YL Gas Chromatography System

YL6500 Gas Chromatograph

USER MANUAL

YL INSTRUMENT CO., LTD.

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Chapter 1. Introduction

1-1. Gas Chromatography

In 1952, James and Martin devised Gas Chromatography(GC). After then, the technology has been developed rapidly and has been used in many fields such as analytical chemistry and biochemistry. This rapid progress has been made by introducing well developed capillary columns in it. These days, we can easily analyze each component in a mixture by using GC-MS which uses Mass Spectrometer(MS) as a detector.

Gas chromatography involves a sample vaporized and injected onto the head of a chromatographic column. The sample is transported through the column by the flow of inert, gaseous mobile phase. The separation is made during the transportation of the sample carried by gaseous mobile phase in the column. The column itself contains a stationary phase. The mobile phase does not interact with the molecules in the sample unlike other chromatography in GC analysis.

The carrier gas - the mobile phase - must be chemically inert. Commonly used gases are helium, argon, and nitrogen. In gas-solid chromatography, the stationary phase is a solid which has wide surface area and ability to absorb solutes for analysis. And the liquid stationary phase in gas-liquid chromatography is chemically or physically coated on the surface of an inert solid which has wide surface area.

In gas chromatography, the partitioning of solutes between the mobile and stationary phases accounts for the separation of solutes. In GC, partition ratio depends on vapor pressure of solutes, chemical characters of each component, and an affinity for the stationary phase. Since these are all effected by temperature, it is very important to choose the stationary phase of a column to get good results by optimizing analysis condition. Also, temperature programming of a column is greatly important due to the same reason.

1-2. Characters of the YL6500 GC

The YL6500 GC is a rectangular box, approximately 605 mm width, 455 mm height, 550 mm depth. The weight is 55 kg. It has a control panel for operation on the right side.
The control panel has a LCD screen for input of the operating conditions and display of the input data, a LED for display of the status of the equipment, and a keypad for operation of the equipment and input of variables. There are a power button below the control panel and a column oven on the left side of it. You can open the oven by pushing a button which is below it.

The inlet is on the left side of the equipment. You can connect it with the auto sampler. The detectors are on the right-upper side. The following four figures are the schematic diagrams of the YL6500 GC. You can install each three inlets and detectors and four valves to handle gas samples. You can also set the volume of gas by using the Advanced Pneumatic Control(APC) system which is connected with the inlets and detectors. The APC is on the right side of back of the equipment .

Fig. 1-1. YL6500 GC

1-2-1. Advanced Pneumatic Control (APC)

The Advanced Pneumatic Control system (APC) is designed to achieve the automatism and accuracy of gas chromatography. It digitalizes the control of the volume of gas, which improves the accuracy of an experiment and reduces the time for analysis.

You can use the APC in every inlets and detectors. Also, you can use various modes such as Split/Splitless, Constant flow/pressure, and Ramped flow/pressure to obtain better results. The APC controls the volume of hydrogen and air to reduce the exhaust of combustible gas – hydrogen - in a detector like a Flame Ionization Detector (FID), which uses gas. This character of the APC improves the safety and allows automatism like auto ignition.

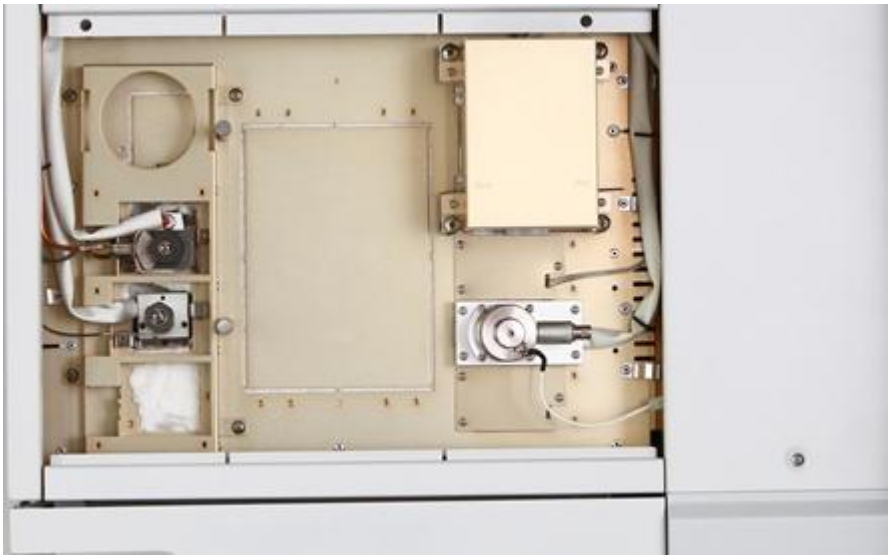


Fig. 1-2.Top of YL6500 GC.

1-2-2. Control of system

The YL6500 GC has a colorful touch screen (LCD) on the control panel which enables you to control temperature of inlets, oven, and column and flow of gas. This graphic color LCD applies an intuitive interface, large screen with the easy touch screen input to allow users to control GC in easy and comfortable.

Also, YL6500 GC provides a function setting the auto operation according to the time. You can store all equipment status, volume of flow, and temperature program as “Method-n”, and the feature of Time Control enables you to save time. Besides these features, you can enjoy a lot of other functions for users. More details will be described from Chapter 3.

LCD: Colorful touch screen to strengthen a user interface with a finger-tip.

Keypad : Keypad for entering values

Start/Stop button

Power button

Figure 1-3 Control panel of the YL6500 GC

1-2-3. Inlet

The inlets of the YL6500 GC is designed for having no loss of sample and reaching the set temperature rapidly. The maximum temperature for a packed column is 450 ℃, and for a capillary column is 400 ℃. You can install various inlets such as Packed Inlet, Capillary Inlet, and On-Column Inlet. The maximum number of inlets to be installed on one GC is three.

1-2-4. Detector

In the YL6500 GC, you can equip various detectors to detect different types of samples. To provide precise results, the YL6500GC is equipped with a detector which has high linearity and reproducibility of signal. Also, it has an accurate function which controls temperature to prevent sample from liquidizing and to improve sensitivity.

1-2-5. Valve for gas sample

For automation of gas sampling, valves can be installed separately for a gas sample injection. These can be equipped up to four additionally with common inlets

Also, multi-positioning vales are available up to two for applications of various gas samples.

1-2-6. Column oven

The YL6500 GC promotes the retention time of sample and the accuracy of experiments by improving the precision and accuracy of temperature of column oven. The temperature of oven is controlled in a unit of 1 ℃ from room temperature up to 450 ℃. If you want to operate the instrument below room temperature, you have to install extra cooling equipment which uses liquid nitrogen or carbon dioxide.

1-3. Specifications of YL6500 GC

System requirement

Electricity: 220 VAC 60Hz, 3.5kW

Gas Chromatograph YL6500 GC Oven System Module

(1) Usable volume : 14L

(2) Automatic cooling under processor control

(3) Temperature operating range : (ambient to 450℃ or more)

-80℃ ~ 450℃ (with LN_2 cryogenic cooling)

-55℃ ~ 450℃ (with LCO_2 cryogenic cooling)

(4) Temperature set-point Resolution: 1℃

(5) Programming: 25 ramps, 26 steps temperature program

(6) Maximum set-point heating rate: 100℃/min

(7) Run time: Maximum 9,999 min

(8) Cool down rate: 6.5 minutes from 450℃ to 50℃

(9) Temperature program method: Maximum up to 20

(10) Temperature stability: ±0.01℃ (Isothermal), ±0.1℃ (Gradient)

(11) Improved column conditioning function : Automatic set of split flow up to 5 ml/min on column conditioning

(12) Prevention of oven malfunction (Over heating)

(13) Heated Devices : 10 heated zones standard

A. 1 heated oven zone

B. 3 heated injector zone

C. 3 heated detector zone

D. 2 heated valve zone

E. 1 heated methanizer zone

Injector (Maximum no. of inlet installation: three)

(1) Packed inlet

1) Maximum Temperature : 450 ^{o}C
2) Pressure setting range : 0.01 ~ 100 psi
3) Total flow setting range : 0.1~100 ml/min
4) Flow stability < ±0.05ml/min
5) Pressure stability < ± 0.05psi
6) Temperature setpoint : 1℃

7) Temperature stability < ±0.1℃

(2) Capillary inlet (Split/Splitless)

1) Maximum Temperature : 400°C
2) Pressure setting range : 0.01 ~ 100 ml/min
3) Total flow setting range : 0.1 ~ 400 ml/min N_2
   0 ~ 1000ml/min He
4) Splitless time setpoint : 0.01min
5) Total Flow stability < ±0.05ml/min
6) Pressure stability < ± 0.05psi
7) Temperature setpoint : 1℃
8) Temperature stability < ±0.1℃

(3) On-column inlet

1) Maximum Temperature : 450 °C
2) Pressure setting range : 0.01 ~ 100 psi
3) Total flow setting range : 0.1 ~ 100 ml/min
4) Flow stability < ±0.05ml/min
5) Pressure stability < ± 0.05psi
6) Temperature setpoint : 1℃
7) Temperature stability < ±0.1℃
8) Temperature programming up to 5 steps

Advanced Pneumatic Control

(1) Up to 6 APC blocks for inlets, detectors or auxillary gases

(2) Increased precision and accuracy in pressure and flow rate

: Increased the number of times sampling for flow and pressure

: Increased the number of times controlling for valves

: Shockproof APCs

: Automatic compensation for temperature & pressure in installation condition

(3) Up to 6 APCs can be installed and up to 18 channels of APC

(4) Flow setpoint : 0.1ml/min

(5) Pressure setpoint : 0.01psi

(6) All gas flows controlled by APC

(7) Available gasses : N_2, He, H_2, Air, Ar/CH_4

(8) Board for use of APC control : APC Main B/D

(9) Constant Flow

(10) Constant Pressure

(11) Programmed Flow : 5 steps

(12) Programmed Pressure : 5 steps

(13) Leak detection

(14) After an alarm for a shortage of gas, it automatically saves the method to method no. 0 and shuts down

(15) Gas saver

Detector

- Maximum no. of detector installation: three
- Data Acquisition Rate : 200 Hz

(1) Flame Ionization Detector

1) 450 ^{o}C maximum operating temperature

2) Automatic flame ignition

3) Temperature set-point : 1 ℃

4) Ignition message: Alarm message as 'Flame out'

5) MDL : 2.0 pg carbon/sec

6) Linear dynamic range : 10^{7}

7) Temperature stability : ± 0.1℃

8) Air : 0~500 ml/min

9) H_2 : 0~100 ml/min

10) Make-up gas : 0~100 ml/min

(2) Thermal Conductivity Detector

1) 400^{o}C maximum operating temperature

2) Flow through cell : 4 Rhenium-Tungsten filaments

3) MDL : 2.5 ng/ml (Standard), 400 pg/ml (uTCD)

4) Filament protection

5) Temperature set-point : 1 ℃

6) Micro-cell (Option)

7) Ref : 0~100 ml/min

8) Make-up : 0~100 ml/min

(3) Nitrogen-Phosphorous Detector

1) 400^{o}C maximum operating temperature

2) MDL : <0.4 pg N/sec (Azobenzene)

<0.2 pg P/sec (Malathion)

3) Dynamic Range for N : > 10^4

4) Dynamic Range for P : > 10^4

(4) Flame Photometric Detector

1) 300^{o}C maximum operating temperature

2) Minimum detection limit for S : < 20 pg S/sec

3) Minimum detection limit for P : < 0.5 pg P/sec

4) Dynamic Range for S : > 10^3

5) Dynamic Range for P : > 10^4

6) S/C Selectivity : 10^5

7) P/C Selectivity : 10^6

(5) Pulsed Discharge Detector

1) 400^{o}C maximum operating temperature

2) PDHID, PDECD mode is selectable

3) APC not supported

4) Helium ionization mode(PDHID) : Linearity – 10^5

MDL (organic compound : low ppb)

(permanent gas : low ppm)

Electron capture mode(PDECD) : Linearity – 10^5

MDL – 10^{-15}

Dopant gas : 3% Xe in Helium

(6) Electron Capture Detector

1) Maximum Operating Temperature : 400 ℃

2) Minimum Detection Limit for S : 10 fg/sec

3) Linear Dynamic Rage : > 10^4

Chapter 2. Installation

2-1. Environment for installation

You have to check the environment for installation before opening the box of the YL6500 GC.

2-1-1. Place for installation

The YL6500 GC is approximately 605 mm width, 550 mm depth, and weighs 55 kg. Although you can usually use ordinary experimental desk, you had better check whether it support the YL6500 GC or not.
To operate the YL6500 GC normally, it is recommended to maintain the indoor temperature between 15℃ and 25 ℃, and humidity between 50% and 60%.

2-1-2. Oven outlet

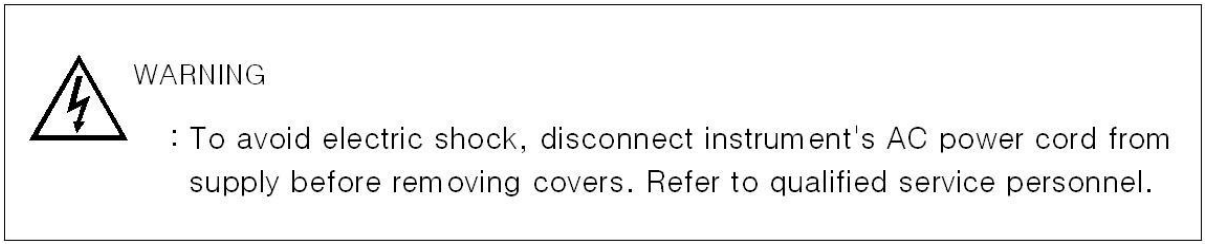
The column oven of the YL6500 GC draws off warm air from the back side of the equipment. Therefore, you should install the equipment away from a wall at least 30 cm, and keep something which can be harmed by warm air away from the back side.

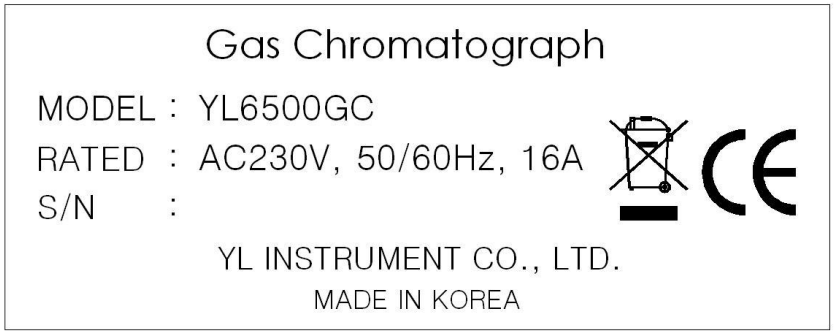
2-1-3. Safety sign

There are following signs about safety on the surface of the equipment. Please, take the advice of messages when you operate or repair the equipment.

When you open the cover of the equipment, it is safe not to turn it on.

You have to keep something which must not be heated such as wires, gas lines, and chemicals away from the back side, because the oven exhausts hot air from the back side. Also, you must not touch the hot inlets and detectors.





2-2. Check

Firstly, check the condition of the box of equipment and then inspect the status of it. If you find any flaw, feel free to contact us.

Secondly, check the Packing List attached on the box whether there are any missing items or not. If you find out any missing, do not hesitate to contact us.

2-3. Power

2-3-1. Power voltage

The YL6500 GC is adjusted to be used in 220 VAC (5%), 60 Hz, and the consuming voltage is 3.5 KW. It is recommended to install the Auto Voltage Regulator (AVR) – capacity 5.0 KW - in a place where the change of voltage is severe.

2-3-2. Power cable

You can provide the equipment with power and ground because the offered power cable contains the ground line and plug also has ground.

2-3-3. Ground

For safety, the IEC (the International Electro technical Commission) recommends to ground the equipment. Therefore, you should check the connection of ground before installing the equipment. If there is no ground line in a power socket, you have to connect the extra ground line to the ground part of a plug.

2-3-4. Check the power

1) Do not turn on the euipment, if it is not on the normal place.
2) Check the power switch located on the right-down side of the equipment whether it is “OFF” or not.
3) Turn on the quipment.
4) When you turn on the equipment, you can check the status of the system controller on the LCD because the YL6500 GC operates self-check function.
5) Watch for the screen whether there is an error message or not.
6) If there is no error message when turning on the equipment, turn it off again and then continue to install it.

2-4. Used gas

2-4-1 Selection of gas

1. Carrier gas

When selecting a TCD, you use helium as carrier gas and nitrogen or argon in the case of analysis of hydrogen. Also, when you select a FID, helium, nitrogen, or hydrogen is possible for carrier gas. But hydrogen is rarely used because explosion may occur. Therefore, when using both a TCD and FID, you have to obtain helium and nitrogen. Besides, other detectors (ECD, NPD, FPD) usually use nitrogen and helium, too. But you have to use optimal carrier gas when taking a special detector like PDD.

2. Purity of gas

Although there are some exceptions according to usage or detectors as the following table 2-1, you have to use pure carrier gas normally above 5N(99.9995%)

Table 2-1. Tips for carrier gas

Detector	Gas	Usage	Purity	Remark
TCD	Helium	Carrier gas	99.995%	Purchasing it
	N_2 or Ar	Carrier gas	99.995%	When detecting H_2
FID	Nitrogen	Carrier gas	99.995%	Purchasing it
	Helium	Carrier gas	99.995%	Purchasing it with nitrogen selectively
	Hydrogen	Support gas	99.995%	Purchasing it
	Air(dry)	Support gas	Best Grade	Purchasing it
ECD	Nitrogen	Carrier gas	99.9995%	Purchasing it
	Ar/CH_4	Carrier gas	Best Grade	Purchasing it with nitrogen selectively
NPD	Helium	Carrier gas	99.9995%	Purchasing it
	Nitrogen	Carrier gas	99.995%	Purchasing it with helium selectively
	Hydrogen	Support gas	99.995%	Purchasing it
	Air(dry)	Support gas	Best Grade	Purchasing it
PDD	He	Carrier gas	99.995%	Purchasing it
	3% Xe/He	Dopant	Best Grade	Purchasing it

3. Support gas for a detector

1) When selecting FID, NPD, or FPD detector, you have to use air and hydrogen in addition to support gas.
2) Use air in a cylinder. If you have to use a compressor, be sure to install an oil filter and a filter for excluding water and to connect an exit Fitting to 1/8 inch tube.
3) If using a hydrogen generator, you can lower not only the price but also the danger. For this, you must use ultra-pure hydrogen and refresh a desiccator periodically.

2-4-2. Connection with a gas tube

You have to use 1/8 inch metal tubes in outer diameter (O.D.) for all gases. If you use a plastic tube, oxygen in air can infiltrate it and other impurities like plasticizer can harm a column and a detector. Also, you must not use old tubes contaminated by oils because they pollute gas tubes connected to the equipment.

1. Accessories for connection

1) Regulator

Use regulator suitable for the types of gas and shape or size of cylinder. If possible, use it having CGA(Compressed Gas Association) numbers.

2) Moisture Trap

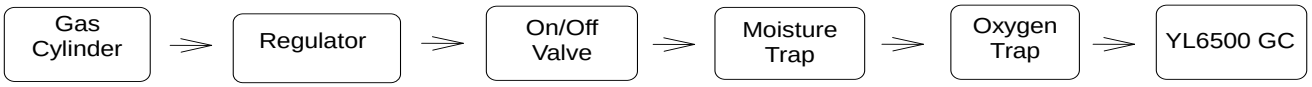
A moisture trap filled up with zeolite 5A is installed between carrier gas and the equipment. By allowing nitrogen or helium containing water to flow through it or eliminating water by heating in vacuum, you can activate it. If you use detectors with high sensitivity like ECD, it is recommended to activate it just before using.

3) Oxygen Trap

Even a little oxygen in carrier gas can harm a column, especially a capillary column with very little stationary phase and lower the performance of a EDC detector. Therefore, if selecting an ECD, be sure to use an oxygen trap. This trap is installed next to a moisture trap.

2. Connection with a gas tube

Connect the equipment with gas tubes as followings.



On/Off Valve : The On/Off Valve is not essential, but useful. You must install it to the outlet of a regulator.

Moisture trap : You need a moisture trap for carrier gas, and must install it with every detectors. Install it to the outlet of a regulator or a shut off valve.

Oxygen Trap : You need an oxygen trap for carrier gas. When using an ECD detector or a capillary column, you must install it. Generally, it is essential for a packed column, too. Install it to the outlet of a moisture trap.

Connect the tube for carrier gas to the back-right side of the YL6500 equipment, “CARRIER”. When selecting FID as a detector, connect each tube for air or hydrogen to “AIR” or “HYDROGEN”. If using other valves for gas sample, connect the tube for compressed air or nitrogen to “VALVE GAS”.

3. Check for leakage

1) Connect all gas tubes. (Do not open the cylinder valve yet.)
2) Adjust the pressure controlling valve in order to make the second-order pressure gauge of the regulator to zero.
3) Open the cylinder valve. If there is a opening and closing valve, open it, too.
4) Adjust the pressure controlling valve in order to make the second-order pressure gauge of the regulator to about 50-70 psi.
5) Check gas leakage from the connection part of the regulator and the cylinder to the backside of the YL6500 GC by using the bubbles.
6) When finding some leakage, tighten the connection part and check the leakage again.
7) See whether the pointer of the pressure gauge goes down or not when closing the outlet of GC and the cylinder valve. If the pointer goes down at once, there is some leakage. Therefore, you must check the leakage again.
8) If you can not find any leakage, continue to install the equipment.

Chapter 3. Screen

3-1. Structure of Control Panel

The control panel of YL 6500GC is LCD(Liquid Crystal Display) and this offers an intuitive interface with a wide touch screen so that operators can control easily and comfortably. The user interface of YL6500 GC is more enhanced than previous YL`s GC. The screen configuration is divided into three parts, and users are able to enter lots of parameters at the same time, therefore, it leads to shortening the screen and button settings. The change of settings can be made with tapping on screen and a key pad.

3-1-1. Home screen

The wide LCD screen conveys a lot of information at the same time. There are three main icons on Home screen, which are [System], [GC], and [Display]. [System] icon is to set the system. [GC] icon is to set parameters. [Display] icon is to show current status of GC.

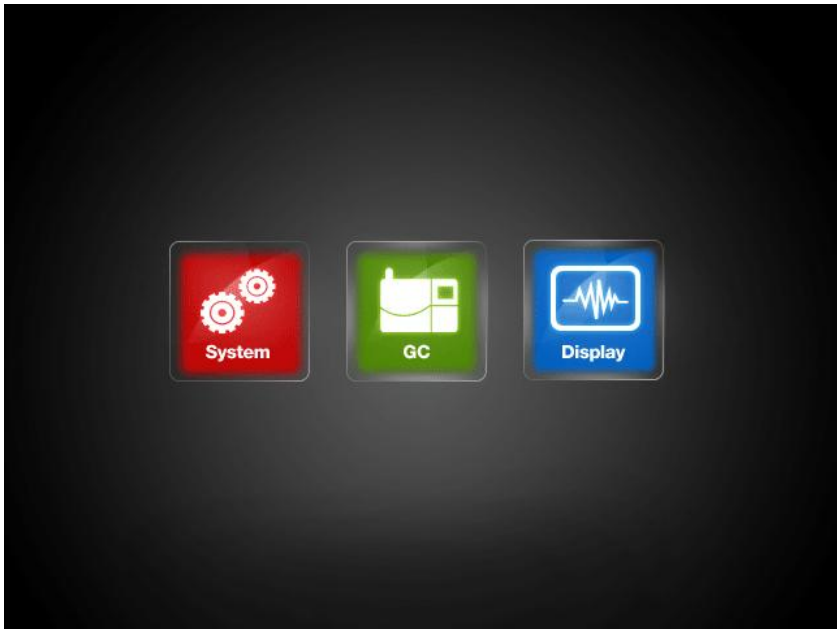


Fig 3-1. Home screen

- **SYSTEM Screen**

[System] screen is to check the basic information and environment setting of instrument, run the self-diagnosis function, calibrate the temperature and flow rate, and control the other special functions. System screen is configured with seven tab screens, and each tab screen can be displayed by tapping one of tabs on the top.

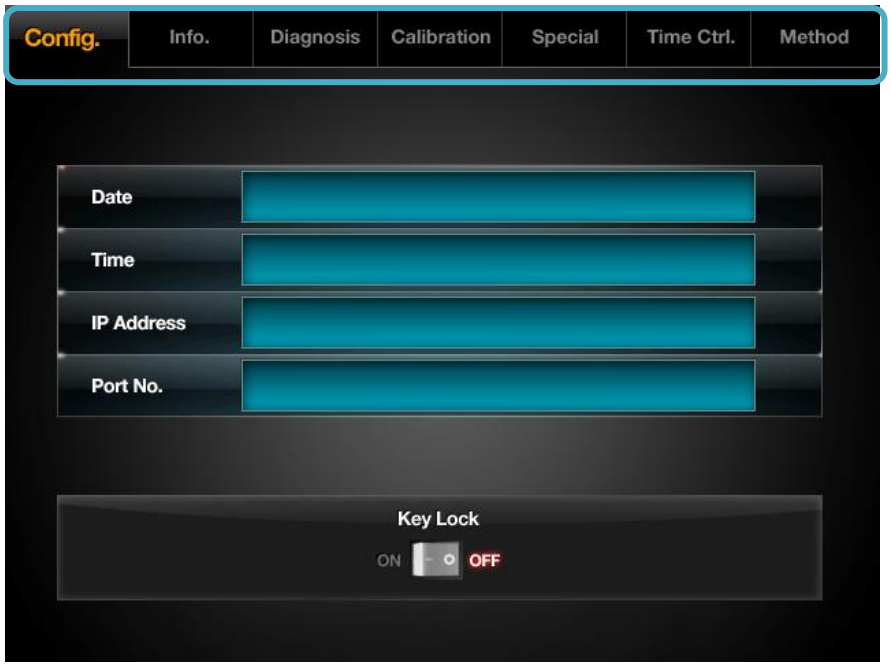


Fig. 3-2 System screen

- **GC Screen**

[GC] screen is to set parameters. Each item screen is moved by tapping the top tabs, and parameters are entered by the key pad. Oven, Injector, Detector, and Signal tabs are activated with the default and Valve and Methanizer tabs are activated depending on whether these are installed or not.

Fig 3-3. GC Control panel screen

- **DISPLAY Screen**

[Display] screen is to indicate the current status of GC. The current status is displayed at the upper left-hand and right-hand. It displays [READY], [NOT READY], or [RUN] at the upper left-hand side depending on the current status. When a special function is operated, it displays [POST RUN], [READY] [RUN], [GAS SAVER], or [COLUMN COND] at the upper right-hand side. While an analysis is running, it displays an oven temperature, a step for oven program, run time, and run time in progress. In the case of a repeat analysis, it shows the number of total analysis and current analysis.

YL6500 GC can be installed up to three inlets and three detectors, and each is displayed as [FRONT], [CENTER], [REAR] depending on the its position. You can see temperatures, flow rates, and signals for all inlets and Detectors, and if tapping on one of inlets or detectors tab, it displays the temperature, flow rate, and signal in order. Not installed inlets and detectors are deactivated.

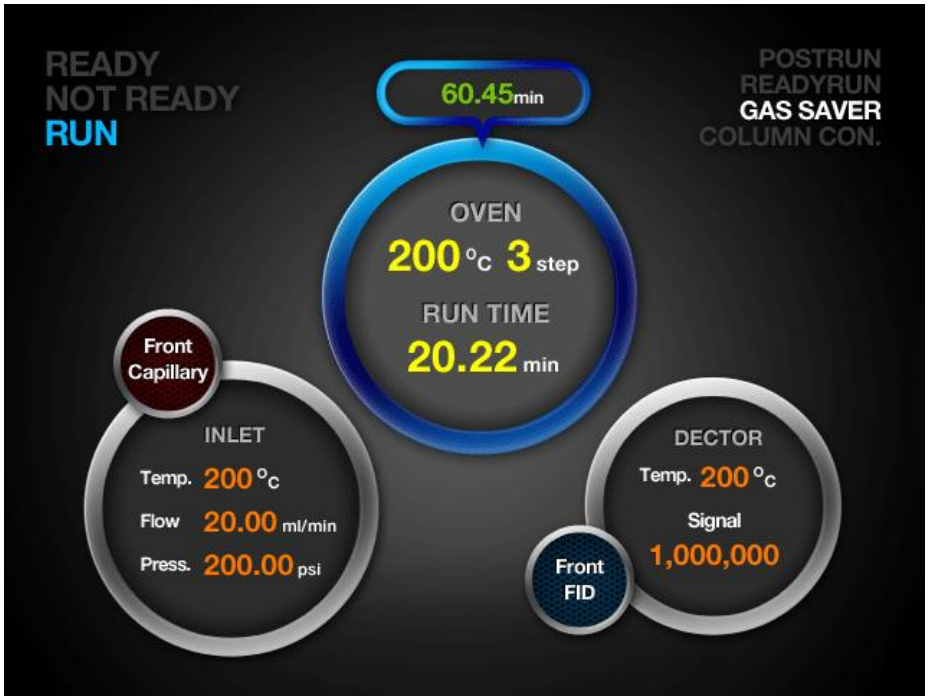


Fig 3-4. Display screen

*** Status message**

Message	Description
Ready	Once all parameters reach to settings then the equilibration time elapses.
Not Ready	Either an entered temperature or flow rate does not reach to set point.
Run	During an analysis
Post Run	When cleaning the column after finishing an oven program
Ready Run	When operated by isothermal run after starting an analysis
Gas Saver	Reducing the flow through a split vent during an analysis

3-1-2. Operation Key

YL6500 GC has a key pad for entering settings and each function. There are twenty three keys on the key pad and it is configured with numeric keys and menu keys which are located LCD screen below.

- **HOME**

This key is used to move the initial screen consists of three icons [System], [GC] and [Display].

- **Ready Run**

This key is used to be back to the initial parameter of split flow after an analysis is done.

If [Gas saver] mode is operated to capillary inlet, parameters return to initial status, but split flow retains as saved in [Gas saver] mode in order to save the gas continuously. At this time, if pressing [Ready Run] key, split flow is set to the initial value so that a split injection is performed normally.

- **ON / OFF**

Makes each function activated or deactivated. It is used for turning on or off the each function.

- **START**

This key is used to start the process of GC analysis. When injecting a sample manually, press the [Start] key after an injection. If Autosampler is installed with GC, no need to press the [Start] key because Autosampler sends a start signal automatically to GC.

- **STOP**

This key is to used to stop the process of GC analysis. When pressing [Stop] key during an analysis, the analysis stops then all parameters return to the initial state.

- **CANCEL**

This key is used to delete all settings you entered.

- **ENTER**

This key is used to enter the settings. It is not activated if pressing the [ENTER] key only. You need to select [ON] key in order to its activation.

- **POWER**

This [Power] key is used for turning on or off the LCD screen, not a power supply switch.

3-1-3. LED Status

YL6500 GC displays the current status of instrument by using three LEDs on the top of control panel. [POWER] LED light indicates whether power is supplied or not, [READY] LED lights up when actual value reaches to set point, and [RUN] LED light is on during the running.

- **POWER**

[POWER] LED is turned on if the power is supplied to the instrument. If not, [POWER] LED is off.

- **READY**

Once all of the conditions reach to set point then the equilibration time elapses, [READY] LED is on.

- **RUN**

When pressing [Start]key , [RUN] LED is turned on, and an analysis is running.

3-1-4. Screen settings and parameters entering

- **Screen setting**

When you enter parameters for items installed on your GC, select each item and enter desired value on LCD screen by using a touch pen supplied with GC or touching softly on it with your finger.

- **Operating function**

To operate a desired function, touch on a desired item`s tab then select [ON]. To stop the operation, select [OFF].

- **Parameter entering**

To enter parameters such as temperature or flow rate, touch on a screen you want to enter. Input parameters in where cursor is on, using numeric keys and then press [Enter] key.

3-2. SYSTEM Screen

The following is a schematic diagram for System display. Configuring each function of GC and operating special function are performed on this screen.

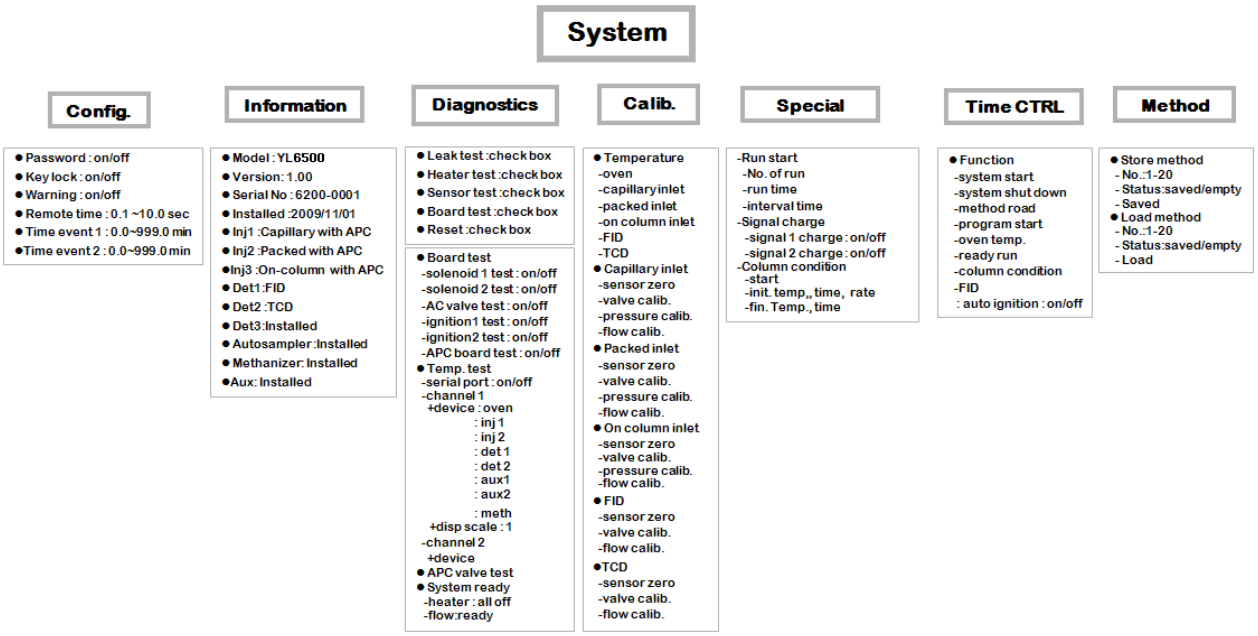


Fig.3-5 Schematic diagram of System display

3-2-1. Configuration

Enter the environment settings of GC system, for instance, the current date, time. IP Address, Port No. and Password, Key Lock, Key beep, and Autosampler settings are set on this configuration screen.

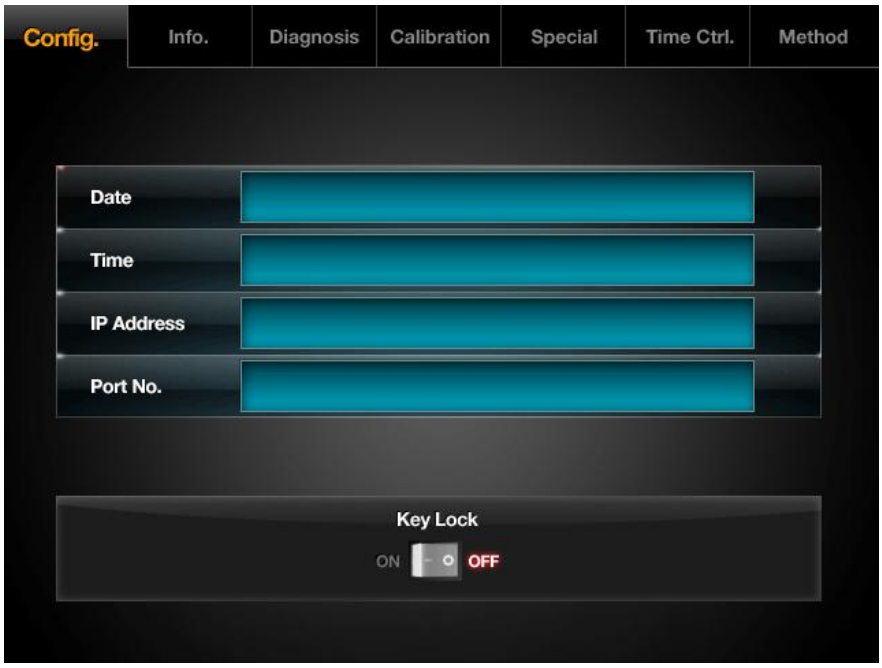


Fig. 3-5. Configuration screen of YL6500 GC

- **Date**

Enter the current date depending on your local date. The initial date is based on the delivery date from the factory. It can be altered with a eight-digit. The format is YYYY/MM/DD.(Year/Month/Day)

- **Time**

Enter the current time depending on your local time. The initial time is based on the delivery date from the factory. It can be altered with a six-digit. The format is HOUR/MM/SS.(Hour/Minute/Second)

- **Port No.**

Enter a Port No. The initial value is based on the delivery at factory.

- **Key Lock**

[Key Lock] makes the Key function locked. If selecting [ON], keys do not work. Initial setting is [OFF].

3-2-2. Information

[Information] screen displays the basic information of your GC system such as model name, firmware version, and what items are installed. Any parameter can not be entered on this screen.
If Valve is installed on GC, [NEXT] key is activated at the bottom and you can move to [Valve] screen with this key to check the status of Valve.

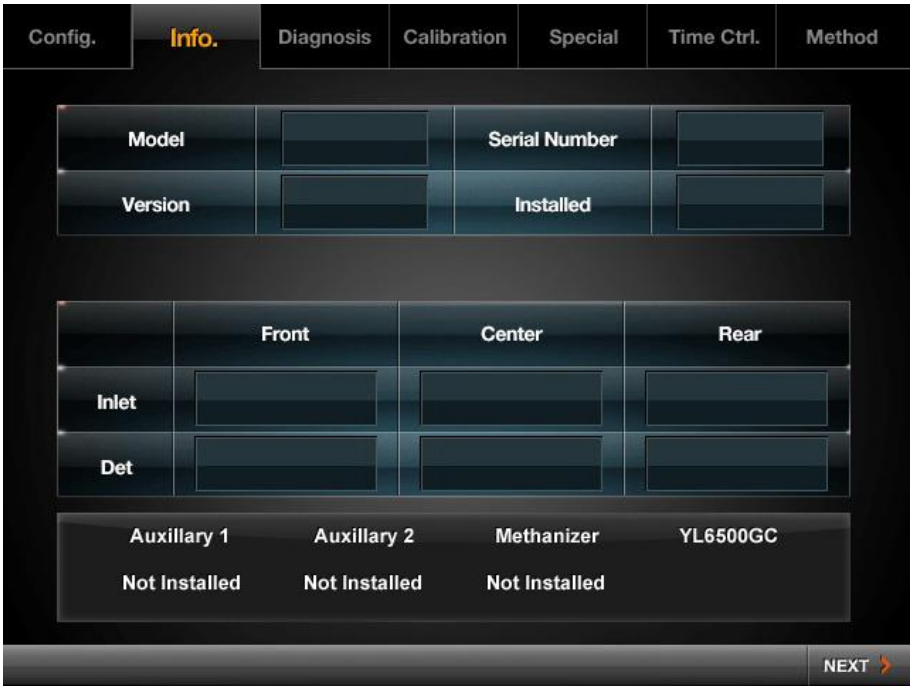


Fig. 3-6. Information screen of YL6500 GC

- **Model**

It is to indicate the Model name of GC

- **Version**

It is to indicate a F/W Version which is the control type used in producing an equipment. If ROM version is upgraded or downgraded, displayed version is changed automatically.

- **Serial No**

It is to indicate a serial number which is unique for each instrument. Users can not change arbitrarily this settings.

- **Installed**

It is to indicate the date when an instrument is delivered from the factory. Users can not change arbitrarily

this settings.

- **Inlet (Front, Center, Rear)**

It is to indicate inlet types installed in an instrument depending on their position. Total three inlets are able to be installed.

- **Detector(Front, Center, Rear)**

It is to indicate detector types installed in an instrument depending on their position. Total three detectors are able to be installed.

- **Auxillary**

It is used to check the temperature of Auxillary items. If Auxillary items (valve, methanizer) are not installed, it indicates as [Not Installed].

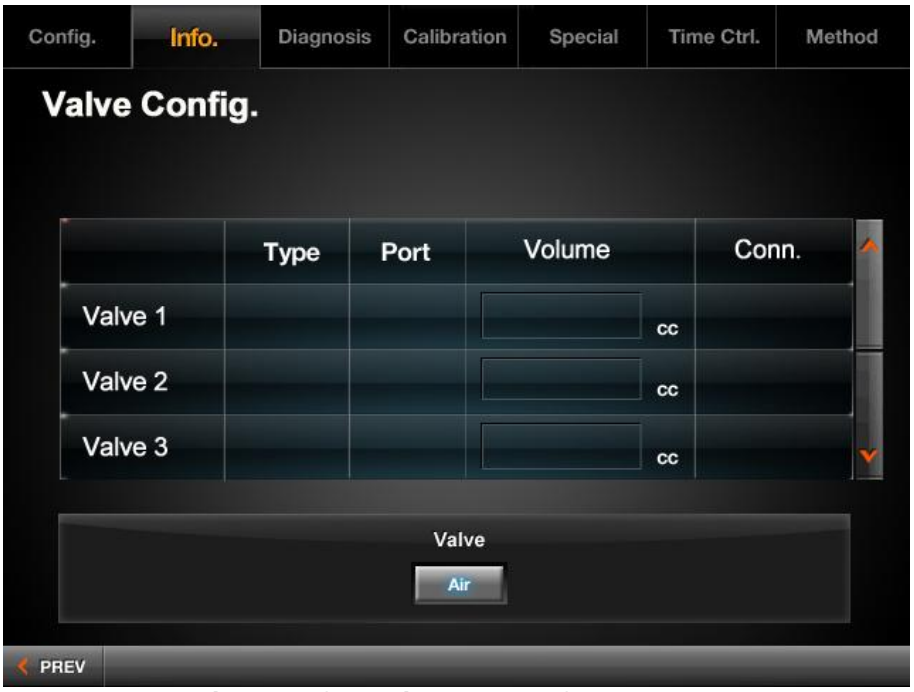


Fig 3-7. Information screen of YL6500 GC

- **Valve**

It is used to check the type of valve, the number of port, and loop volume. If not installed, tabs are deactivated.

3-2-3. Diagnosis

YL6500 GC has a self-diagnosis function. This self-diagnosis function is very useful to inspect the status of instrument. Various functions are available on [Diagnosis] screen and these functions are to check oven, APC, inlet and detector, and output for solenoid valves movement on Main board. Note that, this procedure could affect serious problems to the system if an unqualified person performs.

Fig 3-8. Diagnosis screen of YL6500 GC

- **APC**

APC self-diagnosis checks the function of APC controlling flow rate of inlet and detector. You may check the function of Voltage, Leak, APC Sensor and APC valve on [APC] screen.

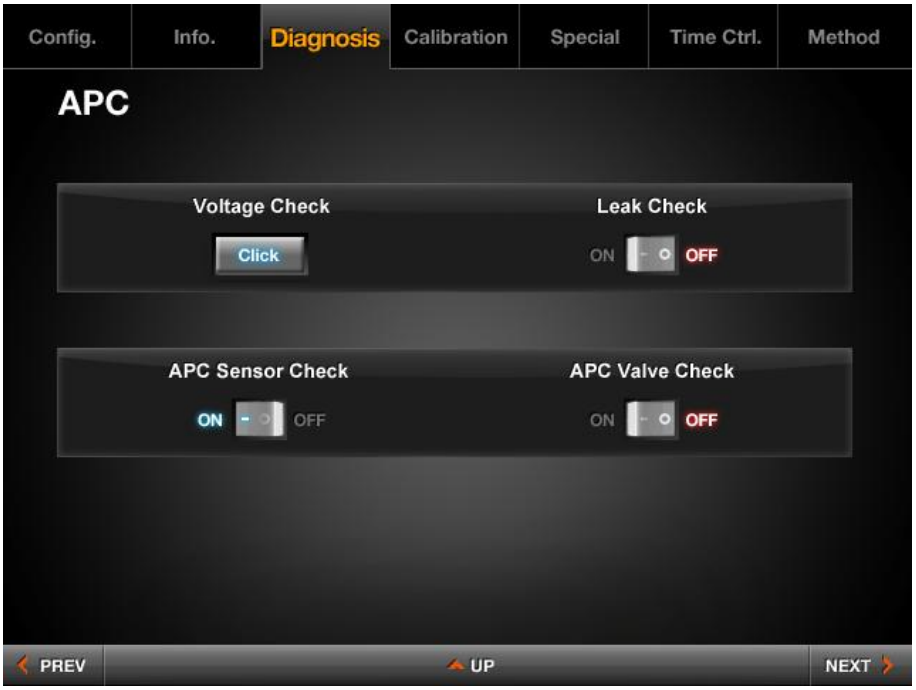


Fig 3-9. Diagnosis screen of YL6500 GC (APC)

- **Inlet**

Inlet self-diagnosis checks the flow rate and temperature of installed inlets.

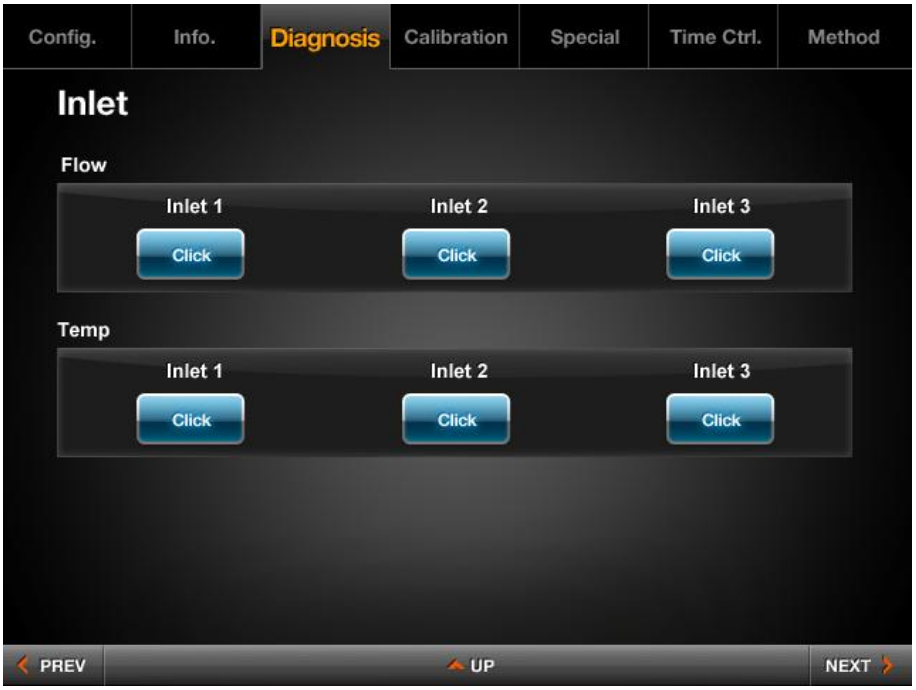


Fig 3-10. Diagnosis screen of YL6500 GC (Inlet)

- **Oven**

Oven self-diagnosis checks the function of oven. You may check the function of Voltage, Valve, Position Valve, DA, Zero Crossing, and Temp. on [Oven] screen.

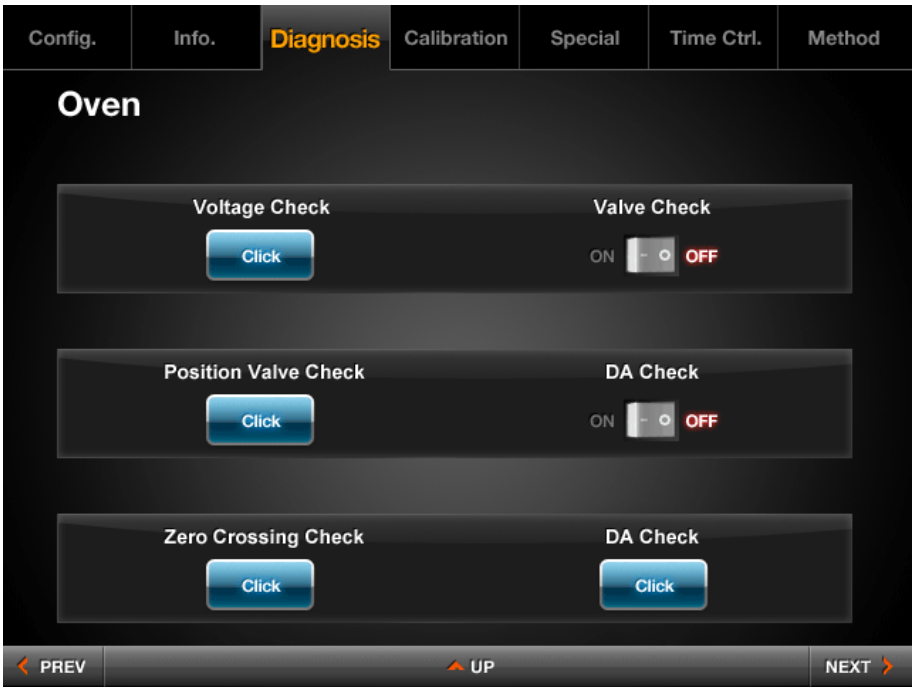


Fig 3-11. Diagnosis screen of YL6500 GC (Oven)

- **Detector**

Detector self-diagnosis checks the function of installed detectors. Checking option is different depending on the type of detectors. You may check the function of ADC, Power, and Ignition ECD Reference Current on [Detector] screen.

3-2-4. Calibration

This screen is to calibrate the temperature and flow rate for oven, injector, and detector. Displayed icon is different depending on your GC`s configuration. You can move each item by tapping one of items you want to calibrate.

<Caution>
This procedure should be performed very accurately. Therefore, the instrument encounters serious problems if an unqualified person performs. This procedure can be performed by only trained and qualified service engineers.

- **Oven**

The function of [Oven Calibration] is to calibrate the temperature. You can move each calibration option with arrow keys at the bottom and touching tab buttons on the top. After completing the calibration, [END] message is displayed. In case that an error occurs during calibration procedure, [Fail] message is displayed and you need to perform again from the beginning. The current temperature of oven shows on [Act] tab. To calibrate the temperature of oven, set the oven to a temperature of 100℃, then wait until the temperature is stable. After stabilization, measure the actual temperature of oven using an approved thermometer. Input settings for temperature on [Set] tab, then input measured temperature on [Measured] tab. After setting these parameters, press [ON] key to perform the temperature calibration.

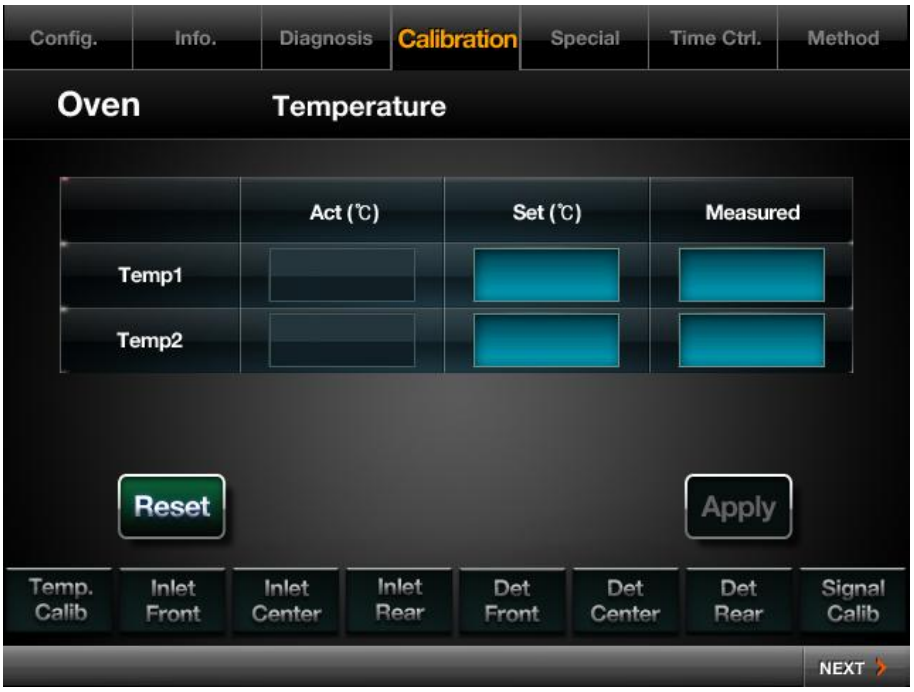


Fig 3-12. Calibration screen(Oven) of YL6500 GC

- **Inlet**

Calibrates the temperature, proportional valves and flow rate for inlet on [Inlet] tab. [Pressure Calib.] function is added to Capillary inlet.

✓ Temp

Calibrates the temperature of inlet. The current inlet temperature displays on [Act] tab. To calibrate the temperature of inlet, set the inlet to a temperature of 100℃, then wait until the temperature is stable. After stabilization, measure the actual temperature of inlet using an approved thermometer. Input settings for temperature on [Set] tab, then input measured temperature on [Measured] tab. After setting these parameters, press [ON] key to perform the temperature calibration.

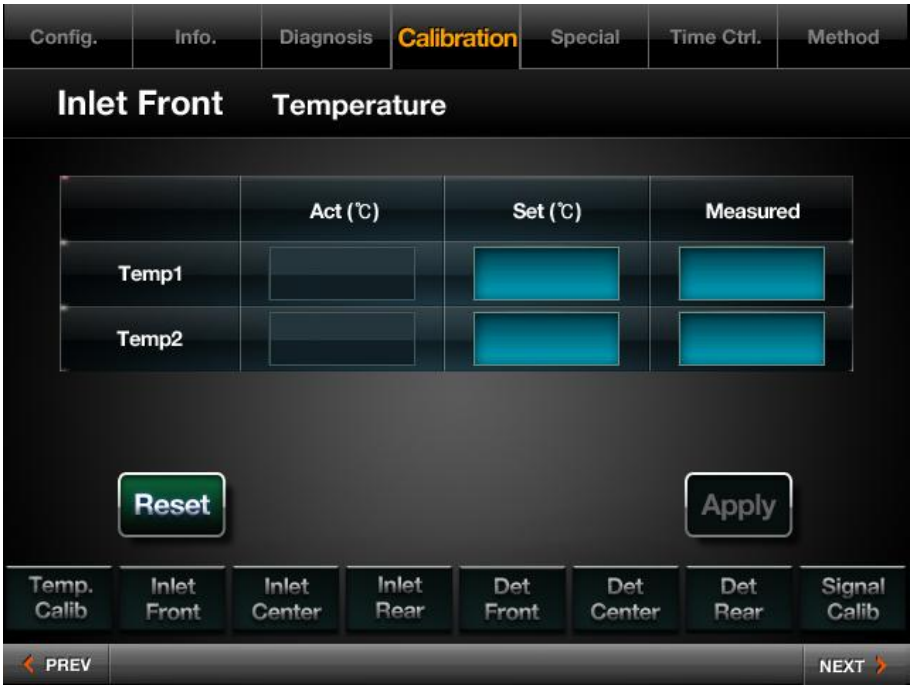


Fig 3-13. Calibration screen of YL6500 GC (Inlet temperature)

✓ Sensor Zero

The function of [Sensor Zero] is to perform zero adjustment for Flow sensor. This procedure should be performed under condition of atmospheric pressure, no gas flow, and room temperature.

Fig 3-14. Calibration screen of YL6500 GC (Sensor zero)

✓ Valve Calibration

The function of [Valve Calibration] is to calibrate valves installed on APC block. This procedure should be performed under condition of gas flow and room temperature. To perform this procedure, press [ON] key on the key pad. During this procedure, each voltage of Valve 1, Valve 2, and Valve 3 is displayed on the screen and calibrated automatically.

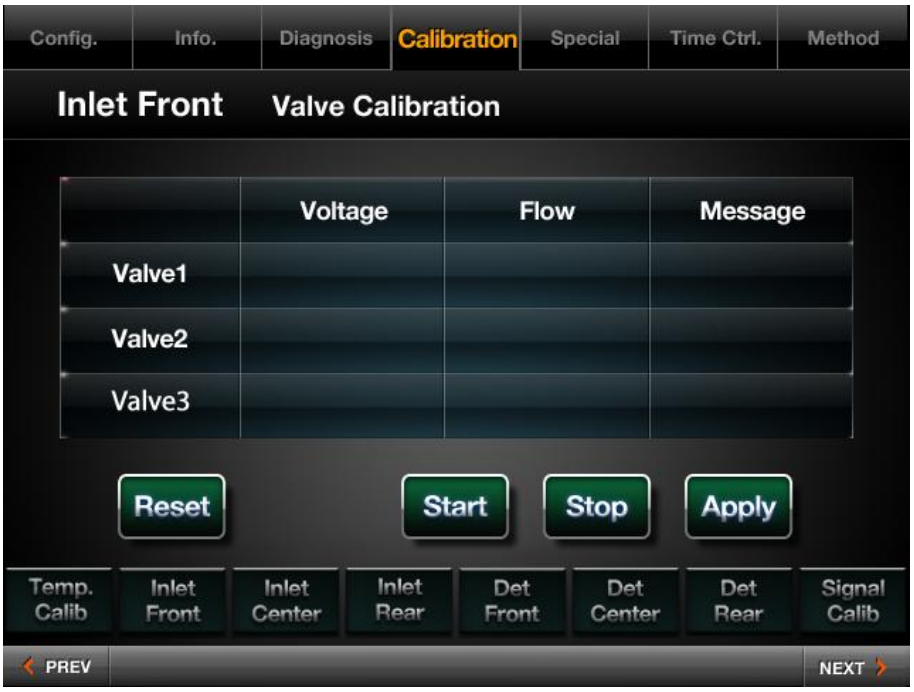


Fig 3-15. Calibration screen of YL6500 GC (Valve calibration)

✓ Flow Calibration

Flow calibration should be performed by an approved flow meter and you have to keep this flow in good condition carefully. Capillary inlet calibrates the flow rate of Purge and Split. Packed and On-column inlets calibrate only flow rate of Column. Actual flow rate is displayed on [Act], and input flow rate of Split and Purge then wait until flow rate of Purge and Split reaches to set point.

When flow rate is stable, measure actual flow rate using an approved flow meter. Input a parameter for flow rate on [Set] tab, then input measured flow rate on [Measured] tab. After setting these parameters, press [ON] key to perform flow calibration.

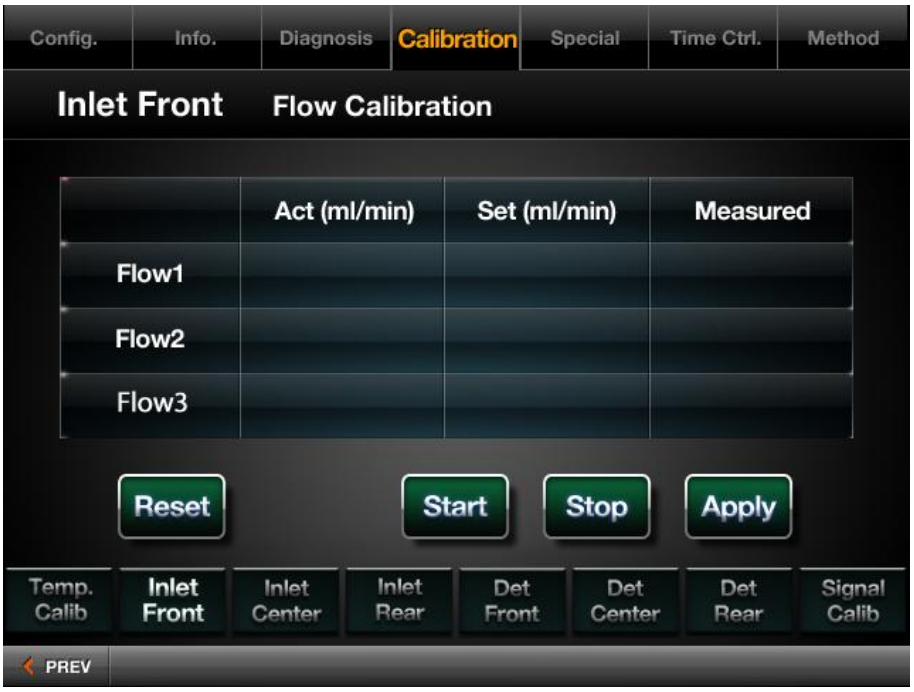


Fig 3-16. Calibration screen of YL6500 GC (Flow calibration)

- **Detector**

Calibrates the temperature, proportional valves, and flow on [Detector] tab. The sort of used gas is different depending on detector, therefore, firstly select the gas used for your detector then start to do calibration procedure. All procedure is same with Injector section.

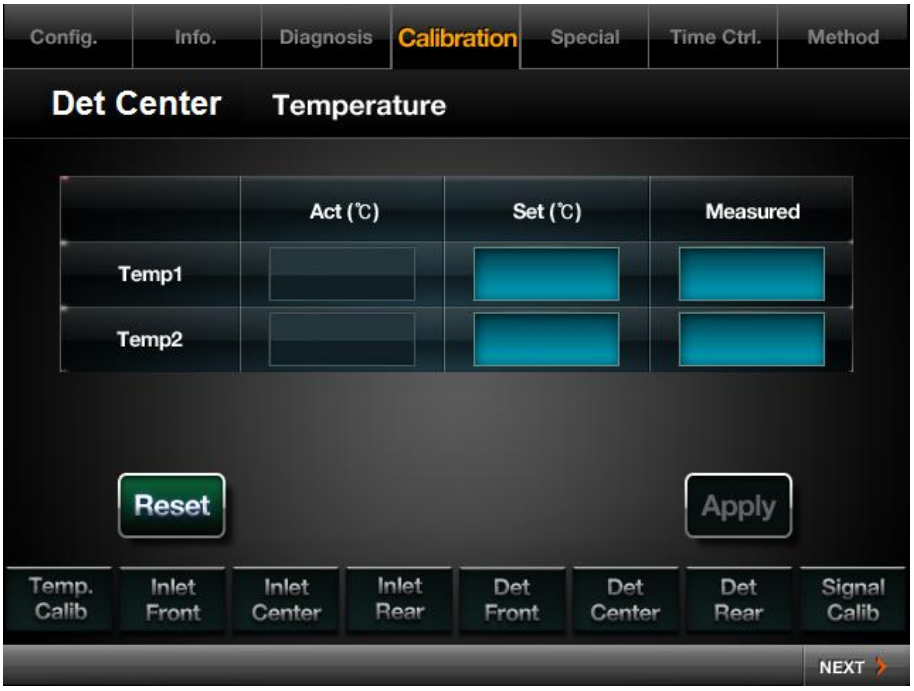


Fig 3-17. Calibration screen of YL6500 GC (Detector)

3-2-5. Special

This screen displays special functions for user convenience. The special function consists of 5 functions and you can move by tapping [NEXT] arrow at the bottom.

- **Run Start**

This is to run Auto repeat analysis function. You can set the number of running, run time, and cycle time.

✓ No. of Run

It displays the number of running. If you set this number, you don't need to press the [Start] key for each run. The maximum available number is 9,999.

✓ Run time

Set the running time for each analysis. You can not set this run time arbitrarily. The run time, which is set on the oven programming stage, is displayed.

✓ Cycle time

Set the [Cycle time] for each run. Cycle time should be longer than Run time or same with it. GC operates the next run when cycle time elapses.

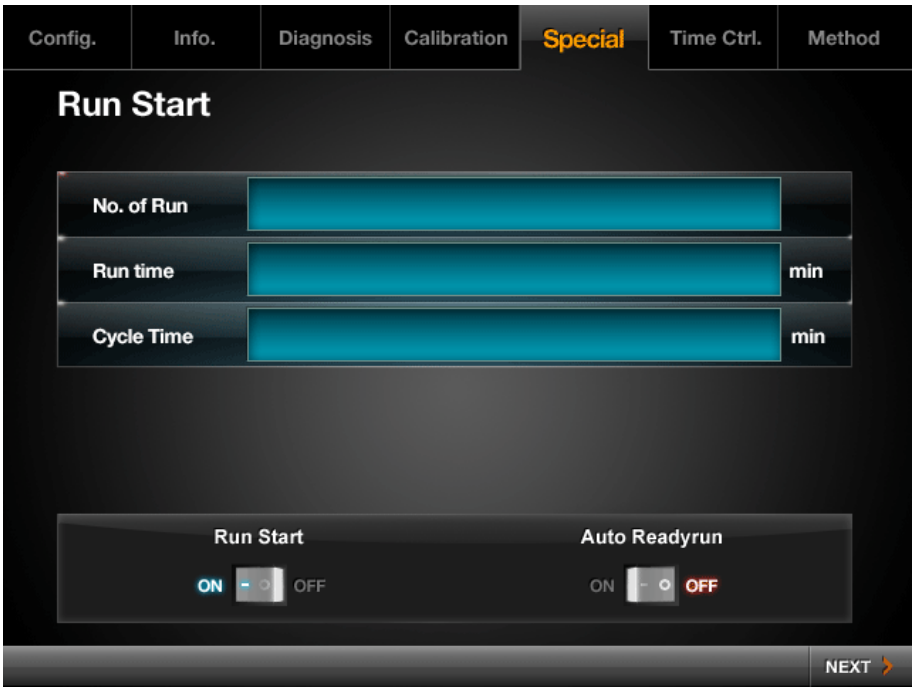


Fig 3-18. Special function screen of YL6500 GC (Run Start)

- **Post Run**

  [POST RUN] function is to clean the inside of a column after finishing an analysis operated by an oven program. You have to properly set treatment temperature in [Temp] tab and treatment time in [Time] tab according to the kind and features of a column. If not use this function, set the treatment time in [Time] tab to 0.0 min.

Fig 3-19. Special function screen of YL6500 GC (Post Run)

- **Remote Access**

You can set functions which send a simple signal to operate external devices. You can set the time for delaying a signal from an external device and that for starting an external event by sending a signal to an external.

Fig 3-20. Special function screen of YL6500 GC (Remote Access)

3-2-6. Time Control

YL6500 GC offers [Time control] function which enables you to operate your GC by a given time. A clock inside of GC makes GC run according to the time control program. When tapping on [Time control] tab, a table is displayed, and you can set DATE, TIME, and Function. You are able to make up to twenty steps.

- **Date**

Enter the date when you will operate with a six-digit. The date is a YY/MM/DD format. (Year/Month/Day)

- **Time**

Enter the time when you will operate with a six-digit number. The time is a HH/MM/SS format. (Hour/Minute/Second) Entered times are classified automatically in chronological order.

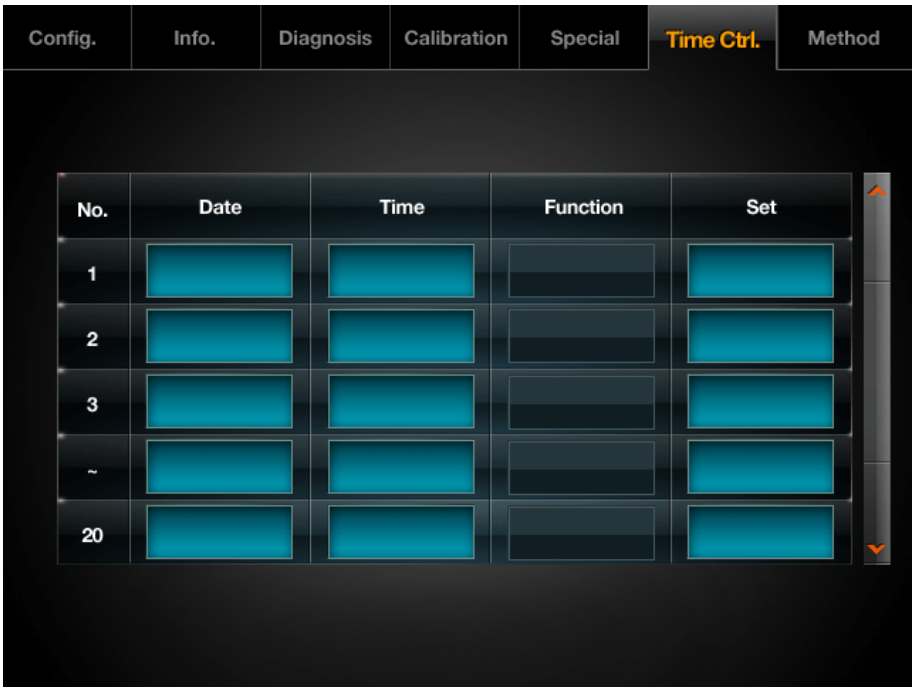


Fig 3-21. Time Control screen of YL6500 GC

- **Function**

Enter the function you want to operate automatically. If tapping on [Function] tab, you are able to select necessary functions from the Function list.

✓ Function List

Function	Description
System Start	To turn on the YL6500 GC. This function is available if the instrument is turned off by the [Time control] function.
Shutdown	To turn off the YL6500 GC.
Method Load	To load saved parameters(Method). If selecting this function, you are asked a method number.
Program Start	Same function with [Start] key, To operate a program.
Oven Temp	To set temperature for oven
Ready Run	To operate Ready Run function.

3-2-7. Method

The function is to load all parameters as Method file you saved. The [Method] function lets you recall and use all environment settings such as a temperature program, a flow program, parameters for a detector, signal, a valve, etc. by saving them at a time in a method number. Therefore, you do not need to remember all parameters for an analysis. Total 20 method files can be saved. The color of [Method] button is changed depending on whether it is saved or not, and it is able to overwrite altered parameters into an exist method file. If tapping on [RESET] key, all method files are deleted.

Fig 3-22. Method screen(1) of YL6500 GC

If touching one of method numbers, it shows as below screen whether it is saved or not. To save the method file, tap on [SAVE], and to load a method file, tap on [LOAD]. If you want to close this screen, tap on [CLOSE] at the upper right-hand.

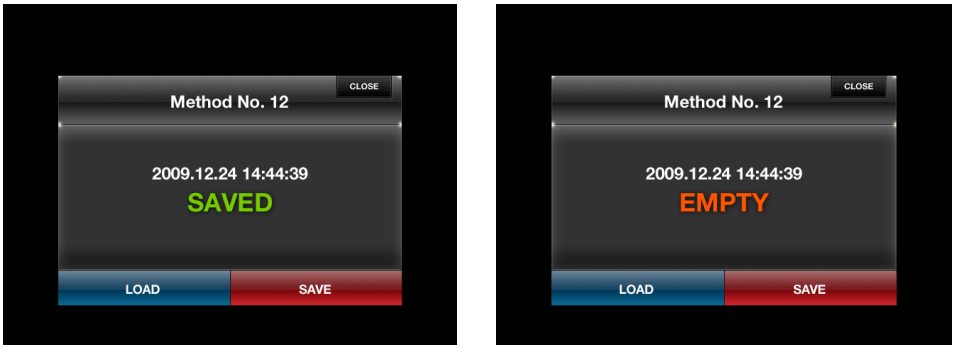


Fig 3-23. Method screen(2) of YL6500 GC

3-3. GC Control Screen

This is to set up the analysis condition. Each tab on the top side of screen is to be shifted and the value can be entered by a keypad. Tab activation differs according to the system configuration. The signal of inlet and detector is activated according to the type and the order.

3-3-1. Oven

There are 2 tabs which are [Config.] to set the basic environmental condition of oven and [Setting] to set the analysis condition.

- **Config.**

This is to set the basic environmental condition.

- ✓ Maximum Temp

  You can set a maximum oven temperature.

- ✓ Equilib. Time

  The Ready LED lights on when there is no change in set-up temperature within the Equilib. Time. The Equilib. time setpoint can be 0.1 to 9,999 minutes.

- ✓ Cryogenic System

  If you want to use a Cryogenic system, turn on this function and select one among LN_2 and LCO_2.

- **Setting**

This is to set up an analysis condition of oven. The current temperature is indicated in [Act]. Enter the settable temperature into [Set]. Depending on Oven Mode, the oven program will be activated. You can put the temperature ramping rate, setpoint temperature and specified period of time. (-) temperature can be set when the Cryogenic System is ON. Temperature programming is available up to 25 ramps and the maximum run time is 9,999 min.

Fig 3-24. Oven Control Screen of YL6500 GC(Configuration)

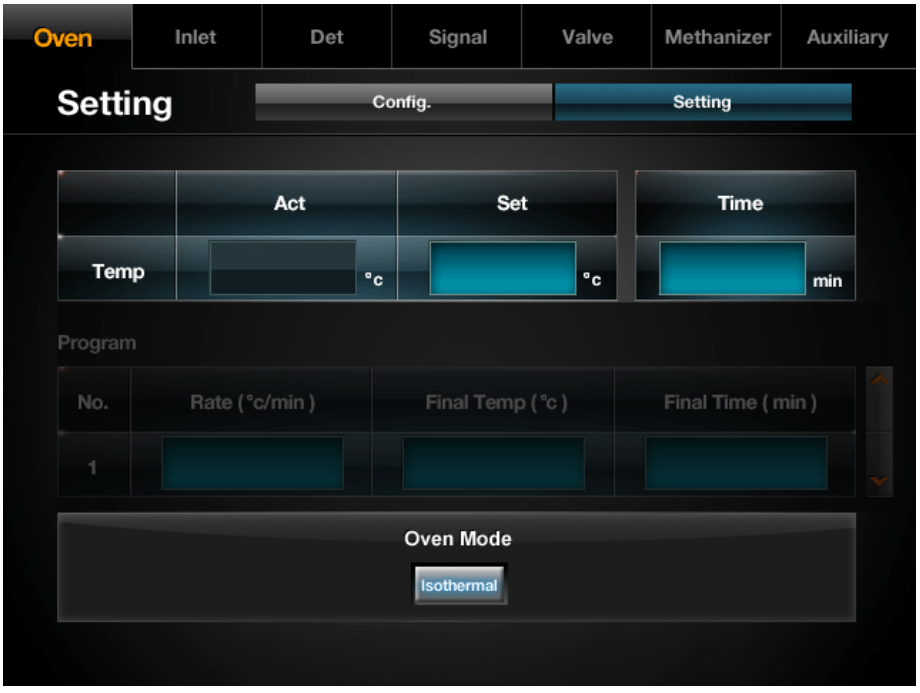


Fig 3-25. Oven Control Screen of YL6500 GC (Isothermal)

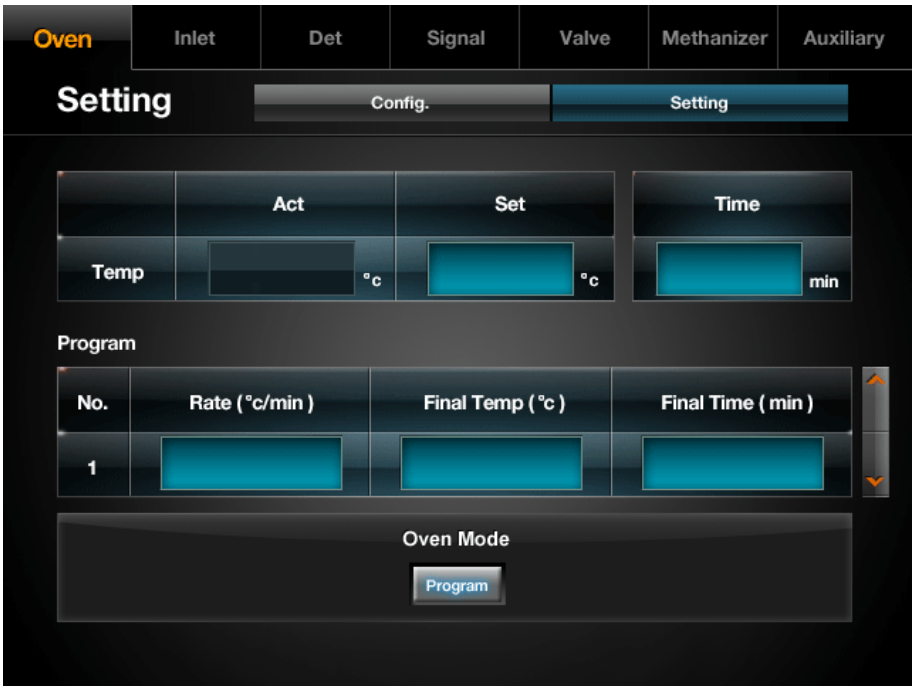


Fig 3-26. Oven Control Screen of YL6500 GC (Temp. Programming)

✓ Temp.

Enter either the initial temp. or the temp for an isothermal run. To set temperature programming, enter the settable temperature and press [ON].

✓ Time

It's the specified period of time in the set temperature.

✓ Rate

This is the ramping rate of temperature. The maximum ramping temp. is 100 ^{o}C/min. When you set the rate to 0.0, the temperature programming is not applied from the next step.

✓ Final Temp.

It's the final temperature that is to be reached at the ramp rate.

✓ Final Time

It's a specified period of time to hold the temperature at the final temperature.

✓ Oven Mode

There are isothermal and program (temperature programming) modes. The ramp temperature program is up to 25 steps and the maximum run time is 9,999 min.

3-3-2. Capillary Inlet

There are three tabs of **Config**. of **Inlet** and it's activated depending on the inlet location. The **Inlet** is composed to two tabs which are **Config**. to set the basic environmental condition and **Setting** to se the analysis condition.

- **Config.**

Fig 3-27. Control screen of YL6500 GC (Capillary Inlet)

✓ Column Setup

Enter the installed column specification such as column length, inside diameter and film thickness.

✓ Gas Saver

This function reduces the flow of carrier into the inlet and out the split vent after the injection is complete to avoid unnecessary gas consumption. In this condition, the column flow rate and pressure are maintained while split vent flows decreases. This is to be applied to the capillary inlet.

✓ Gas Saver ON/OFF : Select either turn Gas Saver ON or OFF

Time : Enter the time that Gas Saver starts based on run time.

Flow : Enter the split flow when Gas Saver function is on.

✓ Carrier Gas

There are 5 kinds gases to be selected as a carrier gas which are N_2,He,H_2,Ar and Ar/Me.

✓ APC Mode

There are 4 column flow control modes(APC modes) which are Constant Flow, Constant Pressure, Programmed Flow and Programmed Pressure.

- **Setting**

This is to set up an analysis condition of inlet. The current temperature, column flow and pressure of inlet are indicated in [Act]. When you set APC mode to Programmed Flow or Programmed Pressure, you'll be able to program the flow and pressure. Programmed Flow and Programmed Pressure can be set up to 5 steps. You can select parameters in Injection Mode and the screen configuration is changed depending on Injection Mode.

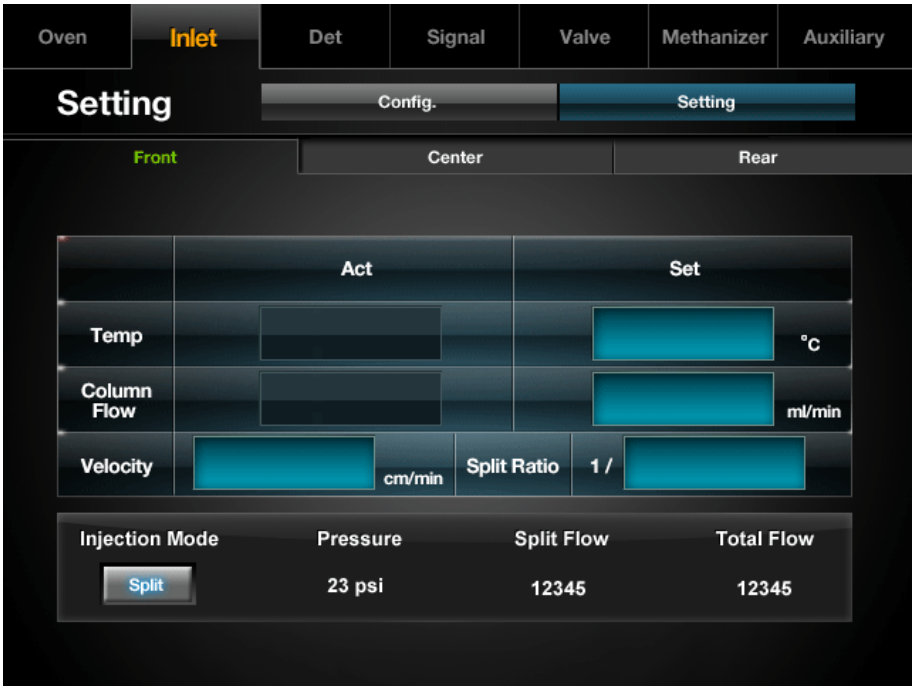


Fig 3-28. Control screen of YL6500 GC (Capillary Inlet)

✓ Temp.

Set the temperature of inlet.

✓ Column Flow

Set the column flow. Settable flow range is 0.1~100 ml/min.

✓ Velocity

This is to indicate the column flow to a velocity.

✓ Split Ratio

This split ratio determines the injected sample amount which is injected into a column and the amount which is drained into the split vent line. If you input a split ratio, the equipment controls Split Flow and Total Flow automatically according to the ratio.

✓ Injection Mode

- Split : A split injection method is generally used to inject small quantity of a sample into a capillary column. It makes very small quantity reach at the column according to split ratio of the injected sample.

- Splitless : A splitless injection method is suitable for a microanalysis of a solute which has high boiling point and dissolved in a solvent having low boiling point. And the method is used when the concentration of a sample is low. This method lets all sample go into a column during some time and then takes the split injection type.

In a splitless mode, Split Ratio turns to Split on Time. This time(Unit : min) is the period of operation in a splitless mode to a split mode. Setting this parameter to 0.0 turns the operation to a split mode right after the start of analysis.

3-3-3. Packed Inlet

- **Config.**

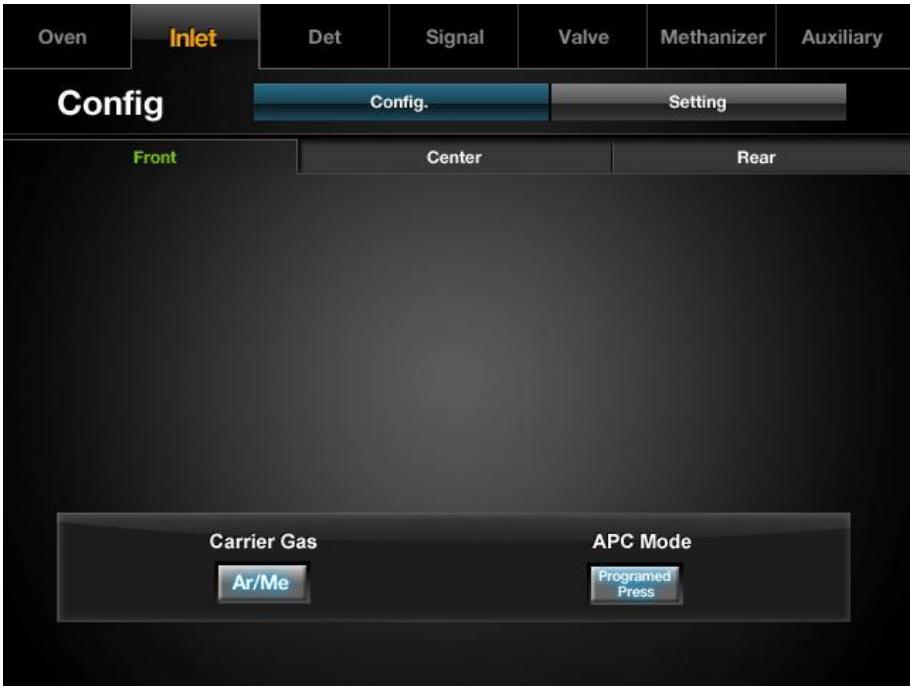


Fig 3-29. Control screen of YL6500 GC (Packed Inlet)

✓ Carrier Gas

There are 5 kinds gases to be selected as a carrier gas which are N_2,He,H_2,Ar and Ar/Me.

✓ APC Mode

There are 4 column flow control modes(APC modes) which are Constant Flow, Constant Pressure, Programmed Flow and Programmed Pressure.

- **Setting**

Fig 3-30. Control screen of YL6500 GC (Packed Inlet)

✓ Temp.

Set the temperature of inlet.

✓ Column Flow

Set the column flow. Settable flow range is 0.1~100 ml/min.

✓ Pressure

This indicates the pressure at the column.

3-3-4. On-column Inlet

- **Config.**

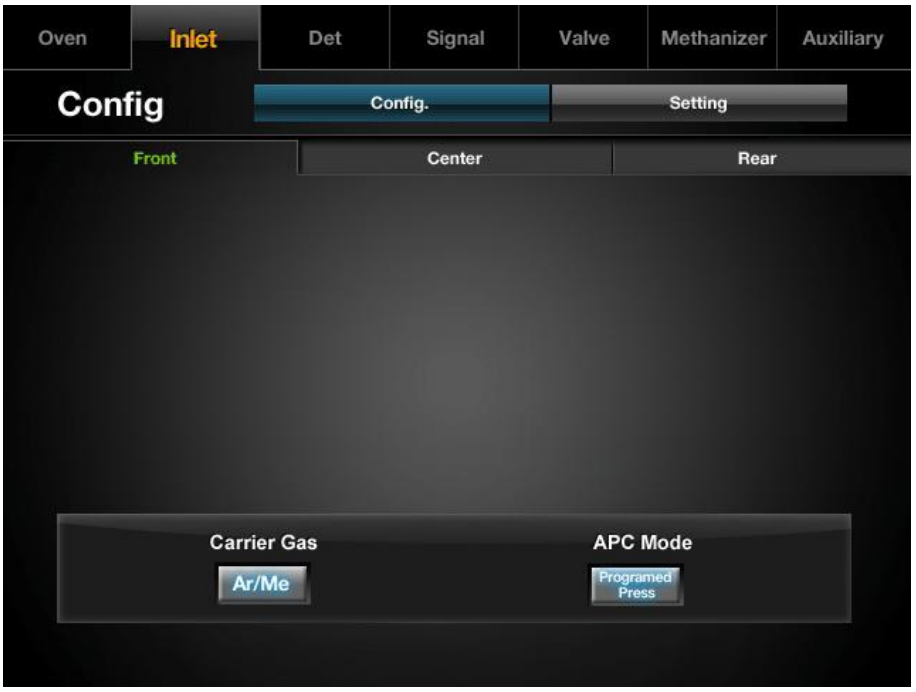


Fig 3-31. Control screen of YL6500 GC (On-column Inlet)

✓ Carrier Gas

There are 5 kinds gases to be selected as a carrier gas which are N_2,He,H_2,Ar and Ar/Me.

✓ APC Mode

There are 4 column flow control modes(APC modes) which are Constant Flow, Constant Pressure, Programmed Flow and Programmed Pressure.

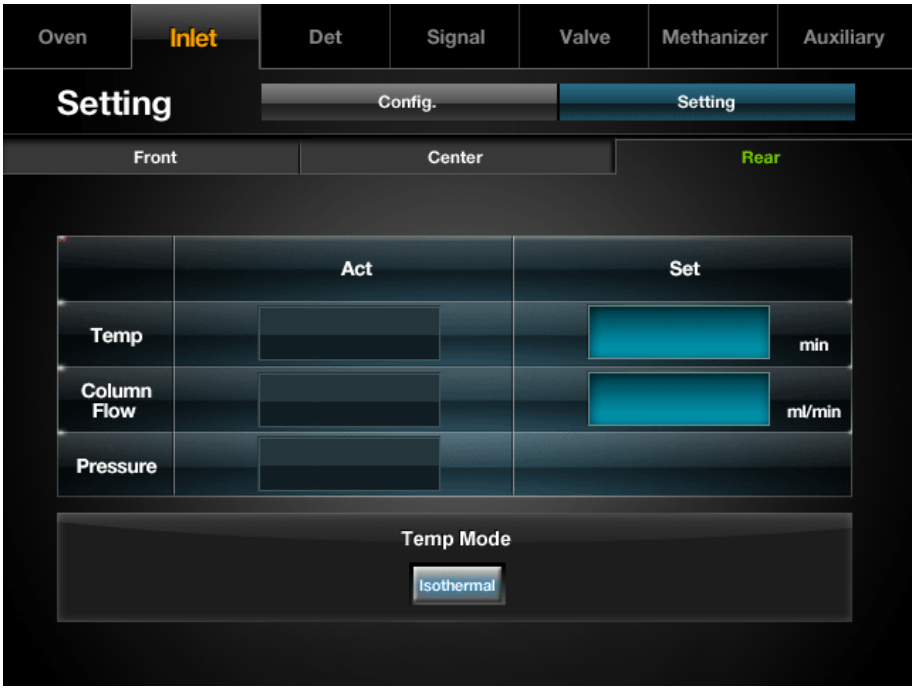


Fig 3-32. Control screen of YL6500 GC (On-column Inlet)

Fig 3-33. Control screen of YL6500 GC (On-column Inlet)

- **Setting**

  ✓ Temp.

  Set the temperature of inlet.

  ✓ Column Flow

  Set the column flow. Settable flow range is 0.1~100 ml/min.

  ✓ Pressure

  This indicates the pressure at the column.

  ✓ Temp. Mode

  It's able to set a temperature programming for an on-column inlet. There are 3 modes available for a temperature programming which are Isothermal, Program and Track Oven. In a Program Mode, it's able to set a temperature programming up to 5 steps.

3-3-5. Flame Ionization Detector

This is to set parameters for a flame ionization detector.

- **Config.**

  ✓ Lit offset

  "Lit offset" is a standard value for acknowledging an ignition failure when "Auto ignition" function is set. That is to say, when signal values are under a set value, the ignition is repeated. (Default : 5)

  ✓ Delay Time

  It is recommended to set a delay time to have enough time for the outside temperature of FID to reach over 150℃ . If not, there could be some moisture inside of FID block even if FID temperature already reached this point. Once FID reaches over 150℃, FID will start ignition after this time by flowing an auxiliary gas.

Fig 3-34. Control screen of YL6500 GC (FID)

✓ Ignite Flow

Set a flow rate of air. The default value is 100ml/min.

✓ Ignite Temp

This is the temperature that FID can attempt the auto ignition. The auto ignition only works after reaching this temperature.

✓ Makeup Gas

Select a Makeup Gas which is mostly a same gas as a carrier gas. Pressing button changes the gas type. There are 5 kinds gases to be selected as a carrier gas which are N_2,He,H_2,Ar and Ar/Methane.

✓ Connection

Select the inlet that is connected to a detector.

✓ Signal Autozero

When this is set to ON, a signal starts from '0' in an analysis.

- **Setting**

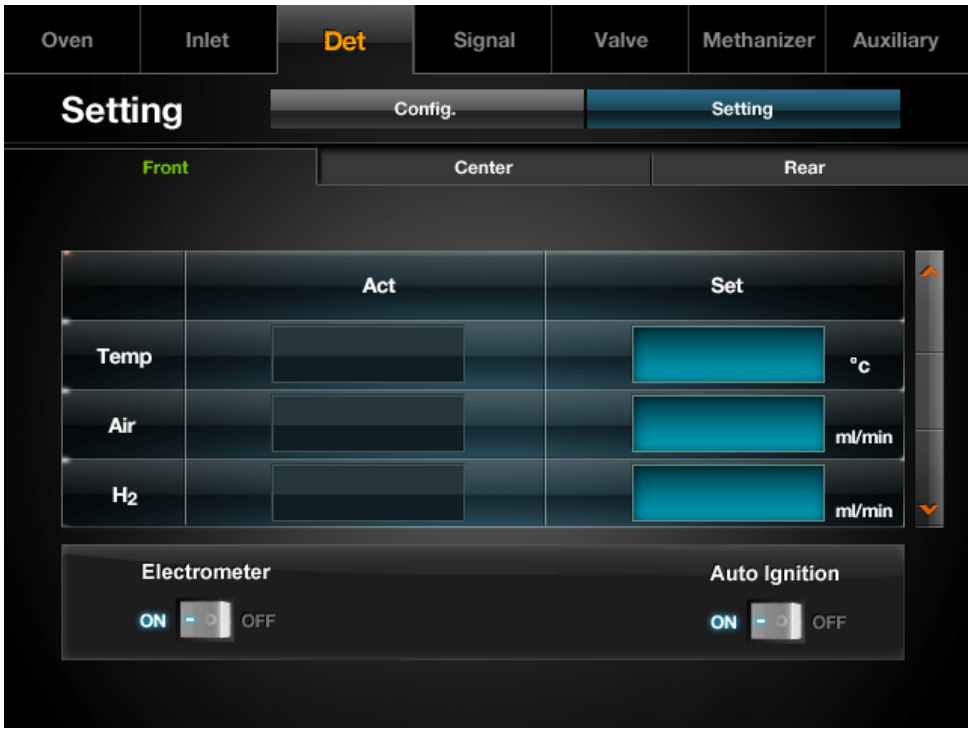


Fig 3-35. Control screen of YL6500 GC (FID)

✓ Temp.

Set a temperature of detector. It is recommended to set over 150 ˚C for FID to be ignited.

✓ Air

Settable flow rate range of air is 0~ 500ml/min. Mostly, the air flow rate is set in 300~400 ml/min.

✓ H2

Settable flow rate range of hydrogen is 0~100mL/min. Mostly, the H_2 flow rate is set in 20~40 ml/min.

✓ Mkup Gas

Settable flow rate range of Mkup gas is 0~100ml/min. Mostly, the Mkup gas flow rate is set in 20~40 ml/min. This is same as the carrier gas.

✓ Electrometer

This has to be ON to get a signal from a detector.

✓ Auto Ignition

When you set auto ignition function at a FID, ignition starts automatically. Firstly, select [ON] at

[Electrometer] and [Auto Ignition] after setting temperature of a FID. Then, air and hydrogen flow automatically and auxiliary gas is ready to flow. When temperature of a detector reaches a setting value, mixture of air and hydrogen flow and ignition start after [Ignition Delay Time] elapses.

If ignition fails even with trials more than 15 times, you can see an error message which tells you to check the system and auto ignition stops.

3-3-6. Thermal Conductivity Detector

- **Config.**

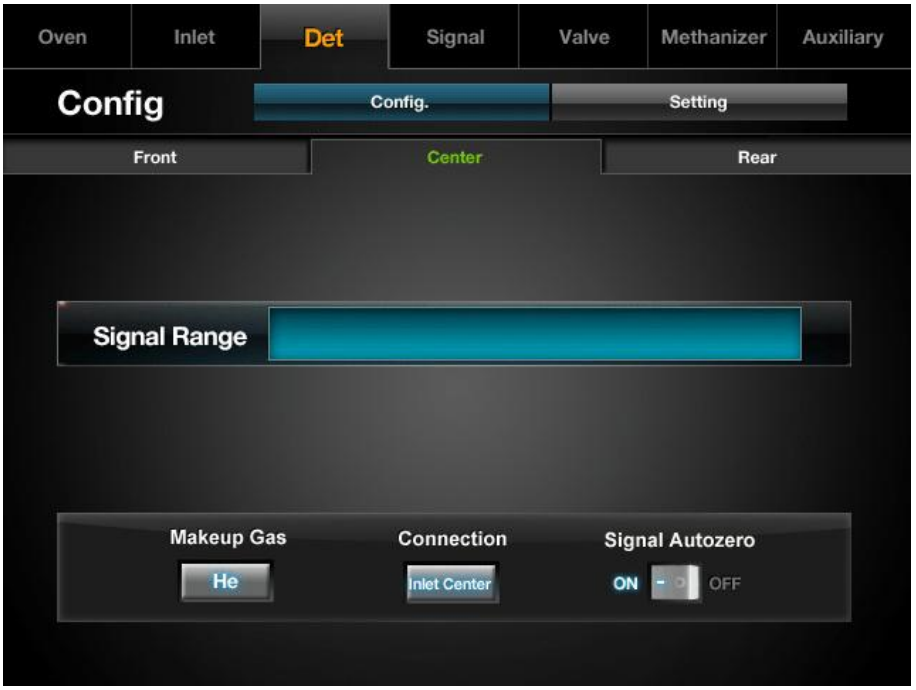


Fig 3-36. Control screen of YL6500 GC (TCD)

✓ Signal Range

Set a signal acquisition range. Settable range is 0.0 to 1000000.

✓ Makeup Gas

Select a Makeup Gas which is mostly a same gas as a carrier gas. Pressing button changes the gas type. There are 5 kinds gases to be selected as a carrier gas which are N_2,He,H_2,Ar and Ar/Methane.

✓ Connection

Select the inlet that is connected to a detector.

✓ Signal Autozero

When this is set to ON, a signal starts from '0' in an analysis.

- **Setting**

Fig 3-37. Control screen of YL6500 GC (TCD)

Fig 3-38. Control screen of YL6500 GC (TCD)

✓ Temp.

Set a temperature of detector.

✓ Ref. Flow

Set the flow rate that is toward the cell flowed standard current.

✓ Makeup Gas Flow

Set the flow rate that is toward the cell flowed samples.

✓ Sense

This parameter is to change the sensitivity of TCD by changing the current toward to the cell.

Fig 3-39. Control screen of YL6500 GC (TCD)

✓ Polarity

This function changes the polarity direction of a peak of a TCD. You can use this function when analyzing the component which has larger thermal conductivity than a carrier gas. You can change the direction of a peak only when the above component emerges and return the direction to the original (+) again.

If “Polarity Change” is “OFF”, the polarity is always “+”. If you want to change polarity, press ON at “Polarity Change”.

3-3-7. Electron Capture Detector

- **Config.**

Fig 3-40. Control screen of YL6500 GC (ECD)

✓ Makeup Gas

Select a Makeup Gas which is mostly a same gas as a carrier gas. Pressing button changes the gas type. There are 5 kinds gases to be selected as a carrier gas which are N_2,He,H_2,Ar and Ar/Methane.

✓ Connection

Select the inlet that is connected to a detector.

✓ Signal Autozero

When this is set to ON, a signal starts from '0' in an analysis.

- **Setting**

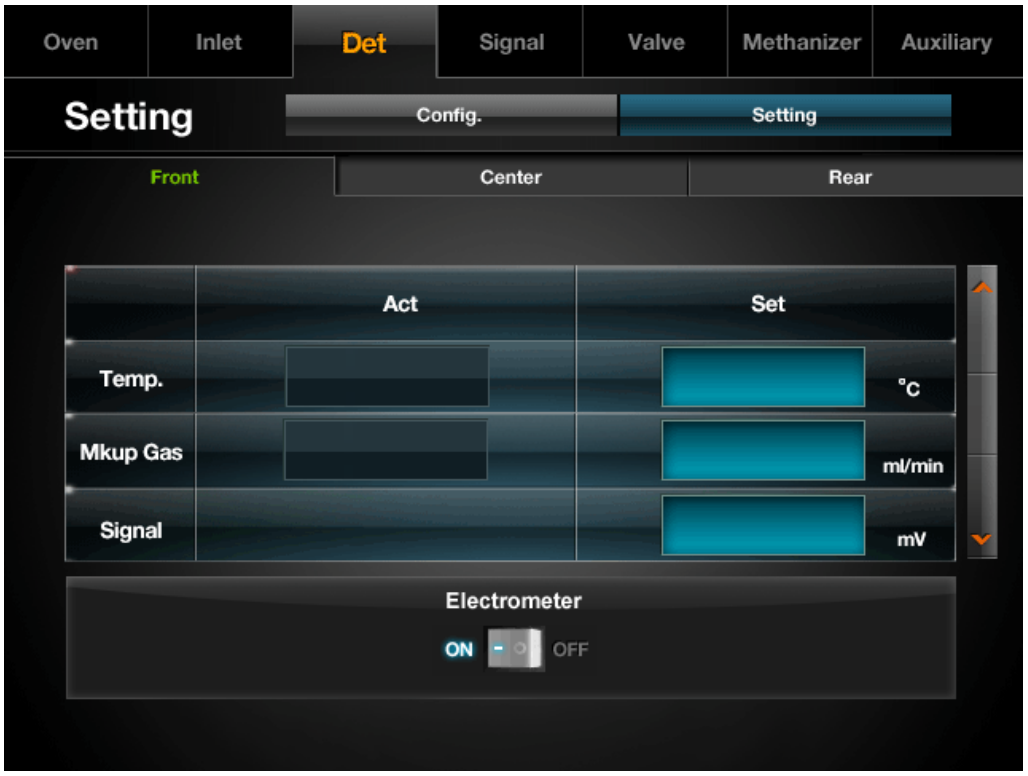


Fig 3-41. Control screen of YL6500 GC (ECD)

✓ Temp.

Set a temperature of detector. It is recommended to set over 150 ˚C for FID to be ignited.

✓ Mkup Gas

Settable flow rate range of Mkup gas is 0~100ml/min. Mostly, the Mkup gas flow rate is set in 20~40 ml/min. This is same as the carrier gas.

✓ Electrometer

This has to be ON to get a signal from a detector.

3-3-8. Signal

- **Config.**

Fig 3-42. Signal Screen of YL6500 GC

✓ Signal Zero

The detector signal is red from some point after subtracting this Zero value.

✓ Signal Range

Set a signal acquisition range. Settable range is 0.0 to 1000000.

✓ Signal Change

When this is ON, the detector output can be changed.

Fig 3-43. Signal Screen of YL6500 GC (Signal change)

3-3-9. Valve

- **Config.**

Fig 3-44. Valve Screen of YL6500 GC

✓ Type

Select the type of valve installed on GC. There are 2 types of valve which are LSV and GSV.

✓ Port

Set the no. or port.

✓ Volume

Set the size of sample loop.

✓ Conn.

Select the inlet that is connected to a valve.

3-3-10. Methanizer

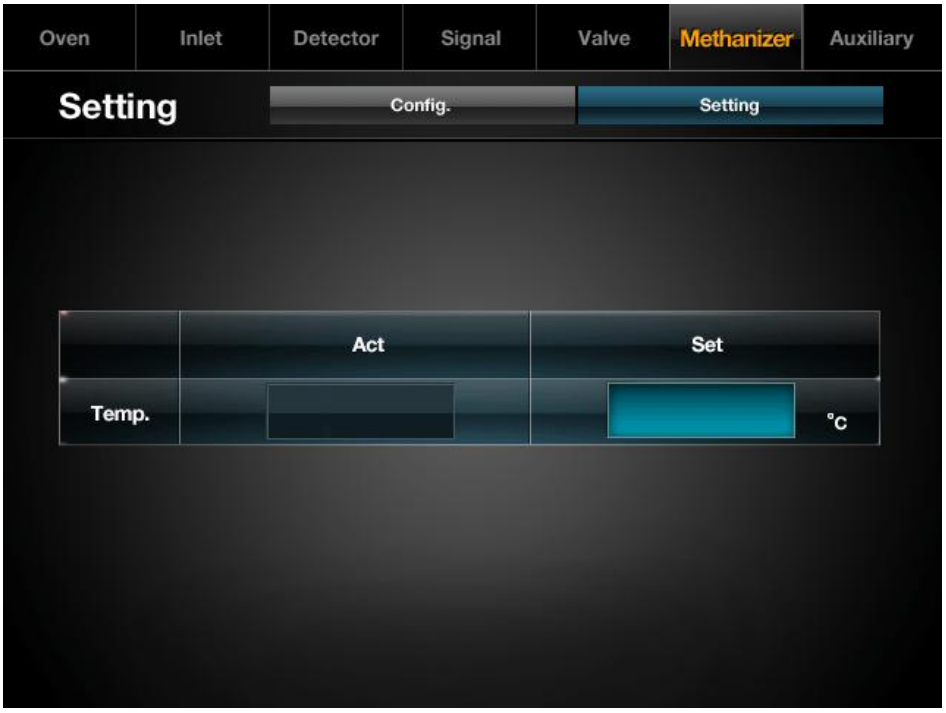


Fig 3-45. Methanizer screen of YL6500 GC

✓ Temp.

Set a temperature of a methizer.

3-3-11. Auxillary

- **Config.**

Fig 3-46. Auxiliary screen of YL6500 GC

✓ Max Temp.

Set a maximum temperature of Auxiliary.

- Auxiliary 1 is a maximum temperature of valve 1 & 2.
- Auxiliary 2 is a maximum temperature of valve 3 & 4.

- **Setting**

Fig 3-47. Auxiliary screen of YL6500 GC

✓ Temp.

Set a temperature of Auxiliary.

Chapter 4. Sample inlet and carrier gas controls

4-1. Introduction

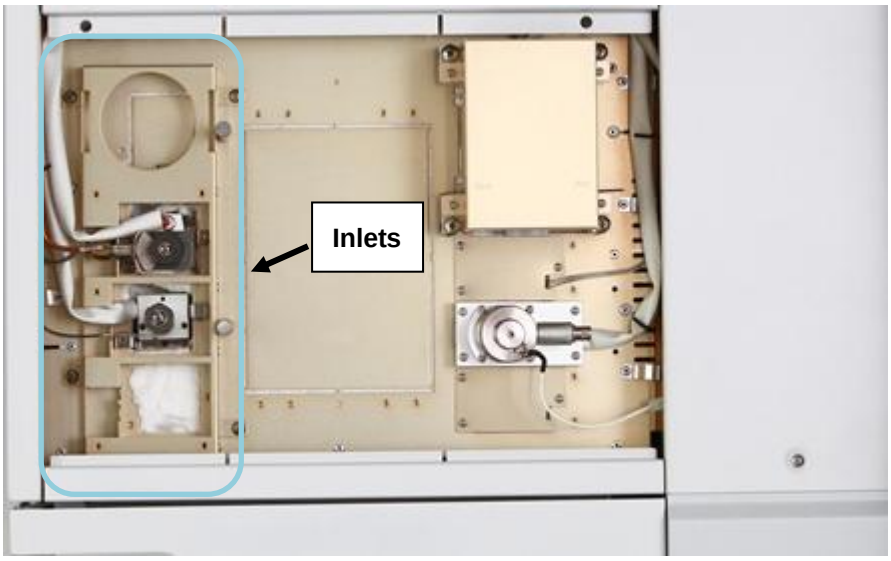


Fig 4-1. Inlets of YL6500GC

In the YL6500 GC, you can install up to 3 inlets as shown in the above figure and maximum inlet temperature is 450℃. We assume in this manual that the nearest one to the front side is as Front, the middle one is as Center, and the back one is as Rear. There are two inlet systems. One is an inlet for a packed column and the other is an inlet for a capillary column as an inlet system. If necessary, you can install an on-column inlet.

There are many ways to inject a sample into a chromatography column. An inlet system is determined by components of a sample and the type of a used chromatography column. The role of an inlet is to change a phase of all or parts of a sample into a gas phase in order to inject it into a chromatography column.

Generally, inlet temperature is set about 20℃ higher than temperature to volatilize a sample component which has the highest temperature to be volatilized and also about 20~50℃ higher than column temperature. To inject a sample, you can use a micro syringe through a septum which is at the end of an inlet. Although a septum has an ability to prevent a sample from leaking, you have to change it after using it many times. Also, an injection of a sample is made in a moment because a peak width should be short to have a good result of a quantitative analysis by letting a peak be separated well from an adjacent peak.

The YL6500 GC has the APC (Advanced Pneumatic Control) systems which control all gas flowing electronically to have high accuracy and convenience. The APC can be installed up to 6 at inlets, detectors, etc., and enables all inlets to be set in terms of flow and column pressure.

Table 4-1. Inlet specifications of YL6500 GC

Item	Packed Injector	Capillary Injector
Maximum Temperature	450℃	400℃
Pressure Setting Range	0.01-100 psi	0.01- 100 psi
Total Flow rate range	100 mL	0 - 400ml/min N_2, 1 - 800ml/min H_2 or He
Accessories	Septum, Liner, 1/4'Ferrule	Septum, Liner, O-ring Graphite ferrule

4-2. Packed column inlet

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You can inject an liquid sample (typically 0.1-10μl) into the inner heating region in a packed column inlet by using a micro syringe piercing a silicon rubber septum at the end of the inlet. The vaporized sample in the inlet is carried into a column by carrier gas.

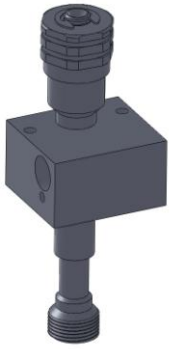


Fig 4-2. Packed Inlet

4-2-1 Structure of the inlet

A heater and a sensor is installed to maintain high temperature in a packed column inlet. You can use a capillary column at a packed column inlet and then have to use an adapter. You can use even capillary columns which have large inside diameter, 0.53mm or 0.75mm, at a packed column inlet by using an adapter for a capillary column.

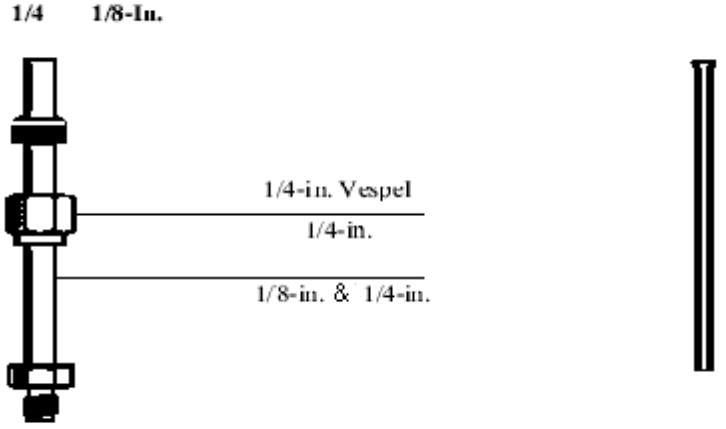


Fig 4-3. Liner & Insert for a packed column inlet

4-3. Capillary column inlet

Generally, a column having the inside diameter less than 750μm is used at a capillary column inlet, and the inside diameter of used columns is typically less than 320μm. Since a capillary column has smaller surface area than a packed column, even a 1μl sample may harm the column. Therefore, you have to inject 1μl or less than. To inject this small quantity, you can use a split injection method which lets only portion of a sample go into a column at an inlet itself. When the concentration of a sample component is very low, you can use a splitless injection method which reconcentrates the sample during some time and then the sample goes into a column. You can control these methods by using the keypad and LCD screen on the front side.

4-3-1. Split Injection

A split injection method is generally used to inject small quantity of a sample into a capillary column. It makes very small quantity reach at the column according to split ratio of the injected sample. Generally, the maximum sample quantity which does not carry an overload at a inside diameter 0.25mm WCOT (Wall Coated Open Tubular) column is 10-50ng per the component of the sample. That for a WCOT column which has large inside diameter and a SCOT (Support Coated Open Tubular) column is 1-10μg per the component of the sample. Therefore, high split ratio (50:1 ~ 500:1) is suitable for a WCOT column having high analysis ability, low split ratio (10:1 ~ 50:1) is proper for a WCOT column which has big sample capacity and large inside diameter and a SCOT column.

A sample is injected into the inside heating region through a septum. Carrier gas will mix the vaporized sample well at an inlet having high temperature. At a split point, some of a sample go into a chromatography column and the rest is pushed out through a split vent line. You can control the flow which flows in a column and the flow of a split vent by using LCD screen and number keys on the key pad. The purge vent is kept 3ml/min constantly. The split ratio is defined as the following.

Split Ratio = (Split Flow + Column Flow) / Column Flow

When analyzing a harmful sample, you can connect the end of a split vent to a fume hood or a trap. For a split of a sample in the case of split injection method, there may be quantitative errors when some components of a sample are not fully vaporized. And if inlet temperature is too high, there may be loss of components due to chemical decomposition or new chemicals which are not in an original sample may happen. A split injection method is not suitable for a microanalysis because most of a sample is wasted.

4-3-2. Splitless Injection

A splitless injection method is suitable for a microanalysis of a solute which has high boiling point and dissolved in a solvent having low boiling point. And the method is used when the concentration of a sample is low. This method lets all sample go into a column during some time and then takes the split injection type.

4-3-3. Structure of the capillary column inlet

A capillary column inlet is composed of an inlet for injecting a sample and the APC which controls the flow of the inlet as the following figure.

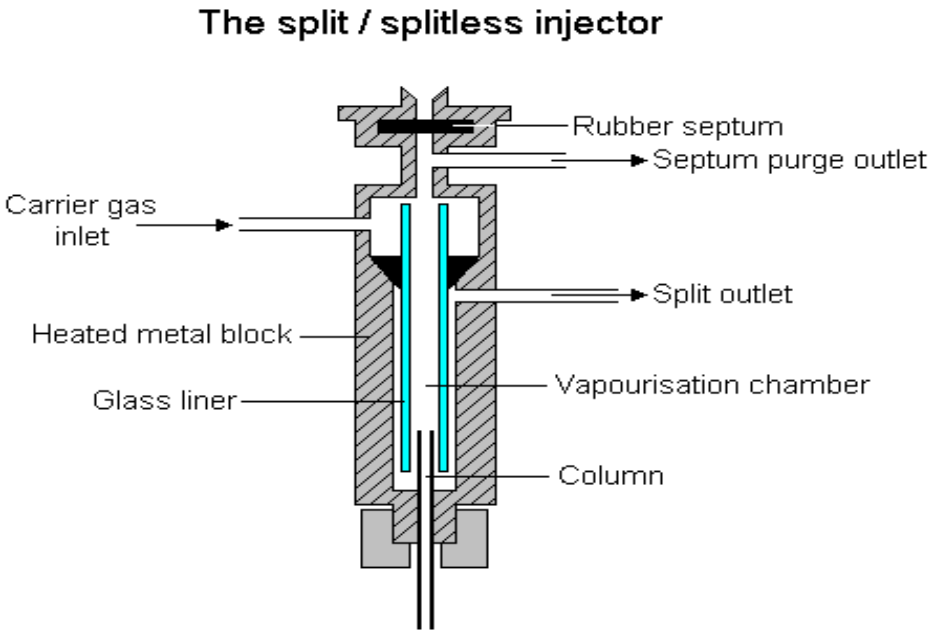


Fig 4-4. Structure of a capillary column inlet

There are basically two glass inserts for a capillary column inlet in the YL6500 GC. One is the cup type for a split and the other is the straight type for a splitless. Be sure to use them according to their uses.

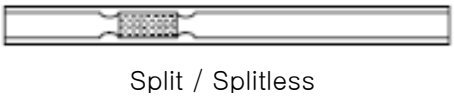


Fig 4-5. Capillary Glass Insert

4-4. Introduction for the Advanced Pneumatic Control (APC)

The APC(Advanced Pneumatic Control) means a system which controls gas stream electronically. The system controls the flow of gas used for a detector as well as the carrier gas used in the YL6500 GC. The APC uses electronic valves and sensors which control the flow of gas. There are four control modes - Constant Flow, Constant Pressure, Programmed Flow, and Programmed Pressure – for controlling the flow.

4-4-1. Constant flow

The constant flow mode keeps flow rate constant during an analysis. Generally, if gas temperature will be changed, gas pressure or flow rate of gas also varies. Therefore, this mode will change the gas pressure to keep flow rate constant when temperature varies.

4-4-2. Constant pressure

The constant pressure mode keeps the pressure acting on a column constant during an analysis. Therefore, this mode will change flow rate somewhat when column temperature varies.

4-4-3. Programmed flow

You can program the flow rate of carrier gas electronically in an analysis process by using the ramped flow mode as you program the temperature of an oven in terms of time in a GC analysis. The programming step is available up to five steps.

4-4-4. Programmed pressure

The ramped pressure mode can electronically program the gas pressure which acts on a column during an analysis. Also, the programming step is available up to five steps.

4-4-7. Gas saver

The gas saver function prevents gas from wasting by reducing the flow through a split vent during an analysis after injection of a sample. Although the flow through a split vent is reduced, the pressure acted on a column and the flow through the column remain constant. You can use this function in every setting (Split/Splitless/pulsed split/pulsed splitless) and manage on the LCD screen.

Gas Saver ON/OFF : To turn on/off the gas saver function.
Flow : To set split flow when using the gas saver function.
Time : To set when the gas saver function starts. Input the value on the basis of run time.

After starting “Run” by injection of a sample, the split flow remains constant during the indicated time in this function and then the gas saver function starts. In the case of the Splitless, if the “Saver Time” is smaller than the “Split On Time”, the value is not valid.

4-5. Flow control of carrier gas

The YL6500 GC has three inlets. Generally, we designed a capillary column is for the [Front] or [Center] and a packed column is for the [Rear]. Therefore, we are assuming that the inlet is connected with a capillary column in the Front and the other inlet is connected with a packed column in the Center.
In order to control the flow of carrier gas, you must choose the type of carrier gases, the environment of APC system, and then other details.

- Choose the type of carrier gas at the bottom. The displayed type of carrier gas is changed whenever touching on it.
- Choose a control method for the flow on APC Mode.
- Select the detector location connected to the inlet on Detector conn.

4-5-1. Choosing carrier gas

You can choose the kind of carrier gas by touching on Carrier gas tab.

Choose one carrier gas among these types, 

by touching on this icon until it displays the desired type.

4-5-2. Environment setting for the APC

You can set the dimension of column, and the position of injector and detector connected to the column.

1) Dimension setting of a column

Input the length, diameter, and film thickness of a used column by touching on tabs and number keys on keypad, and then press [Enter] key. You have to input these values because flow rate is determined by length and diameter in a capillary column.

2) Choosing detector

Select the detector location connected to the inlet on Detector conn. by touching on this icon repeatedly until it displays the desired type.

4-5-3. Function setting of the APC

You have to make a function setting of APC before setting flow or pressure of carrier gas. You may choose a control method for flow on APC Mode by touching on this icon.

1) Choosing Flow Mode

You can choose four control modes for the carrier gas flow by touching on APC Mode icon.
The modes are determined by selecting one among four modes (Const Flow, Const Press, Ramped Flow, Ramped Press). The carrier gas flow is changed by temperature. If you select the mode of Const Flow, the pressure will be adjusted automatically and the rate of flow will be remained at a certain value. In the mode of Const Press, the rate of flow will be a little changed according to temperature because the pressure of the column is adjusted constantly. If using a packed column, you can select only the mode of Const Flow not the mode of Const Press.

If you select Ramped Flow mode, the mode screen contains entries for setting up the program. With this mode, you can control the flow of carrier gas according to time. You begin with entering the initial flow rate (InitFlow), initial flow quantity, and initial time (InitTime) on the key screen. The unit of flow rate is ml/min, and time is expressed in min. In the next step, you can input the value of ‘Rate 1” in order to change the flow rate. The unit is ml/min.

Enter the desired value of the first step flow rate (Final Flow1) and the remained time reaching the flow rate. If desired more, enter the second value into the second row. Repeatedly, you can set upto 5 steps.

With the mode of “Ramped Press”, you can set up the program similarly. But if choosing a packed column, you cannot do this.

2) Display of flow rate of carrier gas

You can see ‘Velocity” on the Inlet setting screen. This parameter means the flow rate of carrier gas in the case of a capillary column.

3) Function setting for Gas Saver

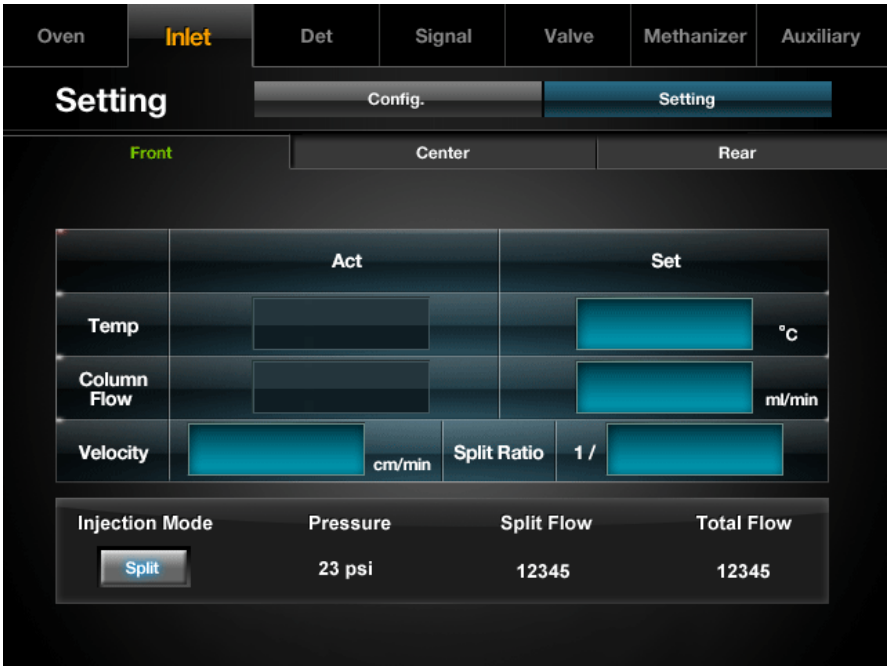
You can select the function “Gas Saver”. This function can be used only for a capillary column. This prevents the waste of carrier gas by reducing the quantity of flow through the Split Vent during the runs. If you select this function, the column pressure and quantity of flow is kept constantly even though the quantity of flow through the Spit Vent is reduced.

Select [ON] on Gas Saver, and input the values of time and quantity of carrier gas that flow through the Split Vent during the function.

Gas Saver ON/OFF : To turn on/off the gas saver function.
Flow : To set split flow when using the gas saver function.
Time : To set when the gas saver function starts. Input the value on the basis of run time.

4-5-4. Function setting for a capillary column inlet

Set inlet temperature, flow of carrier gas, sample injection method, etc. If touching on Front tab of Inlet, you can see the following window.



1) Temp

Set the temperature parameter of the inlet. To determine the temperature of the inlet, firstly touch on Set tab and input desired value (the unit is °C) using number keys then press Enter key. The maximum temperature is 450 °C, and you can set the temperature in units of 1 °C. But you can set up it until 400 °C in the inlet for a capillary.

Press [ON] key on key pad to heat it until the set point. Then you can see the set point instead of “OFF”. If you want to change the temperature during the heating period, just put the desired value and press ENTER】.

2) Injection Mode

In this step, you can determine the way of injecting a sample for the inlet of a capillary. There are two sample injection modes – Split and Splitless. Choose one mode between of them by pressing Injection Mode icon.

3) Pressure

You can set up the column pressure in the case of Constant Pressure mode when determining the function of APC. The input range is between 0.01psi and 100 psi. To set up the pressure value, just input the desired value and press [ENTER] key regardless of “OFF’ or a certain value.

4) Col Flow

You can determine the flow rate through the column in the case of using a packed column or the Constant Flow mode in the function of APC. The rate range is between 100ml/min and 0.1ml/min. To set the value, just put the desired value and press [Enter] key without regard to the case of “OFF” or a certain value.

5) Split injection mode

a. Split Ratio

This split ratio determines the injected sample quantity which is injected into a column and that which is injected into the split vent line. If you input a split ratio, the equipment controls split flow and total flow automatically according to the ratio.

b. Split Flow

This determines flow rate which flows to an exit of an inlet after being splitted. If you change a split flow, a split ratio and a total flow are changed according to this because a column flow is a fixed value.

6) Splitless injection mode

a. Split On Time

This is the time when returns to a split mode after being operated in a splitless mode (The unit is min.). If you input 0.0 at this, a split mode starts immediately when pressing [START] key.

7) Total Flow

A total flow is a value which is added a column flow, a split flow, and a purge flow (exited to a septum). If you change a total flow, a split ratio and a split flow is changed because a column flow is fixed.

4-5-5. Function setting for a packed column inlet

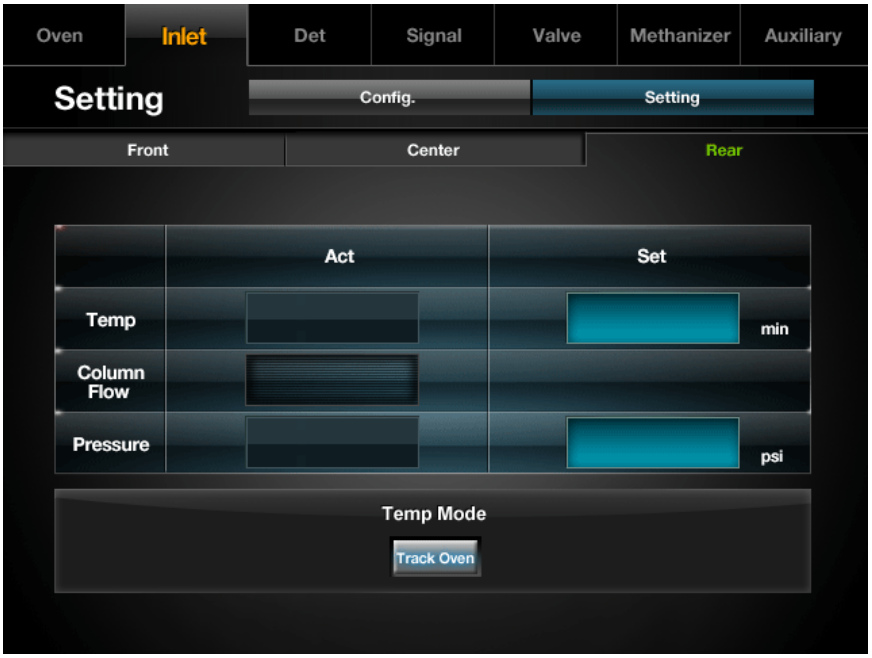
When selecting a packed inlet, you need to input only inlet temperature and flow rate of carrier gas as the following window shown by using number keys.

Temp : To set inlet temperature

Pressure : Displays the current pressure applied on a column (Not to set it)

Flow : To show the flow rate through a column and set it

4-5-6. Function setting for a packed on-column inlet



Chapter 5. Oven and column

5-1. Oven

An oven is a device which improves accuracy and confidence of an analysis result by controlling column temperature exactly for an analysis. The standards of the YL6500 GC are followings.

- Maximum temperature 450℃
- Minimum temperature –80℃ (Using liq. N_2)
- Maximum ramping rate 100℃/min
- Temperature programming 25 steps
- Maximum running time 9,999 min

5-1-1. Structure of an oven

An oven controls temperature using a heater and a fan when a flap door at the back side of the oven is opened or closed. It cools up to +5℃ higher than room temperature without extra cooling device. The oven temperature is measured by using a platinum RTD (resistant temperature detector). If analyzing a sample under room temperature, you need extra cooling device such as liquid nitrogen (-80℃) or dry ice (-50℃). Temperature programming is available up to 25 steps and maximum running time is 9999min. In order to open the oven door, just push a button on the right-down side of the oven door. This oven is designed to stop to operate an oven fan and a heater when the oven door is opened during running in order to protect a user and the equipment.

5-1-2. Environment setting for oven

In order to operate the oven, firstly you have to make environment setting and operating condition for it by tapping on LCD screen and using numeric keys on key pad.

To access the following screen, tap on [GC] icon at [Home] display then touch on [Config.] tab of [Oven] item. In order to input parameters, tap on each tab, input parameters by using numeric keys then press [Enter] key.

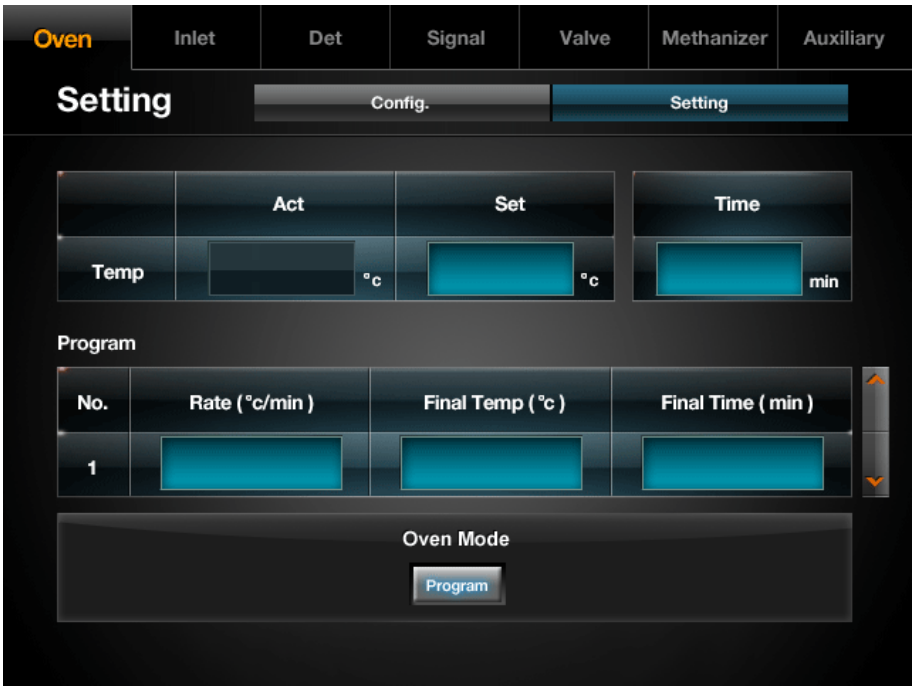
The items about environment setting of an oven are shown in the following table.

Table 5-1. Oven Capabilities

Setting item	Setting value	Description
Maximum Temp	0~450℃	To set maximum temperature of an oven
Equilib. Time	0.0~9999	A ready ramp will light up if temperature is not changed during equilibrium time after reaching a set temperature. This is available up to 9999 min from a decimal point.
Cryogenic System	On/Off	To turn on/off a cryogenic cooling system. You can control a valve for liquid nitrogen if this is “ON”.
Coolant	LN_2/Co_2	To select the type of gas used for cryogenic option.

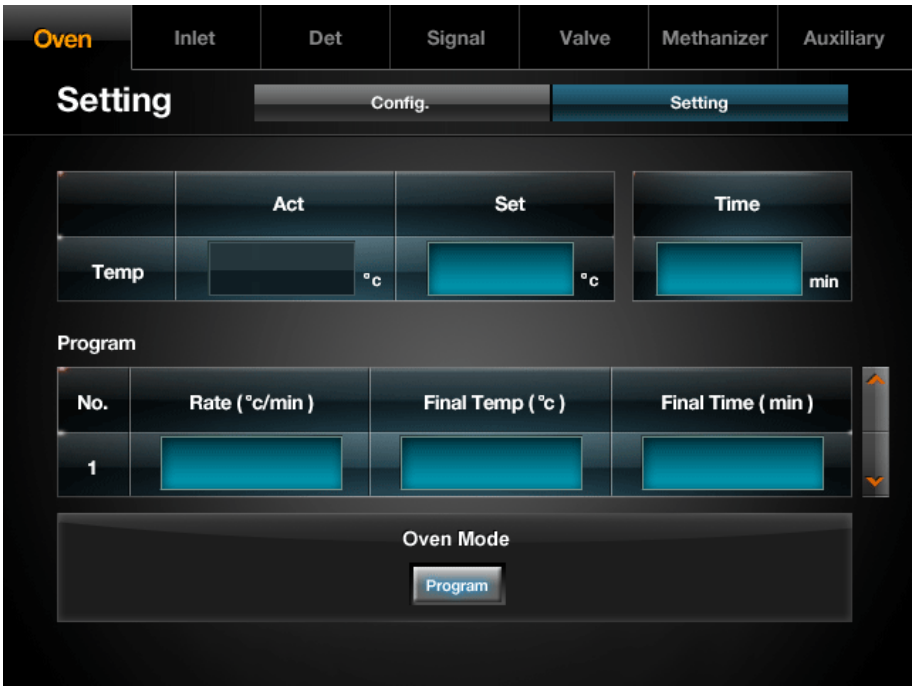
5-1-3. Use for an oven temperature controller

After tapping on [Setting] tab and selecting [Oven mode] as [Program], enter the oven parameters for program mode on the following window.



On the above window, The temperature on [Set] tab indicates an initial temperature of program mode. Initially, it displays “OFF”. In order to set the initial temperature, tap on [Set] tab and enter parameter with numeric keys and then press [ON] key. As soon as pressing [ON] key, the oven starts to heat immediately. **That is to say, if a temperature value is shown on the [Set] tab, the oven heater is operating.**

“InitTime” means the time (min) which goes from pressing [Start] key in order to operate a ramping program to starting ramping. After this time, the ramping program starts. (In the case of an isothermal analysis, this is running time for an analysis.)



By pressing [Enter] key after inputting each value at [Rate], [Final Temp], and [Final Time] in the first row, you can make the first step ramping program. If tapping on a down arrow, you can see the second step ramping program window. You can make the ramping program as the first step program. If you input 0.0 at [Rate] in the second row, the other step ramping programs will be invalid as well as the second step program. If you continue, you can make the ramping program until the 25th step.

Item	Unit	Explanation
Temp on [Set] tap	^{o}C	Initial temperature or temperature during an isothermal analysis
Time	minute	Time which goes during maintaining initial temperature for an isothermal analysis, Or run time for an isothermal analysis
Rate	^{o}C/min	Ramping rate of an oven at each step, Maximum effective ramping rate is 100 ^{o}C/min. If you input 0.0, the next steps will be invalid from the step.
Final Temp	^{o}C	Final temperature which is reached by heating at the former ramping rate.
Final Time	minute	Time which maintains the final temperature
N = 1~25, Commonly maximum time is 9,999 min.		

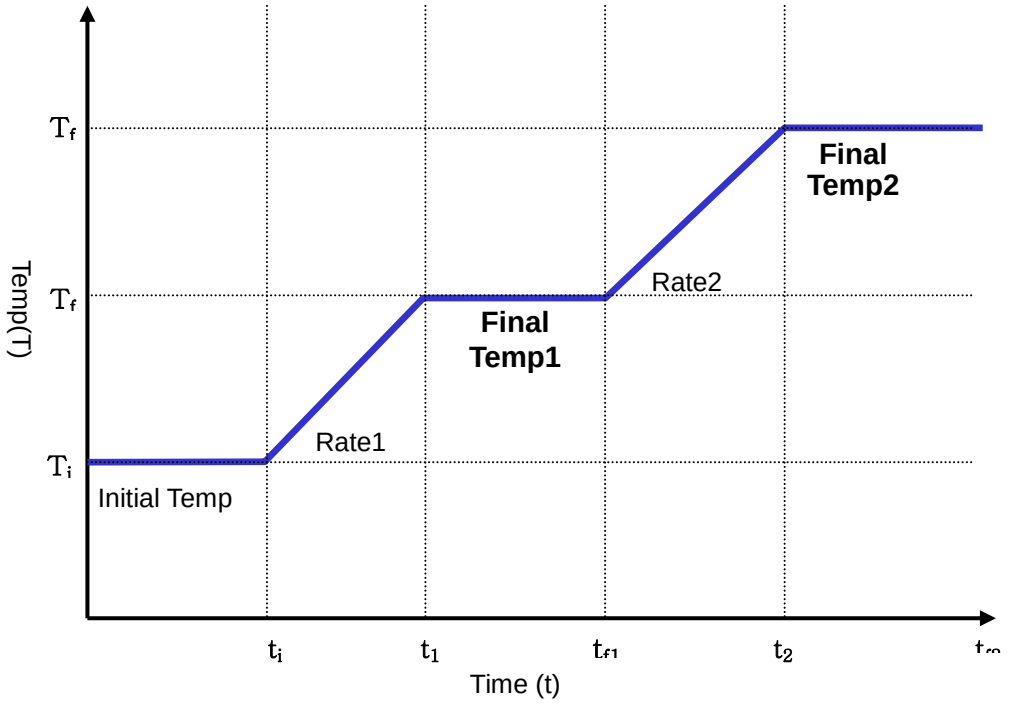
At first, an oven is at initial temperature. But, if you press [Start] key, a ramping program will start. When ramping program goes, the error range between measured temperature and set program is less than ±0.5 ^{o}C.

The following figure is a graph about changes in terms of time and temperature when temperature programming runs.

Explanation of the following figure “ti” is the time which is set at the above “ init time” and “t1” and “t2” are times which go to each “final temp1” and “final temp2” at “Rate1” and “Rate2”.

Also, “tf1” and “tf2” mean each “final time1” and “final time2” which go at final temperature.

Ti = initial Temp, Tf1= final Temp 1, Tf2= final Temp 2



5-1-4. Post run function

The following is [Post run] screen. To access this function, tap on [System] icon at [Home] display, touch on [Special] tab and then tap on [NEXT] arrow at the bottom until this screen is displayed.

You can use the [POST RUN] function to clean the inside of a column after finishing analysis according to an oven program. You have to properly set treatment temperature on [Temp] tab and treatment time on [Time] tab according to the kind and features of a column. If you will not use this function, set the treatment time [Time] as 0.0.

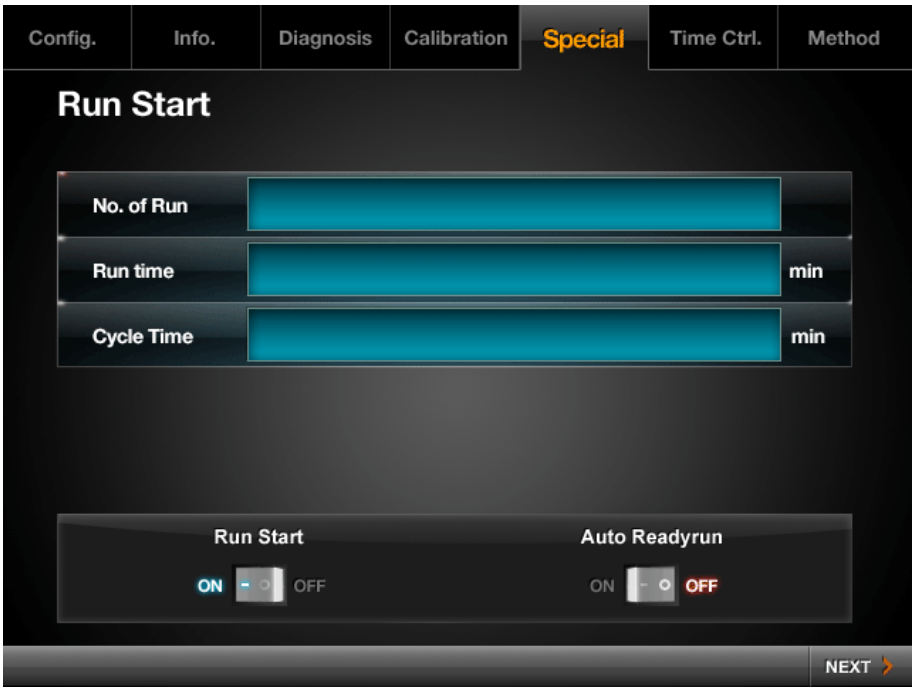
5-1-4. Returning to ready status

You can use [Ready Run] key for the case which the functions such as [Gas saver] and [Splitless] are set in a program for a capillary column inlet. If these functions are set, you have to set the flow conditions for operating these as initial values after finishing a GC program.

If you want to use this function, set the flow condition as initial value for operating a sample analysis program by pressing [Ready Run] key after finishing a sample analysis. And then, inject a sample. If [Ready Run] runs, you can see [Ready Run] message on [Display] screen.

5-1-5. Auto ready status

You can operate the instrument automatically without pressing [Ready Run] key after every analysis is done by the following procedure. If tapping [Special] tab at [System], you can see the following window. Then, if you select [Auto Readyrun] to [On], the equipment operates automatically.



Default : Auto ready status

5-2. Column

A column plays an important role in separating components in a sample. It is installed between an inlet and a detector. You have to check out a fitting, and a liner, an adapter, and an insert which are used at an inlet, and a detector to install a column. Especially, in the case of a 1/4 inch glass column, you have to use the column according to standards because the column is chosen according to the distance between an inlet and a detector.

5-2-1. Choosing a column

Columns are divided into several parts according to what they are made of. Even though they are made of same materials, they are also divided into several parts according to purposes of usage and kinds of stationary phase.

Columns are divided roughly into packed columns and capillary columns. A packed column is packed with something and a capillary column is a column whose inside is coated with stationary phase. Columns are divided into GSC and GLC according to the status of materials which is packed in or coated in a column.

1) Packed Column

A packed column is made of stainless steel, glass, nickel, etc.. the average of length is about 6~20 feet. Normally, the outside diameter (O.D) is 1/8 inch, 3/16 inch, or 1/4 inch and the inside of diameter (I.D) is 2mm ~ 4mm. Stationary phase which is packed in this column is a solid itself or a solid on which nonvolatile liquid is coated. Generally, resolution is low. The packed stationary phases are methyl silicon, molecular sieves, , porous polymer, etc. and other special materials are used for the packed stationary phase.

2) Capillary Column

Generally, the inside diameter (I.D) of a capillary column is about 0.1-0.53mm, and very thin film is coated on the inside of the column. Since pressure depression of gas according to length is not very large in the case of a capillary column, the column can be very long (10-100m). Therefore, the theoretical plate is greatly large. But, the quantity of a sample which can be injected is very small. A capillary column is mainly made of fused silica and also unusually made of stainless steel.

3) GSC

This column is packed with solid stationary phase and mainly used for separating inorganic gas or hydrocarbon having low molecular weight by absorption at room temperature. The used stationary phase is charcoal, silica Gel, zeolite, molecular Sieve, porous polymer, etc..

4) GLC

If stationary phase is liquid, liquid is coated on solid support, which is packed in a column. The quantity of liquid should be enough to be coated on the solid support. But, too much reduce efficiency of the column. Also, since retention time is proportion to the quantity of liquid, small quantity enable you to analyze a sample quickly. Liquid have to resolve components of a sample and have different partition ratio according to the components. Also, it have to be nonvolatile and be stable so that it does not react with a sample chemically.

Fig 5-1. Capillary Column

Fig 5-2. Packed Column

5-2-2 Fitting

You need some kinds of fittings to install a column at GC. The following table summarizes features of typical fittings.

Table 5-2. Fittings which are used when installing a column

Materials	Application	Features
Stainless Steel Ferrule	Metal packed column Capillary column Connecting tubing	Long length of life span and possibility to be used in high temperature, Notice: Too tightening may harm a column, an inlet, and a detector.
Brass Ferrule	Metal column Connecting tubing	Possibility to be used in over 250℃ Possibility to be leaked when ramping
Graphite Ferrule	All packed column Capillary column	Possibility to be used up to 400℃, Long length of life span, High performance, Possibility to be installed or removed easily, Strong absorption, No use at a path of a sample
Teflon Ferrule	All packed column Capillary column	Possibility to be used in low temperature (~250℃), Possibility to be installed or removed easily, Possibility to be leaked when ramping No effect on a sample – for a sensitive column
Vespel Ferrule	All packed column Capillary column Especially, glass column	Possibility to be used up to 350℃, High endurance and non-active to fire Pay attention to using a sensitive column
Silicon O-ring	Glass column	Possibility to be used up to 250℃, It is not good at high-sensitive analysis.

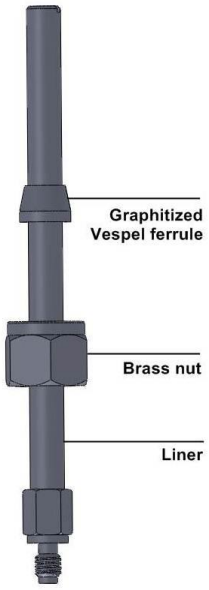
5-2-3. Liner, Adapter, Insert

A liner and an adapter are installed at the inside of an oven and are used to connect a column with an inlet and a detector. An insert is used only for an inlet. You have to install these before installing a column.

1) Liners and inserts for a packed column inlet

There are two liners for a packed column inlet. One is 1/8inch for a packed column, and the other is 530μm for a capillary column. Also, a glass insert is used to reduce reactivity to a sample. Moreover, a glass insert which includes silicides and silanized glass wool is used to catch residue of a nonvolatile substance. When changing a liner and an adapter, you need a 1/4 inch vespel ferrule and a 1/4 inch nut. Also, when using ferrule, you had better not to use a metal ferrule because you have to replace a liner too when leakage happens.

You can install an insert as the following procedure.



a. Be careful not to contaminate the surface of an insert.

b. Place the insert into a liner for a packed column inlet with making the trumpet part on the end of the insert up.

c. Install a liner for a packed column inlet at an inlet.

d. Check the leakage with “SNOOP”.

Fig 5-3. Installation of liner for Packed inlet

2) Adapter for a detector

You need an adapter when connecting a packed column or a capillary column with a detector. Although you need an adapter for a 1/8” metal column, you do not need it for a 1/4” glass column as an inlet. When changing a liner and an adapter, you need a 1/4 inch vespel ferrule and a 1/4 inch nut. Also, when using ferrule, you often use a metal ferrule because you have to replace a liner too when leakage happens.

3) Insert for a capillary column inlet

You can install an insert as the following procedure.

1) Be careful not to contaminate the surface of an insert.
2) Remove an insert retainer nut.
3) If there is an already installed insert, remove the insert by using a tool like a pincette.
4) Confirm a new insert.
5) Position a graphite ferrule or a Viton O-ring at the insert so that 2 to 3mm extend pasts the other end of a mixing chamber.
6) Slide an insert into an inlet and push gently to put the insert into the inlet fully.
7) Tighten an insert retainer nut lightly by hand, and then tighten it with an inlet tool to prevent leakage.
   - When leakage happens, tighten it again with an inlet tool. (Too strong force may take off a nut.)

4mm ID, 78.5 x 6.3 mm OD

Fig 5-4. Glass insert for capillary column inlet

5-2-4. Installation of a packed column

1) 1/4 inch glass column

Install a well-fitted column (The distance between an inlet and a detector is 9.0 inch for 6500GC) because this column will not be bent. You can use a ferrule or an O-ring for a glass column. Use a ferrule for most analysis. If using an O-ring, use it for low temperature analysis. When packing a column or glass wool, space fully so that a syringe is not caught at an inlet. Pack it spacing about 40mm because a jet is located on the upside of a column in a detector. The method is described as followings.

1) Put a nut and a ferrule into a column. If you put an O-ring before putting a nut, it prevents the column from damage.
2) Slide the column into an inlet and a detector fully.
3) Take down about 1-2mm and tighten a column nut by hand.
4) Use a wrench to tighten the column nut just to prevent leakage.
5) Check leakage by using bubbles. If necessary, tighten it more.

2. 1/8 inch metal column

It is easy to install if using a proper fitting, liner, and insert because general metal column is flexible. Use two wrenches to prevent a liner itself from turning when tightening a column nut. Tighten the column nut with fixing one wrench at the liner itself and the other at the column nut.

5-2-5. Installation of a capillary column

Position a column hanger at the upper inside of an oven and then put a capillary column at the hanger. This column hanger is designed to be placed at two grooves located at the upper inside of an oven. These grooves are going to be located on the center of an oven when the hanger is settled. You can install a capillary column by using a column nut and a graphite ferrule. You had better to use different graphite ferrules according to inside diameter (I.D.)of a capillary column. It is recommended to use a 1.0mm(I.D.) ferrule for a 10.53mm(I.D.) column and to use a 0.5mm(I.D.) ferrule for a column less than 0.32mm(I.D.) Connect a column with a detector after connecting with an inlet to install the column.

1) Put a column nut and a ferrule into a column.
2) Cut the 1-2cm end of the column (a contaminated portion) with a column cutter after putting the nut and the ferrule.
3) Strip the end of the ferrule up to 0.4-0.6 mm and put a mark on the column under the column nut.
4) Slide the column into an inlet, and tighten it with a wrench after sliding the column up to the mark.
5) When you connect a column with a detector such as FID, TCD, and NPD, put a column nut and a ferrule into a column with the same method for an inlet.
6) Slide the column into a FID fully and pull down it about 2mm. Then, tighten it with a wrench.
7) Check leakage with “SNOOP”. If there is leakage, tighten it again.

5-2-6. Initialization of column

Since mostly a new column contains volatile pollution which is absorbed in the atmosphere, you have to eliminate these to obtain a stable base line. In the initialization of a column, operate the equipment with connecting the column to an inlet, but do not connect it to a detector. When doing this, close the detector with a cap or a plug.

1) For the case of a packed column

The flow rate of carrier gas is 30ml/min for a 1/8 inch column and 50ml/min for a 1/4 inch. Temperature of a column should be in maximum temperature for the column though it is maximum temperature for analysis or 30℃ higher than this. When finishing column conditioning, flow carrier gas until an oven cools. And then, start analysis after connecting a column with a detector and setting an analysis condition.

2) For the case of a capillary column

Be careful not to change temperature rapidly and raise it slowly to prevent stationary phase from leakage because a capillary column has very small stationary phase. Although flow is differ according to a column, generally the flow rate of carrier gas is 5-20ml/min for a 530μm (I.D.) column and 2.0ml/min for a 320μm (I.D.). Temperature of an oven should be lower than maximum temperature for a column though it is maximum temperature for analysis or 30℃ higher than this.

Chapter 6. Detector

6-1. Introduction

You can install two detectors in the YL6500 GC. Generally, a Flame Ionization Detector (FID) and a Thermal Conductivity Detector (TCD) are used. In this manual, we assume you have installed a FID at [Front] and a TCD at [Rear].

Besides, you can install a Nitrogen Phosphorous Detector (NPD) which has good sensitivity especially for nitrogen or phosphorous, an Electron Capture Detector (ECD) which captures high energy electrons (β-rays) produced by using a radioactive isotope (^{63}Ni), a Pulsed Discharge Detector (PDD), a Flame Photometric Detector (FPD), etc. according to analysis purposes. More details are explained in the supplement.

Generally, detectors are divided into universal detectors and selective detectors. If a detector can detect all components like a TCD, it is a universal detector. On the other hand, selective detectors are detectors which can detect only certain components according to use and the above all detectors except for a TCD belong to them.

Besides, synthetic detectors such as AED(Atomic Emission Detector), FT-IRD, MS(Mass Analyzer), etc. are used.

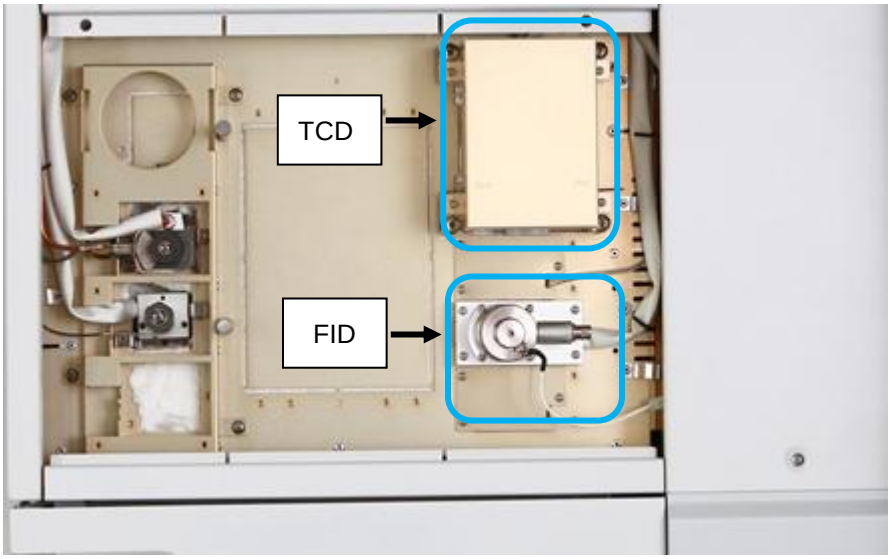


Fig 6-1. Top of GC equipped with FID/TCD

6-2. Flame ionization detector

A Flame Ionization Detector (FID) measures the quantity of ions which are produced when a sample coming out from a column is flamed by hydrogen and air. Carbon atoms of an organic compound (except for carbons of a carbonyl or carboxyl compound) produce CH radicals, and they can produce CHO^+ ions by hydrogen-air flame.

$$CH + O \rightarrow CHO^+ + e^-$$

Therefore, a FID detects signals in proportion to the numbers of carbon atoms. CHO^+ ions which are produced by flames carry current to an anode above the flame. Therefore, current which flow from a cathode under the flame to an anode are measured by a recorder. A FID can detect all organic compounds, but volatile compounds and gas such as O_2, CO_2, H_2O, NH_3, etc. can not be detected. A FID can not detect or rarely detects the following compounds.

Table 6-1. Materials which are not detected with a FID

Group	No detectable materials
Gases	Hydrogen, Nitrogen, Oxygen, Ammonia Carbon Dioxide, H_2O
Volatile compounds	Carbonyl sulfide, Carbon disulfide,
Halogen group	Silicon Halides, Cl_2,

6-2-1. Sensitivity

Detector's response to an organic compound is proportion to sample's weight and sensitivity is about 10^2 times higher than it of a TCD. Generally, you may use N_2 as carrier gas (You can also use He gas.). But when you use a capillary column, it is recommended to use H_2 or He gas as carrier gas. Also, you can have good detection result when using N_2 gas as support gas to increase flow velocity when a sample goes into a detector.

Sensitivity of a detector responses to the ratio of H and carrier gas(N_2) very sensitively. The ratio should be in 1:7~10. It is recommended to adjust ratio to obtain what has the best sensitivity because the best ratio may be different according to equipments. When you use a capillary column, flow rate of carrier gas is the sum of flow rate in the column and that of auxiliary gas.

6-2-2. Flow of fuel gas

In a FID, hydrogen and air are used to make flames. Generally, it is recommended to adjust the ratio to 1 : 7 or 1 : 10, flow rate of hydrogen to 30-40 ml/min, and that of air to 300-400 ml/min to make ignition

easily. Although flow rate of hydrogen is the fact which greatly influences ignition and sensitivity of a detector, that of air is less sensitive than that of hydrogen and so enough air to make ignition is OK. Flow rate of air is enough when it is about 7-10 times of that of hydrogen. For example, it is no matter that flow rate of air is a bit large when concentration of a sample is thick, but it is recommended that it of air is small when the concentration is thin. There are some cases not to be ignited well when flow rate of carrier gas is large. Then, increase flow rate of hydrogen a little.

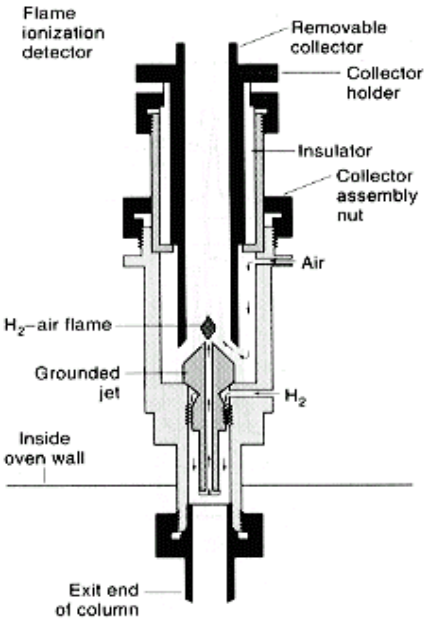


Fig 6-2. Structure of FID

Reason to make auxiliary gas flow

To make detection limit of a FID better, it is recommended to make flow rate of carrier gas about 20ml/min for a capillary column. But, since flow rate of a capillary column mostly less than about 10ml/min, it is recommended to flow auxilary gas to make flow rate more than at least 20ml/min. This auxiliary gas is used to prevent a sample's component which passes a column from diffusing at a detector. Such diffusing decreases accuracy of quantitative analysis beacause it induces peak broadening phenomenon. Therefore, auxiliary gas is used to prevent such phenomenon.

6-2-3. FID components

1) FID Jet

You have to replace a FID Jet according to the type of a used column (a packed column or a capillary column). If you are going to use a packed column after using a capillary column, you have to change a jet.

Table 6-2. FID Jets

Jet Tip's inside diameter	Used column
0.018 inch	A packed column
0.011 inch	A capillary column

A 0.011 inch jet shows the greatest sensitivity of a detector when a capillary column is used. When using a 0.018 inch jet for a packed column, you have to pay attention to use it because flames of a FID may be distinguished.

2) Collector

A collector is a place where ions which are ionized at a FID come together. It is located around the upper

of a jet and is made of metal. The ions which are gathered at this are carried to a FID board and produce signals.

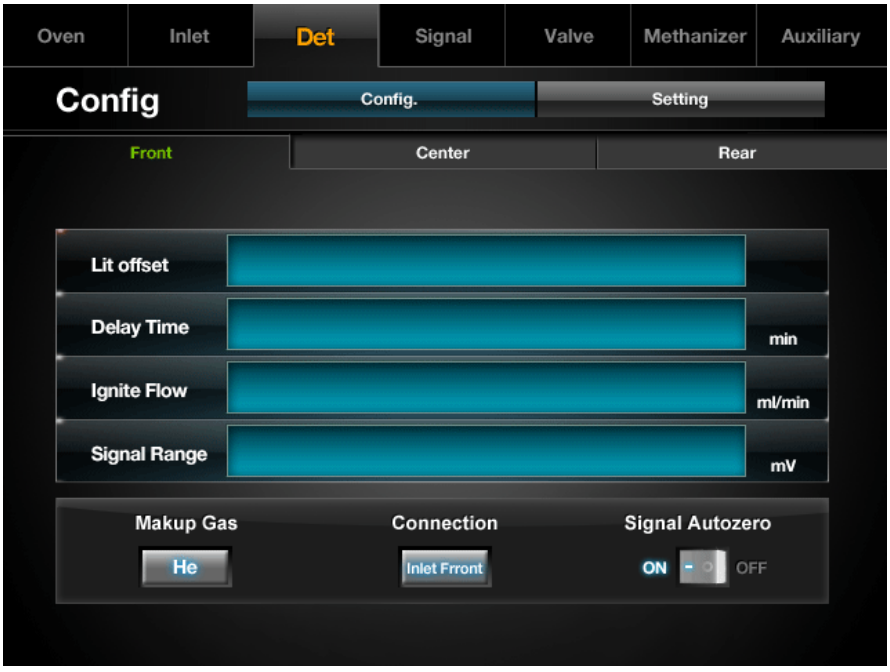
3) Ignitor

An ignitor is heating wires to ignite a FID. The end of this is connected with a power of electricity and the other end is grounded to the FID body. If current flow through the heating lines, the wires produce heat and ignite fuel gas.

6-2-4. Environment setting for the FID

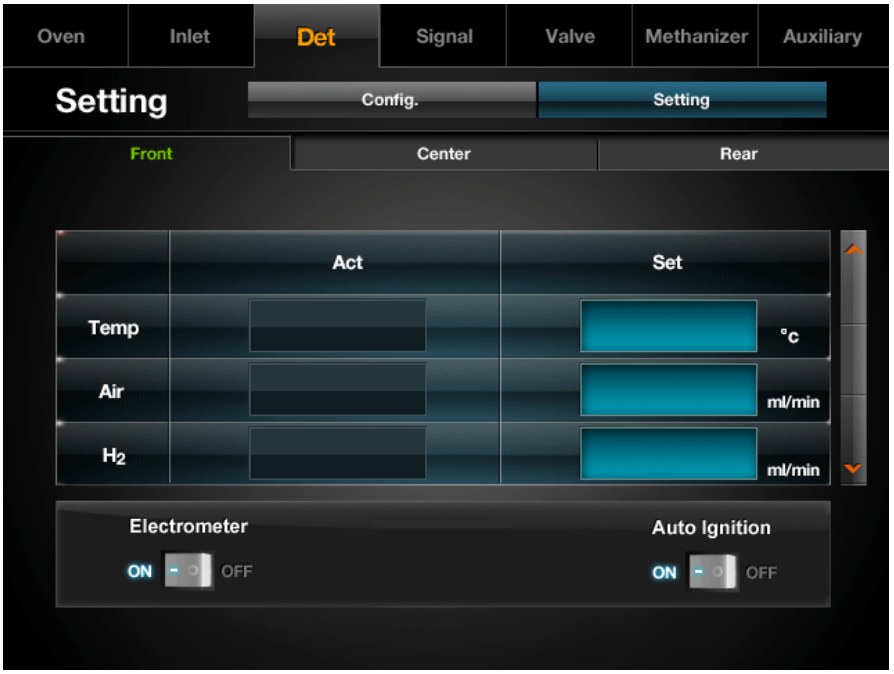
You have to make an environment setting of the detector before using a FID. If tapping on [Config.] tab of [Front] detector, you can see the following window.

The detail items are explained in the following table. To input a value, press [Enter] key after entering each parameter. If you want to change the sort of makeup gas, press [Makup Gas] tab until it displays a desired gas.



Item	Detail item	Explanation
Makup Gas	He H_2 N_2 Ar/CH_4	Select the kind of auxiliary gas. Generally, use the same kind of carrier gas as a make-up gas. If tapping [Makup Gas] tab, you can choose the kind of gas used for FID.
Lit offset	0.0~9999 (counts)	[Lit offset] is a standard value for acknowledging an ignition failure when “Auto ignition” function is set. That is to say, when signal values are under a set value, the ignition is repeated. (Default : 5)
Delay Time	0~999(min)	It is recommended to have delay time for raising temperature of outside before making ignition because moisture may be produced at a FID block when ignition is made before that of outside is not high enough. Although temperature of a FID is above 150℃, that of outside may not be so high. That is to say, after spending delay time after reaching 150℃, ignition starts by flowing auxiliary gas.
Ignite Flow		Set the flow rate for air when starting the ignition. Generally, set 100ml/min.

6-2-5. FID procedure for operating



Set necessary parameters on [Setting] of [Det] tab where FID is positioned. To input a value, tap on each tab, input parameters using numeric keys and press [Enter] then [On] key. If you want to change

parameters during the operation, just tap on a tab you want to change and input parameters using numeric keys then press [Enter] key.

Item	Item value	Explanation
Temp	0~ 450 (℃)	Set temperature of a detector. You have to set proper temperature because ignition is possible above 150 ˚C.
Electrometer	ON/OFF	When it is “ON”, a detector produces signals.
Auto Ignition	ON/OFF	You can turn on/off the auto ignition function.
Air Flow	0~500 (mL/min)	You can adjust flow rate of air among 0~ 500mL/min. Frequently used range is 300~400 mL/min.
H_2 Flow	0~100 (mL/min)	You can adjust flow rate of hydrogen among 0~100mL/min. Frequently used range is 20~40 mL/min.
Mkup(N_2)	0~100 (mL/min)	You can adjust flow rate of make-up gas among 0~100mL/min. Frequently used range is 20~40mL/min.

Auto ignition function : When you set auto ignition function at a FID, ignition starts automatically. Firstly, select [ON] at [Electrometer] and [Auto Ignition] after setting temperature of a FID. Then, air and hydrogen flow automatically and auxiliary gas is ready to flow. When temperature of a detector reaches a setting value, mixture of air and hydrogen flow and ignition start after [Ignition Delay Time] elapses.

If ignition is confirmed (Auto confirm), the ignition process stops and ready status for an analysis starts. If you want to confirm the ignition, just check the signal on [Signal] tab by pressing a down arrow key.

If you install a capillary column, flow make-up gas (MkUp gas) properly.

If ignition fails even with trials more than 15 times, you can see an error message which tells you to check the system and auto ignition stops.

6-3. Thermal conductivity detector

6-3-1. Introduction

A thermal conductivity detector (TCD) is included in a universal detector because it can detect all materials which have different thermal conductivity from that of carrier gas. Thermal conductivity is a measure how well a material carries heat according to temperature difference. The higher thermal conductivity, the faster heat moves.

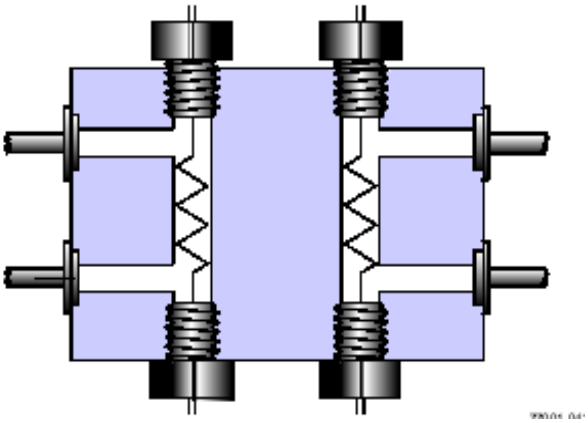


Fig 6-3. Structure of TCD cell

In a TCD, rhenium-tungsten filament is installed toward gas which come from a column. This filament produces a constant resistance when carrier gas flow at a constant velocity. But, temperature of the filament rise when thermal conductivity of gas is reduced. This is happened when a solute which has lower thermal conductivity than carrier gas comes from a column. The rising of temperature increase electric resistance of the filament. Therefore, an analysis is performed by measuring the resistance change of a filament in a path for a sample on the basis of resistance of a filament in a path only for carrier gas.

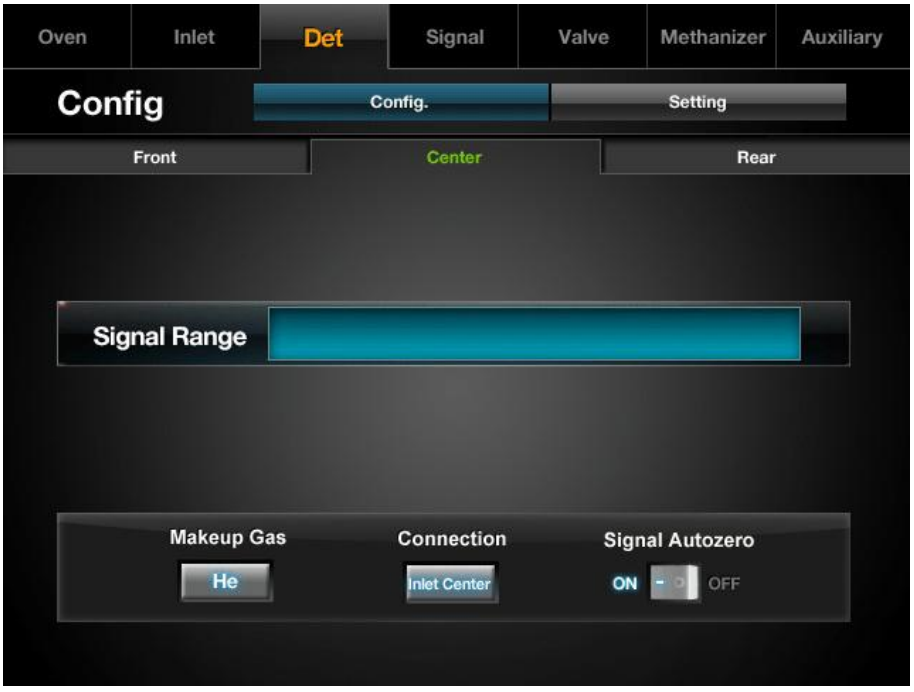
It is recommended that the difference of thermal conductivity between a sample component and carrier gas is big because response of a detector result from the change of thermal conductivity of flow. Generally, hydrogen (H_2) and helium (He) are used as carrier gas because their thermal conductivities are big. Although hydrogen have the best sensitivity because it has the largest thermal conductivity, you have to be careful to use it due to danger of explosion. Therefore, helium is used as carrier gas mostly. Although nitrogen and argon are used as carrier gas, they are used only for a special analysis because they are used in low sensitivity for avoiding damages of a filament and shows low response to most materials. You have to be careful to treat a filament of a TCD because it is apt to be damaged by air or oxygen.

6-3-2. Connection with a column

Two columns are used in a TCD. One is an analysis column and the other is a reference column. An analysis column is installed at the front side – the outside when opening the door of an oven of the YL6500 GC- and a reference column is installed at the back side. Although it is good to use just the same column with an analysis column as a reference column, normally an empty column is used for a reference column. When the GC is delivered, a 1/16 inch column is installed already. You can use the GC only with an analysis column. When you install a capillary column for a precise analysis, pay attention to eliminate an adapter which is installed on a TCD for Make-up. Refer to chapter 4 to know a method to install a column.

6-3-3. Environment setting for the TCD

Make an environment setting on the following window by tapping on [Config.] of [Det] tab where TCD is positioned.



Select the kind of make-up gas(Mkup/REF) by taping on [Makup] tap. Be sure to choose the same kind with carrier gas. Every time you press this tab, one of the available gases(He, N_2, Ar, H_2, 5%CH_4/Ar) is displayed.

6-3-4. Use for the TCD

Set necessary parameters on [Setting] of [Det] tab where TCD is positioned. To input a value, tap on each tab, input parameters using numeric keys and press [Enter] then [On] key. If you want to change parameters during the operation, just tap on a tab you want to change and input parameters using numeric keys then press [Enter] key.

1) Attention for TCD use

You have to flow carrier gas through an analysis column and a reference column before turning on a TCD by selecting [ON] at [Filament] tab. This treatment is for preventing a filament from oxidizing due to air. Therefore, you have to pay attention for [Ref. Flow] and [MkUp Gas Flow] on the above window and column flow from the inlet connected to TCD not to be “OFF”.

2) Temperature setting

You have to set enough temperature of a detector for a sample which comes from a column not to be condensed. But, a TCD has better sensitivity when temperature of the detector is low.

3) Flow rate setting

Ref Flow : Item for setting reference gas flow which is flow rate toward a TCD reference resistance.

MkUp Flow : You have to flow make-up gas for minimum flow rate because flow rate of carrier gas is mostly less than 10ml/min when using a capillary column.

You have to make each flow rate be more than 10ml/min at least. If flow rate is less than 10ml/min, the power of a filament is cut off automatically to protect a TCD cell. And you have to make each flow rate of carrier gas through a reference column and an analysis column be same to obtain stable signals when analyzing a sample. Therefore, refer to the flow rate value from the inlet connected to TCD.

4) TCD variable setting

Filament: If you select [ON] at [Filament] tap, an inner filament of TCD is powered, and then you can perform an analysis. Confirm whether carrier gas flows or not before selecting [ON]. That is to say, you have to pay attention for ref. gas flow, makeup gas flow (in case of capillary column) and column flow from the inlet connected to TCD not to be “OFF”. A filament can be turned on only in flow rate which is more than 10ml/min.

Sense: This adjusts current which is for a filament of a TCD. The control range is between 0 and 6. When this value is 6, sensitivity is the greatest. But, if it is 0(zero), you can not measure signals. The limited range of Sense value depends on the type of carrier gas you will use. (Ar: 3-4, N_2: 4-5, He: 5-6)

5) Polarity change

This function changes the polarity direction of a peak of a TCD. You can use this function when analyzing the component which has larger thermal conductivity than that of carrier gas. You can change the direction of a peak only when the above component emerges and return the direction to the orignal(+) again.

If [Polarity] is selected to [OFF], the polarity is always “+”. If you want to change polarity, select [ON] at [Polarity] tab.

If you want to change initial status of polarity, press [NEXT] arrow key on [Setting] tap and choose Initial State to “-” or “+”.

You can make Polarity change program on the above window.

For example, If you input 1.5 min in the first [Time] tap, 1min in the second [Time] tap and select initial state to “+”, polarity changes to negative(-) after 1.5min (Time1) then the it changes to positive(+) again after 1.0min(Time2). If a value of [Time] tab is 0.0, polarity will not change.

6-4. Electron Capture Detector

6-4-1. Introduction

The ECD detects a sample using a radioactive beta particle (electron) emitter. It is frequently used in analysis of pesticides and polychlorinated biphenyls. It also responds to peroxides, anhydrides, nitriles, nitrates, conjugated carbonyls, ozone and organometallics.

The emitted electrons, through repeated collisions with the carrier gas molecules, generate a plasma of positive ions, radicals and several low energy secondary electrons. The ECD essentially consists of two electrodes with the column effluent flowing between them. The emitted electrons, through repeated collisions with the carrier gas molecules, generate a plasma of positive ions, radicals and several low energy secondary electrons.

These thermal electrons are captured by molecules having high electron affinity to form negatively charged species. The reduction in the standing or background current due to removal of thermal electrons by electron capturing compounds, forms the basis of the detector response.

These processes can be schematized as below.

1) The radioactive isotope Ni63 → β particle (The electron with high energy)
2) β particle + carrier gas → With low energy
3) Cx (Halogenated compounds) + The outermost electron → Cx^-(anion) + Energy
4) The measurement of reduction in the standing current by electron capturing compounds

6-5. Detector Output Signal

On a [Signal] tab, It's able to set the zero value, the output signal range of detector signal and signal change function when there are more than 2 detectors to be used.

There are two terminals written as "Signal 1" and "Signal 2" at the back side of the equipment. Basically, these terminals carry signals of a detector as analog signals between 0 and 5 volt. You can display or process the signals by connecting a recorder or other proper devices. You can use many functions about test signals, blank signals, signal width adjustment, etc. for your convenience of signal treatment and signal management. You can set these functions by pressing【SIG】.

Here, if you make **"Value"** be "ON", signals which go to a terminal are shown up. And you can see the signal values.

If you see the back side of the equipment, you will see terminals which carry signals of a detector to the outer and they are labeled "DA 1", "DA 2" and "DA 3". Although normally signals of detector-1 (Front) are carried to the "DA 1" terminal, detector-2 (Center) are carried to the "DA 2" terminal, and detector-2 (Rear) are carried to the "DA 3" terminal, you can change the order by signal conversion function.

This function makes the "DA 1" terminal select signals of detector-1, detector-2 and detector-3 electronically. But it does not make component which is separated in column-1 enter detector-1, detector-2 and detector-3 selectively.

6-5-1. Range

The output of signals (5V) is in proportion to concentration of a sample. Actually, if a concentration of a sample is high, the equipment weaken signals accepted from a detector. Otherwise, it strengthen the signals for analysis. You can use this function by "Range". To use this function, press Config. Tab on [Signal] and put the value in "Range". The Minimum value of range is 0 and the maximum is 10. If the range value increases 1 unit, the output becomes 1/2 of the former. That is to say, area of peak on a chromatogram decreases by 1/2, if the range value increases by 1 unit. Therefore, if you will calculate quantities of each component by comparing areas of a sample and reference materials, you have to set

each range to be same.

6-5-2. Zero

If there are no components to be analyzed, principally background has to be zero because output signals are not detected. But, in effect, the background is more than zero due to contamination of column or carrier gas and especially deflection of a detector itself, etc. To correct this error, you can use “zero” function. Press Config. Tab on [Signal] and enter the value that you want to subtract from signals of a detector.

6-5-3. Signal Change

If you want to change the output of signals, tap on [Setting] at [Signal] tab then you can see the following window. You can select a detector position which accepts signals initially and input signal conversion time.

If you input a value in the first [Time] tab, a detector in parentheses will be changed to other detector position which is different from an initial detector. For example, signals come from a TCD, but they come from a FID after 4.5 minutes. If you again input a value in the second [Time] tab, the detector in parentheses will be changed to the initial detector (TCD). In this way, you can make signal conversion of a detector up to 5 steps.

If you want to remove a signal conversion time which is inputted already, just input 0.0min to [Time] tab.

For example, if you want to input again from the beginning, just input 0.0 in the first [Time] tab.

You cannot change the time while program is running.

Chapter 7. Gas sampling valve

7-1. Introduction

You can inject gas sample into an inlet directly with a syringe in Gas Chromatography. But, this method has not good reproducibility. Therefore, you need an extra injection device. You can use a gas sampling valve to inject gas sample. The device is installed at the fore part of a column. The maximum valve number is 4 in the YL 6500 GC, and the program is possible up to 30.

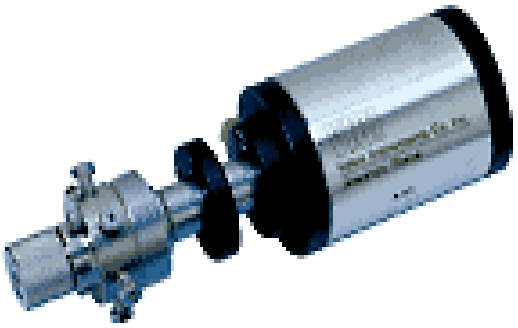


Fig 7-1. Gas Sampling Valve

7-2. Valve types

Injected gas sample is contained in a sampling loop and then, it is carried by carrier gas into an analysis column to be analyzed. You need a valve which can change the path of gas variously for the process such as containing a sample in a loop, carrying it through carrier gas, etc.. Generally, 6-ports or 10-ports valves are frequently used as a valve which treats gas sample. Although the connecting method is various according to the purpose, basically a valve for a capillary column is installed when GC is delivered.

When you treat the gas which is condensed at room temperature due to its high boiling point, you have to raise temperature of a valve before using. Since maximum temperature and pressure are different according to valves, you have to properly choose one according to samples. There are some methods such as a manual method, an air actuated, an electric actuated, and a micro-electric actuated method to operate valves. Here, explanation is done on the basis of an air actuated method.

2-position air actuator is attached at the top to open or close a valve for gas sample. An air actuator is operated by air or nitrogen and the proper pressure is 40-80 psi.
Basically, the capacity of an installed sample loop for injecting a sample is 250μL. Therefore, if you want others having different capacity, you have to order them.

7-3. Environment setting for a valve

If tapping on [Config.] at [Valve] tab, you can see the window for environment setting for a valve.

You can choose either [LSV] (Liquid sampling valve) or [GSV] (Gas sampling valve) by tapping on [Type] icon.

Enter the number of port and the volume of sample loop size, and select the inlet position where valve is connected by tapping on [Conn.] tab.

7-4. Operating a valve

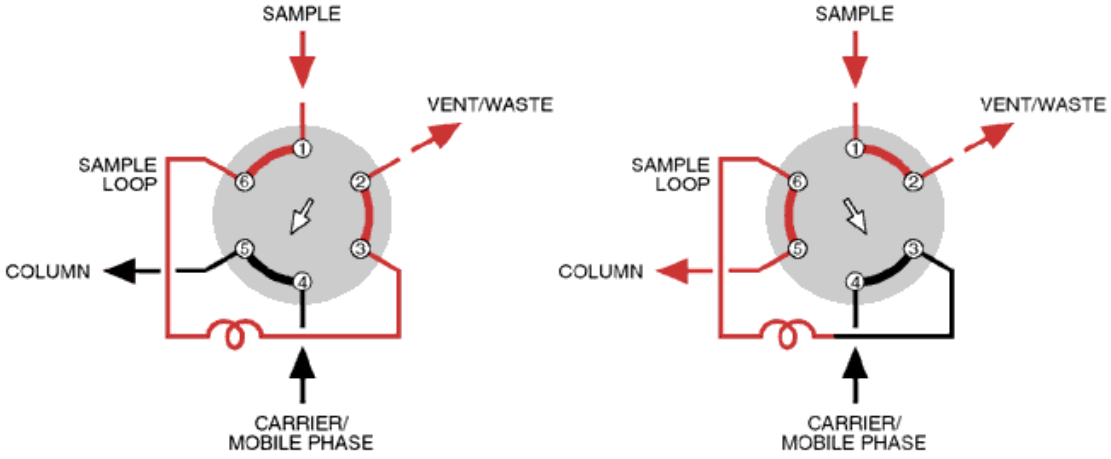
7-4-1. Setting temperature

The YL6500 GC has three auxiliary heater. You can control the auxiliary heater at [Auxiliary] tab and also adjust temperature of a valve at here. Since the auxiliary heater block can contain normally up to two gas sampling valves, select numbers which are suitable among the auxiliary controllers AUX1, AUX2, and AUX3 according to the number of the used valves and temperature which will be set.

7-4-2. Valve program

You have to set a valve program in order to operate valves. If tapping on [Setting] at [Valve] tap, you can select the initial position [On or Off] and time events for each valve. The available event steps are total 30 steps.

(The time events are based on “RUN” time.)



You can see the procedure how a sample is loaded on a sample loop using a 6-ports valve and carrier gas carry the sample at the above figure.

Chapter 8. GC operating procedure

We have explained the basic principle and the operating method of the equipment for each part in the above chapters. But we will explain the operation procedure synthetically to operate the equipment actually in this chapter. You have to check prerequisites and necessary parts for operating the equipment well. After this, you can operate the equipment easily by knowing the following checklist and simple procedure for operating it.

8-1. Check list before operating

8-1-1. Check regular voltage

The YL6500 GC needs stable power source. Therefore, it has an auto voltage regulator. Moreover, if you use an extra power device for an electricity failure, you can prevent experimental results from vanishing due to the power failure. Since the maximum spending power is 3.5 kW, it is recommended to use the auto voltage controller whose output voltage is 220V and maximum power is 5kW.

8-1-2. Connecting a power cable, a signal, and a remote line

Confirm that the power cable is connected correctly and GC is connected with the data system rightly. Refer to chapter 2 (Installation) for method connecting the cables.

8-1-3. Preparing gas

Select proper carrier gas used for GC according to the type of an used detector and its purity has to be high (more than 99.995%). If necessary, install traps for eliminating moisture or oxygen and a refiner additionally. Also, prepare extra auxiliary gases. (Refer to chapter 2.)

8-1-4. Preparing a column

When you analyze a sample in gas chromatography, the most important part is an analyzing column. The columns differ according to components which is analyzed. You can choose the column by using a reference or an experiment. After selecting a column, install a column and take an initialization step before using it. Refer to chapter. 5 for the column initialization.

8-2. Operation flow

1. Confirm a check list before operating the equipment.
2. Install a column which is proper for an analysis purpose to the equipment.
3. Flow gases needed for an analysis.

   Carrier gas(N_2 or He): 50-70psi

   Auxiliary gas(Air or H_2): 50-70psi
4. Switch on the power of the YL6500 GC.
5. Set the dimension of an installed column and the kind of carrier gas.

   ※ GC Control Screen -> Inlet(Front/Center/Rear) -> Config

   · Set the column specification : Enter the length, inside diameter and the film thickness of the column

   · Select a carrier gas at “Carrier Gas”.

   · Select the flow control modes : Constant flow/pressure, Programmed flow/pressure
6. Input the pressure or flow rate of a column.

   ※ GC Control Screen -> Inlet(Front/Center/Rear) -> Setting

   · Set a pressure or flow rate of column :

   - Enter a flow rate(ml/min) in a constant flow
   - Enter a pressure(psi) in a constant pressure.
   - Split/Splitless mode setting : Select either split or splitless mode. Enter a split ratio in a split mode. Enter a split on time in a splitless mode.
7. Input a temperature of each heated area such as an inlet, a detector and an oven.

   ※GC Control screen → Inlet/Detector/Oven -> Setting

   If you want to use a TCD, enter a temperature only after a checking column flow rate and a reference cell flow rate are on to a TCD.
8. Input a flow rate and necessary factors of a detector

   FID: Autoignition, Electrometer ON

   TCD: Set a flow rate of REF/Mkup gas – Filament ON
9. Stabilize a column.

   Check the signals to be stabilized after it reaches to the setting a temperature.
10. Run a chromatography data system to display the acquisition window.
11. Enter acquisition conditions to the CDS(Autochro 2/ AutoChro3000 / YL-Clarity).
12. Press【START】button right after injecting a sample after confirming the baseline to be stabilized.

8-3. Operating method of the equipment

8-3-1. FID analysis

1. Confirm the check list before operating the equipment.
2. Install a column which is proper for an analysis purpose at the equipment.
3. Open the cylinder of carrier gas, hydrogen and compressed air, which are needed for an analysis. Set the pressure of a gauge at 60psi.
   Carrier gas (N_2, He): 60psi
   Auxiliary gas (Air): 60psi
   Auxiliary gas (H_2): 40psi
4. Switch on the power of the YL6500 GC
5. Enter the length and inside diameter of a capillary column and select the detector which is connected to the column.
6. Enter the type of carrier gas.
7. Input the pressure or flow rate of a column.
8. Enter a temperature of inlet, detector and oven. The detector temperature should be more than 150℃ to be ignited.
9. Initialize and stabilize a column
10. Turn on the electrometer and the auto ignition of a FID, and set make-up gas (Mkup gas) when installing a capillary column.
11. Check signals after confirming the ignition of the system.
12. Run a chromatography data system to display the acquisition window.
13. Enter acquisition conditions to the CDS
14. Press【START】button right after injecting a sample

8-3-2. TCD analysis

1. Confirm the check list before operating the equipment.
2. Install a column which is proper for an analysis purpose at the equipment.
3. Open the cylinder of carrier gas He (Or N_2/Ar) and set the pressure of a gauge between 50 and 70psi. If you analyze hydrogen, use Ar (or N_2) as a carrier gas.
4. Switch on the power of YL6500 GC.
5. If you use a capillary column, input the length and inside diameter of the column and select a detector position which is connected with it.
6. Enter the type of carrier gas.
7. Input the pressure or flow rate of a column.
8. Set a reference gas (REF gas) of a detector. And if you use a capillary column, set the flow rate of make-up gas (Mkup gas).
9. Enter the temperature of an inlet, a detector and an oven. And then, press “Filament” [ON] after stabilizing a flow rate and a temperature.
10. Stabilize the column.
11. Set the value of TCD SENSE.
12. Run a chromatography data system to display the acquisition window.
13. Enter acquisition conditions to the CDS
14. Press【START】button right after injecting a sample

8-3-3.ECD analysis

1. Confirm the check list before operating the equipment.
2. Install a column which is proper for an analysis purpose at the equipment.
3. Open the cylinder of carrier gas, N_2 and set the pressure of a gauge between 50 and 70psi.
4. Switch on the power of YL6500 GC.
5. If you use a capillary column, input the length and inside diameter of the column and select a detector position which is connected with it.
6. Enter the type of carrier gas.
7. Input the pressure or flow rate of a column.
8. Set a makeup gas (Mkup gas) of a detector and a flow rate.
9. Enter the temperature of an inlet, a detector and an oven. And then, press “Electrometer” [ON] after stabilizing a flow rate and a temperature.
10. Stabilize the column.
11. Run a chromatography data system to display the acquisition window.

12. Enter acquisition conditions to the CDS.
13. Press【START】button right after injecting a sample

8-3-4. Finishing operation after an analysis.

It's required to eliminate impurities remained in a column after finishing an analysis. Therefore, perform "Post run" process by increasing a temperature of oven to clean out the column.

After all process, turn off the instrument by the following procedure.

1. Turn off a detector.
   FID : Autoignition OFF
   TCD : Filament OFF
2. Set a temperature of an oven to be 50 ℃.
3. Make the temperature of an inlet, a detector, a valve, etc. be off.
4. Once the temperature of oven reaches to 50 ℃, turn off the flow of each inlet.
5. Make the flow of a detector be off.
   ECD : Make up gas OFF
   TCD: (After temperature is below 60 ℃.) REF , Mkup gas OFF
6. Separate the column from the detector after temperature of an oven is below 50 ℃ and that of a detector is below 150 ℃.
7. Turn off power.
8. Close the valve of a gas cylinder.

Chapter 9. Maintenance

When using the equipment, sometimes you are faced with the need to maintain and repair it. This chapter describes maintenance procedures and requirements of YL6500 GC.

9-1. Introduction

9-1-1. Maintenance list

Maintenance lists with time to replace are summarized in the following table.

Table 9-1. Maintenance lists with time to replace			Time to replace
Oven and heated zones	Confirming the connection part of a column		If necessary
	Column conditioning due to peak tailing		If necessary
	New column conditioning		Whenever a column installation
	Replacing an oven heater sensor		If necessary
	Replacing a heater sensor of an inlet and a detector		If necessary
Inlet	Gas trap for mobile phase		Every 2 months
	Packed inlet	Confirming a leakage of mobile phase gas	If necessary
		Confirming a leakage of an inlet	If necessary
		Septum	Every 30~50 uses and if necessary
		Cleaning an inlet and a liner	If necessary
	Capillary inlet	Confirming a leakage of mobile phase gas	If necessary
		Confirming a leakage of an inlet	If necessary
		Septum	Every 30~50 uses and if necessary
		Cleaning an inlet	If necessary
		Cleaning an insert and packing it	Whenever noticing a leakage
		Replacing an insert O-ring	Whenever noticing a leakage
		Vent line conditioning	Whenever noticing a leakage
		Replacing a column seal	Whenever noticing damage
Detector	FID	Confirming a leakage	If necessary
		Cleaning	If necessary
		Replacing a seal	If necessary
		Cleaning a collector	Whenever signals are high or if necessary
	TCD	Confirming leakage	If necessary
		Cleaning	If necessary
	NPD	Confirming leakage	If necessary
		Cleaning	If necessary
		Replacing a seal	If necessary
		Position correction of a collector	Whenever replaced and if necessary
	ECD	Confirming leakage	If necessary
		Cleaning	If necessary
APC		Replacing a split trap	Every 6 months and if necessary
		Replacing a block	Whenever facing troubles
		valve/flow calibration	If necessary

9-1-2. Basic maintenance

Since the YL6500 GC controls temperature of an oven, an inlet, and a detector by electricity, be careful to manage the heating and electric parts.

1. Do not touch an inlet when the equipment is running or temperature of it is high.
   If necessary, touch it carefully with gloves.
2. In operation, hot wind comes from the back side of the equipment. Therefore, keep enough space from the wall when istalling it. If there are plastic or rubber materials in operation, they may be damaged.
3. If you have to open the back cover, do it after turning off all sources of electricity. You can open it only with a cross-shaped driver. You can remove the cover of the equipment and the left, right, and back cover with the cross-shaped driver. Also, if the connected tubes such as a tube for mobile phase gas, that for air, and that for hydrogen are eliminated already, you can easily remove parts of the flow controller located on the left side of the equipment only with the cross-shaped driver.
4. If necessary, clean the surface of the equipment using warm water or a detergent with smooth clothes. Be careful not to flow solvent like water into the equipment.
5. After operating, it is recommended to take a post-treatment always not to be the remnants of a sample in a column. (Performing【Post run】)

9-1-3. Trap maintenance

A trap for eliminating impurities of carrier gas is connected from a carrier gas cylinder to the equipment. Generally, a moisture trap is connected to the exit of the carrier gas cylinder and an oxygen trap is connected from the equipment to the moisture trap.

1) Moisture Trap

A S-shaped moisture trap is used to eliminate a trace level of moisture which is in carrier gas. The moisture is harmful to the mobile phase of a column when the equipment is operating in high temperature and induce the deterioration of a detector like an ECD. This moisture trap is generally packed with “Molecular Sieve 5A”. If carrier gas contain very big contamination which is difficult to be absorbed on “Molecular Sieve 5A”, you can use “Molecular Sieve 13X” or “Activated Charcol” as an absorber.

This moisture trap can be used again by conditioning in an oven. For conditioning, connect the moisture trap to an inlet after closing the exit of a detector in the same method for column conditioning. Then, switch on the oven at 350℃ for at least 3 hours with flowing N_2 or He at 60ml/min. If you want to pack the trap again, clean it after removing the old packing materials it with new “Molecular Sieve 5A”.

2) Oxygen Trap

An oxygen trap is used to eliminate a little oxygen which is in carrier gas. The oxygen harm a capillary column. Moreover, when using a high sensitive detector like an ECD, you have to use the trap because oxygen is a hindrance in the ECD. But this trap can not be used again after conditioning and have to be replaced.

9-2. Inlet

This is to maintain a carrier gas system connected to the back of the equipment, an inlet for a packed column (a packed inlet), and that for a capillary column (a capillary inlet).

9-2-1. Packed column inlet

1. Leakage check for carrier gas

Leakage check for carrier gas is done with the exception of connecting parts of a column. This is because you can check the connecting parts using “SNOOP”. If you can not find any leakage in this check procedure, check the leakage of a detector.

ⓐ Turn off all detectors.

ⓑ Make all parts related to temperature be at room temperature and turn off them.

ⓒ Do not open a gas cylinder.

ⓓ Remove a column and block an inlet with "CAP".

ⓔ Open the cylinder and adjust the second gauge pressure to be 50psi.

ⓕ Set to constant pressure mode.

ⓖ Set the volume of total flow

ⓗ Wait about 10 minutes after the volume of total flow decreases up to zero. Check the pressure gauge. If pressure does not drop more than 1~2 psi, it means there is no leakage of an inlet.

ⓘ If you find any leakage, check the leakage part by examining all connecting parts with "SNOOP".

2. Leakage check for an inlet

The possible leakage parts of the inlet are the septum and the column connection part. The leakage of the column connection part is easily examined with "SNOOP". Also, you can inspect the leakage of the septum by replacing it to a new one.

3. Replacing a septum

Generally, you have to replace a septum every 30~50 injections. Especially, if you use it at 250℃above, you have to change it more frequently. When replacing, close the carrier gas cylinder. To replace a septum, input a septum to "Septum Retainer Nut" and tighten it a little by hand. And then, raise a temperature and tighten it again.

If the used septum is coated with Teflon, install it with making the coated surface be toward downside of an inlet.

4. Cleaning an inlet

Remove a column and a liner after eliminating the septum retainer nut and the septa while the inlet temperature is to be room temperature. The liner contains a glass insert. If the glass insert is contaminated, wash it with chromic acid first and then in order of distilled water, methanol and acetone. After that, dry it up fully at 150℃.

Clean the liner which is removed from the inlet with 0.1% nitric acid and wash it with water. And then, make ultrasonic cleaning with methanol.

9-2-2. Capillary column inlet

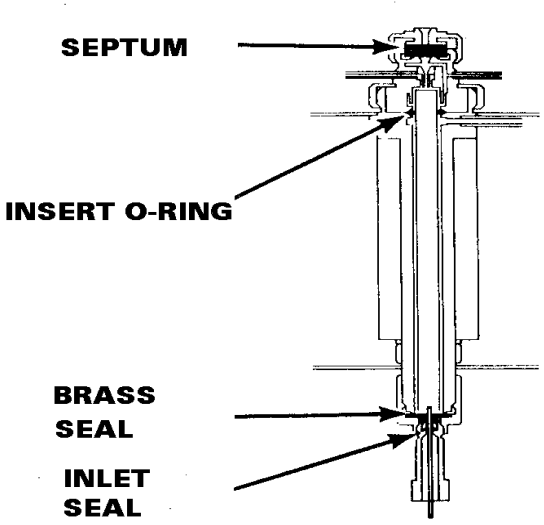


Fig. 9-1. Expected leaking areas of inlet

1. Leakage Check for carrier gas

If you want to check the leakage of carrier gas, it is needed to find whether the leakage happens at “APC” or at the inside of an inlet. If you can not find any leakage at the two parts, check the leakage of a detector.

ⓐ Turn off all detectors.

ⓑ Make all parts related to temperature be at room temperature and turn off them.

ⓒ Remove a column and block an inlet with “CAP”

ⓓ Set the column dimension to 0, 0.

ⓔ Set to the constant pressure mode.

ⓕ Set the pressure to 20 psi and the split ratio to 1.0.

ⓖ Make sure that the pressure and the total flow are stabilized enough and turn them off. And then, immediately block the purge vent and the split vent which are located on the upside of the APC block with a plug.

ⓗ Check whether there is a drop in pressure or not for about 5 minutes.

ⓘ If the pressure is in the range of 19 ~ 20 psi, it means that there is no leakage.

ⓙ If the pressure increase, it means that the leakage is from a total valve. But, if the pressure decrease below 19 psi, it means that the leakage is from an inlet. If you find any leakage of the inlet, check all connecting parts with "SNOOP".

2. Leakage check for an inlet

If you find the leakage of an inlet, you can expect 4 area where the leakage are from : a septum, a column connection part, an O-ring, and a brass seal.

1. You can examine the leakage of a septum and a column connection part with "SNOOP".
2. If you can not find any leakage at them, replace an O-ring of an insert.
3. Check the leakage again. If the pressure drops down more than 1 psi for 5 minutes, it means there is a the leakage and you have to replace a brass seal.

Fig. 9-2. brass seal

3. Replacing a septum of an inlet

To replace a septum, decrease the pressure first, and then, remove “Septum Retaining Nut" and the old septum. Tighten the new septum by hand to make “Snap-Ring” of “Septum Retaining Nut” be up about 1mm above the nut.

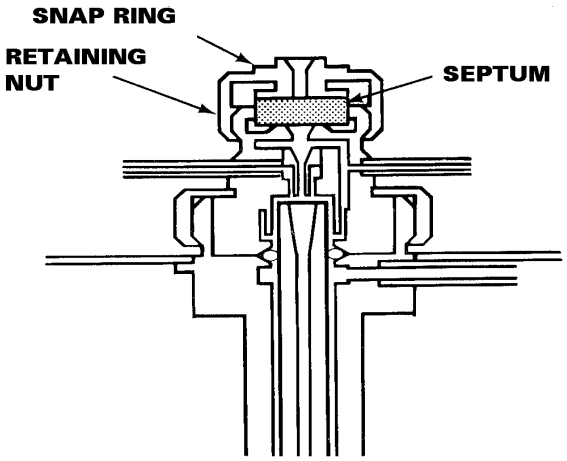


Fig. 9-3. Septum of capillary inlet

4. Cleaning an inlet

A capillary inlet is designed to slide an insert into it. Although you do not have to clean the inside of the inlet fully, it is very important to clean the insert of the inlet. Therefore, you have to clean or replace the insert at least every 3 months. If necessary, clean up the inlet. You have to disconnect carrier gas connection tube which is connected already and remove the insert which is installed at “Insert Retaining Assembly” to clean up the inlet. Then, clean the inlet with a proper brush. If necessary, wash the inlet with

neutral detergents or methanol. After cleaning, dry up the inside of the inlet fully by flowing carrier gas with connecting carrier gas connection tube.

5. Cleaning a glass insert

The glass insert has to be clean without absorption of any contaminants like particles. If this is contaminated, the performance of the entire inlet is reduced. That is to say, problems such as a peak tailing, unstableness of baseline, etc. are induced. To clean the insert, remove the inlet assembly and disconnect an O-ring with the insert. If this insert is "Split Type", remove the packed materials. Since the contaminants are carbon compounds which will not dissolve in organic solvent, clean the insert with chromic acid firstly. And wash it with distilled water, methanol, and acetone. Then, dry up it fully at 150℃ and install it.

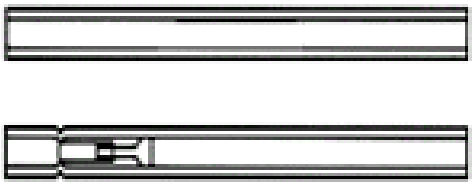


Fig. 9-4 glass insert

If you want to pack the insert again, put the treated glass wool in the nozzle part inside the insert and pack “10% OV-1 on 80/100 mesh Chromosorb WHP” in it. And then, put the glass wool in it again. Since about 2 inch syringe has to be used, keep about 35mm space when packing.

Check the O-ring before puting the insert in the inlet. If there is no wrong, install an O-ring at the 5 mm upside of the insert and input the insert in the inlet. And then, slide it as down as possible.

9-3. Oven

The oven is a device which controls temperature of a column. (Refer to chapter 5.) If the oven gets organic materials or dirts in it, there will be soots or dirty marks since its temperature is very high. Therefore, when you do not use it, clean it using a dried rag with methanol, aceton, etc.. You can replace heaters and sensors in the heating parts including the oven whenever necessary.(You had better ask the service department to replace them.

9-3-1. Columm condition check and maintenance

1. Check column connection parts

Most gas leakage happen at column connection parts. This is mainly due to heat change. You can check the leakage with "SNOOP". When you check the leakage, the examining parts have to be at room temperature. If the temperature is high, you can not confirm the leakage because “SNOOP” can not form bubbles.

2. Peak tailing of a capillary column

You can confirm the status of a column using peak tailing phenomonon. For this, the used column should be apt for the sample analysis. Since you can get “Test Sheet” in the column box when buying a capillary column, you have to check it. And if possible, it is better to try with the test component. If the peak of a sample componet shows peak tailing though that of the test component is OK, you can conclude that the column does not fit well with the component of the sample. You can confirm the peak tailing by magnifying the peak when collecting data with the Autochro2/Autochro 3000/YL-Clarity.

Except for the column, you have to check a glass insert. This is because small septum fragments which are produced in the glass insert by repeating of injection can cause the peak tailing. If the peak tailing still apears even after the above examinations, you have to clean a detector because it means the inlet has no problem.

3. Column conditioning

Contamination of a column is mainly induced by remants which is not volatilized. Even a new column has volatile pollutants which is absorbed from air. Especially, the column which is leaved alone for a long time without colsing the end of it, is sure to have pollutants. Therefore, you have to make column conditioning before using it.

Since colunm conditioning differs according to the kinds of columns, refer to data obtained when buying a column. Column conditioning means the thermal conditioning which is happened while carrier gas is flowing through the column. You can make the column conditioning by the following procedure.

Select “COLUMN CONDITION” among items after pressing【SPECIAL】in the GC control screen.

Do not connect a column with a detector before column conditioning and close the input of the detector with a cap to prevent it from contamination.

For most columns, Set initial temperature to 50℃ and initial time to 30min. And then, Set the maximum temperature depending the kinds of the columns.

Do not raise temperature of an oven more than the maximum tempearture of the column. This is because such action decreases the life time of the column and damages the column rapidly. Especially, if using a capillary column, you must not raise the temperature rapidly and have to use it under the maximum temperature of column. Set “Fin Temp” under the maximum temperature of the column and raise the temperature at about 3℃/min. slowly for column conditioning.

9-4. Detector

9-4-1. Flame ionization detector (FID)

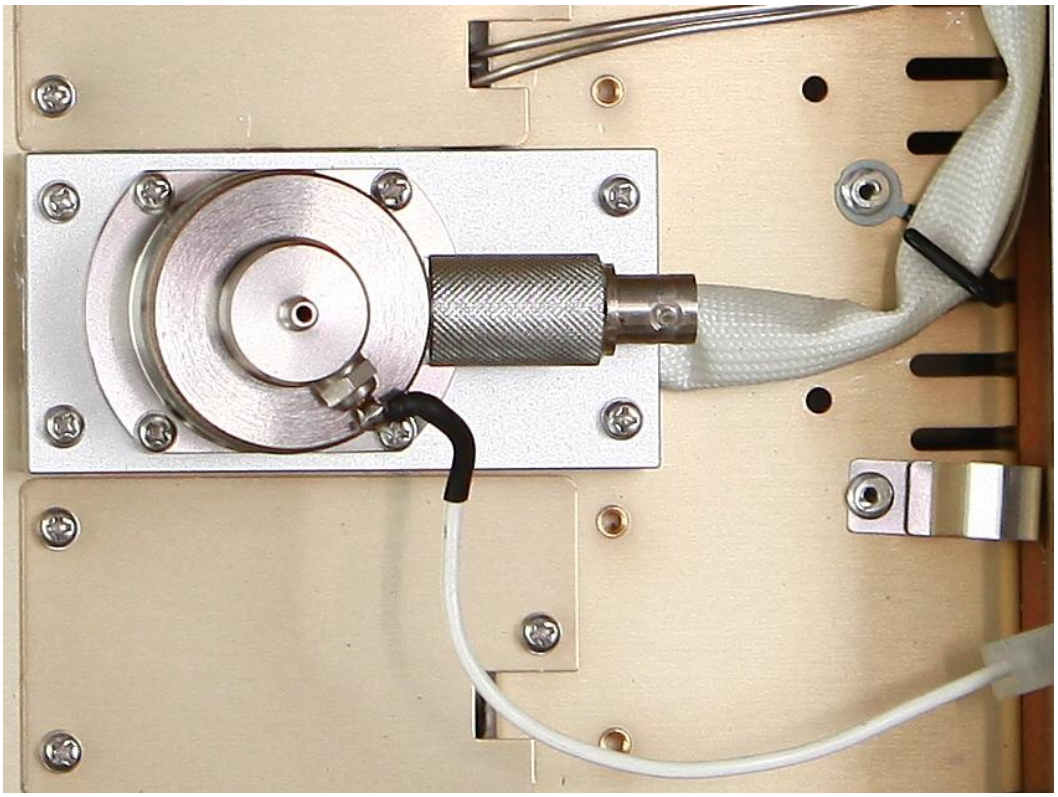


Fig 9-5. Picture of the installed FID

1. Replacing a jet

If using a FID, you have to change a jet depending on the kind of the used column (a packed column and a capillary column)

Table 9-2. Type and explanation of a jet

Jet type (Inside diameter of a nozzle)	Explanation
0.011 inch (0.28 mm)	For a capillary inlet.
0.018 inch (0.45 mm)	For a packed inlet.
1. You have to install a jet before installing a column. If you replace a column, you have to change a jet before installing the column. 2. It's essential to use a 0.011 in. jet for a capillary column. If you use it for a packed column, the ignited flame may be distinguished.	

2. Cleaning

A collector and a jet may be contaminated by white silica or black carbon materials from a column. These contaminators decrease the sensitivity and induce “Noise” and “Spike” phenomena. Therefore, you have to clean them to remove these materials.

Turn off the detector and adjust the temperature to be room temperature. Close all cylinders for gases used for the detector. Open the upper-cover of the equipment.

You can clean them as the following procedure.

ⓐ Switch off the equipment and disconnect the power cable from the electric outlet.

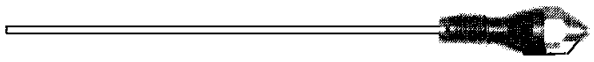


Fig 9-6. FID Jet

ⓑ Remove a signal connector from a FID body. .

ⓒ Disconnect the connector which is connected with the equipment and the ignitor.

ⓓ Loosen the collector nut by hand.

ⓔ Check the gasket whether it is damaged or not by inspecting the bottom of the collectror mount after removing 4 screws holding it.

ⓕ Disconnect the collector body, the upper collector insulator, and the low collector insulator which are installed in the collectror mount.

ⓖ Spin counterclockwise the jet installed in the FID body with a jet driver and pull out it with a pincette.

ⓗ Clean the upside of the FID body with solvent, compressed air, or nitrogen.

ⓘ Check the damage of the jet and replace it with a new jet which is proper for the column. Then, tighten it with a jet driver.

※ Reference ※

Do not use any tool except a driver.
If tightening it heavily, the jet and the FID base may be damaged. Tighten it by hand with a driver and spin it about 1/4 turn again.

ⓙ Assemble the collector. The high collector insulator which the bottom part is long is located under the collector body. The low collector insulator is flat and a round shape and is located above the collector.

ⓚ Put the assembled collector assembly in the collector mount.

ⓛ Install the collector mount on the FID base and fix it with 4 screws.

ⓜ Tighten the collector nut by hand and connect the ignitor connector.

ⓝ Spin a screw hold the FID board and make up the right side panel.

Fig 9-7. Disassembled FID collector

ⓞ Run the equipment and check the signal without ignition whether it is “0” or not. If the signal is "1,000,000", it means that the detector and the board have short circuit. Therefore, install the board again and check the signal.

※ Reference ※

If the signal shows “1,000,000”, generally there is water which is produced by air and hydrogen in the collector. Therefore, remove water. This is because air and hydrogen flow in the low temperature of the detector. Open the cylinders of air and hydrogen always when the temperature of the detector is more than 150℃, and ignite then.

3. Operating an ignitor

If the ignition of a detector is not stable, you can hardly see the red flames at the exit of the detector. For this case, you have to check the ignitor or the related circuit. If “Ignition failure” happens more than 3 times when you operate the auto ignition function, you can think that the ignitor may be cut.

Firstly, measure the resistance between the terminal of the middle cable of the ignitor and the case with the DVM. If the measured resistance is infinity, replace the ignitor because it is cut.

You can replace the ignitor as the following procedure.

ⓐ Turn off the detector with closing the cylinder of the gas for the detecor. But, turn on the equipment.

ⓑ Disconnect the connector connected with the equipment and the FID ignitor. If you want to disconnect it, squeeze the narrow part of the connector so that the part hanged on the other side connector is loosed.

ⓒ loose the ignitor with a 5/16” spanner.

ⓓ Clean the part for installing which is loosed to install a new ignitor.

ⓔ Put the new ignitor in the part for installing. When putting it, be careful for the copper washer not to fall out.

ⓕ Make sure that it is installed tightly and connect it with the connector.

4. Ignition trouble

If the following facts are observed, you can conclude that the jet is damaged. The facts are that the flow rate of the detector is correct, that there is no gas leakage, that the flow rate of carrier gas is right, that the ignitor runs well during ignition, and that the detector is not contaminated.

※ Reference ※

If you have ignition failure when using He as carrier gas, it is because the flow rate of carrier gas which is added with AUX gas. In this case, decrease the flow rate of carrier gas which is added with AUX gas as low as possible. And if the flow rate of carrier gas is high, increase the flow rate of hydrogen a little.

If you can not find any outside damage of jet, the damage is the jet’s inside break which is produeced by tigthening it too tightly. Therfore, you have to replace it. If ignition failures keep on happening in spite of changing the jet, the problem is due to the detector’s inside damage which is produced by tightening the jet heavily. In this case, you have to replace the detector.

9-4-2. Thermal Conductivity Detector (TCD)

When you use a TCD, it is very important to protect the filament which is made of tungsten-rhenium. Be sure to keep it from oxygen. And since the sensitivity is high when temperature is low, you have to use it under low temperature.

When using the TCD, you may fine the movement of a baseline, the incrase of noises, the unexpected peaks, etc. This phenomenon may be caused by a sample or the packed materials of a column which enter into the TCD and contaminate it. In this case, you have to make the thermal cleaning which is known also as “bakeout”. For the bakeout, make sure that there are no leakages of carrier gas or contaminations of each inlet and detector.

1. Thermal cleaning procedure for a TCD

ⓐ Turn off the detector. Press【OFF】on the filament item.

ⓑ Disconnect the column from the detector and close the detector fitting.

ⓒ Flow reference and makeup gas at 20~ 30mL/min.

ⓓ Set temperature of the detector to 400℃ and maintain the temperature during some hours.

ⓔ Decrease temperature slowly up to room temperature while maintaining the gas flow.

For the TCD, if a filament is cut, you have to replace the whole TCD block. If you can find “OVER CURRENT” when using the TCD, firstly make the value of “SENSE” be smaller than the current value. If “OVER CURRENT” keeps on happening in spite of the smaller values, you have to check both the board and the cell. If “OVER CURRENT” is not shown when decreasing the value of【TCD SENSE】, you have to adjust only VR2 of the board. If the filament of the TCD is cut, “OVER CURRENT” is generally shown on the detector’s screen. In this case, it happens whatever the value of 【TCD SENSE】is. For this, just measure the resistance of the TCD cell. Here, you can know the method which tells how to measure and check the TCD status.

(1) Measuring a cell resistance

① Turn off the TCD and do not flow gases.

② Switch off power of the euipment.

③ Disconncet the line which is connected between the TCD and the TCD board.

④ Select a proper voltmeter which can measure resistance and adjust it to measure resistance (Ω).

⑤ Measure resistances of each line. You have to meausre them when the temperature of the detector is room temperaure. This is because the resistance changes according to temperature.

⑥ If anything is measured as “0” among the above values, it means that a filament is cut. In this case, replace the TCD cell. Refer to the “TCD” part of the chapter 11 for replacing the TCD cell.

⑦ When the measured value is greatly differ with the above value, be ready to replace it beacasue its feasures are changed.

(2) Adjustment of the VR of the board

There are three volume resistances(VR101) in the TCD board.

♠ VR101 – This resistance is used to increase and decrease signal values of a detector. In the default, the signal values are set between 20,000 and 30,000 at TCD SENSE 7 (Based on N_2).