 Screw cap
4．capillary column　5．Sealing gasket(capillary　6．M5，φ1.6 Screw cap

Figure 5-2 the scheme of split injector connection

5.3 Connection of makeup gas adaptor and hydrogen flame detector

Connection of makeup gas adaptor and FID detector is as following (see Figure 5-3):

1. Fit Φ6 graphite gasket (GC0042) to Capillary makeup connection (GC0057).
2. Insert Capillary makeup connection to FID connection as deep as possible.
3. Hold this position, tighten nuts with connection manually, then tighten up with a spanner.
4. Control makeup pipe from the left side of the central column leads, Catheter tip in the head, respectively, Add M8 × 1, φ2.1 Screw cap (GC0021) and sealing gasket (capillary transition φ2) (GC0038), Insert the capillary makeup connection side of import, and then tightened with spanner.



1．detector（A）　2．graphite gasket（φ6，graphite）　3．M12×1，φ6.2 Screw cap
4．Makeup gas adaptor　5．graphite gasket（Capillary）　6．M5，φ1.6 Screw cap
7．capillary column　8．graphite gasket（Capillary transition）　9．M8×1，φ2.1 Screw cap
10．Makeup pipe　11．Column compartment inside hole　12．Capillary injector

Figure 5-3 Connection diagram of makeup gas adaptor

5.4 Capillary column installation

After split flow vaporization tube is connected with make-up gas adaptor, capillary column can be installed. Fig 5-7 is the diagram of capillary column installation. GC1120 capillary column analysis system is adapted to all kinds of capillary, such as glass capillary column, flexible quartz capillary column (molten silica capillary column) etc. Flexible quartz capillary column 0.375~0.45mm. Different capillary packed column gasket should be used for different type of capillary column. Please refer to table 5-1.

If wide bore capillary column (such as inner diameter 0.53mm, 0.75mm, etc) is selected, user can expand gasket by drilling, the driller diameter should be approximate to capillary outside diameter.

Column Size	Gasket	Accessory No
Glass capillary column (Φ0.9~Φ1mm)	Graphite washer with steel jacket (ID Φ0.9mm)	GC0036
Flexible quartz capillary column (ID Φ0.9mm)	Graphite washer with steel jacket (ID Φ0.35mm)	GC0037
Note: For capillary column with a inner diameter of 0.05~0.25mm, its outside diameter is 0.375mm; inner diameter 0.32mm, outside diameter is 0.45mm; inner diameter 0.53mm, outside diameter is 0.69mm		

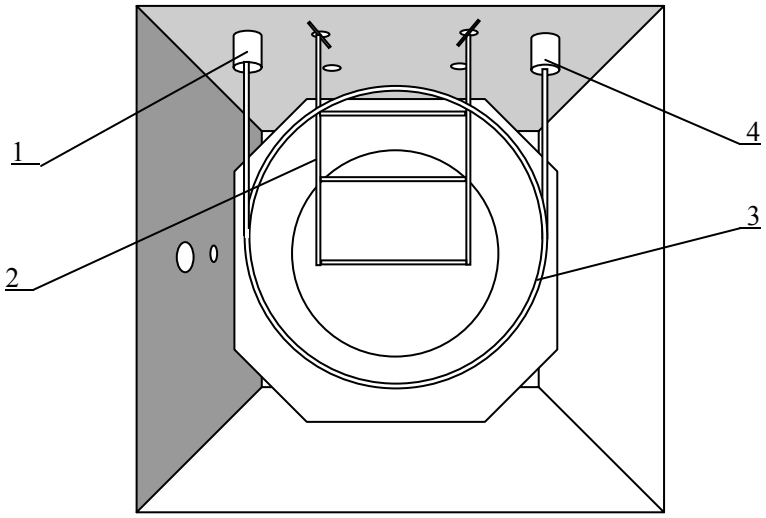
Table 5-1

Capillary column installation procedures are as follows:

1. Capillary column support（GC0061）inserted at 2 round holes on the upper front of column compartment (column compartment has 2 groups before and after the four round holes, the user can choose as a convenient Group 2 round holes inserted capillary column support), see Figure 5-4. And then wrapped the framework of capillary column, capillary column installed in the capillary column support.
2. Fit Φ1.6 Screw cap (GC0047), capillary gasket to both ends of capillary column in turn.
3. Push one end of column into Joint sets, capillary column head should exceed split point (about 30mm around). Hold this position, tighten up nuts by spanner (note: do not tighten too

much, or capillary column will break down), see Figure 5-2.

4. Push the other end of Capillary column into Capillary makeup connection, column head should exceed makeup gas point. Hold this position, tighten up nuts by spanner., see Figure 5-3.



1．Capillary injector　　2．capillary column support（GC0061）

3．capillary column　　4．FID detect

Figure 5-4 Capillary column support

5.5 Split flow injection capillary column analysis operation

Split flow injection capillary column analysis is the common analysis method. The advantages are its applicableness to all kinds of sample, wide concentration range, good qualitative characters to common sample; and because of its instant injection, it has high column efficiency.

GC1120-6 is double capillary injection configure. It use "carrier gas flow A" and "carrier gas flow B" knobs to adjust capillary total gas flow. Makeup gas flow adjust by the “total flow adjust” and " makeup adjust" knob. Split flows adjust by “Split adjust” and “Aux adjust1” knobs. Septum purge flow adjust by “Septum purge” and “Aux 2” knobs.

GC1120-1 and GC1120-4 use "carrier gas flow A" knob to adjust capillary total gas flow. Makeup gas flow adjust by the "makeup adjust" knob.

GC1120-5 adjust total gas flow by “total flow adjust” knob. Makeup gas flow adjust by the "makeup adjust" knob. These two adjusting knob installed at the top of the gas system on the panel (" carrier gas flow A "knob of the flow and the relationship between the number of laps, please check the Attachment 1 in a" carrier gas flow A, B "column," makeup adjust "of the flow and the relationship between the number of knob ring, please check the Attachment 1 in a" makeup adjust "column).

Operation procedures are as follows:

1) Connection and operation of FID detector, FID amplifier, recorder, data processor or chromatography workstation is the same as those of packed column FID (see chapter 4).
2) Adjust “carrier flow A” knob (flow control valve to control capillary column total flow) to required flow, equal to split flow required plus column flow. At this stage pre-column pressure gauge should have pre-column pressure indication. If pre-column pressure indication reaches 0.3 MPa, open “split” knob clockwise, until pre-column pressure indication becomes less than 0.3 MPa. Long term pre-column high pressure (>0.3 MPa) will lead to flow control valve damage.
3) Set temperature of detector, injector, column compartment to required value, and start temperature programming process.
4) When all temperatures increase to set value, open hydrogen and air needle valve knob (air flow A, hydrogen flow A), adjust them to required flow value.
5) Adjust “makeup adjust” knob to proper value (about “2~6”circles generally, make-up gas flow 2ml/min~37ml/min).
6) GC1120-1, GC1120-4 and GC1120-5 has a septum purge function, by the " septum purge "

knob to adjust (knob-conditioning system installed in the gas at the top of the panel) for the exclusion of excess carrier gas and the removal of the silicone rubber caused by the ghost peak (Purge flow normally divide about 3ml/min). Purge flow rate determination method of divide is based on the "purge" in soap film flow meter connected to or receive electronic digital flow meter. 5.6 see the use of soap film flow meter.

7) Adjust “split” knob (split flow needle valve, turn clockwise, larger indication value means large split flow velocity and split flow ratio). To measure split flow velocity, connect it with a soap film flow meter or electronic digital flow meter. Please refer to paragraph 5.6, use of soap film flow meter.

8) Linear velocity measurement: the linear velocity through column is measured by the following method. Inject a component that will not be retained by the stationary phase (typical sample is CH_4), use “WATCH” key on computer panel (please refer to chapter 2,2-10 page) to measure the retention time. If a data processor or chromatograph workstation is used, retention time will be calculated automatically. Linear velocity is determined by the flowing formula:

$$\text{Average linear Velocity } \overline{\mu}\ (cm/\sec) = \frac{\text{column length } L(m)\times 100}{\text{retention time } t(\sec)}$$

9) Calculation of column volume flow rate in capillary: because of the troubles of measuring volume flow at the outlet side of capillary, the column volume flow rate can be calculated approximately as follows:

$$Fc = 15\ \pi d^2 \overline{\mu}$$

式中：Fc ——column volume flow rate，ml/min

$\overline{\mu}$ ——mean linear velocity of carrier gas，cm/sec

d ——inner diagram of capillary column，cm

Or take off the make-up gas connection (connected with capillary column) from the joint of FID detector, connect it with the soap film flowmeter hose, then the flow rate can be measured directly by soap film flowmeter. But remember to shut off the make-up gas by adjusting “carrier flow B” to less than 1 circle.

10) Measurement of split flow ratio: for normal capillary column (0.22 mmID~0.32mmID), general separate flow ratio is from 50:1 to 500:1; for wide bore diameter and thick liquid film, the ratio is from 5:1 to 500:1; for small diameter from 50 um to 100 um, the ratio is more than 1000:1. The formula to calculate the ratio is:

$$\text{split flow ratio} = \frac{\text{split flow} + \text{Column Volume Flow}}{\text{Column Volume Flow}}$$

Example: column volume flow rate is 1ml/min, “split” outlet side measured by soap film meter (detained in part 5.6) or data flow meter is 199ml/min, so the split flow ratio is 200:1

Example 2: Inner diameter of column is 0.31mm, the mean linear velocity is, $\bar{\mu}$ =13.2cm/sec，he measured split flow rate is 54ml/min, so the column volume

$Fc=15\times3.14\times(3.1\times10^{-2})^2\times13.2=0.6ml/min$ split flow ratio $=\frac{54+0.6}{0.6}=91:1$

11) If the ideal linear velocity is not obtained and the expected retention time or split flow ratio is unsuitable, the carrier gas flow should be adjusted, then inject and calculate. Then adjust “split” knob of split flow, measure split flow and calculate split flow ratio, until an ideal result is reached.

12) After the base line is stabilized, start to inject and analyze.

13) because GC1120 carrier gas flow control valve provides total flow ahead of capillary column, a simple and approximate method can be used to measure split flow ratio, i.e. after getting the volume flow rate, it is not necessary to measure split flow rate, the following equation can be used directly:

$$\text{split flow ratio} = \frac{\text{total flow}}{\text{Column Volume Flow} + \text{purge flow}}$$

Notes

If the user directly into the large diameter capillary column, just close the "split" valves so that the "split" of the flow of "0" can be. In order to achieve better results, the user can the large diameter capillary column directly into the kind of special quartz tube liner.

Warning

When carried out by capillary analysis and the use of dangerous chemicals, diverted from the export of gas to be ventilated cabinet or the access of the chemical purification of the corresponding tube. A longer period of time, should be to remove parts installed in shunt flow control valve on the back-pressure adsorption tube (filters), replacement of the adsorbent, adsorption tube a little b-side to fill the glass wool.

5.6 Soap film flowmeter

Soap film flowmeter can be used to measure flow rate for split flow and post column flow (makeup gas output or FID nozzle).

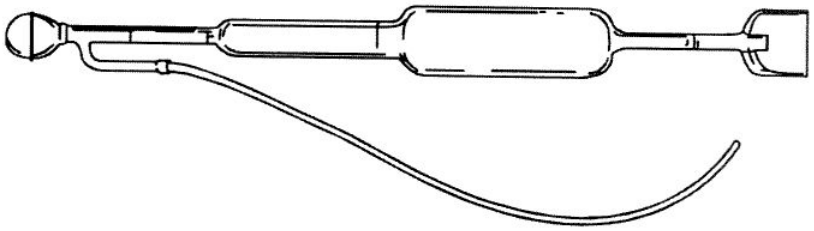


Figure 5-5 Soap film flowmeter sketch

Please refer to Figure 5-5 for soap film flowmeter sketch.

A soap film flowmeter has three velocity ranges: 1, 10 and 100ml/min. Suitable for measuring low (carrier gas) and high (FID air) flow rate.

Soap film flowmeter is the most basic and reliable tool to measure gas flow rate. When gas is passing the flowmeter, it will produce a bubble in pipe. The bubble movement in the pipe indicates the gas flow rate. Most soap film flowmeter have several sections, each section with a different diameter to suit different ranges of gas flow rate.

Measuring procedures:

1) Connect one end of hose to soap film flowmeter.
2) Connect the other end of the hose to gas source.
3) Inject soapsuds or leak test solution in the small ball of the soap film flowmeter.
4) Hold soap film flowmeter vertically. Press the small ball in the bottom to produce a soap film bubble.
5) When soap film start passing through the minimum graduate on the soap film flowmeter, press [WATCH] to start timing function.
6) Press [WATCH] again while the soap film passing a certain graduate of a certain range to stop timing function.
7) Flow rate unit: ml/min. If timing stopped at the first range graduate, the value of flow rate is 1/t of the indication; if stopped at the second range, the value is 10 times of 1/t; if stopped at the third range, the value is 100 times of 1/t.
8) Press [CE] and repeat steps 4) to 7) for at least once to confirm the validity of the flow rate

Notes

Soap film flow meter(GC0090) should be prepared or purchased by user.

6 Maintenance

6.1 Instrument maintenance

Proper maintenance can not only ensure instrument performance but also lengthen life of the instrument. Users should pay attention to the following instructions for maintenance:

{A}. Instrument should work strictly under specified conditions. Special measurements should be taken if the instrument working conditions do not comply with the specified ones.

{B}. The instrument should be operated strictly according operating rules. Creasy dirt, organics and other foreign matters are not allowed to enter detector and pipe to avoid pipe block or instrument malfunction.

{C}. Column operating temperature should be lower than the maximum allowable operating temperature of the solution on the absorbent. For high sensitive applications, column temperature should be much lower.

{D}. GC1120 gas pressure requirements are specified as follows:

carrier gas, 343,000Pa (3.5kg/cm^2~6kg/cm^2)

air.29,400Pa~588,000Pa(3kg/cm^2~6kg/cm^2);

hydrogen, 196,000Pa~343,000Pa (2kg/cm^2~3.5kg/cm^2).

If hydrogen is used as carrier gas for GC1120, then its pressure should be controlled to 343,000Pa (3.5kg/cm^2).

6.2 Hydrogen flame ionization detector cleaning

Take FID overall case apart, take off the electrode and insulated gasket, clean them with acetone or alcohol, and then dry them. If FID is heavily contaminated, immerse the disconnected parts into solution of ultrasonic cleaning. After ultrasonic cleaning, wash them with water and then alcohol, and finally dry them. When re-assembly them, make sure that the ignition filament be positioned around the nozzle and be prevented from being in contact with earth. The ignition filament should be lower than nozzle to avoid filament overheating and thus ensures detector sensitivity. If FID is contaminated by the solution on the absorbent, clean it with a solvent that may dissolve the contaminant.

Procedures to take apart the FID are as followed: unscrew the screws, take off the upside set. Use spanner (GC0071) to unscrew the collector sign seat. And then use the spanner to open a small nut, take cap, then remove the collector can be followed by two upper and lower insulation gaskets, the final use the spanner to o rotation with the nut fixed ignition filament(ignition cable from out of), taking the ignition filament.

To replace or remove the nozzle cleaning, you can spin with screwdriver under the pressure plate installed four M3 screws, remove the protective shield and the internal glass wool, and then remove the emitter cable, at this time there is no shielding material on the nozzle, use 8mm socket wrench (GC0093) spin out of the nozzle (GC0059). FID structure refer to Figure 3-1

Warning

A new Sealing gasket(septa) (GC0060) should be used when replacing a nozzle. Tighten the nozzle to avoid leak.

6.3 Injector cleaning

Injector, especially vaporization pipe, is to be contaminated easily. Therefore, it is important to clean injector.

Capillary injector cleaning methods are as follows: first spin under heat abstraction cap, take the needle guide and silicon seal (GC0035), then spin loose nut on position sleeve, pull out the position sleeve, and finally come up with a Split quartz liner tube (GC0056) and silicone rubber sealing gaskets(GC0032) or graphite sealing gaskets (GC0041), parts of the above cleaning with acetone or alcohol and drying. Removed Capillary column and Joint sets, the inside wall of injector tube can be cleaned with acetone or alcohol for several times. Then use a large volume of carrier gas to blow off the cotton fiber and dry the solvent. And then installed in accordance with section 5.2.2 capillary column dedicated to injector.

6.4 Chromatographic signal diagnose and trouble shooting

Please refer to Table 6-1 for chromatograph output message diagnose and trouble shooting.

Table 6-1

Malfunction	Diagnose	Checking and repair
1. No peak	1. Amplifier power source disconnected	1. Check amplifier and fuse
	2. Ionization line off	2. Check ionization line
	3. No carrier gas	3. Check carrier gas path, plugged or exhausted
	4. Recorder poor contact	4. Check recorder wiring
	5. Recorder malfunction	5. Check operating manual to exclude malfunction
	6. Injection temperature too low, sample not vaporized	6. Increase injector temperature
	7. Micro-injector plugged	7. Replace injector
	8. Injector septa leak	8. Replace septa
	9. Column connection loosened	9. Tighten column
	10. No flame (FID)	10. Ignition
	11. FID polarizing voltage unconnected or poor contact	11. Connect polarizing voltage, or exclude polarizing voltage poor contact
2. Normal retention time with decreased sensitivity	1. Attenuation too much	1. Decrease attenuation, increase high resistance
	2. Not enough sample quantity	2. Increase sample quantity
	3. Sample injection waste	3. Ensure sample is injected into system completely
	4. Injection syringe leak or plugged	4. Replace injector or unplug injector
	5. Carrier gas leak, especially injector leak	5. Leaking test
	6. Flow of hydrogen and air improper (FID)	6. Adjust hydrogen and air flow
	7. Detector no high voltage (FID)	7. Check or connect high voltage
3. Tail peak	1. Injection temperature too low	1. Adjust sample temperature
	2. Injection tube contaminated (sample or septa residue)	2. Clean sample tube
	3. Column oven temperature too low	3. Increase column temperature
	4. Unskilled sample injection	4. Improve sample injection skill, inject fast, take out fast
	5. Improper column (sample reacts with absorbent or solution on the absorbent)	5. Choose proper column

Table 6-1 (continue)

Malfunction	Diagnose	Checking and repair
4. Tongue peak	1. Column overloaded, too much sample 2. Sample condensed in the system	1. Decrease sample quantity 2. Increase column temperature, select a proper injector, column and detector temperature
5. No separated peak	1. Column temperature too high 2. Column too short 3. Solution on absorbent runs off 4. Improper stationary phase 5. Carrier gas rate too high 6. Unskilled injection	1. Decrease column temperature 2. Select a longer column 3. Change column or age column 4. Choose proper column 5. Decrease flow rate of carrier gas 6. Improve sample injection skills
6. Dome peak	1. Exceeds detector linear range 2. Recording damping too much	1. Decrease sample quantity 2. Adjust recorder damping
7. Flat peak	1. Amplifier input saturated 2. Change of zero position of recorder gearing	1. Decrease sample quantity and amplifier sensitivity 2. Check recorder zero position, or compare with other recorder
8. Zigzag base line	1. Flow control valve diaphragm fatigue 2. Cylinder pressure regulating valve output pressure change	1. Replace diaphragm or repair the valve 2. Adjust the pressure regulating valve
9. No injection and baseline changes in single direction (FID)	1. Detector temperature too low 2. Column heating stopped or out of control	1. Increase detector temperature to above 100°C, clean the detector, or increase the detector temperature to 200°C to drive away steam 2. Check temperature control system and heating filament Pt resistance
10. Pen tossing at a certain position	1. Recorder slide resistance contaminated	1. Clean slide resistance
11. Baseline mutates	1. Power plug poor contact 2. Exterior electrical field interference 3. Improper hydrogen, air flow rate (FID)	1. Plug firmly into the power outlet 2. Exclude electrical field interference 3. Adjust flow rate of hydrogen and air
12. Baseline drifts suddenly	1. Recorder low sensitivity 2. Recorder poorly earthed	1. Adjust recorder sensitivity 2. Ensure the recorder and the whole machine are properly earthed
13.Retention time increased, sensitivity decreased	1. Carrier gas flow velocity too low 2. Carrier gas flow rate changes after injection 3. Injector septa leaking	1. Increase carrier gas velocity, if gas path plugged, exclude it 2. Replace injection septa 3. Replace injector septa
14.Negative peak	1. Sample is injected to the wrong column 2. Switch position is wrong	1. Inject sample to a proper column 2. Change the switch position

Table 6-1 (continue)

Malfunction	Diagnose	Checking and repair
15. Baseline fluctuates irregularly under constant temperature	1. Instrument positioned improperly	1. Position the instrument on a proper table free of vibration and severe air convection
	2. Instrument not properly earthed	2. Instrument and recorder are properly earthed
	3. Solution on the absorbent runs off	3. Select a proper solution for the absorbent, column should be fully aged, do not raise the column temperature to the maximum allowable temperature of the solution on the absorbent, especially for detector of high sensitivity
	4. Carrier gas leaking	4. Leak detecting
	5. Detector contaminated	5. Clean detector
	6. Improper carrier gas flow rate	6. Adjust the carrier gas flow control valve to have a proper carrier gas flow rate, the cylinder pressure should be within the range of $50kg/cm^2$ to $150kg/cm^2$
	7. Improper hydrogen and air (FID)	7. Adjust hydrogen and air flow rate
	8. Amplifier unstable	8. Check and repair amplifier
	9. Recorder malfunction	9. Disconnect recorder signal cable and make it short circuit with metal wire. Repair it according to recorder specification if it is not in good condition
16.Extra peak *peak width of half-height increases suddenly	1. Previous sample high concentration peak	1. Inject sample when the previous solutes leaves the column completely
	2. Water or other condensate peak during temperature rising	2. Install a new cleaner or regenerate the cleaner under appropriate operating conditions
	3. Air peak	3. Exclude air in the injector
	4. Sample decomposed	4. Decrease injector temperature
	5. Sample contaminated	5. Ensure sample is free of impurities
	6. Sample reacts with solution on the absorbent or the stationary medium	6. Use another column
	7. Column head glass fiber or injector contaminated	7. Replace the column head glass fiber or clean the injector
	8. Injection septa contaminated or low molecule composition runs off	8. Dryer the septa under 200°C for 16 hours
17.Pen returns back to a position lower than the baseline and flame extinguished	1. Sample quantity too high	1. Decrease sample quantity
	2. Hydrogen or air flow rate too low	2. Adjust flow rate of hydrogen and air
	3. Carrier gas flow velocity too high	3. Choose an appropriate carrier gas flow velocity
	4. Flame nozzle contaminated or plugged	4. Clean flame nozzle (or un-plugged the flame nozzle)
	5. Hydrogen is used up	5. Ensure hydrogen supply

Table 6-1 (continue)

Malfunction	Diagnose	Checking and repair
18.Stepped peak not regressed to zero (flat head peak), pen shifts wiggly	1. Recorder pen gaining, improper damping 2. Improperly earthed 3. Sight AC signal feedback to recorder	1. Calibrate recorder gain and damp 2. Properly earth the instrument and the recorder 3. A 0.25f/250V capacitor, positive or negative input connected to earth is needed if necessary. (Note: Do not connect the capacitor to the positive or negative of the signal cable)
19.Baseline deviates zero	1. Recorder zero position abnormal 2. Column loss (FID) 3. Detector contaminated 4. Recorder malfunction	1. Short circuit recorder input with a filament, adjust to zero 2. Choose a column with minimum loss 3. Clean detector 4. Repair recorder
20.Pointed peak at irregular spacing	1. Dust or foreign substance burnt in the flame irregularly (FID) 2. Insulator leakage or high resistance connection relay leakage 3. Amplifier malfunction 4. Unstable flame	1. Discharge water from the pipe work, replace or activate the desiccant of the hydrogen filter 2. Detect leakage 3. Clean impurity in the flow path. Increase column temperature if contaminated 4. Adjust flow rate of hydrogen and air
21.Burr peak at regular spacing	1. Water condensed in hydrogen path 2. Gas leakage 3. Gas flow plugged 4. Unstable flame	1. Discharge water from the pipe work, replace or activate the desiccant of the hydrogen filter 2. Detect leakage 3. Clean impurity in the flow path. Increase column temperature if contaminated 4. Adjust flow rate of hydrogen and air
22.Baseline fluctuates periodically	1. Poor detector temperature 2. Improper column temperature control 3. Improper carrier gas flow 4. Carrier gas flow rate and pressure too low 5. Improper air and hydrogen adjustment (FID)	1. Improve Pt resistance control precision 2. Improve Pt resistance control precision 3. Adjust carrier gas flow 4. Replace carrier gas cylinder 5. Adjust air and hydrogen flow
23.Baseline drift in one direction only	1. Detector temperature increases or decreases significantly 2. Amplifier zero drift 3. Column temperature increases or decreases significantly 4. Carrier gas runs out	1. Keep detector temperature constant, temperature change at startup is normal 2. Repair amplifier 3. Keep detector temperature constant, temperature change at startup is normal 4. Replace carrier gas cylinder

Table 6-1 (continue)

Malfunction	Diagnose	Checking and repair
24.Baseline noise to much	1. Column contaminated or too much column loss	1. Replace column
	2. Carrier gas contaminated	2. Replace or regenerate carrier gas filter
	3. Carrier gas velocity too high	3. Adjust carrier gas flow velocity
	4. Carrier gas leakage	4. Detect leakage
	5. Improperly earthed	5. Instrument properly earthed
	6. High resistance contaminated	6. Clean the high resistance
	7. Recorder slide wire contaminated	7. Clean the slide wire
	8. Recorder malfunction	8. Repair recorder if recorder input end has noise when short circuited
	9. Injector contaminated	9. Clean injector and septa residue
	10. Hydrogen flow velocity too high or too low (FID)	10. Adjust hydrogen flow velocity
	11. Air flow velocity too high or too low (FID)	11. Adjust air flow velocity
	12. Air or hydrogen contaminated	12. Replace hydrogen and air filter
	13. Water condensed in FID	13. Increase FID temperature to remove water
	14. Detector cable poor contact	14. Replace or repair cable
	15. Detector insulation decreases (FID)	15. Clean detector
	16. Detector probe, nozzle or base contaminated	16. Clean detector
25.Baseline changes after temperature programming	1. Column loss increases as temperature increases	1. Choose proper column or age column
	2. Column flow velocity not calibrated properly	2. Calibrate column flow velocity
	3. Column contaminated	3. Replace column
	4. Solutions on absorbents are not the same for the two columns	4. Weight of solutions on the absorbents of the two columns should be equal
26.Irregular baseline changes during temperature rising	1. Column loss too much	1. Choose a proper column. Column operating temperature should be much low than maximum allowable temperature for solution
	2. Improper operating conditions	2. Choose proper operating conditions
	3. Column contaminated	3. Replace column
	4. Septa caused strange peak during temperature rising	4. Dry septa for 16 hours at 200°C before usage

7 TCD Detector

7.1 GC1120 Thermo Conductivity Detector (TCD) working principle

GC1120 TCD has four symmetrical cavities inside a metal block, each equipped with a heat-sensitive component (GC1120-TCD heat-sensitive components consist of four rhenium tungsten filaments, each has a resistance of about 90Ω under ambient temperature). Of the four cavities, two are reference cells and the others are measuring cells. Please refer to Figure 7-1 for TCD structure. Heat-sensitive components inside the reference cells and the measuring cells form four arms of a Wheatstone bridge. An adjustable DC regulator supplies power to the Wheatstone bridge.

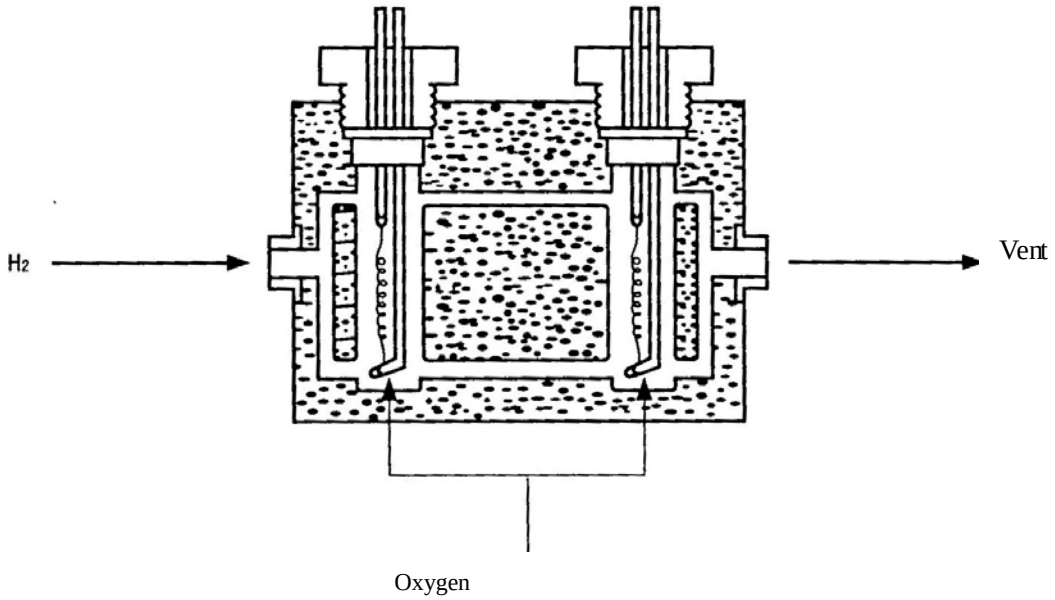


Figure 7-1 TCD structure sketch

Only carrier gas goes through the TCD reference cells. Components that exit the column are carried through the measuring cells by a carrier (normally hydrogen). Since compound conductivity is different from that of the carrier gas, the resistances of the heat-sensitive components in the measuring cells change accordingly. The Wheatstone bridge therefore becomes unbalanced and a voltage proportional to concentration is produced and sent to chromatograph workstation or data processor for signal recording and calculation. Please refer to Figure 7-2 for GC1120-TCD working principle.

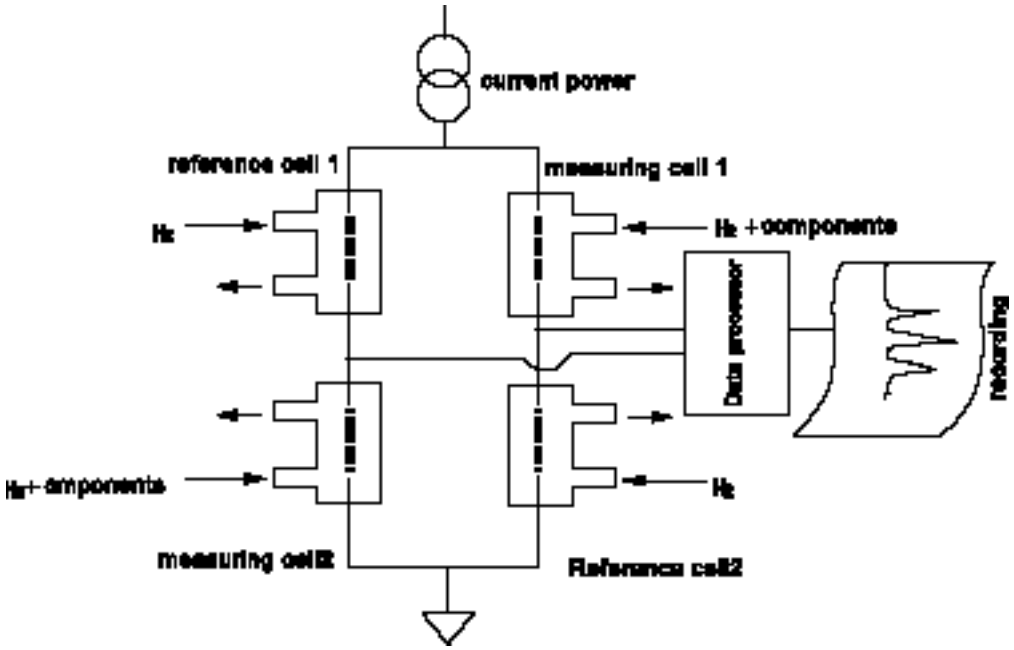


Figure 7-2 GC1120-TCD working principle sketch

7.2 GC1120 TCD specifications

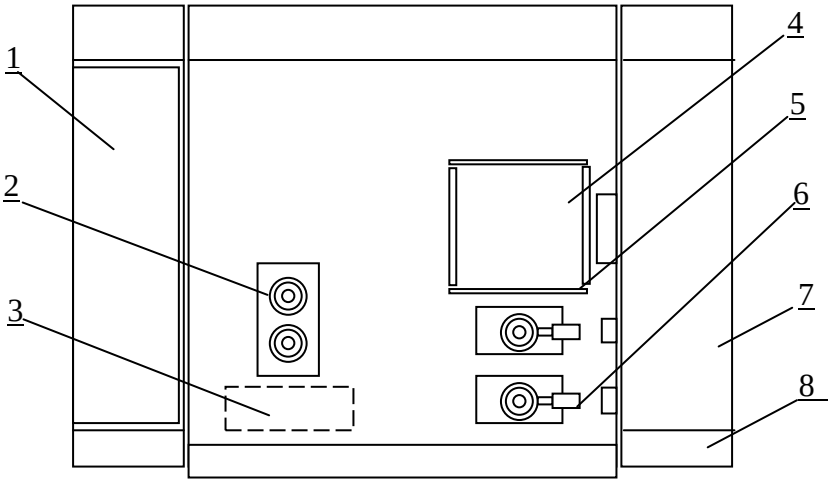
Sensitivity	≥5000mV·ml/mg
Highest sensitivity	≥10000mV·mL/mg（Electronic zoom）
Noise	≤20μV
Drift	≤60μV/30min
Linear dynamic range	$\geqslant 10^4$

Startup stability: the instrument is to be in normal operation three hours after startup.

7.3 GC1120 TCD installation

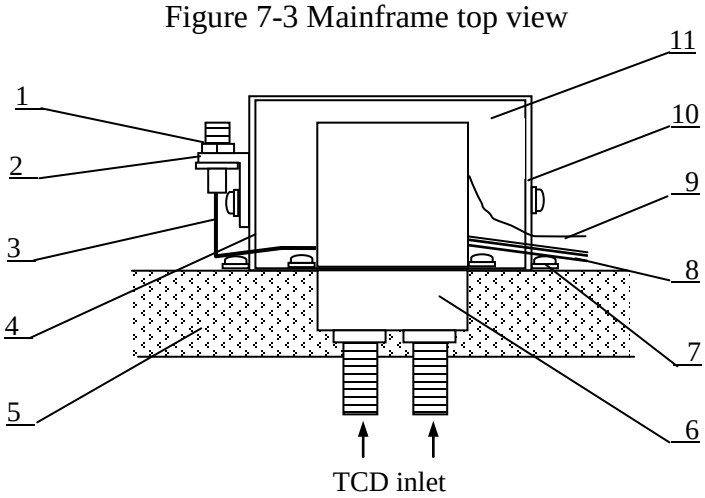
GC1120 TCD installation procedures are as follows:

1) Up swing the cover at the top of the column compartment.
2) Cut the connection strip between the metal board behind the ionization chamber and the installation board by a clamp. Take off the metal board.
3) Place TCD at that position and fasten the four bolts by a screwdriver (Note: the TCD should be oriented in such a way that its cable terminals face the electrical box). Please refer to Figure 7-3 and Figure 7-4.



1. Gas control
2. Injector
3. Column compartment
4. Metal board
5. Connection strip
6. Ionization chamber
7. Electrical box side board
8. Electrical box

Figure 7-3 Mainframe top view



1. M8×1 screw nut
2. Mount
3. Gas discharge tube
4. Angular pieces (left)
5. Glass fiber insulation
6. TCD detector
7. 4× φ 3 screw and spacer
8. TCD current and signal cable
9. TCD heating component cable
10. Angular pieces (right)
11. Insulation cover

Figure 7-4 TCD installation sketch

4) Unscrew the fix bolts at the bottom of the side board of the electrical box. Take

off the side board.

5) Draw the TCD Pt resistance cable, TCD electrical heating component cable, and TCD current and signal cable into the electrical box through a round hole at the back of the electrical box.

6) Connect the TCD Pt cable and TCD electrical heating component cable respectively to the corresponding terminals on the microprocessor main board (please do this according to signs printed on the board: connect the TCD Pt resistance cable to two 10-core sockets, marked as “TCD” on top of the board. Connect the TCD heating component cable to two 8-core sockets, marked as “TCD H” on top of the board. Power of the heating component is 65W, resistance is 740Ω. The two connection cables are red high-temperature proof cables. The Pt resistance has a resistance of 110Ω under ambient temperature. The two cables are silver-plated brass wires sheathed in high-temperature proof glass fiber conduit.).

7) Plug the TCD current and signal cable (5-core, spacing 3.96mm) to the XS3 socket (5-core, spacing 3.96mm) on the back of the current regulator board (please refer to chapter 7.5 for current regulator board installation) at the bottom of the electrical box.

Note

The bottom of TCD should not be in contact with column compartment wall directly. Asbestus or glass fiber should be used as insulation.

Warning

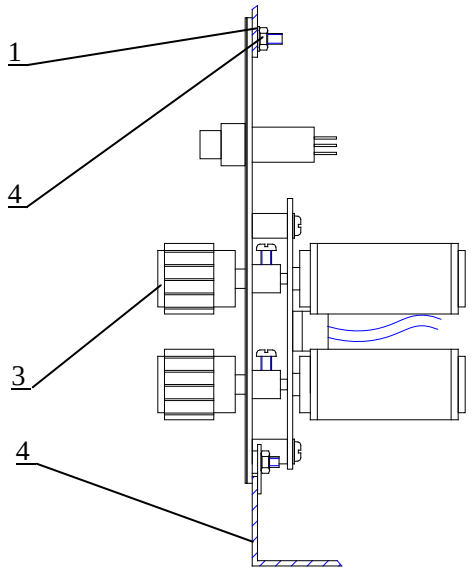
It is prohibited to connect the TCD Pt resistance cable to the sockets of the TCD electrical heating component or vice versa. Otherwise the microprocessor board and the instrument may be damaged.

7.4 GC1120-TCD current regulator installation

GC1120-TCD current regulator consists of a TCD current regulator panel (zero adjusting electrical board) and a TCD current regulator board. The installation procedures are as follows:

1) Open the brown glass plexiglas door under the electrical box. Unscrew the two screws on the blank board. Take off the blank board.

2) Take TCD current regulator panel (zero adjusting electrical board) out of the GC1120-TCD current regulator package. Install the panel on to the position where the existing blank board sits. Fasten the two screws on the board. Please refer to Figure 7-5.



1 . φ 3 spacer
2. φ 3 screw
3 GC1120-TCD current regulator panel (zero adjusting)
4. Electrical box

Figure 7-5 Schematic drawing for parts under the electrical box

3) Take the TCD current regulator electrical board out of the GC1120-TCD current regulator package. Install it horizontally on to the four support columns at the bottom of the electrical box (It is possible that GC1120-FID amplifier circkuit board has been installed under these four support columns. The TCD current regulator electrical board should be installed in such way that one side of the electrical board, which welded with XS2, XS3 and XS4, faces the back of the electrical box). Fasten the board with the φ3mm screws by a screw driver (the four screws have been pre-screwed into the top of the four support columns).

4) Plug the 2-core plug (spacing 2.54mm, for input of current regulator on/off signal), which is connected with the [Current Switch] on the TCD current regulator panel, into the XS5 socket on the TCD current regulator electrical board. Plug the 2-core plug (spacing 3.96mm, for input of ~45V AC), which is connected with a transformer at the bottom of the electrical box, into XS2 socket on the TCD current regulator electrical board. Plug the 3-core plug (spacing 2.54mm, for TCD signal output), which is connected with the "DET2/TCD" socket under the electrical box, into XS7 socket on the TCD current regulator electrical board.

5) Take a cable with a length about 500mm out of the GC1120-TCD current regulator package (the cable has a 5-core plug at both ends, spacing 2.54mm). Plug one end of the cable into the XS8 socket on the zero adjusting electrical board behind the TCD current regulator panel, the other end into the XS4 socket on the TCD current regulator electrical board. Thus the zero adjusting signal and the [Current Switch] indication light signal are connected.

6) Plug the 40-core strip cable, which is connected with the microprocessor electrical board and FID amplifier, into the XS1 socket on the TCD current regulator electrical board (if FID amplifier electrical board has been installed under the electrical box, then the 40-core strip cable should be drawn from the back of the TCD current regulator electrical board).

7) Cover the side board of the electrical box. Fasten the two screws at the bottom of the side board of the electrical box with a screwdriver. Installation of the TCD current regulator completes. Please refer to Figure 7-6.

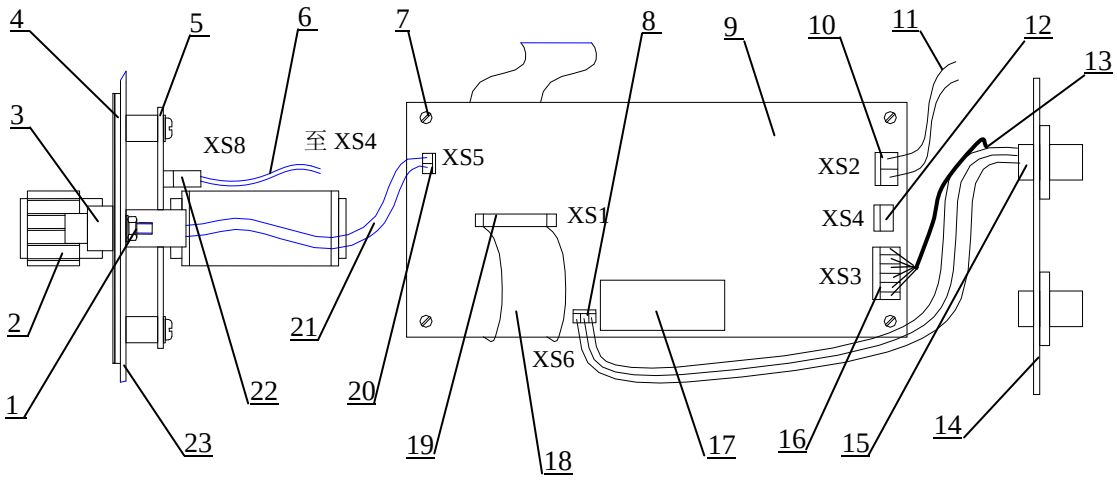


Figure 7-6 GC1120-TCD current regulator installation sketch (part, plan)

1. Panel screw
2. [Zero] knob
3. [Current Switch] button
4. TCD current regulator panel
5. Zero adjusting electrical board
6. Cable with a length about 500mm (the cable has a 5-core plug at both ends, spacing 2.54mm)
7. 3-core socket XS7 (spacing 2.54mm)
8. TCD current regulator electrical board
9. FID amplifier electrical board
10. 2-core socket XS2 (spacing 3.96mm)
11. Support column
12. 2-core plug (spacing 3.96mm, for input of ~45V AC), connected with a transformer
13. TCD current and signal cable and 5-core plug (spacing 3.96mm, for TCD working current and bridge output signal)
14. ϕ 3mm screw for electrical board fixation
15. Bottom of the side of the electrical box
16. 5-core socket XS3 (spacing 3.96mm, connects with TCD current and signal cable)
17. 5-core socket XS4 (spacing 2.54mm, connects with one end of the cable with a length of about 500mm)
18. 3-core socket (marked as “DET2/TCD”, the cable behind connects with a 3-core socket with a spacing of 2.54mm)
19. 40-core strip cable and plug
20. 40-core strip cable socket XS1
21. 2-core socket XS5 (spacing 2.54mm)
22. 5-core socket XS8 (spacing 2.54mm, connects with one end of the cable with a

length of about 500mm)

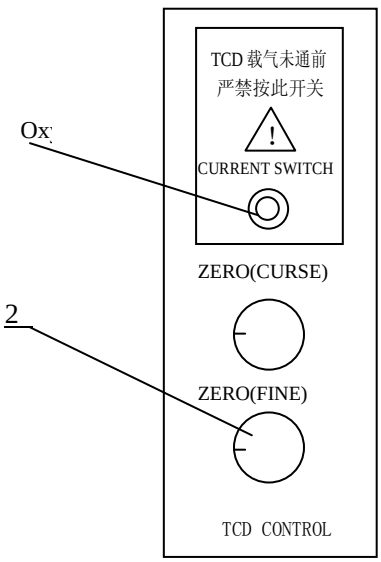
23. [Current Switch] button, cable and 2-core plug (spacing 2.54mm, for current regulator switch signal input)

7.5 GC1120-TCD current regulator panel and setting

Please refer to Figure 7-7 for GC1120-TCD current regulator panel layout. The functions of the switches, knobs and indication light on the panel are as follows:

[Zero]knob————[Zero] knob has “coarse” and “fine” tunes with wide and limited adjusting range respectively. Tuning the [Zero] knob can compensate some degree the unbalance of the TCD bridge so that the chromatograph baseline and the chromatogram recorded by recorder, data processor or chromatograph workstation can be adjusted to an appropriate position.

[Current Switch]knob—When TCD is selected and TCD working current is set on the microprocessor temperature control panel, press [Current Switch] button and the indication light on the left side will turn on. This shows that the TCD working current has gone through the four rhenium tungsten filaments of the TCD. The warning message printed on the right side of the button reads “ Not press this button unless carrier Gas is connected ”. The message reminds you to check if carrier gas is connected to TCD before you press the button. Once the [Current Switch] is pressed, it will be effective until the power supply for the mainframe machine shuts down. However, when the power supply for the mainframe is switched on for the first time, the [Current Switch] is off and so is the indication light.



1. Current switch
2. Zero adjusting knob

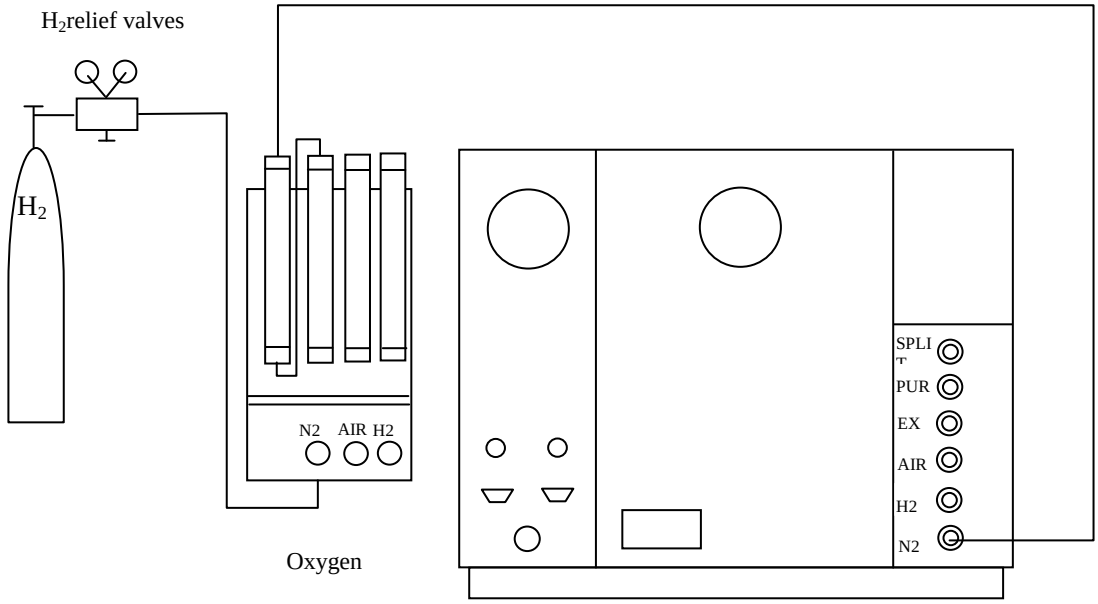
Figure 7-7 GC1120-TCD current regulator panel layout

7.6 GC1120-TCD external gas path connection

Carrier gases appropriate for TCD are hydrogen and helium. Nitrogen and argon normally are not used as carrier for TCD. If nitrogen or argon is to be used, the current regulator should be set at a current less than 80mA. Otherwise, strange peak may occur. Please refer to GC1120-TCD external gas path connection. Please refer to Chapter 4.3 of the GC1120 operating manual for more details. The difference is that TCD only needs one path of purified gas supply (H_2) for the “Carrier” inlet at the bottom of the left side of the mainframe machine.

Warning

The lab should be well ventilated and no fire is allowed when hydrogen is used as TCD carrier.



7-8 GC1120-TCD Connection Diagram of outer Gas Path

7.7 Column installation when GC1120-TCD is selected

Please refer to chapter 4.4 (packed column installation) of the GC1120 operating manual for column installation. Please note that two columns should be connected to two GC1120-TCD inlets. The other ends of the two columns should be connected to the two outlets of the packed column injectors.

Warning

Before the column compartment is heated, two packed columns should be connected, hydrogen valve should be opened and leaking test should be carefully done. Otherwise serious explosion may occur if hydrogen comes into contact with working heating filaments in the column compartment.

7.8 GC1120-TCD thermostatic analysis operation

The operation procedure of the GC1120-TCD thermostatic analysis is as follows:

1) Install TCD and TCD current regulator
2) Connect the external carrier gas (H_2) path and carry out leaking test
3) Install two aged column (from packed column injector to TCD)
4) Connect the power supply cable of the recorder, data processor or chromatograph workstation. Zero adjust the recorder.
5) Connect the signal cable of the recorder, data processor or chromatograph workstation. One end of the signal is to be connected with the recorder or data processor input end, the other end with “DET2/TCD” socket at the bottom of the electrical box of the mainframe machine (same as the connection of FJ-2000 chromatograph workstation).
6) Open the carrier gas supply valve, adjust the low voltage pressure rod until the H_2 pressure gauge reaches an indication between 0.35 MPa and 0.5 MPa. Adjust the two carrier flow valve knobs (please refer to Figure 1-5 in chapter 1.7 of the GC1120 operating manual) on the carrier control panel at the front left side of the mainframe machine. Adjust carrier flow A and B to the volume needed (Please refer to the dial-flow rate curve cards for the number of adjustment circles needed for the dial knob. Please note that H_2 curve cards should be consulted for this case).
7) Turn on the mainframe machine switch. Set temperatures for column compartment, TCD and injector as described in chapter 2 of the GC1120 operating manual and commence temperature control.
8) When the temperature set values are reached, press [DETSELECT] [4] [ENT] in sequence to select TCD as the current working detector.
9) Press [CURRE] [150] [ENT] in sequence to set the TCD working current to 180mA. The value, 150mA, is only an example. An appropriate TCD working current should be selected according to specific chromatograph conditions. Please refer to chapter 2.2.4 of GC1120 operating manual for “Detector parameters”.
10) Press [Current Switch] on the TCD current regulator panel and the indication light on the left turns on.

11) Turn on the switch of the recorder or the data processor. Then turn on the switch of the recorder pen. Set working range of the recorder, e.g. 1mV. (For chromatograph workstation, this step can start after step 6).)
12) Adjust the “Zero” knob of the TCD current regulator so that a chromatograph base line is achieved for recording. Injection can start after the base line is stabilized.
13) Press [POLAR] to change peak direction. Please refer to chapter 2.2.4 for polarity setting.

Note

TCD bridge current should be considered when setting the TCD temperature. The larger the current, the higher the thermoconductivity temperature. Thermoconductivity temperature shoud be set to at least 70℃; bridge current 100mA.
Though TCD can work under a maximum current of 200mA. Increase carrier (H_2) flow rate accordingly when operating under high current. TCD life will be shortened and rhenium tungsten filaments be oxidized due to high current.

Warning

Strict rules should be followed, that is “Turn on carrier gas (H_2) supply first, then start heating and then put through current”, before TCD can start any work. When no carrier gas is supplied for TCD, it is not allowed to set bridge current and to press the [Current Switch] button on the current regulator panel. Otherwise rhenium tungsten filaments will be burnt.

Note

Four rhenium tungsten filaments installed in TCD cavities are consumables that are not warranted for one year. Therefore please be careful and operate according to instruction while using TCD.

7.9 GC1120-TCD programmed temperature operation

GC1120-TCD programmed temperature operation is similar to GC1120-TCD thermostatic operation. The difference is that the column compartment is temperature programmed. Please refer to chapter 2 of the GC1120 operating manual for programmed temperature operation. Please pay attention to the following items for TCD programmed temperature operation:

a) TCD temperature should be 20℃ to 30℃ higher than column temperature to prevent post-column condensation of the high boiling components.

b) Hydrogen should be strictly de-oxidized to prevent TCD strange peak occurring during temperature program. De-oxidation tube may be used de-oxidation. Please refer to Figure 7-9 for de-oxidation tube connection. Pure nitrogen (oxygen concentrate $\leq$ 1ppm) shall also be used as carrier gas.

c) Bridge current normally is set at less than 130mA to prevent TCD baseline drift during temperature program.

d) If serious baseline drift occurs, adjust carrier flow of one of the gas paths slowly so that the baseline can be compensated.

Note

The installation and use of de-oxidation tube should fully follow the procedures stipulated in the operating manual. In-coming air may affect the tube life.

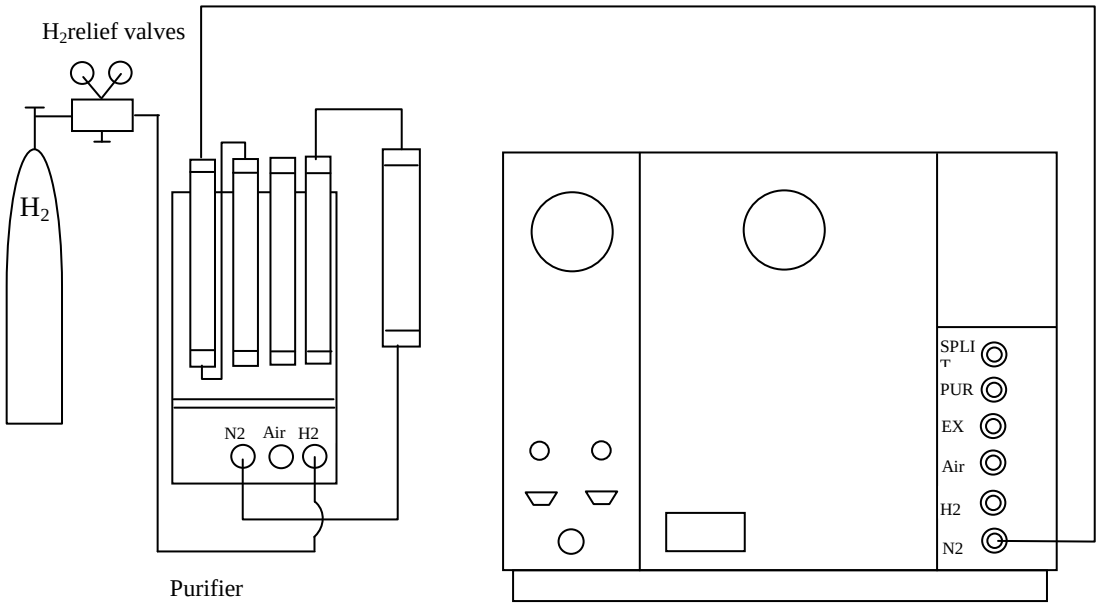


Figure 7-9 De-oxidation tube connection sketch

7.10 GC1120-TCD detector maintenance

Please pay attention to and follow the following items for use of GC1120-TCD:

1) It is prohibited to set TCD working current and press [Current Switch] button while carrier gas is not being supplied so that rhenium tungsten filaments burning can be avoided.
2) Do not connect the post-column carrier gas into TCD while the columns are being aged for the first time. Vent it into the column compartment. For safety purpose, nitrogen instead of hydrogen should be used for column aging. The aged column should be connected and hydrogen should be used. It is not allowed to set TCD working current and press [Current Switch] button during column aging.
3) TCD is a critical chromatograph part. Untrained operator should not dismantle or install rhenium tungsten filaments to avoid unnecessary loss.
4) It is not necessary to dismantle the rhenium tungsten filaments for cleaning if TCD is contaminated by sample. Disconnect the column, fill the TCD with solvent (ethanol, acetone, etc.) from TCD inlet. The solvent will flow from TCD outlet to container. Repeat several times. Blow the solvent out by using a clean gas. Then heat the TCD to a temperature at least 30% percent higher than the solvent boiling temperature. Purge with a clean gas and finally install the column and restore carrier supply to start normal operation again.

Figure 7-1: GC1120-TCD malfunction diagnose and trouble shooting.

Phenomenon	Diagnose	Trouble Shooting
No peak or small peak	1. Current not set 2. Tungsten filaments burnt 3. TCD current regulator poor connection 4. Syringe not gas tight or plugged 5. Injector septa not gas tight 6. Inappropriate carrier gas 7. Current regulator malfunction	1. Set current according to operating manual 2. Replace tungsten filaments 3. Connect TCD properly 4. Replace syringe 5. Replace septa 6. Choose an appropriate carrier gas 7. Contact manufacturer for maintenance
Can not adjust to zero	1. Tungsten filaments resistance not match 2. come in contact with internal wall 3. Tungsten filaments contaminated 4. TCD current regulator poor connection	1. Contact manufacturer for maintenance 2. Contact manufacturer for maintenance 3. Clean the tungsten filaments (please refer to “TCD maintenance”) 4. Connect TCD properly
Base line noise too big	1. Carrier gas impurity (impurity is crucial under high current working condition) 2. TCD contaminated 3. TCD column un-aged or aged incompletely 4. Injector septa not gas tight 5. Leaking at the connection of gas path and column 6. TCD working current too high 7. Current regulator malfunction	1. Purify the carrier gas, e.g. de-oxidation, desiccation and purification, etc. 2. Clean TCD cell and injector 3. Age the column at a temperature 10℃ to 30℃ higher than working temperature 4. Replace septa 5. Detect the leaking point and tighten it 6. Decrease working current or increase attenuation of recorder or data processor 7. Contact manufacturer for maintenance

Attachment 1：Gas Flow Sheet

Unit: ml/min

Knob Circles	Carrier flow A、B （Carrier control valve）		H_2 flow A、B (H_2 needle valve)	Air flow A、B (Air needle valve)	Makeup
	N_2	H_2	H_2	AIR	N_2
1	0	0	0	0	0
1.4	0	0	0	0	0
1.8	0.8	1.6	0	0	0
2	1	2	0	2	0
2.4	2.0	4.0	1.25	7	0
2.8	3.0	6.0	3.0	20	0
3	4.26	8.78	4.0	27.45	1.8
3.4	6.10	13.5	7.5	47.0	3.15
3.8	9.2	20.0	13	70.0	9.0
4	11.21	23.50	16.88	83.0	12.9
4.2	13.0	28.0	21.0	95.0	16.1
4.4	15.0	34.0	26.5	110.0	18.5
4.8	20.0	43.0	37.5	143.0	33.3
5	22.27	48.99	43.90	159.38	37.35
5.2	25.0	56.0	52.0	178.0	43.4
5.4	28.0	63.5	60.0	198.0	49.9
5.6	31.0	71.0	68.0	215.0	57.5
5.8	34.0	80.0	78.0	233.0	65.7
6	37.22	88.47	87.41	252.31	72.7
6.2	41.0	98.0	98.0	274.0	81.1
6.4	44.0	108.0	110.0	293.0	90.0
6.6	48.0	118.0	120.0	315.0	90.2
6.8	52.0	128.0	135.0	338.0	107.1
7	55.57	139.47	145.19	358.15	117.6
7.2	60.0	152.0	157.5	380.0	126.3
7.4	64.0	162.0	172.0	405.0	146.2
7.6	68.0	166.0	185.0	427.0	156.0
7.8	72.0	190.0	200.0	450.0	164.8
8	76.86	202.91	216.86	474.36	178.2
8.2	81.5	218.0	230.0	500.0	184.2
8.4	86.0	232.0	250.0	522.0	197.0
8.6	90.0		265.0	550.0	209.0
8.8	95.0			575.0	215.0
9	100.0			600.0	235.0